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Clinical Technique/Case Report

Ceramic Fragments and Metal-free Full Crowns: A Conservative Esthetic Option for Closing Diastemas and Rehabilitating Smiles

ME Miranda • KA Olivieri • FJ Rigolin
RT Basting

Clinical Relevance

The reestablishment of the anterior teeth's esthetic harmony can be obtained with excellent clinical results by using the same porcelain for different tooth preparations, such as for laminate veneers and full metal-free crowns.

SUMMARY

Dental ceramics make it possible to restore anterior teeth that have been esthetically compromised, presenting a high resistance to wear, biocompatibility, color stability, and low thermal conductivity. The development of different types of ceramic and techniques for adhesive cementation have made it possible to produce more conservative restorations without involving the healthy dental structure and with minimally invasive preparation, such as the bonding of ceramic fragments. The purpose of this article is to describe a clinical case in which diastemas were closed by using nanofluorapatite ceramic (e.max Ceram, Ivoclar-Vivadent) fragments on teeth 7 and 10 with minimal tooth preparation and metal-free ceramic crowns (e-max Ceram) reinforced with zirconia copings through a computer-aided design/computer-aided manufacturing system (Lava, 3M-ESPE) on teeth 8 and 9.

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INTRODUCTION

A harmonious and attractive smile gives back a person's self-esteem and allows for a more favorable first impression. The smile is a person's most important facial expression, and the anterior teeth are the most direct complement to this harmony.[1]

The advent of new ceramic materials has led to a greater demand for their use as they enhance biometric qualities such as enamel structure, biocompatibility, longevity, mechanical strength, and chemical stability.[2] In some situations where the dental structure is absent or has little coronal structure remaining, it may be necessary to recommend a crown. One can consider metal-free crowns to obtain a better esthetic, using reinforced ceramic (with alumina, zirconia, or lithium disilicate, among others), which provides more mechanical strength.[3]

Laminate veneers are also commonly suggested for reestablishing the cosmetic shape of the teeth, to close diastemas and correct dental alignment, or to produce small alterations in color.[4] The use of laminate veneers was made possible once a technique was developed to treat ceramic with hydrofluoric acid, in combination with the application of a bonding agent (silane), to obtain an adhesive interface with resin cements[5] after conservative tooth preparation.[6] However, in situations where the teeth are healthy there is no need to carry out dental preparation. There is only a need to correct dental alignment or to correct the shape, form, or harmony of the smile when the person has diastemas. In these cases, refractory feldspathic porcelain or pressed ceramics are used to build ceramic fragments. These fragments, by way of an adhesive cementation technique, are bonded to the dental structure through resin cementation systems, allowing such fragments to resist fracture.[7]

However, in some clinical situations it is necessary to use different ceramic restorations on the same person, such as fragments, laminate veneers, and full crowns. Use of different types of restorations can present different degrees of translucency and opacity and may compromise the esthetic results of the ceramics.[8]

Thus, the objective of this article is to present a clinical case in which ceramic fragments and metal-free full crowns were used to close diastemas and reestablish the shape and esthetics of the upper anterior teeth with the same type of ceramic (e-max Ceram, Ivoclar-Vivadent, Schaan, Liechtenstein).

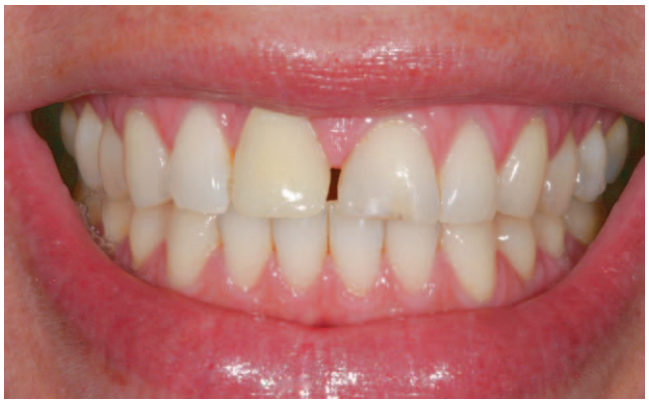

Figure 1. *Anterior teeth, frontal view.*

CASE REPORT

A 38-year-old woman presented with a chief complaint of diastemas between her upper central incisors and between the central incisor and the lateral incisor on the right side (Figure 1). The patient stated that she had had a dental implant in the right central incisor region and a ceramic crown made approximately 15 years ago. The left central incisor presented a class IV resin restoration with staining, loss of translucency, wear, and the presence of microinfiltration. The patient wished to improve the esthetics of her anterior teeth and close the diastemas.

Good oral hygiene was observed during the clinical exam. Periodontal examination revealed no gingival inflammation or bleeding during probing. The patient's medical history presented no contraindications to dental treatment.

After the clinical examination, impressions were made of both arches using stock trays and irreversible hydrocolloid (Hidrogun, Zhermack, Badia Polesine, Italy) and poured in type IV cast stone (Kromotypo4, Lascod, Firenze, Italy).

The upper cast was used for diagnostic wax-up and then duplicated. The diagnostic wax-up and the duplicate cast were presented to the patient, and the treatment plan was discussed. It included two metal-free full crowns in ceramic reinforced by zirconia copings for the upper central incisors and two ceramic fragments with a nanofluorapatite ceramic base for the upper lateral incisors.

An impression with condensation silicone material (Speedex, Coltene-Whaledent, Altstatten, Switzerland) was obtained using an aluminum impression tray from the duplicate cast of the diagnostic wax-up. This stone cast was later used to simulate the final result of the proposed treatment using self-

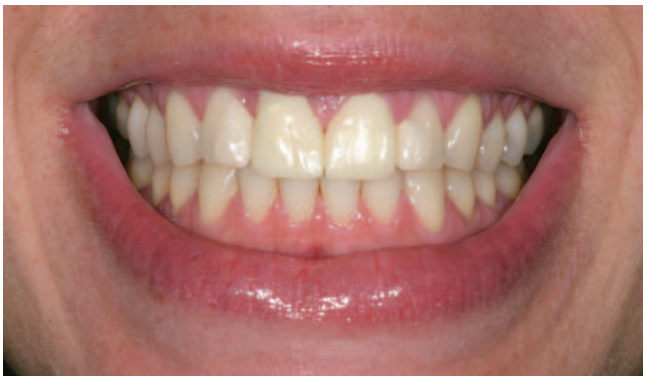

Figure 2. *Mock-up of the patient's mouth using self-polymerizing acrylic resin.*

polymerizing acrylic resin in color 62 (Snap Parkell, Edgewood, NY, USA) (Figure 2). After the patient approved the esthetic result, the teeth were prepared and the impression was made using a vinyl polysiloxane material (Elite HD, Zhermack) (Figure 3). To improve the esthetic condition, a computer-aided design/computer-aided manufacturing system was used to fabricate the coping for the central incisors using zirconia (Lava System, 3M-Espe, St Paul, MN, USA). The Ceramic was then fired over the copings to make the crowns (e.max Ceram, Ivoclar-Vivadent, Schaan, Liechtenstein). The ceramic fragments were made with the same nanofluorapatite ceramic base (e.max Ceram, Ivoclar-Vivadent) (Figure 4).

For final cementation, self-conditioning resin cement (Rely X U100, 3M-Espe) was used for the metal-free crowns in the upper central incisors under rubber dam isolation.

The ceramic fragments were conditioned with 9.5% hydrofluoric acid (DMG, Hamburg, Germany), two layers of silane were applied (Monobond-S,

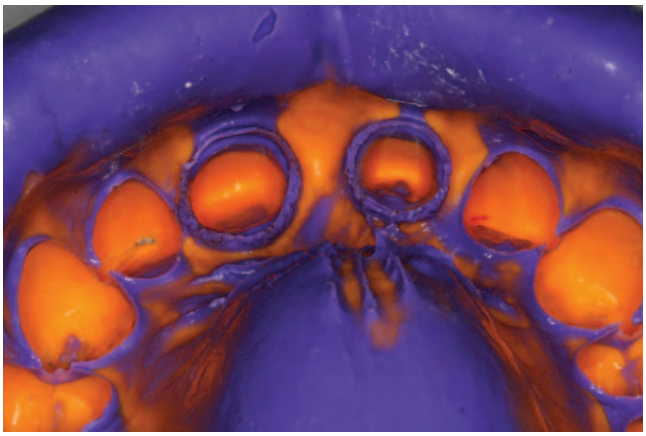

Figure 3. *Impression made using addition type silicone.*

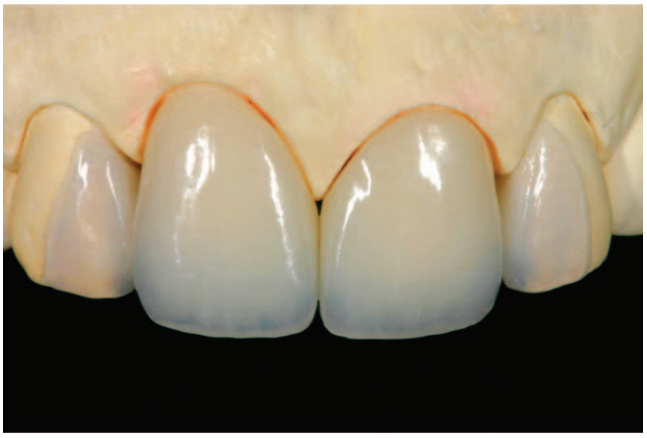

Figure 4. *Anterior teeth: ceramic fragments on teeth 7 and 10 and metal-free crowns on teeth 8 and 9.*

Ivoclar-Vivadent), and finally the adhesive application (Bond, 3M-ESPE) was carried out. On teeth 7 and 10, conditioning with phosphoric acid at 37.0% (Bisco Inc, Schaumburg, IL, USA) was performed and, after abundant water rinsing, an enamel adhesive was applied (Bond, 3M-ESPE). The cementation was done with a resin cement's base paste (Variolink II, Ivoclar-Vivadent) which was photoactivated for 40 seconds (Figure 5). The final presentation can be seen in Figures 6 through 8.

DISCUSSION

For a smile to be considered esthetically attractive, there must be a harmonious balance between shade, shape, and texture. With the objective of correcting a lack of esthetic harmony in the smile, such as with conditions like diastemas, peg teeth, and alterations in color and tooth size, ceramic restorations have frequently been used.[9]

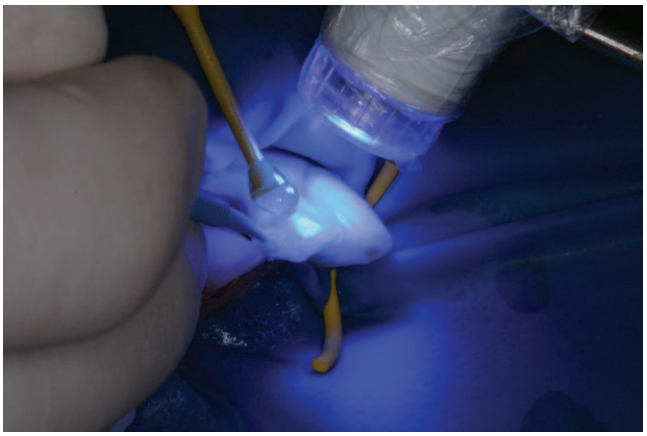

Figure 5. *The resin cement paste after photoactivation on tooth 7.*

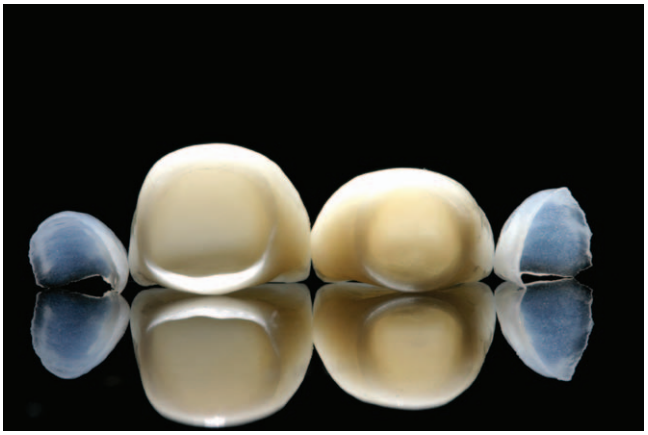

Figure 6. *The translucence of the ceramic fragments and the metal-free crowns.*

Composite resins were traditionally used to correct and close diastemas and to improve teeth with shade alterations. However, because of their low clinical longevity and susceptibility to pigmentation problems and marginal fractures, professionals have opted for laminate veneers and ceramic fragments.[10,11] Ceramic fragments present a low occurrence of loss of adhesion and fractures[10,11] and possess a mechanical strength similar to that of the natural tooth.[12] Because of this, we decided to make ceramic fragments for teeth 7 and 10 so as to preserve the remaining dental structure.

Piemjai and Arksornnukit[13] measured the resistance to compression of laminate veneers of 0.5 and 1.0 mm thickness when bonded to the enamel and to the dentin using different resin cements. They observed similar results between the self-polymerizing and photoactivated cements when cemented

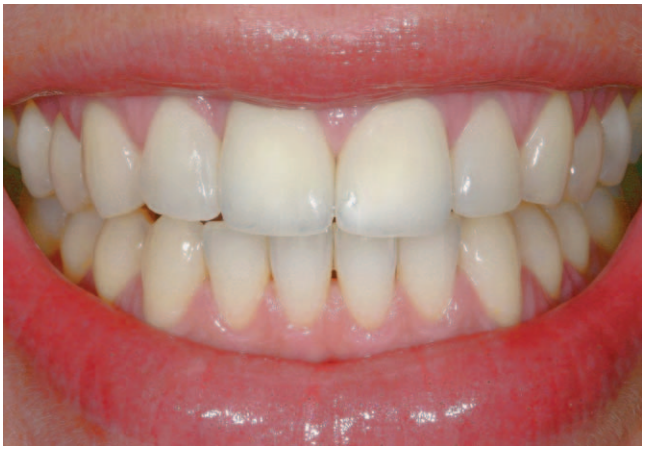

Figure 7. *Anterior teeth after treatment.*

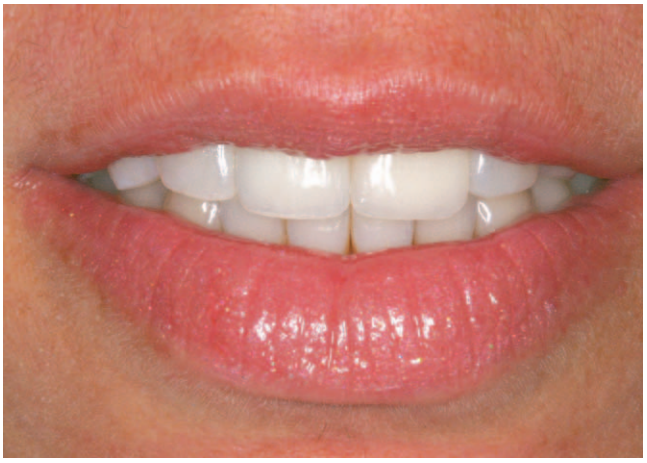

Figure 8. *The final presentation, a natural smile.*

in enamel. In this case, we opted to use a photoactivated cementation system, which has the advantages of greater clinical longevity, greater bonding strength due to the substrate being enamel, and non-alteration of cement color over time; in addition, because the ceramic fragments are very thin (0.3mm), they allow for adequate transmission of light and polymerization of the resin cement.[13]

For the upper central incisors, we decided to make metal-free crowns because of the implant present in the region of tooth 8 and because there was little remaining coronal structure in tooth 9. The advantage of crowns made with zirconia copings by the CAD/CAM system is that they have good mechanical strength and are more opaque; because of this, they are recommended for teeth that have color alterations or a metallic substrate.[14] However, because they are acid resistant, it is not possible to use adhesive cementation. Thus, we decided to use a self-adhesive cement, taking into consideration the fewer number of clinical steps for this procedure. Moreover, this cement combines with the hydroxyapatite of the dental substrate (for tooth 9) which does not promote the formation of a hybrid layer, and provides adequate bonding strength to the zirconia infrastructure.[15]

The use of different ceramic systems in an extensive restorative treatment can compromise the final result in terms of the ceramic shade and translucency. Because of this, in this clinical case we opted to use the same ceramic (e-max Ceram) for the fragments (teeth 7 and 10) and for the covering material in the zirconia copings (teeth 8 and 9), thereby gaining a very satisfactory esthetic result.

CONCLUSION

The demand for a beautiful smile is a reality, and the use of ceramic fragments with a nanofluorapatite ceramic base and full crowns in ceramic reinforced with zirconia can restore the esthetics via a very conservative and minimally invasive restorative treatment.

Acknowledgement

Our appreciation to CDT Cristiano Soares for his meticulous work making the ceramic restorations.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 31 August 2012)

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Clinical Research

Longevity of Direct Restorations in Stress-Bearing Posterior Cavities: A Retrospective Study

Y-J Rho • C Namgung • B-H Jin
B-S Lim • B-H Cho

Clinical Relevance

In posterior stress-bearing occlusal cavities, the longevity of resin composite restorations (RCs) was lower than amalgam restorations, while the clinical performance of the restorations in use was not different. RCs must be observed with periodic follow-ups for early detection and timely repair of failures.

SUMMARY

The aims of this retrospective clinical study were to compare the longevities of direct posterior amalgam restorations (AMs) and resin composite restorations (RCs) that were subjected to occlusal stresses and to investigate variables predictive of their outcome. A total of 269 AMs and RCs filled in Class I and II cavities of posterior teeth were evaluated with Kaplan-Meier survival estimator and multivariate Cox proportional hazard model. Seventy-one retreated restorations were reviewed from dental records. The other 198 restorations still in use were evaluated according to modified US Public Health Service (USPHS) criteria by two investigators. The longevity of RCs was significantly lower than that of AMs (AM = 8.7 years and RC = 5.0 years, $p<0.05$), especially in molars. The prognostic variables, such as age, restorative material, tooth type, operator group, diagnosis, cavity classification, and gender, affected the longevity of the restorations (multivariate Cox regression analysis, $p<0.05$). However, among the restorations working in oral cavities, their clinical performance evaluated with modified USPHS criteria showed no statistical difference between

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both restoratives. In contrast to the short longevity of RCs relative to AMs, the clinical performance of RCs working in oral cavities was observed to be not different from that of AMs. This suggests that once a RC starts to fail, it happens in a rapid progression. As posterior esthetic restorations, RCs must be observed carefully with periodic follow-ups for early detection and timely repair of failures.

INTRODUCTION

Amalgam and composite resin are the most widely used direct filling materials in posterior stress-bearing areas.[1,2] For posterior stress-bearing occlusal surfaces, especially for Class II restorations, amalgam is still the most commonly used material in some countries.[1,3-4] However, the use of amalgam is declining gradually due to patients' esthetic demands and concern over the hazards of mercury.[3-5]

There has been controversy with the longevity of amalgam and composite resin as posterior restorations. Generally, in cross-sectional retrospective studies, amalgam restorations (AMs) exhibited better longevity than composite resin restorations (RCs).[1,6,7] A prospective randomized clinical trial also showed a higher seven-year survival rate in AMs than in RCs.[8] Moreover, even in the reports that showed no significant difference in the longevity of the two restoratives, the replacement rates for RCs were significantly higher than those for AMs.[9,10] However, unlike the results of other retrospective studies, a better 12-year survival rate and a comparable 10-year survival rate of RCs compared to AMs were also reported by a small group of well-motivated practitioners.[11-12] Manhart and others[13] also reported comparable annual failure rates between the two materials from meta-analysis of the survival rates of direct posterior restorations (amalgam 3.0%, composite resin 2.2%).

As reasons for amalgam replacement in the posterior area, poor margins and resulting secondary caries were ascribed to biting force and creep.[14,15] Low-frequency cyclic stresses caused by mastication and thermal changes during ingestion of hot and cold food induce creep.[16] When composite resin is used in a posterior stress-bearing area, it may also be subjected to the same situation. Cyclic loading was found to lead to significant decreases in fracture strength and the fatigue limit of the adhesive itself at the adhesive-dentin interface.[17] Therefore, whenever composite resin is used in a posterior stress-bearing area, the effect of the occlusal stresses being applied to the weakest adhesive-dentin interface should be considered with respect to the aging of the restorations.

Despite the improvement in composite resin materials and bonding techniques, composite resin is still used as a direct posterior filling material without any basis on scientific clinical evidence.[3,18] In fact, the use of composite resin to restore stress-bearing surfaces of molar and premolar teeth may still be controversial due to the individual practitioner's concerns over unpredictability, microleakage, unacceptable wear, postoperative sensitivity, time and technique sensitivity in moisture control and placement, and control of polymerization shrinkage stress.[1,3,19] Comparative data on the longevity of AMs and RCs as direct restorations under similar conditions are needed, especially in posterior stress-bearing areas.

The aims of this retrospective clinical study were to compare the longevities of direct posterior AMs and RCs that were subjected to occlusal stresses in stress-bearing areas and to investigate variables predictive of their outcome. For the purposes, the longevity of AMs and RCs that were placed into Class I and II cavities by multiple operators working in a dental school and their prognostic variables were evaluated retrospectively.

MATERIALS AND METHODS

This study is a part of the cross-sectional clinical study that was performed retrospectively in the Department of Conservative Dentistry, Seoul National University Dental Hospital, from July 6, 2009, to August 28, 2009. The project was approved by the Institutional Review Board of the Seoul National University Dental Hospital. In order to compare the longevity of direct AMs and RCs that were filled into the cavities under continuous occlusal forces, data on 269 AMs and RCs placed into Class I and Class II cavities of posterior teeth of 140 patients were selected and evaluated with survival analysis.

Selection criteria included the patients who had appointments during the study period and, among them, who had restorations placed in the department. Prior to a patient's visit, information on the patient and treatment was collected from dental records. Patient information included gender, age, and medical and dental history. From the records, old AMs and RCs that had been directly placed into Class I and Class II cavities of premolars and molars were selected. In order to exclude restorations used for an interim purpose, those restored within one year were not included in this study. Restorative

material, cavity classification, tooth type, reason for treatment, and date of treatment were recorded as treatment information.

If there was a record on retreatment or subsequent treatment, such as extraction, endodontic treatment, and prosthodontic treatment that could affect the integrity of the restoration, the date and reason for the subsequent treatment were recorded. The restoration was regarded as an event case, and its longevity was determined as the period from the initial treatment to the subsequent treatment. If there was no record of retreatment and subsequent treatment, the patient was clinically evaluated under informed consent before the appointed treatment of the visit. When a restoration was still in function and its characteristics were consistent with the record, the restoration was evaluated by two investigators according to modified US Public Health Service (USPHS) criteria (Table 1). If there was a disagreement between the investigators, it was resolved by consensus. When the restoration was rated as Alpha or Bravo, the restoration was considered censored. Its censored life span was determined as the period from the initial treatment to the date of examination. Related information was also collected from the records. When the restoration was still in the oral cavity but rated as "clinically unacceptable" Charlie in a single criterion of the modified USPHS criteria, it was recommended for retreatment and regarded as a failure. The longevity of those restorations were also calculated from the initial treatment to the date of examination (Table 2). In cases where it was unclear whether there had been subsequent treatment on the existing restoration or whether the characteristics of the restoration agreed with the record, the cases were excluded from the study.

Statistical analyses were performed with SPSS version 18.0 (SPSS Inc, Chicago, IL, USA). To evaluate the longevity of the AMs and RCs, survival analysis was performed using Kaplan-Meier survival estimates. The effect of the assumed prognostic variables on the survival of the two restorative materials was analyzed using a multivariate Cox proportional hazard model by simultaneously entering all the variables. Reasons for the failures of restorations were compared based on the records and clinical examination results. Finally, using chi-square test/Fisher's exact test between the restorations rated as clinically acceptable (Alpha and Bravo grades) and those rated as unacceptable (Charlie grade) in each USPHS criterion, the clinical performance of both restorations remaining in oral cavities were compared.

RESULTS

Among 374 Class I and Class II posterior AMs and RCs, 105 (28.1%) restorations were excluded from the study due to disagreement between their characteristics and their records. A total of 269 (71.9%) restorations that were placed between 1986 and 2008 in 140 patients were included in this study. Fifty-nine patients were male, and the remaining 81 patients were female. The ages of the patients at treatment were 10-78 years with a mean age (±SD) of 46.9 (±16.0) years and those at evaluation were 15-81 years with a mean age of 53.4 (±16.7) years. There were 131 AMs and 138 RCs (Table 2). Systemic diseases were found in 52 patients (37.1%) with hypertension (24 patients), diabetes (seven patients), and hepatitis (seven patients) being the most prevalent. Patients with disabilities and past dental history related to difficulties in maintaining general oral hygiene, such as multiple caries or xerostomia, were excluded from the study. The number of AMs and RCs delivered in each year and the proportion of each restoration in each year are presented in Figure 1. According to the data, the first direct RC in a stress-bearing cavity of posterior tooth was observed in 1996, and the number and proportion of RCs gradually increased. After 2003, the proportion of RCs exceeded that of AMs.

In total, the median survival times of AMs and RCs in the occlusal stress-bearing cavities of posterior teeth were 8.7 and 5.0 years, respectively, and their survival estimates were significantly different (log rank test, $p<0.05$; Table 3 and Figure 2a). With respect to the classifications, the median survival times of Class I and Class II AMs were 10.0 years and 6.9 years, respectively (log rank test, $p<0.05$; Table 3 and Figure 2b), whereas Class I and Class II RCs were not statistically different (median survival times of 3.3 and 5.4 years, respectively; Table 3 and Figure 2c). With respect to the materials, Class I restorations showed significantly different survival estimates between AMs and RCs (log rank test, $p<0.05$; Table 3 and Figure 2d). However, Class II restorations did not exhibit a difference (Table 3 and Figure 2e). Compared to the AMs that were statistically not different, RCs were significantly different between premolars and molars (Breslow test, $p<0.05$; Table 3). There were no significant differences in the survival estimates of both materials between with and without systemic diseases (Breslow test, $p<0.05$; Table 3). When the patients

Table 1: *The Modified US Public Health Service Criteria Used in the Study*

Category	Rating		Criteria
	Success	Failure	
Retention	Alpha		Present
	Bravo		Partial loss
		Charlie	Absent
Color match	Alpha		No mismatch to the adjacent tooth structure
	Bravo		Slight mismatch but clinically acceptable
		Charlie	Esthetically unacceptable mismatch
Marginal discoloration	Alpha		No discoloration on the margin
	Bravo		Superficial discoloration on the margin
		Charlie	Deep discoloration penetrating in a pulpal direction
Secondary caries	Alpha		No caries present
		Charlie	Caries present
Wear (anatomic form)	Alpha		Anatomy resembles the original restoration
	Bravo		Anatomy exhibits a change in contour but does not require replacement
		Charlie	Excessive wear with dentin exposure requiring replacement
Marginal adaptation	Alpha		Continuity at the margin (no ledge or ditch)
	Bravo		Slight discontinuity detectable with an explorer but does not require replacement
		Charlie	Marginal ledge or crevice requiring replacement
Postoperative sensitivity	Alpha		Absent
		Charlie	Present

had systemic diseases, there was no difference in the survival estimates between both materials. However, among the patients without systemic diseases, AMs exhibited a longer median survival time than RCs (log rank test, $p<0.05$; Table 3). On the other hand, there were significant differences between AMs and RCs with respect to operator, diagnosis, age-groups, gender, and the location in maxilla or mandible (Table 3).

According to the multivariate Cox proportional hazard model and the Wald statistics, the age-group, restorative material, tooth type, operator group, diagnosis, cavity classification, and gender affected the lifetime of the restorations in a descending order ($p<0.05$; Table 4). The location of teeth in the maxilla or mandible and the presence or absence of systemic diseases did not exhibit significant influence on the overall survivals. Among the age-groups, teenagers and those in their 70s had higher risks than the other age-groups, except those in their 40s. With AMs as reference, the relative risk of RCs significantly increased 2.28 times. The relative risk of molars increased 2.45 times compared to premolars. In comparison to Class I restorations, the relative risk of Class II restorations increased 1.63 times. In comparison to males, the relative risk of females was 0.65 times that of males. Among the operator groups, the student group exhibited significantly lower risks than the professor and the resident groups, but the relative risks of the professor group and the resident group were statistically not different. Among the diagnostic categories, the restorations placed due to pulpal problems exhibited the highest risks compared to those restored due to other reasons, but the relative risks of the restorations due to primary reasons and replacements were statistically not different.

Regardless of the longevity of the restorations, from the treatment records of the retreated restorations and the clinical evaluation of the restorations working in oral cavities according to the USPHS criteria, 73 (55.7%) of the 131 AMs were deemed as failures, while 58 (42.1%) of 138 RCs were determined as failures (Table 2). Forty-four AMs and 27

Table 2: *Distribution of Amalgam and Resin Composite Restorations Included in This Retrospective Cross-Sectional Clinical Study for the Survival Analysis of Direct Posterior Class I and II Restorations Subjected to Occlusal Stress*

Restorations	Status	No. (%)			Event
		Amalgam	Composite	Sum	
Replaced		44 (33.6)	27 (19.6)	71 (26.4)	Event
Survived in the mouth	Failure (Charlie)[a]	29 (22.1)	31 (22.5)	60 (22.3)	Event
	Clinically acceptable (Alpha or Bravo)[a]	58 (44.3)	80 (58.0)	138 (51.3)	Censored
Total		131 (100)	138 (100.1)	269 (100)	

[a] *If the restoration was rated a clinically unacceptable grade of Charlie in any one of the seven criteria, the restorations were determined as failures and classified to an event case. The restorations rated as Alpha or Bravo were considered as clinically acceptable and classified to be censored.*

RCs had records for replacement. Among them, the reasons for replacement were recorded in 33 AMs and 21 RCs. In cases of replaced restorations, the most common reasons for failures in AMs were loss of the restoration (36.4%), fracture of the restoration (27.3%), and secondary caries (21.2%). In RCs, they were secondary caries (38.1%), loss of retention (23.8%), and fracture of the restoration (14.3%). However, in the failure cases of the 29 AMs and 31 RCs that were retained in oral cavities but determined as failures due to the Charlie grade even in a criterion of the modified USPHS criteria, the most common failure reason was secondary caries for both restorative materials. Ill-fitting margins and a loss of retention followed for both restoratives. Some of them were rated as Charlie in more than one criterion. Among 87 AMs and 111 RCs remaining in oral cavities, the clinical performance of the restorations that were evaluated using USPHS criteria was statistically not different between both restorations in all the criteria (Table 5).

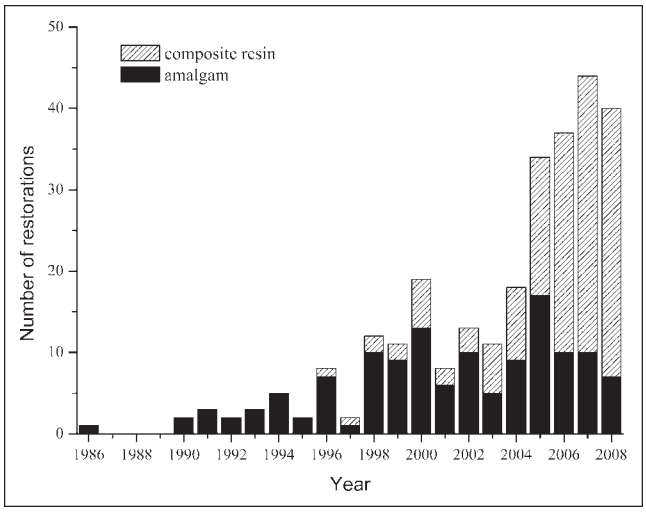

Figure 1. *The number of amalgam and resin composite restorations delivered in each year.*

DISCUSSION

In this study, the longevity of AMs was significantly greater than that of RCs (Table 3). Generally, AMs had better longevity and required less repair or replacement than RCs.[6-8,20,21] Burke and others[6] and Mjör and others[20] reported the median survival times of Class I AMs, Class II AMs, Class I RCs, and Class II RCs as 7.4~10, 6.6~11, 3.3~6, and 4.6~6 years, respectively. Forss and others[7] reported 12 years for AMs and 5 years for RCs. Although this study was performed in a department of a university-based hospital, the longevity data of this study were quite similar to the values reported in those studies based on the general practice settings. The longevity data of this study were also shorter than those obtained from clinical studies practiced by a single dentist or a small group of highly motivated practitioners.[11,21] The reasons for the relatively short longevity of the restorations included in this study may be explained from two aspects. First, most retrospective studies collected data from responses to questionnaires and regarded all the remaining restorations as censored. However, in this study, those restorations rated as Charlie even in one criterion of the USPHS criteria were regarded as failures. The strict criteria to determine the survival may have reduced the longevity values of this study. Second, the fact that a high proportion of molar teeth were included in our study also decreased the longevity of posterior direct restorations (19 premolar and 112 molar in AMs; 49 premolar and 89 molar in RCs). In most other studies, they did not separate the longevity values into premolars and molars.[6,7,20]

Table 3: *Median Survival Times of Amalgam and Resin Composite Restorations According to Prognostic Variables**

Prognostic Variables	Amalgam	Composite
Material	8.7 (7.8~9.6)^{A}	5.0 (3.2~6.8)^{B}
Cavity classification		
I	10.0 (7.9~15.5)^{aA}	3.3 (2.5~10.5)^{aB}
II	6.9 (2.9~12.7)^{bA}	5.4 (2.5~10.4)^{aA}
Tooth type		
Premolar	11.2 (10.8~12.7)^{aA}	8.7 (3.3~12.5)^{aA}
Molar	8.3 (3.7~13.7)^{aA}	3.3 (2.2~10.4)^{bB}
Systemic disease		
Absent	8.7 (4.0~13.7)^{aA}	5.2 (2.7~10.4)^{aB}
Present	6.9 (2.8~11.2)^{aA}	3.2 (2.4~11.0)^{aA}
Operator		
Professor	8.1 (3.9~13.7)^{bA}	5.0 (2.5~12.5)^{aA}
Resident	8.3 (3.7~12.7)^{bA}	3.3 (2.3~10.4)^{aB}
Student	13.3 (10.0~15.5)^{aA}	NA^{A}
Diagnosis†		
Primary reason	9.1 (3.9~13.7)^{aA}	5.0 (2.5~10.4)^{aB}
Replacement	8.3 (4.0~8.7)^{aA}	NA^{aA}‡
Pulp pathosis	NA^{bA}	NA^{aA}
Gender		
Male	8.0 (2.8~13.7)^{aA}	3.7 (2.2~10.5)^{aA}
Female	8.9 (4.0~13.3)^{aA}	6.5 (2.5~10.4)^{aB}
Arch		
Upper	8.4 (3.9~13.3)^{aA}	3.6 (2.7~10.4)^{aB}
Lower	10.0 (3.9~13.7)^{aA}	5.4 (2.5~11.0)^{aB}
Age		
10	10.0 (2.9~NA) ^{abA}	1.2 (1.1~1.4)^{bB}
20	10.8 (2.1~12.9) ^{abA}	8.7 (NA~NA)^{aA}
30	12.7 (9.1~14.3)^{aA}	10.4 (3.3~10.5)^{aA}
40	8.0 (5.1~8.7) ^{abA}	2.7 (2.2~3.3)^{aB}
50	7.9 (3.5~17.8) ^{bA}	4.4 (2.8~5.2)^{aA}
60	11.2 (4.0~13.7) ^{abA}	5.4 (2.2~12.5)^{aA}
70	0.4 (NA~NA)^{cA}	3.0 (2.3~NA)^{abB}

** Median survival times are presented in years, and the numbers in the parentheses are the survival times in years at 25% and 75%. Different small superscript letters indicate a significant difference in the longevity within the column, and different capital superscript letters indicate a significant difference in the longevity between the two restoratives within the row.*
† Treatment reasons (diagnoses) were divided into three categories: primary reasons, replacements, and pulpal problems. The primary lesions, such as caries, attrition, abrasion, erosion, tooth fracture, diastema, and esthetic problems, were grouped as primary reasons. Filling body fracture, partial retention loss, and secondary caries were grouped as replacements. Pulpal pathosis and hypersensitivity were included as pulpal problems.
‡ NA means that the value of survival time is not available due to lack of event cases.

However, in our study, a high proportion of molar teeth that had shorter lifetimes than premolars decreased the longevity of AMs and RCs compared to other studies.

Many factors, such as operators with various clinical experience, tooth type, location of the tooth, size of the restoration, and age, may affect the longevity of the restorations.[6,13] Compared to Class II AMs, Class I AMs were reported to have a longer survival time and lower failure rate.[22] Our study also showed that the longevity for Class I AMs was significantly longer than that of Class II AMs. However, there was no significant difference between the longevities of Class I and Class II RCs. This result is contrary to the earlier study, which reported higher failure rates in Class II RCs than in Class I RCs.[23] However, it corresponds to the fact that some studies report shorter longevities for Class I restorations (three to four years) than for Class II restorations (four to seven years).[20,22] Compared to Class I AMs, the relatively short longevity of Class I RCs may result from the drawbacks of adhesion, such as the weakest link of bonding, high configuration factor of the box-shaped cavity, poor resistance to polymerization shrinkage stress, and low-grade continuous occlusal stresses.[17,24,25] The results suggest that the longevity of the posterior direct RCs under occlusal stresses may be determined by the adhesive as the weakest link rather than the restorative material and cavity classification, and the suggestion needs to be further investigated.

In the multivariate analysis, the relative risk of failure for molars was 2.45 times higher than premolars (Table 4). There was no difference in the longevity of AMs in premolars and molars. However, RCs of premolars exhibited significantly higher longevity than those of molars (Breslow test, $p<0.05$; Table 3). Previous studies showed that adhesive restorations were more successful in premolars and in the non-stress-bearing areas than molars and stress-bearing areas.[20,23] Simecek and others[10] also reported a higher incidence of replacement for both AMs and RCs in molars than premolars. In our study, with respect to the materials, there was no significant difference in premolars. However, the RCs in molars exhibited less than half the median survival times of the AMs in molars (AMs 8.7 years, RCs 3.3 years; Table 3). In the multivariate analysis, the restorative material and tooth type contributed the most to longevity, except the age-groups (Wald statistics; Table 4). Contrary to the AMs, the longevity of the RCs in

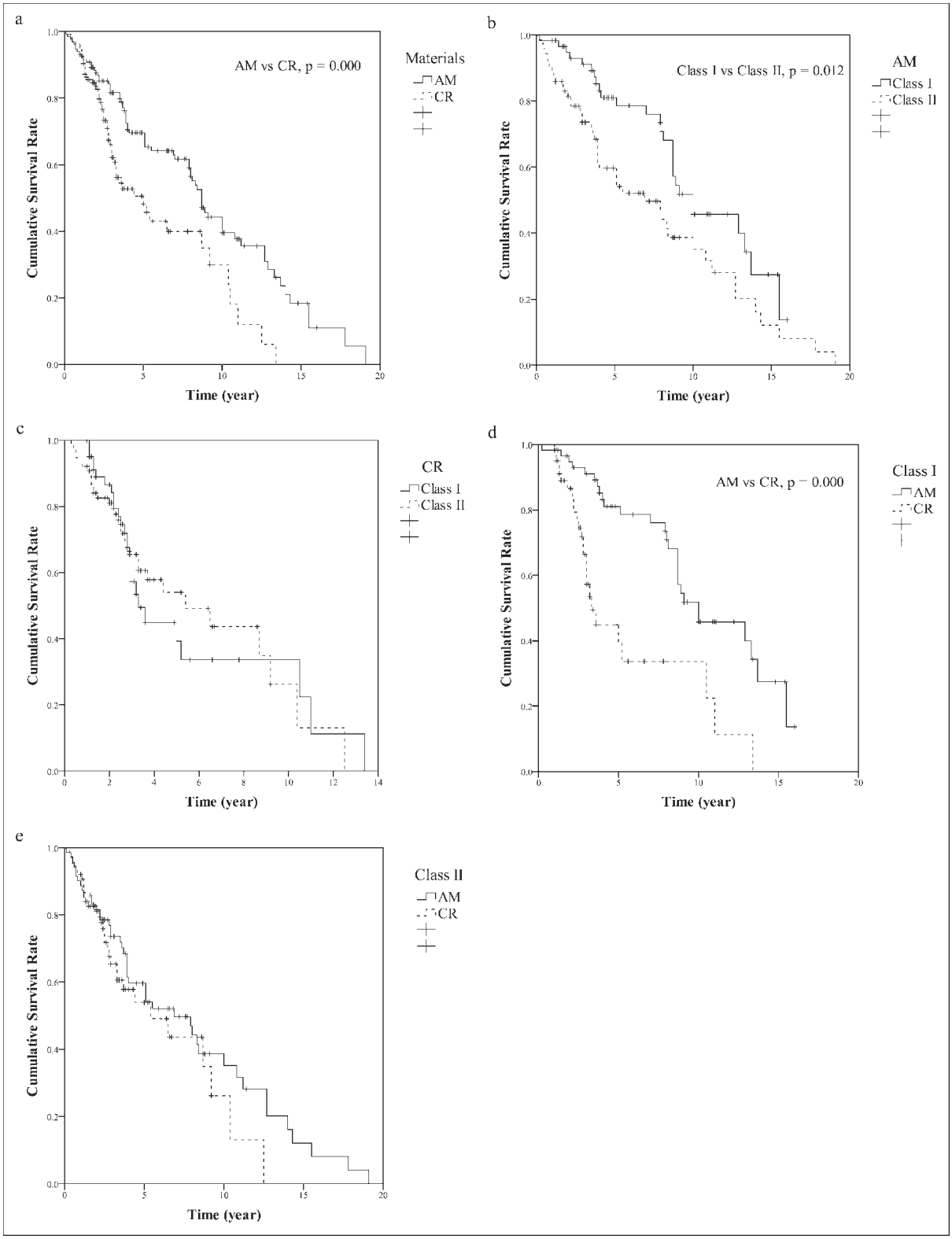

Figure 2. *Cumulative survival estimates of direct posterior Class I and Class II amalgam restorations (AMs) and resin composite restorations (RCs). (a): AMs and RCs. AMs exhibited significantly higher survival estimate than RCs (log rank test, $p<0.05$). (b): AMs. There was a significant difference between Class I and Class II restorations (log rank test, $p<0.05$). (c): RCs. There was no significant difference between Class I and Class II restorations. (d): Class I restorations. There was a significant difference between AMs and RCs (log rank test, $p<0.05$). (e): Class II restorations. There was no significant difference between AMs and RCs.*

Table 4: *Relative Risk of Failure in Posterior Direct Amalgam and Resin Composite Restorations According to Prognostic Variables*[a]

Variables	p-Value	Relative Risk	95% CI		Wald Statistics
			Lower	Upper	
Age	0.000				25.153
10		1.00			
20	0.014	0.33	0.14	0.80	
30	0.000	0.14	0.06	0.34	
40	0.106	0.53	0.25	1.14	
50	0.010	0.37	0.17	0.79	
60	0.002	0.26	0.11	0.62	
70	0.225	0.46	0.13	1.61	
Material	0.000				13.696
Amalgam		1.00			
Composite		2.28	1.47	3.53	
Tooth type	0.001				11.885
Premolar		1.00			
Molar		2.45	1.47	3.53	
Operator	0.005				10.704
Professor		1.00			
Resident	0.140	1.39	0.90	2.14	
Student	0.014	0.36	0.16	0.81	
Diagnosis	0.010				9.284
Primary reason		1.00			
Replacement	0.416	0.75	0.37	1.51	
Pulp problem	0.005	8.69	1.91	39.51	
Cavity classification	0.023				5.149
I		1.00			
II		1.63	1.07	2.49	
Gender	0.030				4.681
Male		1.00			
Female		0.65	0.44	0.96	
Arch	0.140				2.180
Maxilla		1.00			
Mandible		0.75	0.52	1.10	
Systemic disease	0.764				0.090
Absent		1.00			
Present		1.07	0.70	1.63	

Abbreviation: CI, confidence interval.
[a] *The data in this table were obtained through the multivariate Cox proportional hazard model. According to the Wald statistics, the variables affecting the lifetime of the restorations were presented in a descending order. The arch and the systemic diseases did not exhibit significant influence on the survivals ($p > 0.05$). In each variable, the relative risk of each group showed the ratio of the risk compared to the first group with a relative risk of 1.00.*

molar teeth was significantly lower than in premolar teeth. Due to advances in composite materials, bonding techniques, and operator experience, there is now a decreasing trend in the failure rate of RCs.[3,4,18] However, as seen from the result of this study, the longevity of RCs was significantly lower than that of AMs, especially in molars. The longevity of the restoration might have been affected by the tooth type due to reasons such as the inherent difficulties of accessing molar teeth during treatment, the large size of the restorations, and the heavy occlusal forces. In spite of the significantly shorter longevity of RCs than AMs, the observation that the clinical performance of the restorations working in oral cavities showed no statistical difference between both restorative materials (Table 5) suggested the rapid progressing nature of the failure of RCs. Therefore, as posterior esthetic restorations, RCs must be observed carefully with periodic follow-ups for early detection of failures and for a timely repair procedure.

Among the systemic diseases, hypertension, diabetes, and hepatitis were the most prevalent ones. Systemic diseases were too diverse with small sample sizes to be evaluated for each disease. Therefore, systemic diseases were evaluated only for their presence or absence in this study. The presence of systemic diseases had no significant effect on the longevity of both the AMs and RCs (Tables 3 and 4). Only in the healthy patient group without systemic diseases did RCs have shorter median survival time than AMs (Table 3). The patient factors that raise the caries risk with respect to the longevity of restorations may be xerostomia, dietary habits, oral hygiene, oral flora, and root exposure through the effect of salivary secretion.[26] In this study, because patients with systemic diseases who may have issues with salivary secretion were excluded, the relative risk of the group was not different from the group without systemic diseases. Further studies are needed on the effect of the presence of systemic diseases on the longevities of restorations and the relationship between individual systemic disease and the longevity of restorations. By the same token, treatment reasons (diagnoses) as a prognostic variable were divided into three categories: primary reasons, replacements, and pulpal problems. The primary lesions, such as caries, attrition, abrasion, erosion, tooth fracture, diastema, and esthetic problems, were grouped as primary reasons. Restoration body fracture, partial retention loss, and secondary caries were grouped as replacements. Pulpal pathosis and hypersensitivity were included as pulpal problems. Although there was no

Table 5: *Comparison of the Clinical Performance Between the Restorations Filled With Amalgam and Composite Resin Evaluated on the Basis of the Ratings of the Modified US Public Health Service Criteria*

Criteria	Chi-Square Test/Fisher's Exact Test[a]			
	Total		Odds Ratio	
	χ^2	*p*	AM/RC	95% CI
Retention	0.160	0.824	0.837	0.350~2.001
Color match[b]	—	—	—	—
Marginal discoloration[b]	—	—	—	—
Secondary caries	2.007	0.180	0.623	0.323~1.202
Wear (anatomic form)	NA[c]	1.000[a]	0.804	0.158~4.089
Marginal adaptation	1.064	0.396	0.647	0.282~10485
Postoperative sensitivity	NA	1.000[a]	0.778	0.153~3.952

Abbreviations: AMs, amalgam restorations; CI, confidence interval; NA, not available; RCs, resin composite restorations.
[a] When the expected incidence in more than one cell was less than 5, the result of Fisher's exact test was selected.
[b] The statistical results for the criteria "color match" and "marginal discoloration" were not available because the data on the two criteria were obtainable for RCs but not for AMs.
[c] The χ^2 value and odds ratio were not calculated, as there was more than one cell showing no incidence in the 2 × 2 tables.

significant difference between the relative risks of the restorations due to primary reasons and replacements, the relative risk of the restorations delivered due to pulpal problems was significantly higher than the other reasons (Tables 3 and 4).

The reason for failure of the restorations were evaluated in two ways, that is, from the treatment records and from the clinical evaluations according to the modified USPHS criteria for retreated cases and restorations working in oral cavities. For the retreatment cases, the loss of retention, fracture of the restoration, and secondary caries were the reasons for replacement in AMs. Secondary caries was the most frequent reason for replacement of RCs, followed by the loss of retention and fracture of the restorations. For the restorations working in oral cavities, because the restoration was classified as failure when it was rated as Charlie in any one of the seven modified USPHS criteria, the failure reasons for each restoration could be numerous. The most frequent reason for a clinically unacceptable Charlie grade was secondary caries in both types of materials, followed by marginal adaptation and loss of retention. This was consistent with other studies, in which secondary caries and fracture of the restorations were the main reasons for the failure irrespective of the restorative materials.[6,20] In AMs, the restorations with poor marginal adaptation were more numerous than those with retention loss. However, in RCs, the restorations with poor marginal adaptation were less frequent than AMs. This result met well with the low marginal strength of dental amalgam. Occurrence of marginal discoloration as well as poor marginal adaptation in RCs can be attributed to the polymerization shrinkage of the resin composite itself, long-term degradation of adhesion, and accumulation of fatigue from continuous occlusive forces.[17,25] Although AMs exhibited poor marginal adaptation, they might have better longevity than RCs due to the increased marginal seal by corrosion products and creep mechanisms.[27,28]

This study had several limitations. This study was performed retrospectively on a limited number of patients who visited our department during a period of eight weeks. The sample size was relatively small compared to other studies in which several thousands of restorations were collected using questionnaires by mail.[6,7,20] Moreover, the treatment records did not contain all the necessary data, such as missing data on the reason for replacement and on the techniques and materials, such as adhesives and restoratives. This made a large portion of valuable information unavailable. These were definite shortcomings of this study compared to a prospective controlled study. On the contrary, this study, performed in a department of a university hospital, had several advantages. First, the restorations were delivered by operators with various experience, including students, residents, and professors. Hav-

ing various operators performing the restorations may have a definite advantage over a single dexterous operator or a small group of well-motivated practitioners in preventing biases.[11,12,21] Second, because the data were collected from patient records at a university hospital, treatment protocols were relatively standardized, and the longevity calculated from the record was accurate and reliable. Third, compared to the general practitioner–based retrospective study, nearly three-fourths of the restorations were evaluated clinically by two trained examiners according to the widely used criteria. The evaluation of the clinical status of the restorations must have been more consistent and reliable than other studies. Finally, in this study, because the restorations rated as clinically unacceptable Charlie even in one criterion were ethically recommended to be replaced, they were classified as event cases. As a result, the longevity of the restorations in this study was relatively short due to the strict criteria on the event case compared to the studies based on the responses of general practitioners to the requested questionnaires.

CONCLUSIONS

This study evaluated the effect of the variables that might be related especially to the occlusal stresses, such as material, cavity classification, tooth type, gender, arch, and age. The longevity of RCs was significantly lower than that of AMs, especially in molars. In spite of short longevity of RCs, the clinical performance of RCs working in oral cavities was not different from that of AMs. This suggests that once a RC starts to fail, it happens in a rapid progression. As posterior esthetic restorations, RCs must be carefully observed with periodic follow-ups for early detection and timely repair of failures.

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Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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Influence of Examiner Experience on Clinical Performance of Visual Inspection in Detecting and Assessing the Activity Status of Caries Lesions

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Clinical Relevance

The methods of caries activity assessment associated with the International Caries Detection and Assessment System have similar performance independent of the examiners' levels of experience. However, assessments based on the clinical features of the lesions are less time consuming.

SUMMARY

Our hypothesis was that a method of caries activity evaluation based on the clinical features of the lesions would be less time consuming but more influenced by the examiner's experience than the scoring system used in association with the International Caries Detection and Assessment System (ICDAS). Thus,

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the aim of this study was to evaluate the performance of three groups of examiners with different levels of experience using two different methods to assess the activity status of caries lesions by visual inspection. A cross-sectional study in a dental office setting was performed selecting 18 children, aged three to eight years, who had sought dental treatment at a dental school. Examinations to detect caries lesions were performed using visual inspection by six examiners with different levels of experience: two undergraduate dental students, two specialists in pediatric dentistry, and two graduate students. The examiners used ICDAS and two different methods to assess caries activity: using an additional score system or considering the examination of clinical features. Two benchmark examiners examined the children in a joint session, and their consensus was considered to be the reference standard. The sensitivity, specificity, and reproducibility were calculated for different thresholds: all, cavitated, and active caries lesions. Multilevel analyses were performed to compare the different methods and examiners. No differences were observed among the examiners, either in detecting all lesions and cavitated lesions or regarding the activity assessment. The methods of assessing activity status performed similarly, but the time spent on examinations was shorter for the method evaluating clinical features. In conclusion, the experience of examiners does not significantly influence the performance of visual inspection, and both methods of assessing activity status result in similar diagnostic accuracy.

INTRODUCTION

Visual examination is the most commonly used method for caries lesion detection because it is an easy technique that is routinely performed in clinical practice.[1] The method has demonstrated high specificity, and it is the only validated method used to assess the activity status of caries lesions.[1-3] However, it has presented low sensitivity and reproducibility,[1,4] the latter because of the subjectivity of the procedure.[5,6]

In this context, attempts to improve the accuracy of caries detection methods have been proposed,[7] such as the development of scoring systems. The International Caries Detection and Assessment System (ICDAS), based on visual inspection, allows for the accurate recording of the severity of carious lesions, from noncavitated stages to frank cavitation.[7] The system was developed for use in clinical research, in clinical practice, and for epidemiological purposes.[8] However, for caries activity assessment, ICDAS must be used in combination with other procedures.[7]

One of the adjunct methods for caries activity evaluation is based on the examiner's interpretation of the clinical features of the lesions.[9] For this approach, the examiner evaluates some clinical parameters that are more often associated with active or inactive caries lesions (clinical characteristics assessment [CCA]). Another option is the lesion activity assessment (LAA), which is used in association with ICDAS and is performed by assigning numerical values (points) to three clinical parameters: visual appearance, whether the lesion is in a plaque stagnation area, and surface texture. The sum of these three independent scores is then used to determine whether the lesion is active or arrested.[10]

These two methods are established on the basis of different aspects of cognitive processes involved in diagnostic reasoning in clinical settings. The first method, based on the evaluation of the clinical features of the lesions (CCA), represents a descriptive approach to the decision-making process. The examiner evaluates the clinical features of the lesions and mentally weights each characteristic to reach a decision about the lesion activity. On the other hand, the assignment of numerical values for different clinical parameters to reach a decision concerning caries lesion activity is part of the prescriptive theory. Predetermined values are attributed to each clinical parameter, and the sum of the values indicates whether the lesion is active or inactive.

The descriptive theories about decision making are closer to clinical reality because clinicians are more accustomed to drawing on past experience to reach a diagnosis.[11] Moreover, this process is less time consuming and more practical to perform as part of a daily clinical routine.[11,12] Nevertheless, this process is greatly influenced by the professional's experience. Novice clinicians use a hypothetic-deductive reasoning strategy to reach a decision. This is a stepwise process and therefore consumes more time. On the other hand, experts frequently use cognitive shortcuts to make diagnoses, a process called heuristics.[11,12] Another limitation is that unchecked heuristic reasoning can lead to cognitive bias and diagnostic errors.[11,13]

Our working hypothesis was that the method based on CCA of lesions would be less time consuming but more greatly influenced by the examiner's experience regarding its reliability and accuracy. Conversely, the LAA method would present similar accuracy and reproducibility independent of the examiner's expertise. To test this hypothesis, we aimed to evaluate the performance of three groups of examiners with different levels of experience in assessing caries lesion activity using two different approaches associated with ICDAS. We also investigated the influence of the examiner's experience on ICDAS scoring.

METHOD AND MATERIALS

This study was approved by the Committee for Ethics in Research, Dental School, University of São Paulo. Each participant's guardian signed a positive consent form.

Sample Selection and Dental Examinations

The inclusion criteria were children with primary or mixed dentition and who had sought dental treatment at our school. Thus, eighteen children, aged three to eight years old, who presented at the Dental Clinic of the Dental School, University of São Paulo, were selected.

The assessments were performed by six examiners with different levels of experience: two undergraduate dental students, two specialists in pediatric dentistry, and two graduate students. The two undergraduate students were in their last year of study at the School of Dentistry of the University of São Paulo. The specialists had completed the specialization course at the same school, and they had at least four years of experience. The graduate students were enrolled in the PhD course in Pediatric Dentistry at the School of Dentistry of University of São Paulo, and they had at least six years of experience. Furthermore, only the graduate students had experience in using ICDAS in previous research.

The examiners were randomly divided in two groups (one dental student, one specialist, and one graduate student per group) using a coin toss. One group was first trained to use the method based on CCA of the lesions, and the other group used the LAA system. After the first series of examinations, the criteria were reversed.

Previous training sessions were conducted by two benchmark examiners (MMB and FMM). The training consisted of a lecture about ICDAS and about the adjunct activity criteria. Different sessions for each group were established. Then, hands-on training was conducted using 20 extracted primary teeth. Immediately after examining each tooth, the participants reviewed their scores by comparing them with the scores of the experts. There was a discussion about doubts and difficulties in carrying out the examinations. To avoid bias, each group of examiners underwent independent training using only one system of activity assessment. On a second occasion, 15 days later, the group that had used one index was trained to use of the other index, using the same methodology. The system used by each group for the first examination was randomly selected.

Examination Method

After the first training session, the examinations were performed. Before the examination, the teeth were carefully cleaned with a rotating bristle brush and a pumice/water slurry. Visual inspection was performed with the subjects positioned in a dental unit with operating light illumination, a 3-in-1 syringe, plane dental mirror (Duflex, Rio de Janeiro, Brazil) and WHO periodontal probe (Hu-Friedy, Rio de Janeiro, Brazil).

The surfaces of all of the teeth in the oral cavity, both primary and permanent teeth, were the experimental units. The criteria used were ICDAS and adjunct methods of activity assessment: CCA and LAA, depending on each group.

The approach based on the CCA of the lesions considered several clinical aspects, such as opacity, loss of luster, roughness, and location of the lesion (plaque stagnation area or not). The clinical characteristics associated with active or inactive caries lesions, according to different ICDAS scores are described in Table 1.

The other system was based on assigning different values for the lesion according to three characteristics: ICDAS score, whether it occurred in plaque stagnation area, and surface texture. After scoring the lesion, the examiners totaled the points and classified the lesion as active or inactive. A detailed description of the method is presented in Table 1.

First, one system was used for each subject according to the group of examiners. Fifteen days after the end of these examinations, other training sessions were held, and the group that performed the evaluation of activity using CCA of the lesions was trained to use the other system (LAA), and the other group was trained to perform the evaluation based

Table 1: *Criteria Used for Caries Activity Assessment Based on Evaluation of Clinical Characteristics Assessment[9] and Based on Numerical Values[10]*

International Caries Detection and Assessment System (ICDAS) Score	**Active Lesions**	**Inactive Lesions**
Clinical characteristics assessment (CCA)		
1, 2, or 3	Rough surface on gentle probing; plaque stagnation area; enamel surface whitish/yellowish opaque with loss of luster	Enamel surface is whitish, brownish, or black and may be shiny; hard and smooth surface on gentle probing. Nonplaque stagnation area
4	Probably active	
5 or 6	Soft surface on gentle probing	Hard surface on gentle probing, shiny aspect
Parameter	**Description**	**Numerical Values (Points)**
Lesion activity assessment (LAA)		
Clinical appearance	ICDAS 1, 2 (brown lesions)	1
	ICDAS 1, 2 (white lesions)	3
	ICDAS 3, 4, 5, or 6	4
Plaque stagnation area	Plaque stagnation area (pits, fissures, or cavities)	3
	Nonplaque stagnation area (flat pits and fissures or smooth surfaces)	1
Surface texture	Rough or soft surface on gentle probing	4
	Smooth or hard surface on gentle probing	2
Total	4-7 = inactive lesions; >7 = active lesions	

on CCA. Then, at another appointment, the examiners blindly re-examined all of the children using the system that they had not used in the first examination. The evaluation was performed independently by each group of examiners during different weeks, applying the previous training to the criteria of the activity. The examiners were unaware of their own scores and of each other's scores. The duration of each examination was measured using a digital stopwatch for all of the examinations.

