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Editorial

Clinical Trial Registration

As editor of Operative Dentistry, one of my driving motivators is a desire to maintain the journal's stature as an upper echelon dental journal. In an ever-changing publishing world, required decisions go far beyond whether or not a given manuscript should be accepted for publication. For instance, our editorial staff considers recommendations made by the International Committee of Medical Journal Editors (ICMJE), an organization that outlines ethical principles and other publication guidelines for authors and editors in the health sciences.

In recent years, the ICMJE has advocated for the registration of all randomized clinical trials in searchable electronic databases available to the public at no cost.[1] Because there is no peer review mechanism associated with the registry, these databases hold information concerning the protocols but exclude the results of the trials. One reason for this suggested policy is the belief that a greater level of transparency will enhance the likelihood that a trial will be completed as planned and therefore increases the reliability of the evidence that the trial provides. A description of the required information for the database is included in the United States Federal Drug Administration Amendments Act of 2007.[2]

As with many such initiatives that touch multiple complex issues, this registry has proven to be controversial largely because it makes public, on a broad scale, the path of investigation that a particular research team is taking. In dentistry, many of these trials involve products that do not have patent protection because of their similarity to other existing products. Utilizing a public registry prospectively, before recruiting subjects, could arguably hinder a potential market advantage for dental manufacturers. Of note, the registry guidelines specifically state that feasibility or pilot studies are not intended to be covered. Rather, only larger efficacy studies that carry more clinical relevance for the public are targeted. On the positive side, registering randomized clinical trials provides an additional level of oversight to the appropriate conduct and reporting of the work. These clinical trials are designed to answer specific questions. Registered trials could assist editors and reviewers in discerning whether the aims of a trial were met or whether, retrospectively, data was manipulated or omitted in order to provide a positive result.[3]

At a recent Chicago meeting of journal editors and other interested parties, several journals (including the Journal of Dental Research, the Journal of the American Dental Association and the Journal of Periodontology) made it known that they have instituted a registration requirement. At least one journal is requiring registration, but not prospectively. But, it was clear that not all journals were excited about moving in this direction.

One hundred seventy-four dental publications are included in the PubMed/Medline's National Library of Medicine Catalog[4] and the American Dental Association holds over 600 journal titles in its library.[5] Each of these publications must make a decision about whether it is important for them to require this registration and I fully expect that there will be some who will opt out. Thus, clinical trial registration will become another factor that differentiates our dental publications. I believe that Operative Dentistry must continue to be a reliable source of the best available evidence for decisions regarding oral health. It seems to me that focusing on a high level of credibility in today's world of multiple information sources would uphold the reputation of this journal for the members of its supporting academies.

Moving forward, authors submitting to Operative Dentistry will be asked to provide registration information that our editors, reviewers, and staff can validate. (This registration will be in addition to the requirement of providing appropriate approval from a human subjects oversight committee given prospectively for any study using human subjects or tissues.) Completion of the registration process is a fairly innocuous task and can happen on any publically accessible database such as NIHClinicalTrials.gov. More specific instructions will be included in the journal's instructions to authors.

It is my hope that clinical trial registration will help keep this journal, and thus our sponsoring

Academies, recognized as desiring to provide optimal care for the people we serve. I am grateful for the many researchers around the world who contribute to our quest for knowledge, no matter whether they work in the private or public sector. I look forward to continuing to publish the best of that work.

1. International Committee of Medical Journal Editors. Uniform requirements for manuscripts submitted to biomedical journals: publishing and editorial issues related to publication in biomedical journals: obligation to register clinical trials. Retrieved online April 30, 2013 from http://www.icmje.org/publishing_10register.html.
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Jeffery A. Platt, Editor

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Clinical Tech/Case Report

Multidisciplinary Approach for the Treatment of Extensive External Cervical Resorption After Dental Trauma

TP Alves • TRC Soares • SC Barreto
H Fried • GDS Pereira • LC Maia
AEC Santos

Clinical Relevance

The prognosis of severe cases of external cervical resorption is normally unfavorable due to the injury location and treatment difficulty, and in most cases, the injured tooth is indicated for extraction. It is extremely important to preserve the injured tooth in young patients both for the psychologic aspect and for maintaining function and esthetics.

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SUMMARY

External cervical resorption (ECR) is a sequela of dentoalveolar trauma that may cause functional, esthetic, and psychologic alterations. The aim of this study was to report a successful multidisciplinary treatment approach performed in a 12-year-old patient who presented with posttraumatic ECR associated with extensive opened cavity, pulp necrosis, and periapical lesion of tooth number 9, with an initial unfavorable prognosis. Crown lengthening was done to enable restoration of vestibular surface with resin composite, forming a barrier that allowed endodontic treatment. Afterwards, a prefabricated fiberglass post was cemented and esthetic restoration was performed using the adhesive technique and direct composite veneer. Reconstructive periodontal surgery was performed to correct irregular gingival contour. After treatment and successive follow-up sessions, it was concluded that although the

tooth had been indicated for extraction, low invasive direct techniques were effective to recover function and esthetics and to maintain the tooth in the oral cavity.

INTRODUCTION

Dental trauma is considered an emerging public health problem that normally affects children and adolescents,[1] and it has a strong negative impact on their quality of life.[2] The main challenge of treating traumatized permanent teeth is related to preserving the tooth and minimizing possible clinical and radiographic sequelae.[1] This frequently requires careful multidisciplinary planning associated with follow-up sessions.

There is a consensus that dental trauma occurs more frequently in children, particularly boys, and in the anterior region of the maxilla,[3] most commonly affecting the central incisor teeth.[4] Overjet above 5 mm and inadequate lip sealing are significant risk factors for traumatic lesions.[2,3] The most common causes are related to falls, collisions, sports activities, violence, and road traffic accidents.[1,5,6] Several sequelae are associated with dental trauma, and their severity is directly related to the force and direction of the impact.[7] Studies have shown a prevalence of posttraumatic injuries to permanent teeth that range from 3.9% to 58.6%.[8] Among the types of injuries, the most frequent are pulp necrosis, internal and external resorption, calcific metamorphosis, and ankylosis.[1]

External dental resorption is characterized as an irreversible loss of cementum, dentin, and bone, which may be classified according to the location of the injury.[9,10] When this affects the cervical portion of the tooth it is called external cervical resorption (ECR). In some cases, restorative and endodontic treatment may be performed effectively using composites that satisfactorily restore patient esthetics.[11]

The prognosis of severe cases of ECR is normally unfavorable due to the location of the injury and difficulty of treatment, and in most cases, the injured tooth is indicated for extraction.[9] Therefore, the aim of this study was to report the success of a conservative multidisciplinary treatment approach with a patient who presented severe ECR associated with pulp necrosis, periapical lesion, and gingival retraction due to anterior dental trauma.

CASE REPORT

A 12-year-old girl was referred to the Dental Trauma Surveillance Center of the School of Dentistry of the Federal University of Rio de Janeiro. The history comprised the report of "a hole in the front tooth" and an injury to the tooth as a result of a bicycle accident one year earlier.

Immediately after the accident, the patient underwent treatment by other professionals to restore the fractured tooth without taking any radiographs. However, the restoration fractured one week later and it had to be redone. Three months after the injury, the patient had pain and edema in the region and was taken to an emergency service where endodontic access was performed to alleviate the symptoms. The patient's guardian was instructed to seek an endodontist in order to continue treatment. However, treatment was not immediately performed. Instead, orthodontic treatment began 4 months after the injury, which had to be interrupted due to the appearance of cervical resorption in the vestibular face of the traumatized tooth (Figure 1A,B).

During the clinical examination at the Dental Trauma Surveillance Center, an extensive cavity on

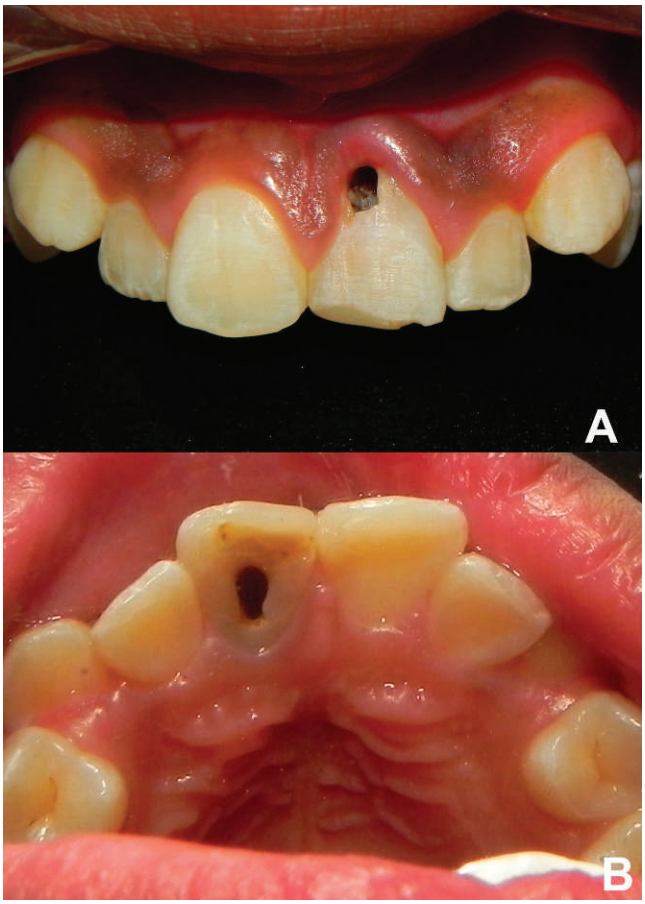

Figure 1. *Initial photos: external cervical resorption associated with caries. (A): Frontal view. (B): Occlusal view.*