Reference Standard

After all of the examinations, two senior lecturers who had experience in caries activity assessment and in using ICDAS also examined the children in a joint session, and the consensus of these examiners was considered the reference standard for all of the surfaces. These benchmark examiners (MMB and FMM) reached a decision about ICDAS score for each surface, and they used both methods together for caries activity assessment (LAA and CCA). Then, they decided only whether the lesions were active or inactive. These results were considered the reference standards.

Statistical Evaluation

Concerning the activity assessment, both systems, LAA and CCA, were compared by calculating intraclass correlation coefficients (ICC), sensitivity, and specificity using the results of benchmark examiners as references. Sensitivity and specificity values were compared using multilevel Poisson regression analyses. For these analyses, we considered five different levels in the following hierarchical order: examiner (level 5), patient (level 4), teeth (level 3), tooth surface (level 2), and evaluation (level 1). The outcomes for specificities and sensitivities were, respectively, false-positive (FP) results (true negative as a reference) and false-negative (FN) results (true positive as a reference). The independent variables were the method of caries activity assessment, the level of the examiner's experience, and the order in which the method was performed. When the analyses found a statistically significant association, we calculated prevalence ratios (PR) and respective 95% confidence intervals (CIs) for false FP or FN results. Appropriate statistical software was used for the multilevel analyses (MLWin 2.10, Centre for Multilevel Modeling, Bristol, UK).

Table 2: *Reliability (Intraclass Correlation Coefficients [ICCs]), Performance (Sensitivity and Specificity), and Time Spent in Examination (Mean ± SD) of Different Examiners According to the Level of Experience in Assessing Caries Lesions Activity Using Two Different Methods*[a]

Examiners (Ex)	Undergraduate Students		Specialists		Graduate Students	
	Ex 1	Ex 2	Ex 1	Ex 2	Ex 1	Ex 2
ICC						
CCA	0.650	0.759	0.650	0.712	0.705	0.693
LAA	0.690	0.646	0.679	0.724	0.774	0.686
Sensitivity						
CCA	0.769	0.641	0.741	0.538	0.819	0.641
LAA	0.728	0.663	0.659	0.622	0.703	0.741
Specificity						
CCA	0.869	0.972	0.889	0.983	0.898	0.940
LAA	0.912	0.912	0.930	0.948	0.945	0.913
Time of examination × (min)						
CCA	12.0 (3.3) A		9.6 (2.8) B		9.9 (2.8) B	
LAA	14.5 (3.2) A		11.0 (3.4) B		12.5 (4.2) B	

Abbreviations: CCA, clinical characteristics assessment; LAA, lesion activity assessment.
[a] *There were no statistically significant differences in specificity or sensitivity values among the methods or examiners according multilevel Poisson regression analyses (p>0.05). Means (SD) in the same line that do not share a letter are significantly different (p<0.05). There were significant differences between the methods for undergraduate and graduate students (p<0.05).*

The durations of the examinations were compared using two-way analysis of variance for repeated measures. With regard to the ICDAS scores, the ICCs were calculated by comparing the results of the benchmark examiners and the results of examiners with different levels of experience. Sensitivity and specificity were calculated at two thresholds: all lesions (ICDAS $\geq$ 1) or cavitated lesions (ICDAS $\geq$ 3). Multilevel Poisson regression analyses were also performed to compare the results regarding the levels of the examiners' experience. A receiver operating characteristic (ROC) analysis was also performed at both thresholds, and the areas under the curves were calculated. The level of significance was set at $p<0.05$.

Intra- and interexaminer agreement were calculated using weighted kappa statistics, considering the ICDAS scores for the examiners with similar levels of experience.

RESULTS

Overall, 11 (61.1%) boys and 7 (38.9%) girls, with a mean age of 5.3 years (standard deviation [SD] = 1.4), participated in the study. A total of 1734 surfaces of primary and permanent teeth in 18 children were examined. The children presented with dmf-t plus DMF-T ranging from 2 to 13 (mean ± SD = 8.2 ± 3.7). Considering the reference standard examination, 1264 surfaces (72.9%) were classified as sound, 32 surfaces (1.8%) were given an ICDAS score of 1, 117 (6.7%) were given a score of 2, 38 surfaces (2.2%) were given a score of 3 of ICDAS, 7 (0.4%) were given a score of 4, and 80 (4.6%) and 136 surfaces (7.8%) were given scores of 5 or 6, respectively. Moreover, 39 surfaces (2.2%) were restored, and five teeth (21 surfaces, 1.2%) were extracted because of extensive caries lesions. Considering the carious lesions, 326 were classified as active lesions, and 84 were inactive.

Therefore, we considered the sound surfaces plus inactive caries lesions (n=1348) versus active caries lesions (n=84) to obtain the data about caries activity assessment methods (Table 2). For the analysis related to the ICDAS scores (Table 3), lesions classified as score 1 to 6 of ICDAS were considered decayed (n=410) and sound surfaces (n=1264) as nondecayed at the all-lesions threshold. At the cavitated lesions threshold, surfaces scored 3 to 6 of ICDAS were considered decayed (n=261) and surfaces classified as 0, 1, or 2 of ICDAS nondecayed (n=1413).

Regarding the assessment of activity (Table 2), LAA showed similar or slightly better ICC than CCA compared to the classifications of the benchmark examiners. However, independent of the method of activity assessment and of examiner experience, the criteria used in the first examinations had significantly higher sensitivity values (PR, FN; 95% CI = 1.43; 1.10-1.87; p=0.008), but significantly lower specificity values (PR, FP; 95% CI = 0.54; 0.31-0.93; p=0.011).

Table 3: *Accuracy of the Examiners Obtained Using the International Caries Detection and Assessment System (ICDAS) Considering the Evaluation of the Benchmark Examiners as Reference Standard at Two Different Thresholds: All Lesions (ICDAS ≥ 1) or Cavitated Lesion (ICDAS ≥ 3)*[a]

Examiners	Undergraduate Student	Specialists	Graduate Student
Intraclass correlation coefficient	0.801-0.890	0.858-0.878	0.877-0.890
All lesions			
Sensitivity	0.794-0.812	0.794-0.835	0.810-0.863
Specificity	0.913-0.931	0.858-0.959	0.908-0.919
Area under receiver operating characteristic (ROC)	0.875-0.884	0.885-0.889	0.886-0.914
Cavitated lesion			
Sensitivity	0.813-0.841	0.825-0.877	0.785-0.825
Specificity	0.966-0.979	0.972-0.981	0.983-0.986
Area under ROC	0.927-0.933	0.932-0.955	0.932-0.943

[a] *Range of parameter values obtained in two series of examinations with two examiners according to the level of experience. There were no statistically significant differences in specificity or sensitivity values among the examiners according multilevel Poisson regression analyses (p>0.05).*

Furthermore, the examinations performed by dental undergraduate students were of significantly longer duration than those performed by the more experienced examiners. For all of the examiners, assessments using LAA criteria were significantly longer, varying from 12% (specialist) to 20% longer for the graduate students (Table 2).

With regard to the comparison with the reference standards, similar ICC values were observed among the groups of examiners (>0.8). At the cavitated threshold, the undergraduate students and specialists, in general, showed higher sensitivity and lower specificity than the graduate students. In detecting all caries lesions, however, graduate students showed a slight increase in sensitivity, but the areas under the ROC curves were similar at the two thresholds in the different groups of examiners (Table 3).

Regarding the ICDAS scores, we found that the inter- and intraexaminer kappa values were high, independent of the examiners' clinical experience (Table 4).

DISCUSSION

Because visual inspection is a subjective method for detecting caries lesions and for assessing their activity, the examiner's experience can influence the accuracy of the method. Two methods of assessing the activity of caries lesions have been published for use associated with the ICDAS.[9,10] One method (CCA) is based on a descriptive theory of cognitive processes, while the other is based on prescriptive theories.[11,12] Therefore, we aimed to investigate the influence of examiners' experience on the assessment of the activity caries lesions using these two different approaches and on the detection of caries lesions using ICDAS. Visual scoring systems for caries detection, such as ICDAS, are also based on a prescriptive theory of clinical decision making.[11]

With regard to caries activity assessment, we observed that examinations based on the numerical scoring of lesions took longer than examinations using the other method. Furthermore, for both methods, the more experienced examiners performed the examinations more quickly than the undergraduate students. These findings partially proved our hypothesis because methods based on prescriptive theories are usually more time consuming. Moreover, it would be expected that examinations performed by inexperienced clinicians would be of longer duration because they are used to employ hypothetical-deductive processes in comparison with

Table 4: *Agreement (Weighted Kappa Values) Obtained by Examiners According to their Level of Experience in Detecting Caries Lesions Considering the Scores on the International Caries Detection and Assessment System (ICDAS)*[a]

Examiners	Undergraduate Students	Specialists	Graduate Students
Intraexaminer	0.826-0.900	0.842-0.886	0.890-0.915
Interexaminer	0.838-0.839	0.821-0.837	0.837-0.839

[a] *Values obtained in two series of examinations.*

examinations performed by more experienced dentists.[11,12]

Concerning performance, the LAA method showed a slightly higher ICC than the method using the clinical features of lesions. This finding could be due to the more objective nature of the LAA, while the method of individual interpretation is more susceptible to subjectivity. However, this superiority was not significant. This finding is corroborated by the sensitivities and specificities obtained by both methods.

With regard to the examiner's experience, there was no significant influence of either method. One interesting finding concerned the order of the methods for assessing caries activity (at the first or second session). This order was actually more important than the method used. The criteria used for the first examination presented higher sensitivity, while the second session of examinations had higher specificities. This finding could be explained by the extensive training of the examiners in assessing the caries activity statuses of the lesions. In our study, when the examiners performed activity assessments at a second session, they had already carried out the examinations previously. Therefore, they were more highly trained than the first time. They could have maintained a residual effect from the first method, particularly because both methods for caries assessment consider similar clinical characteristics.

The similarity between the methods could explain the absence of an influence of the examiner's experience on performance. The small number of evaluators can be considered a limitation of our study, as only two examiners at each level of experience were used. This choice was made because a larger number of examiners would imply a more lengthy assessment for each child, making it more tiring and stressful for the young children who participated in this study. Furthermore, it would have increased the number of sessions, which could have led to dropouts.

Considering caries detection, previous studies—conducted before ICDAS had been proposed—found significant differences related to the dentist's experience. In a study in which caries lesions were evaluated according to the examiners' interpretations, with no use of scoring systems, less experienced dentists more accurately performed both the visual and the exploration methods.[14] This is an example of a descriptive approach to reaching a decision. It is likely that the less experienced examiners used hypothetic-deductive processes and that the experts used heuristic processes, which are more susceptible to bias. Conversely, other studies have shown that undergraduate students, performing visual inspection with no scoring system, yield lower specificity and reliability.[15,16]

We observed in the present study that the level of experience of the examiners had little influence on the performance and agreement of caries detection processes using ICDAS. These different results obtained with ICDAS and in the previous studies[14-16] were probably the results of the different approaches used to perform caries detection. It would be expected that the results with ICDAS would be less influenced by the examiners because a detailed description of each condition was provided for the examiners, as observed in our study. Another study using ICDAS corroborated our findings.[17] Similar results were obtained when the examiners used objective methods, such as the laser fluorescence method.[15]

In fact, one goal of using visual scoring systems is the minimization of the inherent subjectivity of clinical examinations.[1] Nevertheless, although the use of ICDAS and other visual scoring systems seems to be logical, clinicians (mainly more experienced ones) think differently from each other in clinical settings. They are accustomed to drawing on past clinical experience to make a diagnosis, and therefore they can be more resistant to learning about prescriptive methods of clinical examination. Therefore, several attempts should be made to teach dentists how to perform ICDAS to improve their diagnostic accuracy and to standardize the caries diagnostic process throughout the world.[7] The ICDAS e-learning program could be an alternative because it has improved the performance of dental students in detecting occlusal caries lesions.[18]

This was the first study to investigate methods of caries detection and activity assessment considering the different approaches of the theories behind clinical decision making. More studies should be performed to evaluate how clinicians are accustomed to making their diagnoses with regard to caries lesions and to propose more realistic methods with sufficient accuracy and reliability.

In conclusion, the examiner's experience does not significantly influence the performance of visual inspection for the detection and assessment of caries lesion activity after extensive training. Furthermore, both methods of caries activity assessment demonstrate similar validity, but the LAA method is more time consuming.

Conflict of Interest

The authors certify that they have no financial or other personal interest in any product, service or company mentioned in this article.

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Two-Year Clinical Performance of a Low-Shrinkage Composite in Posterior Restorations

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Clinical Relevance

Clinical performance of the Filtek Silorane Restorative System was similar to that of methacrylate-based restorative systems used in this study after two years of clinical use. Teeth restored with Adper Scotchbond SE + Filtek Z250 showed a trend toward higher marginal staining.

SUMMARY

Objectives: **The aim of this study was to compare the two-year clinical performance of three restorative systems in posterior restorations, which included a low-shrinkage composite and both etch-and-rinse and self-etch adhesive strategies.**

Materials and Methods: **After signing an informed consent, 25 patients received three Class I (occlusal) or Class II restorations performed with one of three restorative systems: Filtek Silorane Restorative System, Adper Scotchbond 1 XT (a two-step etch-and-rinse adhesive) with Filtek Z250, and Adper Scotchbond SE (a two-step self-etch adhesive) with Filtek Z250. All materials were applied following the manufacturer's instructions. Two blind observers evaluated the restorations at three different moments (baseline; and after one and two years) according to the US Public Health Service modified criteria. Kruskal-Wallis test and Mann-Whitney U-test were used to compare the behavior of the restorative systems, while Friedman and Wilcoxon tests were applied to analyze the intra-system data ($p<0.05$).**

Results: **The three restorative systems showed a statistically similar clinical performance at two years. Intra-system comparisons between baseline and two years showed declining marginal adaptation scores in the restorations placed with all systems. In addition, marginal staining and surface roughness scores were**

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lower after two years for the restorations placed with Adper Scotchbond SE + Filtek Z250.

Conclusions: **Although the clinical performance of Filtek Silorane was considered acceptable after two years, no advantage of the silorane-based resin over the methacrylate-based composite was found. Teeth restored with Adper Scotchbond SE showed a tendency for marginal staining, which may compromise the final color of the restorations.**

INTRODUCTION

The polymerization shrinkage of resin composites remains the main drawback of these widely used materials for the restoration of posterior teeth. The volumetric reduction due to polymerization generates stress within the material, at the adhesive interface, and in the tooth structure.[1] The physical mismatch between the shrinkage-prone restorative material and the stiffer tooth structure may result in undesirable clinical situations, such as microleakage, marginal staining, gap formation, postoperative sensitivity, and enamel microcracks or cusp deflection.[2,3]

With this background, several strategies aimed at reducing polymerization shrinkage have been proposed. One of them, the substitution of the most commonly used methacrylate-based monomers[4] for resins with a lower polymerization rate, has been an option since Filtek Silorane was introduced. However, information about the lower polymerization rate of Filtek Silorane is controversial. *In vitro* studies have reported a significantly lower cusp deflection after restoration of MOD preparations with a silorane-based resin composite in comparison to methacrylate-based resins.[5,6] According to the manufacturer's information, volumetric shrinkage is supposed to not exceed 1%. However, recent research has found it to be higher (1.4%)[5,7] and close to the 1.7% total volumetric shrinkage attributed to Filtek Z250, a methacrylate-based resin composite.[7] Additionally, Filtek Silorane did not provide better results than methacrylate-based materials in clinical investigations.[8,9]

Filtek Silorane is part of an integral restorative system that includes a proprietary and specific two-step self-etch adhesive. This adhesive strategy has become increasingly popular, as self-etch adhesives are more user friendly and less technique sensitive and may reduce postoperative sensitivity[10,11] compared to etch-and-rinse adhesives. Nevertheless, their adhesion to enamel, especially those with low acidity (mild self-etch adhesives), is still not comparable to that of etch-and-rinse adhesives, which are considered the "gold standard."[10,12,13]

Clinical trials represent the ultimate test to adequately measure the clinical effectiveness and durability of adhesives and resin composites.[14] This is of paramount relevance, as there is no clinical evidence to back the deleterious effect of polymerization stress on restoration longevity.[15] As mentioned above, few studies have analyzed the clinical performance of the Filtek Silorane Restorative System. The present study was initiated in 2008, and the results of the one-year clinical performance revealed that the application of Filtek Silorane did not provide any advantage over a methacrylate-based resin composite.[9] However, more conclusive outcomes would be expected from a longer period of clinical use. Accordingly, the aim of this study was to compare the two-year clinical performance of three restorative systems in posterior restorations: the low-shrinkage silorane resin composite with its proprietary adhesive and a widely studied methacrylate resin composite, Filtek Z250, used either with a two-step etch-and-rinse adhesive or with a two-step self-etch adhesive. The null hypothesis tested was that there would be no differences in clinical behavior for the three restorative systems after two years.

MATERIALS AND METHODS

Before participating in the study, subjects signed a written informed consent. Both the consent and this research protocol had been previously reviewed and approved by the Ethics Committee of Rey Juan Carlos University.

All patients, with ages ranging from 18 to 60 (average 29.8), required at least three Class I (occlusal) and/or Class II restorations (Table 1). The dental health status of patients was normal in all other respects. Specific exclusion criteria were as follows:

- Fewer than 20 teeth
- History of existing tooth sensitivity
- Periodontal disease (CPITN values higher than 2)
- Extremely poor oral hygiene with evident accumulation of plaque or calculus within the gingival pocket or within the tooth and/or gingival margin
- Bruxism
- Known allergy to resin-based materials or other materials used in this study
- Pregnancy or breast-feeding

Table 1: *Number of Evaluated Restorations by Location (Tooth) and Extension (Class) for Each Restorative System*

Restorative System	Number of Restorations	Tooth		Class		
		Premolars	Molars	I	II	
					MO/OD	MOD
Filtek Silorane Restorative System	25	12	13	12	10	3
Adper Scotchbond 1 XT + Filtek Z250	25	8	17	14	10	1
Adper Scotchbond SE + Filtek Z250	25	13	12	12	12	1
Total (%)	75	33 (44)	42 (56)	38 (50.6)	32 (42.6)	5 (6.6)

- Chronic use of anti-inflammatory, analgesic, and psychotropic drugs

Further, exclusion criteria for the teeth to be restored were as follows:

- Nonvital teeth
- Abutment teeth for fixed or removable prostheses
- Teeth without a normal occlusal relationship with natural dentition or without at least one adjacent tooth contact.

Bitewing radiographs of the teeth to be restored were taken preoperatively, unless the patient had radiographs taken within the previous year. There was an even distribution of the restorations that replaced existing restorations with clinical or radiographic signs of recurrent caries or esthetic failures and restorations that were performed to treat primary caries lesions.

All operative procedures were performed by the same operator (BB). Restorations were placed under local anesthesia with rubber dam isolation. The cavity design was restricted to eliminate carious tissue from primary caries lesions or to remove the restorative material when existing restorations were replaced. Cavities were prepared using diamond burs (Komet-Brasseler, Lemgo, Germany) with no intentional bevels on enamel cavosurface margins. In deep cavities, dentin was covered with a resin-modified glass ionomer cement (Vitrebond, 3M ESPE, St. Paul, MN, USA). An appropriate matrix system (Palodent, Dentsply DeTrey, Konstanz, Germany) and wood wedges were applied to the cervical margins of proximal preparations.

The restorative systems evaluated in this study were the Filtek Silorane Restorative System, Adper Scotchbond 1 XT + Filtek Z250, and Adper Scotchbond SE + Filtek Z250 (Table 2). Initially, the three restorative systems were randomly assigned to each of the three teeth in which restorative treatment was needed, regardless of the characteristics of the tooth and restoration class. However, interference in the randomization procedure within patients was occasionally carried out with the purpose of equally distributing materials into some important variables (tooth type and position, restoration class and size) in such a way that the influence of those factors was minimized.[16] All adhesive systems were applied according to manufacturer's instructions (Table 2). Resin composites were placed in 2-mm increments using an incremental layering technique. Each increment was light cured for 20 seconds using a LED Demetron I polymerization unit (Kerr, Orange, CA, USA) with a minimum light output of 550 mW/cm^2.

After polymerization, coarse finishing was accomplished with carbide burs under water cooling and, if needed, with a #12 blade and aluminum oxide disks (Sof-Lex, 3M ESPE). Final finishing of the occlusal surface was accomplished with polishing points (Enhance and PoGo, Dentsply DeTrey).

Clinical Evaluation

All restorations were evaluated after one week (baseline), one year, and two years for the following parameters: color match, retention, marginal adaptation, anatomic form, surface roughness, marginal staining, sensitivity, and secondary caries (Table 3). Pre- and postoperative sensitivity was determined with a dental syringe placed 2 cm from the tooth surface. Two clinicians (LC and EC) evaluated the restorations blindly at each recall using the modified United States Public Health Service criteria as adapted by Wilson and others[17] (Table 3). When disagreements arose during evaluations, the examiners had to reach a consensus. To help with the evaluation of marginal discoloration, intraoral color photographs were collected at baseline and at the recall appointments. Clinical photographs consisted of digital images at 1.3× magnification taken with a Nikon D80 camera with a 105-mm Micro-Nikkor lens (Nikon USA, Melville, NY, USA).

The statistical analyses were carried out with the IBM SPSS 19 (IBM Corporation, Armonk, New York,

Table 2: *Materials Used in the Study (3M ESPE)*

Adhesives (Batch No.)	Composition	Instructions for Use	Type
Silorane System Adhesive (also known as LS System Adhesive or P90 System Adhesive) (Primer: 8AP; Adhesive: 8AK)	Primer: phosphorylated methacrylates, Bis-GMA, HEMA, water, ethanol, silane-treated silica filler, Vitrebond™ copolymer, initiators, stabilizers	Primer: application for 15 seconds with black microbrush, followed by gentle air dispersion and 10 seconds of light curing	Two-step self-etch
	Adhesive: hydrophobic DMA, phosphorylated methacrylates, TEGDMA, silane-treated silica filler, initiators, stabilizers	Adhesive: application with green microbrush followed by gentle air dispersion and 10 seconds of light curing	
Adper Scotchbond 1 XT (also known as Adper Single Bond Plus or Adper Single Bond 2) (318655)	HEMA, Bis-GMA, GDMA, water, ethanol, silane-treated silica nanofiller, photoinitiator	Acid etch: phosphoric acid (Scotchbond™ Etchant, 3M ESPE): 35% (15 seconds); rinse (10 seconds); blot excess water using a cotton pellet or minisponge; do not air-dry	Etch-and-rinse
		Adhesive: apply two to three consecutive coats of adhesive for 15 seconds with gentle agitation using a fully saturated applicator; gently air thin for five seconds to evaporate solvent; light cure for 10 seconds	
Adper Scotchbond SE (also known as Adper SE Plus) (Liquid A: 7AF; Liquid B: 8AL)	Liquid A (colored wetting solution): water, HEMA, surfactant, rose bengal dye	Liquid A: apply to the cavity so that a continuous red-colored layer is obtained on the surface	Two-step self-etch
	Liquid B (Adhesive): UDMA, TEGDMA, TMPTMA, HEMA phosphate and MHP, bonded zirconia nanofiller, initiator system based on camphorquinone	Liquid B: scrub into the entire wetted surface of the bonding area during 20 seconds; red color will disappear quickly, indicating that the etching components have been activated; air-dry thoroughly for 10 seconds; apply second coat to the entire bonding surface; light air application; light cure for 10 seconds	
Resin composites	**Organic matrix**	**Inorganic filler**	
Filtek Silorane (8BH)	3,4-epoxycyclohexylethylcyclopolymethylsiloxane, bis-3,4-epoxycyclohexylethylphenylmethylsilane, yttrium fluoride (15%), camphorquinone, iodum salt, stabilizers, pigments.	Silanized quartz particles: 50% vol, 70% weight	
		Size: 0.1-2 μm	
Filtek Z250 (7LY)	Silane-treated ceramic, bisphenol A polyethylene glycol diether dimethacrylate, UDMA, Bis-GMA, TEGDMA, Water <2%	Quartz and zirconia particles: 60% vol, 78% weight	
		Size: 0.01-3.5 μm (0.6 μm average)	

Abbreviations: UDMA, urethane dimethacrylate; GDMA, glycerol 1,3-dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; Bis-GMA, bisphenol A diglycidyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; TMPTMA, trimethylolpropane trimethacrylate (hydrophobic TMA); MHP, methacrylic phosphates.

USA) for Windows software using the Kruskal-Wallis test and Mann-Whitney nonparametric U-test to compare the behavior of the three restorative systems at baseline, one year, and two years. Friedman and Wilcoxon nonparametric tests were used to compare the data obtained for each restorative system at each evaluation period. The level of confidence was set at $\alpha<0.05$.

RESULTS

A total of 75 restorations were placed in 25 patients. The distribution of the restorations was similar between Class I (38) and Class II (37) cavities (Table 1). All patients attended the one-year recall (100% rate), although it decreased at the two-year assessment (96% rate), as one patient did not return for the recall. The results are summarized in Table 4.

Comparison of the Performance of the Three Restorative Systems at Two Years

All restorative systems resulted in a percentage of Alfa ratings of 100% at two years for the categories of retention, anatomical form, sensitivity, and secondary caries. However, Alfa ratings for color match, surface roughness, and, especially, marginal adaptation decreased for all the restorative systems, although this reduction did not result in statistical

Table 3: *Modified United States Public Health Service Criteria Used*[a]

Criteria	Code	Definition
Color match	Alfa	Restoration matches adjacent tooth structure in color and translucency.
	Bravo	Mismatch is within an acceptable range of tooth color and translucency.
	Charlie	Mismatch is outside the acceptable range.
Retention	Alfa	Full retention.
	Bravo	Partial retention.
	Charlie	Restoration is lost.
Marginal adaptation	Alfa	Restoration closely adapted to the tooth. No crevice visible. No explorer catch at the margins, or there was a catch in one direction.
	Bravo	Explorer catch. No visible evidence of a crevice into which the explorer could penetrate. No dentin or base visible.
	Charlie	Explorer penetrates into a crevice that is of a depth that exposes dentin or base.
Anatomic form	Alfa	Restorations continuous with existing anatomic form.
	Bravo	Restorations discontinuous with existing anatomic form, but missing material not sufficient to expose dentin base.
	Charlie	Sufficient material lost to expose dentin or base.
Surface roughness	Alfa	Surface of restoration is smooth.
	Bravo	Surface of restoration is slightly rough or pitted but can be refinished.
	Charlie	Surface deeply pitted, irregular grooves and cannot be refinished.
	Delta	Surface is fractured or flaking.
Marginal staining	Alfa	No staining along cavosurface margin.
	Bravo	<50% of cavosurface affected by stain (removable, usually localized).
	Charlie	>50% of cavosurface affected by stain.
Sensitivity[b]	Alfa	None.
	Bravo	Mild but bearable.
	Charlie	Uncomfortable, but no replacement is necessary.
	Delta	Painful. Replacement of restoration is necessary.
Secondary caries	Alfa	Absent.
	Bravo	Present.

[a] *Based on Wilson and others.*[17]
[b] *Postoperative sensitivity at baseline was registered one week after the restoration insertion.*

differences. Marginal staining was not significantly different among the three restorative systems, although only 62.5% of the restorations inserted with Adper Scotchbond SE + Filtek Z250 were rated Alfa.

Baseline Versus Two-Year Evaluation for Each Restorative System

Filtek Silorane Restorative System—Marginal adaptation was significantly worse at two years than at baseline, as 7 of 23 restorations were rated Bravo (p=0.01). The deterioration of the marginal integrity was statistically significant at the one-year assessment and remained significant at two years. On the other hand, marginal staining increased in the last 12 months since two more restorations were rated Bravo at two years, which was close to statistical significance (p=0.59). Only one restoration showed a true color modification over time, and two restorations did not match the adjacent tooth structure because of the yellowish and very opaque aspect of the Filtek Silorane resin composite. One restoration showed signs of clinical failure (fracture of the restorative material, exposure of dentin, and presence of secondary caries) after one year of clinical use, being replaced prior to the two-year evaluation.

Adper Scotchbond 1 XT + Filtek Z250—Color match, marginal staining, and surface roughness parameters resulted in worse rankings at two years although with no statistical repercussion. A significant reduction of the marginal adaptation was detected at two years (p=0.04). No Charlie ratings were assigned to this restorative system for any of the criteria. Postoperative sensitivity (slight discomfort associated with cold beverages) was found in one patient during the first week after the restoration was placed but relapsed thereafter.

Adper Scotchbond SE + Filtek Z250—Marginal adaptation (p=0.005), marginal staining (p=0.005),

Table 4: *Number of Evaluated Restorations in Each Criterion for Each Experimental Group*

Criteria	Code	Materials								
		Baseline			One Year			Two Years		
		Filtek Silorane RS	Adper Scotchbond 1 XT + Filtek Z250	Adper Scotchbond SE + Filtek Z250	Filtek Silorane RS	Adper Scotchbond 1 XT + Filtek Z250	Adper Scotchbond SE + Filtek Z250	Filtek Silorane RS	Adper Scotchbond 1 XT + Filtek Z250	Adper Scotchbond SE + Filtek Z250
Color match	Alfa	23	25	23	22	24	22	20	22	21
	Bravo	2	—	2	3	1	1	3	2	1
	Charlie	—	—	—	—	—	2	—	—	2
Retention	Alfa	25	25	25	24	25	25	23	24	24
	Bravo	—	—	—	1	—	—	—	—	—
	Charlie	—	—	—	—	—	—	—	—	—
Marginal adaptation	Alfa	24	25	25	17	21	18	16	20	16
	Bravo	1	—	—	7	4	7	7	4	8
	Charlie	—	—	—	1	—	—	—	—	—
Anatomic form	Alfa	25	25	25	24	25	25	23	24	24
	Bravo	—	—	—	—	—	—	—	—	—
	Charlie	—	—	—	1	—	—	—	—	—
Surface roughness	Alfa	23	24	25	22	22	21	20	20	20
	Bravo	2	1	—	2	3	4	3	4	4
	Charlie	—	—	—	—	—	—	—	—	—
	Delta	—	—	—	1	—	—	—	—	—
Marginal staining	Alfa	25	25	23	23	22	16	19	21	15
	Bravo	—	—	2	1	3	8	3	3	8
	Charlie	—	—	—	1	—	1	1	—	1
	Delta	—	—	—	—	—	—	—	—	—
Sensitivity	Alfa	25	24	24	25	25	25	23	24	24
	Bravo	—	1	1	—	—	—	—	—	—
	Charlie	—	—	—	—	—	—	—	—	—
	Delta	—	—	—	—	—	—	—	—	—
Secondary caries	Alfa	25	25	25	24	25	25	23	24	24
	Bravo	—	—	—	1	—	—	—	—	—

and surface roughness (p=0.046) were significantly worse at two years than at baseline. All these criteria showed the same trend, as the significant differences were detected in the first year of clinical use and remained stable at the two-year assessment.

Additionally, color stability decreased at the two-year assessment, resulting in a near statistically significant difference (p=0.05). This restorative system was the only one receiving one Charlie rating for color match.

One patient experienced postoperative sensitivity after restoration placement, which disappeared gradually after a few days.

DISCUSSION

In the present study, the three restorative systems resulted in statistically similar clinical parameters after two years. However, regarding the intra-system comparisons between the baseline and two years for each restorative system, all exhibited a statistically lower number of Alfa ratings for marginal adaptation after two years.

Marginal adaptation is influenced mainly by the polymerization shrinkage of the resin composite and the adhesive type,[18] so both factors might have influenced the clinical results of this study. Ideally, marginal adaptation, as an exclusive consequence of polymerization shrinkage and resulting stress, should be assessed at baseline because both take place during the placement of the restoration. However, clinical consequences, such as wear and integrity of the adhesive interface, might have also modified the marginal adaptation during the two years of clinical use.

Polymerization shrinkage of resin composites is considered a potentially harmful factor for the integrity of the restoration at the margins and, consequently for clinical success and longevity of direct restorations because of the release of stresses onto the adhesive interface.[19] The dynamics of the transmission of the stress shrinkage are affected by the cavity configuration.[20] High C-factor values are expected to emerge in Class I and II cavities,[20] where the application technique of the resin composite is also a factor that may influence the bonding effectiveness.[21] Class I and class II preparations were selected in the present study because of the specific recommendation of Filtek Silorane Restorative System for posterior restorations. Additionally, an incremental technique was used for all restorations, as it has been demonstrated to benefit bond strength of both methacrylate-based[22,23] and silorane-based resin composites.[21]

The present study did not find any clinical benefit of Filtek Silorane, the resin composite with reduced polymerization shrinkage. The final value of polymerization shrinkage for this resin composite has been recently questioned since it resulted in a very similar volumetric reduction rate compared to that of Filtek Z250.[5,6] Moreover, Filtek Silorane showed higher elastic modulus and polymerization stress values than those of Filtek Z250.[5] These investigations contradict the idea that less shrinkage polymerization contributes to lower polymerization stress values, as would be originally expected,[24] and confirm that reduced shrinkage *per se* guarantees neither the attenuation of stress effects in restored teeth[5] nor the interfacial integrity of the restoration.[7] These *in vitro* data are strongly in line with the findings of previous clinical studies.[15,25]

Regarding the clinical outcomes in the literature of the Filtek Silorane Restorative System, they are still scarce and contradictory. While a recent publication has detected a worse marginal adaptation than that of a methacrylate-based composite (Ceram•X),[8] another research project found satisfactory performance after two years (84% of optimal marginal adaptation).[26] The present study complements a research project in which one-year results found an acceptable clinical performance of Filtek Silorane, although marginal adaptation was not as stable as that of an etch-and-rinse two-step adhesive combined with a methacrylate-based resin composite (Adper Scotchbond 1 XT + Filtek Z250).[9] Analysis of data obtained from the two-year clinical assessment revealed that the advantages of the

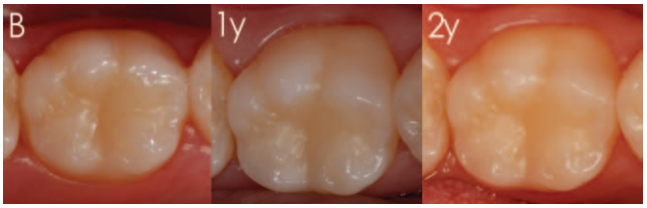

Figure 1. *First mandibular molar. Occlusal restoration with Adper Scotchbond 1 XT and Filtek Z250. This restoration preserved its original aspect after one and two years. No signs of adhesive deterioration were found. B, baseline; 1Y; one-year recall; 2Y, two-year recall.*

Adper Scotchbond 1 XT + Filtek Z250 restorative system measured at one year had disappeared at two years. The basic and most important clinical finding is in agreement with the peer-reviewed literature: the silorane-based resin composite provides adequate clinical performance that does not surpass the behavior of methacrylate-based materials.[8,9,26]

Results from the one-year evaluation were in part explained by the use of different adhesive strategies. In regard to the two-year outcomes, it is noteworthy that the restorations performed with only the etch-and-rinse adhesive obtained the highest number of Alfa ratings for marginal adaptation (Figure 1). Nevertheless, in this case, it did not lead to a statistically better outcome than that achieved with self-etch adhesives, which agrees with previous clinical research in which a similar clinical performance was observed between self-etch and etch-and-rinse adhesives.[27,28] Many of the marginal defects detected in the present study appeared to result from the fracture of thin flashes of resin composite that extended to noninstrumented enamel surfaces adjacent to the cavity margins.

The adhesive system that accompanies Filtek Silorane requires separate light curing of the primer and the bonding resin, thereby establishing the bonding mechanism to hard dental tissues in the first application step, resembling one-step self-etch adhesives. This primer has a relatively high pH (2.7) and contains the Vitrebond copolymer, which has been reported to be able to chemically bond to the calcium within the hydroxyapatite,[29,30,31] as explained by the "adhesion-decalcification" concept.[32] According to this, Mine and others[33] observed a tight superficial interaction and a very slight inter- and intracrystallite demineralization with subsequent resin infiltration when bonded to enamel. In regard to dentin, it has been demonstrated that the Filtek Silorane adhesive system

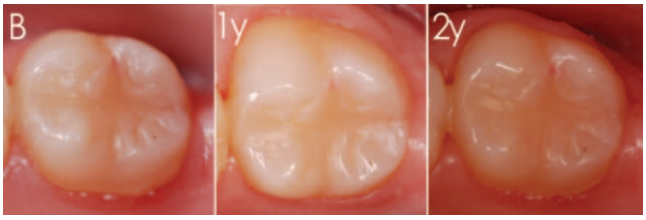

Figure 2. *Second mandibular molar. Occlusal restoration with Adper Scotchbond SE and Filtek Z250. Marginal staining located at the buccal central margin was observed from baseline and remained stable during the following assessments, being rated Bravo (<50% of cavosurface is affected). B, baseline; 1Y; one-year recall; 2Y, two-year recall.*

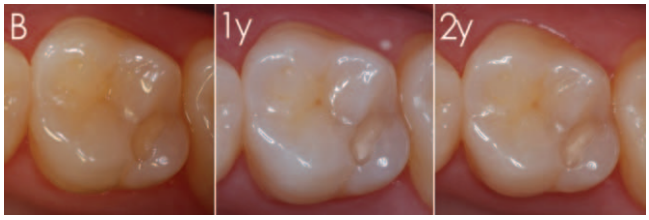

Figure 3. *First maxillary molar. Occlusal restoration with Filtek Silorane Restorative System. This restoration was rated Bravo for color parameter in all the evaluations because of the poor esthetic characteristics of the composite resin. No signs of adhesive deterioration were found. B, baseline; 1Y; one-year recall; 2Y, two-year recall.*

provides a tight, stable, and water-resistant adhesion to dentin.[31,33]

Besides marginal adaptation, intra-system comparison also detected a deterioration of the surface roughness and marginal staining parameters after two years for the restorations performed with Adper Scotchbond SE + Filtek Z250. Surface roughness should not be different from that recorded with Adper Scotchbond 1 XT + Filtek Z250 since both systems included the same resin composite, which was always applied, finished, and polished in the same way. In fact, the results at two years were exactly the same for both restorative systems, so the statistical repercussion could be ascribed to their different values at baseline.

Meanwhile, marginal discoloration may be considered a clinical sign indicating that a restoration is prone to failure or, at least, that the adhesive interface degrades with time.[34] Although Adper Scotchbond SE is a strong self-etch adhesive (pH = 1),[35] with high etching ability, marginal discoloration and color changes were detected, as it has been previously reported for another strong self-etch adhesive.[34] Adper Scotchbond SE is similar to a one-step self-etch adhesive, as Liquid A is a HEMA-water solution with no etching ability that turns from pink to yellow after the application of Liquid B, containing the acidic monomers. However, this activation may lead to an incomplete conversion of the acidic monomers and their inclusion in a HEMA-rich (with an enhanced susceptibility to hydrolytic degradation) and still pink-colored adhesive interface. This possibility was corroborated by the presence of the characteristic pink shade in most of the stained margins around Adper Scotchbond SE + Filtek Z250 restorations (Figure 2).

High color instability after water immersion has been revealed for a self-etch adhesive (One-Up Bond F) with a very similar color-change mechanism to that of Adper Scotchbond SE.[36] Moreover, it was concluded that discoloration of the adhesive interface may affect the color appearance of the entire restoration,[36] which is highly consistent with what evaluators found in the present study, as all Adper Scotchbond SE + Filtek Z250 restorations that rated Bravo or Charlie for color match showed variable saturation of pink, even at the baseline evaluation (Figure 2).

Regarding Filtek Silorane, the two Bravo ratings for color match at baseline were caused by the poor esthetic characteristics of the silorane resin composite (Figure 3). As mentioned before, this restorative system has been especially designed for restorations in posterior teeth, where the esthetic demand is less critical. Consequently, the manufacturer provides only four shades. The evaluators deemed these restorations too yellow and very opaque, with a very different translucency from that of tooth structure (Figure 3). Both restorations were also rated Bravo in the subsequent follow-up assessments; therefore, only one restoration showed a real color modification over time. These observations derived from the *in vivo* analysis are consistent with *in vitro* research demonstrating low translucency[37] and high color stability of silorane resin composite compared to those of methacrylate-based resin composites.[38]

CONCLUSIONS

The clinical performance of the Filtek Silorane Restorative System was found acceptable after two years. Despite the limitations of this study, the clinical outcomes led to the perception that the Filtek Silorane Restorative System did not provide any detectable advantage for the evaluated criteria compared to the methacrylate-based restorative systems used in this study. Teeth restored with Adper Scotchbond SE + Filtek Z250 showed a trend

toward higher marginal staining, which may compromise the final color of the restorations.

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Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article *except for the following*: Dr Elena Cabrera reports personal interests in 3M ESPE for the following reasons: Dr Cabrera has been affiliated with 3M ESPE Iberia since 1 July 2011. At the time this research project was carried out, this author was affiliated exclusively with Rey Juan Carlos University. This author has not participated in the writing of this manuscript, but she agrees with the contents.

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The Effect of Perioperative Ibuprofen Use on Tooth Sensitivity Caused by In-Office Bleaching

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Clinical Relevance

The anti-inflammatory drug ibuprofen reduced the intensity of tooth sensitivity up to one hour after in-office bleaching treatment.

SUMMARY

Objective: This study determined the effect of the administration of perioperative ibuprofen 400 mg on tooth sensitivity caused by in-office bleaching.

Methods: A triple-blind, parallel-design, randomized clinical trial was conducted on 30 adults who received placebo or ibuprofen before and after bleaching. The drugs were administered three times per day for 48 hours; the first dose was given one hour prior to the bleaching treatment. Two bleaching sessions with 35% hydrogen peroxide gel were performed with a one-week interval. Tooth sensitivity was recorded on two scales: visual analogue and five-point verbal rating scale up to 48 hours after bleaching. The shade evaluation was performed with a visual shade guide and spectrophotometer, before and 30 days after bleaching. The absolute risk of tooth sensitivity and its intensity were evaluated by Fisher exact and Mann-Whitney tests, respectively. The shade changes were evaluated by Student's t-test.

Results: Both groups showed similar absolute risk of tooth sensitivity ($p>0.05$). Lower tooth sensitivity was observed in the experimental group only up to one hour postbleaching ($p=0.04$). Similar tooth sensitivity was observed in the other periods of time.

Conclusion: The perioperative use of the anti-inflammatory ibuprofen was not able to avoid tooth sensitivity but reduced its intensity up to one hour after bleaching.

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INTRODUCTION

The desire for whiter teeth has made tooth bleaching one of the most sought-after cosmetic procedures in dentistry. Available bleaching modalities include dentist-prescribed at-home bleaching and dentist-supervised in-office bleaching.[1] Even though the at-home bleaching system is the most frequently recommended treatment for vital teeth, some patients do not want to use a bleaching tray or do not want to wait two to three weeks to see the results of the treatment.[2] Thus, another bleaching option, the in-office bleaching procedure, is often requested.

However, the in-office procedure using 35% hydrogen peroxide has a long history of tooth sensitivity and gingival irritation.[2,3] Incidence levels of tooth sensitivity have been reported to be as high as 87%,[4-6] which seems to result from the easy passage of the peroxide through the enamel and dentin to the pulp,[7] causing pulpal damage with inflammation.[8] Pulp tissue damage caused by dental bleaching is likely to lead to the release of cell-derived factors, such as adenosine triphosphate[9] and prostaglandins, which excite or sensitize pulpal nociceptors.[10]

The use of desensitizing agents such as fluorides and potassium nitrate before or after bleaching was capable of reducing the experience of tooth sensitivity during the bleaching treatment.[2,5,11,12] However, this approach adds another step to the bleaching protocol, which is against the clinicians' preference for simplification. Another approach recently investigated was the preoperative use of ibuprofen,[13] which is a nonsteroidal anti-inflammatory drug capable of blocking the cyclooxygenase (COX) pathway.[14] Although this clinical alternative seemed promising, the preoperative use of a single dose of ibuprofen 400 mg before the in-office bleaching protocol was shown to reduce tooth sensitivity only during but not after the treatment period.[13] As mentioned by the authors of the previous study,[13] the half-life elimination of ibuprofen is two to four hours, and thus, more than one dose may be required to keep the ibuprofen serum level sufficiently high for optimum analgesic effect.

Therefore, the current study tested the primary hypothesis that the preventive and perioperative use of ibuprofen 400 mg for 48 hours, starting one hour before the bleaching session, would reduce the absolute risk of tooth sensitivity. A secondary hypothesis tested was that the use of this anti-inflammatory drug would reduce the intensity of sensitivity without affecting the bleaching efficacy and shade change.

MATERIALS AND METHODS

This clinical investigation was approved (protocol No. 17836/2010) by the scientific review committee and the committee for the protection of human subjects of the local university. The experimental design followed the Consolidated Standards of Reporting Trials statement.[15] Based on preestablished criteria, 30 volunteers from the city of Guarapuava, Paraná, Brazil were selected for this study and signed a term of free and informed consent to participate. Two weeks before the bleaching procedures, all the volunteers received a dental screening and dental prophylaxis using a rubber cup with pumice and water slurry.

Study Design

This was a randomized, placebo-controlled, triple-blind, parallel-group clinical trial, with an equal allocation rate to receive one of two treatments. The study took place in the clinic of the Brazilian Association of Dentistry in Guarapuava, Paraná, from January 2011 to February 2011.

Inclusion and Exclusion Criteria

Patients included in this clinical trial were at least 18 years old and had good general and oral health. Participants were recruited by means of radio and television advertisement. A total of 247 participants were examined, in a dental chair, to check if they met the inclusion and exclusion criteria (Figure 1). The participants should have at least eight maxillary and mandibular anterior teeth that were caries free and without restorations on the labial surfaces. The central incisors were C2 or darker as judged by comparison with a value-oriented shade guide (Vita Lumin, Vita Zahnfabrik, Bad Säckingen, Germany). Participants who had undergone tooth-whitening procedures, had preexisting anterior restorations, were pregnant/lactating, had severe internal tooth discoloration (tetracycline stains, fluorosis or pulpless teeth), had bruxism habits, or had any other pathology that could cause sensitivity (such as recession or dentin exposure) were excluded from this study to minimize confounding experimental variables or side effects from bleaching. Participants who reported a history of health problems in the stomach, heart, kidney, or liver or participants using any continuous drug with anti-inflammatory and antioxidant action were also excluded from the study.

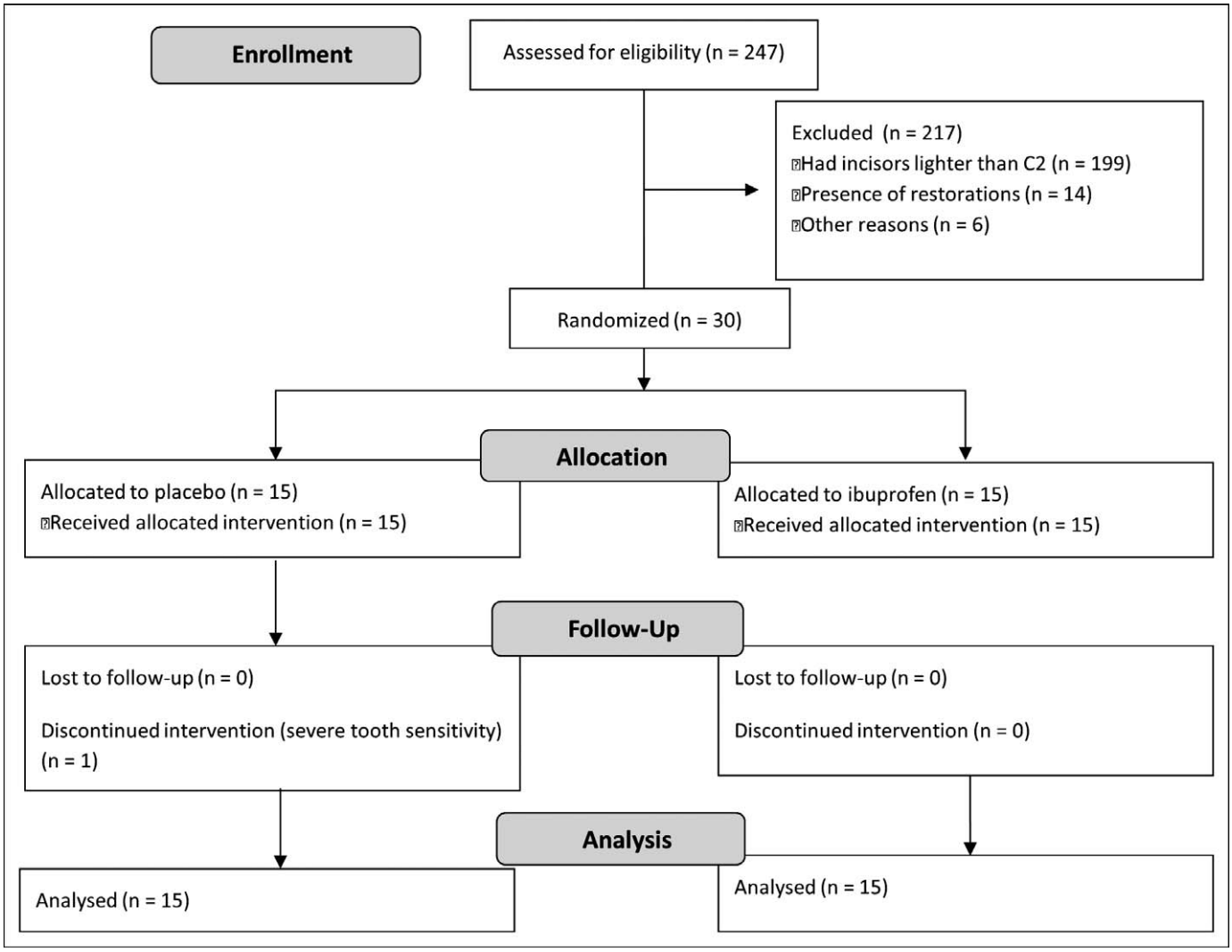

Figure 1. *Flow diagram of the clinical trial including detailed information on the excluded participant.*

Sample Size Calculation

The primary outcome of this study was the absolute risk of tooth sensitivity. This risk was reported to be approximately 87% for the bleaching product Whiteness HP Maxx (FGM, Joinville, SC, Brazil).[4] Thus, 30 patients were required to have an 80% chance of detecting, as significant at the two-sided 5% level, a decrease in the primary outcome measure from 87% in the control group to 41% in the experimental group.

Study Intervention

Participants were randomly stratified by sex into the placebo and ibuprofen groups. The randomization process was performed by computer-generated tables by a third person not involved in the research protocol. Details of the allocated groups were recorded on cards contained in sequentially numbered, opaque, sealed envelopes. These cards were prepared by a third person not involved in any of the phases of the clinical trial. Once the participant was eligible for the procedure and completed all baseline assessments, the allocation assignment was revealed by opening this envelope by this third person. Neither the participant nor the operator knew the group allocation, with both being blinded to the protocol.

The participants from the placebo group received a placebo (Talco pharma S M-200 Henrifarma Prod Quím Farm LTD, São Paulo, SP, Brazil), and participants from the ibuprofen group received a dose of nonsteroidal anti-inflammatory ibuprofen 400 mg (Uniprofen, União Quím Farm Nacional S/A, Embu-Guaçu, SP, Brazil). All participants were

watched to ensure that they took the drugs or placebo one hour prior to treatment. Other doses of placebo or ibuprofen (400 mg) were administered every eight hours after the first dose during a period of 48 hours. When it was time for the participants to take the other doses of medicine and placebo, the research assistant called all patients to remind them to take the drugs. This procedure was done to increase adherence to the protocol.

The gingival tissue of the teeth to be bleached was isolated from the bleaching agent using a light-polymerized resin dam (Top Dam, FGM, Joinville, SC, Brazil). The 35% hydrogen peroxide gel (Whiteness HP Maxx, FGM) was used in three 15-minute applications for both groups following the manufacturer's directions. The in-office bleaching agent was refreshed every 15 minutes during the 45-minute application period. Two bleaching sessions, with a one-week interval, were performed on each patient. All participants were instructed to brush their teeth at least three times a day using fluoridated toothpaste (Sorriso Fresh, Colgate-Palmolive, São Paulo, SP, Brazil).

Shade Evaluation

Shade evaluation was recorded before and 30 days after the bleaching treatment using two methods: the subjective evaluation using a shade guide (Vita Lumin, Vita Zahnfabrik) and an objective evaluation using the spectrophotometer (Easyshade, Vident, Brea, CA).

For the subjective examination, the 16 shade guide tabs were arranged from highest (B1) to lowest (C4) value, making the minimum qualifying shade C2 as number 7 (seventh tab on the value-ordered arrangement). Although this scale is not linear in the truest sense, the changes were treated as though they represented a continuous and approximately linear ranking for the purpose of analysis. The measurement area of interest for shade matching was the middle one third of the facial surface of the anterior central incisor. For calibration purposes, five patients who were not included in the sample because they were used in the pilot study participated in the training phase of this study. The two examiners, blinded to the allocation assignment, scheduled these patients for bleaching and evaluated their teeth against the shade guide at baseline and 30 days after the procedure. The two examiners were required to have an agreement of at least 85% (Kappa statistics) before beginning the study evaluation.[5] The shade comparison before and after treatment is given by the difference between the baseline and 30-day shades (ΔSGU).

For the objective evaluation, a preliminary impression of the maxillary arch was made using dense silicone Adsil (Vigodent S/A Ind Com, Rio de Janeiro, RJ, Brazil). The impression was extended to the maxillary canine and served as a standard shade measurement guide for the spectrophotometer. A window was created on the labial surface of the molded silicone guide for the central incisor to be evaluated. The window was made using a metal device with well-formed borders and radius of 3 mm.[3,16] The measurement was made by only one operator, in all 30 patients, using Vita Easyshade (Easyshade, Vident) before and 30 days after the bleaching therapy. The shade was determined using the parameters of the Easyshade device, which indicated the following values: L*, a* and b*, in which L* represents the value from 0 (black) to 100 (white) and a* and b* represent the shade, where a* is the measurement along the red-green axis and b* is the measurement along the yellow-blue axis. The shade comparison before and after treatment is given by the differences between the two shades (ΔE),[3,16] which is calculated using the formula $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$.

Tooth Sensitivity Evaluation

The patients recorded their perception of tooth sensitivity during the first and second bleaching sessions using two pain scales. A five-point verbal rating scale (0 = *none*, 1 = *mild*, 2 = *moderate*, 3 = *considerable*, and 4 = *severe*)[4,5] and a visual analog scale[17-19] (using a 10-cm horizontal line with words *no pain* at one end and *worst pain* at the opposite end) were used in this study. The subjects were asked to record whether they experienced sensitivity during the treatment up to one hour after the bleaching, from one hour to 24 hours and from 24 hours to 48 hours after bleaching, individually, for both maxillary and mandibular arches. As two bleaching sessions were performed, the worst score/numerical value obtained in both bleaching sessions was considered for statistical purposes. The values were arranged into two categories: overall percentage of patients who reported tooth sensitivity at least once during treatment, regardless of the assessment point (absolute risk of tooth sensitivity), and tooth sensitivity intensity at each of the assessment points. These values were computed for both the maxillary and mandibular arches.

Table 1: *Comparison of the Number of Patients Who Experienced Tooth Sensitivity on the Maxillary and Mandibular Arches During the Bleaching Regimen in Both Groups Along With Absolute and Relative Risks*[a]

Treatment	Maxillary Arch				Mandibular Arch			
	Tooth Sensitivity (No. of Participants)		Absolute Risk (95% CI)	Relative Risk (95% CI)	Tooth Sensitivity (No. Participants)		Absolute Risk (95% CI)	Relative Risk (95% CI)
	Yes	No			Yes	No		
Placebo	12	3	80 (55-93)	0.92 (0.67-1.3)	14	1	93 (70-98)	1.16 (0.87-1.6)
Ibuprofen	13	2	87 (62-96)		12	3	80 (54-93)	

[a] *Fisher test,* p=*1.0 for maxilla and* p=*0.59 for mandibular arches.*

Statistical Analysis

The analysis followed the intention-to-treat protocol and involved all participants who were randomly assigned.[15] The statistician was blinded to the study groups. The agreement between examiners' objective evaluation was evaluated using the kappa statistics. The primary outcome of absolute risk of tooth sensitivity was compared using Fisher exact test (α=5%). The relative risk and the confidence interval for the effect size were calculated.

Tooth sensitivity intensity (secondary outcome) was also statistically analyzed. The mean/median and standard deviation/interquartile range of the two pain scales were calculated. The data sets of tooth sensitivity intensity were plotted on histograms and inspected for normal distributions. Some data did not appear to be normally distributed. Therefore, nonparametric statistical tests were used to compare the various treatments. Statistical analyses of two pain scales comparing the two groups at the three different assessment points were performed using the Mann-Whitney U test. Comparisons between times within each group were performed using the Friedman tests. In all statistical tests, the significance level was 5%.

The data from the placebo group were used to compare the absolute risk of tooth sensitivity and its intensity between the maxillary and mandibular arches. For the former, Fisher exact test at a level of 5% was used. For the latter, the Mann-Whitney U-test was used at the same level of significance.

Color change, another secondary endpoint, was used to assess the efficacy of the bleaching treatment associated with the perioperative use of ibuprofen. The ΔSGU, ΔL, Δa, Δb, and ΔE values of both groups were evaluated by Student *t*-test. In all statistical tests, the significance level was set at alpha of 5%.

RESULTS

The mean age (years) of the participants in this study was similar between the groups (placebo: 26.4 $\pm$ 6.8, and ibuprofen: 32.9 $\pm$ 9.9 years). Fifty-three and 33% of the participants from the placebo and ibuprofen groups were male. Figure 1 depicts the participant flow diagram in the different phases of the study design.

Tooth Sensitivity

The data from 30 patients were used in this study, following the intention-to-treat analysis. In regard to the absolute risk of tooth sensitivity (primary outcome), no significant difference was observed between groups, as seen in Table 1 (p=1.00 for maxilla and p=0.59 for mandibular arches). The relative risk, along with the 95% confidence interval, is also evidence that the use of the experimental drug had no effect on the tooth sensitivity reduction.

In the comparison between the dental arches, no significant difference was detected for both groups, with regard to the absolute risk of tooth sensitivity ($p>0.05$) and tooth intensity ($p>0.05$).

Most of the tooth sensitivity complaints occurred within the first 24 hours ($p<0.001$), and none of the participants experienced pain after 24 hours. When using the visual analogue scale, the tooth sensitivity intensity (Table 2) was less intense for ibuprofen than the placebo group in both arches only up to one hour (p=0.04 for maxillary and p=0.008 for mandibular arches). This was also observed for the five-point verbal rating scale in the mandibular arch (p=0.006), but not in the maxillary arch, which showed no statistical difference between groups (p=0.06).

Significant whitening was observed in both study groups by means of the subjective and objective evaluation methods ($p<0.001$). Whitening of approximately 5.3 and 4.3 shade guide units was detected for placebo and ibuprofen groups, respectively (Table 3), and a variation of 7.8 to 5.9 in the ΔE was observed for the placebo and ibuprofen groups, respectively. The results of the subjective (visual shade guide) and the objective evaluation (spectro-

Table 2: *Comparison of Tooth Sensitivity Intensity Experienced on the Maxillary and Mandibular Anterior Teeth by Patients From the Treatment Groups at Different Assessment Points Using the Pain Scales*[a]

	Five-Point Verbal Rating Scale (0-4)[b]				Visual Analog Scale (0-10)[c]			
	Maxillary Arch		Mandibular Arch		Maxillary Arch		Mandibular Arch	
	Placebo	Ibuprofen	Placebo	Ibuprofen	Placebo	Ibuprofen	Placebo	Ibuprofen
Up to 1 hour	2 (1/3)^{aA}	1 (0/2)^{aA}	3 (1/4)^{aA}	1 (0/2)^{bA}	3.3 ± 2.9^{aA}	1.5 ± 2.0^{bA}	4.3 ± 3.6^{aA}	1.5 ± 2.4^{bA}
1 hour to 24 hours	2 (0/2)^{aB}	2 (1/3)^{aA}	2 (0/3)^{aB}	2 (1/3)^{aB}	2.4 ± 2.7^{aB}	3.0 ± 2.8^{aB}	3.7 ± 3.6^{aA}	3.2 ± 3.0^{aB}
24 hours to 48 hours	0 (0/0)^{aC}	0 (0/0)^{aB}	0 (0/0)^{aC}	0 (0/0)^{aC}	0.0 ± 0.0^{aC}	0.0 ± 0.0^{aC}	0.0 ± 0.0^{aB}	0.0 ± 0.0^{aC}

[a] *Comparisons are valid only within the same pain scale. At each period, the two treatments were compared with the Mann-Whitney U-test, and differences are represented by different superscript lowercase letters. For each treatment, the three periods were compared with the Friedman test (α=0.05), and differences are represented by different superscript uppercase letters.*
[b] *Medians (first/third interquartile) values.*
[c] *Means and standard deviations.*

photometer) matched the hypothesis of equality between the groups after bleaching ($p \approx 0.6$ for both methods).

DISCUSSION

It has been reported that tooth-whitening solution applied to human mandibular incisors was shown to cause structural damage to the pulp, such as disruption of the odontoblast layer and the production of inflammatory infiltrates.[8] Tissue damage triggers the creation of bradykinin[20] and prostaglandins.[21,22] Each of these compounds either activates or sensitizes nociceptors and causes pain.[20,21]

The category of prostaglandins, which has been known to play a critical role in the pathogenesis of pulpal disease, involves the COX pathway. There are at least two variants of COX, the COX-1, which is involved in physiological functions and inducible COX-2, which is believed to be involved in the inflammatory response[14] and has been responsible for the production of prostaglandins mediating inflammation and pain due to pulp inflammation.[21]

Table 3: *Change Between Baseline and 30-Day Assessment (Means and Standard Deviations) for ΔSGU, ΔL, Δa, Δb, and ΔE for the Two Treatment Groups*[a] *for the Maxillary Arch*

	Placebo	Ibuprofen
Subjective evaluation (delta visual shade guide)		
Δ shade guide units	5.3 ± 1.9 A	4.3 ± 2.7 A
Objective evaluation (spectrophotometer)		
CIELab		
ΔL	2.2 ± 1.6 A	0.7 ± 4.4 A
Δa	−1.3 ± 1.4 A	−0.7 ± 0.8 A
Δb	−7.0 ± 3.2 A	−3.9 ± 1.6 B
ΔE	7.8 ± 3.3 A	5.9 ± 2.1 A

[a] *Comparisons are valid only within rows. Similar uppercase letters indicate statistically similar means (Student t-test, α=0.05).*

Ibuprofen is a nonsteroidal anti-inflammatory drug, and its mechanism of action results from acetylating the COX enzyme, which in turn inhibits the synthesis of prostaglandins.[22] However, contrary to one's previous expectation, the use of a nonsteroidal anti-inflammatory drug (ibuprofen 400 mg) in a preventive approach was not capable of avoiding the tooth sensitivity arising from bleaching. The lack of efficacy of ibuprofen in preventing tooth sensitivity could be that several other inflammatory mediators,[22] apart from COX-1 and COX-2, are probably involved in the inflammatory reaction in the pulp tissue that leads to pain and symptoms of neurogenic inflammation.

For instance, bradykinin[20] and substance-P have long been known to be involved in the process of pulp pain and inflammation.[23] Unfortunately, ibuprofen cannot prevent the production of these mediators. If many mediators act synergistically to produce both pain and inflammatory reaction after bleaching, an anti-inflammatory drug that inhibits several initial inflammatory events, such as the glucocorticoids,[24] may be more effective in reducing tooth sensitivity arising from bleaching. Chrousos and others[25] reported that some side effects have been reported with short-term oral glucocorticoid therapy such as insomnia, mild mood changes, stomach upset, facial flushing, and weight gain; however, these events are not often observed when glucocorticoid therapy is performed in young, healthy adults, and it is considered a relatively safe procedure. This strategy should be a focus of future clinical investigations that attempt to reduce tooth sensitivity caused by bleaching.

Dental pulp is densely innervated with sensory afferents with conduction velocities in the A"β, Aδ, and C-fiber range.[26] Thus, hydrogen peroxide may cause direct cellular damage to nerve cells via free radicals and other reactive oxygen species[27] produc-

ing lipid peroxidation of membrane protein and nucleic acid oxidation.[28,29] If this is the cause of tooth sensitivity, the use of an anti-inflammatory drug, such the one investigated in the present study, would not reduce the pain experience.

It is worth mentioning, however, that the intensity of tooth sensitivity in the ibuprofen group was lower during the first hour after bleaching in the present investigation, similar to the findings observed in the study by Charakorn and others.[13] This means that this medicine can be used to reduce the intensity of tooth sensitivity only during and immediately after the bleaching session.

However, no significant difference between groups was observed in the following assessment points. In Charakorn and others' study,[13] this finding was explained by the decreasing amount of ibuprofen in patients' serum as time passes. However, the results of the present investigation do not support this hypothesis since ibuprofen was administered in six doses during a period of 48 hours after bleaching and not in a single dose, as in Charakorn and others' study.[13] This finding suggests that the prostaglandins produced by COX[14] probably occur in the first hours after bleaching. As time goes by, other inflammatory mediators, not inhibited by the ibuprofen, may be expressed and trigger the tooth sensitivity.

Costa and others[8] in a histological pulp evaluation after bleaching, showed notable damage to the pulp tissue of mandibular incisors but not premolars. In a literature review, Haywood[30] reported that tooth sensitivity from bleaching usually affects the smaller teeth, such as the maxillary lateral incisors and the mandibular incisors, although the present clinical study found no significant difference in tooth sensitivity intensity or in the absolute risk of tooth sensitivity between dental arches; in other words, the tooth sensitivity may be related to the thickness of the hard dental tissues rather than to the arch itself. Further studies should be conducted to identify the most painful teeth and follow up their vitality in the long run after bleaching, suggesting that tooth sensitivity intensity may be a reliable signal of significant changes in the pulp tissue.

With regard to the bleaching outcome, the results of this study indicated that both groups demonstrated equivalent and significant tooth shade enhancement when compared with baseline values (Table 3). It is difficult to make a comparison of shade change after in-office bleaching with studies in the existing literature, because of the different methods of measurement (shade guides and spectrophotometer) and different units of measurement (CIELab system, shade guide units, etc) used. However, studies that used 35% hydrogen peroxide and reported their results in shade guide units usually observed an overall shade change of five to eight shade guide units after two bleaching sessions,[4,5,6] which is in agreement with the results of the present investigation.

Finally, one could not ignore that the small sample size of this study is a clear limitation of the present study. The study was designed to find a high effect size, that is, reduction in 50% of the tooth sensitivity among participants in the experimental group. Thus, it can be concluded that an effect as large as this was not observed, but one cannot rule out the fact that smaller effect sizes do exist. Conducting the same experimental design using larger sample sizes should be encouraged to rule out this hypothesis. Moreover, the selected sample was mainly composed of young participants, which also limits the ability to generalize for older adults.

CONCLUSION

The perioperative use of ibuprofen 400 mg for a period of 48 hours, starting one hour before in-office bleaching treatment, does not reduce the absolute risk of tooth sensitivity but may reduce the tooth intensity up to one hour after the bleaching session without jeopardizing the whitening effect.

Acknowledgments

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Laboratory Research

Effect of a Chlorhexidine-containing Adhesive on Dentin Bond Strength Stability

C Sabatini

Clinical Relevance

A novel chlorhexidine-containing adhesive may offer comparable dentin-resin bond stability to the use of 2.0% chlorhexidine digluconate applied as a therapeutic primer while also providing a simplified clinical application technique.

SUMMARY

Purpose: The present study aimed to investigate a novel adhesive system containing 0.2% chlorhexidine digluconate (CHX) for its ability to improve the stability of the adhesive interface compared with the use of 2% CHX as a therapeutic primer. Furthermore, the study aimed to confirm the inhibitory properties of these CHX concentrations (0.2% and 2.0%) on dentin matrix metalloproteinase activity by gelatin zymography.

Methods: Superficial dentin substrate for bonding was obtained from 120 non-carious human molars. A conventional adhesive Peak LC Bond and a CHX-containing adhesive Peak Universal Bond were used either in combination with 35% phosphoric acid (etch-and-rinse approach) or with self-etching primer (self-etch approach) for evaluation of the variables CHX treatment (2.0% therapeutic primer and 0.2% adhesive), adhesive approach (etch-and-rinse and self-etch), and storage time (24 hours and six months). A bonding jig was used to fabricate composite cylinders, which were stored for either 24 hours or six months, after which shear bond strength (SBS) was evaluated using a notched-edge testing device. A three-way analysis of variance and a Student *t*-test with a significance level of $p<0.05$ were used to analyze the data. Extracts from concentrated demineralized human dentin powder were subjected to sodium dodecyl sulfate polyacrylamide gel electrophoresis and incubated in the presence of 0.2% and 2.0% CHX.

Results: No significant effect of CHX treatment, adhesive approach, storage time variables, or their interactions on mean SBS was

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demonstrated ($p<0.05$). No significant difference between the control and the CHX-treated groups was detected for either adhesive technique at 24 hours or six months ($p<0.05$). No significant variation in mean SBS was detected after six months of storage ($p<0.05$). Zymographic analysis revealed bands of enzymatic activity for the group demineralized with phosphoric acid and complete inhibition of gelatinolytic activity for the groups treated with 0.2% and 2.0% CHX.

Conclusions: CHX demonstrated inhibition of dentin proteolytic activity. However, when CHX was incorporated into a commercially available adhesive or used as a therapeutic primer, no difference in bond strength was observed at baseline or after six months of storage relative to the control group without CHX.

INTRODUCTION

Despite improvements made to the chemistry of contemporary dentin adhesives, resin-dentin interfaces still deteriorate over time.[1-7] A correlation between the presence of active endogenous matrix metalloproteinases (MMPs) in dentin matrices and premature degradation of hybrid layers has been demonstrated.[8,9] Different forms of MMPs have been identified in dentin matrices by zymographic analyses, with MMP-2 and MMP-9 being the most prevalent.[8,10,11] When exposed to an acidic environment, such as the one created by the application of acidic adhesive resins, these enzymes, initially secreted as pro-enzymes, become active proteinases.[12] These host-derived MMPs have been shown to degrade sub-optimally infiltrated collagen fibers once they have been activated through bonding procedures.[9,13]

Currently, bonding to dentin can be achieved through an etch-and-rinse (ER) or a self-etch (SE) approach. Etch-and-rinse adhesives have a pH too high (2.5-4.5) to etch through the smear layer and underlying dentin, hence the requirement for a separate acid-etching step with 32%-37% phosphoric acid (0.1-1.0). Subsequent infiltration of the resin monomers into the demineralized dentin matrix typically leaves collagen incompletely encapsulated at the bottom of the hybrid layer,[14] which is then susceptible to proteolytic degradation by host-derived MMPs. Self-etch adhesives, with a higher pH (1.0-2.7), can still etch through the smear layer. Despite the shallow etching pattern, optimal hybridization and high bond strengths have been reported with these formulations.[15] Activation of precursor MMP forms has been demonstrated with both ER[16] and SE[12, 13] adhesive systems.

Chlorhexidine digluconate (CHX), a cationic antimicrobial agent, has demonstrated successful inhibition of dentin MMP collagenolytic activity in zymographic studies when used in concentrations of 0.2% and 2.0 %.[17-19] Clinically, reduced degradation of the dentin-adhesive interface has been shown with the use of 2% CHX as a therapeutic primer before the application of the adhesive *in vivo*[20-24] and *in vitro*.[18,25-28] However, this represents an additional step in the bonding sequence. Recently, alternative approaches incorporating CHX into the phosphoric acid or into the primer/adhesive formulation have been proposed to improve its retention and effectiveness while simplifying the clinical application technique. A study demonstrated reduced bond degradation after six months when 2% CHX was incorporated into a conventional 37% phosphoric acid.[29] Incorporation of CHX into the primer has shown conflicting results of improved stability,[30] but also no effect,[31] on the stability of the adhesive interface after one year. The data on the subject are inconclusive.

Therefore, the objective of the present study was to investigate a novel adhesive system containing 0.2% CHX for its ability to improve the stability of the adhesive interface over time compared with the use of 2.0% CHX as a therapeutic primer. Specific aims of our study included the following: 1) to evaluate dentin shear bond strength (SBS) of the novel adhesive containing 0.2% CHX when applied either as an ER or an SE approach at both 24 hours and six months, compared with the topical application of 2.0% CHX; and 2) to confirm the previously demonstrated inhibitory properties of 0.2% and 2.0% CHX on dentin MMP activity by gelatin zymography. The following null hypotheses were evaluated: 1) there would be no difference in SBS between groups treated with the novel adhesive containing 0.2% CHX, topical 2.0% CHX, and use of no CHX; and 2) there would be no difference in bond degradation after six months for either group.

MATERIALS AND METHODS

Zymographic Analysis

Dentin Extraction—Six healthy human molars from donors of unknown age were obtained under a protocol approved by the State University of New York's Institutional Review Board and stored in a 0.5% NaCl solution containing 0.02% sodium azide

at 4°C for no more than one month after extraction. The crowns were separated from the roots at the cement-enamel junction. Coronal dentin blocks were obtained after complete removal of the enamel and pre-dentin tissues by means of a diamond bur in a high-speed handpiece under air-water spray. A slow-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) was used to obtain smaller dentin sections, which were frozen in liquid nitrogen and triturated into fine dentin powder in a high-speed mixer mill (MM400, Retsch, Newton, PA, USA) at 30 Hz for 10 minutes. Fine dentin powder (4 g) was obtained from six molar crowns. The powder was stored dry at −70°C until ready to use.

Aliquots of 1 g each of dentin powder were randomly assigned to one of four groups: 1) mineralized dentin (control); 2) demineralized dentin; 3) demineralized dentin incubated in 0.2% CHX; and 4) demineralized dentin incubated in 2.0% CHX. Groups 2-4 were demineralized by mixing the dentin powder with a 1% phosphoric acid water solution for 10 minutes. The four groups received five rinses of 1 mL of distilled water to eliminate acid remnants. To extract the protein content from the dentin powder, precipitates were re-suspended in 4 mL of extraction buffer (50 mM Tris-HCL, pH 6.0 with 5 mM $CaCl_2$, 100 mM NaCl, 0.1 mM $ZnCl_2$, 0.1% Triton X-100, 0.1% Triton X-114, 0.02% NaN_3) containing a protease inhibitor free of ethylenediamine tetra-acetic acid (Rosche Diagnostics, Indianapolis, IN, USA) for 24 hours at 4°C per Breschi's protocol.[18] The vials were centrifuged at 8,500 rpm (Sorvall RC 6 Plus Centrifuge, Thermo Scientific, Asheville, NC, USA) for 15 minutes at 4°C, and the supernatants were carefully collected by aspiration. Protein content was precipitated with 25% trichloroacetic acid and centrifuged at 8,500 rpm for 10 minutes at 4°C. The precipitate was stored at −70°C until ready to use for zymographic analysis.

Gelatin Zymography—To assess gelatinolytic activity, concentrated samples from dentin powder protein extracts were electrophoresed under non-reducing conditions on a 7.5% sodium dodecyl sulfate-polyacrylamide gel electrophoresis containing 2g/L gelatin substrate. The precipitates were re-solubilized in loading buffer (2% sodium dodecyl sulfate [SDS]; 125mM Tris-HCL, pH 6.8; 10% glycerol; and 0.001% bromophenol blue), and the samples were separated by electrophoresis for 30 minutes at 300 V, 32 mA, and 6 W. After electrophoresis, the gels were washed in 2.5% Titron X-100 for 15 minutes to remove SDS and activated with 2 mM p-aminophenylmercuric acetate for one hour at 37°C. After activation, Groups 3 and 4 were incubated in aqueous solutions of 0.2% and 2.0% CHX, respectively, for 30 minutes. All groups were incubated in zymography buffer ($CaCl_2$, NaCl, and Tris-HCL, pH 8.0) for 48 hours at 37°C to allow development of enzymatic activity. The gels were then stained in 0.2% Coommassie Brilliant Blue, and de-stained in de-staining solution (50% methanol, 40% acetic acid, and 10% water). Gelatinolytic activity was evidenced as unstained bands on a blue-stained background.

Shear Bond Strength

One hundred and twenty recently extracted, non-carious human molars were used to obtain superficial dentin substrate for bonding. The crowns were separated from the roots with a slow-speed diamond saw and embedded in a chemically polymerized methacrylate (Fastray, HJ Bosworth, Skokie, IL, USA) with the facial surface exposed and ground flat on a model trimmer to reveal superficial dentin, which was finished with 320-, 400-, and 600-grit silicon carbide abrasive paper (Buehler). The specimens were stored in deionized water at 4°C until ready to be used. One hour before bonding, the specimens were acclimatized to room temperature (23 ± 2°C) and refinished with 600-grit abrasive paper to expose fresh dentin.

Two adhesive resins were used in this study, a conventional adhesive (Peak LC Bond, Ultradent, South Jordan, UT, USA) and a novel adhesive containing 0.2% CHX (Peak Universal Bond, Ultradent). Both are 7.5 % nanofilled, phosphated, adhesives containing hydroxyethyl methacrylate with ethyl alcohol as the carrier, and can be used either in combination with 35% phosphoric acid (Ultra-Etch, Ultradent) for a two-step ER technique or with self-etching primer (Peak SE primer, Ultradent) for a two-step SE technique. The study variables included CHX treatment (2.0% therapeutic primer and 0.2% adhesive), adhesive approach (ER and SE), and storage time (24 hours and six months). Specimens were equally and randomly assigned to six groups (Table 1) with a sample size of 10 as follows: Group 1, phosphoric acid treatment followed by conventional adhesive (PA+PLC, ER control); Group 2, phosphoric acid treatment followed by topical 2% CHX and conventional adhesive (PA+CHX+PLC); Group 3, phosphoric acid treatment followed by CHX-containing adhesive (PA+PU); Group 4, self-etching primer followed by conventional adhesive (PSE+PLC, SE control); Group 5, topical 2% CHX followed by self-etching

Table 1: *Study Groups, Category, and Application Procedures per Manufacturer Recommendations*[a]

Group	Description	Code	Category/Application Procedure
ER technique			
1	35% H_3PO_4 followed by conventional adhesive resin (control)	PA+PLC	• Apply 35% H_3PO_4 (15 s); rinse (5 s); leave dentin moist • Scrub Consepsis onto moist dentin and air dry (Group 2 only) • Scrub adhesive onto dentin (10 s); gently air dry to leave a thin uniform layer (10 s) • Polymerize (10 s if >600 mW/cm^2; 20 s if <600 mW/cm^2)
2	35% H_3PO_4 followed by rewetting with 2% CHX and conventional adhesive resin	PA+CHX+PLC	
3	35% H_3PO_4 followed by CHX-containing adhesive resin	PA+PU	
SE technique			
4	Self-etching primer followed by conventional adhesive resin (control)	PSE+PLC	• Scrub Consepsis onto moist dentin and air dry (Group 5 only) • Scrub Peak SE primer onto moist dentin (20 s); thin/dry (3 s) • Scrub adhesive onto dentin (10 s); gently air dry to leave a thin uniform layer (10 s) • Polymerize (10 s if >600 mW/cm^2; 20 s if <600mW/cm^2)
5	2% CHX followed by self-etching primer and conventional adhesive resin		
	CHX+PSE+PLC		
6	Self-etching primer followed by CHX-containing adhesive resin	PSE+PU	

Abbreviations: ER, etch-and-rinse; SE, self-etch; CHX, chlorhexidine; PA, phosphoric acid; PLC, Peak LC Bond; PU, Peak Universal Bond; PSE, Peak SE Primer.
[a] *Self-etching primer (Peak SE Primer); conventional adhesive resin (Peak LC Bond); CHX-containing adhesive resin (Peak Universal Bond); aqueous solution of 2% CHX (Consepsis , Ultradent, South Jordan, UT, USA).*

primer and conventional adhesive (CHX+PSE+PLC); Group 6, self-etching primer followed by CHX-containing adhesive (PSE+PU).

The adhesives were applied and polymerized according to manufacturer's instructions with a light-emitting diode light-curing unit (Bluephase 16i, Ivoclar-Vivadent, Amherst, NY, USA) with a power density of 1,600 mW/cm^2. The specimens were placed on a bonding jig (Ultradent) with a cylindrical mold of standardized dimensions (2.38 mm in diameter and 2 mm in height). Composite cylinders were fabricated with resin composite (Filtek Z100, 3M ESPE, Lot# N196007, St Paul, MN, USA) in shade A2 by application of only one increment no greater than 2 mm and polymerized for 20 seconds. The specimens were stored in distilled water containing 0.02% sodium azide at 37°C for either 24 hours or six months, after which SBS was evaluated. A calibrated testing device (Ultratester, Ultradent) loaded at a crosshead test speed of 1mm/min and a load cell of 1,000 lb (453.6 kg) was used. A notched-edge crosshead matching the diameter of the bonded cylinder was used to apply the testing load. The load required to debond the specimen was recorded and expressed in megapascals (MPa), and descriptive statistics were determined.

Because the data were normally distributed (Kolmogorov-Smirnov test), a three-way analysis of variance (ANOVA) was used to analyze the effect of the variables CHX treatment, adhesive approach, and storage time. Student *t*-tests were used to evaluate differences between 24 hours and six months for each of the individual groups. A significance level of $p<0.05$ was used for all tests. All statistical analyses were performed with the Statistical Package for Social Sciences (SPSS) version 16.0 (SPSS Inc, Chicago, IL, USA).

Analysis of the Mode of Failure—Modes of failure were analyzed by observation by a single trained examiner (C.S.) with a stereomicroscope (Nikon SMZ-U, Melville, NY, USA) at a magnification of 50×. Representative images of the different failure modes were recorded with a field emission scanning electron microscope (Hitachi SU-70, Hitachi, Kre-

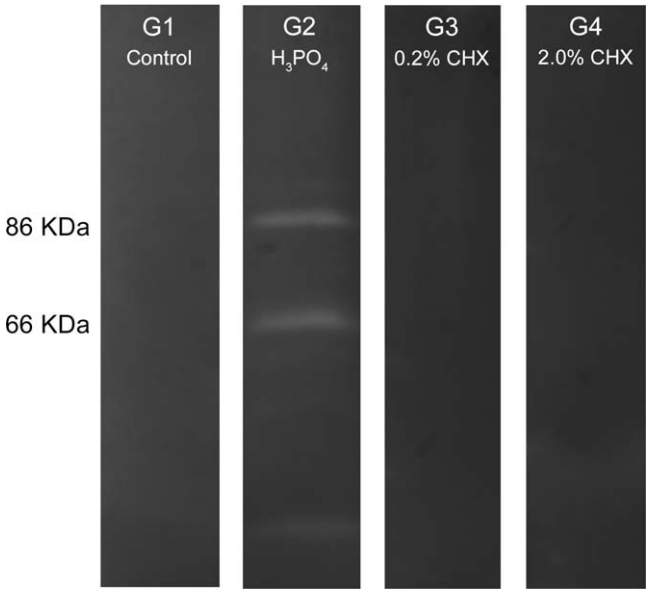

Figure 1. *Gelatin zymogram from concentrated dentin protein extracts. (G1) Mineralized dentin (control). (G2) Demineralized dentin in H_3PO_4 (bands correspond to MMP-2 and MMP-9 isoforms). (G3) Demineralized dentin in H_3PO_4 and incubated in 0.2% CHX. (G4) Demineralized dentin in H_3PO_4 and incubated in 2.0% CHX.*

feld, Germany) in backscattered electron mode at 50×. The fractured surfaces were classified as follows: 1) cohesive in dentin, 2) adhesive, 3) cohesive in composite, and 4) mixed failure, defined as the combination of different failure modes resulting from failure across the interfacial layers.

RESULTS

Gelatin Zymography

When protein extracts were analyzed by gelatin zymography (Figure 1), no activity was detected for the control group with untreated mineralized dentin powder. Bands of enzymatic activity were evidenced for the group that was demineralized with phosphoric acid but received no further treatment with inhibitor (Group 2). These bands corresponded to active MMP-2 and MMP-9 isoforms. Complete inhibition of enzymatic activity was shown with both 0.2% and 2.0% CHX (Groups 3 and 4, respectively).

Shear Bond Strength

Three-way ANOVA (Table 2) revealed no significant effect of the variables CHX treatment, adhesive approach, storage time, and any of their interactions on mean SBS values ($p<0.05$). Table 3 summarizes the mean SBS values and failure mode distribution for all study groups at 24 hours and six months of storage. No significant difference between the control and the CHX-treated groups was detected for either adhesive technique at 24 hours or six months ($p<0.05$). Student *t*-test comparisons between 24 hours and six months for each of the individual groups revealed no significant variation in mean SBS values after six months of storage ($p<0.05$). Mixed-type fracture was the most prevalent type of failure mode observed, irrespective of CHX treatment, adhesive approach, or storage time (Table 3).

DISCUSSION

The present study evaluated the effect of incorporating 0.2 % CHX into a commercially available adhesive blend, used either as an ER or SE approach, compared with the topical application of 2% CHX. Both null hypotheses were accepted as no differences were observed among treatment groups at either testing period; nor were differences in bond degradation observed after six months for each of the individual groups. Because the two main concentrations of CHX evaluated in this study were 0.2% and 2.0%, zymographic analysis was also conducted to confirm the previously demonstrated inhibitory properties of CHX.[17-19] Our results are in agreement with previous studies, which have shown activation

Table 2: *Three-way ANOVA Results*

Source of Variation	DF	SS	MS	F	P
Adhesive	2	15.713	7.856	0.0728	0.930
Composite	1	117.216	117.216	1.086	0.300
Time	1	316.875	316.875	2.936	0.089
Adhesive × composite	2	142.229	71.114	0.659	0.519
Adhesive × time	2	23.994	11.997	0.111	0.895
Composite × time	1	73.320	73.320	0.679	0.412
Adhesive × composite × time	2	185.729	92.864	0.861	0.426
Residual	108	11654.604	107.913		
Total	119	12529.680	105.291		

Abbreviations: DF, degrees of freedom; SS, sum of squares; MS, mean squares; F; f obtained; P, *probability.*

Table 3: *Mean SBS Results and Failure Mode Distribution for the Six Study Groups at 24 hours and Six Months of Storage*[a]

	Group	Storage	SBS (MPa)	Failure mode (%)			
			Mean ± SD	A	D	R	M
ER	1	24 h	40.6 ± 15.7	30	10	0	60
		6 mo	46.4 ± 7.4	0	30	0	70
	2	24 h	40.5 ± 8.5	10	30	0	60
		6 mo	42.2 ± 11.9	0	40	10	50
	3	24 h	46.0 ± 7.2	0	20	0	80
		6 mo	43.6 ± 11.1	0	20	20	60
SE	4	24 h	39.5 ± 9.9	20	30	10	40
		6 mo	42.0 ± 10.5	0	10	10	80
	5	24 h	39.6 ± 11.1	10	20	20	50
		6 mo	45.1 ± 6.4	0	30	10	60
	6	24 h	37.4 ± 11.7	0	30	20	50
		6 mo	43.9 ± 9.8	10	20	0	70

Abbreviations: ER, etch-and-rinse; SE, self-etch; A, adhesive; D, cohesive in dentin; R, cohesive in resin; M, mixed; SBS, shear bond strength.
[a] *No significant differences in mean SBS among groups for either storage period ($p<0.05$). No significant differences in mean SBS between baseline and six months for each of the individual groups ($p<0.05$).*

of endogenous dentin MMPs after treatment with phosphoric acid.[32] Furthermore, both 0.2% and 2.0% CHX demonstrated complete inhibition of dentin proteolytic activity as determined by gelatin zymography validating previous studies.[17-19]

Chlorhexidine, applied either topically or incorporated into the adhesive, did not appear to affect bond strength for either adhesive approach at 24 hours or six months, suggesting that the antimicrobial may be safely combined with the resin monomers contained in the adhesive tested. Our results confirm those from previous studies, which have shown that CHX, applied topically[20,26-28] or into the primer,[30,31] has no effect on the immediate bond strength. Moreover, CHX, applied either topically or into the adhesive, did not appear to affect the stability of the bonds over time, as demonstrated by the non-significant differences in mean bond strength values between 24 hours and six months for each of the individual groups. A number of studies evaluating the stability of adhesive interfaces treated with CHX have demonstrated favorable results of reduced bond degradation.[23-28] However, these studies primarily report on the topical use of CHX before adhesive application; thus, direct comparisons of these results and those from our study are not possible as our study aimed to investigate CHX when incorporated into an adhesive blend rather than topically between the conditioning and adhesive application. Recently, incorporation of CHX into the primer or the adhesive blend has been proposed to optimize its retention and effectiveness while simplifying the clinical application procedures. Conflicting results of improved stability,[30] but also no effect[31] on the stability of adhesive interfaces, have been reported when CHX was admixed into the primer. Similarly, fair comparisons between these results and those from our study are not possible as differences in study methodology, materials evaluated, and primarily in

the actual vehicle for the delivery of CHX vary among studies. The vehicle for the delivery of CHX in our study was the actual adhesive resin rather than the primer. We speculate that this may have limited its interaction with the hybrid layer where MMP inhibition is required. However, even when incorporated into the primer, CHX inhibitory properties may be of limited duration because of its unknown substantivity to dentin. A recent study demonstrated outstanding substantivity of CHX to human dentin[33]; however, results only up to two months of storage were reported in this study, and thus, the long-term role of CHX in the preservation of the bonds requires further investigation when incubated over longer storage periods. Further research is also needed to better understand the anti-proteolytic effects and substantivity properties of CHX and other proposed synthetic inhibitors, so that their use can be optimized, leading to longer-lasting adhesive restorations.

Despite the several benefits of CHX, the inability to stabilize the water soluble, non-covalently bound compound in the adhesive interface may limit its long-term anti-proteolytic benefit.[34] At the same time, polymer plasticization that may further expose collagen fibrils over time requires that MMP inhibiting agents remain available at the interface for effective anti-proteolytic properties over the long-term life of the restoration. Attempts to stabilize CHX into the resin matrix have been proposed by incorporating the compound into the adhesive blend with the belief that the resin matrix can act as a reservoir for the slow release of CHX over time. However, extensive testing of the specific interactions between CHX and the specific adhesive monomers in the different adhesive blends should precede such mixtures, as adverse effects on the adhesive mechanical properties,[35] which may outweigh its anti-proteolytic benefit, may be derived from incorporating CHX into the adhesive blend. Moreover, the nature of the interaction between CHX and adhesive is known to be product specific as it depends on the specific monomeric composition. A study showed that dissolving increasing concentrations of CHX into resin blends with different levels of hydrophobicity decreased their elastic modulus by 27%-48%.[35] Degradation of adhesive interfaces is known to be the combined result of polymer plasticization and collagenolytic activity by host-derived dentin MMPs. Thus, a polymeric network with lower elastic modulus is presumably more susceptible to early plasticization and, as a consequence, to premature failure. Future studies should be undertaken to evaluate which types of resin can be safely combined with this antimicrobial and with other anti-proteolytic agents.

Other aspects may also have contributed to the observed results of non-significant differences between the control and CHX-treated groups in the present study. A storage time of six months may not have been sufficient to detect the effects of hydrolytic degradation of the adhesive interface if the larger surface area of the specimens used for SBS tests is taken into consideration. A study by Kiyomura[36] reported that storage times between 2 and 4 years were required to detect the effects of hydrolytic degradation for specimens of large surface area, such as those used in SBS tests, because of the required longer diffusional distances from the cavosurface margin. Compared with microtensile tests, SBS tests are also known to be less discriminating in their ability to detect differences, perhaps requiring considerably larger sample size to be able to detect differences. A recent review by Braga and others[37] reported that of 100 recently conducted bond strength studies, 59% used a sample size of 10, 15% used a sample size between five and eight, and 26% used a sample size between 11 and 25. In our study, a sample size of 10 was used and no significant differences were detected among groups. A power analysis revealed that a sample size greater than 30 would have been able to detect differences in the range of 4 MPa. However, considering the mean SBS range observed in our study (~40 MPa), a difference of 4 MPa may not be of great clinical relevance. Nonetheless, SBS tests remain useful in the preliminary screening of adhesives, and their simplicity warrants consideration. Useful information can be derived from these data provided that the limitations of the test are understood and its results not overemphasized.

In our study, the most prevalent type of failure mode was mixed type, which, considering the overall high mean bond strength values observed in our study, the type of test, and what is known about the stress distribution created by SBS testing, is not surprising. In general, it is known that in the presence of strong bonds, the fracture path starts in resin composite propagating across the adhesive joint and then into dentin.[38] This suggests that the quality of the bond between the materials present at the interface is such that it surpasses the cohesive strength of its individual components and the adhesive strength between the interfacial layers, yielding an adhesive interfacial assembly that exhibits greater strength when acting as a single

body rather than as separate layers. Moreover, the reported fracture modes may only be considered "apparent" as confirmation of "true" failure modes would require the use of sophisticated surface chemistry analysis instead of only high-magnification microscopic evaluation.[39]

Ongoing efforts continue toward gaining a better understanding of the role of MMPs in adhesive interfaces degradation and the anti-proteolytic benefits that may be derived from CHX and other inhibiting agents. Several benefits that may be derived from the use of CHX, namely anti-bacterial, anti-proteolytic, re-wetting, and buffering properties, deserve further investigation. Aspects relative to the specific adhesive approach, pH, and monomeric composition of the adhesive system, as well as the concentration and application protocol for the delivery of CHX, need to be further investigated to be able to maximize its retention and effectiveness while minimizing its potential adverse effects on the polymer network. Furthermore, several other mechanisms that may also contribute to the degradation of adhesive interfaces, namely permeation of dentin fluid esterases into the adhesive interface and proteolytic degradation by cysteine cathepsins, also require further investigation. Most of the available evidence pertaining to the biologic aspects involved in the degradation of adhesive interfaces is derived from laboratory studies. Although *in vitro* studies are effective at isolating the effect of individual variables in the overall process, ultimate validation of the potential therapeutic benefits that may be derived from incorporating inhibiting agents into the adhesive interfaces can only be obtained from clinical studies.

CONCLUSIONS

Within the limitations of this *in vitro* study design, the following can be concluded:

- CHX, in the concentrations of 0.2% and 2.0%, inhibited dentin proteolytic activity as determined by gelatin zymography.
- When CHX was incorporated into a commercially available adhesive or used as a therapeutic primer, no difference in bond strength was observed either at baseline or at six months of storage relative to the control group without CHX irrespective of adhesive approach.

Conflict of Interest

The Authors of this manuscript certify that they have no proprietary, financial or other personal interest of any nature or kind in any product, service and/or company that is presented in this article.

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Bulk-fill Resin-based Composites: An *In Vitro* Assessment of Their Mechanical Performance

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Clinical Relevance

In an attempt to speed up the restoration process, a new class of resin-based composite (RBC) material, the bulk-fill RBC, was recently introduced on the market, enabling up to 4- or 5-mm thick increments to be cured in one step. Their mechanical properties vary relative to those of flowable and nonflowable nanohybrid and microhybrid RBCs.

SUMMARY

The study aimed to assess the mechanical performance of seven bulk-fill RBCs (Venus Bulk Fill, Heraeus Kulzer; SureFil SDR flow, Dentsply Caulk; x-tra base and x-tra fil, VOCO; Filtek Bulk Fill, 3M ESPE; SonicFill, Kerr; Tetric EvoCeram Bulk Fill, Ivoclar Vivadent) by determining their flexural strength (σ), reliability (Weibull parameter, m), flexural modulus ($E_{flexural}$), indentation modulus (Y_{HU}), Vickers hardness (HV), and creep (Cr).

The significant highest flexural strengths were measured for SonicFill, x-tra base, and x-tra fil, while x-tra base, SureFil SDR flow, and Venus Bulk Fill showed the best reliability. The differences among the materials became more evident in terms of $E_{flexural}$ and Y_{HU}, with x-tra fil achieving the highest values, while Filtek Bulk Fill and Venus Bulk Fill achieved the lowest. The enlarged depth of cure in bulk-fill RBCs seems to have been realized by enhancing the materials' translucency through decreasing the filler amount and increasing the filler size. The manufacturer's recommendation to finish a bulk-fill RBC restoration by adding a capping layer made of regular RBCs is an imperative necessity, since the modulus of elasticity and hardness of certain materials (SureFil SDR flow, Venus Bulk Fill, and Filtek Bulk Fill) were considerably below the mean values measured in regular nanohybrid and microhybrid RBCs.

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The class of bulk-fill RBCs revealed similar flexural strength values as the class of nanohybrid and microhybrid RBCs, and significantly higher values when compared to flowable RBCs. The modulus of elasticity ($E_{flexural}$), the indentation modulus (Y_{HU}), and the Vickers hardness (HV) classify the bulk-fill RBCs as between the hybrid RBCs and the flowable RBCs; in terms of creep, bulk-fill and the flowable RBCs perform similarly, both showing a significantly lower creep resistance when compared to the nanohybrid and microhybrid RBCs.

INTRODUCTION

Time-saving restorative materials are an ongoing demand for posterior applications. A new resin-based composite (RBC) material class, the bulk-fill RBCs, has been introduced in the past few years. They are an attempt to speed up the restoration process by enabling up to 4- or 5-mm thick increments to be cured in one step, thus skipping the time-consuming layering process. Bulk-fill RBCs are also marketed as restoratives that are particularly well suited for patients with limited compliance. Moreover, the rheology of these materials is thought to have changed, thus allowing a better adaption to the cavity walls and resulting in a self-leveling effect. For the same purpose, a sonic-activated bulk-fill RBC was also launched on the market (SonicFill, Kerr, Orange, CA, USA). In spite of the stated improved adaption to the cavity walls, microleakage analysis attested to a similar performance for bulk-fill RBCs (SDR, Dentsply Detrey, Konstanz, Germany), and x-tra base, VOCO, Cuxhaven, Germany) as for conventional RBC (GrandioSO, VOCO) in standardized Class II cavities.[1] The marginal integrity of posterior RBC (CeramX Mono, Dentsply; Tetric EvoCeram, Ivoclar Vivadent, Schaan, Liechtenstein; Filtek Supreme XT, 3M ESPE, Seefeld, Germany; and Venus Diamond, Heraeus Kulzer, Hanau, Germany) fillings to enamel and dentin, made with and without a 4-mm flowable base (SDR, Dentsply), was also similar, both, before and after thermomechanical loading.[2] However, the manufacturer's statements with regard to the incremental thickness were confirmed in *in vitro* studies, as the degree of cure and the micromechanical properties were shown to remain constant within a 4-mm layer at a irradiation time of up to 20 seconds (SDR, Dentsply; Venus Bulk Fill, Heraeus Kulzer).[3]

A main concern of curing large increments is a potentially increased polymerization shrinkage stress at the tooth-material interface. A bulk-fill material in its experimental version (SDR, Dentsply) revealed, however, that it had the lowest shrinkage stress and shrinkage-rate values in comparison to regular flowable and nonflowable nanohybrid and microhybrid methacrylate-based RBCs and a silorane-based microhybrid RBC.[4,5] Moreover, it was shown that bulk-fill flowable RBCs (SDR, Dentsply; x-tra base, VOCO) significantly reduced cuspal deflection in standardized Class II cavities compared with a conventional RBC (GrandioSO, VOCO) restored in an oblique incremental filling technique.[1] Regarding mechanical performance, bulk-fill materials (SDR, Dentsply) proved to be more rigid (higher modulus of elasticity) and more plastic (higher plastic deformation and creep values) when compared to regular flowable RBCs, and generally with lower mechanical properties than regular nanohybrid or microhybrid RBCs.[4] Other studies found, however, that bulk-fill RBCs exhibited a creep deformation within the range of regular RBCs.[6] They also found that the flexure strength, water uptake, and biocompatibility of bulk-fill RBCs (x-tra fil, VOCO) were comparable to conventional RBCs.[7]

The first bulk-fill material on the market, SureFil SDR flow (or SDR on the European market), as well as Venus Bulk Fill, x-tra base, and Filtek Bulk Fill, require an additional final capping layer made of regular RBCs, while other materials in the same category (SonicFill, Tetric EvoCeram Bulk Fill, and x-tra fil) can be placed without it. This different application of materials belonging to the same material class confuses many practitioners since they assume the materials' behavior would be similar.

The aim of this study was, therefore, to assess the mechanical performance of a new material class—the bulk-fill RBCs—at the macro and micro scale, and to compare its performance with an already published material database[8] determined under identical conditions, comprised of modern flowable and nonflowable nanohybrid and microhybrid RBCs.

The null hypotheses were: 1) there would be no significant difference in macromechanical (flexural strength [σ] and flexural modulus [$E_{flexural}$]) and micromechanical (Vickers hardness [HV], indentation modulus [Y_{HU}], and creep [Cr]) properties among the bulk-fill RBCs; and 2) there would be no significant difference in the above mentioned properties among the material class of bulk-fill RBCs and the class of flowable and nonflowable nanohybrid and microhybrid RBCs.

MATERIALS AND METHODS

The seven bulk-fill RBCs on the market up to the present (Table 1) were analyzed. Only SonicFill was sonic activated; this was done with an oscillating handpiece (step 3), as recommended by the manufacturer.

The flexural strength (σ) and flexural modulus ($E_{flexural}$) were determined in a three-point bending test (n=20). Therefore, 140 samples were made by compressing the composite material between two glass plates with intermediate polyacetate sheets, separated by a steel mold having an internal dimension of $2 \times 2 \times 16$ mm. Irradiation occurred on the top and bottom of the specimens, as specified in ISO 4049:2009 standards[9]; the time of the light exposures was 20 seconds, with three light exposures, overlapping one irradiated section no more than 1 mm of the diameter of the light guide (1241 mW/cm^2, Elipar Freelight 2, 3M ESPE, Seefeld, Germany) to prevent multiple polymerizations. After removal from the mold, the specimens were ground with silicon carbide paper (grit size P 1200/4000 [Leco]) to remove protruding edges or bulges, and then stored for 24 hours in distilled water at 37°C. The samples were loaded until failure in a universal testing machine (Z 2.5, Zwick/Roell, Ulm, Germany) in a three-point bending test device, which was constructed according to the guidelines of NIST 4877 with a 12-mm distance between the supports.[10] During testing, the specimens were immersed in distilled water at room temperature. The crosshead speed was 0.5 mm/min. The universal testing machine measured the force during bending as a function of deflection of the beam. The bending modulus was calculated from the slope of the linear part of the force-deflection diagram.

Micromechanical Properties

Fragments larger than 8 mm (n=10) from the three-point bending test specimens of each group were used to determine the micromechanical properties (HV, Y_{HU}, Cr) according to DIN 50359-1:1997-10[11] by means of a universal hardness device (Fischerscope H100C, Fischer, Sindelfingen, Germany). Prior to testing, the samples were polished with a grinding system (EXAKT 400 CS, EXAKT, Norderstedt, Germany) using silicon carbide paper P 2500 followed by P 4000. Measurements were done on the top (n=10) of the slabs, about 4 mm away from the breaking edge, with six measurements per sample. The test procedure was carried out with controlled force, and the test load increased and decreased with a constant speed between 0.4 mN and 500 mN. The load and the penetration depth of the indenter were continuously measured during the load-unload-hysteresis. The universal hardness is defined as the test force divided by the apparent area of the indentation under the applied test force. From a multiplicity of measurements stored in a database supplied by the manufacturer, a conversion factor (0.0945) between universal hardness and HV was calculated by the manufacturer and entered into the software such

Table 1: *Materials, Manufacturer, and Chemical Composition of Matrix and Filler as Well as Filler Content by Weight (Wt) and Volume (Vol)*

Bulk Fill RBCs	Manufacturer, Color, Batch	Resin Matrix	Filler	Filler Wt%/Vol%
Tetric EvoCeram Bulk Fill nanohybrid RBC	Ivoclar Vivadent, IVA, P48872	Bis-GMA, UDMA	Ba-Al-Si glass, prepolymer filler (monomer, glass filler, and ytterbium fluoride), spherical mixed oxide	79-81 (including 17% prepolymers)/ 60-61
Venus Bulk Fill nanohybrid RBC	Heraeus Kulzer, Universal 010026	UDMA, EBPDMA	Ba-Al-F-Si glass, SiO_2	65/38
SureFil SDR flow flowable base RBC	Dentsply Caulk, Universal, 100407	Modified UDMA, TEGDMA, EBPDMA	Ba-Al-F-B-Si glass and St-Al-F-Si glass as fillers	68/44
x-tra base hybrid RBC	VOCO, universal, V 45226	Bis-GMA, UDMA		75/
x-tra fil hybrid RBC	VOCO, universal 1202359	Bis-GMA, UDMA, TEGDMA		86/70.1
SonicFill nanohybrid RBC	Kerr, A3, 4252497	Bis-GMA, TEGDMA, EBPDMA	SiO_2, glass, oxide	83.5/
Filtek Bulk Fill nano RBC	3M ESPE, universal N387662	Bis-GMA, UDMA, Bis-EMA, Procrylat resins	Zirconia/silica, ytterbium trifluoride	64.5/42.5

Abbreviations: Bis-EMA, Bisphenol-A polyethylene glycol diether dimethacrylate; Bis-GMA, Bisphenol-A diglycidyl ether dimethacrylate; EBPDMA, ethoxylated Bisphenol-A-dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

that the measurement results were indicated in the more familiar HV units. Y_{HU} was calculated from the slope of the tangent of the indentation depth-curve at maximum force. By measuring the change in indentation depth with a constant test force, a relative change in the indentation depth can be calculated. This is a value for the Cr of the materials.

Field Emission Scanning Electron Microscope

The structural appearance of the filler was established by a field emission scanning electron microscope (Zeiss Supra 55 VP, Zeiss NTS GmbH, Oberkochen, Germany) on unsputtered samples (Figure 1). Therefore, one fragment of the three-point bending test specimens of each group was ground and polished (P 4000) prior to examination. The backscattering method allows a distinction to become apparent between filler with different densities as well as to assess the fillers' sizes and morphologies.

Statistical Analysis

The Kolmogorov-Smirnov test was applied to verify that the data were normally distributed. The results were compared using one-way and multiple-way analysis of variance (ANOVA) and Tukey post hoc test (α=0.05). A multivariate analysis (general linear model with partial eta-squared statistics) assessed the effect of material, filler volume (%), and filler weight (%) on the mechanical properties (version 20.0, SPSS Inc, Chicago, IL, USA). A Pearson correlation analysis among the tested parameters was conducted, while the flexural strength data were additionally examined by means of a Weibull analysis.

A common empirical expression for the cumulative probability of failure P at applied stress is the Weibull model: $P_f(\sigma_c) = 1 - \exp[-(\sigma_c/\sigma_0)^m]$ where σ_c is the measured strength, m is the Weibull modulus, and σ_0 is the characteristic strength, which is defined as the uniform stress at which the probability of failure is 0.63. The double logarithm of this expression is: $\ln \ln[1/(1-P)] = m\ln \sigma_c - m\ln \sigma_0$ By plotting $\ln \ln[1/(1-P)]$ vs $\ln \sigma$, a straight line results with the upward gradient m.

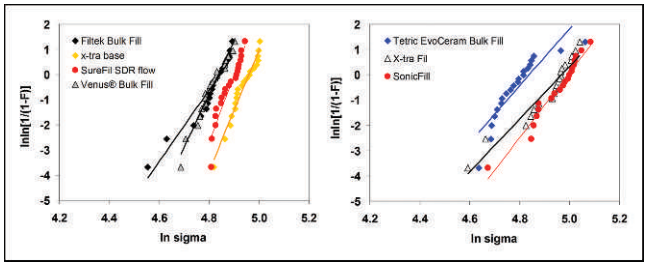

Figure 1. *Weibull analysis.*

RESULTS

Post hoc multiple pairwise comparisons with Tukey test ($p<0.05$) showed the significantly highest flexural strength values for SonicFill, x-tra base, and x-tra fil (Table 2). In terms of the material's reliability, expressed by the Weibull modulus (m), two groups can be distinguished, one comprising x-tra base, SureFil SDR flow, and Venus Bulk Fill, which are characterized by a very high Weibull modulus varying between 21.1 and 26.1, and the rest of the materials, showing a moderate reliability, with Weibull modulus values varying between 10.4 and 14.2 (Figure 1; Table 2). The differences among the materials became more evident in terms of $E_{flexural}$ and indentation modulus Y_{HU}. x-tra fil achieved the significantly highest values, whereas Filtek Bulk Fill and Venus Bulk Fill achieved the lowest. Moreover, an excellent correlation was measured between $E_{flexural}$ and Y_{HU} (Pearson correlation coefficient = 0.91). There was also a very good correlation within the micromechanical properties (Y_{HU} – HV = 0.94; Y_{HU} – Cr = –0.76; and HV – Cr = –0.64, whereas the correlation within the macromechanical properties was only moderate (FS – $E_{flexural}$ = 0.47).

The influence of the parameters bulk-fill RBC (material), filler volume, and filler weight were analyzed in an ANOVA multivariate test (Table 3). The filler volume and filler weight data were taken as indicated by manufacturers. The macromechanical properties (flexural strength and modulus of elasticity in flexural test) and the micromechanical properties (indentation modulus, Vickers hardness, and creep) were selected as dependant variables. The significance values of these three main effects were less than 0.05, indicating that they all contribute to the model. The results show that the strongest influence of the above mentioned parameters on the mechanical properties (higher eta square values) was reflected in the $E_{flexural}$ and Y_{HU}, followed by HV and Cr, while the influence on σ was moderate. Generally, the strongest influence on the measured properties was performed by the filler volume, followed by the filler weight, followed by material.

The material class of bulk-fill RBCs revealed similar flexural strength values when compared to the class of nanohybrid and microhybrid RBCs, and significantly higher values when compared to the class of flowable RBCs. $E_{flexural}$, Y_{HU}, and HV place

Table 2: *Mechanical Properties (mean values with standard deviation in parentheses) Measured at Macroscopic Scale—Flexural Strength (σ) With Weibull Statistic (m = Weibull parameter, σ_0 = Characteristic Strength, R^2 = Regression Coefficient) and Flexural Modulus ($E_{flexural}$)—and Microscopic Scale—Indentation Modulus (Y_{HU}), Vickers Hardness (HV), and Creep (Cr)**

Bulk-fill RBC	σ, MPa	Weibull Statistic			$E_{flexural}$, GPa	Y_{HU}, GPa	HV, N/mm²	Cr, %
		m	σ_0, MPa	R^2				
Tetric EvoCeram Bulk Fill	120.8^{a} (12.7)	11.2	126.4	0.80	4.5^{B} (0.8)	13.4^{d} (0.8)	78.4^{C} (6.7)	3.5^{ab} (0.2)
Filtek Bulk Fill	122.4^{ab} (9.6)	14.2	126.9	0.92	3.8^{A} (0.4)	9.3^{b} (0.2)	48.4^{B} (1.3)	4.3^{d} (0.1)
Venus Bulk Fill	122.7^{ab} (6.9)	21.1	126.1	0.97	3.6^{A} (0.4)	7.7^{a} (2.0)	38.1^{A} (11.8)	4.6^{e} (0.4)
SureFil SDR flow	131.8^{bc} (5.8)	26.6	134.5	0.91	5.0^{B} (0.4)	11.3^{c} (0.5)	54.2^{B} (1.9)	4.0^{c} (0.2)
x-tra fil	137.0^{cd} (14.4)	10.4	143.8	0.92	9.5^{E} (0.6)	22.2^{g} (1.7)	133.5^{D} (32.0)	3.4^{a} (0.3)
x-tra base	139.4^{cd} (7.0)	24.0	142.5	0.96	6.0^{C} (0.9)	14.4^{e} (1.1)	85.1^{C} (11.2)	3.6^{b} (0.3)
SonicFill	142.8^{d} (12.9)	12.9	147.5	0.96	6.9^{D} (0.6)	15.9^{f} (0.7)	82.0^{C} (4.7)	3.6^{b} (0.2)

Abbreviations: RBC, resin-based composite.
** Superscript letters indicate statistically homogeneous subgroups within each column (Tukey test, α=0.05).*

the bulk-fill RBCs between the hybrid RBCs and the flowable RBCs. In terms of Cr performance, the bulk-fill RBCs and the flowable RBCs were similar, both showing a significantly lower creep resistance when compared to the nanohybrid and microhybrid RBCs.

Analyzing the amount of filler in the four material classes revealed a lower filler load in bulk-fill RBCs compared to the nanohybrid and microhybrid RBCs. Compared to the class of flowable RBCs, higher weight filler load was found for the bulk-fill RBCs, while the filler volume was similar in both material categories (Table 4).

DISCUSSION

Though advertised as a new material class, bulk-fill RBCs seem to not differ essentially in their chemical composition from regular nanohybrid and microhybrid RBCs.[8] They contain monomers like Bis-GMA, UDMA, TEGDMA, and EBPDMA in their organic matrix as well as regular filler systems (Table 1). In SureFil SDR flow, the organic matrix also contains a patent-registered urethane dimethacrylate with incorporated photoactive groups able to control polymerization kinetics[5] (SDR technology = stress decreasing resin). In Tetric EvoCeram Bulk Fill, the manufacturer states that, besides having a regular camphorquinone/amine initiator system, it has introduced an "initiator booster" (Ivocerin) able to polymerize the material in depth. However, there are few details concerning the polymerization mechanism or the chemical nature of the initiator. No changes in the polymerization initiating system are specified for the other bulk-fill materials; thus, the enlarged depth of cure must have been regulated by improving the materials' translucency. A simple approach in doing this is to reduce the amount of fillers since translucency and the amount of filler particles correlates linearly.[12] The statistical comparison among the material classes bulk-fill, nanohybrid, and microhybrid RBCs, with regard to the filler amount, confirms this assumption (Table 4). Besides, the translucency of dental materials is also influenced by the difference in the refractive indices between the filler particles and the resin matrix,[13,14] which determines how light is scattering within a material.[15] Similar refractive indices of the components of a RBC, as demonstrated for Bis-GMA and silica filler particles, were shown to improve translucency in experimental dental materials.[16] Apart from these considerations, the dimension of fillers was increased in many bulk-fill RBCs (x-tra fil, x-tra base, SureFil SDR flow, and SonicFill) (Figure 2) to a

Table 3: *Influence of Material, Filler Volume, and Weight on the Mechanical Properties—Flexural Strength (σ), Flexural Modulus ($E_{flexural}$), Indentation Modulus (Y_{HU}), Vickers Hardness (HV), and Creep (Cr)[a]**

Parameter	σ, MPa	$E_{flexural}$, GPa	Y_{HU}, GPa	HV, N/mm²	Cr, %
Bulk-fill RBC	0.406	0.912	0.963	0.791	0.795
Filler vol%	0.279	0.943	0.968	0.794	0.852
Filler wt%	0.368	0.918	0.965	0.792	0.794

Abbreviations: RBC, resin-based composite.
[a] Table contains the partial eta-square values. The higher the partial eta-squares, the higher the influence of the selected factor on the measured properties.
** The influence of all parameters was statistically significant (α=0.05).*

Table 4: *Mechanical Properties (mean values with standard deviation in parentheses) Measured at Macroscopic Scale—Flexural Strength (σ) and Flexural Modulus ($E_{flexural}$)—and Microscopic Scale—Indentation Modulus (Y_{HU}), Vickers Hardness (HV), and Creep (Cr) as Well as Filler Weight (Wt) and Volume (Vol) for Different RBC Categories**

RBC Type	σ, MPa	$E_{flexural}$, GPa	Y_{HU}, GPa	HV, N/mm²	Cr, %	Wt, %	Vol, %
Microhybrid RBC	131.2^{A} (29.8)	7.3^{a} (2.6)	14.9^{A} (4.9)	87.0^{a} (28.8)	3.6^{A} (0.5)	78.5^{a} (4.0)	62.8^{A} (12.5)
Nanohybrid RBC	125.9^{A} (32.6)	6.3^{b} (2.1)	14.8^{A} (5.5)	90.9^{a} (35.6)	3.6^{A} (0.5)	78.2^{a} (7.9)	63.8^{A} (8.7)
Bulk fill	131.1^{A} (13.3)	5.6^{c} (2.0)	13.5^{B} (4.6)	74.3^{b} (32.6)	3.9^{B} (0.5)	73.1^{b} (8.0)	51.0^{B} (12.2)
Flowable RBC	119.3^{B} (25.8)	4.2^{d} (1.3)	10.6^{C} (3.6)	65.8^{c} (28.9)	3.8^{B} (0.6)	69.9^{c} (8.2)	51.1^{B} (10.6)

Abbreviations: RBC, resin-based composite.
** Superscript letters indicate statistically homogeneous subgroups within each column (Tukey test, $\alpha=0.05$).*

size of 20 μm or more, which decreases, at a similar filler amount, the total filler surface and, consequently, the filler-matrix interface. Thus, light scattering at the filler-matrix interface is reduced, allowing more light to penetrate the material and to better cure the RBCs in depth. Moreover, four of the analyzed bulk-fill RBCs are denoted as nano or nanohybrid RBCs (Table 1), containing a certain amount of low-sized fillers. With dimensions below the wavelength of visible light (390 to 750 nm), nanoparticles are unable to scatter or absorb visible light, which is an important aspect in light curing and improves translucency and esthetics.[17]

Assuming that the bulk-fill RBCs are adequately cured and the mechanical properties within the incremental thickness are constant,[3] the mechanical stability in stress-bearing areas of fillings restored with this material class is still an open question, since, so far, long-term clinical studies are not available. Comprehensive reviews of the past years, analyzing the reasons of clinical failures in RBC restorations, indicate an increased trend in material fracture.[18,19] Moreover, the mechanical properties of modern RBCs are significantly weaker and less fracture resistant than those sold in the 1970s and 1980s, before the major push to minimize particle size occurred,[20] which brings into question whether modern RBCs are strong enough under clinical conditions. When comparing the material classes, the bulk-fill RBCs showed significantly lower mechanical properties, except for flexural strength, than the nanohybrid and microhybrid RBCs (Table 4). Since it is a parameter of decisive importance, it is most important to note that the modulus of elasticity is lower in the bulk-fill RBCs than in the nanohybrid and microhybrid RBCs. A material with a low modulus of elasticity, particularly when placed in load-bearing areas, will result in a higher deformability under masticatory stresses. This will cause, as a final consequence, catastrophic failures.