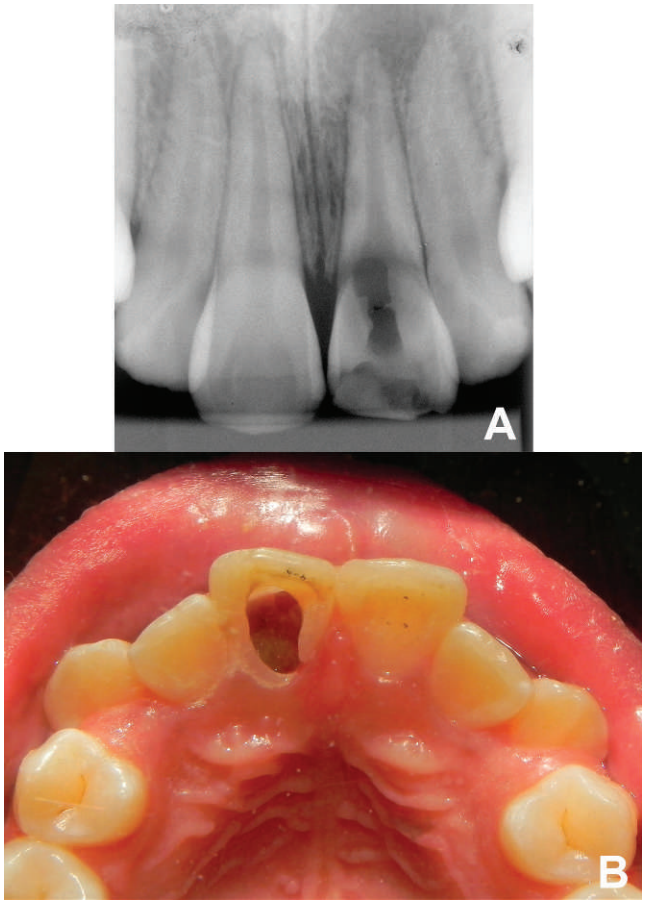

Figure 2. *(A): Initial radiograph. (B): Occlusal view after removal of decayed tissue.*

the vestibular surface in the cervical third was found on the upper left central incisor (tooth number 9) as well as gingival retraction associated with subgingival caries, which made the tooth very fragile. On the lingual surface, endodontic access without temporary sealing material was found in addition to the presence of a carious lesion (Figure 1A,B). The presence of an extensive carious lesion in the coronal region was seen in the radiograph, as well as extensive areas of ECR and a radiolucent image in the periapex suggesting a periapical lesion (Figure 2A). The extensive treatment of this case was carried out by a multidisciplinary team, which included professionals specialized in pedodontics, endodontics, restorative dentistry, and periodontics.

The first stage of the treatment consisted of exposing and removing the carious lesion (Figure 2B) and restoring the vestibular area of the transfixed tooth number 9. First, a periodontal flap procedure was carried out to expose the affected area, and a rubber dam was used to facilitate visualization without interfering with the biologic space (Figure 3A,B,C). After complete removal of the caries, a physical barrier was made with restorative composite resin (NT premium, Vigodent, Rio de Janeiro, RJ, Brazil) on the vestibular wall, which allowed the necessary sealing to perform endodontic treatment. Since the tooth was fragile, the use of a clip was not indicated, and so, the rubber dam was used with the aid of a light-cured gingival protector Top Dam (FGM, Joinvile, SC, Brazil) (Figure 3D).

For the endodontic treatment, chemical-mechanical preparation with 5.25% hypochlorite and hand files was used to dissolve and remove the necrotic tissues. After removing the smear layer with 17% EDTA, obturation was performed using the lateral condensation technique (Figure 4A,B), and a plug was made below the cervical resorption area with gray MTA-Angelus (Angelus, Londrina, PR, Brazil) leaving sufficient space for the cementation of a glass fiber-reinforced No. 1 (Exacto, Angelus) using self-curing resin cement (Fill Magic Dual, Vigodent).

Fifteen days after the canal was obturated, the tooth became darkened in comparison with its homologue (Figure 5A), and professional external dental bleaching was applied using 35% carbamide peroxide gel (Whiteness, FGM) (Figure 5B) in two sessions with an interval of seven days between them (Figure 5B). After waiting 15 days for complete elimination of all residual oxygen, the esthetic restorative process was begun, through selective wear of vestibular enamel in order to perform a composite veneer. The resins A3.5, A3, and A2, transparent incisal with opaque incisal halo B1 resin, and dentin A2 resin, (NT Premium, Vigodent) were used to reconstruct the dental esthetics according to the treatment plan (Figure 6). After restoration was finalized and occlusion was assessed, occlusal wear, finishing, and initial polishing were carried out (Figure 7).

When the patient returned for reassessment, a need for mucogingival surgery was indicated due to the irregular gingival contour and disharmonious esthetics (Figure 8a). Therefore, a free connective tissue graft obtained from the mucosa portion of the palate measuring 5 mm $\times$ 10 mm was enveloped in the gingival contour of tooth number 9 (Figure 8B,C). The patient returned one week later to have the sutures removed and returned again after one month for gingival contour reassessment. Two weeks after the reassessment, finishing and final polishing of the restoration was done.

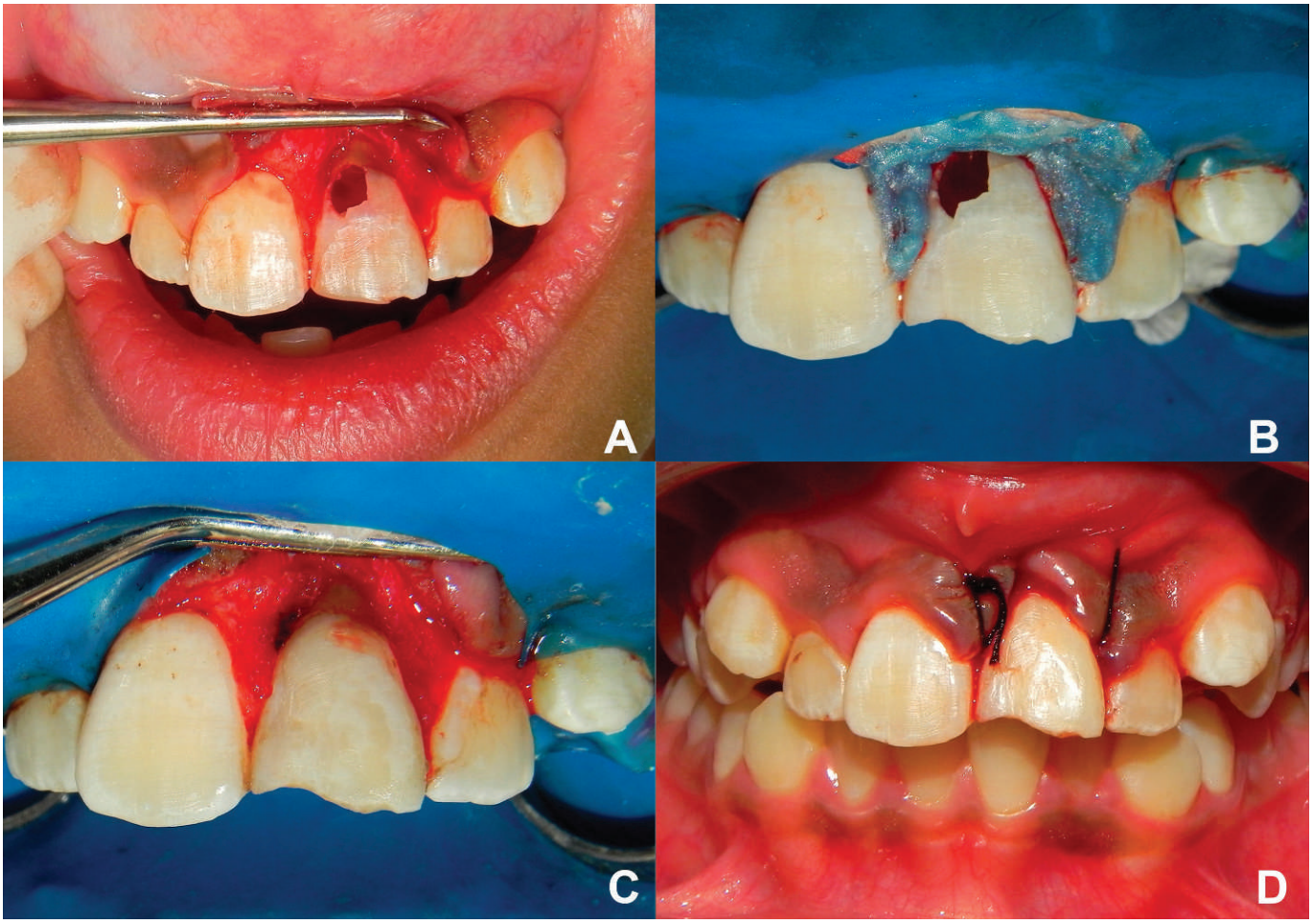

Figure 3. *(A): Surgical exposure to resorption. (B): Absolute isolation with rubber dam and gingival barrier. (C): Restoration with composite resin of the vestibular. (D): Repositioning gum.*

Along the treatment, the patient received instructions on basic care with hygiene and diet. Follow-up sessions with clinical and radiographic exams took place every three months. After one year of follow-up, no evidence of pulp or periapical pathoses or further resorption processes were found. Furthermore, the periapical lesion was regressing. The conservative multidisciplinary and low invasive treatment allowed preservation of the anterior tooth, which had been indicated for extraction by other professionals (Figures 9 and 10).

DISCUSSION

Significant structural loss of the dental crown associated with ECR in permanent maxillary anterior teeth may have functional and esthetic consequences that might lead to more severe psychologic problems.[2] Therefore, the need for an interdisciplinary approach for injured anterior teeth has been emphasized.[12] In the present case it became clear that without the participation of several specialists, the planned treatment would not have been possible.