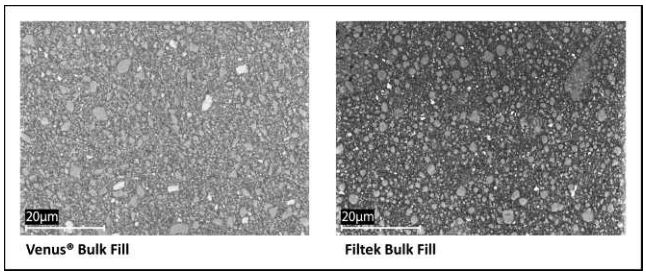

Figure 2. *Structural appearance of the filler established by field emission scanning electron microscopy (backscattering mode).*

Within the bulk-fill RBCs, the material with the highest filler content, x-tra fil, (Table 1) also achieved the highest modulus of elasticity, while a lower filler content was clearly reflected in lower Y_{HU} and $E_{flexural}$ values (Filtek Bulk Fill and Venus Bulk Fill). Thus, the excellent correlation between filler amount and modulus of elasticity measured for RBCs in previous studies[8,21,22] is confirmed. An exception is Tetric EvoCeram Bulk Fill, which shows moderate values for the modulus of elasticity, albeit having a high filler content. It must, however, be considered that Tetric EvoCeram Bulk Fill also contains prepolymerized fillers, which is included in the total filler amount. Thus, the inorganic filler content, which in effect increases the modulus of elasticity, is consistently lower (Table 1).

The material's reliability, expressed in this study by the Weibull modulus (m), neither correlated with the filler amount (Table 1) nor with the filler shape or dimension. Of the three materials with high reliability, x-tra base and SureFil SDR flow included very large fillers (>20 μm) in their formulation, while the filler system in Venus Bulk Fill resembled the structure of regular RBCs (Figure 2). Also, the sonic-activated bulk-fill RBC SonicFill, with a supposed improved flowability and therefore reduced surface defects able to initiate crack propagation, showed only a moderate reliability. As a consequence, additional rheologic measurements are necessary to evaluate the effect of filler size and amount on the flowability of bulk-fill materials.

When using bulk-fill materials, the manufacturers indicate to either finish the restoration by adding a capping layer made of regular RBCs (SureFil SDR flow, Venus Bulk Fill, x-tra base, and Filtek Bulk Fill) or to place the bulk-fill RBCs without capping (SonicFill, Tetric EvoCeram Bulk Fill, and x-tra fil). While in terms of flexural strength the reason for this indication is not evident (Table 2), the measured values for the modulus of elasticity, indentation modulus, and hardness, except for x-tra base, clearly confirm manufacturer indications. Bulk-fill RBCs like SureFil SDR flow, Venus Bulk Fill, and Filtek Bulk Fill reached HV values (38.1-54.2 N/mm^2) considerably below the mean values measured in regular nanohybrid and microhybrid RBCs (90.9 N/mm^2 and 87.0 N/mm^2, respectively); thus, an additional final capping layer is necessary. Moreover, the very large particle size in four of the analyzed materials (Figure 2) could increase surface roughness[23] and renew the discussion about abrasion, attrition, and wear in RBCs.[24]

Both tested null hypotheses are therefore rejected. The measured properties allow a direct comparison of the bulk-fill RBCs with regular RBCs and place them, as a material category, between the hybrid (nano and micro) RBCs and the flowable RBCs. It must, however, be considered that the flexural strength was measured in this study on 2-mm thick samples, as specified in ISO standards, while bulk-fill RBCs are clinically applied in larger increments. Since the degree of cure and the micromechanical properties were shown to remain constant within a 4-mm layer in two of the materials analyzed in this study (SureFi SDR flow and Venus Bulk Fill),[3] it can be assumed that under proper polymerization conditions, a 4-mm increment placed with these materials in bulk or by using an incremental technique would present similar properties.

CONCLUSIONS

The manufacturers' indication to finish a bulk-fill RBC restoration by adding a capping layer made of regular RBCs is a necessity, since the indentation modulus and hardness of particular materials (SureFil SDR flow, Venus Bulk Fill, and Filtek Bulk Fill) were considerably below the mean values measured in regular nanohybrid and microhybrid RBCs.

The measured mechanical properties place the bulk-fill RBCs, as a material category, between the nanohybrid and microhybrid RBCs and the flowable RBCs, suggesting a similar or even inferior clinical behavior of bulk-fill RBCs compared to nanohybrid and microhybrid RBCs. Within the class of bulk-fill RBCs the differences in mechanical properties among the RBCs are, however, large.

Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 19 December 2012)

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Fracture Resistance of Teeth Restored With All-ceramic Inlays and Onlays: An In Vitro Study

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Clinical Relevance

In clinical practice, as tooth preparation increases with onlay vs inlay techniques, the fracture resistance of the tooth may decrease depending on the restoration material used.

SUMMARY

Fracture resistance of inlays and onlays may be influenced by the quantity of the dental structure removed and the restorative materials used. The purpose of this *in vitro* study was to evaluate the effects of two different cavity preparation designs and all-ceramic restorative materials on the fracture resistance of the tooth-restoration complex. Fifty mandibular third molar teeth were randomly divided into the following five groups: group 1: intact teeth (control); group 2: inlay preparations, lithium-disilicate glass-ceramic (IPS e.max Press, Ivoclar Vivadent AG, Schaan, Liechtenstein); group 3: inlay preparations, zirconia ceramic (ICE Zirkon, Zirkonzahn SRL, Gais, Italy); group 4: onlay preparations, lithium-disilicate glass-ceramic (IPS e.max Press); and group 5: onlay preparations, zirconia ceramic (ICE Zirkon). The inlay and onlay restorations were adhesively cemented with dual polymerizing resin cement (Variolink II, Ivoclar Vivadent AG). After thermal cycling (5° to 55°C × 5000 cycles), specimens were subjected to a compressive load until fracture at a crosshead speed of 0.5 mm/min. Statistical analyses were performed using one-way analysis of variance and Tukey HSD tests. The fracture strength values were significantly higher in the inlay group (2646.7 ± 360.4) restored with lithium-disilicate glass-ceramic than those of the onlay group (1673.6 ± 677) restored with lithium-disilicate glass-ceramic. The fracture strength values of teeth restored with inlays using zirconia ceramic (2849 ± 328) and onlays with

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zirconia ceramic (2796.3 ± 337.3) were similar to those of the intact teeth (2905.3 ± 398.8). In the IPS e.max Press groups, as the preparation amount was increased (from inlay to onlay preparation), the fracture resistance was decreased. In the ICE Zirkon ceramic groups, the preparation type did not affect the fracture resistance results.

INTRODUCTION

The presence of extensive carious lesions, unsatisfactory restorations, and tooth fractures results in controversy regarding the optimal restorative procedure.[1,2] Indirect restorations have more desirable physical properties than direct composite restorations because they are fabricated under relatively ideal laboratory conditions.[3] When an indirect restoration is determined to be the best treatment option, the clinician must then determine the geometric configuration of the cavity preparation.[1,4,5] Cusp coverage seems to be the most controversial point with respect to the final cavity preparation design for posterior teeth. The mechanical properties of the restorative materials should be considered before choosing the cavity design.[6,7] Cavity preparation is directly related to decrease of cusp stiffness.[8] The depth and width may affect cusp deflection and tooth fracture strength.[9] According to the cusp coverage, the types of restorations can be classified as inlays (no cusp is covered), onlays (at least one cusp is not covered), or overlays (all cusps are covered).[10]

The adhesive technique allows dental professionals to restore the morphology, esthetic appearance, and original mechanical loading capacity of natural teeth.[11,12] The use of ceramics with adhesive techniques permits the preservation of tooth structure and more esthetic restorations in posterior teeth.[13-16] Zirconia is a crystalline dioxide of zirconium. Its mechanical properties are very similar to those of metals, and its color is similar to tooth color. Zirconia crystals can be organized in three different patterns: monoclinic, cubic, and tetragonal.[17] Yttrium-stabilized zirconia, also known as tetragonal zirconia has become available for use in dentistry through computer-aided design/computer-aided manufacturing (CAD/CAM) or copy-milling techniques, and provides excellent mechanical performance, superior strength, and fracture resistance compared to other ceramics.[18,19] However, the use of some types of ceramics remains limited in the posterior region, where extensive masticatory forces are applied.[20,21] In 2005, an improved pressed-ceramic material called IPS e.max Press (Ivoclar Vivadent AG, Schaan, Liechtenstein) was introduced to the market. There are limited data available on IPS e.max Press (Ivoclar Vivadent AG) ceramic. This pressed ceramic is intended to expand the range of applications for IPS Empress 2 (Ivoclar Vivadent AG). While it features similar physical properties to previous materials, its translucency has been improved. The IPS e.max Press (Ivoclar Vivadent AG) system is comprised of high-stability framework material that consists of lithium-disilicate (Li_2O-$2SiO_2$). The restorations can be customized by using either a layering technique based on fluorapatite glass ceramic or by using the staining technique.[22,23]

The parameters for cavity design should be consistent with principles of adaptation, resistance, and retention, occlusion, and esthetics.[1,9,24-26] Adhesive luting procedures can reinforce teeth and minimize deleterious effects of cusp flexure, thus increasing crown stiffness as an outcome of adhesive effects, cohesive resistance, and stress distribution.[6,15,26-28] In *in vitro* studies, a number of factors may interfere with resistance to fracture, such as the tooth embedment method, type of load application device, and crosshead speed.[29-34] Thus, the experimental methods used for *in vitro* analyses do not accurately represent real clinical conditions in which failures occur primarily due to fatigue.[35-38] To minimize the discrepancy between experimental assessments and clinical failures, different methods have been used, such as the joint use of mechanical tests and fracture mode analyses according to predefined scales.[39-44]

The objective of this *in vitro* study was to determine and compare the fracture strength and the failure modes of lithium-disilicate glass-ceramic and yttrium-stabilized zirconia-based ceramic inlay and onlay restorations. The null hypotheses tested were: 1) the preparation type does not affect the fracture resistance of the tooth-restoration complex and 2) the type of restoration material does not affect the fracture resistance of the tooth-restoration complex.

MATERIALS AND METHODS

In this *in vitro* study, 50 freshly extracted, sound, caries-free human mandibular third molars (wisdom teeth) were used. Calculus and soft-tissue deposits were removed with a hand scaler. The teeth were cleaned using a rubber cup and fine pumice water slurry, and examined to detect any preexisting defects. Only intact, noncarious, and unrestored teeth were included in the study. The teeth were

stored in distilled water until use, and were removed only during the test procedure. The roots were covered with a 0.2-mm layer of a polyether impression material (Impregum Garant L Duasoft, 3M ESPE AG, Seefeld, Germany) to simulate the periodontal ligament, and embedded in an autopolymerizing acrylic resin (Meliodent, Heraeus Kulzer GmbH, Hanau, Germany) up to 2 mm below the cementoenamel junction. The artificial tooth mobility was evaluated in the horizontal and vertical directions by use of a Periotest instrument (Periotest, Siemens AG, Bensheim, Germany). The Periotest value of the embedded teeth was standardized at a value less than or equal to +7 in order to simulate the natural dentition.[45] The teeth were randomly divided into five experimental groups (n=10) as follows:

- group 1: intact teeth, no treatment (control group) (IT);
- group 2: teeth with inlay restorations with lithium-disilicate glass-ceramic (IPS e.max Press, Ivoclar Vivadent AG) (I-e.max);
- group 3: teeth with inlay restorations with zirconia ceramic (ICE Zirkon Zirkonzahn SRL, Gais, Italy) (I-Zirkon);
- group 4: teeth with onlay restorations with lithium-disilicate glass-ceramic (IPS e.max Press, Ivoclar Vivadent AG) (O-e.max); and
- group 5: teeth with onlay restorations with zirconia ceramic (ICE Zirkon Zirkonzahn SRL) (O-Zirkon).

Using a 6° taper diamond rotary cutting instrument (Inlay Preparations Set 4261, Komet, Lemgo, Germany), two different preparations, with rounded internal angles, were defined. Tooth preparations were made by the same operator (SS) with the recommended sequence of specific diamond burs (Inlay Preparations Set 4261, Komet) under constant water-cooling. To ensure standardized cavity preparations, a parallelometer (Paraskop, Bego, Bremen, Germany) was used. The parallelometer has a milling arm, designed as a multifunctional arm, and a lever for a drilling tool. A motor is integrated in the base for control for the milling unit. The isthmus floor of the mesio-occluso-distal (MOD) inlay cavities was prepared following principles for ceramic and indirect composite resin preparations as described elsewhere.[46] The pulpal floor was prepared to a depth of 2.5 mm from the occlusal surface; the occlusal isthmus was 2.5 mm wide, and buccolingual widths on the mesial and distal boxes were similar to the width of the occlusal isthmus. Each box had a gingival floor depth of 1.5 mm mesiodistally and an axial wall height of 2 mm. Margins were prepared with 90° cavosurface angles. The onlays were prepared using basic techniques, the mesiobuccal and distobuccal cusps were reduced by 2 mm according to the anatomic shape of the occlusal surface, and the buccal margins were finished as 1-mm rounded shoulder design (Figure 1A and 1B).

A two-stage impression was made of each prepared tooth using a polyvinyl siloxane impression material (Elite HD, Zhermack SpA, Badia Polesine, Italy). After 2 hours, the impressions were poured using type IV stone (Durone, Dentsply, Petrópolis, RJ, Brazil). A technician fabricated all restorations using a standardized technique following the manufacturer's instructions. To fabricate the IPS e.max Press (Ivoclar Vivadent AG) restorations, a die spacer was applied to the cavity surfaces at a distance of 1.5 mm away from the marginal areas. The wax frameworks were sprued and invested with a speed investment material (IPS PressVEST Speed, Ivoclar Vivadent AG). A lithium-disilicate glass-ceramic ingot (IPS e.max Press, Ivoclar Vivadent AG) was heated and pressed into an investment mold in the furnace (EP 600, Ivoclar Vivadent AG) after burnout of the wax analog. After divestment with glass polishing beads at 4-bar pressure, fine divestment was performed with glass polishing beads at 2-bar pressure. The pressed frameworks were immersed into 1% hydrofluoric acid (Invex Liquid, Ivoclar Vivadent AG) and cleaned in an ultrasonic cleaner (Whaledent Biosonic Jr, Whaledent International, New York, NY, USA) using distilled water for 15 minutes. After cleaning, the fabricated frameworks were veneered with layering ceramic (IPS e.max Ceram A2 Dentin, Ivoclar Vivadent AG). Analogs of ICE Zirkon (Zirkonzahn SRL) frameworks were fabricated with specific light-polymerizing composite build-up materials (T Rigid, Zirkonzahn SRL). The ICE Zirkon (Zirkonzahn SRL) frameworks were milled in "green" ceramic condition. The frameworks then sintered in a sintering oven (Keramikofen 1500, Zirkonzahn SRL) at 1500°C for 2 hours. The ICE Zirkon (Zirkonzahn SRL) frameworks were veneered with low-fusing ceramic (Ceramic Dentine A2, ICE Ceramic, Zirkonzahn SRL). Completed ceramic restorations were adhesively cemented using a dual-curing fine-particle hybrid composite (high viscosity) (Variolink II, Ivoclar Vivadent AG).

The bonding of the teeth was performed according to the following procedure. The teeth were etched with 35% phosphoric acid (Total Etch, Ivoclar

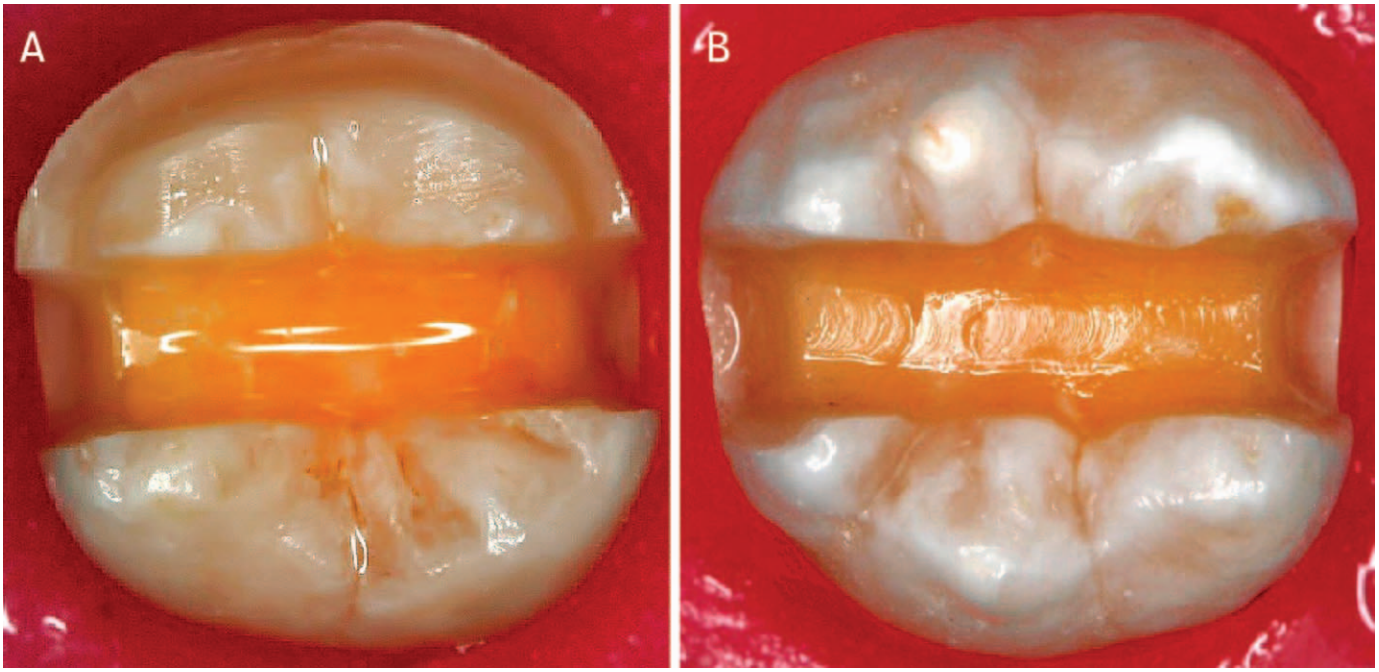

Figure 1. *(A):* Representative onlay preparation. *(B):* Representative inlay preparation.

Vivadent AG) followed by a rinse with water. Excess water was removed in accordance with the wet-bonding technique.[12] Syntac primer (Ivoclar Vivadent AG) was applied for 15 seconds and gently air-dried. Syntac adhesive (Ivoclar Vivadent AG) was applied for 10 seconds and gently air-dried. A bonding agent (Heliobond, Ivoclar Vivadent AG) was applied and left unpolymerized (polymerized together with Variolink II). The same procedure was followed for each tooth.

The bonding of inlay and onlay ceramics was performed according to the following procedures. The zirconia-based specimens were etched for 180 seconds with Metal/Zirconia Primer (Ivoclar Vivadent AG). Hydrofluoric acid, 4.5% (IPS ceramic etching gel, Ivoclar Vivadent AG) was applied to the IPS e.max Press (Ivoclar Vivadent AG) surfaces for 60 seconds, rinsed thoroughly for 60 seconds, and air dried for 10 seconds. The interior surfaces of all specimens were silanized with porcelain primer (Monobond S, Ivoclar Vivadent AG) for 60 seconds and gently air dried. A bonding agent (Heliobond, Ivoclar Vivadent AG) was applied and left unpolymerized (polymerized after inlay placement).

Dual-polymerizing resin cement (Variolink II Base, Ivoclar Vivadent AG) was mixed with its catalyst (Variolink II high viscosity, Ivoclar Vivadent AG) in equal parts for 15 seconds, applied to the ceramic surface, cemented, and light polymerized. The ceramic specimen was cemented perpendicular to the pretreated surface using finger pressure, and excess material was removed with an explorer. The specimens were light polymerized with a minimum light intensity of 650 mW/cm^2 (Elipar Freelight 2, 3M ESPE AG) from the facial and occlusal directions for 20 seconds in each direction. The cement margin was finished using flexible polishing discs (Sof-Lex XT Pop-On, 3M ESPE AG). The specimens were exposed to thermal cycling at temperatures of 5°C and 55°C for a total of 5000 cycles. The dwell time at each temperature was 30 seconds, and the transfer time from one bath to the other was 2 seconds.

The teeth were subjected to axial compressive loading using a metal sphere of 6-mm diameter applied vertically and centered on the occlusal surface of the restoration at a crosshead speed of 0.5 mm/min in a universal testing machine (TSTM 02500, Elista Ltd, Istanbul, Turkey). In order to reduce the local force peaks, 0.5-mm thick tin foil (Baoji Taihe Nonferrous Metal Co, Ltd, Shaanxi, China) was inserted between the metal sphere and the occlusal surface of the restoration. The force (N) required to fracture the restoration and the mode of fracture were recorded. The mode of fracture for each specimen was classified according to Burke[24] as follows:

- mode I: isolated fracture of the restoration;
- mode II: restoration fracture involving a small tooth portion;

Table 1: *Mean Fracture Resistance Values (SDs) and Statistical Categories of All Experimental Groups (n=10)*

Group	Code	Failure Load Mean (N)*
1	IT	2905.3 (398.8)[a]
2	I-e.max	2646.7 (360.4)[a]
3	I-Zirkon	2849.0 (328.0)[a]
4	O-e.max	1673.6 (677.0)[b]
5	O-Zirkon	2796.3 (337.3)[ac]

** Groups with different superscript letters are statistically significantly different according to the Tukey HSD test (p<0.05).*

- mode III: fracture involving more than half of the tooth, without periodontal involvement; and
- mode IV: fracture with periodontal involvement.

The results were analyzed by one-way analysis of variance (ANOVA) and Tukey HSD tests, and were considered statistically different at $\alpha = 0.05$.

RESULTS

The means and standard deviations for the fracture resistance of the test groups are shown in Table 1. The one-way ANOVA showed that there were statistically significant differences among the groups. The Tukey HSD test revealed that group 4 (1673.6 ± 677 N) showed significantly lower fracture strength values than the other groups. No significant differences were observed between the fracture strength values of the inlay groups (group 2 [2646.7 ± 360.4 N] and group 3 [2849 ± 328 N]) and the control group (group 1 [2905.3 ± 398.8 N]) (p=0.264). However, there were statistically significant differences between the fracture strength values of the onlay groups (groups 4 and 5) (p=.000). The O-Zirkon samples showed higher fracture strength values than those of O-e.max. There were significant differences between the I-e.max and O-e.max groups (p=.001); however, there were no significant differences between the I-Zircon and O-Zircon groups (p=.727). The mode of fracture for each group is shown in Table 2.

DISCUSSION

In this study, standardized inlay and onlay preparations were made for each molar tooth, and the restorations were adhesively cemented. The effects of cavity design and ceramic type (zirconia and lithium-disilicate–based ceramics) on the fracture resistance of restored teeth were evaluated.

Table 2: *Mode of Fracture of Restored Specimens According to Burke[24]*

Mode of Failure[a]	I-e.max	O-e.max	I-Zirkon	O-Zirkon
I	5	1	1	—
II	5	6	3	—
III	—	3	5	6
IV	—	—	1	4

[a] Mode I: isolated fracture of the restoration; mode II: restoration fracture involving a small tooth portion; mode III: fracture involving more than half of the tooth, without periodontal involvement; mode IV: fracture with periodontal involvement.

The results of this study revealed that there were no significant differences between the fracture strength values of the inlay groups; however, there were significant differences between the fracture strength values of the onlay groups. When the I-Zirkon and O-Zirkon groups were evaluated, the preparation type (inlay vs onlay preparation) did not affect the fracture strength values when compared with those of the control group. However, in the groups restored using IPS e.max Press, the O-e.max group (group 4) demonstrated significantly lower fracture strength values. Therefore, the null hypotheses that neither the preparation type nor the type of restoration material affects the fracture resistance of a tooth-restoration complex were partially rejected.

In the present study, the unprepared molars achieved the highest mean fracture strength values of 2905.3 ± 398.8 N. This value correlates with findings of a study conducted by Soares and others,[1] in which an average fracture strength value of 3143.1 ± 635.5 N was achieved in mandibular molars. However, the fracture strength values of the inlay and onlay groups in this study, except for the O-e.max group, were higher than the results of their investigation using feldspathic ceramic material for partial ceramic restorations in lower molars.[1] In the present study, the similarities between the fracture strength values of the inlay groups and the control group might be explained by the minimal reduction of the dental structure for an inlay preparation. The stabilization of a prepared tooth by placing a ceramic inlay using adhesive technique is considered to be a proven procedure in numerous

in vitro studies[31,32] and explains the high fracture strength values. Although the mean fracture strength of the O-e.max group was significantly lower than the other groups, the mean fracture strength value of the O-Zirkon group was as high as that of the control group. This results from the mechanical structure of yttrium-stabilized zirconia, which is a glass-free, high-strength polycrystalline ceramic material with a flexural strength greater than 1000 MPa and fracture toughness of 9 to 10 $MPa \cdot m^{1/2}$.[17-19] It seems that the high strength of zirconia material may have compensated for the strength loss of the tooth resulting from the onlay cavity preparation. The lithium-disilicate–based onlay restoration-tooth complex did not withstand compressive loads as high as the zirconia-based onlays.

There are conflicting results in the literature regarding the fracture resistances of teeth restored with inlay and onlay ceramics. The results of O-e.max in the present study contrasted with those reported by Yamanel and others[33] who stated that the onlay design is more effective in protecting tooth structures than the inlay design. Conversely, Morimoto and others[26] reported that the fracture strength of teeth restored with inlay and overlay feldspathic ceramics with cusp coverage was similar to that of intact teeth. Soares and others[1] stated that the fracture resistance values of posterior inlay and onlay leucite-reinforced ceramic restorations were significantly higher than those of intact teeth. Habekost and others[14] investigated the fracture resistance of premolars restored with partial ceramic restorations using two different brands of feldspathic porcelain. Their results indicated that the inlays generated a significantly higher fracture resistance than onlay designs, but lower than that of intact teeth. Stappert and others[22] investigated all-ceramic partial coverage restorations for molars made of IPS e.max Press and demonstrated that their fracture resistance was comparable to that of natural unprepared teeth. In their more recent study, Stappert and others[15] investigated the masticatory fatigue loading and fracture resistance of different all-ceramic partial coverage restorations on natural molars. They found that fracture resistance values for maxillary molars restored with ProCAD/Cerec 3 were similar to those of intact teeth, but were significantly higher than those of IPS Empress and IPS e.max Press. The results of this study are consistent with Stappert and others[15] since the mandibular molars restored with ICE Zirkon onlays had higher fracture strength values than those of teeth restored with IPS e.max Press. However, the results of the present study conflict with those of Cubas and others,[34] who found that the fracture strength values of onlays with In-Ceram cores did not differ from those of feldspathic onlays with a total onlay preparation.

In addition to fracture resistance, it is also important to analyze the fracture modes. In this study, fractures in the I-e.max and O-e.max groups were observed in the restoration itself or in the restoration involving a small tooth portion. Soares and others[13] reported similar results using feldspathic ceramic in extensive inlays, and Burke[24] also affirmed that ceramic fractures before the natural tooth. In the I-Zirkon and O-Zirkon groups, fracture was observed in both the restoration and in the tooth. More severe fractures occurred in both the restoration and the tooth in the ICE Zirkon groups when compared with the IPS e.max Press groups. However, the more severe fractures that were observed in the ICE Zircon groups were in excess of what may ever happen in the oral environment.

Cavity preparation should be based primarily on the preservation of dental structure and on physical properties of the restorative materials. Khera and others[38] studied the effect of preparation depth, isthmus width, and interaxial dentin thickness on the potential for tooth fracture. They concluded that the depth of the preparation was the most critical factor in tooth fracture, whereas the width of the isthmus alone was the least critical. In the current study, ceramic inlays reinforced the dental structure of teeth that were prepared with one half of the intercuspal width, obtaining stiffness values that were similar to those of intact teeth. The lowest fracture strength values were recorded in the lithium-disilicate glass-ceramic onlay groups. Using this restorative material, preparations resulting in a greater loss of tooth structure appear to decrease the fracture resistance of the tooth-restoration complex. In zirconia-based ceramic groups, there were no significant differences in fracture strength values between different cavity preparation designs. With respect to limitations of this study, it was observed that fracture resistances of partial coverage restorations are material-dependent.

Restorations can fracture because of crack formation and propagation, which is especially true for ceramic restorations.[25] As preparations increase in size, the remaining tooth structure weakens, and occlusal loads induce greater cusp deflection. Some researchers have suggested that optimal restorations in teeth with large Class II MOD preparations

are onlays that include cuspal coverage to reduce cuspal flexion under load.[26] Debate remains regarding the point at which onlays should be recommended instead of bonded inlays.[25]

It is difficult to determine which restorative material would be ideal for the restoration of posterior teeth.[40] In the past decades, indirect metal or amalgam restorations were the first choice for the restoration of partially destroyed teeth.[34,41] Magne and others[8] evaluated the fatigue strength of compromised molars restored using CAD/CAM composite resin inlays/onlays with and without fiber-reinforced immediate dentin sealing. They concluded that onlays (with or without fibers) increased the fatigue resistance of compromised molars. Dalpino and others[42] examined the fracture resistance of teeth restored with direct and indirect composite resin and indirect ceramic restorations. They found that bonded indirect ceramic restorations fractured at higher loads than direct and indirect composite resin restorations. A bonded indirect restoration using ceramic is the ideal option for restoration of teeth weakened by wide cavity preparation.[43] The advantage of posterior composites is that they can be placed in one appointment, while ceramic inlays usually require two appointments because of the time required for fabrication in the laboratory.

Resin cement used in adhesive restorations is elastic and tends to deform under stress, resulting in a higher resistance to fracture. Therefore, success of ceramic inlays is absolutely dependent on the creation of an uncompromised adhesive-tooth-ceramic interface.[44] Moreover, the elastic modulus of the luting agent may also affect the fracture strength values of the teeth restored with ceramic inlays and onlays. Cubas and others[34] found that luting agents with higher elastic modulus increased the fracture strength values of partial ceramic restorations.

This study also has some limitations. The continuous vertical load applied to the teeth in this study is not typical of clinical loading.[36] In terms of *in vivo* loading, the masticatory cycle consists of a combination of vertical and lateral forces, subjecting the ceramic to a variety of off-axis loading forces.[37] Cyclic loading may more accurately reproduce fatigue failures observed clinically. Other *in vitro* tests, such as stress distribution analysis, tension tests, and clinical studies need to be conducted to determine fracture strengths of various ceramic restorations with and without cuspal coverage.

CONCLUSIONS

Within the limitations of this study, the following conclusions were drawn:

1) Cuspal coverage decreased the fracture resistance of the posterior tooth and lithium-disilicate glass-ceramic restoration complex.
2) Teeth restored with zirconia ceramic inlays or onlays demonstrated fracture resistance similar to that of intact teeth.
3) The fracture modes in lithium-disilicate glass-ceramic samples were generally restricted to the restoration itself. Conversely, the fracture modes of zirconia samples generally involved both the restoration and the tooth.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service and/or company that is presented in this article.

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Influence of Different Surface Treatments on Bond Strength of Resin Composite Using the Intrinsic Characterization Technique

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Clinical Relevance

Surface treatments can adversely reduce the bond strength between resin composite and light-cured characterizing materials. Maintenance of the air-inhibited surface layer of resin composite is still the best alternative for optimizing the bond between resin composite and light-cured characterizing materials, therefore simplifying the clinical steps for performing dental characterization procedures.

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SUMMARY

Objective: This study evaluated the influence of different surface treatments on the resin bond strength/light-cured characterizing materials (LCCMs), using the intrinsic characterization technique. The intrinsic technique is characterized by the use of LCCMs between the increments of resin composite (resin/thin film of LCCM/external layer of resin covering the LCCM).

Materials and Methods: Using a silicone matrix, 240 blocks of composite (Z350/3M ESPE) were fabricated. The surfaces received different surface treatments, totaling four groups (n=60): Group C (control group), no surface treatment was used; Group PA, 37% phosphoric acid for one minute and washing the surface for two minutes; Group RD, roughening with diamond tip; and Group AO, aluminum oxide. Each group was divided into four

subgroups (n=15), according to the LCCMs used: Subgroup WT, White Tetric Color pigment (Ivoclar/Vivadent) LCCM; Subgroup BT, Black Tetric Color pigment (Ivoclar/Vivadent) LCCM; Subgroup WK, White Kolor Plus pigment (Kerr) LCCM; Subgroup BK, Brown Kolor Plus pigment (Kerr) LCCM. All materials were used according to the manufacturer's instructions. After this, block composites were fabricated over the LCCMs. Specimens were sectioned and submitted to microtensile testing to evaluate the bond strength at the interface. Data were submitted to two-way analysis of variance (ANOVA) (surface treatment and LCCMs) and Tukey tests.

Results: ANOVA presented a value of $p<0.05$. The mean values (±SD) for the factor surface treatment were as follows: Group C, 30.05 MPa (±5.88)a; Group PA, 23.46 MPa (±5.45)b; Group RD, 21.39 MPa (±6.36)b; Group AO, 15.05 MPa (±4.57)c. Groups followed by the same letters do not present significant statistical differences. The control group presented significantly higher bond strength values than the other groups. The group that received surface treatment with aluminum oxide presented significantly lower bond strength values than the other groups.

Conclusion: Surface treatments of composite with phosphoric acid, diamond tip, and aluminum oxide significantly diminished the bond strength between composite and the LCCMs.

INTRODUCTION

Light-cured characterizing materials (LCCMs) are fluid composites containing small load concentrations with colorants or unfilled resins with color tints. They are generally used to characterize occlusal fissures and sulci;[1,2] cover metal posts, darkened teeth, and teeth with metal pigments;[3] and mimic the chromatic characteristics of the tooth.[1,4] An LCCM may be presented in various pigments, such as brown, black, red, ochre, and white, among others, with the purpose of providing a direct restoration with a more natural and harmonious appearance in relation to the adjacent teeth.[5]

The application of LCCMs may be done by means of two techniques: extrinsic or intrinsic. With the extrinsic technique, the LCCM is applied with an explorer tip or brush after the restoration is finished and polished, with the LCCM flowing into the sculpted sulci. The LCCMs must be applied in thin films (<0.2 mm) followed by light activation. However, as LCCMs have low resistance to abrasion because of the smaller (or without) quantity of inorganic load incorporated into the organic matrix, they easily become worn when submitted to occlusal contacts and the restoration soon loses the characterization.[6–8]

To prevent the early loss of the effect of characterization, it is recommended that LCCMs should be covered with an external layer of resin composite. This modification in the application of LCCMs is what characterizes the intrinsic technique. With the intrinsic technique, LCCMs are used between the increments of resin composite. The LCCMs are applied and light activated on the previously light-activated resin composite; they are then covered with one or more layers of resin composite until the restoration is finished.[9] The average thickness of the additional layer(s) varies between 0.2 and 0.5 mm. Pucci and colleagues[10] observed that the intrinsic technique may lead to adverse alteration in the physical or mechanical properties of resin composite, weakening the resin composite/LCCMs/resin composite interfaces, which may promote early failure and compromise the durability of the restoration.

Traditionally, resin composites are compounds with dimethacrylate monomers that cure via a free-radical–induced polymerization. This free-radical polymerization reaction can be inhibited by atmospheric oxygen, resulting in a superficial layer of soft, sticky, liquid-like consistency and poorly polymerized monomer, referred to as an oxygen-inhibited layer. The oxygen inhibited layer is beneficial to interfacial resin-to-resin bonding during incremental technique because interfacial bonding can be increased due to the consistency of the oxygen-inhibited layer which increases the contact area between two contacting polymer increments.[11,12] Additionally, the oxygen-inhibited layer can let the polymers of both layers combine to form an interdiffused zone. This zone is marked by the formation of chemical bonds due to copolymerization.[11,12]

To increase the bond strength between the layers of resin, weakened by the use of the LCCMs, a possible alternative would be to perform surface treatment on the resin composite to optimize the resin composite/LCCM bond. Therefore, the aim of this study was to evaluate the influence of different surface treatments on the bond strength of resin composite and LCCMs using the intrinsic characterization technique.

Table 1: *Materials Used and Their Compositions*

Material	Manufacturer	Composition
Diamond tip 4103	KG Sorensen, Cotia, SP, Brazil	Stainless steel and grains of natural diamond with controlled dimensions.
Magic Acid Gel	Vigodent, Rio de Janeiro, RJ, Brazil	37% phosphoric acid
Airborne particle abrasion with aluminum oxide particles	Microetcher ERC, Danville Engineering, San Ramon, CA, USA	Aluminum oxide of 50 μm
Resin composite Filtek Z350	3M ESPE, St Paul, MN, USA	Bis-GMA (bisphenol A-glycidyl-methacrylate), UDMA (urethane dimethacrylate), TEGDMA (triethyleneglycol dimethacrylate), Bis-EMA (glycidyl ethoxylate dimethacrylate), 20 nm nanosilica filler, agglomerates of primary zirconia/silica particles with 5-20 nm fillers (78.5% by weight)
Kolor Plus	Kerr, Orange, CA, USA	Uncured methacrylate ester monomers, inert mineral fillers, photoinitiatiors, and stabilizing additives
Tetric Colors	Ivoclar/Vivadent, Schaan, Liechtenstein	Bis-GMA, UDMA, and TEGDMA (86% by weight); silanized silicone dioxide (12%–13 % by weight); catalyzers, stabilizers, and pigments (<2% by weight)

METHODS AND MATERIALS

Two hundred and forty blocks of resin composite (Filtek Z350/shade A3, 3M ESPE, St Paul, MN, USA) measuring $4 \times 4 \times 4$ mm were fabricated with the use of a silicone matrix. The composite, shade A3, was inserted into the silicone matrix using the incremental technique and light polymerized for 40 seconds per increment at an intensity of 500 mW/cm^2 (Curing Light XL 3000, 3M Dental Products, St Paul, MN, USA).

The resin composite blocks were divided into four groups (n=60) according to the type of surface treatment performed:

Group 1: control group, no surface treatment was used;

Group 2: Composite surface was treated with 10% phosphoric acid (Magic Acid Gel, Vigodent, Rio de Janeiro, RJ, Brazil) for one minute, followed by water rinsing for two minutes and air-drying;

Group 3: Composite surface was treated with diamond tip 4103 (Kg Sorensen, Cotia, SP, Brazil) standardized by five repetitions;

Group 4: Composite surface was sandblasted with 50 μm aluminum oxide particles (Micro-etcher ERC, Danville Engineering, San Ramon, CA, USA) for 10 seconds.

Next, each group was divided into four subgroups (n=15), according to the type of LCCM used:

Subgroup WT: Application of White Tetric Color pigment LCCM (Ivoclar/Vivadent, Schaan, Liechtenstein);

Subgroup BK: Application of Brown Kolor Plus pigment LCCM (Kerr, Orange, CA, USA);

Subgroup WK: Application of White Kolor Plus pigment LCCM (Kerr);

Subgroup BT: Application of Black Tetric Color pigment LCCM (Ivoclar/Vivadent).

For application of the surface coloring agent, a silicone mold measuring 4×4 mm and 0.4 mm high was used. The coloring agents were applied with a microbrush and light polymerized for 40 seconds at an intensity of 500 mW/cm^2 (Curing Light XL 3000).[6] The light unit tip was at a distance of 5 mm from the coloring agents. The materials used in the study and their compositions are listed in Table 1.

On the light-activated coloring agents, resin composite blocks (Filtek Z350/shade A3, 3M ESPE)

Table 2: *Results of ANOVA for Three Factors*

Factor	Degrees of Freedom	F	p
Surface treatment	3	18.47	0.000*
light-cured characterizing material (LCCM)	3	2.51	0.0656
Surface treatment × LCCM	9	4.37	0.0001*

** Significant differences.*

measuring 4 × 4 × 4 mm were made with the use of a silicone matrix. The specimens were immersed in water at 37°C for 48 hours.

After this, the specimens were submitted to thermomechanical wear (ER 37000, Erios, São Paulo, SP, Brazil). Mechanical cycling was performed with a 60N load and 100,000 cycles, with the force applied on the specimen perpendicular to the surface at the resin/LCCM interface. Simultaneously, the specimens were submitted to 100,000 cycles of thermal cycling at temperatures of 5°C, 37°C, and 55°C for 30 seconds each.

Parallel sections measuring approximately 1 mm were made using a diamond disc attached to a Labcut 1010 (Extec Technologies Inc, Perris, CA, USA) cutting machine. Sections were made at low speed under water cooling to prevent stress induction at the bond interface.

Specimens (≈6 sticks per block) were attached to a microtensile device in a universal testing machine EMIC (DL-1000, São José dos Pinhais, PR, Brazil) with a 10 kg load cell at a crosshead speed of 1 mm/min, according to the ISO 11405 Standard (Dental materials – Guidance on testing of adhesion to tooth structure). Data, expressed in megapascals (MPa), were submitted to parametric two-way analysis of variance (ANOVA) (surface treatment and LCCM) and Tukey post-hoc test, at a 5% level of significance.

Table 3: *Mean Bond Strength Values (±SD) for the Factor Surface Treatment and Tukey Test Results for All Groups*

Surface Treatment	Mean MPa Values (±SD)	Homogeneous Groups*
Control	30.05 (±5.88)	A
Phosphoric acid	23.46 (±5.45)	B
Diamond tip	21.39 (±6.36)	B
Aluminum oxide	15.05 (±4.57)	D

** Means accompanied by the same letters presented no statistically significant differences.*

The fractured specimens were analyzed under a stereomicroscope Stemi 2000 (Karl Zeiss, Jena, Germany) at 50× magnification for determining fracture type: adhesive, fractures in which the failure occurred at the composite (first block)/surface treatment/LCCM interface in more than 75% of the analyzed area; cohesive in resin, fractures in which the failure occurred at the LCCM/composite interface (second block, without surface treatment) or only in resin composite in more than 75% of the analyzed area; or mixed, fractures for which there was no predominance greater than 75% of any type of failure.

RESULTS

The results of three-way ANOVA are shown in Table 2. ANOVA showed a value of $p<0.05$ for the surface treatment factor and interaction between factors, which indicated that there were significant differences among the groups.

The bond strength means (±SD) and the results of Tukey test for the factor surface treatment are shown in Table 3. The control group presented significantly higher bond strength values than the other groups. The groups in which surface treatment was performed with the diamond tip and acid etching presented significantly higher bond strength than the group in which aluminum oxide was used.

The bond strength means (±SD) and Tukey test results for the interaction between factors are shown in Table 4. The control group associated with the White Kolor Plus pigment and Black Tetric Color pigment LCCMs presented significantly higher bond strength values than the group in which surface

Table 4: *Mean Bond Strength Values (±SD) for the Interaction Between the Factors and Tukey Test Results for All Groups/Subgroups*

Surface Treatment	Light-cured Characterizing Material	Mean MPa Values (±SD)	Homogeneous Groups*				
Control	Kolor Plus White	34.45 (±5.88)	A				
Control	Tetric Color Black	33.75 (±4.06)	A				
Control	Tetric Color White	28.93 (±4.14)	A	B			
Phosphoric acid	Kolor Plus Brown	27.81 (±5.13)	A	B	C		
Diamond tip	Tetric Color Black	25.38 (±5.62)	A	B	C	D	
Phosphoric acid	Tetric Color White	24.62 (±3.58)	A	B	C	D	
Diamond tip	Tetric Color White	24.19 (±5.87)	A	B	C	D	
Aluminum oxide	Kolor Plus White	23.26 (±5.51)	A	B	C	D	
Control	Kolor Plus Brown	22.34 (±6.08)	A	B	C	D	
Phosphoric acid	Tetric Color Black	21.35 (±6.01)		B	C	D	
Diamond tip	Kolor Plus White	20.75 (±5.48)		B	C	D	E
Phosphoric acid	Kolor Plus White	19.67 (±3.55)		B	C	D	E
Aluminum oxide	Tetric Color White	17.76 (±4.90)		B	C	D	E
Diamond tip	Kolor Plus Brown	15.72 (±5.0)			C	D	E
Aluminum oxide	Kolor Plus Brown	13.89 (±3.7)				D	E
Aluminum oxide	Tetric Color Black	8.75 (±2.74)					E

** Means accompanied by the same letters presented no statistically significant differences.*

treatment was performed with phosphoric acid associated with the Black Tetric Kolor and White Kolor Plus LCCMs, than the group in which surface treatment was performed with a diamond tip associated with the White Kolor Plus and Black Kolor Plus LCCMs, and than the group in which surface treatment was performed with aluminum oxide associated with the White Tetric Kolor, Brown Kolor Plus and Black Tetric Color LCCMs. The control group associated with the White Tetric Color LCCM presented significantly higher bond strength values than the group in which surface treatment was performed with the diamond tip associated with the Brown Kolor Plus LCCM and than the groups in which surface treatment was performed with aluminum oxide associated with the Brown Kolor Plus and Black Tetric Color LCCMs.

Figure 1 shows the representative graph of the mean bond strength values for all the groups/subgroups.

With regard to the fracture results, the values shown in Table 5 were obtained. Adhesive fractures at the resin/surface treatment/LCCM interface were higher than the cohesive fractures (LCCM/composite

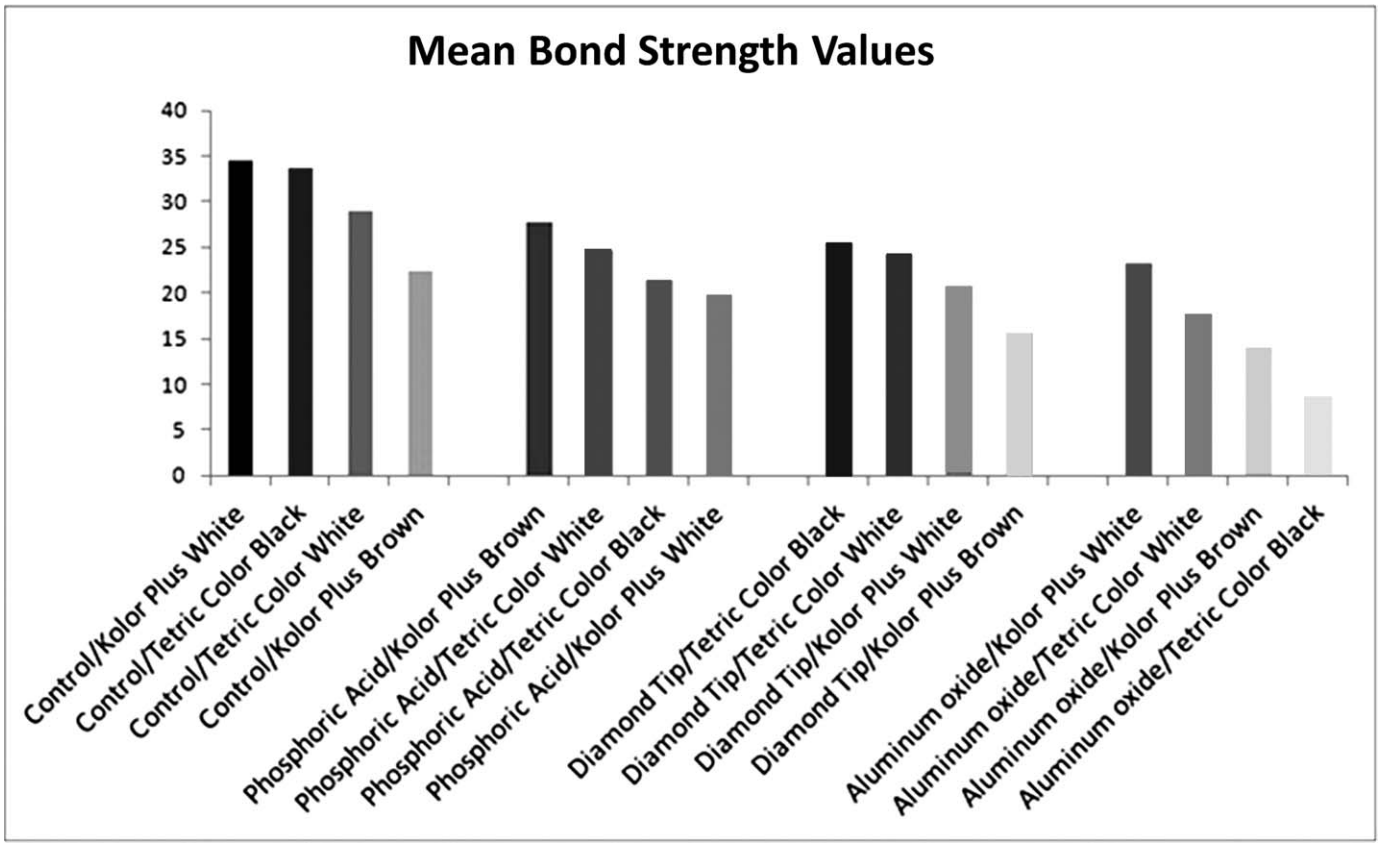

Figure 1. *Representative graph of the mean bond strength values for all the groups and subgroups.*

or only in composite) and suggest failure in the adhesive process.

DISCUSSION

Different surface treatments have been reported for improving the bond between the repair resin composite and the existing resin composite restoration. Roughening composite bonding surfaces with high-speed diamond burs can produce irregularities on the resin composite surface. This has resulted in an increased surface area for micromechanical retention, thereby increasing the repair bond strength.[13] Sandblasting with aluminum oxide increases the surface irregularity and wetting potential of the repair composite,[13] and sandblasting with aluminum oxide is an efficient and cost-effective procedure for repair of aged composite restorations.[14] Surface treatment with phosphoric acid at 37% removes residues on the surface, which facilitates the bond between layers of composites.[15,16] It also seems to remove the organic contamination of the composite surface.[17]

The intrinsic characterization technique of resin composites using surface LCCMs is routinely used in the dental office. In 2011, Pucci and colleagues[10] observed that the use of Tetric Kolor (White and Black pigments) and Kolor Plus (White and Brown pigments) LCCMs with the intrinsic technique significantly reduced the bond strength between the resin composite layers. Therefore, the aim of this study was to observe whether different resin composite surface treatments used to improve repair procedures could optimize the bond strength between resin composite and LCCMs used in the intrinsic technique.

The results of the present study demonstrated that the control group, in which no surface treatment was performed on resin composite before the use of LCCMs, presented higher bond strength values compared with the groups in which surface treatment was performed with phosphoric acid, diamond tips, and aluminum oxide. LCCMs are resin composites that have a high concentration of organic matrix and small concentrations of load. Resin composite polymerization begins with chemical chain reactions by breaking apart the double carbon bonds for the formation of polymers.[18] The bond strength between successive increments of

Table 5: *Classification With Regard to Fracture Type (Number of Sticks)*

Surface Treatment	Light-cured Characterizing Material	Adhesive Fracture	Cohesive Fracture	Mixed	Sticks Lost
Control	Tetric Kolor White	33	36	27	0
Control	Tetric Kolor black	30	39	27	3
Control	Kolor Plus White	33	36	30	0
Control	Kolor Plus Brown	45	39	24	0
Phosphoric acid	Tetric Kolor White	99	0	0	0
Phosphoric acid	Tetric Kolor black	63	36	0	3
Phosphoric acid	Kolor Plus White	57	36	3	0
Phosphoric acid	Kolor Plus Brown	75	27	0	0
Diamond tip	Tetric Kolor White	66	24	0	0
Diamond tip	Tetric Kolor black	90	0	0	3
Diamond tip	Kolor Plus White	63	24	3	0
Diamond tip	Kolor Plus Brown	60	39	0	0
Aluminum oxide	Tetric Kolor White	57	30	0	0
Aluminum oxide	Tetric Kolor black	63	27	0	6
Aluminum oxide	Kolor Plus White	57	33	3	0
Aluminum oxide	Kolor Plus Brown	54	30	0	3
Total		945	456	117	18

resin composite occurs because of the presence of an oxygen-inhibited layer of polymerization. This layer is viscous and has unreacted methacrylates that, by means of covalent bonds, will link to the polymer chains, optimizing the bond strength between substrates.[19–21]

The groups in which surface treatment was performed with phosphoric acid, diamond tips, and aluminum oxide presented lower bond-strength values than the control group because of the removal of the oxygen-inhibited layer, leaving the inorganic matrix on the surface without unreacted methacrylates[19–21] and negatively influencing the bond strength between the substrates. Therefore, these techniques are proposed for improving the bond to the repair of aged resin composite and fresh resin composite.

The results of the present study demonstrated that the group in which surface treatment was performed with aluminum oxide presented significantly lower bond strength values than the groups in which surface treatment was performed with the diamond tip or phosphoric acid. The purpose of surface treatment by the mechanical method (air-

borne aluminum oxide particle abrasion and diamond tip) is to create porosities on the surface to increase micromechanical retention between the layers of the substrate. The objective of chemical surface treatment using phosphoric acid is to perform cleaning of the resin surface to be repaired, thereby improving the bond strength between the resin composite layers.

Some authors [22,23] have affirmed that airborne aluminum oxide particle abrasion promotes greater irregularity on the surface of the substrate to be repaired, when compared with the use of a diamond tip alone. Consequently, airborne particle abrasion increases the surface area, optimizing micromechanical retention between the resin composite layers.[22,23] In addition, several authors have affirmed that the use of airborne aluminum oxide particle abrasion could result in a bond strength close to the cohesive strength of the original resin.[22,23]

However, the oxygen inhibited layer of polymerization is viscous. The reduction in bond strength obtained with surface treatment with aluminum oxide could probably be attributed to the adherence of aluminum oxide particles on this surface layer with unreacted methacrylates, which prevented an effective bond between the LCCMs and resin composite.

The results of the present study also demonstrated that the control group associated with Kolor Plus White and Tetric Color Black LCCMs presented significantly higher bond strength values than the groups in which surface treatment was performed with phosphoric acid associated with Tetric Kolor Black and Kolor Plus White LCCMs, than the groups in which surface treatment was performed with a diamond tip associated with Kolor Plus White and Kolor Plus Black LCCMs, and than the groups in which surface treatment was performed with aluminum oxide associated with Tetric Kolor White, Kolor Plus Brown, and Tetric Color Black LCCMs. As previously described, this may be because removal of the surface layer of inhibited air induced greater fragility of this layer, which negatively influenced the bond strength between the substrates.[18-21]

In addition, the control group associated with the Tetric Color White LCCM presented significantly higher bond strength values than the group in which surface treatment was performed with the diamond tip associated with the Kolor Plus Brown LCCM and than the groups in which surface treatment was performed with aluminum oxide associated with the Kolor Plus Brown and Tetric Color Black LCCMs. Therefore, these results indicate the importance of the presence of an oxygen-inhibited layer of polymerization to improve the bond strength between successive increments of resin composite.[18–21]

Furthermore, our results showed that the control group associated with the Kolor Plus Brown LCCM was grouped differently from the other control groups. This result might be attributed to the color of the pigment present in the composition of the Kolor Plus Brown LCCM; according to Pucci and colleagues,[10] the darker-pigmented LCCMs showed a greater influence between layers of composites decreasing the cohesive strength of the composite. Beyond that, even though the Kolor Plus Brown LCCM has an organic matrix similar to the composites, the quantity of inorganic filler or the quantity of the pigment present in this material may have affected the cohesive strength of the Kolor Plus Brown LCCM and composite more than the others LCCMs tested. Also, there may be some incompatibility between darker pigment and organic filler in these LCCMs, which could reduce radical polymerization of methacrylate C=C bonds. Unfortunately, the manufactures do not provide these specifications of the LCCMs studied.

With regard to the fracture type, it was observed that the adhesive fractures at the resin/surface treatment/LCCM interface were predominant to the cohesive fractures (LCCM/composite or only in composite) in the groups in which surface treatment was performed. Therefore, these results suggest that there was failure in the adhesive process at the interface in which surface treatment occurred, probably because of the removal of the surface layer of inhibited air, inducing greater fragility of this layer.[18–21]

In addition, it could be observed that the cohesive fracture modes were more evident with the control groups than the surface treatment groups. These results confirm the statistically higher cohesive force among the resin interfaces for control groups without surface treatment, probably because of the presence of an oxygen-inhibited layer of polymerization, which forms a better bond at the interfaces.[18–21]

The results of the present study demonstrated that surface treatments with diamond tips, aluminum oxide, and phosphoric acid significantly reduced the bond strength between the resin composite and LCCMs. Maintenance of the air-inhibited surface layer of resin composite is still the best alternative for optimizing the bond between resin

composite and LCCMs, therefore simplifying the clinical steps for performing dental characterization procedures.

CONCLUSION

According to the methodology used and the data obtained, it may be concluded that surface treatments of composite with phosphoric acid, diamond tips, and aluminum oxide significantly diminished the bond strength between resin composites and LCCMs. The groups in which surface treatment was performed with the diamond tip and acid etching presented significantly higher bond strength than the group in which aluminum oxide was used.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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Effect of Postoperative Peroxide Bleaching on the Marginal Seal of Composite Restorations Bonded With Self-etch Adhesives

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Clinical Relevance

If postoperative bleaching is expected, composite restorations should be bonded preferably with well-proven etch-and-rinse adhesive systems.

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SUMMARY

The aim of this study was to determine the effect of peroxide bleaching on the marginal seal of composite restorations bonded with several adhesive systems. Combined cylindrical Class V cavities located half in enamel and half in dentin were prepared on the buccal and lingual surfaces of human molars. The cavities were bonded with the self-etch adhesives Clearfil SE-Bond (CLF), Adper Prompt (ADP), and iBond (IBO) and an etch-and-rinse adhesive Gluma Comfort Bond (GLU) and restored with a microhybrid composite Charisma. Experimental groups were treated 25 times for eight hours per day with a peroxide bleaching gel Opalescence PF 20, while the control groups were stored in distilled water for two months and then subjected to a microleakage test using a dye penetration method. Scanning electron microscopy was used to investigate the etching and penetration abilities of the adhesives and morphology of debonded resto-

ration-enamel interfaces after the microleakage tests. Statistical analyses were performed using nonparametric Kruskal-Wallis, Mann-Whitney, and Wilcoxon tests at $p=0.05$. The microleakage of all GLU groups was low and not significantly affected by peroxide bleaching. Low microleakage was recorded for CLF control groups, but after bleaching, a small but significant increase in microleakage at the enamel margin indicated its sensitivity to peroxide bleaching. For ADP and IBO control groups, the microleakage at the enamel margins was significantly higher than for GLU and CLF and exceeded that at the dentin margins. Bleaching did not induce any significant changes in the microleakage. Electron microscopy analysis indicated that in our experimental setup, decreased adhesion and mechanical resistance of the ADP- and IBO-enamel interfaces could be more important than the chemical degradation effects induced by the peroxide bleaching gel.

INTRODUCTION

Tooth discolorations caused by exogenous factors, such as smoking, absorption of pigments from foods and drinks, or frequent mouth washing with antimicrobial rinses, can be removed by peroxide bleaching. Some studies, however, show that reactive oxygen species released from peroxide products may attack not only the staining moieties captured in the enamel structure but also hard tooth tissues and reconstruction materials. In contrast, little attention has been paid to the effect of postoperative peroxide bleaching on the durability of the adhesive interface between composite restorations and tooth tissues. The results obtained by bond strength[1-3] and microleakage[4-10] measurements, however, are contradictory and do not clearly illustrate this effect.

Adhesion between the tooth tissues and restoration materials has been tested with a wide variety of experimental approaches. In bond strength measurements, the composite build-ups are often made on flattened surfaces of teeth. The configuration factor (C-factor), defined as the ratio of the bonded to unbonded surface, and thus the polymerization shrinkage stress effects, are low in such restorations.[11] Under these conditions, the adhesive interface is primarily challenged by the ambient environment. With microleakage tests, however, the C-factor is significantly higher, and thus the adhesive interface is stressed not only by the environment but also by shrinkage stress, which can accelerate degradation of the interface.

In a previous study,[3] the resistance of an adhesive interface against peroxide bleaching degradation was investigated by the shear bond strength tests at a C-factor of approximately 0.30 using four self-etch and etch-and-rinse adhesive systems that represented typical currently used adhesives associated with different working protocols. A decrease in the bond strength indicated degradation of the adhesive interface created with the one-step self-etch adhesives Adper Prompt and iBond.

In the present study, we focused on evaluating the resistance of an adhesive interface created with the same adhesive systems used in the previous bond strength test, but using the microleakage test instead. It was assumed that degradation of the adhesive interface due to peroxide bleaching would be more pronounced under a higher shrinkage stress than that observed with a bond strength measurement. The null hypothesis was that the marginal seal of the composite restorations would not be impaired by peroxide bleaching.

MATERIALS AND METHODS

The two-bottle, two-step Clearfil SE Bond (CLF; Kuraray Medical Inc, Okayama, Japan); the two-bottle, one-step Adper Prompt (ADP; 3M ESPE AG, Seefeld, Germany); and the one-bottle, one-step iBond (IBO; Heraeus Kulzer GmbH, Hanau, Germany) self-etch adhesives were used. These adhesives were compared with that associated with the one-bottle, two-step etch-and-rinse Gluma Comfort Bond, combined with an etching gel, Gluma Etch 20 Gel (GLU; Heraeus Kulzer GmbH). All restorations were made with a microhybrid composite Charisma (shade A2, Heraeus Kulzer GmbH) to avoid any unwanted effects of varying composites. The home bleaching peroxide gel Opalescence PF 20% (Ultradent Products Inc, South Jordan, UT, USA), containing 20% carbamide peroxide, was used in the study. All materials came from the same production batches. Their composition and application protocols are summarized in Table 1. After delivery, the restorative materials were stored at 10°C to slow down hydrolysis of self-etch adhesives.[12] The bleaching gel was stored at 4°C and was used in the first quarter of its shelf life to minimize spontaneous peroxide gel decomposition during the testing period.

Forty human noncarious third molars extracted for orthodontic reasons were used in the study. After extraction, the teeth were cleaned and stored at a

Table 1: *Materials Used and Their Working Protocols*

Material	Manufacturer	Chemical Composition	Application[a]
Adhesive system			
Gluma Comfort Bond (GLU)	Heraeus Kulzer GmbH, Hanau, Germany	Etchant: Gluma Etch 20 Gel (phosphoric acid 20%)	e (20 s), r, d (1-2 s), 3× b (15 s), w (15 s), d, c (20 s)
		Bond: HEMA, 4-META, polyacid, ethanol, photoinitiators, polyacrylic acids	
Clearfil SE Bond (CLF)	Kuraray Medical Inc, Okayama, Japan	Primer: MDP, HEMA, hydrophilic dimethacrylate, camphorquinone, *N,N*-diethanol-*p*-toluidine, water	p (20 s), d, b, d, c (10 s)
		Bond: MDP, bis-GMA, HEMA, hydrophobic dimethacrylate, camphorquinone, *N,N*-diethanol-*p*-toluidine, silanated colloidal silica	
Adper Prompt (ADP)	3M ESPE AG, Seefeld, Germany	A liquid: methacrylated phosphoric esters, bis-GMA, initiators based on camphorquinone, stabilizers	m (A+B), a (15 s), d, a, d, c (10 s)
		B liquid: water, HEMA, polyalkenoic acid, stabilizers	
iBond (Gluma inside) (IBO)	Heraeus Kulzer GmbH	4-META, UDMA, glutaraldehyde, acetone, water, photoinitiators, stabilizers	3× a, w (30 s), d, c (20 s)
Composite material			
Charisma	Heraeus Kulzer, GmbH	bis-GMA, TEGDMA, UDMA, barium fluoride glass, silicon dioxide, initiators, stabilizers, pigments	c (20 s)
Bleaching gel			
Opalescence PF 20	Ultradent Products Inc, South Jordan, UT, USA	Carbamide peroxide 20 weight %, sodium fluoride 0.25 weight % potassium nitrate	25× 8 h

Abbreviations: bis-GMA, bisphenol A diglycidyl methacrylate; 4-META, 4-methacryloxyethyl trimellitic anhydride; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.
[a] *Application protocol with a recommended time (in seconds): a, application; b, bonding; c, light curing; d, drying/spreading; e, etching; m, mixing; p, priming; r, rinsing; w, waiting.*

temperature of approximately 4°C in 0.5 % chloramine-T solution for one week. They were then stored in distilled water for up to six months at this same temperature.[13] On the buccal and lingual tooth surfaces, 80 standardized cylindrical Class V cavities, with diameters of approximately 3 mm, depths of approximately 1.5-2.0 mm, and a C-factor of 3-4, were cut by one operator using a spherical coarse diamond bur (100 μm, Hager & Meisinger, Neuss, Germany) under water cooling. The cavities were finished with a cylindrical tungsten carbide bur (Acurata G+K Mahnhardt Dental, Thurmansbang Germany) in a high-speed handpiece and were cooled with an air-water spray. The burs were replaced after preparation of 10 cavities. One half of the cavity was located in enamel and the other half in dentin, with a cavosurface angle of approximately 90°. No beveling was made at the cavity margins. Ten teeth, divided into experimental and control groups of 10 cavities, were randomly chosen for each adhesive system. Both the adhesive systems and the composite material were applied by the same operator, strictly following the manufacturer's recommendations. The cavities were restored incrementally, the first increment placed occlusally up to approximately half of the cavity depth and the second increment placed gingivally in contact with the first increment. The last increment restored the

anatomical shape of the tooth. Each increment was polymerized for 20 seconds using an Elipar TriLight halogen lamp (3M ESPE AG) with a power intensity of 800-850 mW/cm^2 that was checked periodically using a calibrated handheld radiometer EVT 460 (Preciosa, Jablonec and Nisou, Czech Republic). After polymerization the restorations were slightly polished with silica-carbide sandpaper P1200 with a particle size of 15 μm (Buehler Ltd, Lake Bluff, IL, USA) under water cooling with an Ecomet III polishing machine (Buehler Ltd) to prepare defined restoration margins without overlaps.

The experimental groups were subjected to 25 bleaching cycles. Each cycle included application of approximately 0.1 g of bleaching gel on the restoration margin. The teeth with the gel were wrapped in moisture-resistant Parafilm foil (Parafilm M, Alcan Packaging, Chicago, IL, USA) and stored in a 100% relative humidity environment. After eight hours, the peroxide gel was carefully removed from the tooth surface under running water using a soft toothbrush, and the teeth were stored in distilled water until the next application. To prevent microbial growth, each tooth was stored in 20 mL of water with approximately 100 ppm of sodium azide per 1 L of distilled water. The control teeth were stored in similarly treated distilled water (replaced every four to five days) for the two months during which the bleaching tests took place in the experimental groups. All of the exposures were performed at 37°C. After the bleaching program was finished, the apices and the surfaces of the teeth were carefully sealed with two layers of nail varnish and one layer of sticky casting wax, except for a 1-mm zone around the restoration margin. The teeth were immersed for 24 hours in a 2% methylene blue aqueous solution at 23°C and were then rinsed, dried, and fixed in polyethylene rings with the self-curing methylmethacrylate resin Spofacryl (Spofa-Dental, Jičín, Czech Republic). The restorations were cut into three parts, in the occlusal-cervical direction, using an Isomet low-speed saw equipped with a water-cooled diamond wafering blade (Buehler Ltd).

Microleakage Evaluation

The depth of dye penetration was evaluated using a Nikon SMZ 2T optical stereomicroscope with 10-20× magnification. A 5° microleakage score was implemented for enamel and dentin, with scoring criteria as follows: 0 = no dye penetration; 0.5 = penetration up to one-fourth of the cavity depth; 1 = penetration up to one-half of the cavity depth, typically equal to the whole depth of the enamel layer on the enamel margin; 2 = penetration over one-half of the cavity depth to its floor; and 3 = penetration including the cavity floor. Evaluation was performed by three calibrated subjects, and the consensual value was considered in the case of score variances. For each restoration, the highest scores on the enamel and dentin margins were used for statistical evaluation.[14] With the exception of the ADP and IBO experimental groups 10 scores were obtained for the remaining groups. As a result of dye penetration through the apices one restoration had to be eliminated in each of these two groups. The Kruskal-Wallis analysis of variance tests by ranks, followed by multiple comparisons of mean ranks, were used for the identification of significant differences in the microleakage of teeth restored using different adhesives. Within each adhesive system, the Mann-Whitney *U*-test, corrected for ties, was used to analyze the effect of bleaching on the enamel and dentin margins. Lastly, the Wilcoxon matched-pairs test was used to evaluate differences in the microleakage observed at the enamel and dentin margins. All of the statistical analyses were performed using statistical software (Statistica 10, StatSoft Inc, Tulsa, OK, USA) with a significance level of 0.05.

Scanning Electron Microscope (SEM) Analysis

To evaluate the etching and penetration abilities of the adhesive systems, eight (n=2) more restorations were placed in the same way as the teeth that were prepared for the microleakage tests. After 24 hours in distilled water, the restorations were sectioned into two parts in an occlusal-cervical direction, demineralized in 6 N HCl for 24 hours, and then immersed into 5% NaOCl for 10 minutes to eliminate organic substances from the composite surface.[15] The composite surface was rinsed with distilled water, cleaned in an ultrasound bath, air-dried, sputter-coated with gold, and examined using a SEM (SEM, Jeol 5500, Tokyo, Japan). To investigate the morphology of the enamel-composite interface created with ADP and IBO, where increased dye penetration indicated marginal failure, tooth tissues adjacent to the restoration were broken off by gentle force, and the surfaces of both the enamel and the composite were analyzed with a SEM.

RESULTS

Table 2 shows the distribution of the microleakage scores and the results of the statistical evaluations. The results of the Kruskal-Wallis analyses indicate

Table 2: *Microleakage Scores at the Enamel and Dentin Margins and Statistical Analysis Results. Score 3 Was Not Observed in Any Group*[a]

Adhesive	Enamel						Dentin						WLC Test
	Microleakage Score				K-W Test	M-W Test	Microleakage Score				K-W Test	M-W Test	
	0	0.5	1	2			0	0.5	1	2			
GLU													
BG	5	3	2	0	A	NS	3	7	0	0	A	NS	NS
W	5	2	3	0	A		3	4	3	0	A		NS
CLF													
BG	1	1	7	1	A	0.030	6	1	3	0	A	NS	0.043
W	4	3	3	0	A		4	3	3	0	A		NS
ADP													
BG	0	0	6	3	B	NS	0	8	1	0	A	NS	0.008
W	0	0	8	2	B		5	4	1	0	A		0.008
IBO													
BG	0	0	8	1	B	NS	1	6	2	0	A	NS	0.02
W	0	0	7	3	B		3	7	0	0	A		0.005

Abbreviations: BG, bleached groups; W, control groups stored in water; K-W, Kruskal-Wallis statistics to test differences among adhesives; M-W, Mann-Whitney to test differences between BG and W groups; WLC, Wilcoxon to test differences between enamel and dentin margins.
[a] *Different letters within each column indicate a significant difference, NS, nonsignificant difference;* p = *0.05.*

that microleakage at the enamel margin of the GLU and CLF control groups stored in water was low and significantly smaller than that of the ADP and IBO groups ($p<0.0001$). With these two adhesives, scores of 1 or higher indicated microleakage within the whole thickness of the enamel layer. At the dentin margin of the control groups, no significant differences between adhesives were found ($p>0.65$). After bleaching, a slight yet significant ($p<0.030$) increase in microleakage at the enamel margin for CLF was revealed by the Mann-Whitney *U*-test (Table 2). This increase, however, did not affect the differences among the adhesives, which varied in the same order (GLU=CLF<ADP=IBO) as did the control groups ($p<0.0003$). At the dentin margin, no significant differences were detected after bleaching ($p>0.20$). The Wilcoxon pairs test revealed significantly higher microleakage at the enamel margin than at the dentin margin for the control groups of ADP ($p<0.008$) and IBO ($p<0.005$); these tests also demonstrated increased enamel, compared with dentin microleakage, after bleaching for the CLF ($p<0.043$), ADP ($p<0.008$), and IBO ($p<0.02$) groups.

Figure 1 shows the morphology of the enamel and dentin interfaces, which characterize the abilities of adhesive systems to demineralize and penetrate into the tooth tissues. For GLU, well-developed, long resin tags in enamel and in dentin, with clearly visible lateral branches, were observed (Figure 1a). The resin tags formed in the ADP and CLF enamel and dentin interfaces shortened and were less distinct compared to those associated with the GLU (Figure 1b,c). In the IBO-enamel interface a shallow

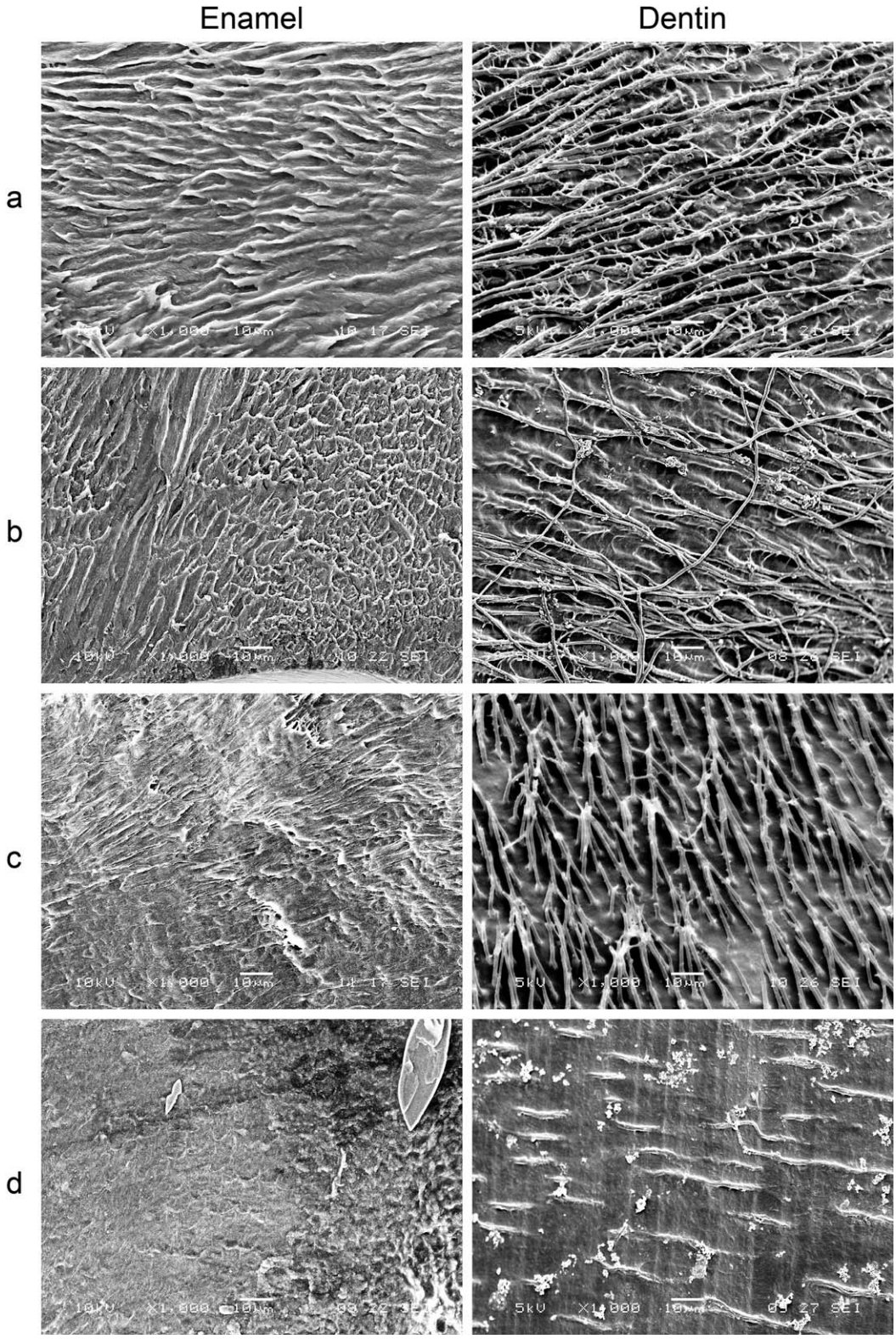

Figure 1. *Morphology of enamel and dentin interfaces treated with various adhesive systems. Tooth tissues removed from the composite restoration surface by HCl and NaOCl: (a) GLU with long resin tags in both enamel and dentin interfaces. Resin tags in dentin with lateral branches parallel to the Class V cavity walls; (b) ADP with short tags in the enamel; (c) CLF with less distinct resin tags and the short dentin lateral branches; (d) IBO with poorly developed resin tags in the enamel and dentin interface and extensive porosity below the enamel-dentin junction.*

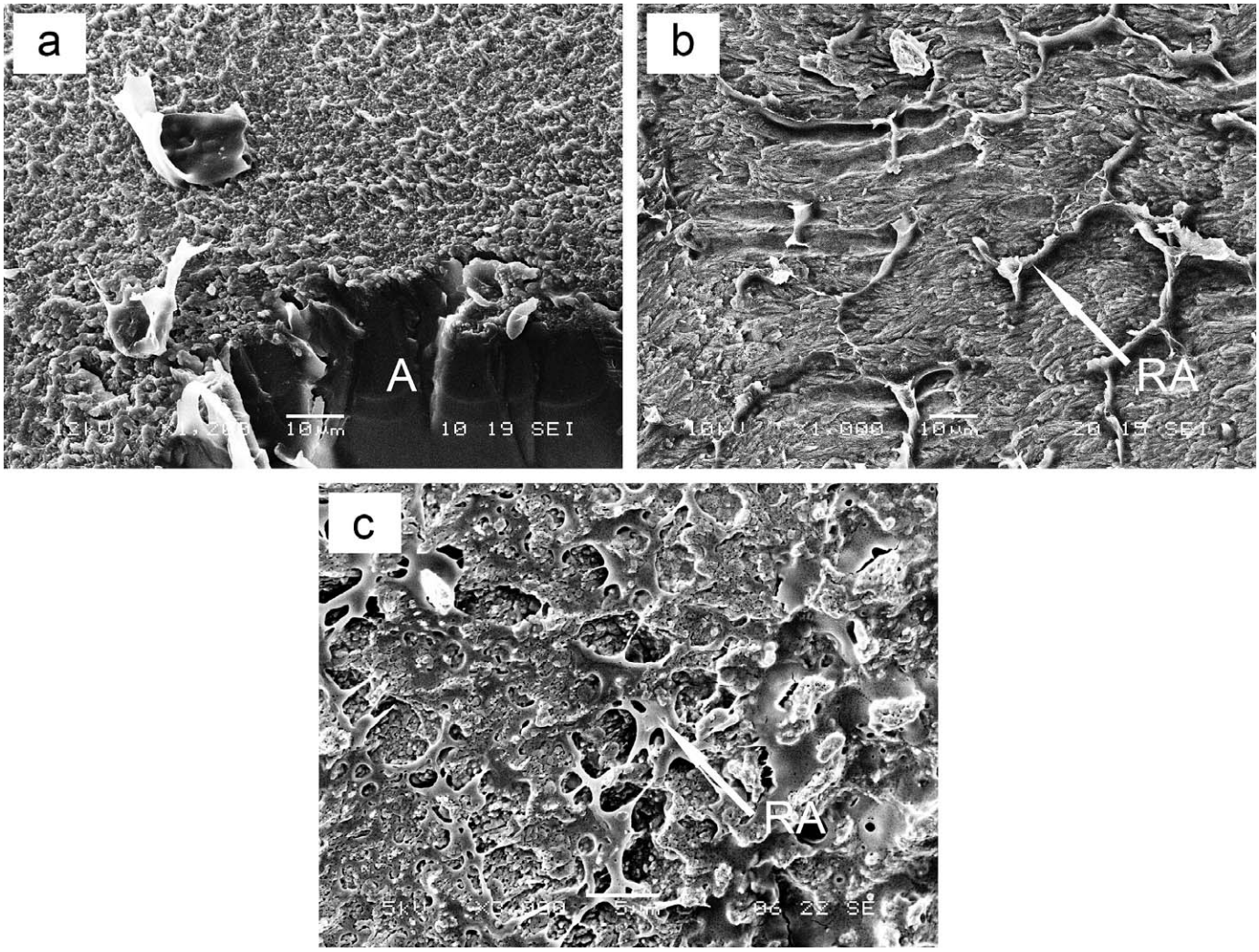

Figure 2. *Composite-enamel surfaces of ADP bonded restorations after peroxide treatment and microleakage test, composite surfaces: (a) distinct prismatic structure on the top of the adhesive layer, (b) remnants of ADP film pulled up from the enamel structure, (c) area of adhesive film disrupted with voids. A, adhesive resin; RA, a thin film of adhesive's remnants disrupted by voids.*

prismatic structure could be recognized (Figure 1d), and in the dentin interface, only a small number of poorly developed resin tags were formed (Figure 1d). With IBO, extensive porosity was observed at deeper locations of the cavities below the dentin-enamel junction (Figure 1d). Analysis of the composite and enamel surfaces, where the dye penetrated along the ADP-bonded cavity, revealed their prismatic structure (Figure 2a), indicating failure at the enamel-adhesive interface. However, some areas of enamel or composite surfaces were covered with remnants of a thin film of the adhesive, pulled out from the enamel structure (Figure 2b) and disrupted by small voids (Figure 2c). Similarly analyzed surfaces of the IBO restorations displayed a prism-less texture and discrete grinding grooves representative of failure at the enamel-adhesive layer interface. Below the enamel-dentin junction extensive porosity in the adhesive layer was found (Figure 3a,b).

DISCUSSION

Frequent esthetic bleaching of vital teeth with peroxide products may adversely affect soft and hard oral tissues,[16,17] restoration materials, and the quality of the marginal seal. The risk of marginal failure due to degradation of the adhesive interface by oxygen radicals is particularly relevant for self-etch systems, which are prone to increased water sorption[18] and thus may be more susceptible to the penetration of small oxygen molecules than are etch-and-rinse systems. Scission of the three-dimensional polymer network, the addition of oxygen radicals to unpolymerized monomers' double bonds, or reaction of radicals with ester groups might accelerate the

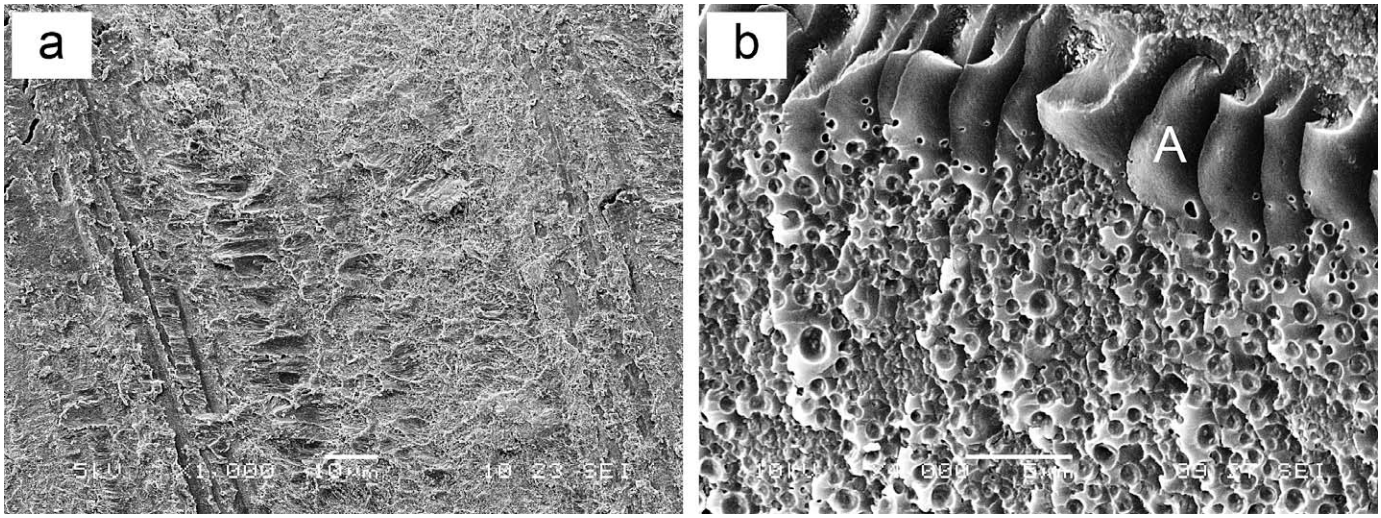

Figure 3. *Composite-enamel surfaces of IBO bonded restorations after peroxide treatment and microleakage test, composite surfaces: (a) top of the adhesive layer without prismatic structure, grinding grooves caused by cutting burs are clearly visible; (b) extensive porosity at the bottom of the adhesive layer at deep parts of cavities below the enamel-dentin junction. A, adhesive resin.*

degradation of the adhesive interface in a similar fashion to that of the polymer matrix of composite materials.[19]

Peroxide-induced degradation of the adhesive interface, however, has not yet been fully elucidated. Inconsistent results in the literature may be due to the different adhesives and bleaching systems studied, differences in bleaching protocols, and the various test methods employed, including, typically, bond strength and microleakage measurements. To clarify some of these discrepancies, the effect of bleaching on marginal integrity was evaluated in this study using the identical adhesives, peroxide bleaching gel, and application protocols of the previous research performed using the shear bond strength measurement.[3] We supposed that comparison of the bond strength results and microleakage should contribute to our understanding of the effects of peroxide bleaching on the composite-tooth tissue interfaces and help to clarify differences in adhesive performance often found with these methods.[20] The adhesives used differed in composition and application protocols (Table 1). The self-etching systems were represented by a two-step mild aggressive CLF, in which an acidic primer and hydrophobic bond are applied to tooth tissues in separate steps. ADP is a strongly acidic one-step self-etch adhesive containing hydrophilic 2-hydroxyethyl methacrylate and hydrophobic monomers. However, volatile solvents, such as ethanol or acetone, which usually serve to remove water from an adhesive layer, are not included in its formulation (Table 1). While these two adhesives contain phosphoric acid esters as self-etch primers, a mild all-in-one adhesive IBO is based on a weak acidic organic monomer, 4-methacryloxyethyl trimellitic anhydride. In its composition acetone is used as a solvent to facilitate water removal (Table 1). These adhesives were compared with the well-proven etch-and-rinse adhesive GLU.[21-23] Microleakage tests performed on the control groups stored in water showed that none of the adhesives guaranteed a perfect marginal seal and that the enamel margins created with ADP and IBO were more susceptible to failure than were those of GLU and CLF. After bleaching, a small but significant increase ($p<0.030$) in microleakage was found for CLF at the enamel margin only. Hence, the null hypothesis stating that bleaching would not deteriorate the marginal seal was rejected.

Similar degradation of the marginal seal due to bleaching was reported for the etch-and-rinse system Single Bond,[8] at the enamel margin, and for Prisma Universal Bond III[7] and the self-etch adhesive Prompt L-Pop,[9] at the dentin margin. On the other hand, no adverse effect of bleaching on marginal seal was reported for the etch-and-rinse adhesives Scotchbond and Single Bond lines[4,5,9,10] and self-etch adhesive iBond.[9]

The higher microleakage at the enamel margin than at the dentin margin found for ADP and IBO (Table 2) also differs from the results of the majority of the other related studies, which usually report a lower resistance at the dentin margin.[20] The factors that may affect microleakage are the location, shape, and volume of the cavity, which control the polymerization shrinkage stress and its distribution at

the cavity-restoration interface.[11] In our experiment, standardized cylindrical Class V cavities were prepared with a cavosurface angle of 90° without enamel beveling, which improves the marginal seal[24] by increasing the enamel-restoration bonded area in a more favorable orientation, perpendicular to enamel prisms.[25] Therefore, the results of our study cannot be compared with the results in which cavities with enamel beveling were prepared. [7,9] Other relevant factors include the type and coarseness of preparation burs, which affect the properties of the dentin and enamel smear layers.[26-28] The durability of the bond between the composite restoration and tooth tissues requires optimal demineralization and infiltration of the enamel and dentin by the adhesive components. These factors may be especially significant for mild self-etch adhesives, which possess a lesser ability to deeply demineralize the enamel smear layer than do the strong self-etch or etch-and-rinse systems.[28] Mechanical strength of polymerized adhesives can also play a significant role in the adhesive's performance.[29] This strength may be deteriorated by water residues in the adhesive, which decrease its degree of polymerization, the presence of residual low-molecular substances acting as plasticizers, or the occurrence of structural defects in the adhesive layer. For example, bubbles and voids of various origins can act as stress concentrators, [30,31] initiating failure of the adhesive interface. As depicted in Figure 1, the demineralization and penetration abilities of the tested adhesives differed significantly. A distinct prismatic structure, as well as the presence of long resin tags in enamel and dentin interfaces, signify the optimal demineralization and deep penetration of GLU-bonding monomers into the etched tooth structures. Therefore, we can assume that the strong demineralization capacity of the phosphoric acid etchant of GLU, along with the deep penetration of the adhesive into the microporosities in enamel and dentin, produce a good marginal seal and confer stability in both water and peroxide bleaching gel. A good marginal seal created with CLF can be attributed not only to its stronger demineralization abilities but also to its unique formation of a hydrolytically stable chemical bond between the 10-methacryloyloxydecyl dihydrogen phosphate monomer and the calcium of tooth tissue hydroxyapatite.[32-34] However, increased microleakage at the enamel margin after bleaching might indicate a potential degradation of the interface under a combined effect of shrinkage stresses and a peroxide bleaching gel. The lower resistance of the enamel margin sealed with ADP is difficult to explain because the opposite behavior should be expected as a result of ADP's more pronounced demineralization and penetration properties (compared with those of CLF). Analysis of the morphology of composite and enamel surfaces after the microleakage test revealed the complicated failure behavior of this adhesive interface under shrinkage stress in both environments. Remnants of the plastically deformed film of ADP, protruding from the prismatic enamel surface (Figure 2b) and in some areas disrupted by the voids (Figure 2c), indicated lower mechanical resistance of the ADP layer. It might be caused not only by structural defects in the adhesive layer but also by inappropriate polymerization resulting from the presence of water residues in the adhesive[35] or incompatibility of ADP acidic components with basic amines of the composite initiation system.[36] If the shrinkage stress of the composite material develops faster and exceeds the mechanical strength of ADP or its bond to the tooth tissues, a marginal failure can occur before the interface is degraded by the bleaching gel. On the other hand, IBO possesses a weaker organic acid in its formulation as a self-etch primer (Table 1), and it has a limited ability to create microporosities in enamel (Figure 1d), resulting in a low resistance of the enamel margin to the shrinkage stress.

Class V restorations at the cemento-enamel junction are produced in a strongly anisotropic substrate. One part of the restoration is bonded to enamel with a high elastic modulus, while the other part is bonded to dentin with a substantially lower elastic modulus. In addition, the bond strength between the substrates and the composite restoration can differ significantly, leading to heterogeneous stress distribution at the tooth-restoration interface and a difference in its stress resistance. If the weakest link between the restoration and tooth tissues breaks, it can be expected that the configuration factor of the restoration and shrinkage stresses acting in the opposite margins will decrease. Thus, if the enamel margin fails as in restorations made with ADP and IBO, stresses at the dentin margin should decrease accordingly. At a lower shrinkage stress, degradation of the dentin interface might thus be slower. With regard to this hypothesis, our results cannot fully exclude the possibility of a degradation of the ADP and IBO interfaces by the bleaching gel found by the bond strength measurement.[3]

Although originally developed to evaluate the sealing ability of nonadhesive restorations, such as amalgam fillings, microleakage tests on Class V

cavities are often used for testing adhesive restorations in which the mechanical properties of substrates and their interaction with an adhesive play key roles. To eliminate the effect of heterogeneous stress distribution, Class V cavities for microleakage tests should be prepared with margins in dentin or in enamel. If modeling a clinical situation, the cavity margins should be beveled, as recommended by standard preparation procedures, and the results should be analyzed with respect to the fact that the durability of cavity margins may depend not only on the adhesive bond strength but also on other factors specific to the test set-up. Thus, the obtained results may differ from the results of other *in vitro* tests.

CONCLUSIONS

Within the limitations of this *in vitro* study, it can be concluded that a marginal seal resistant to postoperative bleaching can be created with etch-and-rinse adhesive GLU. On the other hand, a small but significant increase in the microleakage of two-step self-etch adhesive CLF at the enamel margin might indicate its susceptibility to degradation during peroxide bleaching. Compromised bonding performance of one-step self-etch adhesives ADP and IBO could mask their degradation in peroxide bleaching gel and could be the reason for their apparent resistance in the bleaching gel environment.

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Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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The Evaluation of Working Casts Prepared from Digital Impressions

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Clinical Relevance

In general, the iTero scanner system displayed excellent extraoral performance. However, the polyurethane casts of the iTero system showed relatively low reproducibility, which indicated that the system should be used with caution for working cast fabrication.

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SUMMARY

Objective: The aim of this study is to evaluate the reproducibility of working casts of a digital impression system by comparing them with the original, virtual, and rapid prototyping casts.

Materials and Methods: A total of 54 cast sets in clinically stable occlusion were used. They were scanned by an iTero intraoral scanner and converted into STL format virtual casts. Rapid prototyping casts and polyurethane casts were fabricated from the iTero milling system based on the virtual casts. Several horizontal and vertical measurements were performed from the four types of casts, that is, original stone casts, virtual casts, rapid prototyping casts, and polyurethane casts of iTero. Measurement error, intraclass correlation coefficient (ICC), and differences among the casts were calculated and compared.

Results: Casts from iTero milling machines exhibited greater dimensional differences and lower ICC values than did other casts. In addition, many of the measurements of the iTero working casts showed statistically sig-

nificant differences in comparison to the three other types of casts. In contrast, there were no statistically significant differences between the virtual and original casts.

Conclusion: Virtual casts made by the iTero intraoral scanner exhibited excellent reproducibility. However, the casts from the iTero milling machine showed greater dimensional differences and lower reproducibility compared to other types of casts.

INTRODUCTION

More than 20 years have passed since digital dentistry emerged as a computer-assisted design/computer-assisted manufacturing (CAD/CAM) system for the fabrication of indirect dental restorations.[1,2] At the initial launch, the restorations created by the system were rather rudimentary from the contemporary perspective. However, with the advancement of digital technology, the field of digital dentistry continues to expand.[3]

Digital dentistry in restorative fields includes primarily two types of equipment, acquisition media and manufacturing media.[3] Presently, several systems are equipped with chair-side intraoral scanner units to function as acquisition media during in-office restorative procedures,[4] while many CAD/CAM systems are still limited to the realm of dental technicians and bench-tops.[5] The digital impression technologies of in-office systems have their own unique working principles, light sources, and imaging types. Some systems require a coating powder over the scanning area.[6]

Among the imaging systems, the iTero system (Cadent, San Jose, CA, USA) is based on a parallel confocal imaging principle. For this imaging technique, parallel beams of light are sent through a small hole before contacting the object that is to be scanned. The beams hit the object at the perfect focal length, bounce off of the object, and return through a small hole. Ultimately, these beams of light hit a sensor and are converted to a digital image.[7] This system can capture 100,000 beams of parallel red laser light at 300 focal depths that are spaced approximately 50 μm apart. This spacing allows for an approximate scanning depth between 13 and 15 mm. In total, the system captures approximately 3.5 million data points for each scanned arch.[3] In addition, the acquired image data can be saved as unencrypted STL (Stereo Lithography interface) files that can be used with many other CAD/CAM systems, in contrast to other in-office systems that use proprietary encrypted files for specific platforms.[4]

Another noted feature of this system is the lack of a requirement for a dedicated coating before scanning, which is a clinical advantage. In fact, the iTero system can be used solely as a digital impression system for traditional crown and bridge dentistry. After confirming the scanned virtual cast, the file is sent to a center for cast fabrication. This system uses a subtractive technique for the fabrication of the cast and die, which utilizes a computer numerical-controlled five-axis milling system.

To achieve an accurately fitting prosthesis, precision must be maintained in every step involved in conventional casting: impression, die, wax pattern, investment, and casting.[8] Although CAD/CAM systems do not require all of these steps, obtaining an accurate impression is the critical part of prosthesis fabrication. Whether it is digital or conventional, the overall goal of a dental impression is to produce an exact negative three-dimensional (3D) replica of the hard and soft tissues of the oral cavity.[9] In addition to the impression, accurate working casts are also essential for precisely fitting restorations. The reproducibility of die and casts from the prepared and antagonist sides is one of the decisive factors of not only the fit of definitive restorations, but also for chair-side intervention time.

While digital impressions have recently become more widely used, the accuracy of working models made from digital impressions has not been thoroughly evaluated. Therefore, the aim of this study is to evaluate the accuracy of working models of the iTero system by comparing them with the original, virtual, and rapid prototyping (RP) models.

MATERIALS AND METHODS

Type IV gypsum (Neoplumstone, Mutsumi Chemical Industry Co Ltd, Yokkaich, Japan) cast sets were prepared following alginate impressions taken by 100 first-year students at the School of Dentistry, Seoul National University, Korea (Figure 1a). Casts with missing teeth other than the third molars or those having more than 3 mm of crowding or arch length discrepancies were excluded from the study. In addition, casts with any kind of restorations or carious defects in the posterior segment, which includes the second premolar, first molar, and second molar, on either the preparation or antagonist side were also excluded. After this selection occurred, a total of 54 cast sets judged to be in clinically stable occlusions were used in the present

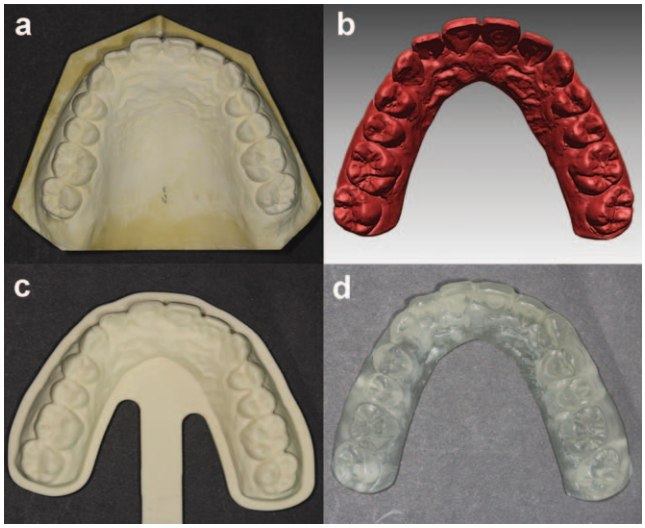

Figure 1. *Comparison of four types of casts: (A) original stone casts; (B) virtual casts in the form of a STL file; (C) polyurethane casts created using the iTero system; and (D) casts prepared by rapid prototyping.*

study. The ages of the participants ranged from 23 to 26 years, with a mean age of 24.3 years. All of the subjects provided written informed consent. This study was approved by the institutional review boards of the College of Dentistry at Seoul National University.

All of the casts selected were scanned by an iTero intraoral scanner. To make the diagnostic model, the digital image capture mode was selected to "Reference model" type to acquire the image of the full arch without the selection of any abutment. Each individual tooth was then scanned on both the buccal and lingual aspects. The anterior, right, and left posterior bites were captured to determine the occlusal relationship. Any undercut area of the cast that failed to produce a clear image was filled using the "Add scans" tool. After the scan process was completed, the captured image was sent to the iTero center for image adjustment and conversion to a STL file (Figure 1b). On the basis of STL data, a polyurethane block was milled by a five-axis milling machine (VF-2TR, Haas Automation Inc, Oxnard, CA, USA). The axes federate, repeatability accuracy, and positioning accuracy of the milling machine were 1000 ipm (25.4/min) on the X, Y, and Z axes; 0.00001 inch; and 0.00002 inch, respectively (Figure 1c).

The STL file was also imported into a prototyping software (Clinet Software) and submitted to a RP machine (Project HD 3000, 3D Systems, Rock Hill, SC, USA) for the fabrication of the actual sized model. The accuracy of a RP machine ranges between ±0.001 and 0.002 inches (0.025~0.05 mm) per inch. This system works with the 3D System's patented Multi-Jet Modeling (MJM) technology and uses two materials (part and support material). A transparent acrylic photopolymer (VisiJet® EX200 Plastic, 3D Systems) was developed specifically as the build material, and a white, nontoxic wax material (VisiJet® S100 Wax, 3D Systems) was used for hands-free melt-away support. The 3D model was built by overlaying in HD mode of resolution of a 32 μm layer of resin polymerized with ultraviolet light curing. The completed RP cast was placed into an oven to burn out the support wax at a temperature of 60°C (Figure 1d).

Horizontal and vertical measurements were performed to evaluate the reproducibility of the working casts. For the horizontal parameters, measurements were made between the distal incisal tips of the bilateral central, lateral incisors, and bilateral canine tips, and the bilateral buccal cusp tips of the premolars and bilateral mesiobuccal cusp tips of the molars (Figure 2a). For the vertical parameters, the distances between the most cervical points and cusp tips of the canine and buccal cusp tips of the second premolars were measured (Figure 2b). The virtual 3D casts were measured and analyzed using specialized software (Rapidform 2004, ver PP2, INUS Technology, Seoul, Korea), and the original casts and two kinds of working casts were measured using digital point calipers with a resolution of 0.01 mm (Mitutoyo, Kawasaki, Japan). Measurements of each cast were made three separate times by a single observer over a three-week period. To test the reliability, 10 virtual casts and 10 actual casts were randomly selected and measured again one month after the initial measurement.

The technical errors of measurement using the original Dalberg formula and intraclass correlation coefficients (ICCs) were calculated among the measurements using language R. With the measurements from original cast sets as a reference, the

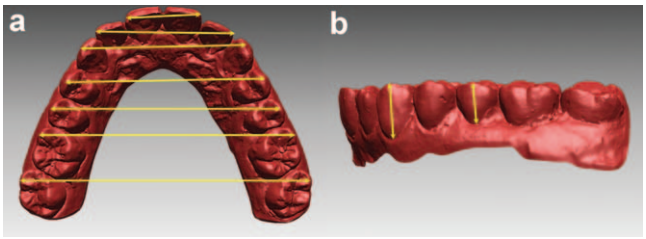

Figure 2. *Examples of measured parameters. (A) Horizontal parameters and (B) vertical parameters.*

differences and percentage differences of measurements from the other three casts were calculated. SPSS software (SPSS for Windows, version 12.0, Chicago, IL, USA) was used to perform paired-sample *t*-tests to determine the significant differences of the measured parameters among the casts. Significance was predetermined at the 0.05 and 0.01 levels of confidence.

RESULTS

Intraexaminer reliability coefficients ranged from 0.994 to 0.999 for virtual measurements and from 0.989 to 0.997 for real measurements. In terms of root mean square values, the random errors of estimation were less than 0.03 mm for virtual measurements and 0.07 mm for real measurements. None of the variables were significantly different between test and retest measurements.

Errors of measurements and the ICCs among the measurements from the four types of cast are summarized in Table 1. In general, casts from iTero milling machines exhibited greater root mean square errors and lower ICC values than other casts. The comparison of iTero casts with the other three types of casts showed errors in terms of the Dalberg formula ranging from 0.033 mm to 0.261 mm, with ICC values ranging from 0.912 to 0.999. In contrast, the comparison among the other three casts exhibited error that ranged from 0.002 mm to 0.040 mm, with ICC values ranging from 0.997 to 1.000. Most of

Table 1: *Errors of Measurement Calculated Using Dahlberg Formula and Intraclass Correlation Coefficients (ICCs)*

	PU vs RP		PU vs OC		PU vs VC		RP vs OC		RP vs VM		OC vs VM	
	rmse, mm	ICC	rmse, mm	ICC	rmse, mm	ICC	rmse, mm	ICC	rmse, mm	ICC	rmse, mm	ICC
Horizontal distance measurements between maxillary teeth												
11 to 21	0.149**	0.954	0.146**	0.957	0.146**	0.956	0.008*	1.000	0.008*	1.000	0.004	1.000
12 to 22	0.201**	0.980	0.203**	0.98	0.203**	0.98	0.007	1.000	0.006	1.000	0.005	1.000
13 to 23	0.169**	0.984	0.169**	0.984	0.170**	0.984	0.004	1.000	0.004	1.000	0.003	1.000
14 to 24	0.169	0.992	0.168	0.992	0.168	0.992	0.009	1.000	0.009	1.000	0.003	1.000
15 to 25	0.257	0.985	0.260	0.985	0.261	0.985	0.007	1.000	0.007	1.000	0.004	1.000
16 to 26	0.182**	0.998	0.182**	0.998	0.184**	0.998	0.006	1.000	0.007	1.000	0.003	1.000
17 to 27	0.200**	0.998	0.198**	0.998	0.198**	0.998	0.007	1.000	0.007	1.000	0.003	1.000
Subtotal average	0.190	0.984	0.214	0.985	0.215	0.958	0.007	1.000	0.007	1.000	0.004	1.000
Horizontal distance measurements between mandibular teeth												
31 to 41	0.116**	0.912	0.115**	0.912	0.115**	0.912	0.008	1.000	0.008	1.000	0.002	1.000
32 to 42	0.152**	0.974	0.154**	0.974	0.151**	0.975	0.007	1.000	0.008	1.000	0.010	1.000
33 to 43	0.129**	0.998	0.125**	0.998	0.225*	0.994	0.008*	1.000	0.008*	1.000	0.039	0.999
34 to 44	0.133*	0.998	0.135*	0.998	0.134*	0.998	0.004	1.000	0.004	1.000	0.003	1.000
35 to 45	0.119**	0.999	0.124**	0.999	0.126**	0.999	0.007*	1.000	0.008*	1.000	0.003	1.000
36 to 46	0.191**	0.998	0.188**	0.998	0.188**	0.998	0.005*	1.000	0.006*	1.000	0.002	1.000
37 to 47	0.188**	0.998	0.186**	0.998	0.205**	0.998	0.005	1.000	0.005	1.000	0.040	1.000
Subtotal average	0.147	0.982	0.147	0.982	0.163	0.982	0.006	1.000	0.007	1.000	0.014	1.000
Vertical distance measurements in teeth from most cervical points to (buccal) cusp tips												
13	0.116	0.985	0.122	0.984	0.116	0.985	0.027	0.999	0.027	0.999	0.023	0.999
15	0.158	0.975	0.161	0.974	0.161	0.974	0.008	1.000	0.009	1.000	0.006	1.000
23	0.043	0.997	0.044	0.997	0.045	0.997	0.008	1.000	0.009	1.000	0.004	1.000
25	0.169	0.959	0.173	0.958	0.170	0.959	0.008*	1.000	0.008*	1.000	0.006	1.000
33	0.051**	0.998	0.055**	0.998	0.056**	0.997	0.008*	1.000	0.008*	1.000	0.003	1.000
35	0.036**	0.999	0.033**	0.999	0.034**	0.999	0.006	1.000	0.008	1.000	0.004	1.000
43	0.076**	0.996	0.074**	0.996	0.072**	0.996	0.007	1.000	0.007	1.000	0.007	1.000
45	0.034**	0.998	0.078*	0.992	0.079*	0.992	0.031	0.998	0.035	0.997	0.009	1.000
Subtotal average	0.085	0.988	0.093	0.987	0.092	0.987	0.013	1.000	0.014	1.000	0.008	1.000
Total average	0.138	0.985	0.141	0.985	0.146	0.985	0.009	1.000	0.009	1.000	0.008	1.000

Abbreviations: OC, original stone casts; PU, polyurethane casts prepared by iTero™ system; rmse, root mean square of errors; RP, casts prepared by rapid prototyping; VC, virtual casts in form of STL file.
** Denotes statistically significant difference ($p<0.05$); ** Denotes statistically significant difference ($p<0.01$).*

the measurements of the iTero casts showed statistically significant differences in comparison to the other three types of casts. The RP casts showed statistically significant differences in comparison to the virtual and original casts in several measurements only at the 0.05 significance level. In comparison to the virtual and original casts, there were no statistically significant differences.

With the measurements from the original casts as references, the differences and percentage differences of measurements from the other three casts are summarized in Table 2. The iTero casts exhibited greater absolute differences from the original casts than did the other two casts. The average difference from the original casts to the iTero casts ranged from 0.033 to 0.250 mm, and the average percent difference ranged from 0.240% to 1.344%, whereas the differences between the other two casts ranged from 0.002 to 0.016 mm, and percent difference ranged from 0.003% to 0.179%.

DISCUSSION

The importance of reproducibility of impression material and working casts has not been diminished even though the recent introduction of digital dentistry has started a new era of powder-free dental clinics by changing the method of impression. In contrast to other systems, the iTero system can be used only as a digital impression tool for the fabrication of working casts for conventional laboratory work. This use meets the need of conventional fabrication when the milled prosthesis is not prop-

Table 2: *Average Absolute Difference (Standard Deviations) from Original Casts (as References)*

	VC		PU		RP	
	Absolute Difference, mm	**% Absolute Difference**	**Absolute Difference, mm**	**% Absolute Difference**	**Absolute Difference, mm**	**% Absolute Difference**
Horizontal distance measurements between maxillary teeth						
11 to 21	0.003 (0.005)	0.021 (0.019)	0.171 (0.061)	0.994 (0.404)	0.009 (0.007)	0.053 (0.024)
12 to 22	0.005 (0.006)	0.017 (0.011)	0.224 (0.110)	0.756 (0.333)	0.005 (0.003)	0.017 (0.012)
13 to 23	0.004 (0.002)	0.010 (0.006)	0.205 (0.069)	0.576 (0.279)	0.004 (0.003)	0.011 (0.007)
14 to 24	0.004 (0.003)	0.008 (0.007)	0.189 (0.082)	0.422 (0.180)	0.008 (0.006)	0.018 (0.024)
15 to 25	0.003 (0.004)	0.006 (0.005)	0.122 (0.076)	0.240 (0.084)	0.006 (0.005)	0.011 (0.007)
16 to 26	0.004 (0.003)	0.007 (0.003)	0.227 (0.067)	0.407 (0.139)	0.007 (0.004)	0.012 (0.008)
17 to 27	0.003 (0.004)	0.005 (0.003)	0.248 (0.098)	0.395 (0.121)	0.006 (0.004)	0.010 (0.006)
Subtotal average	0.004 (0.004)	0.011 (0.008)	0.198 (0.081)	0.541 (0.221)	0.006 (0.004)	0.019 (0.012)
Horizontal distance measurements between mandibular teeth						
31 to 41	0.002 (0.002)	0.014 (0.008)	0.139 (0.025)	1.274 (0.427)	0.008 (0.005)	0.074 (0.034)
32 to 42	0.007 (0.004)	0.031 (0.026)	0.197 (0.047)	0.886 (0.234)	0.008 (0.004)	0.035 (0.024)
33 to 43	0.007 (0.005)	0.024 (0.018)	0.149 (0.051)	0.549 (0.197)	0.008 (0.008)	0.031 (0.018)
34 to 44	0.004 (0.003)	0.012 (0.005)	0.157 (0.066)	0.451 (0.170)	0.003 (0.005)	0.009 (0.008)
35 to 45	0.003 (0.003)	0.008 (0.004)	0.168 (0.034)	0.420 (0.074)	0.007 (0.004)	0.017 (0.011)
36 to 46	0.002 (0.002)	0.003 (0.003)	0.250 (0.076)	0.525 (0.121)	0.005 (0.004)	0.010 (0.007)
37 to 47	0.007 (0.006)	0.012 (0.009)	0.231 (0.084)	0.422 (0.127)	0.005 (0.005)	0.009 (0.006)
Subtotal average	0.005 (0.003)	0.015 (0.010)	0.184 (0.055)	0.647 (0.194)	0.006 (0.005)	0.024 (0.015)
Vertical distance measurements in teeth from most cervical points to (buccal) cusp tips						
13	0.002 (0.004)	0.021 (0.025)	0.050 (0.022)	0.395 (0.188)	0.016 (0.012)	0.172 (0.115)
15	0.006 (0.003)	0.061 (0.031)	0.130 (0.016)	1.344 (0.276)	0.009 (0.007)	0.089 (0.073)
23	0.004 (0.003)	0.044 (0.027)	0.060 (0.022)	0.677 (0.304)	0.009 (0.009)	0.093 (0.090)
25	0.008 (0.004)	0.079 (0.022)	0.033 (0.005)	0.342 (0.056)	0.008 (0.006)	0.083 (0.069)
33	0.002 (0.004)	0.023 (0.029)	0.071 (0.046)	0.726 (0.295)	0.009 (0.007)	0.090 (0.070)
35	0.004 (0.002)	0.047 (0.031)	0.042 (0.020)	0.580 (0.236)	0.008 (0.004)	0.113 (0.117)
43	0.007 (0.004)	0.072 (0.039)	0.082 (0.050)	0.834 (0.335)	0.008 (0.006)	0.084 (0.064)
45	0.010 (0.007)	0.131 (0.076)	0.082 (0.065)	1.100 (0.608)	0.014 (0.012)	0.179 (0.158)
Subtotal average	0.005 (0.004)	0.060 (0.035)	0.069 (0.031)	0.688 (0.287)	0.010 (0.007)	0.113 (0.095)
Total average	0.005 (0.004)	0.030 (0.019)	0.147 (0.055)	0.628 (0.235)	0.008 (0.006)	0.055 (0.042)

Abbreviations: PU, polyurethane casts prepared by iTero™ system; RP, casts prepared by rapid prototyping; VC, virtual casts in form of STL file.

erly created or preferred. In addition, the working cast provided by the system can be used during the adaptation of the prosthesis.

In this respect, the accuracy of working casts of the system has to be evaluated. In the present study, the comparison between original and virtual casts demonstrated the performance of the intraoral scanner, while the comparison between virtual and milled casts and between virtual and prototyped casts exhibited the reproducibility of the polyurethane milling system and RP machine.

According to the results of the present study, the iTero scanner system provided reproducible virtual casts. The errors and differences between the original casts were small, a result that coincides with those of previous studies.[10-13] In contrast, the reproducibility of polyurethane casts fabricated by the iTero milling system was not as good as that associated with virtual casts. This was verified by the fact that the casts fabricated by RP machines exhibited smaller differences and much greater ICCs than did polyurethane casts.

Like the conventional stone cast, two steps are required for cast fabrication: impressions and construction. Based on the results, the inaccuracy of casts from the iTero system seems to come from the cast construction phase. While there are specifications for impression materials and gypsum products for cast fabrications, there are no such specifications for digital impressions. Even though a direct comparison is impossible, the accuracy shown by the casts from the iTero system was not ideal when compared with results described in previous literature about conventional systems. For example, when comparing our results with data from irreversible hydrocolloid impression material, which is the most widely used material in dental clinics for antagonist impressions, the system showed no superiority. This inaccuracy not only caused misfits of the prosthesis but it also increased the required amount of chairside adjustment.

In contrast, the performance of the iTero intraoral scanner system seemed to be excellent within the limits of this study. The lack of a requirement for a dedicated spray coating should be stressed as a great advantage of the system. The use of opaque powder spray provides uniform light dispersion and enhances the accuracy. However, the coating seemed to affect the measurement and led to the increase of dimensions according to the times of coating in our unpublished pilot study using other 3D scanners, such as the CEREC Bluecam intraoral scanner (Sirona, Bensheim, Germany) and the extraoral scanner (optoTOP-HE, Breckmann GMBH, Meersburg, Germany). This result partly coincides with that of a previous study,[14] but there is also a report[15] that compared the impressions of powdered teeth vs nonpowdered stone casts and found that the marginal gap of onlay restorations was not different when the optical impression was taken intraorally vs extraorally without powdering. In clinics, spraying and removing excess powder with air causes the additional steps and associated time, and in some cases it is hard to apply as a result of a lack of ideal isolation or depth of the preparation. In addition, careless spraying can cause irritation to sensitive patients.[15]

Several issues related to this study should be pointed out. First, the virtual model fabricated by scan using the intraoral scanner was not in an intraoral environment. In addition, the scanned objects were not real teeth and gums but rather the stone cast. The intraoral environment contains saliva and humidity, and there are various kinds of hindrances associated with it, including less stable positioning of the scanner during the impression-taking process. In addition, human enamel has a different reflective and dispersive property compared to dental cast stone. Therefore, there is a possibility of compromising the accuracy of impression taking when using the intraoral scanner in a clinical situation. The accuracy of intraoral scanning should be tested intraorally in further studies. Second, the measurements were performed on the unprepared casts; the results can be interpreted to represent the accuracy of antagonizing casts. In order to suggest more specific clinical implications, study using casts with prepared dies of varying spans would be recommended.

From a practical point of view, impression-taking time should be considered. In the present study, a substantial amount of time was needed for the full arch digital impression taking using the system compared with the time needed for conventional impression taking. This additional time might be due to the limitations of the operating system or computer hardware and could be alleviated by rapid technological advances in the near future. However, this disadvantage can cancel the overall merit of this system for long-span prostheses, at least for the time being.

RP is a CAD/CAM technology that was originally developed to fabricate prototypes for industrial purposes. This method automatically constructs physical models from computerized 3D data. RP

has recently been successfully applied in various medical fields, such as in the fabrication of implant surgical guides[16,17] and maxillofacial prosthetics[18,19] and in frameworks for removable partial dentures.[20] Recently, the use of RP in the frameworks of fixed restorations was studied,[21] and the results of this study seemed to be promising, even though further improvements are needed.

CONCLUSIONS

The reproducibility of four types of casts was evaluated in this study. Virtual casts made by the iTero intraoral scanner showed excellent reproducibility in general. However, when comparing original stone casts, virtual casts, RP casts, and casts fabricated by the iTero milling machine, the casts from the iTero milling machine exhibited greater dimensional differences and lower reproducibility than did the other types of casts. The reproducibility must be improved, and the system should be used with caution for working cast fabrication. The RP casts showed some promise. The results of the present *in vitro* study did not come from clinical situations; therefore, there should be *in vivo* studies verifying the intraoral performance of the scanner system with prepared teeth.

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Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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Composite vs Ceramic Computer-aided Design/ Computer-assisted Manufacturing Crowns in Endodontically Treated Teeth: Analysis of Marginal Adaptation

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I Krejci

Clinical Relevance

In adhesive dentistry, the computer-aided design/computer-assisted manufacturing composite crown offers a superior option to the ceramic crown in the restoration of endodontically treated anterior teeth.

SUMMARY

Objectives: **To compare the marginal adaptation between ceramic and composite CEREC crowns in endodontically treated teeth restored with endocrowns or with a short or a long post.**

Methodology: **Forty-eight intact maxillary incisors were used. After endodontic treatment, the crowns were sectioned 2 mm coronally to the cemento-enamel junction, which provided a ferrule of 2 mm. The prepared teeth were divided randomly into six groups (n=8). Group 1 was restored with a large fiberglass post,**

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composite core, and ceramic full-coverage computer-aided design/computer-assisted manufacturing (CAD-CAM) crown. Group 2 was restored with a short fiberglass post, composite core, and ceramic full-coverage CAD-CAM crown. Group 3 was restored with a large fiberglass post, composite core, and composite full-coverage CAD-CAM crown (LPCpr). Group 4 was restored with a short fiberglass post, composite core, and composite full-coverage CAD-CAM crown (SPCpr). Groups 5 and 6 were restored with ceramic and composite CEREC machined endocrowns, respectively (EndoCer and EndoCpr). The restored teeth were loaded thermomechanically in a computer-controlled chewing machine. Impressions of each restoration were made in a polyvinylsiloxane material before and after loading. Gold-coated epoxy replicas were prepared for scanning electron microscopy examination at 200× magnification.