Dental trauma is one of the main factors that predispose the development of ECR.[13] The etiology of resorption is possibly the result of an inflammatory response of the periodontal ligament to traumatic or bacterial stimulus.[14] In this case report, the development of ECR was possibly related to dental trauma associated with potential factors of orthodontic treatment. The difficulty of tooth brushing associated with a delay in the diagnosis and treatment of ECR favored the establishment and progression of a carious lesion leading to pulp necrosis and the development of a periapical lesion.

Invasive cervical resorption is difficult to diagnose, and it is even more challenging to identify the extent and nature of the process, especially in cases where the resorptive defect is buccal or palatine in

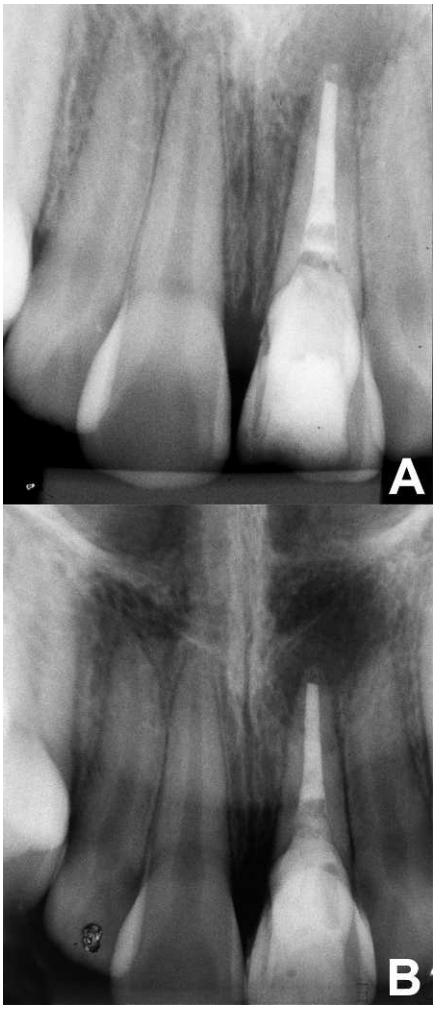

Figure 4. *(A): Radiograph after completion of endodontic treatment. (B): Radiograph after cementation of the glass fiber-reinforced.*

location.[15] When ECR is detected early, conventional and effective endodontic and restorative treatment is possible.[11] However, in severe cases of ECR associated with an extensive cavity, the clinical approach prior to endodontic treatment is complete removal of the existing carious tissue in order to make a more accurate assessment of the tooth. In cases of extensive destruction and lack of sustainable hard tissues, orthodontic traction or surgical exposure of the crown[16] is needed, such as the one performed in this case.

Pulp exposure caused by dentin resorption requires endodontic treatment,[11] which can be made by conventional therapy using gutta-percha filling or new materials such as mineral trioxide aggregate (MTA).[1,17] MTA presents biocompatible characteristics, bacteriostatic effects, and good sealing properties[18]; therefore, in the present case, we chose the

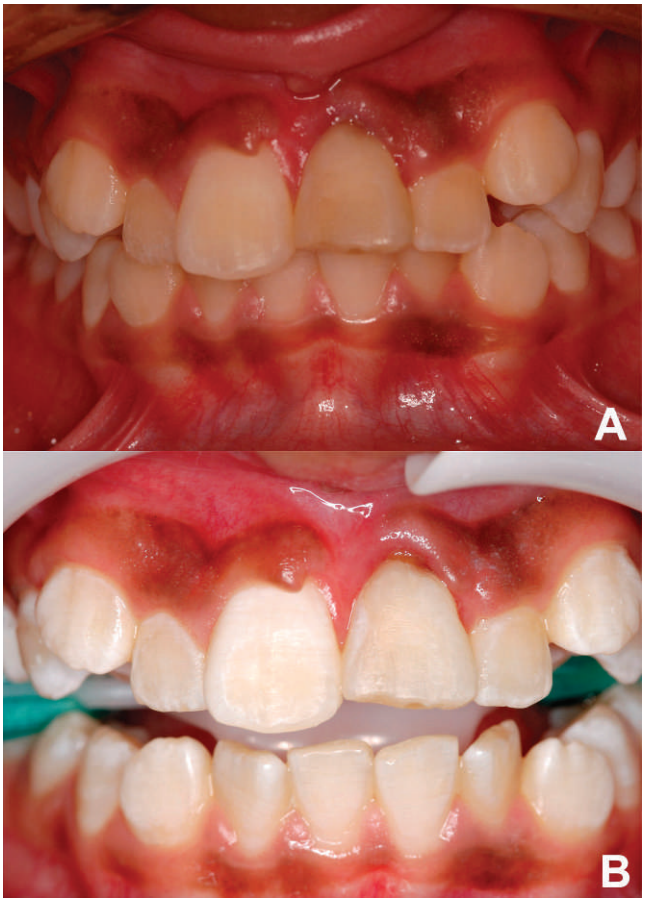

Figure 5. *(A): Darkening of the coronal portion after root canal filling. (B): Final appearance after bleaching.*

endodontic cement and gutta-percha with an MTA plug in the root middle third for the final obturation. Due to the properties of MTA and the fragile condition of the tooth, intracanal medication with calcium hydroxide was not used because it increases the length of clinical treatment and consequently the risk of fracturing the coronal portion.

Apical periodontitis is an inflammation and destruction of periradicular tissues caused by etiologic

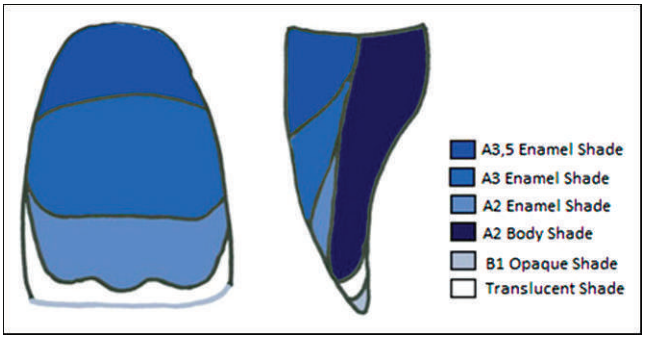

Figure 6. *Diagrams of the resins used.*

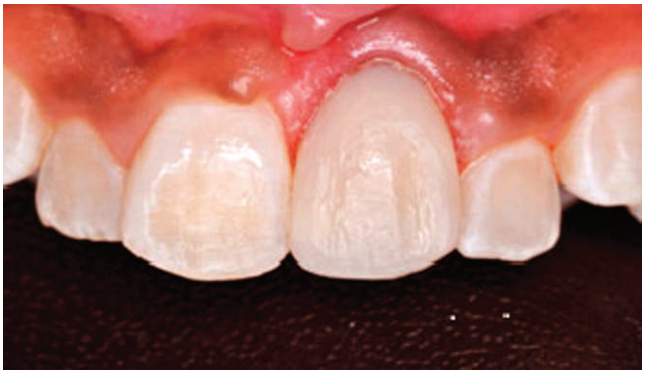

Figure 7. *Facet with composite resin.*

agents of endodontic origin. It is a sequel that is characterized by a radiolucent image in the root apex, similar to a periapical lesion. Its treatment consists of eliminating infection from the root canal.[19] This procedure was performed in the present case using the above-mentioned endodontic treatment with the purpose of reducing the lesion of the traumatized tooth.

One of the main causes of root canal treatment failure is associated with the contamination of the root system between the completion of endodontic treatment and the definite restoration.[20] However, in the present case, immediate restoration was not possible, so resin composite was used as a temporary restoration until completion of treatment. This choice was based on the characteristics of the material chosen, which in addition to presenting satisfactory sealing to infiltration from oral fluid contamination, also presented sufficient resilience and adhesiveness—desirable characteristics to prevent fracture of the coronal remainder. Maximum preservation of healthy tooth structures and the use of restorative materials with mechanical properties similar to the tooth favor greater longevity of complex tooth restorations.[21]