Results: **Loading had a statistically significant effect ($p<0.05$) on the percentage of "continuous margin" in all groups. The LPCpr, SPCpr, and EndoCpr groups showed the highest percentage of continuous margin initially and after loading. The effect of the different post lengths on marginal adaptation was not significant ($p>0.05$).**

Conclusion: **CAD-CAM crowns fabricated from millable composite resin blocks (Paradigm MZ100) offer a superior option to all-ceramic crowns (IPS Empress CAD).**

INTRODUCTION

Advances in adhesive dentistry and technologic developments with computer-aided design/computer-assisted manufacturing (CAD-CAM) technologies have resulted in new systems for dental restoration. Various machinable materials are used currently with CAD/CAM systems to fabricate restorations at the chairside. The CEREC 3 CAD/CAM system was introduced more than 15 years ago and it is the only system that can be used in both clinical practice and the laboratory.[1]

The successful restoration of endodontically treated teeth (ETT) does not depend exclusively on the endodontic treatment; the method of restoration is also important.[2,3] Coronal leakage at the margin of the restoration might result in recurrent caries and failure of both the restoration and the root canal treatment.[4]

For decades, metal-ceramic crowns have been the major type of restoration system used in fixed prosthodontics. Nowadays, all-ceramic anterior crown restorations may be used as an alternative to metal-ceramic crowns.[5,6] In spite of the advantages of all-ceramic restorations, including the esthetic appearance, biocompatibility, and durability they afford, such materials present some disadvantages.[7-9] However, considerable progress has been made in the manufacture of composite resins. Recently, a new resin composite block (Paradigm MZ100, 3M ESPE Dental Products, Seefeld Germany) has been introduced for the CEREC system, which, according to the manufacturer, combines some of the best attributes of ceramics and polymers.[10] Few studies have been performed to evaluate the survival and success rates of single-tooth all-composite resin and all-ceramic complete restorations manufactured with a CAD/CAM system. Rammelsberg and others[11] found that composite resin crowns showed an acceptable survival rate of 96% after three years. However, an excellent marginal adaptation is one of the most important factors for the longevity of esthetic crowns, and further research and evaluation of CEREC 3 composite resin crowns are necessary to improve the probability of clinical success.[12,13]

The quality of the marginal adaptation has been criticized by many researchers, but improvements in the CEREC machine and software have made the fit more acceptable. Numerous studies have evaluated the marginal accuracy of crown restorations and have described promising results. To date, ceramic-based materials have been used with all CAD/CAM systems for anterior teeth. However, there are no reported investigations that have examined the marginal adaptation of CEREC 3 anterior crowns in ETT fabricated from Paradigm Z100 (3M ESPE Dental Products).

The aim of the present study was to evaluate the quality of the marginal adaptation of crowns made out of composite and ceramics on devitalized anterior teeth before and after a thermomechanical fatigue test that simulated a clinical service of 2.5 years. Three types of restorative procedures for the root canal were tested: a 10-mm post, a 5-mm post, and an endocrown. The specimens were loaded at an inclination of 45° with respect to the longitudinal tooth axis. The null hypotheses tested were that 1) There is no effect of fatigue conditions on marginal adaptation; 2) There is no influence of the restorative crown material (ceramic or composite) on the

marginal adaptation at either interface, tooth–luting cement or luting cement–crown; and 3) There is no influence of post length on marginal adaptation.

MATERIALS AND METHODS

Forty-eight sound upper central human incisors that had been stored in 0.1% thymol solution were divided randomly into six equal groups. The bucco-palatal and mesio-distal dimensions and root lengths of all the teeth selected were measured using digital calipers. The inclusion criteria were that the teeth had to be free of carious lesions with complete apexification and straight roots, had to have a crown up to 2 mm above the cemento-enamel junction (CEJ), and had to have an absence of visible fracture lines in the root.

Endodontic Treatment

Before endodontic treatment of each specimen, the root surface was sealed using a filled light-curing adhesive (Lot No. 2957717, Optibond FL, Kerr-Hawe Neos, Orange, CA, USA). The pulp chamber of each tooth was opened following a standardized procedure and the working length was determined visually by placing a size No. 10 K-file (Dentsply-Maillefer, Ballaigues, Switzerland) at the apical foramen. The root canals were instrumented using stainless-steel K-files 10, 15, and 20 (Dentsply-Maillefer) followed by rotary nickel-titanium instruments (ProTaper U®, Dentsply-Maillefer), in accordance with the manufacturer's instructions. All of the canals were prepared up to an F5 size instrument, and instruments were discarded after use in four root canals or if instrument deformation was visible. The root canals were irrigated between instruments with 1 mL of 4.2% sodium hypochlorite. All of the teeth were obturated using the warm vertical condensation technique (System B and Gutapercha Extruder, Elements Obturation Unit™, Analytic Endodontics, Sybron Endo, Orange, CA, USA) using calibrated gutta-percha (Autofit®, Analytic Endodontics) and an endodontic sealer (AH Plus, Dentsply-Maillefer). Following this step, the access cavity was sealed with a light-cured, resin-reinforced glass ionomer restorative (Fuji II® LC, GC America Inc, Alsip, IL, USA). After a setting period of 48 hours, each tooth was fixed on a custom-made metallic holder (Provac FL, Balzers, Liechtenstein), and the root bases were stabilized with a self-curing acrylic resin (Technovit 4071, Heraeus Kulzer GmbH, Wehrheim, Germany).

Root Preparation, Post Selection, and Luting Procedure

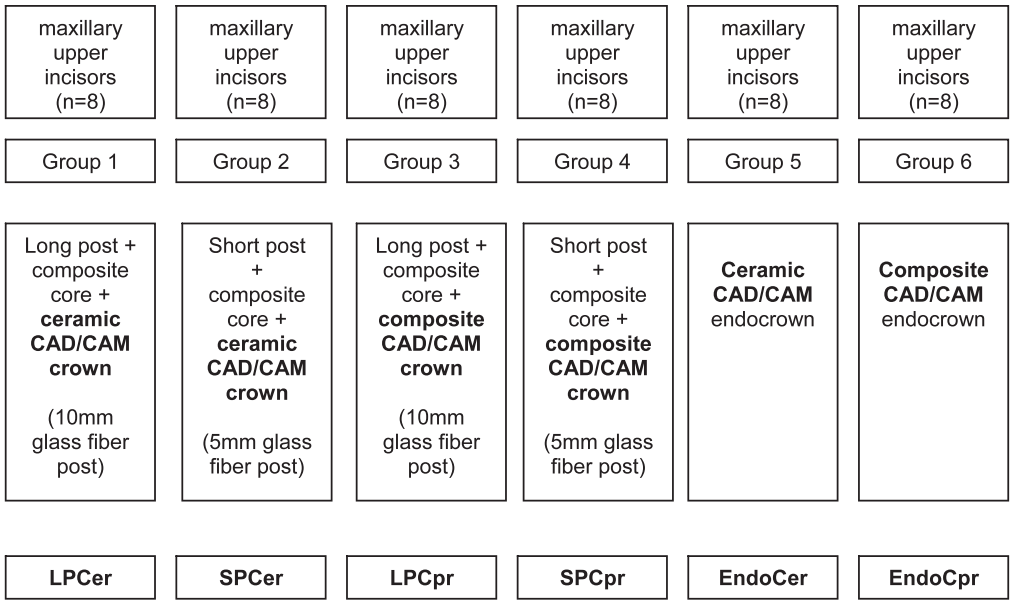
The crown of each tooth was sectioned 2 mm above the CEJ. The prepared teeth were divided randomly into six groups of eight teeth each, as follows: 1) long post, composite core, and ceramic crown (LPCer); 2) short post, composite core, and ceramic crown (SPCer); 3) long post, composite core, and composite crown (LPCpr); 4) short post, composite core, and composite crown (SPCpr); 5) ceramic endocrown (EndoCer); and 6) composite endocrown (EndoCpr) (Table 1).

Translucent glass-fiber posts of a standard size (Lot No. 35052, FRC Postec Plus, Size 3, Ivoclar Vivadent, Schaan, Liechtenstein) were selected for placement in each root canal. Gutta-percha was removed with a size 3 reamer (Ivoclar Vivadent) using a handpiece at 800-1220 rpm. The composition of the adhesive system and restorative materials are detailed in Table 2a and b.

Each post was inserted into the root canal and cut to an adequate length with a diamond rotary cutting instrument, and its incisal end was covered with at least 1 mm of resin composite. The surface of the glass-fiber post was pretreated with etching gel (K-Etchant Gel, Kuraray Europe GmbH) for 15 seconds, sand-blasted with 27-μm silicatized Al_2O_3 powder (CoeJet, 3MEspe, Seefeld, Germany), and silanized (Lot No. 2550, Clearfil Ceramic Primer, Kuraray Europe GmbH) for 60 seconds. The bonding system (Lot No. 41119, Clearfil DC Bond, Kuraray Europe GmbH) was applied to the post and dried with air, which was applied for five seconds using a dental syringe.

All materials used in the root canals were applied using microbrushes. The following bonding protocol was adopted, strictly following the manufacturer's instructions: 37% phosphoric acid (K-Etchant Gel, Kuraray Europe GmbH) was applied to the surfaces of the canal walls for 15 seconds, and the conditioned areas were rinsed thoroughly with water for at least 15 seconds. Water was removed from the rinsed canals with a soft blow of air and paper points. A moist surface was left, to avoid desiccating the dentin. The adhesive (Clearfil DC Bond, Kuraray Europe GmbH) was dispensed onto a disposable microbrush and rubbed immediately onto all root canal surfaces for at least 20 seconds. The solvent was removed by gentle blowing with air from a dental syringe for at least five seconds. The posts were then luted with a dual-cured resin cement (Clearfil Esthetic Cement, Kuraray Europe GmbH), in accordance with the manufacturer's instructions.

Table 1: *Scheme of the Study Design*



The luting cement was applied to the post and to the post space with a microbrush. The posts were seated into the root canals and stabilized, and the excess cement was cleaned up with paper points. The resin cement and the adhesive material were light-cured simultaneously for 60 seconds using a Demi LED Light Curing Unit (Kerr Corp, Middleton, WI, USA) applied in direct contact with the post. To ensure appropriate light intensity, the emitted light was measured before each exposure with the digital radiometer (Bluephase meter, Ivoclar Vivadent) Prior to our study the light intensity measured 1340 mW/cm^2.

Core Preparation

After the posts had been luted, the core was prepared using the same adhesive system (Clearfil DC Bond, Kuraray) and following the same application technique described above. The core was built up using a dual-cured core material (Clearfil Photo Core, Kuraray), which was light-cured for 40 seconds. Transparent matrices (Hawe Striproll, KerrHawe) were used to confine the restorative material. Preparation of the core was finished with diamond burs (Advanced Preparation Set for CEREC Anterior Restorations, Intensiv, Lugano, Switzerland). All crown margins were located in dentin with a ferrule effect of 2 mm. The anatomical shape was reduced with the following minimum requirement: The incisal width of the preparation measured at least 1 mm (based on milling tool geometry) in order to achieve optimal milling of the incisal edge during CAD/CAM processing.

Crown Design and Milling

In group 5 (EndoCer) and group 6 (EndoCpr), ceramic and composite endocrowns, respectively, were prepared with a CAD/CAM system (CEREC 3D, software V2.40 R1800, Sirona, Bensheim, Germany). After crown preparation, the surface was covered uniformly with an antireflecting powder (VITA CEREC Powder, Vita Zahnfabrik, Bad Säckingen, Germany), and a digital impression was obtained with the three-dimensional camera. The digital design and milling of the crowns was performed with the CEREC software. Composite and ceramic crowns were milled from prefabricated blocks (Paradigm MZ100, 3M ESPE, and IPS Empress CAD, Ivoclar Vivadent, respectively) with a cylindrical pointed bur and a step bur size 10. All restorations were milled in "Endo" mode, and a new set of milling burs was used for each group, even though this was not requested by the software manufacturer.

Tooth/Core Preparation for the Luting Procedure

The bonding agent (Clearfil DC Bond, Kuraray) was applied in accordance with the manufacturer's

Table 2: *Mode of Application, Composition, and Manufacturer of the Tested Materials*

Material	Product Name (Manufacturer)	Composition (Main Constituents)	Application Mode	Batch Numbers
a				
Fiber post	FRC Postec Plus (Ivoclar Vivadent, Schaan, Liechtenstein)	Glass fibers (70 vol%), dimethacrylate resin matrix (21 vol%), ytterbium fluoride (9 vol%)		35052
Ceramic blocks	IPS Empress CAD[14]	Components: SiO_2 Additional contents: Al_2O_3, K_2O, Na_2O, CaO, and other oxides, pigments		57343
Composite blocks	MZ100 (3M Espe, Germany)	Conventional hybrid composite resin, Bis-GMA, TEGDMA, and ultrafine zirconia silica ceramic particles as filler Particles have a spherical shape and average size of 0.6 mm		20071221
b				
Dual-cure, resin-based cement system	Clearfil Esthetic Cement (Kuraray)	Clearfil Ceramic Primer: 3-MPS; 10-MDP; ethanol	Apply primer to ceramic and air-dry. Mix equal quantities of pastes A and B. Apply and light-cure for 40 s	13ABA
		Paste A: Bisphenol A diglycidylmethacrylate; TEGDMA; methacrylate monomers; silanated glass filler; colloidal silica		
		Paste B: Bisphenol A diglycidylmethacrylate; TEGDMA; methacrylate monomers; silanated glass filler; silanated silica; colloidal silica; benzoyl peroxide; CQ: pigments		
Adhesive system	Clearfil DC Bond (Kuraray)	K-Etchant gel	Etch for 15 s; rinse with water spray and gently dry with air and paper points; mix liquids A and B (1:1); apply with a brush; gently air-dry for 2-3 s.	41119
		Liquid A: HEMA; MDP; Bis-GMA; DL-camphorquinone; benzoyl peroxide; colloidal silica		
		Liquid B: water; ethanol		
Build-up	Clearfil Photo Core (Kuraray)	Silanated silica, silanated barium, glass, CQ, bisphenol A diglycidylmethacrylate	Apply to the tooth; light-cure for 40 s	2295BA

Abbreviations: Bis-GMA, bisphenol-A-diglycidylether dimethacrylate; CQ, camphorquinone; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethyleneglycol-dimethacrylate; 10-MDP, 10-methacryloxydecyl dihydrogen phosphate; 3-MPS, 3-methacryloxypropyltrimethoxysilane.

instructions: the dentin was etched for 15 seconds with 37.5% phosphoric acid, rinsed abundantly, and air-dried for five seconds, and then the adhesive agent was applied with a light brushing motion for 20 seconds. The composite core was treated by airborne-particle abrasion with 27-μm silicatized Al_2O_3 powder (CoeJet, 3M ESPE). Subsequently, the surface was rinsed with water for 20 seconds and air-dried. Silane (Clearfil Ceramic Primer) was applied to the surface and air-dried after an

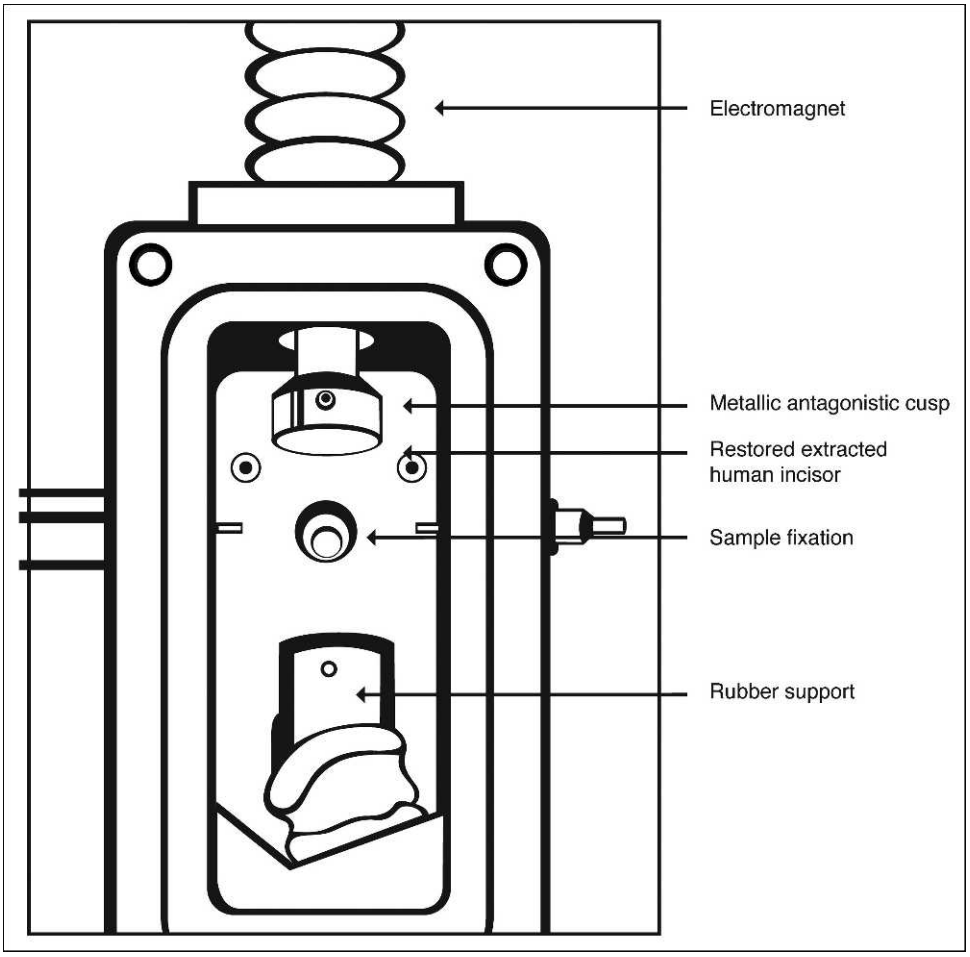


Figure 1. *Custom made device used for occlusal loading of the samples.*

exposure time of 60 seconds. One coat of adhesive resin (Clearfil DC Bond) was then applied to the surface and left unpolymerized until the application of the luting material.

Crown Preparation for the Luting Procedure

In the leucite-reinforced glass-ceramic groups (groups 1, 2, and 6), the internal surface of the crowns was etched with hydrofluoric acid (Vita Ceramic Etch, Vita Zahnfabrik) for 60 seconds. Following this step, silane (Clearfil Ceramic Primer, Kuraray) was applied and blown dry after an exposure time of 60 seconds. Finally, the bonding agent (Clearfil DC Bond) was applied and the excess was blown out. In the microhybrid composite groups (groups 3, 4, and 5), the internal surface of the crowns was treated with 27-μm silicatized Al_2O_3 powder (CoeJet, 3M ESPE). Subsequently, the surface was rinsed with water for 20 seconds and air-dried. A silane (Clearfil Ceramic Primer) was applied and blown dry after an exposure time of 60 seconds. Finally, the bonding agent (Clearfil DC Bond) was applied and the excess was blown out. The crowns from all groups were luted adhesively with a dual-cured luting cement (Clearfil Esthetic Cement, Kuraray) and cured with the same light-curing device mentioned above. Finally, all of the margins were finished and polished under 10× magnification using abrasive discs (Soft-Lex XT, 3M ESPE) and intermittent water spray.

Mechanical Loading, Marginal Adaptation, and Scanning Electron Microscopy (SEM) Evaluation of Samples

The restored teeth were loaded on the palatal surface at an angle of 45° with respect to the longitudinal axis of the root in a computer-controlled chewing machine and were subjected to 600,000 mechanical cycles at 49 N and 1500 thermal cycles in which the temperature varied between 5°C and 55°C (Figure 1). The position of the artificial cusps in the

test chambers of the mechanical fatigue device (Department of Restorative Dentistry & Endodontics and Laboratory of Electronics of the Medical Faculty, University of Geneva) was adjusted to maintain a distance of 1 mm from the top of the core, allowing free initial movement. The artificial cusps that contacted the samples were made of stainless steel, the hardness of which is similar to that of natural enamel (Vickers hardness: enamel=320-325; Actinit stainless steel=315).

Before and after the stress was applied, gold-sputtered epoxy resin replicas of all the samples were fabricated using polyvinylsiloxane impressions (President light body, Coltène-Whaledent, Altstätten, Switzerland). The replicas were used for a semiquantitative analysis of the external adhesive interfaces by SEM (Philips XL 20, Eindhoven, The Netherlands), which was performed at a standard 200× magnification using a custom-made module programmed within the image processing software. Two evaluation parameters were considered, "continuity" (C) and "marginal opening" (MO), in order to enable the quantitative evaluation of marginal adaptation to characterize each portion of the interface.

Statistical Analysis

Data analysis was performed using specific software (Statgraphics 5.0 Plus). The values for marginal adaptation (%) at the interface between the tooth and the luting cement (TC-interface) and between the luting cement and the crown (CC-interface) were introduced as the first dependent variables. The following parameters were introduced as independent variables: testing interval (before loading and after loading), type of material (ceramic or composite), type of restoration (long post, short post, or endocrown), and type of interface (tooth–luting cement or luting cement–crown) (Figure 2).

Multifactorial analysis of variance (ANOVA) and *post hoc* Tukey tests were performed to assess the effect of four independent variables on marginal adaptation for each tested interface after confirming normal distribution with the Leven test ($p<0.05$) and the homogeneity of variance with the Shapiro-Wilks test ($p>0.05$). The level of confidence was set to 95%.

RESULTS

All of the teeth and restorations survived thermomechanical loading in the computer-controlled chewing machine without loss of retention or fracture and could be used for the quantitative analysis of marginal adaptation.

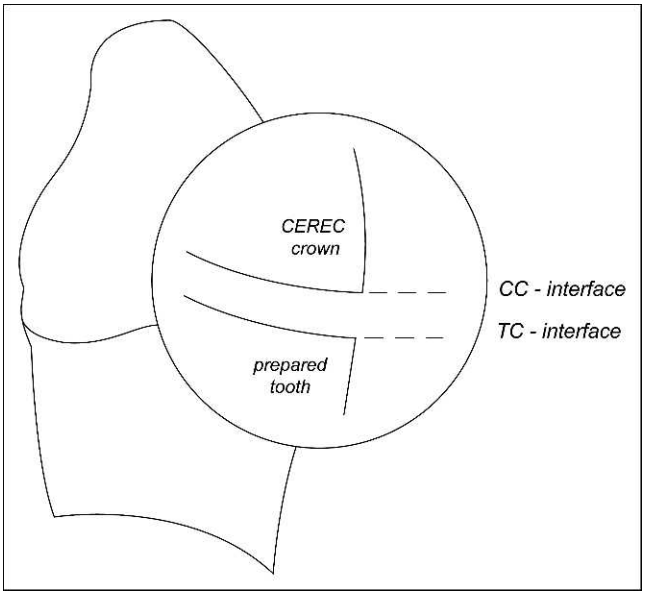


Figure 2. *Tooth–luting cement interface (TC) and luting cement–crown interface (CC).*

The results for marginal adaptation at the tooth–luting cement interface are shown in Table 3. Multifactorial ANOVA revealed a significant effect of testing interval ($p<0.05$) and of the type of material ($p<0.05$) on marginal adaptation.

Before loading, the percentages of continuous margin at the tooth–luting cement interface were greater than 90%, and no significant differences were observed among the different groups ($p=0.062$). However, a trend was observed for better behavior of the composite in comparison with the ceramic material.

After loading, statistically significant differences were detected between the composite (LPCpr, SPCpr, and EndoCpr) and ceramic (LPCer, SpCer, and EndoCer) crowns ($p=0.0001$). The highest scores for marginal adaptation were observed in the LPCpr, SPCpr, and EndoCpr groups, namely, composite crowns restored with long posts, short posts, or endocrowns. The performance of the ceramic crowns at the marginal level was significantly lower, independent of the type of root retention that was used.

The results for marginal adaptation at the luting cement–crown interface are also shown in Table 3. No significant differences among groups were detected either before or after loading ($p=0.9834$). However, the groups LPCpr, SPCpr, and EndoCpr (with composite restorations) showed the highest percentages of continuous margin after loading.

Table 3: *Percentages of Continuous Margins (%CM) at Both Interfaces Before and After Loading for the Different Groups. Small Capital Letters Indicate Statistically Significant Differences Between Materials ($p \leq 0.05$)*

Groups	Tooth–Luting Cement Interface %CM, Mean (SD)		Luting Cement–Crown Interface %CM, Mean (SD)	
	Before Loading	After Loading	Before Loading	After Loading
LPCpr	99.3 (0.85) A	91.3 (6.75) A	98.7 (2.4) A	97.8 (2.63) A
SPCpr	99.2 (0.97) A	85.5 (6.47) A	99.5 (0.62) A	97.7 (1.24) A
EndoCpr	94.4 (6.13) A	80.9 (8.14) A	100 (0.07) A	99.9 (0.00) A
LPCer	94.3 (6.52) A	65.9 (14.18) B	92.9 (7.1) A	95.2 (3.54) A
SPCer	90.2 (12.2) A	57. 7 (18.2) B	89.1 (5.17) A	84.6 (10.18) A
EndoCer	93.9 (5.00) A	68.4 (23.6) B	94.8 (6.47) A	90.1 (4.57) A

Abbreviations: EndoCer, ceramic endocrown; EndoCpr, composite endocrown; LPCer, long post, composite core, and ceramic crown; LPCpr, long post, composite core, and composite crown; SD, standard deviation; SPCer, short post, composite core, and ceramic crown; SPCpr, short post, composite core, and composite crown.

The effect of the different post lengths on marginal adaptation was not significant ($p=0.549$). Thus, the percentages of marginal adaptation were similar in groups restored with long posts, short posts, and endocrowns.

SEM micrographs that are representative of the different groups are shown in Figure 3. The main difference between the ceramic and composite crowns was observed at the tooth–luting cement interface. Dentin cracks could be observed on loaded specimens that had been restored with ceramic crowns, whereas no cracks were evident in the dentin when composite crowns were used as the restorative material (Figure 3).

DISCUSSION

In the *in vitro* study described herein, we compared the marginal adaptation of natural anterior teeth that had been restored by endocrowns, short-post, and long-post retained CAD/CAM composite and ceramic crowns when they were loaded in a computer-controlled chewing machine and evaluated by SEM. Excellent marginal adaptation extends the longevity of restorations.[15,16] Lack of adequate fit is potentially detrimental to both the tooth and the supporting periodontal tissues, as a result of cement solubility or plaque retention.[17] The present study focused exclusively on the quality of marginal adaptation *in vitro* as an indispensable prerequisite for clinical success.[18] Within the limitations of laboratory studies, quantitative analysis of marginal

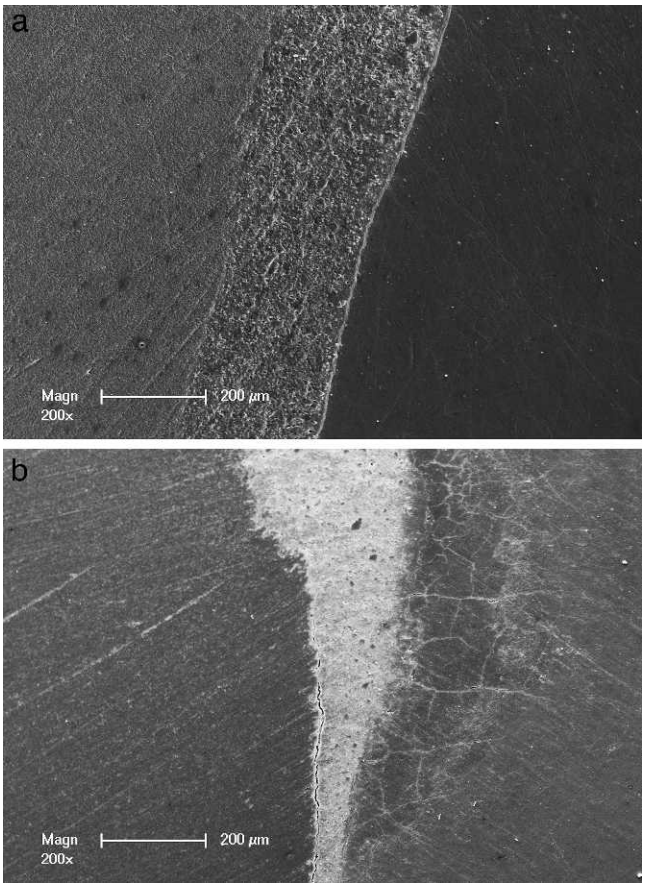


Figure 3. *SEM photographs (200×) of marginal interfaces of a CEREC anterior crown. Left: dentin; right: restoration. (a) Composite anterior endocrown (EndoCpr) after thermomechanical loading. (b) Ceramic anterior endocrown (EndoCer) after thermomechanical loading, with small cracks in the dentin.*

adaptation by SEM has proven to be an exact and reliable method of assessment for the evaluation of the marginal adaptation of adhesive restorations.[19,20] Materials and interfaces normally fail because of stresses and repeated loading. The quality of the SEM analysis is expressed as the percentage of "continuous margin" and "marginal opening" along the total marginal length at both the tooth–luting cement[15] and the luting cement–crown (CC) interfaces. To evaluate marginal adaptation, a replica-based, computer-assisted quantitative SEM analysis of the margin was performed before and after loading. The replica-based approach has several advantages: it is quantitative, nondestructive, and highly discriminatory.[21,22] Although there have been numerous investigations using light microscopy, most authors[23,24] have concluded that SEM imaging provides more appropriate and realistic observations than do light microscopy-based systems of analysis.

Thermomechanical loading was used in an attempt to simulate the oral environment. Stressing the restorations for 600,000 cycles *in vitro* simulates 2.5 years of clinical use. It can be assumed that the results of the present study have a degree of clinical relevance.[16,25,26]

The materials that were chosen for performance assessment, composite blocks and ceramic blocks, are both widely used restorative materials in modern conservative dentistry. In the present study, no artificial periodontium was placed around the abutment roots; the silicone cannot be standardized and varies between 300 and 700 μm, which leads to uncontrolled, and unstandardized, mobility of the abutment teeth. In the clinical situation, increased mobility only occurs in teeth that have severely compromised periodontium, with a loss of attachment of 6 mm or more.[27]

As shown by the SEM analysis of the margin, dentinal adhesion was very successful before loading: the percentages of continuous margins before loading were high for most groups. One explanation could be that all systems that involve etch-and-rinse adhesives combined with conventional luting resin composites result in a very good bond.[28] However, the degree of adhesion changed considerably after loading as a result of marginal degradation at the tooth–luting cement interface. Given that significant differences in marginal adaptation were identified among the groups, the first null hypothesis tested in the study was rejected; marginal adaptation was affected by fatigue conditions. This confirms the results of previous studies[28,29] in which thermomechanical loading resulted in a deterioration of marginal quality.

In the present study there was also a significant influence of the material of the restorative crown (ceramic or composite) on the marginal adaptation of both interfaces, tooth–luting cement[15] and luting cement–crown (LC). Groups LPCpr, SPCpr, and EndoCpr showed the highest percentage of continuous margin after loading. The rigidity of dental restorative materials is considered to be a very important issue when evaluating the adhesive tooth-restoration interface. Composite materials are more resilient than ceramics, and this could have an effect on the stress that is transferred to the margin walls. On the basis of these observations, we also had to reject the second null hypothesis. Even if there is a lack of scientific evidence that correlates dentin cracks with the long-term clinical behavior of ceramic restorations, cracks may be interpreted as a sign of early failure. According to the manufacturer, the IPS Empress CAD block is a conventional feldspathic ceramic, whereas the MZ100 block is a millable composite resin formed of 85% (by weight) ultrafine zirconium-silica ceramic particles that reinforce a highly cross-linked polymeric matrix. The polymeric matrix consists of bisphenol-A- diglycidylether dimethacrylate and triethylene glycol dimethacrylate. Different inherent mechanical properties of the two esthetic materials (ceramic and composite) used for crown fabrication, such as stiffness and flexural strength, might also have influenced the marginal adaptation after thermomechanical loading. The manufacturers report that the modulus of elasticity is approximately 65.4 GPa for the IPS Empress CAD and 30 GPa for MZ100 blocks, whereas the flexural strength is purported to range from 120 to 140 N/mm^2 for the IPS Empress CAD and is reported to be 150 MPa for MZ100 blocks.

Paradigm MZ100 could represent a departure from the more popular ceramic materials. Composites can be more easily adjusted and polished intraorally than can ceramic materials. The repair of ceramic restorations intraorally has not proven to be more than a moderately effective temporary technique. With Paradigm MZ100, the restoration surface can be air-abraded and a hybrid composite can be bonded to the abraded surface. Although it has not been tested for clinical longevity, this affords an easy and efficient intraoral repair procedure for Paradigm MZ100 restorations.

The definition of marginal fit varies considerably among investigators, and often the same term is used to refer to different measurements. In a recent

study, Tsitrou and others[30] showed that the marginal gap of resin composite crowns manufactured with the CEREC 3 system is within the range of clinical acceptance. In a study of posterior teeth, Krejci and others[31] showed an excellent marginal adaptation for adhesive composite restorations. The composite resin crowns might demonstrate higher resiliency with more absorption of load than ceramic crowns; these results are in agreement with the findings of other investigators.[9,32-35] Our results are supported by similar *in vitro* findings. In a recent article,[29] ceramic overlays showed approximately 10% lower marginal adaptation than did composite overlays. Resin composite had a greater stress-dissipating effect than did ceramic.

Given that there is still no consensus on the optimal way to restore ETT, and given that the retention of adhesive restorations is based mainly on adhesion and does not require macroretentive elements,[35] the third null hypothesis, that there is no difference in the marginal adaptation of teeth restored with endocrowns or short or long posts, has to be accepted. Independent of post length, no relationship related to the percentage of continuous margin on both interfaces was found. It can be assumed that the three types of root retention could withstand intraoral masticatory forces to a similar degree.

CONCLUSIONS

In conclusion, thermomechanical loading had a significant effect on the marginal adaptation of both ceramic and composite restorations. CAD-CAM crowns fabricated from millable composite resin blocks (Paradigm MZ100) offer a superior option to all-ceramic crowns (IPS Empress CAD). However, the conclusions drawn from this *in vitro* study must be confirmed by controlled clinical trials before they can be applied as recommendations for routine clinical work.

Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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Departments

Faculty Positions

GROUP PRACTICE FACULTY COLLEGE OF DENTAL MEDICINE – ILLINOIS

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CDMI is seeking full-time clinical faculty to serve in the Group Practice student clinics at the rank of Instructor, Assistant, or Associate Professor to begin February, 2014. Primary responsibility is for the overall well-being of the patients being treated. The Group Practice faculty member has responsibility for instruction and demonstration in one-on-one, small group and plenary settings. The Group Practice faculty will supervise student dentists and provide direct patient care as required by the degree of difficulty of certain cases or for demonstration purposes. There is an opportunity to engage in scholarly activity, as deemed appropriate and as mutually agreed upon by the Group Practice Faculty and CDMI administration.

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Appointment at the Instructor, Assistant, or Associate Professor level will be based on individual experience and credentials. Midwestern University offers competitive salaries and opportunities exist for advancement.

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Midwestern University
Dental Institute
Attn: Dr. Darryn Weinstein
3450 Lacey Rd., Suite 120
Downers Grove, IL 60515

Applications can also be made on-line at:

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CLINICAL DIRECTOR FOR THE KUWAITI MINISTRY OF HEALTH EDUCATIONAL PROGRAM
Kornberg School of Dentistry

Temple University Kornberg School of Dentistry is seeking a Clinical Director for the Kuwaiti Ministry of Health Educational Program.

As part of an agreement with the Kuwaiti Ministry of Health, Temple University Maurice H. Kornberg School of Dentistry is seeking to recruit an assistant/associate professor and director who will supervise the clinical education of dentists in a two-year Advanced Education in General Dentistry program. The candidate must be able to practice dentistry in an AEGD or faculty practice. The candidate must have a DDS/DMD degree from an accredited US Dental School and a minimum of two years post-graduate training in Advanced Education in General Dentistry and/or General Practice Residency, or the equivalent. Candidates with additional education in public health (MPH or DrPH/PhD) and expertise in management of community clinics will be preferred Applicants should have at least two years of experience working in an academic, hospital, community health center or government institution with an in-depth understanding of clinical operations and management, as well as clinical practice experience. Candidates should possess exceptional interpersonal and leadership skills, and experience in teaching and working with international dentists. Candidates should be comfortable providing multidisciplinary oral health care for a wide variety of patients including patients with special needs and medically compromised patients; and advanced restorative, surgical, prosthodontics, periodontics, and surgical

care. Candidates must be eligible for Pennsylvania licensure.

Salary and rank will be commensurate with experience and qualifications. Temple University is an equal opportunity/affirmative action employer. Women and minorities are encouraged to apply. For confidential consideration, interested individuals should send a cover letter, curriculum vitae, and three references to Dr. Lisa Deem, Associate Dean for Admissions, Diversity, and Student Services, Temple University Kornberg School of Dentistry, 3223 North Broad Street, Philadelphia, PA 19140.

ASSISTANT/ASSOCIATE PROFESSOR
Kornberg School of Dentistry

Temple University Kornberg School of Dentistry is seeking applicants for a full-time Assistant/Associate Professor position to serve as the Director of the Division of Operative Dentistry, Department of Restorative Dentistry to begin July 1, 2013. The candidate should have experience and demonstrated leadership skills in teaching and providing patient care using current evidence-based methods in caries management. The responsibilities for this position will include designing and implementing a new curriculum in cariology and restorative dentistry, clinical supervision of students as general dentists, development of contemporary didactic curriculum materials, lecturing, preclinical instruction, and research in the field of operative dentistry. Applicants should be familiar with current pedagogical teaching methods including evidence-based oral health care. Qualified candidates must have a valid Pennsylvania dental license or the necessary credentials to obtain an institutional teaching license and must have an MSc in Operative Dentistry from a US program.

Send resume to Dr. Lisa Deem, Chair Search Committee, Office of the Dean, Temple University Kornberg School of Dentistry, 3223 North Broad Street, Philadelphia PA, 19140. Temple University is an equal opportunity/affirmative action employer, female and minority applicants are encouraged to apply.

GENERAL PRACTICE
Faculty Position - F30570
School of Dentistry

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Send curriculum vitae and a list of at least three references to: **Dr. Terrence Imbery, Chair of Search Committee, Department of General Practice, School of Dentistry, Virginia Commonwealth University, P.O. Box 980566, Richmond, VA 23298**.

Virginia Commonwealth University is an equal opportunity, affirmative action university, providing access to education and employment without regard to age, race, color, national origin, gender, religion, sexual orientation, veteran's status, political affiliation or disability.

Erratum

Figures one and two were unclear in, FJT Burke, V Singh, and NHF Wilson (*2013*) The Normalized Failure Index: A Method for Summarizing the Results of Studies on Restoration Longevity?. Operative Dentistry: September/October 2013, Vol. 38, No. 5, pp. 488–496. They have been recreated below. You have our apologies for any confusion they might have caused.

$$\frac{\text{Restorations failed}}{\text{Restorations evaluated x No of years' duration}}$$

Figure 1.

$$\frac{\text{Failure Index total}}{\text{Number of failed restorations}}$$

Figure 2.

Letter to the Editor

Sir,

We read with great interest a paper recently published by S Ardu [S Ardu, O Duc, I Krejci, and R Perroud (2013) Amelogenesis Imperfecta: A Conservative and Progressive Adhesive Treatment Concept. *Operative Dentistry* **38(3)** 235-241]. While it is a nice and generally well written case report, there are concerns about this article that we believe need to be addressed.

1. Figure 1c, d, f show a tooth like structure in the region between the maxillary left central incisor and canine, which can be confirmed radiographically in Figure 1g as the root stump of the maxillary left lateral incisor. However, the authors claim that the upper left lateral incisor is missing which is contradictory. Radiographs in Figure 4k reveal that the root stump has now been extracted and replaced by a cantilever bridge.

2. With a root stump of about 16 mm and adequate bone support as evident on the radiograph (Figure 1g), post & core followed by a crown could have been a favourable option for the maxillary left lateral incisor in this case of amelogenesis imperfecta.

3. Although the post treatment results are appreciable, the idea of extracting the root stump in relation to the maxillary lateral incisor in a young individual and placing a three-unit bridge is questionable.

Drs. Munish Goel; Gurmeet Singh Sachdeva; Shikha Bala; Shweta Verma; Neeraj Sharma; & Liza Sachdeva

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The Author's Respond:

The root of the upper left lateral incisor was extracted during the long term maintenance phase (the one where composites were realized). This decision for extraction was made after the patient's (and the patient's parents') refusal of orthodontic treatment. The "IDEAL" treatment plan was, in fact, an orthodontic alignment of the two arches as well as extrusion of the maxillary left lateral incisor root in order to place a post and core restoration followed by a long term provisional crown. Unfortunately, we had to respect the patient's wish and were not able to follow our "ideal" treatment plan.

Due to the refusal of the "ideal treatment plan" by the patient, other options were taken into consideration: an adhesive cantilever bridge for the long term provisional phase and a three unit bridge or an implant for the definitive phase.

The final option was the placement of a disilicate 3-unit bridge which was preferred over implant and crown placement. This decision was made due to the young age of the patient, the possibility of an apical migration of hard and soft tissues (with the consequent esthetic problems) and based on the favorable long term results reported in the literature for three unit bridges.

Drs. Stefano Ardu; Olivier Duc; Ivo Krejci; & Raymond Perroud

Faculty Members

Division of Cariology and Endodontology;

University of Geneva Dental School,

Geneva, Switzerland

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Inorganic and Prepolymerized Filler Analysis of Four Resin Composites

DC Salazar • J Dennison • P Yaman

Clinical Relevance

Selection of a composite resin based on high filler content, may be misleading. The reported filler content may vary, depending on the ratio of prepolymerized resin to inorganic glass particles. A composite material should be labeled with two values: total filler and inorganic filler contents.

SUMMARY

This study determined the filler content by weight percentage of four resin composites and examined the morphology, size, and elemental distribution of the filler particles. Four commercially available light-cured resin composites were evaluated for filler content by weight using ashing in air and acetone dissolution techniques. Ten specimens were analyzed for each material and technique. Specimens for ashing were heated to 650°C for 30 minutes. For the acetone dilution, the uncured specimens were dissolved, centrifuged, and decanted. In addition, scanning electron microscopy evaluation and energy dispersive x-ray spectroscopy analysis were performed to determine morphologic characteristics and elemental distribution, respectively. Filler percentages by weight for Aelite LS, Filtek LS, IPS Empress Direct, and Kalore from ashed in air were 86.44%, 77.86%, 72.17%, and 70.62%, and from acetone dissolution percentages were 85.05%, 75.56%, 78.88%, and 77.73%, respectively. Aelite LS had significantly higher filler content for both techniques. Kalore had significantly lower filler content for the ashing technique (70.62%), and Filtek LS had significantly lower filler content for the acetone dissolution technique (75.55%). Manufacturer reported filler content for Aelite LS (88%) and Filtek LS (76%) approximated the study results for both techniques, while Kalore (82%) and IPS Empress Direct (79%) were only similar for acetone dissolution, indicating higher content of prepolymerized particles. Morphologic examination showed spherical shaped particles for Aelite LS and splintered and irregular shaped particles for all other materials. Aelite LS had the highest filler content for both techniques. Values for filler content by weight using the acetone dissolution were closer to manufacturer reported values.

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INTRODUCTION

Since the first dental resin composites were developed, many efforts to improve their clinical performance have been undertaken. Researchers have suggested that filler content, size, and morphology of the filler particles within a composite resin formulation have the potential to influence the strength, elastic modulus, wear resistance, color matching, and polymerization shrinkage of a composite resin.[1-8] In addition, researchers have reported that increasing the filler particle size will effectively modify not only the pattern and rate of wear, but the restoration's polishability as well. It has also been stated that the greater the particle's size, the greater the potential for wear, which in turn affects mechanical properties of composites.[3,8-10] Thus, it would seem reasonable to expect more studies reporting correlations between mechanical properties and filler particle morphology and size. Perhaps the lack of some investigations is due to the difficulty in determining the exact size of the filler particles within a composite resin.[11] Furthermore, attempts to improve clinical performance and to decrease polymerization stress of methacrylate-based composites have focused on the development of new monomers, such as ring-opening silorane chemistry[12-14] and a new nanofiller technology.[7,15,16]

Only one study has reported measuring filler content by weight for current commercial composites using a technique that preserves prepolymerized particles.[17] Furthermore, there is no standard procedure for verifying a manufacturer's report of filler loading and size except by the least expensive method of ashing in air.[18] Ashing in air is a standard technique that has been used in numerous studies to determine filler content by weight.

Several methods have been suggested to determine the filler loading in resin-based composites: thermogravimetric analysis, gas pycnometry, or ashing in air. Ideally, filler loading should not vary with the different test methods. However, different results have been reported by varying the test method among resin-based composite materials. Factors like organic matrix and inorganic fillers as well as silane coating and prepolymerized particles have been reported to influence the filler content analyses of these materials.[8-10,19-21]

Scanning electron microscopy (SEM) often uses a dissolution technique with acetone or ethyl alcohol to remove the organic matrix from inorganic fillers.[3,8-10,22-25] According to some manufacturers, ashing in air can burn off some of the filler content of composites and thus give false results. To combat this problem, a separation of the matrix and filler using acetone needs to be explored. It is hypothesized that a solvent such as acetone will not break down prepolymerized filler, silane, agglomerates, or clusters from composite formulations. Acetone dilution has been used to remove the organic matrix for SEM analysis in many studies but has not been addressed as a possible technique to determine filler by weight. A solvent such as acetone is an alternative to determine filler by weight when including prepolymerized particles.[17]

The aim of this study was to determine the filler by weight percentage of four different resin composites using both ashing in air and acetone dissolution techniques. A secondary aim is to examine the morphology and composition of the filler particles in each material.

MATERIALS AND METHODS

Four commercially available resin composites (Table 1) were chosen based on their reported filler content by particle size and evaluated in this study for filler content by weight percentage. In addition, energy dispersive x-ray spectroscopy (EDS) analysis and SEM morphologic characterization were performed on the filler particles. Filler percentages obtained were compared against manufacturer's data.

Ten specimens were analyzed for each material and technique. For acetone dissolution, specimens of 0.5 g of each material were mixed with 10 mL of acetone (electronic grade, Fisher Chemical, Fair Lawn, NJ, USA) in a test tube (Pyrex 50 mL, Fisher Scientific, Hanover Park, IL, USA). All tubes were weighed initially empty and then weighed again following loading with the 0.5-g specimen. Tubes were covered with aluminum foil to prevent light exposure. All specimens initially were agitated (Maxi-mix 1, Thermolyne, Dubuque, IA, USA) until all solid was dissolved and verified visually. Agitation continued with a gyratory shaker (G10, New Brunswick Scientific, New Brunswick, NJ, USA) within a controlled temperature chamber (Norlake Scientific, Hudson, WI, USA) at 37°C for 1 hour. Specimen tubes were centrifuged (Centrifuge 5810R, Eppendorf, Hamburg, Germany) for 15 minutes at 4000 rpm and then decanted twice (5 mL pipette, Novamed Inc, Lawndale Skokie, IL, USA) for a total of 9.5 mL. The specimens were left to dry overnight in the temperature-controlled chamber to ensure all acetone evaporated, and weights of the specimens were measured to the nearest 0.001 g the following morning. The entire process was repeated twice to

Table 1: *Material Specifications as Reported by Manufacturer.*

Brand Name	Filler Type	Manufacturer	Batch Number
IPS Empress Direct	Nanohybrid	Ivoclar	M68450; N10129
Filtek LS	Microhybrid	3M ESPE	N182605; N205729
Kalore	Nanohybrid	GC America	0907141; 0907101
Aelite LS	Microhybrid	Bisco	1000007473; 1000011945

ensure dissolution of the organic matrix. The calculation of filler percentage by weight was the same as the ashing technique except that residual material might include prepolymerized fillers, clusters, and possible silane contents. The formula to determine percentage by weight of the specimens after acetone dissolution was as follows:

$$\text{Weight (Wt) percent} = \frac{\text{Wt after dissolution} - \text{Wt of tube}}{\text{Initial Wt of sample}} \times 100.$$

For the ashing in air, the filler content of the selected composites was determined by using a burn out furnace (Neymatic 101, JM Ney Company, Bloomfield, CT, USA). Crucibles were weighed empty and then weighed after specimen loading using an analytical scale (Analytical Standard, AS200-S, O'Haus, Florham Park, NJ, USA). The crucibles (High alumina, 10 mL, Cole-Palmer Instrument Co, Golden, CO, USA) loaded with specimens were introduced in the furnace after the temperature had reached 650°C and left for 30 minutes. The crucibles with the ashed specimens were again weighed on an analytical scale.

Filler percentages from ashing in air and acetone dissolution technique were statistically analyzed by one-way analysis of variance (ANOVA), independent *t*-tests, Tukey multiple comparison, and Pearson correlation.

EDS is a microanalytic technique that is based on the characteristic x-ray peaks that are generated when the high-energy beam of the electron microscope interacts with the specimen. Each element yields a characteristic spectral fingerprint that may be used to identify the presence of that element within the sample. The relative intensities of the spectral peaks may be used to determine the relative concentrations of each element in the specimen. The x-ray signal is detected by a solid-state silicon-lithium detector, and the construction and efficiency of this detector sets a lower limit on the atomic number that may be detected. Generally, elements heavier than carbon are detectable.[22]

For the SEM evaluation, the residual fillers from ashing in air were mixed with acetone to produce a suspension that was placed on an aluminum SEM stub and allowed to dry. Morphologic and size evaluations were done using a FEI Quanta 200 3D focused ion beam workstation and environmental scanning electron microscope with an EDS system at magnifications of 20,000× and 40,000× at 20-30 kV beam acceleration voltage. The same areas of the samples utilized for SEM observation were additionally x-ray scanned using the same microscope for qualitative and semi-quantitative analyses to determine the elemental distribution profile.

RESULTS

Table 2 shows the means and standard deviations of the materials evaluated by ashing in air and acetone dissolution techniques. Weight percentage of inorganic fillers from ashed specimens ranged from 70.62% to 86.44%. Weight percentage of fillers from acetone dilution specimens ranged between 75.56% and 85.05%. Mean values for each material were significantly different when comparing ashing with acetone dissolution techniques. Both techniques were extremely precise, with standard deviations between 0.1% and 1.1%. Aelite LS and Filtek LS

Table 2: *Filler by Weight (%)*

Product	Manufacturer Values	After Ashing in Air	Manufacturer Difference (%)	After Two Acetone Dissolutions	Manufacturer Difference (%)	Significance (Independent *t*-test)
IPS Empress Direct	79	72.12 (0.1)	9	78.88 (0.5)	0	$p<0.001$
Filtek LS	76	77.86 (0.1)	2	75.56 (0.2)	1	$p<0.001$
Kalore	82	70.62 (0.3)	14	77.73 (0.3)	5	$p<0.001$
Aelite LS	88	86.44 (1.1)	2	85.05 (0.5)	3	$p=0.003$

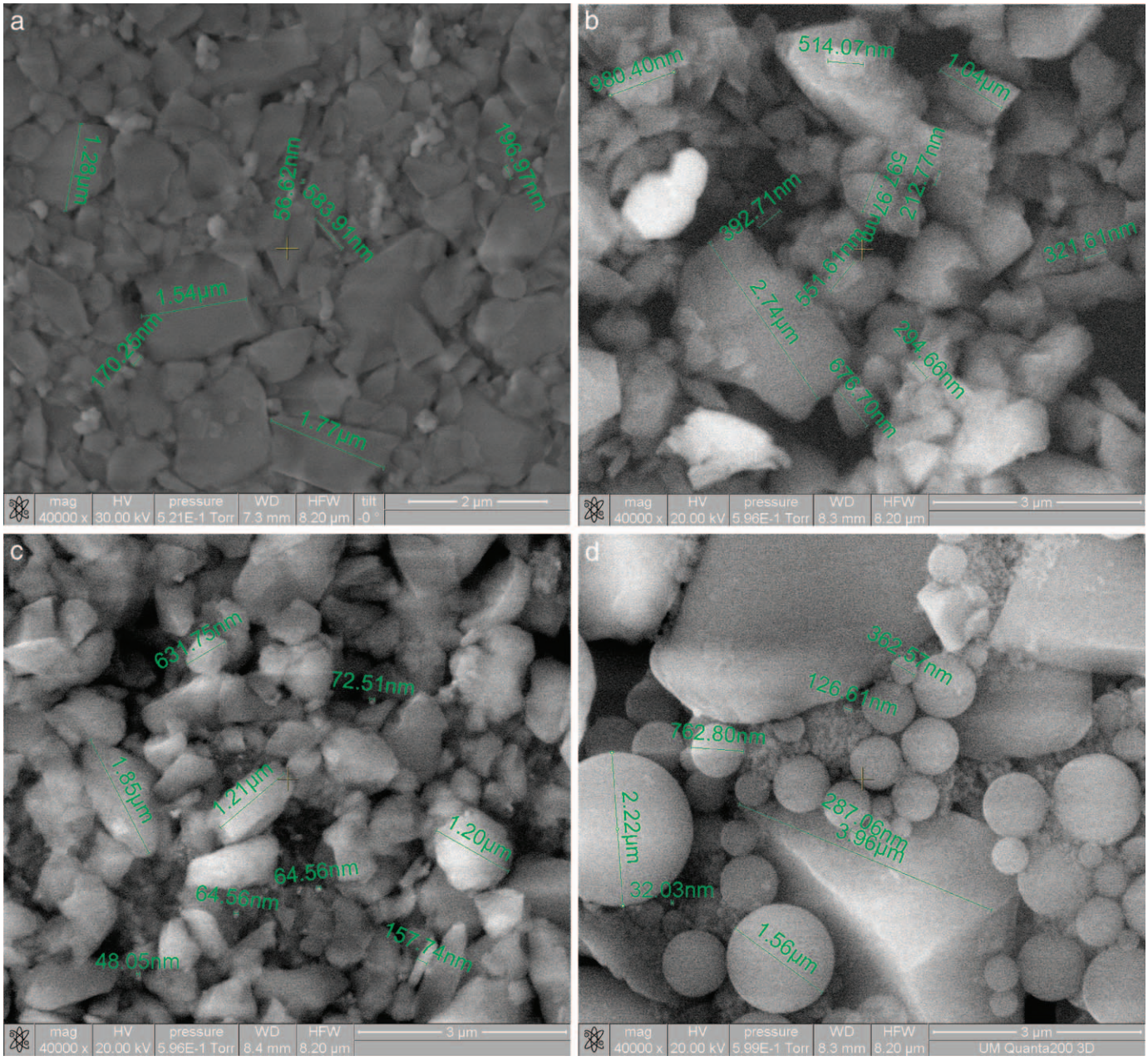

Figure 1. *(A): SEM photomicrographs and filler measurements of IPS Empress Direct composite after acetone dissolution at 40,000× magnification. (B): SEM photomicrographs and filler measurements of Filtek LS composite after acetone dissolution at 40,000× magnification. (C): SEM photomicrographs and filler measurements of Kalore composite after acetone dissolution at 40,000× magnification. (D): SEM photomicrographs and filler measurements of Aelite LS composite after acetone dissolution at 40,000× magnification.*

showed closer results for percentages by weight to manufacturers' reported data when using ashing (2%) and acetone technique (1%-3%). On the other hand, Kalore and IPS Empress Direct showed results closer to manufacturer reported data when using acetone dilution for weight measurements (0%-5%). Aelite LS had a significantly higher loading than the others for weight percentage for both ashing in air and acetone dilution. Both techniques were highly correlated (r=0.72).

Representative SEM photomicrographs (backscattered electron images) of the composites evaluated in this study at 40,000× are shown in Figure 1A through D. Irregular particles (3.96 µm) in conjunction with smaller spherical (32.03 nm-2.22 µm) filler particles and agglomerated particles were observed in Aelite LS. Overall, Aelite LS was the material that

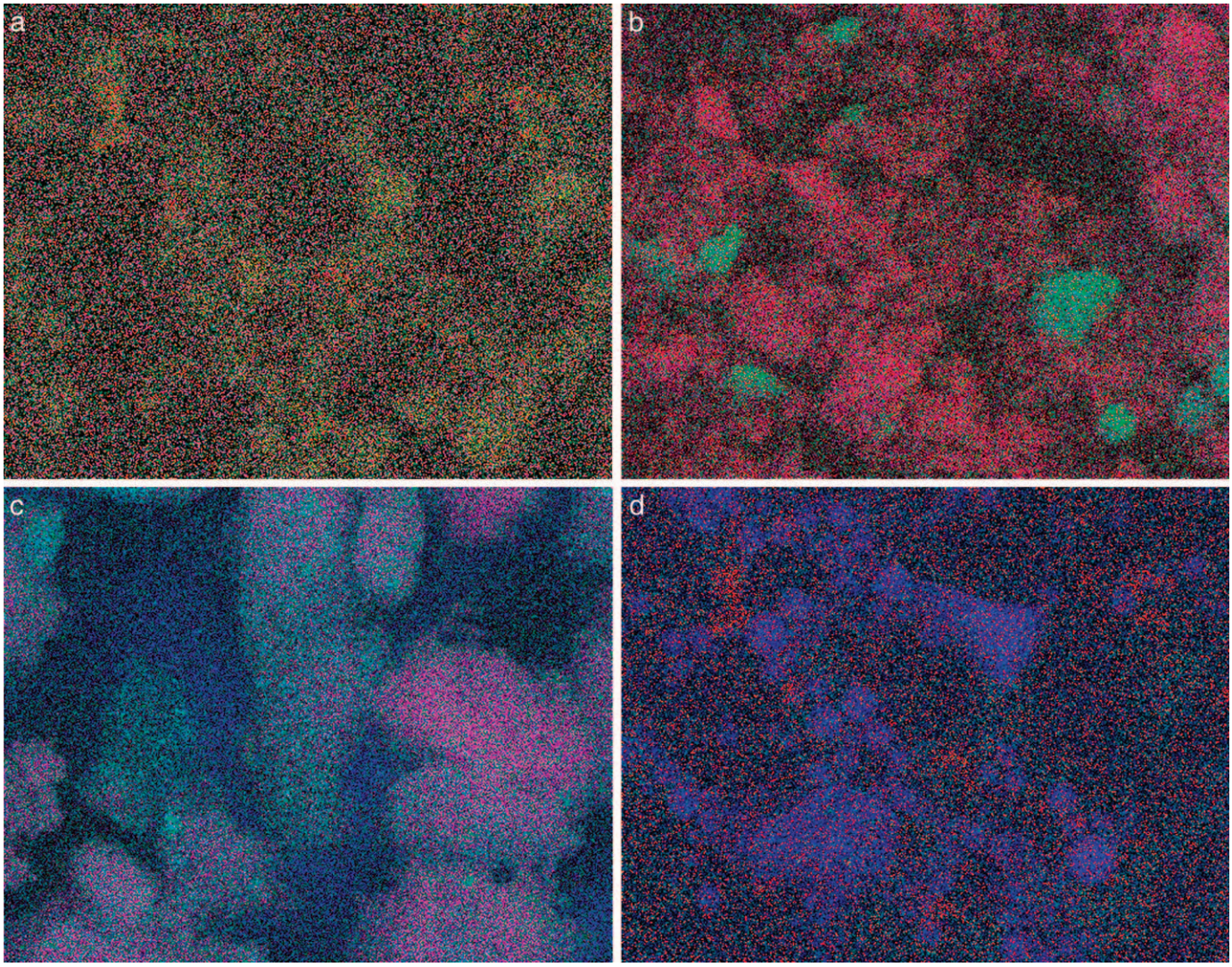

Figure 2 (A): SEM photomicrographs used for EDS analysis and color mapping obtained by the superimposition of every individual element distribution in the IPS Empress Direct composite: Si-yellow, Al-pink, O-purple, F-green, Yb-red, Ba-light blue. (B): SEM photomicrographs used for EDS analysis and color mapping obtained by the superimposition of every individual element distribution in the Filtek LS composite: Si-pink, Mg-blue, Al-purple, F-green, O-red, Y-light blue. (C): SEM photomicrographs used for EDS analysis and color mapping obtained by the superimposition of every individual element distribution in the Kalore composite: Si-pink, Al-purple, Mg-blue, F-green, O-yellow, Yb-light blue. (D): SEM photomicrographs used for EDS analysis and color mapping obtained by the superimposition of every individual element distribution in the Aelite LS composite: Si-blue, Al-red, O-light blue.

showed the biggest (3.96 μm) and smallest filler particles (32.03 nm) (Figure 1D). Irregular and splintered filler particles of different sizes were seen in Filtek LS, IPS Empress Direct, and Kalore. In Filtek LS, most of these particles were somewhat larger (598 nm-2.74 μm), while the remaining particles were between 212.77 nm and 321.61 nm (Figure 1B). Kalore showed the most regularly shaped filler particles among all composites evaluated, with a mix of medium (631 nm-1.85 μm) and small (48 nm-157 nm) size particles (Figure 1C). On the other hand, in IPS Empress Direct, a more irregular shape pattern in the filler particles was observed, with particles typically ranging from 1.28 μm to 1.77 μm, and the smallest particles being around 56.62 nm (Figure 1A). The elements detected in the resin composites are shown in Figure 2A through D and Table 3. Silica, aluminum, and oxygen were found in all of the resin composites tested and were the main components of the fillers.