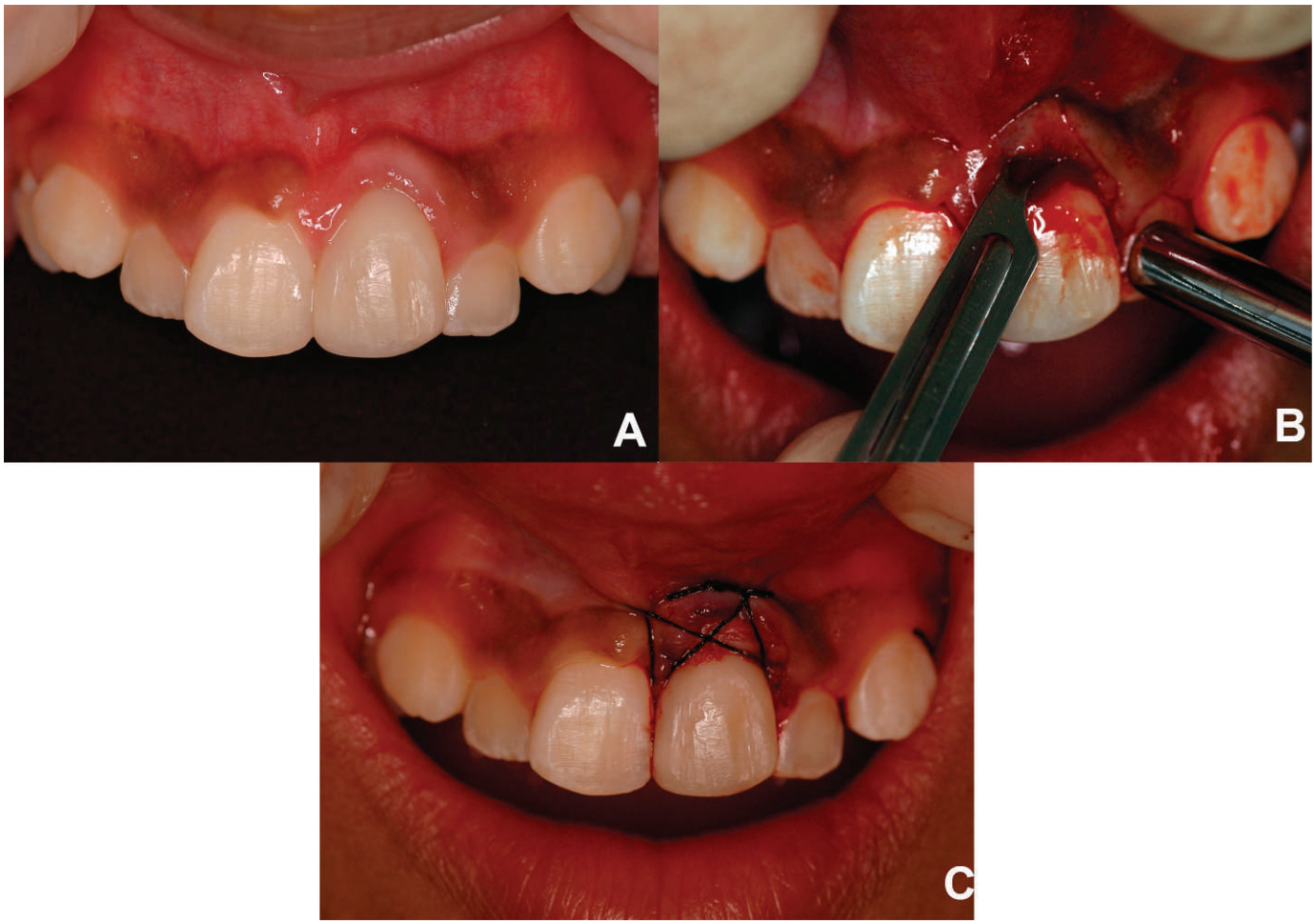

Figure 8. *(A): Irregular gingival contour upon returning for reassessment. (B): Periodontal surgery. (C): Replacement of the connective tissue graft removed from the palate.*

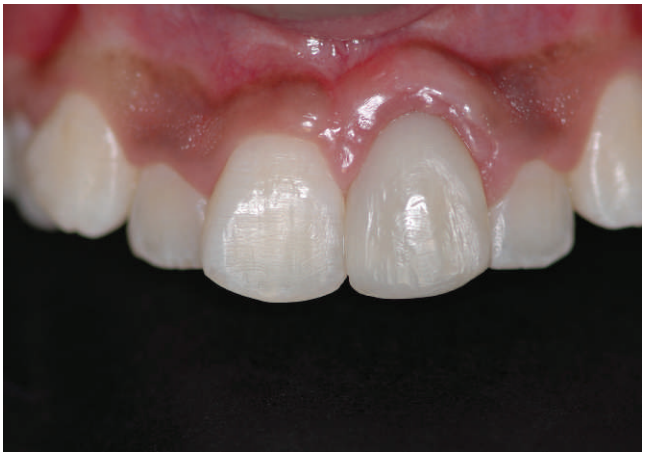

Figure 9. *Clinical aspect after one year of follow-up.*

The nano-composite selected to perform the final restoration had essential characteristics that enabled satisfactory esthetic results, provided high strength and consistency, and allowed ideal polishing to finalize the restoration. Moreover, a fluorescent agent is embedded in this material and so emits visible light when exposed to ultraviolet light. This gives a more natural shine and vitality under different lighting conditions, thus providing a satisfactory esthetic and harmonious smile. A radiopaque composite was chosen to improve the quality of the radiographic follow-up studies. The main purpose of the occlusal adjustment performed after the restoration was to control trauma resulting from the action of functional and parafunctional occlusal forces, eliminating interference, and favoring better long-term dental stability.[22]

After conservative treatment, the gingival contour of a traumatized tooth is usually altered, affecting esthetics. In the present case, the patient had gingival recession, and the recommended procedure was subepithelial connective tissue graft. The soft tissue grafts (gingival graft and subepithelial connective tissue graft) have been used successfully in periodontics for reconstructing areas showing gingival recession, loss of interdental papillae, and alveolar ridge-volume deficiency.[23] The connective tissue coming from the palate or gingiva is capable of inducing keratinization from epithelial cells proliferating on the subepithelial connective tissue graft at the receptor site.[24] However, for long-term success, it is important that there is adequate primary graft fixation, revascularization, and close contact between the graft and receptor.[25]

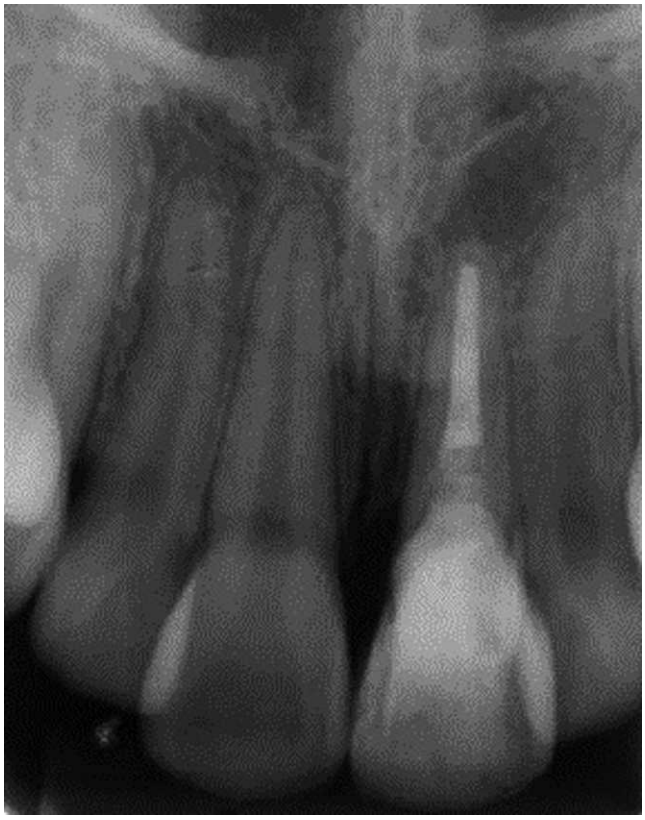

Figure 10. *Radiological aspect after one year of follow-up.*

Connective tissue graft has the same indications and predictability as free gingival graft. However, it has some advantages, mainly in relation to the postoperative period because it is more comfortable due to primary intention healing. In terms of esthetics, connective tissue graft is also superior because there is greater uniformity of color in relation to the tissues adjacent to the receptor site.[26] Therefore, due to those and other advantages, this was the technique chosen for the present case.

Children and adolescents who have suffered severe traumatic injuries in the anterior maxillary region with posterior injuries immediately suffer a decline in their quality of life and in their guardians' quality of life.[26] Therefore, strict criteria in planning and immediate procedures must be carried out effectively and satisfactorily to improve both the children's and guardians' quality of life. Clinical and radiographic follow-up sessions should be carried out periodically, and they are extremely important even after completion of treatment. Thus, future traumatic injuries may be avoided and a multidisciplinary team will be able to intervene efficiently at the right time.[27,28]

The multidisciplinary planning enabled successful esthetic rehabilitation of the smile in the patient with an anterior tooth with an unfavorable prognosis. This case followed conservative therapy for the tooth structure that remained after an extensive external cervical resorption and was associated with careful follow-up sessions. It is extremely important to preserve the injured tooth in young patients both from the psychologic aspect and to maintain function and esthetics.

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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Minimally Invasive Treatment for Esthetic Management of Severe Dental Fluorosis: A Case Report

Y Wang • Y Sa • S Liang
T Jiang

Clinical Relevance

The proposed technique may be a promising alternative to restorative treatment of teeth with severe dental fluorosis.

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SUMMARY

Dental fluorosis is a developmental disturbance of enamel caused by excessive fluoride on ameloblasts during enamel formation. Patients often present to the dentist with a main goal of improving their esthetic appearance. This case report describes a minimally invasive technique for treating a severe case of enamel fluorosis with brown surface aspect and small defects. A selective mega-abrasion and microabrasion were used to recreate macro- and micro- surface morphology, followed by power bleaching, home bleaching, and resin infiltration to improve the esthetic appearance.

INTRODUCTION

Dental fluorosis (DF) is a developmental disturbance of enamel caused by excessive fluoride on ameloblasts during enamel formation.[1] Based on the excellent work of Dean *et al* in the 1940s,[2,3] a strong relation was found between fluoride concentration in

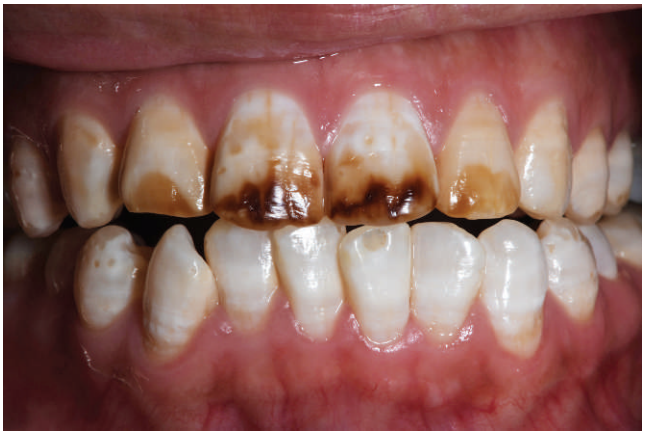

Figure 1. *Initial view of a patient with severe DF.*

drinking water and the prevalence and severity of DF.[4] Because of its important role in the prevention and control of dental caries, fluoride has also been added to milk and salt in recent decades.[5,6] As a result, clinicians are facing an increasing number of patients with DF.[6] DF affects the color and/or structure of enamel, which leads to an unpleasant esthetic appearance.