DISCUSSION

Many methods could be used to study the filler concentration by weight. Thermogravimetry is a technique in which the mass of a material is

Table 3: *Elements Detected and Filler Content of Resin-based Composites*

Product	Filler Particle Size Range	Elements Detected
IPS Empress Direct	57 nm-1.8 μm	Si, Al, O, F, Yb, Ba
Filtek LS	212 nm-2.7 μm	Si, Al, O, Mg, F, Y
Kalore	48 nm-1.9 μm	Si, Al, O, Mg, F, Yb
Aelite LS	32 nm-4 μm	Si, Al, O

Abbreviations: Aluminum (Al); barium (Ba); fluorine (F); magnesium (Mg); oxygen (O); silica (Si); strontium (Sr); ytterbium (Yb); yttrium (Y); and zirconium (Zr).

monitored as a function of temperature and time as a sample specimen is subjected to a controlled temperature program.[11,21,26] The thermogravimetry curve pattern varies according to the materials tested, thus reflecting the variations in organic composition. Ashing in air, on the other hand, is the technique most frequently used to determine the percentage of fillers by weight. It is based on the elimination of the organic fraction of the resin composite by heating the composite to a constant temperature. Some authors ashed the materials in an electric furnace at temperatures that ranged between 600° and 700°C, respectively, for 30 minutes.[3,8-10] Aelite LS (86.44%) and Filtek LS (77.86%) composites showed the highest amounts of inorganic content after ashing in the furnace. When comparing these results with the manufacturer's reported data, the microhybrid materials showed closer results for ashing in air (2%). On the other hand, the nanohybrid composite's (Kalore and IPS Empress Direct) results after ashing showed a greater difference from the manufacturer's data (9%-14%).

Variations within these composites could be explained by the definition of filler content. The filler content reported by manufacturers sometimes includes only the inorganic filler particles, and anything organic is vaporized when the composite is subjected to ashing temperatures. Prepolymerized particles in many composites use the organic matrix and inorganic filler particles, which are first cured into solid blocks and then milled or ground down into sizes ranging from 17 μm to 60 μm.[1,27] The milled particles are added to a nonpolymerized resin along with inorganic particles and dispersed aggregates to increase loading. Prepolymerized fillers are relatively large fillers with less surface area, enabling greater weight filler loading and thereby resulting in less volumetric shrinkage. These larger fillers also prevent the resin matrix from moving as a result of friction between the resin and the prepolymerized filler surface during curing, thereby reducing shrinkage.[28]

Prepolymerized particles present different shapes and sizes, and they can have as much as 50% organic content.[10,11,21,22,24,28,29] It is of interest as to what constitutes filler content with different manufacturers. IPS Empress Direct and Kalore have prepolymerized particles in their filler composition. During ashing in air, organic matrix as well as prepolymerized particles can be vaporized, which can result in significantly lower percentage by weight values (Table 2). Prepolymerized particles with organic content were included in the manufacturer filler calculation of IPS Empress Direct and Kalore, which is reflected using the acetone dissolution technique. The manufacturer of IPS Empress Direct reported 50.2% barium-aluminum-fluorosilicate glass, 9.8% ytterbium trifluoride, and 19.6% prepolymerized particles in its total filler percentage by weight of 79%. On the other hand, Kalore reported a higher filler content, with a total of approximately 50% fluoroaluminosilicate and strontium glass and 32% prepolymerized particles. So, Kalore and IPS Empress Direct are directly related in how their filler percentages by weight were calculated. These nanohybrid composite results after acetone dissolution were closer to the filler weight reported by the manufacturers, and the small differences may be due to some particles lost during each acetone pipette decantation step. However, these small differences were not seen when using ashing in air technique instead of acetone for the same measurement. According to their manufacturer, Aelite LS and Filtek LS do not have prepolymerized particles incorporated in their filler composition, which resulted in the smaller difference between ashing in air and acetone dissolution (less than 3%). The microhybrid manufacturers' reports were very close to the results obtained in this study, and the small differences could be due to loss of some particles during handling of samples from furnace to analytical scale after ashing or loss of particles during decantation in the acetone dissolution. Aelite LS had the highest percentage of filler content by weight for both techniques, while Filtek LS had the lowest percentage of filler by weight after acetone dissolution (75.56%).

Others factors could explain differences found between our data and those given by the manufacturers. The first one is the variable amount of silane. The silanation process plays a main role in the adhesion of the organic resin matrix to the inorganic mineral fillers.[30] Manufacturers and laboratories treat the filler-matrix interface according to their own methods and use different ways to calculate the

percentages of fillers.[8] Some manufacturers seem to evaluate the percentage of fillers by weight before the silanization process of the fillers, while others include the percentage of silane coating in their calculation. In addition, the surface area of the fillers will affect the percentage of silane used—the smaller the fillers, the higher the quantity of silane.

Furthermore, samples from each composite were dissolved in acetone to evaluate the filler content by weight using a technique that preserves the prepolymerized particles. Previous studies had evaluated filler structure and size,[8,10,11,22,24] but only one previous study has determined filler percentage by weight using the acetone dissolution technique.[17] With the new generation of composites, it will be necessary to evaluate these materials considering their prepolymerized particles. A pilot study was done to determine the amount of cycles of acetone dissolution necessary to obtain a stable percentage by weight. No significant differences were found between the second and third dissolution; therefore, two cycles of acetone dissolution were chosen as a standard procedure. Kalore showed the highest difference in filler content by weight for the acetone dissolution technique (77.73%). A similar increase was seen with IPS Empress Direct when comparing ashing in air (72.17%) and acetone dissolution (78.88%). Aelite LS and Filtek LS showed a decreased weight percentage when using acetone dissolution, which may be related to residual nanoparticles suspended in the acetone after centrifuge sedimentation that are lost during pipette aspiration.

For the filler morphology analysis, the samples dissolved in acetone were collected and dissolved a third time for mounting purposes. On the SEM evaluation, all composites showed an irregular to splintered shape except for Aelite LS, which is the only composite in this study that contains spherical particles mixed with irregular-shaped particles. A spherical shape is known to have many advantages such as to allow an increased filler load for composites and also to enhance their fracture strength, surface roughness, and shrinkage strain since mechanical stresses tend to concentrate on the angles and protuberances of the filler particles.[3,8,10,31,32] The spherical particles had different diameters that ranged from 32 nm to 2 μm, while the bigger irregularly shaped particles presented an average size of 4 μm with some nanoclusters and dispersed nanoparticles surrounding the bigger filler particles.

For Filtek LS, the SEM images confirmed a distinct filler morphology of irregularly shaped fillers. The manufacturer of Filtek LS claimed that the radical change in the shape of the fillers, among the Filtek composite varieties, is related to the specific characteristic that the silorane-based organic matrix needs in its composition. Silorane is known to contain quartz particles, which cannot be processed by a sol-gel technique and may explain the more irregular morphology compared with other materials provided by the same manufacturer. According to the manufacturer, the average size range was from 40 nm to 1700 nm; however, the SEM images in this study showed particles that ranged from 200 nm to 3 μm in size. In addition, the images showed a more heterogeneous size pattern when compared to Kalore, the other microhybrid material.

According to the manufacturer, Kalore contains 60% filler content by weight of 400 nm nano-sized modified strontium glass and 20% filler content by weight of 100 nm of lanthanoid fluoride. Prepolymerized nanoclusters of fillers, inorganic fillers, and mono-dispersed particles, contained in this specific nanohybrid composite present size ranges between 16 nm and 17 μm. When the samples were evaluated under SEM, the images showed more regular shaped filler particles whose bigger particles ranged from 632 nm to 1.8 μm and small particles ranged from 48 nm to 158 nm. Among all materials, Aelite LS (32 nm) and Kalore (48 nm) are the materials that had the smallest filler particles in their structure. In addition to Kalore and Fitek LS, IPS Empress Direct also showed irregular shaped particles with large fillers that ranged from 1.28 μm to 1.77 μm and smaller aggregates of particles with sizes from 56.62 nm to 196.97 nm.

In this study, prepolymerized particles were not evaluated for elemental analysis because the components of each composite material were disrupted during the acetone dissolution technique; therefore, further studies should be done keeping the organic matrix of each resin-based composite.

Many modifications have been made recently to the elemental composition of filler particles. Elements such as aluminum and lithium were added to decrease the hardness on these particles, to be able to break them in smaller sizes. Barium, yttrium, zinc, and strontium are added to develop radiopacity. Some manufacturers have claimed that yttrium fluoride can be incorporated to release fluoride ions for anticaries activity.[21] In this study, the EDS analysis of the fillers revealed a variety of elements,

depending upon the individual products. Colors representing each element were picked randomly for each analysis (Table 3, Figure 2).

For IPS Empress Direct (Figures 1A and 2A), the filler particle sizes are well dispersed, and the predominant elements of silica (yellow) and fluoride (green) appear to be concentrated in the larger particles; ytterbium (red) and barium (light blue), however, appear to be in smaller, more dispersed particles. Aluminum is generalized throughout the field, in keeping with the stud background on which the fillers were placed.

For Filtek LS (Figures 1B and 2B), the particles are larger and more distinct (Figure 1B). Silica (pink) and oxygen (red) are heavily concentrated in the more distinct larger particles (Figure 2B), and separate less numerous particles contain a high concentration of fluoride (green) as well as magnesium (blue) and yttrium (light blue) for radiopacity. Again, the aluminum (purple) forms the general background color from the mounting stub.

For Kalore (Figures 1C and 2C), the larger particles appear to contain heavier concentrations of ytterbium (light blue) and magnesium (blue) with some aluminum (purple) microconcentrations within specific particles. Other particles appear to contain heavier concentrations of silica (pink). Small amounts of fluoride (green) were distributed around the edges of most particles, and there was no oxygen (yellow) evident in most fields.

For Aelite LS (Figures 1D and 2D), the predominant element is silica (blue) in both larger and smaller particles, and small concentrations of aluminum (red) in smaller particles, with a general distribution of oxygen (light blue) in low concentrations. There was no evidence of fluoride, magnesium, or a heavy metal ion.

A clinician selects a composite expecting good physical properties and esthetics. If that decision is made based on the manufacturer's reporting of filler weight percentage, the clinician may be misled by assuming that those percentages are inorganic content. This study has shown that the filler weight percentage that is reported may be affected by techniques used to determine that percentage as well as by the inclusion of prepolymerized particles.

CONCLUSIONS

1. Mean values for all materials were significantly different for both ashing and acetone dissolution techniques, but there was a strong correlation between the two tests.
2. Aelite LS had significantly higher filler content for both ashing and acetone dissolution.
3. Kalore had significantly lower filler content for ashing (70.62%), and Filtek LS had significantly lower filler content for acetone dissolution (75.56%).
4. Manufacturer reported filler content for Aelite LS (88%) and Filtek LS (76%) approximated the study results for both techniques, while Kalore (82%) and IPS Empress Direct (79%) were only similar for acetone dissolution, indicating higher content of prepolymerized particles.
5. Morphologic examination showed spherical shaped particles for Aelite LS and splintered and irregular shaped particles for the other materials.
6. The elements detected were silica (Si), aluminum (Al), strontium (Sr), yttrium (Y), magnesium (Mg), fluorine (F), ytterbium (Yb), oxygen (O), zirconium (Zr), and barium (Ba). Si, Al, and O were the common elements in the composition of all four resin composites evaluated.
7. Manufacturers should provide two filler content values for a composite resin material—inorganic filler content and total filler content.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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Marginal Adaptation of Direct Class II Composite Restorations with Different Cavity Liners

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Clinical Relevance

The application of flowable composite as a liner may not improve marginal adaptation and is product dependent. Lining the cavity with a 1-mm-thick layer of a bonding agent improves marginal adaptation but, clinically, may be problematic.

SUMMARY

The aim of this study was to evaluate how cavity linings with different elastic modulus can influence the marginal adaptation (MA) of Class II composite restorations before and after thermo-mechanical loading.

Materials and Methods: Forty Class II cavities with margins extending 1 mm below the cement-enamel junction were prepared in extracted human third molars. In each group except the control group, a lining material of 1-mm thickness was applied to the bottom of the cavity and polymerized before placing the resin composite Herculite XRV Ultra (group A: control; group B: Premise Flowable lining; group C: Herculite XRV Ultra lining; and group D: Optibond FL lining). MA was evaluated (with a scanning electron microscope) before and after loading (200,000 loading cycles). Statistical analysis was done using the Shapiro-Wilks test, the analysis of variance test, and Duncan *post hoc* test at $p<0.05$.

Results: Before loading, the percentages of continuous margins in dentin were superior ($p<0.05$) for groups C and D (71.1% and 87.2%, respectively) compared to groups A and B (55.7% and 48.3%, respectively). After loading, group D (79.8%) was statistically superior in dentin compared to all of the other groups (43.6%, 35.9%, and 54.4%, respectively). In occlusal enamel, no significant difference was found between groups. The percentage of enamel fractures and the percentage of non-continuous margins in proximal enamel were high, with no significant difference between liners. It can be concluded that for the materials used in this study, a 1-mm-thick lining with an extremely low elastic modulus (2-3 GPa) could redistribute shrinkage stress. The

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use of a flowable composite did not significantly improve MA.

INTRODUCTION

Light-cured resin composites (RCs) benefit today by an increasing spectrum of indications; their application has been expanded to include the restoration of anterior tooth wear[1-6] and noncarious cervical lesions.[7-10] Therefore, RCs continuously face challenges associated with specific environments, such as high tensile stresses in cervical areas as well as increased expectations with regard to longevity. Long-term stability is considered to be affected to a significant degree by marginal integrity.[11-13]

The marginal seal of composite restorations represents an increasing concern. Over the years, *in vitro* evaluations of the performance of resin adhesives revealed that microleakage and gap formation, mainly at the dentin-composite interface, did not improve at the same rate as did bond strength values.[13-16] Independent of the bonding capacity of an adhesive system, it seems that adhesive restorations are far from assuring a perfect marginal seal, with degradation in time occurring regardless of the product used.[17] Marginal integrity is considered to be the result of several parameters related to the forces created by curing contraction, as bond strength alone could not be correlated to marginal adaptation (MA) in a study undertaken by Kemp-Scholte and Davidson.[18]

A consensus can be found in the literature with regard to the effect of curing contraction in restoration performance. As a result of the confinement of the restoration, curing contraction has been shown to generate strain along the tooth restoration complex, leading to pre-stressed obturations, which are more prone to long-term degradation. The unfavorable effects of polymerization contraction forces can be evaluated effectively by marginal analysis, as marginal gaps offer proof of the forces generated by polymerization shrinkage or by thermo-mechanical strain, exceeding the bond strength.[15] Other methods of assessing the effects of polymerization contraction are finite element analyses (FEA) of the stress distribution,[10,19-21] measurements of tooth deformation[22-27] and, recently, a three-dimensional (3D)-deformation analysis.[28]

The phenomenon of force development in contracting materials was first described in the dental literature by Bowen.[29] Later, the subject of polymerization contraction stress was studied in depth in an attempt to clarify the complex interaction among the many factors involved. An interplay of polymerization shrinkage, elastic modulus (E-modulus), viscous flow capacity, and conversion degree of the material as well as adhesive ability, cavity configuration, and tensile stresses exerted on the restoration[12,14,19,30-33] is considered responsible for the marginal integrity. The combined action of several factors creates various stresses on the restoration-tooth interface, with consequences such as debonding or strain on the remaining tooth structure creating enamel fissures, fractures,[34] and cuspal movement.[22,25,35] Authors of studies[20,23,27] employing the FEA method also developed maps of polymerization stress accumulation, showing the distribution patterns of the forces and the effect on remaining tooth structures.

The dimensional modification due to shrinkage depends on the number, size, and functionality of the monomers as well as on the filler load. The volumetric contraction undergoes two phases: the phase before and the phase after the gel point. During the first phase, the resin retains its capacity to flow, and, therefore, it compensates the contraction forces by rearrangement of the molecules by preventing strain that would otherwise develop at the interfaces. During the second phase, however, the contraction forces are directed toward the bonded surfaces, leading to, among other things, defects in the MA. These defects occur when the elasticity of the material is not sufficient to compensate for the contraction forces, thus allowing for stress to develop at the interface of the tooth restoration complex. High strain capacity (reduced E-modulus), high viscous flow, and small amounts of polymerization shrinkage lead to reduced gap formation.[19]

Extensive research was done on the implications associated with the E-modulus of the restorative material. Flexibility is defined as the ability of a material to strain without becoming permanently deformed.[36] The higher the E-modulus and the polymerization shrinkage of the composite, the higher the contraction stress will be.[12,20,23,37] According to physical laws and *in vitro* observations, a low E-modulus is supposed to play an important role in stress relief, becoming, therefore, a desired property of the material.[19,20] It is assumed that an increased flexibility would lower stress values on the interface.[19,20] However, both the E-modulus and contraction are related to the filler content: a higher filler load would lead to a lower contraction but to an increase in stiffness as well.[12,14,31,36,38] A way to overcome this difficulty within material composition involves the use of a flexible material as liner under

the restorative material, giving rise to the 'elastic wall' theory. Based on the hypothetical benefits of increasing the compliance of the prepared cavity artificially, low-modulus intermediate layers such as flowable RCs and unfilled adhesives were presented as stress breaking materials.[33,36,38,39]

Different studies[18,20,37,40] measured stress relief when an unfilled resin with increased thickness was used as intermediate stress breaker, and an FEA study[20] was able to create a stress map of the tooth-restoration complex measuring different stress relief values for different thicknesses of the adhesive layers. However, studies evaluating the influence of flowable composites placed as intermediate layers on marginal integrity show conflicting results. Based on previous research, a general opinion prevails indicating that the behavior of a material as a stress absorber cannot be predicted using the E-modulus alone, as polymerization kinetics is a complex material-specific phenomenon.[12,38,41] Therefore, controversy is still present in the literature regarding the supposed positive effect of flowable composites on stress relief and marginal integrity.

Despite this high relativity, some hypotheses have been promoted and need further investigation. Accordingly, some authors[19,37,42] suggest the use of an extremely low E-modulus in order to obtain a significant effect, while others[37,42] suggest placing a pre-cured layer on top of the adhesive regardless of the composition (restorative or flowable RC).

The null hypothesis tested was that no difference in terms of percentage of continuous margin (% CM) exists among restorations with liners of different elasticity. The independent variables that were kept constant were loading condition (before and after loading), cavity lining (no lining; Optibond FL liner; Herculite XRV Ultra; and Premise Flowable) and tooth substrate (occlusal enamel [OE]; proximal enamel [PE]; and cervical dentin [D]). The only tested variable was MA, expressed in % CM.

MATERIALS AND METHODS

Cavity Design

Forty Class II occluso-mesial cavities, with margins extending 1 mm below the cemento-enamel junction, were prepared in extracted human third molars (for each tooth only one cavity was prepared). The teeth were stored in 0.1% thymol solution, and after scaling and pumicing, teeth were randomly assigned to four experimental groups (n=10) and mounted on custom-made specimen holders using a cold polymerizing resin (Technovit 40721, Haereus Kulzer, Wehrheim, Germany). Prior to the mounting, the apices were sealed with an adhesive system (Optibond FL, Kerr, West Collins, CA, USA). Diamond burs (80 μm; Universal Prep set, Intensiv SA, Lugano, Switzerland) were used under continuous water spray. The standardized dimensions for the proximal cavity were as follows: width = 5 mm, height = 6 mm (measured from the tip of the palatal cusp to the gingival margin), depth = 2 mm (mesio-distal width of the gingival floor). For the occlusal cavity, the dimensions were as follows: depth = 3.5 mm (measured from the tip of the palatal cusp) and width (vestibule-oral dimension) = 5 mm.

Materials

For all groups, a nano-hybrid composite (Herculite XRV Ultra) was used for the restoration. A three-step etch-and-rinse system (Optibond FL) with a proven bonding capacity, a 48% filler load, and a very low E-modulus was used as the adhesive. The flowable composite Premise Flowable was chosen for its low polymerization shrinkage relative to other flowable composites and for its high percentage of filler load, assuring good mechanical properties as the lining material is in direct contact with the oral environment (Figure 1). All of the above-mentioned materials were provided by the same manufacturer, therefore assuring compatibility. The properties of the mentioned materials are listed in Table 1.

Restorative Procedures

Restoration was performed immediately after cavity preparation in order to avoid alteration of dental tissues. All cavities were beveled in the occlusal box.

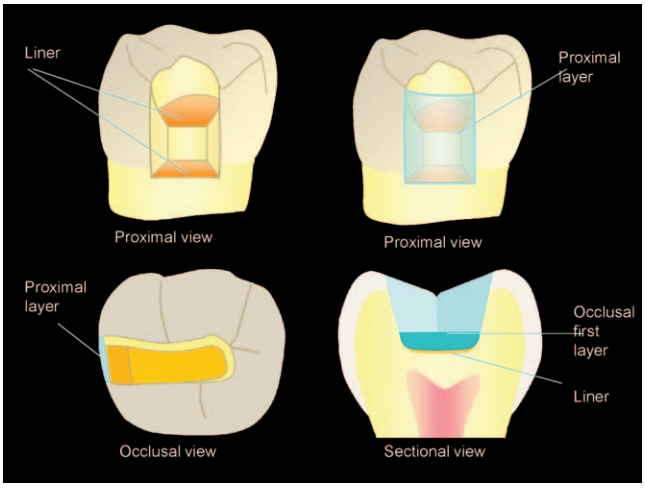

Figure 1. *Application of the liner and layering technique of the RC.*

Table 1: *Material Properties*[a]

Material	Elastic Modulus, GPa	Volumetric Polymerization Shrinkage, %	Filler Load by Weight, %
Herculite XRV Ultra	9.4	2.7	78
Premise Flow	7.55	3.31	72.5
Optibond FL	1-3	—	48[a]

[a] *Data provided by the Kerr Corporation.*

The teeth were randomly assigned to one of the four groups, as follows.

Group A (Control Group)

The cavities were encircled with a metallic matrix band after conditioning enamel and dentin with a 37% H_3PO_4 gel (Gel Etchant, Kerr) for 45 seconds and 15 seconds, respectively. Dental tissues were rinsed and nearly dried with a light air pressure spray (two to three seconds) before the application of the two adhesive components (Primer and Adhesive), following the manufacturer's instructions. The bonding resin was light-cured (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) for 40 seconds. This step was preceded by the buildup of the proximal wall with a unique vertical increment of less than 1-mm thickness (centripetal layering technique). The vertical increment of the proximal box was cured for 40 seconds from the occlusal direction. The matrix band was then removed, and the composite resin was applied using a three-increment layering technique for the occlusal cavity (Figure 1). Each increment in the occlusal cavity was 1.5 mm thick and was individually cured for 40 seconds (light intensity produced by the curing device = ~1000 mW/cm^2).

Group B

The same steps were performed as for group A. After polymerization of the adhesive system, a thin lining layer of Premise Flowable (1-mm thickness) was applied on the bottom of the occlusal cavity and the proximal box (exposed to external environment) and polymerized before the buildup of the proximal wall. The occlusal cavity was further filled, after removal of the matrix band, using the same composite, Herculite XRV Ultra, and the same layering technique as described for group A (Figure 1).

Group C

The same steps were performed as for group A until cavity restoration. After polymerization of the adhesive system, a thin lining layer of the restorative composite Herculite XRV Ultra (1-mm thickness) was applied on the bottom of the occlusal cavity and the proximal box and was polymerized before buildup of the proximal wall. The occlusal cavity was further filled after removal of the matrix band using the same composite, Herculite XRV Ultra, and the same layering technique as described for group A (Figure 1).

Group D

The same steps were performed as for group A until cavity restoration. After polymerization of the adhesive system, a thick layer of the adhesive from the Optibond FL adhesive system (1-mm thickness) was applied as lining material on top of the same Optibond FL adhesive system and was polymerized before the buildup of the proximal wall. The occlusal cavity was further filled after removal of the matrix band using the same composite, Herculite XRV Ultra, and the same layering technique as described for group A (Figure 1).

Evaluation

Seventy-two hours after polymerization, the restorations were finished and polished using flexible disks with different grain sizes (SofLex PopOn, 3M-ESPE, St Paul, MN, USA). Immediately after completion of the polishing procedure, impressions were made of each restoration with a polyvinylsiloxane impression material (President Light Body, Coltene, Alstätten, Switzerland). Subsequently, epoxy replicas were prepared for the computer-assisted quantitative marginal analysis in a scanning electron microscope (SEM; XL20, Philips, Eindhoven, The Netherlands) at 200× magnification. Continuous margins were assessed separately in D (% CM in D), PE (% CM in PE), and OE (% CM in OE), as percent of the total length of the analyzed margins. Total margin length represents the data from all of the margins of a Class II cavity, which is composed of OE, PE, and D. Additionally, enamel fractures (EF) parallel to the margins were documented in the SEM.

After storage at 37°C in water in the dark for two months, the restored teeth were loaded with repeated thermal and mechanical stress in a chewing

Table 2: *Percentage of Continuous Margins (%CM) Located in Dentin*[a]

Group	Description	Before Loading (mean ± SD)	After Loading (mean ±SD)
A	Control (Optibond FL + Herculite XRV Ultra)	55.7 ± 25.1 b,c	43.6 ± 28.2 b
B	Opotibond FL + Premise Flowable + Herculite XRV Ultra	48.3 ± 21.8 c	35.9 ± 28.6 b
C	Optibond FL + Herculite XRV Ultra + Herculite XRV Ultra	71.1 ± 27.8 a,b	54.4 ± 29.6 b
D	Optibond FL + Optibond FL + Herculite XRV Ultra	87.2 ± 19.4 a	79.8 ± 25.4 a

Abbreviation: SD, standard deviation.
[a] *Different letters indicate significant differences between groups of the same column ($p<0.05$). The lowest values of marginal continuity were obtained for groups A and B. Before loading, groups C and D were significantly superior compared to groups A and B but were not significantly different compared to each other. After loading, results showed no difference in marginal adaptation (MA) among groups A, B, and C, with group C being clearly superior.*

machine. Thermo-cycling was carried out in flushing water with temperature changing 600 times from 5°C to 50°C and back with a dwell time of two minutes at each temperature. The mechanical stress comprised 200,000 load cycles transferred to the center of the occlusal surface with a frequency of 1.7 Hz and a maximal load of 49 N. Epoxy replicas were prepared for the computer-assisted quantitative marginal analysis in a SEM, using the same parameters as were used before the loading procedure.

Statistical analysis was done using the Shapiro-Wilks test, and differences between groups were statistically evaluated using analysis of variance and the Duncan *post hoc* test at $p<0.05$. The difference between results related to loading conditions (before and after the fatigue test) was analyzed using the statistical method of the paired *t*-test. MA was the only dependent variable.

RESULTS

Only the means and standard deviations for the percentages of CM and EF are reported, as 'overfilled margins' and 'underfilled margins' did not exceed 5% in any group. The scores for the MA are displayed separately for PE, D, and OE in Tables 2 through 5.

Before loading, the MA in cervical D was superior ($p<0.05$) for groups C and D compared to groups A and B. In PE and OE, all groups showed the same behavior ($p>0.05$) and it was noticed that the % CM in PE was relatively low compared to the % CM in OE (Tables 2 through 5).

After two months of water storage and after thermal and mechanical stressing, the MA decreased considerably in all four groups at a statistically significant level. A paired-samples test showed significant differences ($p=0.000$) between both testing intervals before and after loading in all of the marginal segments tested (occlusal, proximal, and terminal).

Significant differences between groups were found for the criteria % CM in D and % CM in PE. In D, the superiority of the group D was clearly outlined ($p<0.05$) compared to all of the other three groups (Table 2). In PE, significant superiority was found only for group C compared to group A, with no statistically significant difference between the other liners. The % of enamel fractures (% EF) was high after loading, the application of the liners showing no statistically significant improvement of the MA (Table 5).

All restorations, both with and without lining material, presented discontinuous margins. The null hypothesis could be rejected, as statistically significant differences could be demonstrated between groups.

DISCUSSION

The present study was designed to test the classical presumption about the positive effect of flowable composite liners (Premise Flowable in this experi-

Table 3: *Percentage of Continuous Margins (%CM) in Proximal Enamel (PE) Before and After Loading*[a]

Group	Description	Before Loading, mean ± SD	After Loading, mean ± SD
A	Control (Optibond FL + Herculite XRV Ultra)	78.6 ± 3.5 a	30.3 ± 14.1 c,b
B	Opotibond FL + Premise Flow + Herculite XRV Ultra	79.2 ± 23.7 a	35.5 ± 23.5 b,a
C	Optibond FL + Herculite XRV Ultra + Herculite XRV Ultra	79.6 ± 16.4 a	48.6 ± 14.5 a
D	Optibond FL + Optibond FL + Herculite FL	71.1 ± 16.1 a	45.5 ± 9.4 b,a

Abbreviation: SD, standard deviation.
[a] *Different letters indicate significant differences between groups of the same column ($p<0.05$). The % of CM in PE was low before and after loading. The comparison between groups reveals a significant difference only for group C compared to group A and only after loading. No significant difference was found among the three different liners.*

Table 4: *Percentage of Continuous Margins (%CM) in Occlusal Enamel (OE) Before and After Loading*[a]

Group	Description	Before Loading, mean ± SD	After Loading, mean ± SD
A	Control (Optibond FL + Herculite XRV Ultra)	92 ± 13.7	84.5 ± 21.8
B	Optibond FL + Premise Flow + Herculite XRV Ultra	94 ± 10.4	87.3 ± 20.1
C	Optibond FL + Herculite XRV Ultra + Herculite XRV Ultra	98.5 ± 2	92.2 ± 6.7
D	Optibond FL + Optibond FL + Herculite XRV Ultra	97.3 ± 3.6	88.6 ± 8.3

Abbreviation: SD, standard deviation.
[a] *No significant difference in %CM in OE was found between groups either before or after loading.*

ment) and to gain a better understanding of the influence of the E-modulus on marginal sealing of composite restorations before and after artificial mechanical and thermal aging. For this purpose, the study was designed to include a group with a pre-cured layer of an extremely elastic material, such as the adhesive of the adhesive system Optibond FL, as well as a group with a thin lining (1-mm thickness) of the same restorative composite used for the filling (Herculite XRV Ultra). The significant difference between the E-modulus of the three different cavity linings could therefore add more to the knowledge of marginal sealing of composite restorations than could be gained by using only groups with different flowable composites as liners.

SEM is a widely used method with which to evaluate MA. Direct observation by SEM is difficult as a result of the vacuum procedure utilized during SEM, which causes cracks. By using a replica method, artificial gap formation can be avoided.[43] The specimens were polished after 72 hours and stored for two months before thermo-mechanical stressing in order to take into consideration the complex post-polymerization processes, such as ongoing contraction and stress relaxation by water absorption.[44,45]

While several other studies assessed the MA of restorative RC with and without a flowable liner through the dye penetration method, no marginal evaluation by SEM was done on a bonding product placed as liner (in this case the Adhesive of the Optibond FL adhesive system). Also unlike previous studies, our experiment was not designed to compare the effect of different flowable RC liners but rather to assess the influence of an E-modulus within a broad range (2-3 GPa and 9.4 GPa) (Table 1).

A group with the Herculite XRV Ultra used as a 1-mm pre-cured layer was introduced with the purpose of determining to which degree the improvement in MA is due to the pure layering procedure rather than to the properties of the lining materials.

Knowing the unpredictable effect on MA of a flowable composite placed as a liner, regardless of its E-modulus,[12,38,41,46] we preferred to use a flowable RC with rather optimized mechanical properties (high filler load), which could be beneficial to clinical use. Among other flowable RCs, the Premise Flowable has one of the lowest polymerization shrinkage rates, and the filler load is close to that of a restorative RC (72.5%).

The composite placement technique was chosen in order to minimize the polymerization shrinkage stress, especially at the cervical D wall. Therefore, a unique vertical layer was considered to have a minimal contact with the walls of the proximal box and a favorable ratio of unbonded to bonded surface.

The cavity configuration, the adhesive, and the restorative composite were kept constant, with the focus remaining on those parameters related to lining materials.

MA in Dentin

In dentin, the lowest values of marginal continuity were obtained for groups A (no liner) and B (Premise Flowable liner). The application of a thin liner (1

Table 5: *Percentage of Enamel Fractures (%EF) Before and After Loading*[a]

Group	Description	Before Loading, mean ± SD	After Loading, mean ± SD
A	Control (Optibond FL + Herculite XRV Ultra)	4.4 ± 4.8 a	20.6 ± 17.4 a
B	Optibond FL + Premise Flow + Herculite XRV Ultra	4.9 ± 7.7 a	38.8 ± 24.1 b
C	Optibond FL + Herculite XRV Ultra + Herculite XRV Ultra	9.5 ± 18.7 a	25.6 ± 16.3 a
D	Optibond FL + Optibond FL + Herculite XRV Ultra	10.7 ± 11.6 a	30.7 ± 14.8 a

[a] *Different letters indicate significant differences between groups of the same column ($p<0.05$). The %EF in proximal enamel (PE) was high after loading. The comparison between groups reveals no differences before loading. After loading, group B was significantly worse ($p=0.05$) compared to groups A, C, and D, which were not significantly different to each other. A high standard deviation is to be noted as well as a significance level of only $p = 0.05$.*

mm) of Herculite XRV Ultra led to significantly superior MA in dentin only before loading and only compared to the control group, but this finding was not significantly different compared to group D ($p>0.05$). This result is probably due to the benefits of placing a pre-cured layer, as previously described.[37,42] It is worth noting that, while not always statistically significant, the group with Herculite XRV Ultra lining showed generally better MA than did the control group. However, group D was significantly better than all of the groups after loading, indicating that layering is not the only factor contributing to the stress relief (Table 2).

Applying the flowable RC as a liner did not achieve better MA. Before loading, group B was significantly worse than group C. After loading, results showed no difference in MA by using the flowable liner compared to the results achieved by layering the composite (group C) or compared to the control group results (group A). Both before and after loading, group B was significantly worse than group D. A possible explanation, presented by other authors[12,38,41] as well, indicates that the benefits of the elasticity of the flowable liner were probably surpassed by its contraction.

After thermo-cycling, a significant improvement of MA in dentin was obtained only by placing a 1-mm-thick layer of Optibond FL on top of the same bonding product (Table 3). A clear positive effect on MA was observed in dentin before and after loading. This positive influence could be explained by the very low E-modulus of the Adhesive of the Optibond FL system, in the range of 1 to 3 MPa, as was predicted by some authors[20,33,40] and in accordance with other *in vitro* studies[18,36,37] showing a positive effect of thick layers of unfilled resins used as stress breaking liners under restorative composites. One FEA analysis[47] revealed an interesting finding in Class V cavities, in which only a very low E-modulus (2 GPa) was shown to be capable of absorbing tensions along the tooth restoration interface and thus preserving the cohesive and adhesive integrity of the tooth restoration complex.

MA in Enamel

In this study, the adaptation to PE was low and decreased significantly after loading. Some considerations are to be made regarding the evaluation of marginal integrity in PE, as follows:

- In all groups, the % of CM in PE after loading was low compared to OE (Tables 3 and 4). The most important cause is probably the lack of beveling PE, leading, therefore, to a weak adhesion prone to EF.[48] The comparison between groups reveals no differences in MA before loading as well as an almost significantly higher % EF for group B after loading ($p=0.05$), although at a high standard deviation. While the % CM in PE was generally very low, the only significant difference between groups was found after thermo-cycling in group C, which scored higher % CM compared only to group A. It should be noted that no significant difference was found among the three different liners. It would appear that in PE, the most influential on MA was the lack of beveling of the margin. Less important seems to be the type of liner, which was not placed directly on the PE margins. In this particular configuration, the Optibond FL liner seems to have only a local effect in its area of direct application, which is the cervical D margins.
- Contraction seems to contribute as well to the degradation of the marginal integrity. The paired-samples test showed significantly higher values for the % EF and significantly lower % CM in proximal cavities after thermo-cycling compared to before thermo-cycling. It is well documented[23] that areas pre-stressed by polymerization shrinkage show failure under occlusal load. Forces due to contraction created a residual strain within the tooth, leading, after loading, to the formation of cracks in the PE, a finding that is in accordance with those of the FEA analysis of Versluis and others.[27]
- Moreover, as a result of the cavity geometry, the axial enamel in a mesio-occluso-distal (MOD) cavity compensates less for deformation under load, compared to OE margins.[27] Under thermo-mechanical load, sound teeth distribute applied stresses more homogeneously compared to the structurally modified teeth, which show a complex biomechanical behavior due to the interruption of tissue continuity.[23,27,33] Therefore, the high percentage of noncontinuous margins in PE observed after loading could also result from a differential behavior, under load, of the PE walls compared to the OE margins when the continuity of a tooth is interrupted by a MOD cavity.

Therefore, the interaction between shrinkage stress distribution patterns[27,28] and biomechanical balance[23,27] of an occluso-proximal cavity under loading condition, together with strength of adhesion (bevel), creates a complex and differential behavior of proximal and occlusal tooth structures.

Shrinkage stress development is a dynamic, nonlinear process, depending on the complex inter-

action of many material-dependent, cavity-dependent, and restoration placement–dependent factors. Contraction stress in a tooth restoration complex reveals a distribution pattern with forces acting differently along the interface.[27] In this study, the highest percentage of noncontinuous margins was found in cervical D, which is in accordance with the stress distribution pattern depicted by FEA studies.[20,23,27] Polymerization stress maps showed shrinkage stress along the interface, with maximum stress accumulation at the cervical zone, in our case the dentin-composite interface, also known to be the most problematic area for adhesion.

The results of the present study confirm recent findings indicating that a thick adhesive layer with extremely low modulus could relieve stress at the interface, and they reaffirm the unpredictable stress-absorbing effect of flowable composites as liners. Research on properties of flowable composites also showed substantial differences in E-modulus and volumetric shrinkage, indicating flowable composites to be a very inhomogeneous group of materials.[12,38,41,46, 50-54] One study[38] situated the E-modulus in a wide range, with values between 6.5 GPa and 12.5 GPa for 12 different flowable composites. It seems that the performance of flowable composites as a stress buffer remains unpredictable, as polymerization kinetics tend to be material specific and because the optimization of the elasticity and shrinkage, with opposing effects, is difficult to achieve. In other words, despite their low E-modulus, the contraction stress produced by some flowable composites could be high because of their high volumetric shrinkage, which approaches 6% in some products.[12,38,41] Other studies[22,24,55-64] evaluating the influence of flowable composites placed as intermediate layers,[49] on marginal integrity show conflicting results as well.

Based on the present research, it seems that a liner with a very high elasticity applied in a thick layer could redistribute shrinkage stress and contribute to maintaining integrity along the tooth restoration interface, as indicated by previous research.[18,20,33,36,37,40]

Although the % CM after load in cervical D was higher in group D, the % EF in PE was not significantly lower compared to the other groups, indicating that enamel did not benefit from the same stress relief as did the interface. Our results correlate as well with those of other studies[16,18,50,51] reporting that a layer of low modulus composite can lead to redistribution of the forces and some strain relief along the interface but does not improve bond strengths and therefore does not reduce the overall negative influence of curing contraction. In the present study, the liner was applied only on the cervical margin and pulpal wall, not on the buccal and lingual proximal walls. It seems that the liner of Optibond FL was efficient in reducing and redistributing stress at the interface predominantly in the area of its application, where the marginal seal is also known to be the most problematic because of the concentration of stress, which is not uniformly distributed along the cavity walls.[20,21,23] This area is also marked by high variation in the bond strength along the bonded surface.[37,52] In return, no benefit of the stress-relieving effect was found on the enamel, where the bevel seems to play a more important role in assuring a good bonding stability after the fatigue test.

Any attempt to predict the contraction stress development of a material during polymerization based on E-modulus or polymerization shrinkage value alone seems to be misleading.[12,14,27,38,53] This hypothesis is confirmed as well by the present study, in which the flowable composite used as liner did not allow for the expected stress relief, while the performance of the Optibond FL liner was more heavily influenced by its very low E-modulus and was less affected by the high shrinkage. Product-specific properties such as flow capacity and pregel and postgel shrinkage render the stress-relieving effect unpredictable.

However, using only a composition-based approach to relieve shrinkage stress may still be too simplistic. Ensuring optimal dentin bonding and using incremental layers of composite in appropriate configurations are factors within the control range of the practitioner, which could be of importance in assuring a better marginal seal.[54] Accordingly, marginal quality could be optimized by factors such as guidance of the shrinkage vectors, reducing the ratio of bonded to free unbonded restoration surfaces, and minimizing the mass of *in situ*-cured composite.

CONCLUSION

For the materials used in this study, no improvement of MA was found when the flowable composite was used as a liner, both before and after thermomechanical loading. The application of a layer of the same restorative composite used for the restoration with the same thickness as the flowable composite (1 mm) had a positive effect before loading, probably as a result of the benefit of placing a precured layer on the bottom of the cavity before stratification of the

restoration. After thermo-mechanical loading, a significant improvement of MA in D was obtained only by placing a 1-mm-thick layer of the Adhesive of the Optibond FL system on top of the same product used as adhesive. This positive effect may be explained by its very low E-modulus, in the range of 1 to 3 MPa.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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Sealing Properties of Three Luting Agents Used for Complete Cast Crowns: A Bacterial Leakage Study

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Clinical Relevance

The results of this study demonstrated that Rely X Luting Plus, a resin-modified glass ionomer cement, exhibited the most favorable sealing properties against bacteria during a 60-day observation period in comparison to a self-etching resin cement Maxcem Elite and a conventional glass ionomer cement Ketac Cem. The bacterial leakage model used for this experiment proved to be a useful method to determine the sealing properties of luting agents used for cementation of cast restorations.

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SUMMARY

Objective: **To assess the sealing properties of three different luting materials used for cementation of full cast crowns on extracted human premolars.**

Methods: **Thirty noncarious human premolars were prepared in a standardized fashion for full cast crown restorations. All margins were placed in dentin. After impressions of the preparations, stone dies were fabricated on which copings were waxed, which were cast in type III alloy using standardized laboratory methods. Teeth were randomly assigned to three groups of 10 samples each (n=10), for which the following cements were used: 1) a resin-modified glass ionomer cement, Rely X Luting Plus (3M ESPE, St Paul, MN, USA); 2) a self-adhesive resin cement, Maxcem Elite (Kerr Corporation, Orange, CA, USA); and 3) a glass ionomer cement, Ketac Cem (3M ESPE), the latter used as control. After cementation the samples were allowed to bench-set for 10**

minutes, stored in water at 37°C, subjected to thermal cycling (2000×, between 5°C and 55°C, dwell time 35 seconds), and then stored in sterile phosphate buffer for seven days at 37°C. Subsequently, the occlusal surface was carefully reduced until the dentin was exposed. Finishing on wet sand paper removed the gold flash caused by grinding. After sterilization, the specimens were subjected to bacterial microleakage in a dual chamber apparatus for 60 days. Bacterial leakage was checked daily. Data were analyzed using the Kaplan-Meier survival test. Significant pairwise differences were analyzed using the log-rank test followed by Fisher exact test at a $p<0.05$ level of significance.

Results: **Rely X Luting Plus showed the lowest microleakage scores, which statistically differed significantly from Maxcem Elite and Ketac Cem ($p<0.05$).**

Conclusions: **Rely X Luting Plus cement displayed significantly lower microleakage scores than a self-adhesive resin-based and conventional glass ionomer cement.**

INTRODUCTION

Inadequate marginal adaptation of complete cast metal crowns, porcelain fused to metal restorations, ceramic crowns, and inlays/onlays is one of the most important shortcomings that can affect the wash-out of a luting agent, resulting in bacterial penetration between restoration and dentin, thus compromising the durability of a restoration.[1] As demonstrated in a previous study,[2] the choice of an appropriate cementing agent may be a determining factor concerning the degree of microleakage. Unfortunately, contemporary luting agents are not totally effective in preventing marginal bacterial microleakage, which may lead to cement dissolution causing secondary decay, possible pulpal inflammation, and hypersensitivity[3] as well as loss of retention.[4] Currently, glass ionomer, resin, or resin-modified glass ionomer (RMGI) cements are routinely being used by the dental profession. Conventional glass ionomer luting cements bond to dentin by ionic bonds with hydroxyapatite, while resin-based cements bond to dentin through micromechanical interlocking. RMGIs are hybrid formulas and composed of fluoro aluminosilicate glasses, polyacrylic acid, and resin composites and contain photo or chemical initiators and methacrylate monomers.[5] RMGIs bond to dentin through a combined ionic bond between polyacrylic acid and hydroxyapatite and a micromechanical interlocking with collagen and dentinal tubules. Previous studies have shown that resin-based luting materials possess superior mechanical properties over conventional glass-ionomer cements,[6,7] which may be a factor in sealing capacity as well as resistance to displacement.[4] To date, limited information on microleakage at the interface between tooth structure and full cast crowns luted with different types of self-adhesive cements is available.[2,8,9]

The purpose of the present *ex vivo* study was to compare the sealing property of three different luting materials (a resin-modified glass ionomer, a self-etching resin-based cement, and a conventional glass ionomer cement) in complete cast gold crowns by means of a bacterial leakage test. The null hypothesis was that there is no significant difference with respect to marginal leakage among the three tested materials.

MATERIALS AND METHODS

Specimen Preparation

Thirty intact permanent human maxillary and mandibular premolars with fully developed roots, extracted for orthodontic reasons, and showing comparable crown length and size were used. The study received exemption from the Ethics Committee of the Argentine Dental Society. All teeth were prepared by a single operator as described by Pameijer and others.[10] Briefly, the teeth were prepared freehand using a No. 4138 high-speed diamond chamber bur (KG Sorensen, Ind e Com Ltda, SP, Brazil) using copious oil-free water-cooling. The occlusal surface was reduced perpendicular to the long axis, penetrating the dentin, while a medium chamber finish line was established circumferentially in dentin, approximately 0.5 mm short of the cementoenamel junction. All preparations had a height of ±5 mm with a total angle of convergence of approximately 12°, while all line angles were rounded. A new bur was used for every five preparations. From each individual tooth, preliminary impressions were made with Putty Soft, type I body vinyl polysiloxane (President, Coltene/Whaledent, Mahwah, NJ, USA) using individual custom-made acrylic trays. Final impressions were made with light body vinyl polysiloxane type III (President), and dies were poured in type IV extra-hard stone (Densell Mix, GDK, Densell Dental Technology, Buenos Aires, Argentina) following the manufacturers' instructions. The stone dies were trimmed and one coat of die relief (Renfert Pico-Fit, Renfert GmbH, Hilzingen, Germany) was applied,

taking care to stay ±1.5 mm short of the margins of the preparation. Subsequently, wax patterns (Whip Mix Corporation, Louisville, KY, USA) were fabricated to model copings ± 0.5 mm thick with flat occlusal surfaces. Using standard laboratory techniques, the wax patterns were invested in phosphate-bonded investment (Ivoclar Vivadent, Amherst, NY, USA) and cast in type III gold (AlbaDent, Fairfield, CA, USA). The castings were divested, and checked for accuracy under a stereomicroscope (Carl Zeiss, Oberkochen, Germany) at 20× magnification. The internal fitting surface was air abraded with 50 μm aluminum oxide. At all times, other than when they were worked on, the teeth were stored in a sterile phosphate buffer (SPB) solution. After checking for fit and retention, each tooth and matching coping were numbered, individually bagged, and autoclaved. After sterilization, 10 samples (n=10) were randomly assigned to each of three treatment groups.

Cementation

The luting agents used in this study are described in Table 1. In group 1, Rely X Luting Plus (3M ESPE, St Paul, MN, USA), a resin-modified glass ionomer cement, was used. Group 2 was cemented with Maxcem Elite, a self-adhesive resin cement (Kerr Corporation, Orange, CA, USA), and group 3 used Ketac Cem (3M ESPE), a conventional acid-base reaction glass ionomer cement. Prior to cementation the teeth were thoroughly rinsed with sterile oil-free water-spray and dried with filtered compressed air. Care was taken to avoid excessive drying. Cementation was performed under aseptic conditions by a single operator and strictly according to the manufacturers' instructions. The castings were filled with the cement mixture and immediately seated, following which they were kept under finger pressure for 2 minutes, Then, for the samples of group 1 and 2, an oxygen inhibition gel (DeOX; Ultradent Products Inc, South Jordan, UT, USA) was applied to the margins and left in place for 4 minutes, after which excess cement was removed with a scaler. After bench setting for an additional 10 minutes, the marginal fit was checked by visual examination and a probe, and the samples stored for 24 hours in SPB at 37°C. They were subsequently subjected to 2000 thermal cycles in water baths at 5°C and 55°C with a dwell time of 35 seconds and once more stored in

Table 1: *Description of Cementing Agents Used in This Study*

Material/Lot	Material Type	Manufacturer	Composition
Rely X Luting Plus			
Lot N386694	Paste/paste, dual syringe direct dispensing through clicker dispensing system	3M ESPE, St Paul, MN, USA	Paste A: Radiopaque fluoro aluminosilicate (FAS glass) opacifying agent, HEMA, water, proprietary reducing agent
			Paste B: nonreactive zirconia silica filler methacrylated polycarboxylic acid, HEMA, Bis-GMA, water, potassium per sulphate
Maxcem Elite			
Lot 3493741	Paste/paste, dual syringe direct dispensing through a mixing tip	Kerr Corporation, Orange, CA, USA	Glycerol phosphate dimethacrylate co-monomers (mono-, di-, and tri-), functional methacrylate monomers, water, acetone, ethanol, barium, glass-fumed silica, sodium hexafluorosilicate (complete formulation not available)
Ketac Cem			
Lot 460448	Powder/liquid	3M ESPE, St Paul, MN, USA	Powder: glass powder, polycarboxylic acid, pigments
			Liquid: water, tartaric acid, conservation agents

Abbreviations: Bis-GMA, bisphenol A glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate.

SPB for seven days at 37°C. In preparation for the bacterial leakage test, the occlusal surfaces of the gold copings were reduced until the dentin was exposed using a fine high-speed diamond bur with light pressure and copious water-cooling. This was followed by sanding the occlusal surface on wet garnet paper of 400 and 600 grit (3M ESPE) to remove gold flash that may have been caused by the diamond bur. Subsequently, the entire root surface 1 mm below the margin of the copings was coated with two layers of nail polish and the samples subjected to bacterial microleakage.

Bacterial Leakage Setup

For this experiment, a slight modification of the dual chamber test apparatus described by Imura and others[11] was used. The tip of 1.5-mL Eppendorf plastic tubes (upper chamber) was cut and the sample was pushed (crown first) through the opening until approximately one half of the crown protruded through the end of the tube. The junction between the sample and the tube was sealed with sticky wax, making sure the crown margin was situated in the upper chamber. The tubes were put into glass vials (lower chamber) containing 10 mL of sterile trypticase soy broth (TSB; Difco Laboratory, Detroit, MI, USA) in such a way that the occlusal dentin/cement interface was submerged in the broth of the lower chamber (Figure 1). The junction between the tube and the glass vial was then sealed with two layers of cyanoacrylate (Ciano, Anaerobicos IWT, Buenos Aires, Argentina) and finally covered with sticky wax. The entire test apparatus was sterilized with ethylene oxide gas for 12 hours and then incubated at 37°C for 72 hours to verify sterility. If the TSB broth showed turbidity, the test set-up was discarded and replaced by a new one and the process repeated.

Bacterial Leakage Test

This phase of the study was carried out in a microbiology laboratory by a microbiologist under strict sterile conditions. The upper chamber was filled with 1 mL of TSB containing 24-hour growth of *Enterococcus faecalis* ATCC 29212 (10^8 colony-forming units/mL). The inoculated apparatus was incubated for 60 days at 37°C. The upper chamber was reinoculated every five days with fresh cultures of the microorganism. The TSB broth in the lower chamber was checked daily for turbidity, which, when observed, was an indication that bacterial leakage had occurred along the crown/cement or cement/dentin interface. Once turbidity was detected, the day of the observation was recorded. Samples from both chambers were then incubated on blood agar plates to check bacterial viability by morphologic observation and Gram staining. The number of teeth demonstrating bacterial leakage and the days on which leakage occurred were recorded for each group.

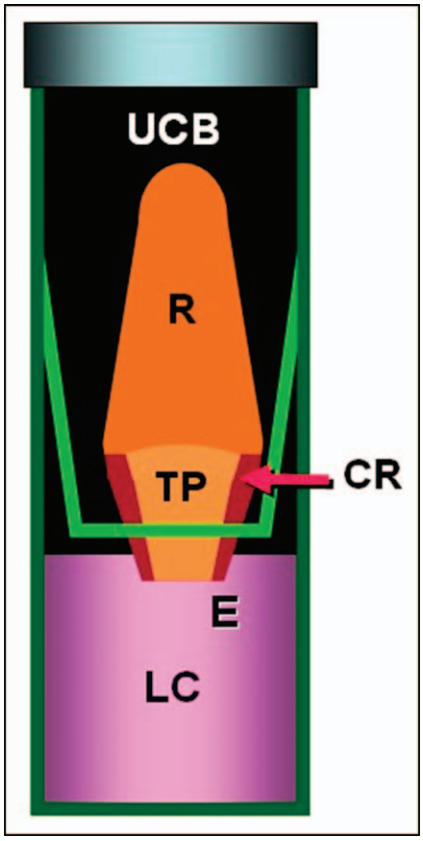

Figure 1. *Schematic set-up of the bacterial leakage system. CR, cast crown; E, exit for bacteria; R, root; TP, tooth preparation; TSB, lower chamber containing TSB broth; UCB, upper chamber conaining TSB broth with* E faecalis.

Statistical Analysis

The length of time until leakage was detected was compared among the three groups using the Kaplan-Meier survival analysis. Significant pairwise differences were analyzed using the log-rank test followed by Fisher exact test. The selected level of statistical significance was $p<0.05$. The presence of *E faecalis* was also checked.

RESULTS

Before the bacterial leakage test, two samples in group 1 and one in group 2 showed evidence of contamination. They were sterilized again according to the procedure described above. The results for the

Table 2: *Samples Showing Turbidity Per 20-Day Interval*

Group/Material	n	Experimental Period, d			
		1-20	21-40	41-60	No Leakage
1. RelyX Luting Plus	10	0	0	1	9 (90.0%)
2. Maxcem Elite	10	0	2	3	5 (50.0%)
3. Ketac Cem	10	0	3	4	3 (30.0%)

three groups are shown in Table 2. In group 1, turbidity did not occur until 55 days (one sample), whereas no leakage was observed in nine samples (90.0%). In group 2, leakage occurred at 38, 40, 42, 43, and 50 days (one sample each). In this group, five samples (50.0%) did not show leakage. In group 3, turbidity was seen at 22 days (one sample), 30 days (one sample), 33 days (one sample), 43 days (two samples), 44 days (one sample), and 47 days (one sample), whereas 3 samples (30.0%) did not show leakage. Fisher exact test revealed that the mean bacterial leakage values for Rely X Luting Plus were significantly lower than those of Maxcem Elite and Ketac Cem ($p<0.05$), while no significant differences were found between Maxcem Elite and Ketac Cem ($p>0.05$). The median survival time for Ketac Cem (ie, absence of bacterial leakage) was 60 days (with a 95% confidence interval of 45.8-74.2), while for Rely X Luting Plus and Maxcem Elite it could not be determined as it was greater than 60 days, the time interval the experiment lasted. Bacteriologic testing of the contents of the lower chamber with the samples that demonstrated leakage revealed viable *E faecalis*.

DISCUSSION

Marginal fit of full cast restorations has always been of concern to clinicians since poor adaptation is related to microleakage.[8] The chemical composition and the type of luting material used for cementing full cast metallic crowns has a bearing on fluid and bacterial microleakage.[2,12,13] Bacterial leakage at the crown margins and between the luting material and dentin is an important factor involved in secondary decay as well as pulpal and/or gingival inflammation.[3,14,15] The use of conventional dyes or stains has provided considerable information about this issue.[8,13,16,17] However, it seems unlikely that testing agents in a molecular state would be totally suitable to provide information on microleakage of particulate matter or aggregates of macromolecules such as large enzymes, bacteria, and/or bacterial by-products.[18] In the present study, we used an *ex vivo* method to analyze the penetration of bacteria at the margins of cast gold crowns luted with different types of cements. Controversy exists in the literature whether or not *ex vivo* and *in vivo* microleakage testing correlate and if results from *ex vivo* experiments can be correlated with the clinical situation.[19] In clinical practice, other factors such as material biocompatibility, ease of use, and specific requirements for each individual case (eg, height of residual tooth structure, preparation angle, and location of the preparation margins) may influence the choice of cement to be used.[20]

E faecalis was chosen because it exists in the normal oral flora in humans and is frequently found in mixed infections with other aerobes and facultative anaerobes.[21] For evaluation, a qualitative approach (presence or absence of turbidity) during a 60-day period was used. Although this observation period was similar to what has been reported in the endodontic literature,[22,23] it should be emphasized that 60 days is a short period considering much higher expectations of longevity of a restoration. In spite of the fact that the experimental conditions used in this study did not include a broader range of factors that affect marginal leakage of full cast crowns in a clinical environment,[24] the test nevertheless allows for a comparison of different materials under strictly controlled conditions.