The treatment plan for DF depends on the severity of disease.[4,7] In cases of severe DF, invasive approaches, such as resin composite restorations, ceramic veneers, or even crowns, are generally chosen. However, the loss of tooth structure weakens its mechanical properties. Thus, especially for young patients, invasive procedures accelerate the destruction of tooth at an early age. Moreover, the aforementioned treatments are relatively expensive. For these reasons, more conservative approaches that are cheaper and less time consuming should be proposed for severe DF.

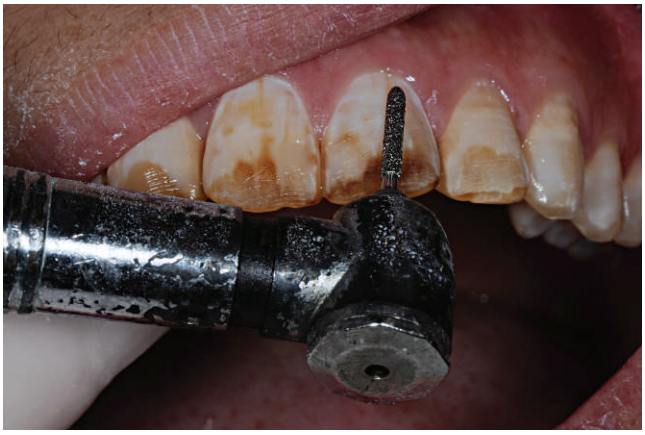

Figure 2. *Mega-abrasion performed with a 105-μm fine diamond bur.*

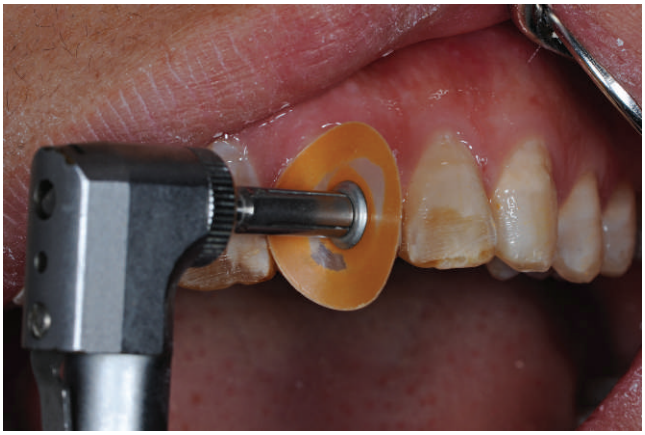

Figure 3. *Surface finishing. (Sof-Lex discs, 3M ESPE)*

The aim of this article is to introduce a minimally invasive treatment for improving the esthetic appearance of teeth afflicted with severe DF.

CLINICAL CASE REPORT

A 28-year-old woman came to our prosthodontics department to improve her dental esthetics. She had been experiencing severe discoloration and surface defects on the anterior teeth for more than 10 years. Clinical assessment revealed that the DF was severe and had a significant impact on the esthetic appearance of the teeth (Figure 1).

Considering the patient's young age, more conservative treatment was proposed to improve the condition of her teeth instead of the conventional restorative approaches. The proposed treatment was based on a combined approach of enamel abrasion, tooth bleaching, and resin infiltration for managing the enamel surface and periodontal scaling to prevent inflammation and improve the gingival status before the formal treatment.

First, mega-abrasion was performed using a high-speed handpiece with a 105-μm fine diamond bur (ML524, Diatech, Altstatten, Switzerland) to remove the superficial 200–400 μm enamel (Figure 2). After that, medium to fine abrasive discs (Sof-Lex, 3M ESPE, St Paul, MN, USA) were used to reshape the enamel surface and remove the sharp angles (Figure 3). Subsequently, a photopolymerizable resin dam (Beyond Technology Inc, Santa Clara, CA, USA) was applied (Figure 4), and a small amount of abrasive paste (Opalustre, Ultradent Products, South Jordan, UT, USA) containing silicon carbamide microparticle paste and 6.6% hydrochloric acid was painted on the affected teeth. The tooth surfaces were then micro-abraded using a specific rubber cup (Oralcups,

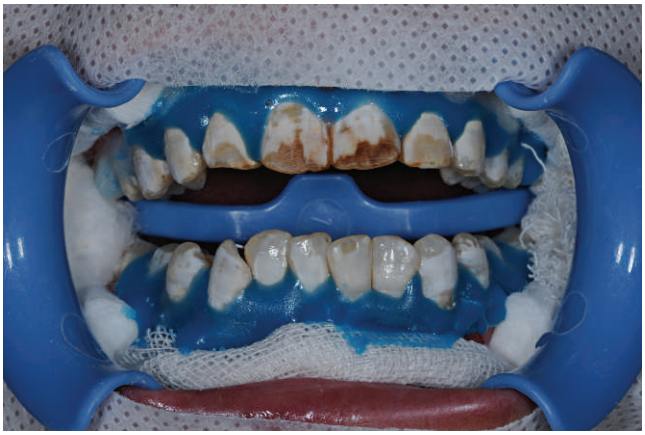

Figure 4. *Photopolymerizable resin dam. (Beyond Technology Inc)*

Ultradent Products) with slight pressure for about 120 seconds (Figure 5).

After microabrasion, an in-office bleaching agent (Opalescence Boost, 38%H_2O_2 Ultradent Products) was performed to alleviate the dark brown tooth color (Figure 6). Then a desensitizing agent (Fluorinated protector, Beyond Technology Inc) was painted and left undisturbed on the surfaces of bleached teeth for five minutes (Figure 7). Subsequently, agent removal with suction, thorough water rinsing, and removal of the photopolymerizable rubber dam were conducted (Figure 8). To better harmonize color, at-home bleaching was suggested to the patient. After applying eight syringes of 10% carbamide peroxide (Ultradent Products), the patient was satisfied with the bleaching effect (Figure 9).

Considering the hypomineralization structure of DF, a standard resin infiltration approach (Icon, DMG Products, Hamburg, Germany) was performed

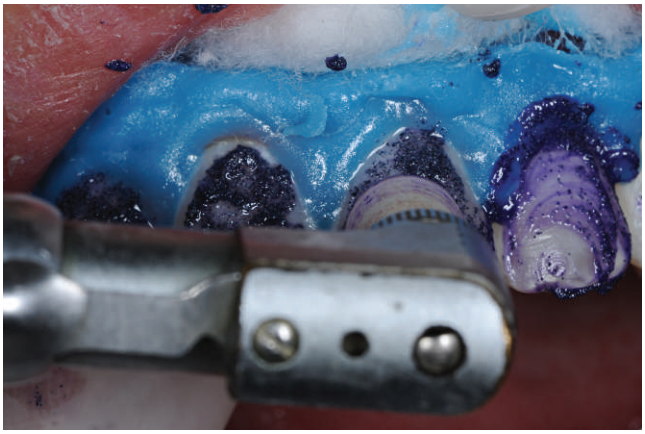

Figure 5. *Microabrasion. (Opalustre, Ultradent Products)*

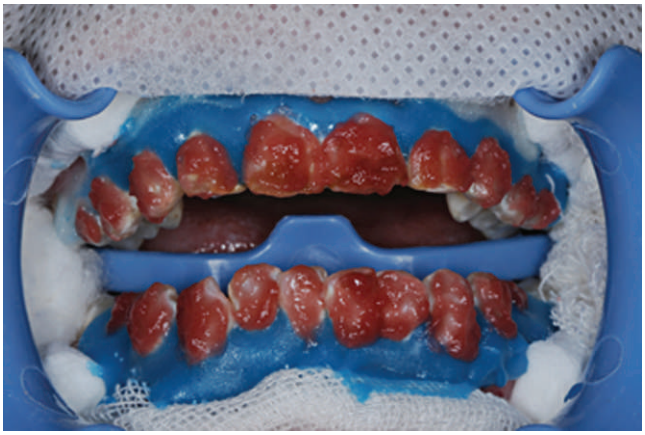

Figure 6. *Application of an in-office bleaching agent. (38% H_2O_2, Ultradent Products)*

to prevent potential enamel caries two weeks after at-home bleaching therapy (Figure 10). Finally, improvement of the esthetic appearance was achieved and remained stable until the 12-month follow-up (Figure 11).

DISCUSSION

The etiology of DF seems to be well known. Excessive consumption of fluoride during critical ages disturbs enamel mineralization, inhibits enamel apatite crystal growth, and interferes with the degradation of enamel matrix proteins, which results in a whitish-brown enamel aspect of the defect of enamel structure, depending on the severity of the DF.