It should be noted that our test was focused only on crown margins placed on dentin. As previously has been demonstrated,[3,25] the degree of microleakage is higher on dentin than on enamel, therefore a worst-case scenario was tested. Another untested variable in this study was the cement thickness between the restoration and dentin. As per

protocol, die-relief spacer was used in order to decrease the seating discrepancies of the castings,[26,27] albeit it has been previously shown that the marginal seal is not negatively influenced by the cement thickness.[25] However, this variable needs further research. Occlusal load stress, which normally occurs under *in vivo* conditions, is another variable that was not tested in the current study and should be taken into consideration when marginal leakage of cemented crowns is tested. Prior to being subjected to bacterial microleakage, all samples had undergone thermal cycling, which is a method to simulate the long-term stresses to which a restoration is exposed under clinical conditions.[13,16,28] However, it has been reported that higher microleakage values were registered when thermocycling was followed by load-cycling[29] an issue that also needs to be investigated more extensively.

The results demonstrated that Rely X Luting Plus had significantly lower leakage scores than Maxcem Elite and Ketac Cem, suggesting that Rely X Luting Plus provided an acceptable marginal seal for up to 60 days. These results are in agreement with Pameijer and others[10] who reported that gold copings cemented with Rely X Luting Plus demonstrated good sealing properties. Furthermore, Rossetti and others[17] reported that Rely X Luting cement (3M ESPE) showed significantly lower leakage than a resin-based luting agent or a zinc phosphate cement. Rely X Luting cement and Rely X Luting Plus have chemically similar formulas and differ only in that the first is a powder/liquid formulation, while the second comes as a paste/paste and is delivered by a clicker dispenser. This chemical similarity let led us to suppose that the physicochemical interactions with the tooth surface are the same for both formulations.

When compared to Rely X Luting Plus, the bacterial leakage rates were significantly more pronounced for Maxcem Elite and Ketac Cem. According to reports in the literature, bacterial penetration indicates that potential gaps are present at the tooth-cement interface that may lead to secondary decay, pulpal inflammation, and periodontal disease.[3,14,15] Maxcem Elite is a contemporary self-adhesive resin cement with 69% filler content by weight. Previous studies[30,31] have shown adhesive failure between materials of resinous nature and dentin. As demonstrated by Frassetto and others,[32] Maxcem Elite exhibited high volumetric contraction stresses and greater shrinkage values than other tested resin cements. In the present study, the leakage patterns registered for Maxcem Elite did not show significant differences with the conventional glass ionomer cement Ketac Cem. Similar to Maxcem Elite, high volumetric contraction stresses and low-bond strength capacity to dentin have also been reported for Ketac Cem.[33] Indeed, the higher bacterial leakage scores by both materials may be explained because of high contraction stresses that may compromise the bonding leading to an increase in microleakage.

The lower sealing ability of Ketac Cem is in agreement with previous observations of Pameijer and others[10] and a study of White and others[12] who reported that only 29% of samples cemented with Ketac Cem revealed zero leakage. Furthermore, Piemjay and others[34] reported that the acid-base cement Ketac Cem exhibited greater microleakage when tested with a 0.5% basic fuchsin dye for 24 hours. The bacterial leakage data reported here also corroborates favorably with a pilot leakage study published previously[35] using methylene blue dye in which Ketac Cem demonstrated high leakage patterns. On the contrary, Tung and Coleman[36] and Coleman and others,[37] reported that, among other luting agents, Ketac Cem was effective in preventing leakage of detectable molecular concentrations of lipopolysaccharide and dextran. These contradicting data from one laboratory to another may be the consequence of using different experimental conditions as well as by variables such as tooth preparation, treatment of the dentin surfaces, and application of the luting material.

In summary, the current study demonstrated that Rely X Luting Plus showed significant differences with Maxcem Elite and Ketac Cem in its ability to better seal the interface crown/cement and cement/dentin against bacterial microleakage, while Maxcem Elite and Ketac Cem showed comparable microleakage results. Therefore, the null hypothesis must be partially rejected.

CONCLUSIONS

Within the limitations of this study, the following conclusions can be drawn:

- The method described here appeared to be a suitable method for testing the sealing ability of luting materials against bacterial microleakage when used for cementation of cast crown restorations.
- Cast crowns cemented with Rely X Luting Plus provided an acceptable marginal seal up to 60 days and showed significantly lower bacterial leakage when compared to Maxcem Elite and Ketac Cem.

• The stability and the sealing properties of the tested materials at the restoration/tooth interface after more than 60 days need to be investigated further.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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Clinical Effects of Exposure to Coffee During At-home Vital Bleaching

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Clinical Relevance

Coffee consumption during bleaching did not affect the effectiveness of dental bleaching.

SUMMARY

The purpose of the present study was to evaluate whether exposure to coffee during bleaching treatment with 16% carbamide peroxide (CP) affects the degree of whitening and tooth sensitivity. Forty patients with central incisors darker than A2 were selected. Participants who did not drink coffee were assigned to the control group (CG), while participants who drink coffee at least twice a day were assigned to the experimental group (EG). For CG, foods with dyes were restricted. For EG there was no restriction on food and patients were asked to make coffee rinses for 30 seconds, four times daily. For both groups 16% CP was used for a period of three hours daily for three weeks. Shade evaluation was assessed visually by Vita classical shade guide and by the Easyshade spectrophotometer at baseline, during bleaching (first, second, and third weeks), and post-bleaching (one week and one month). Patients recorded their sensitivity perceptions by means of the numerical rating scale and 0-10 visual analog scales. Variation in shade guide units and the two colors (ΔE) were evaluated by two-way analysis of variance and Tukey tests (α=0.05). Absolute risk of tooth sensitivity and intensity of tooth sensitivity was evaluated by Fisher exact and Mann-Whitney tests (α=0.05). Effective bleaching was observed for both groups after three weeks, without statistical difference. No difference in terms of risk of tooth sensitivity and intensity of tooth sensitivity was detected between groups. Approximately 57% of the participants experienced tooth sensitivity, which was recorded mainly as "mild." Exposure to coffee during bleaching treatment does not seem to affect the degree of bleaching and tooth sensitivity.

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INTRODUCTION

Dental bleaching is one of the most sought-after clinical procedures at present, because tooth color is considered the most important factor with regard to dental esthetics.[1] It is a safe, conservative, and

effective procedure,[2,3] generally performed with gels containing hydrogen peroxide or carbamide peroxide (CP) in different concentrations.[4,5] Treatment is performed in-office with or without light activation, at home with the use of trays, or by means of a combination of these two modalities.[4,6,7]

In spite of the effectiveness of the tooth whitening technique, this procedure may cause changes, such as an increase in the permeability of dental tissues[8] and demineralization[9] of the tooth enamel surface[10,11]. Therefore, while bleaching treatment is being performed, it is common for professionals to ask their patients to avoid the ingestion of foods and drinks rich in coloring agents, such as coffee, red sauces, red wine, chocolate, tea, beetroot, and acai. Among these coloring agents, coffee is outstanding, as it is a coloring beverage frequently consumed in Western countries.[12]

It has been demonstrated that coffee is capable of causing tooth staining[13] because it has a dark color and an acid pH.[14,15] This would cause an increase in permeability and penetration into the tooth structure during bleaching and may cause interference in terms of the final results of bleaching.[3,16,17] Laboratory studies[13,18] have indicated that teeth submitted to dental bleaching and those exposed to coloring agents in the diet indeed have greater potential for staining. This leads to lower longevity/stability of the whitening effect[19] and induces clinicians to impose dietary restrictions during dental bleaching procedures.

Nevertheless, this is a controversial topic, since other *in vitro* studies[16,20,21] have concluded that the ingestion of colored foods during dental bleaching and over the course of time does not interfere with the results obtained with dental bleaching. Although there are studies[22,23,24] that have attempted to correlate the effectiveness and longevity of dental bleaching with the frequency of ingesting foods rich in coloring matter, a literature search revealed no clinical studies that correlated the effectiveness and longevity of dental bleaching with the consumption of colored foods and beverages during dental bleaching. Therefore, the aim of this clinical study was to evaluate whether the effectiveness of bleaching is diminished by exposure to coffee during home bleaching treatment with 16% CP and whether this treatment affects tooth sensitivity.

MATERIALS AND METHODS

This clinical investigation was approved (Protocol No. 17854/10) by the local ethics committee. Forty subjects who were willing to sign the consent form before the study began were enrolled according to the inclusion and exclusion criteria. All subjects received dental screening and dental prophylaxis two weeks before the bleaching protocol began.

Inclusion and Exclusion Criteria

Patients included in this clinical trial were at least 18 years old and had good general and oral health. Each subject had at least one central incisor with shade A2 or darker, assessed by comparison with a value-oriented shade guide (Vita Lumin, Vita Zahnfabrik, Bad Säckingen, Germany) and spectrophotometer (Easyshade, Vita Zahnfabrik). Patients were excluded from the study if they had undergone previous tooth-whitening procedures, had anterior teeth with restorations on the labial surfaces, had veneers or full crowns, were pregnant or lactating women, were smokers, had gingival recession on anterior teeth, had spontaneous tooth pain, had endodontically treated anterior teeth, had fluorosis or severe internal tooth discoloration, had teeth with noncarious cervical lesions, were under orthodontic treatment, or had bruxism habits.

Experimental Groups

Patients who met the inclusion criteria were asked about their daily coffee consumption. Those who did not drink black coffee were placed in the control group and instructed to not consume colored foods and drinks (coffee, tomato sauce, ketchup, mustard, beets, carrots, chocolate, black tea, acai, or artificial and naturally dye-colored drinks [such as Coca-Cola® and orange and grape Fanta], green tea, jelly, snacks, soy sauce, candies and chewing gum with dyes, grapes, berries, and red wine) one week before treatment began and during the entire period of bleaching therapy.

The participants who reported that they drank black coffee at least twice a day every day were placed in the experimental group. No dietary restrictions were placed on participants in the experimental group. Apart from their daily coffee intake (two to three cups daily), these patients were instructed to make mouth rinses with instant black coffee for 30 seconds (Nescafé® Tradição, Nestlé, Araras, São Paulo, Brazil), four times a day. For this procedure, participants received 8 mg of coffee to be dissolved in 50 mL of warm water (equivalent to one teaspoonful of instant coffee powder in a small coffee cup full of water), in accordance with the instructions for making instant black coffee. They were instructed to perform the first rinse immediately

after removing the bleaching tray. During the day, three more rinses had to be performed, with an interval of four hours between rinses. Participants were instructed to wait at least 15 minutes after coffee rinses before rinsing the mouth with clean water, brushing their teeth, or eating.

The aim of this procedure was to increase the exposure of bleached teeth to black coffee. As a measure of adherence to the experimental protocol, participants were given a diary in which they were asked to take note of the number of coffee rinses performed daily. They were emphatically instructed about the importance of the procedure and the importance of reporting any time they forgot or were unable to perform the coffee rinses.

Bleaching Procedure

Alginate impressions were made of each subject's maxillary and mandibular arch, and these were filled with dental stone. To produce study models, no block-out material was applied to the labial surfaces of teeth.[25-27] A 0.9-mm soft vinyl material, provided by the manufacturer, was used to fabricate the custom-fitted tray that would hold the whitening gel. The excess material from the labial and lingual surfaces was trimmed to 1 mm from the gingival junction.

The tray and 16% CP gel (Whiteness Perfect, FGM Dental Products, Joinville, Brazil) were delivered to each subject, with verbal instructions for use. All subjects were instructed to wear the tray containing the bleaching agent for at least three hours a day for a period of three weeks. After the daily three-hour period they were instructed to remove the tray, wash it with water, and brush their teeth as usual. At this time, participants in the experimental group were instructed to perform the first daily coffee rinse.

With regard to oral hygiene, all participants were instructed to brush their teeth regularly and were asked to not use whitening toothpaste and mouthwash containing peroxides.

Shade Evaluation

The shade evaluation was performed with the use of subjective and objective evaluation methods. For the subjective evaluation, the 16 tabs of the shade guide (Vita Classic, Vita Zahnfabrik) were arranged from highest (B1) to the lowest (C4) value. Although this scale is not linear in the truest sense, for the purpose of analysis, the changes were treated as though they represented a continuous and approximately linear ranking.[4] Two calibrated evaluators (M.R. and S.K.) with agreement of at least 85% determined by weighted kappa statistics recorded the shade of each subject's teeth at baseline, during treatment (after the first, second, and third weeks of undergoing bleaching treatment), and one week and one month after the end of the bleaching protocol.

The area of interest for measurement of tooth color matching was the middle third of the facial surface of the anterior central incisors, according to the American Dental Association guidelines. Shade changes were calculated from the beginning of the active phase through to the individual recall times by calculating the change in the number of shade guide units (ΔSGU), which occurred toward the lighter end of the value-oriented list of shade tabs. In the event of disagreements between the examiners during shade evaluation, a consensus was reached.

An objective shade evaluation was also performed with a digital spectrophotometer (Vita Easyshade, Vita Zahnfabrik) right after the subjective shade evaluation. Before the spectrophotometer measurement, an impression of the maxillary arch was taken with dense silicone paste (Coltoflax e Perfil Cub, Vigodent, Rio de Janeiro, Brazil). The impression was extended to the maxillary canine and served as a standard color measurement guide for the spectrophotometer. For each dental component to be evaluated, a window was created on the labial surface of the molded silicone guide using a metal device with a radius of 6 mm and well-formed borders.

The shade was determined using the parameters of the Easyshade device on which the following values were indicated: L*, a*, and b*, where L* represents the value from 0 (black) to 100 (white) and a* and b* represent the shade, where a* is the measurement along the red-green axis and b* is the measurement along the yellow-blue axis. The color comparison before and after treatment is given by the differences between the two colors (ΔE), which is calculated using the formula[28] $\Delta E = [(\Delta L^*)2 + (\Delta a^*)2 + (\Delta b^*)2]^{1/2}$. The classical Vita shade detected by the spectrophotometer was also recorded.

Tooth Sensitivity Evaluation

Subjects were asked to keep a daily record of whether they experienced sensitivity, using a five-point verbal numerical rating scale (NRS)[29,30] and a visual analog scale (VAS).[4,31,32] For the NRS scale, participants were instructed to choose one of the following scores to represent the intensity of the

tooth sensitivity they felt every day: 0 = none, 1 = mild, 2 = moderate, 3 = considerable, and 4 = severe. For the VAS scale, the participants were instructed to place a line perpendicular to the 10-mm–long line with zero at one end indicating "no sensitivity" and at the 10-mm end indicating "unbearable sensitivity."

To compare the intensity of tooth sensitivity between the groups, the median of the intensity of tooth sensitivity experienced by each patient was calculated throughout the period of bleaching therapy. The overall percentage of patients with tooth sensitivity and the total number of days on which patients experienced tooth sensitivity were also evaluated.

Statistical Analysis

A pilot study was conducted and it was shown that use of the bleaching protocol described in this study would lead to a ΔE value of 4.0 ± 1.0 after three weeks of bleaching. In order to have an 80% chance of detecting significance at the level of 5%, considering an increase in the primary outcome measure from "4" in the control group to "5" in the experimental group, a minimum of 16 participants would be required in each group. In order to accomplish this, considering follow-up losses, 20 participants were selected for each study group.

Data from the 40 patients were used in this study, according to the intention-to-treat analysis.[33] The ΔSGU data obtained from subjective and objective evaluation and the ΔE data were submitted to a two-way repeated-measures analysis of variance (ANOVA) (groups vs assessment time), with the assessment time being the repeated factor (α=0.05). After this, a *post hoc* analysis (Tukey test, α=0.05) was used for pairwise comparisons. Comparison of the absolute risk of sensitivity was made using the Fisher exact test (α=0.05) and comparison of the intensity of tooth sensitivity between groups was made using the Mann-Whitney test (α=0.05) for both pain scales.

RESULTS

A total of 163 participants in the age range of 18 to 40 years were evaluated to select 40 participants that met the inclusion criteria (Figure 1). The mean age (years) of the participants in this study was similar between the groups (control: 22.5 ± 4.8 years; experimental: 23.7 ± 5.8 years). Fifty percent of the participants were women, 11 being in the control group and nine in the experimental group.

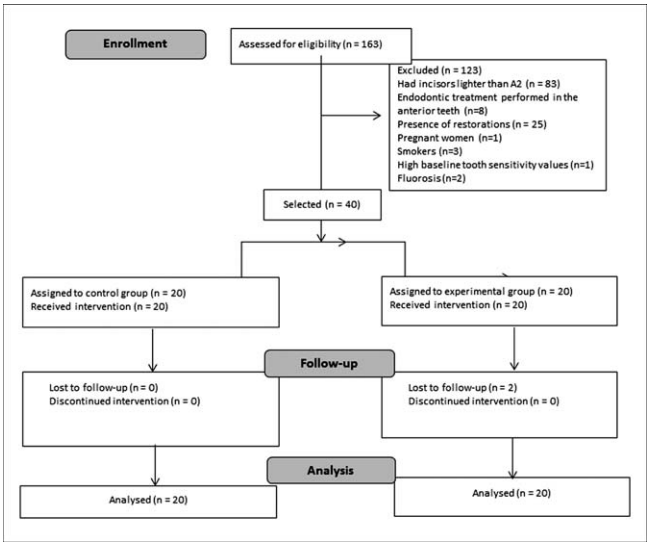

Figure 1. *Flow diagram of the clinical trial, including detailed information on the excluded participants.*

The mean baseline color in the shade guide units was 5.6 ± 1.5 for the control group and 6.3 ± 2.0 for the experimental group.

All participants attended the recall visits during the bleaching protocol. Two participants from the experimental group did not attend the one-week and one-month postbleaching visit. For these participants, the data obtained in the earlier recall visit were attributed, for statistical purposes, according to the recommendation of the intent-to-treat analysis.[33]

Each participant in the experimental group performed a total of 84 coffee rinses, with the exception of five patients who did not perform the rinsing once, two who did not perform it three times, and two who did not adhere to the protocol four times throughout the study.

Shade Evaluation

The two-way ANOVA for the subjective and objective evaluation of ΔSGU values showed that the cross-product interaction between groups vs assessment time (p=0.962 and 0.964, respectively) and the main factor group (p=0.109 and 0.339, respectively) was not statistically significant. Only the main factor assessment time (p<0.001 and p=0.045, respectively) was significant (ie, bleaching efficacy). A higher degree of bleaching was obtained after the third week of treatment, and this was statistically similar to the shade measured at one week and one month postbleaching (Table 1).

Two-way ANOVA for the ΔE values showed that the cross-product interaction group vs assessment

Table 1: *Means and Standard Deviations Between Assessment Points for the Two Treatments in Shade Guide Units (ΔSGU) Assessed by Means of Subjective and Objective Evaluation*[a]

Assessment Time Intervals	Subjective Evaluation		Objective Evaluation	
	Control	Experimental	Control	Experimental
Baseline vs 1 wk	3.0 ± 0.9 A	3.3 ± 1.6 A	3.4 ± 1.9 a	3.9 ± 1.9 a
Baseline vs 2 wk	3.4 ± 1.3 A,B	4.0 ± 1.7 A,B	3.7 ± 2.1 a,b	4.3 ± 1.9 a,b
Baseline vs 3 wk	4.4 ± 1.4 B	4.7 ± 1.6 B	4.3 ± 2.2 b	4.4 ± 2.1 b
Baseline vs 1 wk postbleaching	4.4 ± 1.5 B	4.5 ± 1.5 B	4.2 ± 2.1 b	4.4 ± 2.1 b
Baseline vs 1 mo follow-up	4.4 ± 1.1 B	4.3 ± 1.7 B	4.2 ± 2.1 b	4.3 ± 2.1 b

[a] *Two-way analysis of variance (ANOVA) and Tukey test for each measurement (α=0.05). Means identified with the same uppercase and lowercase letters indicate statistically similar values for the subjective and objective evaluations, respectively.*

time was not significant (p=0.934), but the main factors were (p=0.019 and p<0.001, respectively). A higher degree of bleaching was observed for the control group. With regard to assessment time, a higher degree of bleaching was obtained after the third week of treatment, and this was statistically similar to the shade measured at one week and one month postbleaching (Table 2).

Tooth Sensitivity

No statistical difference between groups was observed in terms of absolute risk of tooth sensitivity (Fisher exact test, p=1.0). The values for absolute risk of tooth sensitivity of the control and experimental groups were 55% (95% confidence interval [CI] 34.2%-74.2%) and 60% (95% CI 38.7%-78.2%), respectively. The intensity of tooth sensitivity was also similar between groups (p=0.529 for NRS and p=0.258 for VAS scales, respectively; Table 3). Approximately 57% of the participants presented with mild tooth sensitivity.

Table 2: *Means and Standard Deviations Between Assessment Points for the Two Treatments in ΔE for the Two Groups*[a]

Assessment Time Interval	Groups	
	Control	Experimental
Baseline vs 1 wk	6.8 ± 2.5 Aa	6.5 ± 3.2 Ab
Baseline vs 2 wk	8.8 ± 2.6 Aa	7.7 ± 3.3 Ab
Baseline vs 3 wk	10.8 ± 3.0 Ba	9.8 ± 2.7 Bb
Baseline vs 1 wk postbleaching	11.0 ± 3.1 Ba	9.5 ± 2.8 Bb
Baseline vs 1 mo follow-up	10.6 ± 2.3 Ba	9.8 ± 2.6 Bb

[a] *Two-way analysis of variance (ANOVA) and Tukey test (α=0.05). Means identified with the same uppercase letters indicate similar values within column. Means identified with the same lowercase letters indicate statistically similar averages within rows.*

DISCUSSION

In the present study, each patient performed mouth rinses with coffee four times a day for 30 seconds for a period of three weeks. In previous laboratory studies, the time of exposure to coffee varied greatly.[19,21] For example, Attia and others[19] immersed the specimens for 15 minutes a day for 28 days, whereas Cardoso and others[21] performed five daily exposures lasting one minute each for 15 days. The time of bleached tooth exposure to coffee in this study was determined by taking into consideration

Table 3: *Medians and First and Third Interquartiles of the Tooth Sensitivity Scores and Pain Scales for the Two Groups*

Group	Five-point Numerical Verbal Rating Scale*	Visual Analog Scale**
Control	1 (0; 1)	0.1 (0; 0.3)
Experimental	1 (0; 1)	0.4 (0; 0.7)

** Mann-Whitney test,* p = *0.529; ** Mann-Whitney test,* p = *0.258.*

the time it took to swallow the coffee.[34] Considering that this probably did not take longer than two to five seconds, four daily mouth rinses performed for 30 seconds each would represent excessive consumption of the beverage.

Researchers are constantly concerned about the possibility that alterations in tooth enamel caused by bleaching agents[10,11] may negatively interfere with the effectiveness of treatment. Indeed, laboratory studies have related that bleaching agents promote alterations in the tooth enamel surface due to the slightly acidic nature[35] and demineralizing potential of bleaching products,[9] and this could favor greater retention of coloring agents in the enamel. However, the results of the present study oppose the widespread idea among clinicians that foods and beverages with coloring agents, such as coffee, may pigment the teeth if used while dental bleaching is being performed.[15] When the ΔSGU values obtained by the subjective and objective methods were compared, a similar degree of bleaching was observed in the two study groups after three weeks of bleaching. These results are in agreement with those of previous laboratory studies[21,36] that investigated the effect of coffee on home and in-office dental bleaching, in which no significant difference in the effectiveness of bleaching was observed when coffee was tested as a coloring beverage during dental bleaching.

Saliva most likely plays a fundamental role in reversing the structural alterations produced by dental bleaching agents. During the use of the bleaching tray, human saliva may act to replace minerals lost by the tooth structure during bleaching[17,37] and may also neutralize the low pH of coffee,[17] which justifies the favorable results obtained even in the presence of coffee. Indeed, in a literature review Attin and others[38] observed that in studies that found a reduction in enamel microhardness, human saliva and fluoridation were not used during dental bleaching. In a comparison between *in vitro* and *in situ* results, Justino and others[39] confirmed the remineralizing effect of human saliva, since the most pronounced alterations, such as depressions in the tooth surface and reduction in microhardness values, were observed in the *in vitro* group. In addition, the bleaching gel used in this study contained sodium fluoride and potassium, which also act as remineralizing agents.

Another important aspect to point out is that the substances that are thought to cause extrinsic staining, such as coffee, are compounds constituted of macromolecular chains and, thus, are hardly capable of permeating through human enamel, which allows only the passage of low-molecular-weight molecules.[40] Tooth enamel functions as a semipermeable membrane that only allows the passage of ions and small molecules,[40] therefore the bleaching process does not occur in the mineralized enamel structure, but possibly as a result of oxidation of the organic tissues of human dentin.[41] Moreover, it is known that extrinsic stains are associated with the adsorption of pigments on the tooth enamel surface as well as with biofilm[42] and can be efficiently removed by means of professional dental prophylaxis.

During the return visits scheduled in this clinical study, it was observed that the effectiveness of dental bleaching was maintained over the course of time. There was no statistically significant difference between the ΔSGU values (Vita subjective and objective) and ΔE values obtained on the conclusion of treatment and those obtained in the postbleaching time intervals evaluated (one week and one month). These results are in disagreement with the results obtained by Attia and others,[19] who observed that the bleaching effect was less stable when human and bovine teeth were exposed to coffee during home bleaching. The fact that the patients maintained oral health in a satisfactory manner, a procedure that was not performed in the study of Attia and others,[19] may have prevented the incorporation of extrinsic stains on the tooth surfaces.

Another adverse effect commonly observed during bleaching is dental sensitivity.[4,7,29,30] No statistically significant difference was found between the two groups evaluated with regard to the dental sensitivity reported by the patients (control group, 55%; experimental group, 60%). The absolute risk of sensitivity presented in this study corroborates the values of other researchers.[29,43] In this study, the majority of the patients who presented with sensitivity defined it as "slight" when it was evaluated by means of the NRS and VAS scales, which is in agreement with the results shown in other studies.[16,29,43]

The low intensity of dental sensitivity in this study may be associated with the fact that the bleaching agent contained potassium nitrate as a desensitizing agent. Potassium nitrate penetrates the enamel and dentin and travels to the pulp, creating a desensitizing effect on the nerve by affecting the transmission of nerve impulses.[44] In addition, the bleaching gel was used for only three hours a day. Cardoso and others[45] verified that the prevalence and intensity of dental sensitivity increased considerably when the

CP-based bleaching gel was used for eight hours a day in comparison with only one hour a day.

Some of the limitations of this study should be mentioned. Unfortunately, the participants were not randomized to the study groups because this procedure was not ethically justified. In spite of this, the findings of this nonrandomized clinical trial are stronger than those obtained in a laboratory setting. In addition, dietary restrictions were only utilized in the control group. If differences were detected among groups we would not be able to discriminate between the effect of coffee exposure and those of the other staining foods from the patient's diet. The patients included in this study presented excellent oral hygiene, had no restoration in the anterior teeth, and had no gingival recessions, which could facilitate the penetration and deposition of coloring matter from the coffee. Therefore, the results of this study cannot be extrapolated to populations with deficient oral hygiene and those presenting with one of the exclusion criteria of this research.

CONCLUSION

Within the limitations of this study, it can be concluded that exposure to coffee during at-home dental bleaching with 16% CP did not affect tooth sensitivity or the effectiveness of dental bleaching.

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Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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Comparison of Enamel and Dentin Shear Bond Strengths of Current Dental Bonding Adhesives From Three Bond Generations

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Clinical Relevance

Dentists use dental bonding agents (DBAs) in most operative/restorative procedures. Knowing the strengths and weaknesses of such commonly used materials helps clinicians choose the appropriate DBA for optimal clinical outcomes.

SUMMARY

Objective: Durability is still a major challenge in adhesive dentistry. One of the biggest areas of development has been to simplify the bonding process by using all-in-one adhesives. The aim of this study was to compare the shear bond strength (SBS) to dentin and enamel of nine dental bonding agents (DBAs) from three generations after simulated aging.

Methods and Materials: For this study, 108 sound extracted human molars were randomly assigned to nine groups (n=12). The sample teeth were mounted in self-cure acrylic resin sectioned to provide paired enamel and dentin samples. All samples were polished with 240 and 600-grit silicon carbide sandpaper and randomly grouped according to the product and substrates (enamel or dentin). Herculite Ultra resin composite cylinders were bonded on each test surface, stored in 100% humidity at 37°C for 24 hours, and then thermocyled for 1,000 cycles at 5°C and 55°C. SBS testing was performed using an Ultratester at a crosshead speed of 0.5 mm/min. Statistical analysis included two-factor analysis of variance, one-sample Wilcoxon and Kruskal-Wallis tests, and the Scheffe post hoc test at an alpha level of 0.05 using SAS version 9.2.

Results: Significant differences in SBS were observed between the sixth- and seventh-generation DBAs (p=0.002) but not between the sixth- and fourth-generation DBAs. Scheffe post hoc tests for the sixth-generation DBAs showed that some DBAs yielded significantly higher enamel SBS than others, but not as much as dentin SBS. As for the seventh-generation DBAs, similar post hoc tests showed

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significant variations in SBS between substrates (enamel and dentin) and DBAs tested. Significant main effects were also found for the different substrates for the fourth-generation ($F[1,96]=10.532$; $p=0.003$) and seventh-generation ($F[1,96]=22.254$; $p<0.001$) DBAs, but not for the sixth-generation DBAs ($F[1,96]=1.895$, $p=0.172$). The SBS was higher on dentin than enamel for the fourth- and seventh-generation DBAs.

Conclusion: As expected, fourth- and sixth-generation DBAs generally showed stronger SBS values than the seventh-generation all-in-one DBAs. The new sixth-generation DBA OptiBond XTR (Kerr) showed strong SBS values to both substrates and performed well in comparison to the other DBAs tested.

INTRODUCTION

The past two decades have seen rapid progress to improve dental adhesive bonding materials and technologies as well as to simplify clinical application for dentin and enamel. The major shortcoming of contemporary adhesive restoratives is their limited durability *in vivo*.[1] The most cited reasons for failure of adhesive restorations are loss of retention and marginal adaptation.[2,3] Consequently, a viable approach to prolong the clinical lifetime of dental adhesives is to focus on improving the long-term stability of the bond of these biomaterials to tooth hard tissues, especially dentin.

The immediate bonding effectiveness of most current adhesive systems is quite favorable regardless of the adhesive used.[4] However, when adhesives are tested in simulated laboratory studies and clinical trials, the bonding effectiveness of some materials appears dramatically low, whereas the bonds of other materials are more stable.[5,6]

Although many advances have been made in adhesive technology since Michael Bonocoure introduced acid etching in 1955 and Ray Bowen introduced bisphenol A glycidyl methacrylate (Bis-GMA) to dentistry in 1962, the bond interface remains the biggest challenge when placing an adhesive restoration.[7] Water sorption is thought to be the main factor destabilizing the adhesive–tooth bond, although the actual interfacial degradation mechanisms are not completely understood.[8,9] Other factors to consider are all the chemical and mechanical challenges inherent to the oral environment, such as moisture, masticatory stresses, changes in temperature and pH, and dietary and chewing habits.[1]

One of the biggest areas of development has been to simplify the bonding process by use of all-in-one adhesives, but according to the review published by Van Meerbeek and others, "the conventional 3-step etch & rinse adhesives and 2-step self-etch adhesives are still the benchmarks for dental adhesion in routine clinical practice."[10] All-in-one adhesives have certainly improved over the past decade, and the development of functional monomers with strong and stable chemical affinity to hydroxyapatite is without doubt a valuable direction to continue for the improvement of dental adhesion.

The aim of this study was to compare shear bond strengths (SBSs) to dentin and enamel of nine dental bonding agents (DBAs) from three generations, including a new sixth-generation Optibond XTR (Kerr, Orange, CA, USA), after simulated aging with thermocycling. The null hypotheses were that there would be no significant differences in SBS among the three DBA generations tested, and that there would be no significant differences in SBS between enamel and dentin substrates from the three DBA generations tested.

METHODS AND MATERIALS

A total of 108 extracted human molars were randomly assigned to nine groups (n=12). The teeth were selected in the following manner: the crowns were assessed under magnification (Surgitel 2.75×, Loupes GSC Corp, Ann Arbor, MI, USA), and the teeth with visible caries, cracks, or tooth structure anomalies were excluded from the study.

The sample teeth were mounted in an Ultradent mold (Ultradent Products, South Jordan, UT, USA) using self-cure polyethyl methacrylate diethyl phthalate (PMDP) acrylic resin (Esschem, Linwood, PA, USA) positioning the facial or lingual surface (whichever was flatter) to be exposed for the enamel bond surface. They were then sectioned mesiodistally at approximately 4 mm distance from the enamel surface. The enamel sections were then remounted in the acrylic molds in order to fit the Ultradent bonding jig so they could be tested for SBS with the Ultratester (Ultradent Products). The remaining portion of the sectioned tooth became the dentin sample. This made for a total of 216 test surfaces providing paired enamel and dentin samples from each tooth. Sample sectioning was done with a water-cooled diamond wheel saw (Leitz 1600, Wetzlar, Germany).

The enamel surfaces were polished with 240-grit silicon carbide (SiC) sandpaper until a flat area of

Table 1: *Dental Bonding Adhesive Systems: Composition, pH, and Protocols* [a]

Product/ Generation	Etch/ Rinse	Primer	Self-etch/ Primer	Adhesive/ Light Cure	Etch-Prime-Adhesive/Light Cure	Primer pH
Optibond FL (Kerr, Orange, CA, USA)/4th	15 s/10 s	15 s scrub	—	15 s/20 s	—	2.0
Optibond FL (alternate) Enamel without primer/ 4th	20 s/10 s 20 s dry	—	—	10 s /20 s	—	—
Optibond XTR (Kerr, Orange, CA, USA)/ 6th	—	—	20s scrub	15s/10s	—	1.6
Clearfil SE (Kuraray America, Inc., New York, NY USA)/ 6th	—	—	20s	Apply/10s	—	2.0
SE Protect (Kuraray America, Inc., New York, NY USA)/ 6th	—	—	20 s	Apply/10 s	—	2.0
Prelude (Danville Materials, San Ramon, CA, USA)/6th	—	—	10 s scrub	10 s scrub/10 s	—	2.0
Xeno IV (Dentsply Caulk, Milford, DE, USA)/ 7th	—	—	—	—	15 s scrub ×2/10 s	2.3
iBond SE (Heraeus Kulzer, South Bend, IN, USA)/ 7th	—	—	—	—	20 s scrub/20 s	1.6
Prompt-L-Pop (3M ESPE, St. Paul, MN, USA/ 7th	—	—	—	—	15 s scrub, second layer/10 s	1.0
Futura Bond DC (VOCO America, Inc., Briarcliff Manor, NY, USA)/7th	—	—	—	—	20 s scrub/10 s	1.5

[a] *Air drying for 10 seconds was used after all primer applications, and gentle air thinning was used when all adhesive applications were done.*

approximately 5 mm in diameter was established. The dentin surfaces were also polished with 240 grit sandpaper and copious amounts of water. Both enamel and dentin surface groups were then polished with 600-grit SiC sandpaper with copious amounts of water. The samples were grouped by substrate pairs and labeled according to the product and substrate (enamel or dentin) for accurate identification. They were then hermetically sealed and stored in 100% humidity at 37°C. Each group was bonded within 24 hours of surface preparation.

Next, the adhesive systems were used according to manufacturers' instructions for each test group as seen in Table 1. Ultradent SBS test molds (Ultradent Products) were used to build resin composite cylinders 2.38 mm in diameter × approximately 2.0 mm in height with Herculite Ultra A2 Enamel (Kerr) bonded on each test surface. The resin composite cylinders were light-cured for 40 seconds using a dental halogen curing light (OptiLux 500, Kerr) that was tested regularly to ensure 420-460 mW/cm^2 for 40 seconds from 1.0 mm distance.

After sample bonding, the specimens were stored in 100% humidity at 37°C for 24 hours then thermocyled for 1,000 cycles at 5°C and 55°C with a 30-second dwell time. The samples were then submitted to SBS testing at room temperature using an Ultratester testing machine (Ultradent Products) set to operate at a 0.5 mm/min crosshead speed until bond failure occurred. Once the SBS testing was done for the fourth-generation enamel group, the enamel surfaces were re-prepared according to the same protocol, rebonded with 20 seconds etch and rinse, submitted to 20 seconds strong air dry, and then bonded by the same protocol using the adhesive only (no primer). In regards to reusing the enamel samples for comparing an alternative protocol, this was done to make an accurate comparison with the same substrate surfaces. Because enamel has a very low level of organic constituents, and the samples were kept under controlled humidity with a short storage time between bond testing and rebonding, we believed these factors would have little effect on the results.

The statistical tests of hypotheses regarding the SBS data were performed using a two-factor (between and within) analysis of variance (ANOVA) of ranked data. Comparisons between dentin and enamel were assessed using statistical procedures

Table 2: *Shear Bond Strength (SBS) Summary*

Generation	Product	Enamel SBS	SD	Dentin SBS	SD
Fourth	Optibond FL (Kerr)	27.1	9.7	38.9	8.6
Fourth	Optibond FL (alternate)[a]	28.1	5.7	NA	NA
Sixth	Optibond XTR (Kerr)	34.1	7.8	33.4	8.8
Sixth	Clearfil Protect Bond (Kuraray)	36.7	5.2	30.7	8.5
Sixth	Clearfil SE Bond (Kuraray)	30.7	8.5	27.6	7.1
Sixth	Prelue (Danvile)	18.5	10.8	34.5	7.4
Seventh	Xeno IV (Detsy Caulk)	14.4	5.4	33.8	9.2
Seventh	Prompt L Pop (3M ESPE)	24.6	9.5	27.1	11.3
Seventh	Futurabond (Voco)	13.1	8.9	14.9	6.7
Seventh	Ibond Self Etch (Heraeus)	14.6	9.7	16.8	13.2

[a] *Alternative protocol without primer.*

for related samples (one-sample Wilcoxon signed ranks test). Comparisons between DBAs were conducted with the Kruskal-Wallis procedure for independent samples and the Scheffe post hoc test for the data+ between bond generations. All the tests of hypotheses were two-sided and conducted at an alpha level of 0.05 using SAS version 9.2 (SAS Institute, Cary, NC, USA). The samples that failed before testing were given a zero value but were included in statistical analysis. This occurred during the thermocycling process and mainly with the seventh-generation bonding adhesive systems.

RESULTS

Table 2 summarizes the results. Significant differences were detected in SBS between groups regardless of the substrates, generations, and DBAs tested ($p<0.001$). Significant differences were also detected in the SBS between some of the enamel groups and between some of the dentin groups ($p=0.002$) as depicted in Figures 1 and 2. Only two of the nine study groups demonstrated a SBS difference that was significantly different from zero when dentin was compared to enamel ($p<0.05$) (Figure 3).

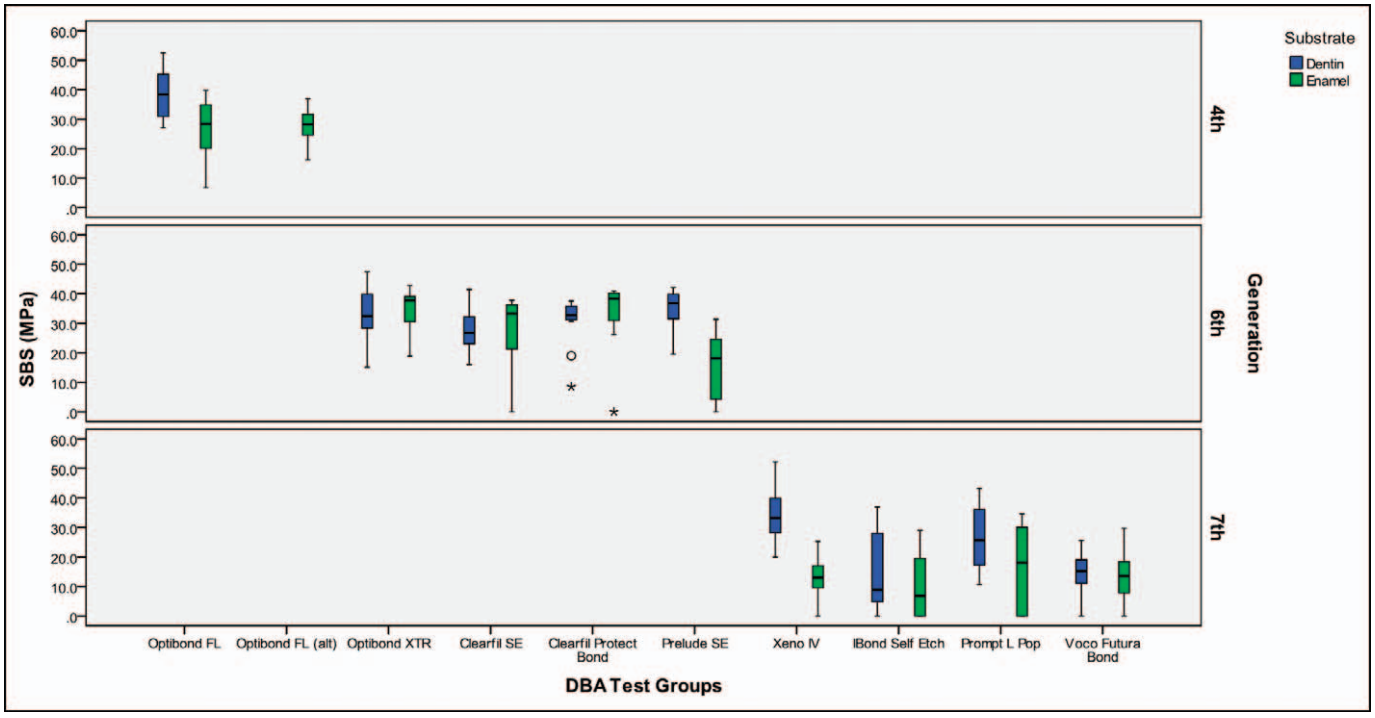

Figure 1. *Shear bond strength of DBAs according to generation (MPa). The * and 0 indicate outliers in the sample. Use of nonparametric statistics and analysis of ranked data reduces the bias of outliers on central tendencies. These data were included in the statistical analysis.*

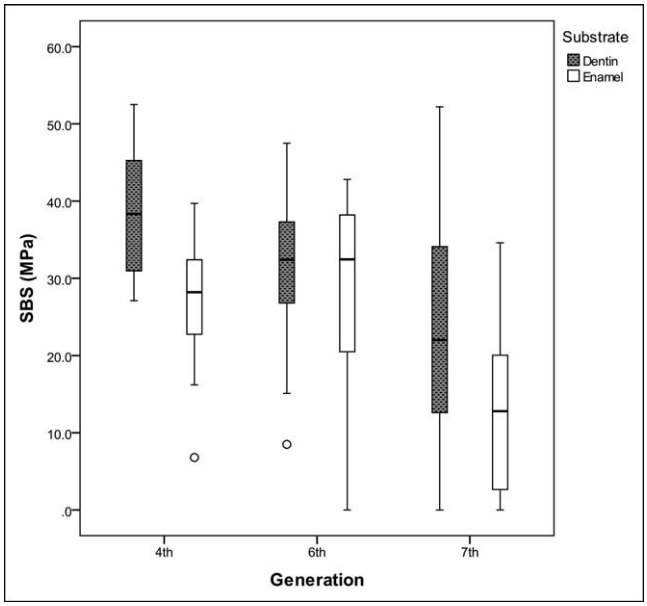

Figure 2. *Shear bond strength of DBAs pooled by generation and substrate. The 0 indicate outliers in the sample. Use of nonparametric statistics and analysis of ranked data reduces the bias of outliers on central tendencies. These data were included in the statistical analysis.*

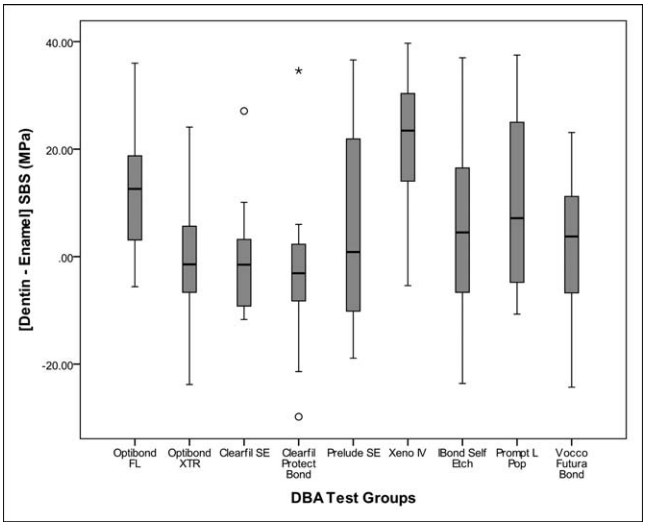

Figure 3. *Shear bond strength comparison of paired dentin and enamel. Values <0 indicate that the enamel SBS values were stronger than the dentin SBS values. The * and 0 indicate outliers in the test groups. Use of nonparametric statistics and analysis of ranked data reduces the bias of outliers on central tendencies. These data were included in the statistical analysis.*

Effect of Product on Bond Strength

The dependent variable for this analysis was the rank of SBS as measured in megapascals (MPa) and was used to test the effect of independent variables, which are the bonding agents, as "adhesive product" or "DBA" and enamel and dentin as "substrate." The SBS measurements were analyzed using a two-factor independent ANOVA within each generation group. The first factor was the adhesive product (nine total; between DBA groups), and the second factor was the substrate (enamel, dentin; within DBA groups). Figure 1 provides a summary of these results. Statistically significant differences in bond strength were observed between the DBAs of the sixth-generation DBAs [$F(3, 96) = 4.202$, $p = 0.008$] and seventh-generation DBAs [$F(3, 96) = 7.199$, $p < .001$], but not between the fourth- and sixth-generation DBAs.

Scheffe post hoc tests for the sixth-generation DBAs showed that Optibond XTR (Kerr, Orange, CA, USA) yielded significantly higher enamel bond strength than Prelude SE (Danville Materials, San Ramon, CA, USA) ($p = 0.037$). Clearfil Protect Bond (Kuraray America, Inc., New York, NY USA) also yielded adequate enamel bond strength, as did Prelude SE, with no statistically significant difference ($p = 0.093$); however; dentin SBS for Prelude SE was among the highest in its generation group. For the seventh-generation DBAs, similar post hoc tests showed that Xeno IV (Dentsply Caulk, Milford, DE, USA) had significantly higher bond strength than IBond Self Etch (Heraeus Kulzer, South Bend, IN, USA) ($p = 0.010$) and Voco Futura Bond ($p = 0.009$). Prompt-L-Pop also had significantly higher bond strength than IBond Self Etch ($p = 0.032$) and Voco Futura Bond (VOCO America, Inc., Briarcliff Manor, NY, USA) ($p = 0.029$). No other pairwise comparison within the sixth- or seventh-generation DBAs yielded notable results.

The SBS measurements were also analyzed using a two-factor independent ANOVA between generations. Figure 4 depicts the results showing that there was no significant difference between pooled generation groups of fourth- and sixth-generation DBAs; however, the pooled generation group SBSs of seventh-generation DBAs were significantly lower than those of the fourth and sixth pooled generation groups.

Effect of Substrate on Shear Bond Strength

Significant effects were also found for the different substrates for the fourth [$F(1, 96) = 10.532$, $p = 0.003$] and seventh [$F(1, 96) = 22.254$, $p < 0.001$] generations; This was shown by the fact that the dentin SBS was significantly higher than the enamel SBS in both the fourth and seventh generations. But for the sixth generation [$F(1,96) = 1.895$, $p = 0.172$],both the dentin and enamel substrates had almost

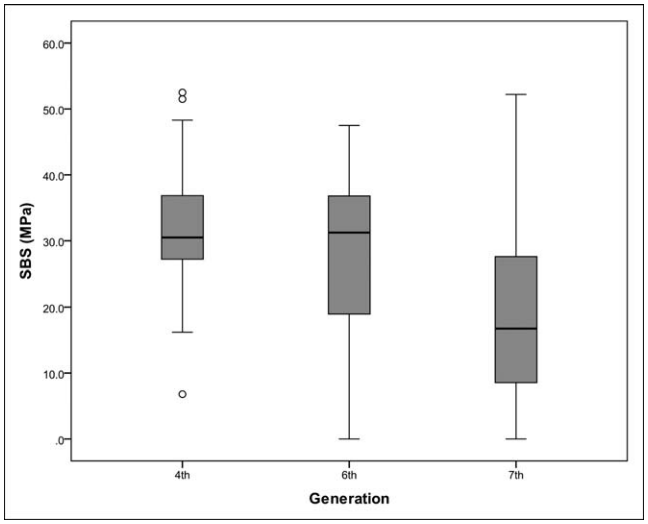

Figure 4. *Shear bond strength of DBAs pooled by generation. There was no significant difference between fourth- and sixth-generation adhesives when substrates were pooled; however, SBS values were significantly lower for seventh-generation adhesives. The * and 0 indicate outliers in the sample. Use of nonparametric statistics and analysis of ranked data reduces the bias of outliers on central tendencies. These data were included in the statistical analysis.*

identical SBS (see Figure 2). Also of interest was that the two DBAs with the highest combined enamel and dentin SBS and lowest variability were Clearfil SE Protect and Optibond XTR. With the alternative bonding protocol of the fourth-generation DBA to enamel (20-second etching time, 20-second air-drying time, and adhesive without primer) a significant SBS value was not observed between the primer plus adhesive manufacturer's recommended protocol and the alternative adhesive only protocol for Optibond FL (Kerr, Orange, CA, USA), 27.5 MPa vs 28.1 MPa, respectively.

DISCUSSION

Difference Between All Groups

As stated in the introduction, the null hypotheses for this study were that there would be no significant SBS differences between the three DBA generations tested, and that there were no significant SBS differences between enamel and dentin substrates from DBAs of the three adhesive generations tested. However, significant differences were detected in SBS between groups regardless of the mentioned variable DBAs ($p<0.001$). This is not surprising as manufacturers are continually trying to come up with DBAs that have improved physical and mechanical properties as well as simplifying the protocol for ease of use. Numerous studies have been done to test these DBAs and to alter the original protocols. Because of variations in the protocols and diverse formulations of primers and adhesives, it is expected that some will simply work better than others. And that is what was observed in this study as well as others.[10-14]

One aspect in the design of this study that was specifically done to add more power to the results was pairing substrate samples. With any study, whether it is *in vitro* or *in vivo*, there will always be confounding factors, such as age of the test tooth, storage media and duration, cultural and environmental factors of the person from whom the tooth was obtained, and sample-processing procedures. These issues are difficult, if not impossible, to account for statistically. To minimize these confounding factors, care was taken to pair the enamel and dentin samples in order that SBS comparisons of these substrates would be more meaningful.

It has been reported that self-etch bonding systems have a significantly lower SBS to enamel than the gold standard total etch fourth-generation adhesives.[15] This was not the case in our study, as our data showed higher enamel bond strengths, though not significant, with some of the self-etch sixth-generation DBAs (Figures 1 and 2). For example some of the highest bonds to enamel in this study were with the new Optibond XTR self-etch, even though it is considered a mild self-etch adhesive with a pH of 2.4. According to the manufacturer, the pH drops to 1.6 during the primer application, resulting in enhanced enamel etching and bonding.

Perhaps this is because the primer has hydrophilic comonomers including mono- and di-functional methacrylate monomers, in a solvent of water, ethanol, and acetone. This three-part solvent is believed to enhance its self-etching capability by facilitating penetration of the hydrophilic monomers into the tooth, which should lead to high bond strengths. The Optibond XTR hydrophobic adhesive layer contains a balanced or neutral chemistry in that it has a pH of 3.3 in the bottle but the pH increases to 6.5-7.0 after application and light curing.[16] This seems to complement the primer by stabilizing the bond in a moist oral environment.

Difference Among Enamel Groups

Significant differences were detected in the SBS between some of the enamel groups, as shown in Figure 1 ($p<0.001$). No significant difference in SBS was observed between the pooled fourth- and sixth-generation DBAs with the enamel substrate (Figure 2). The SBS to enamel was significantly lower ($p=0.003$) with all of the seventh-generation adhe-

sives compared with the previous generations (fourth and sixth) with exception of Prelude SE. The median SBS for the fourth-generation adhesive at the enamel was lower, but not to a statistically significant degree, than that of the sixth-generation adhesives. When the alternative protocol for bonding to enamel (adhesive only to dry etched enamel) was used in this study, the mean SBS increased, but the difference was not statistically significant. Historically, concerns have been expressed regarding the short- and long-term bonding effectiveness of self-etch adhesives to unetched and or unprepared enamel[17] especially the mild self-etch class.

Difference Among Dentin Groups

Significant differences were also detected in the SBS between some of the dentin groups shown in Figure 1 ($p=0.002$). The fourth-generation group had the highest SBS, which was significantly stronger than the sixth-generation groups pooled (Figure 2); however, it was not higher than Prelude by itself (Figure 1), and both the fourth and sixth generations had significantly higher SBS than the seventh-generation groups. These results are expected from review of other studies. In a 2005 review, De Munck and others[7] concluded that the three-step etch-and-rinse adhesives are still the gold standard in durability and that any kind of simplification, such as one-step all-in-one adhesives, results in loss of bonding effectiveness and durability. They stated, "Only the two-step self-etch adhesives approach the gold standard and do have some additional clinical benefits."[7]

This may be due to the finding that dentin collagen fibrils contain inactive proforms of proteolytic enzymes called matrix metalloproteinases (MMPs). When fully mineralized, the MMPs in the dentin matrix are inactive. Most of the one-step all-in-one adhesives are highly acidic. These MMPs are exposed and activated by acid-etching or self-etch primers during adhesive application process.[18] Therefore, the stronger the acid the more MMPs are released, resulting in adhesion breakdown over time. A possible reason the fourth- and sixth-generation adhesives have greater durability than the one-step all-in-one adhesives is that they get better infiltration and adaptation (wetting) to the exposed collagen fibrils because their hydrophilic primer is separate from the hydrophobic adhesive. Other factors have also been proposed for their lower performance, such as inhibition of polymerization of the restorative composite being bonded due to the high acidity of seventh-generation DBAs, an insufficiently thick adhesive layer, phase separation between hydrophilic and hydrophobic ingredients, and resultant sensitivity to hydrolysis.[19]

Depth of penetration of the monomer into the hybrid layer, however, may not be the main determining factor of bond strength. Yoshida and others[20] have described another bonding mechanism in that self-etch adhesives, especially mild ones, demineralize dentin only partially, and that leaves hydroxyapatite partially attached to collagen. As some of the hydroxyapatite remains available for chemical interaction between the functional monomer's carboxyl groups it can form an ionic bond with the calcium of the residual hydroxyapatite. So it is theorized that the less soluble the calcium salt of the acidic molecule, the more intense and stable the molecular adhesion to a hydroxyapatite-based substrate.[20] This certainly could be a big factor in explaining the variance in DBA bond strengths.

In a more recent review, Van Meerbeek and coworkers[10] stated: "While micromechanical interlocking remains the primary adhesive mechanism, mild self-etch adhesives (sixth generation), in particular, may additionally make use of chemical interaction that especially contributes to the long-term stability of the bond. Although one-step adhesives are the simplest to use, their adhesive performance is less than that of multistep adhesives, primarily due to lower bond strength and durability, phase separation phenomena with hydroxy-ethyl-methacrylate (HEMA)-poor/free formulations, enhanced water sorption with HEMA-rich formulations, and a reduced shelf life."

The results of the current study also showed that the sixth-generation adhesives, including the new Optibond XTR, appear to perform very well in comparison to the fourth-generation adhesives. The seventh-generation all-in-one type adhesive systems are more susceptible to water sorption and, as a result of nanoleakage, are more prone to bond degradation and tend to fail prematurely compared with their fourth- and sixth-generation counterparts.[9]

Difference Between Paired (Enamel vs Dentin) Groups

In the present study, the enamel SBS was generally weaker than the dentin SBS in the fourth- and seventh-generation groups, but they were almost equal in the sixth-generation groups (Figure 2). The current study paired the enamel and dentin substrates from the same tooth to allow a more powerful

comparison between the SBS between the two substrates. We found that only two of the nine study groups demonstrated a bond strength difference between the two substrates (Figure 3) that was significantly different from zero when dentin was compared to enamel: Optibond FL (p=0.012) and Xeno IV p=0.008), shown in Figure 3. For these two groups, the bond strength for dentin was significantly higher than that measured for enamel. In a recent study that involved bonding to dentin and enamel, Hanabusa and others[11] found that by pre-etching the enamel with phosphoric acid etch-and-rinse, the enamel bond and dentin bond strengths were almost equal. However the marginal integrity of bonded enamel is still greater than marginal integrity of dentin and less prone to hydrolytic breakdown.[21]

Difference Between Generations (Total)

Significant differences were detected in the bonding strength between the generations when both enamel and dentin substrates were pooled (p=0.002). Both the fourth-generation (p<0.001) and sixth-generation (p<0.001) groups demonstrated significantly higher SBS than the seventh generation. No significant difference was observed between the fourth and sixth generations (p=0.781). Figure 4 depicts the SBS of the generations with enamel and dentin values pooled. In almost every case, the dental bonding systems that combine the primer and adhesive have lower SBS values and longevity, The gold standard in dental bonding is still the fourth generation, followed closely by the sixth generation.[7] In both of these classes of dental bonding agents, the primer and adhesives are placed as two separate steps. Perhaps one of the biggest factors for this is that the chemistry of a one-bottle system is not stable and is prone to hydrolyzation; this weakens the ability of the acidic monomers to etch as they prime the tooth substrate.[22] With this in mind, some manufacturers (eg, 3M ESPE with Prompt-L-pop) have altered the packaging to address this problem with some success.

Difference Between Generations (Enamel vs Dentin)

Another finding in this study that was contrary to the null hypothesis was that significant differences were detected in the enamel bonding strength between the generations (p<0.001). The fourth and sixth generations were not significantly different from each other; however, the sixth-generation DBAs, with their self-etch primers, had higher mean SBS values (Figure 2). One might expect that the fourth-generation etch-and-rinse product would have the highest SBS to enamel because of its deeper etching ability. When bonding to enamel, an etch-and-rinse approach is definitely preferred because the micromechanical interaction appears sufficient to achieve a durable bond to enamel. On the other hand, the mild self-etch (sixth generation) approach seems to provide superior performance when bonding to dentin. These mild self-etch adhesives also chemically interact with residual hydroxyapatite because mild self-etch adhesives demineralize dentin only partially, leaving a substantial amount of hydroxyapatite crystals around the collagen fibrils. This remains available for possible additional chemical interaction.[23] The resulting twofold bonding mechanism, that is, micromechanical and chemical adhesion, is believed to be advantageous and definitely contributes to bond effectiveness and durability.[9] This is similar to the twofold bond mechanism seen with glass ionomer restoratives. This may explain why the mild self-etch (sixth generation) adhesive systems in this study had bonds equal to or higher than those of the fourth-generation etch-and-rinse systems. The all-in-one seventh-generation DBAs, with their stronger pH, do not seem to share the twofold bonding mechanism; therefore, their bond effectiveness and durability are not expected to be as good.

CONCLUSIONS

The null hypotheses of this study that there would be no significant difference with SBS between three adhesive generations of DBAs and between substrates (enamel and dentin) were rejected. According to the results of the current study, there were differences between the SBS of the enamel and dentin substrates and the DBA generations. This study also showed that SBS differed significantly between DBA generations more so than between the substrates tested. As expected from previous studies, fourth- and sixth-generation multistep DBAs generally showed stronger SBS values than the seventh-generation all-in-one DBAs. Also of note, the new sixth-generation DBA, OptiBond XTR, showed strong SBS values to both enamel and dentin substrates and performed well in comparison to the other DBAs tested.

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Conflict of Interest
The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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