Initial mega-abrasion was chosen to remove the superficial layer of fluoride enamel that displayed the most unesthetic color and defective structure. This procedure eliminated deeper stains in the

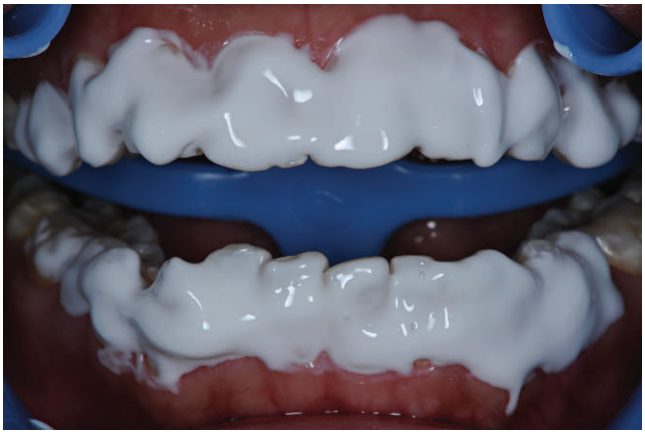

Figure 7. *Application of desensitizing agent. (Fluorinated protector, Beyond Technology Inc)*

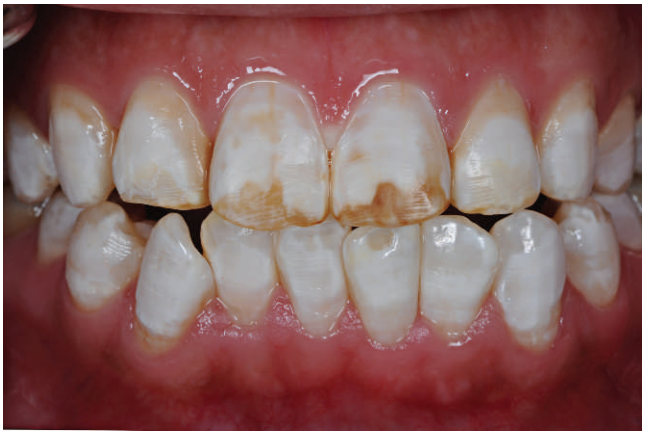

Figure 8. *Facial view after removal of the photopolymerizable rubber dam.*

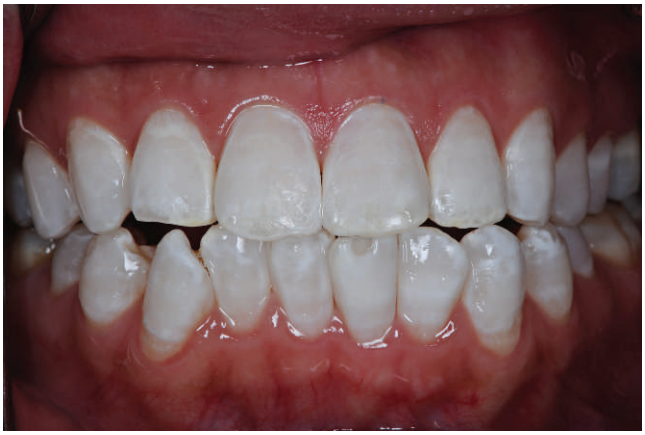

Figure 10. *Facial view after application of the resin infiltration. (Icon, DMG Products)*

enamel and minimized the clinical chair time.[6,8] It has been postulated that even 25% to 33% enamel reduction would probably be unrecognizable and clinically acceptable.[9] Therefore, well-controlled mega-abrasion could be considered as an acceptable approach in severe cases of DF without an unnecessary sacrifice of hard dental tissue.

To produce whiter teeth and harmonize tooth color, microabrasion and vital tooth bleaching were applied subsequent to mega-abrasion. In addition, a fluoride application was performed directly after the in-office bleaching to eliminate potential dental sensitivity.

The involvement of resin infiltration was a novel approach compared with previous DF treatments. The reasoning for this choice was based on the structure of fluorosed enamel and the properties of resin infiltration. In general terms, fluorosed enamel includes areas of diffuse hypomineralization and porosities in the subsurface enamel.[10] When the superficial layer of fluorosed enamel is removed by mega-abrasion and microabrasion, the subsurface enamel is exposed to the air. Then enamel pores act as pathways for bacterial and acid.[11] Thus, filling these pores would be an effective prevention therapy for potential caries. Because the resin infiltrant showed a very low viscosity, low contact angles to enamel, and high surface tensions, it could penetrate porosities and rapidly occlude the pathways.[12] Moreover, because of the similar refractive indices of resin and enamel, resin infiltration would improve the white opaque appearance of pores and reduce the original contrast between pores and enamel caused by light scattering,[13] thus leading to the esthetic appearance of enamel.

The proposed technique cost much less than conventional treatment approaches for severe DF. For instance, an all-ceramic veneer is about 10 times

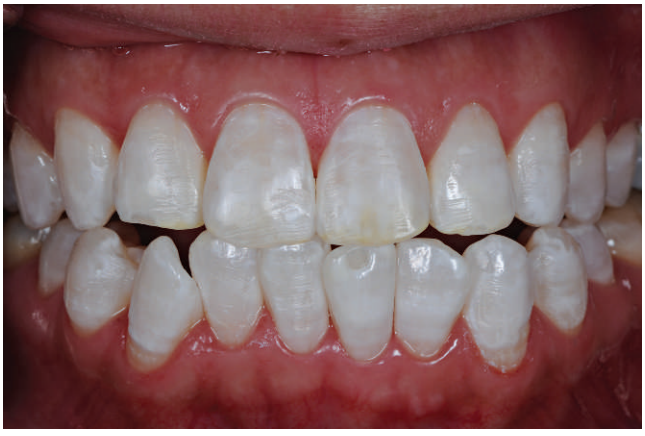

Figure 9. *Facial view after applying eight syringes of 10% carbamide peroxide. (Ultradent Products)*

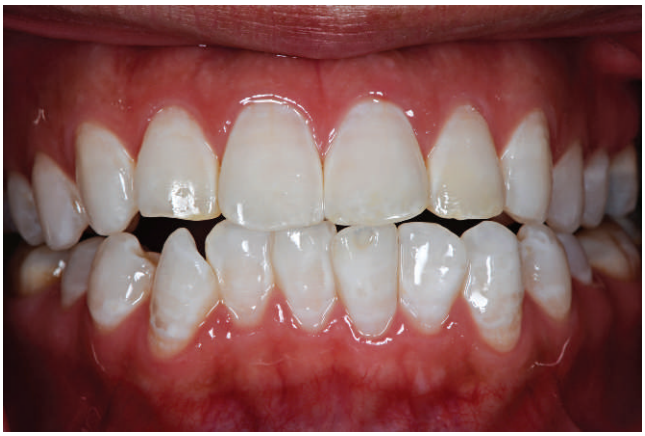

Figure 11. *Patient's appearance at the 12-month follow-up.*

more expensive than the proposed technique if considering the material cost and chair-side time.

CONCLUSIONS

Treatment with enamel abrasion followed by tooth whitening and infiltration application seems to be a minimally invasive therapy for severe DF. Moreover, this treatment is cheaper and less time consuming for patients.

Conflict of Interest

The authors of this article 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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Hypoplastic Enamel Treatment in Permanent Anterior Teeth of a Child

LD Carvalho • JK Bernardon • G Bruzi
MAC Andrada • LCC Vieira

Clinical Relevance

Hypoplastic enamel stain in anterior teeth can seriously compromise the esthetics of a smile. Knowing the etiology of the enamel deficiency is essential for determining the most appropriate treatment approach.

SUMMARY

In some patients with labial white stains involving the enamel and dentin, bleaching associated with a restorative procedure using composites may be an appropriate treatment alternative. Although bleaching makes the teeth and the stain whiter, the staining is less evident and easier to restore. Restorative procedures using adequate composites may then recover the natural optical properties while also providing appropriate mechanical properties, thereby ensuring the longevity of the treatment. In this article, the clinical case of a 9-year-old patient who reported dissatisfaction with her smile because of the presence of hypoplastic enamel staining at the central superior and inferior incisors is reported. The treatment consisted of a bleaching protocol followed by composite resin restorations using the stratification technique. The final esthetic result demonstrated the possibility of obtaining a natural smile with an adequate color and natural-looking restorations, thereby ensuring the esthetics and the patient's functional satisfaction.

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INTRODUCTION

Several factors may compromise esthetics in dentistry. In the case of enamel defects, hypoplastic spots may significantly affect the smile.[1]

Enamel hypoplasia results from incomplete or defective formation of the enamel organic matrix,

usually associated with genetic or environmental factors. It is a disorder caused by a dysfunction in enamel matrix secretion during the mineralization or maturation of this tissue.[2] When the cause of this condition is hereditary, the enamel malformations may come from defects in the genes that encode the proteins related to the mineralization process.[3] Thus, when that happens, there is involvement of both the primary and secondary dentitions in a generalized way. However, when environmental factors interfere in the process, the severity of the defects is directly related to the intensity and duration of the environmental stress and with the number of affected ameloblasts.[4,5] In those cases, the spots may be localized or generalized and may involve only enamel or enamel and dentin. Among the environmental factors that cause stains are some systemic disorders; viral diseases; nutritional deficiencies; trauma; ingestion of chemical substances, such as fluoride or some medicines; and even idiopathic causes.[6]

A dental professional must be prepared to make the right diagnosis of these changes to determine the appropriate treatment. A differential diagnosis with other lesions that cause staining should be performed, including whether the white spots came from caries lesions, trauma, or posteruption wear.[1]

Adequate planning may ensure a conservative, effective, and durable treatment. Sometimes dental bleaching may be a conservative alternative, able to achieve a good result, even though many times the spots do not disappear completaly.[6] The association of abrasion with an acid occasionally improves the result.[7, 8]

In lesions involving enamel and dentin, either with or without the loss of structure, direct restorations may be indicated. This approach may be the most conservative treatment, able to provide an excellent esthetic result and longevity. There is still the possibility of indirect restorations in more severe cases.[1,9-12]

Among the factors to determine the appropriate treatment, the patient's age is relevant. Invasive treatments should be avoided, especially in childhood. However, when the esthetic damages compromise the child's social life, restorative treatment is indicated and should be as conservative as possible. [9]

Composite resin is a restorative material alternative that may restore esthetics with high quality, minimal wear, and durability. The necessity of whitening before providing restorative care should be assessed in each patient, even at a young age. This procedure should be performed before restorative treatments using a low-concentration bleaching gel.

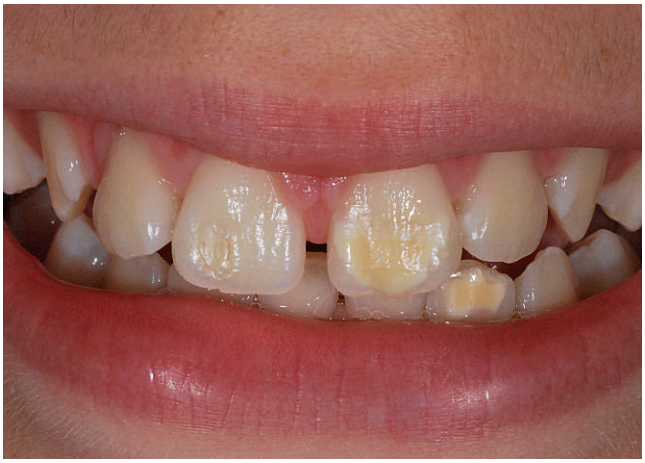

Figure 1. *The patient's smile before treatment. Observe the presence of hypoplastic white spots in the anterior teeth.*

CASE REPORT

A 9-year-old female patient was unsatisfied with her smile because of the presence of white spots on the labial surface of the central incisors (Figure 1). After anamnesis and clinical examination, the teeth were diagnosed as being naturally yellow, and the white spots in the middle and incisal thirds of the upper and lower incisors (teeth 11, 21, 32) were a consequence of trauma to the deciduous teeth, in which the primary tooth injured the growing permanent tooth. The stains were characterized as hypoplastic spots. In the radiographic analysis, no periodontal or periapical alterations were observed. The enamel surface texture was not altered (Figures 2 and 3). A transilluminator device was placed on the palatal surface of each tooth to evaluate the spot depth, allowing the light transmission analysis to define the hypoplastic defects (Figure 4). Thus, it was possible to identify the depth and intensity of the alterations in the dental structures. This technique aids in the treatment approach: the less the light propagation through the affected areas, the greater the depth of the stain. Clinically this is characterized by a high degree of opacity.

The stain opacity confirmed dentin involvement in this patient, necessitating invasive treatment with direct restorations using a composite resin. The amber pigmentation around the white spot complicated the color selection. A specific bleaching protocol was indicated to minimize the yellowness

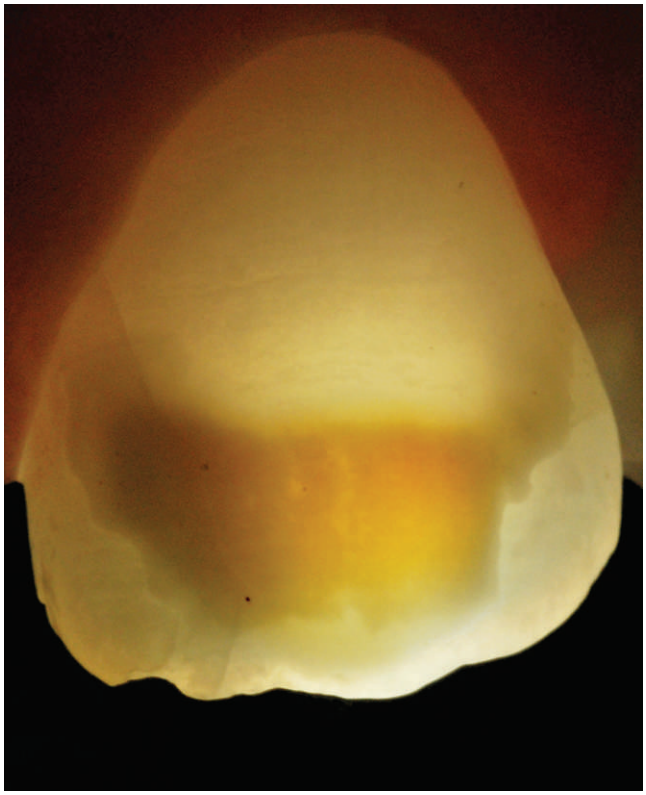

Figure 2. *Using a transilluminator device placed in the palatal surface, it is possible to view the depth of staining and the degree of opacity in the teeth involved.*

of the teeth and stain pigmentation. The tooth was isolated using a gingival barrier, and the 10% carbamide peroxide Whiteness Perfect 10 (FGM Produtos Odontológicos, Joinville, Brazil) was applied only to the affected area. As the patient had no sensitivity or any adverse effects, home bleaching was performed for one hour/day over a span of 14 days (Figure 5).

The mock-up of the restorations was performed seven days after completion of the bleaching. The

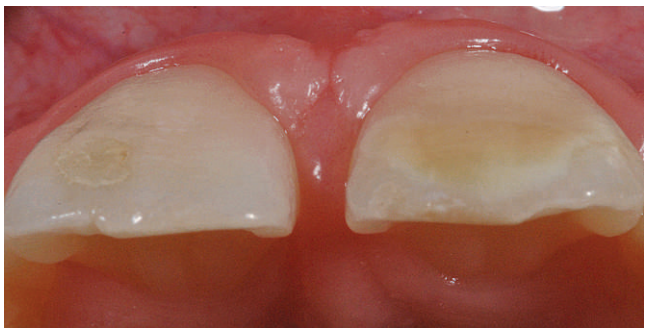

Figure 3. *Incisal view of the upper central incisors. Observe that the teeth are not altered.*

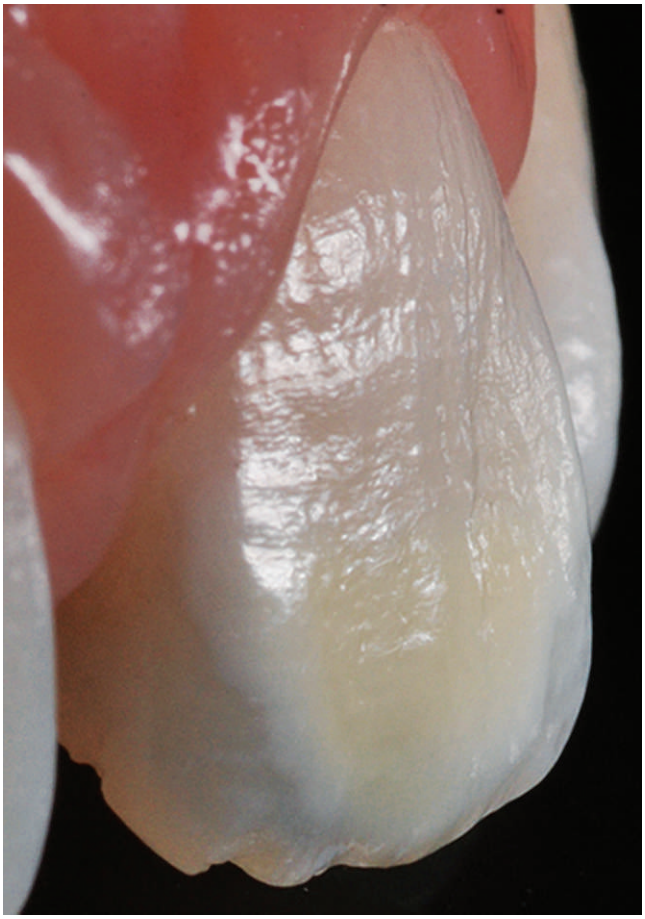

Figure 4. *Proximal view of the central incisors. Note that the surface texture of the teeth is not altered.*

Empress Direct Resin System (Ivoclar Vivadent, Schaan, Liechtenstein), with different levels of translucency, was selected to reproduce the optical effects of the enamel, dentin, and incisal edges.

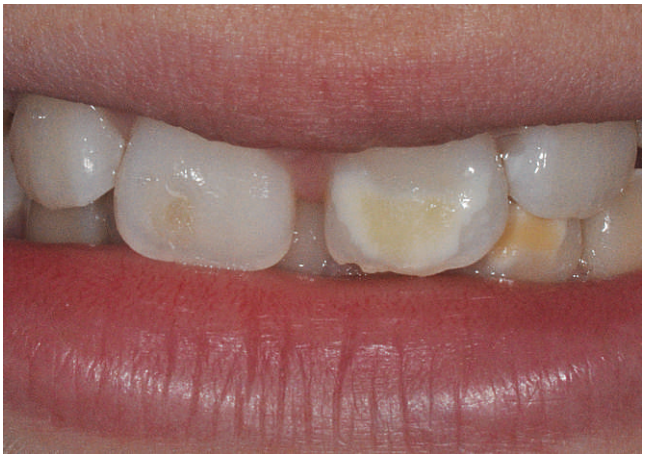

Figure 5. *The upper incisors after the bleaching treatment. The home bleaching was performed only for these four teeth. Observe the increase in color homogeneity.*

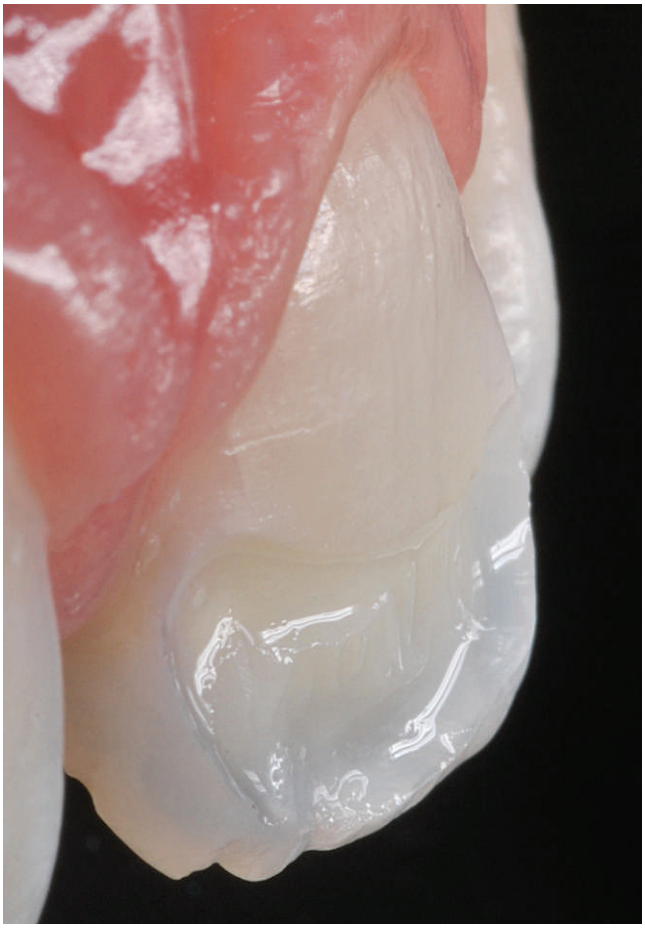

Figure 6. *The proximal view demonstrates the cavity depth. Note the exposure of dentin.*

After a prophylaxis, the enamel color was selected using the system scale positioned in the enamel not affected by the stain. Both the tooth and the color scale were hydrated and natural light was used.

Because the enamel macro- and microstructures were not altered, an acrylic resin matrix was made to copy the enamel surface, ensuring the reproduction of the original surface texture. This matrix was prepared by applying Vaseline (ADV Farma, São Paulo, Brazil) and saturated duralay acrylic resin spheres (Reliance Dental Mfg Co, Worth, IL, USA) on the buccal surface until autopolymerization was complete. The matrix was used at the mock-up and kept in water for the permanent treatment. The cavity was prepared using a diamond bur under water cooling. The whole depth of the hypoplastic stain was removed to eliminate the opacity difference between the affected and unaffected teeth (Figure 6). At this point, the dentin color selection was confirmed and the opalescent shade was selected.

The mock-up was conducted with the selected shades for each layer. Hybridization of dental tissues was performed only at one point at this time. After three days, the color restoration was observed to be correct, allowing the buildup of the final restorations.

The mock-up was removed and the rubber dam was used to restore definitely. The cavity was etched with 37% phosphoric acid for 15 seconds on dentin and 30 seconds on the enamel, extending 1 mm beyond the preparation margins. After rinsing, the dentin was protected with a cotton pellet and the enamel was air-dried. The adhesive system Single Bond 2 (3M ESPE, St Paul, USA) was applied, following the manufacturer's instructions, taking care to remove any excess, especially in the proximal surfaces. The cavity was photocured for 10 seconds.

The restoration was fabricated with composite resins from the Empress Direct kit (Ivoclar Vivadent, Schaan Liechtenstain). For the reconstruction of the artificial dentin, the shade A1D was used at the cavosurface angle associated with the shade B1D applied on the bottom of the cavity. The same composite resin was used to sculpt the dentinal mamelons. This layer was light polymerized for 40 seconds. To reproduce the opalescent effect, the shade trans-opal was placed over the tips of the mamelons, between the dentin composite and the incisal edge. This layer was light-polymerized for 40 seconds. The enamel was reproduced with a high translucent composite resin for bleaching teeth (shade EBL-L). A brush was used to smooth and adapt the composite at the cavosurface margins. Liquid Vaseline was applied on the inner surface of the acrylic matrix, which was placed on the restoration surface. The excess was removed, and the assembly was polymerized for 40 seconds. The enamel surface was adequately reproduced. The photo-polymerization was completed without the matrix.

The procedure was repeated for the other spots. After 24 hours, the surfaces were polished with flexible discs and rubber points. The natural tooth structure was successfully reproduced with the restorations (Figures 7 and 8).

DISCUSSION

Hypoplastic white spots on permanent teeth may be a result of several factors. In the clinical case reported, trauma with the deciduous dentition during the formation of the permanent tooth enamel likely caused the stains on the permanent teeth.

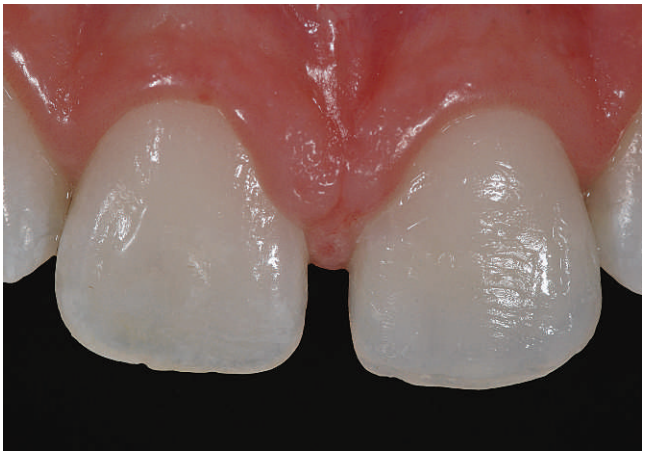

Figure 7. *The final restorations.*

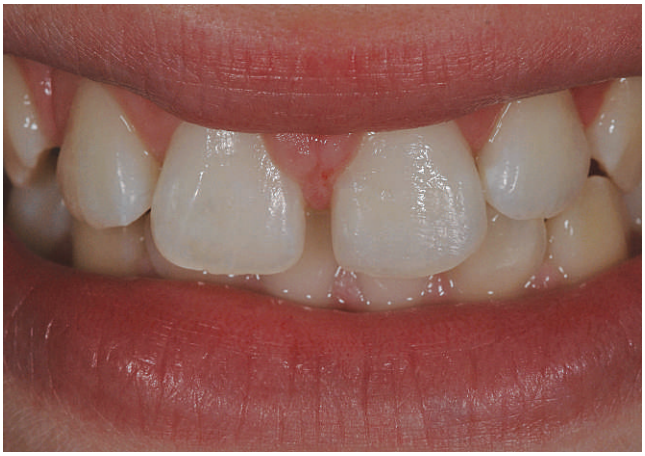

Figure 8. *The final smile.*

Such accidents, relatively common in the first dentition, mainly at the incisors, can cause defects on the surfaces of the permanent successors.[13,14] These stains presented an increased opacity and an amber pigmentation in the contour but no changes in the surface texture.

When localized to the anterior teeth, hypoplastic stains may have psychological and behavioral consequences as a result of the esthetic changes. This may influence the social life of the patient, as the defects alter the tooth structure and affect the appearance of the smile.[9]

It is known that some cosmetic procedures should be avoided and the invasive treatment of these defects should be postponed. However, the stains in the patient described here were generating great dissatisfaction, as the patient wanted to be a model. In less severe cases, bleaching could generate good results. Nevertheless, the enamel and dentin involvement was decisive for choosing the restorative treatment. The teeth had already completed eruption, an essential factor for the procedure.[6] Therefore, the composite restoration was chosen to be the more conservative option because it is associated with excellent mechanical, esthetic, and functional properties. However, the imminent difficulty in achieving perfect esthetics because of the amber pigmentation at the stain margin indicated the need for bleaching. In pediatric patients, it is known that a bleaching treatment should follow the policy determined by the American Association of Pediatric Dentistry (AAPD), which has defined the judicious use of bleaching for vital and nonvital teeth in children.

The procedure must be strictly controlled by the dentist, who should determine the appropriate method and timing of the treatment within the context of an individualized, comprehensive, and sequenced planning, while also considering the side effects of tooth whitening for children and adolescents. The AAPD does not indicate the total arch treatment for patients with mixed dentition.[15] Taking into account that a young patient's pulp is wider than that of an adult patient, and that the diffusion of hydrogen peroxide at high concentrations into dental tissues is extensive,[16-18] a safer treatment was selected for the current patient: 10% carbamide peroxide, reducing the possibility of sensitivity.[19-21] This treatment was performed in an individualized way for this patient: using direct applications on the more pigmented areas followed by application with personal trays for 14 days, and only with the affected teeth.

The mock-up was performed to ensure predictability of the result. By using the stratification technique, [22,23] the opaque shades restored the affected dentin and reconstructed anatomical structures, such as dentin mamelons. The characteristics of the natural dentition colors could be reproduced with the effect shades, reproducing the translucency of the incisal edge, which generally reflects a blue or gray color. When applied on the dentin composite, the incisal edge shade generates a counter-opalescent effect, reflecting an orange hue. A translucent composite was applied on the surface. It is important to consider that, during stratification with composites, the resin enamel thickness must be thinner than the natural enamel to prevent a reduction in the restoration value. Thicker layers of enamel resin generate more gray restorations because of the difference of the refractive index between the resin and the natural tooth.[24] Therefore, each step of the treatment is essential for a successful outcome: diagnosis, planning, and execution.

CONCLUSION

In the cases of hypoplastic spots on anterior teeth, in which color change and increased opacity affect the smile, the use of a combined treatment of bleaching and composite resin restorations can improve the appearance and uniformity of the teeth, restoring the patient's self-esteem. A correct diagnosis of the lesion depth is necessary for proper planning and to ensure a predictable outcome and success in the short and long term.

Conflict of Interest

The authors of this article 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 Research

A 48-month Clinical Evaluation of Fissure Sealants Placed With Different Adhesive Systems

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Clinical Relevance

Etch-and-rinse adhesive systems can be a better choice for ensuring the long-term success of fissure sealants.

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SUMMARY

Aim: **To compare the retention rates of a nanofilled occlusal fissure sealant placed with the use of an etch-and-rinse or a self-etch adhesive over 48 months.**

Materials and Methods: **The authors enrolled 244 teeth, each with no restoration or sealant and no detectable caries, from 16 patients. The sealants were placed with Solobond M two-step etch-and-rinse adhesive or Futurabond NR one-step self-etch adhesive by four previously calibrated dentists using a table of random numbers. After completion of the adhesive application, a nanofilled sealant, Grandio Seal, was applied and light-cured. Two other calibrated examiners, who were unaware of which adhesive had been used, independently evaluated the sealants at baseline and at 12-, 24-, 36-, and 48-month recalls. Each sealant was evaluated in terms of caries formation being present or absent and retention using the following criteria: 1 = completely retained, 2 = partial loss, and 3 = total loss. The**

Pearson χ^2 test was used to evaluate differences in retention rates among the sealants used with different adhesives for each evaluation period.

Results: **The retention rates for sealants in the Solobond M group were significantly higher than those in the Futurabond NR group in all periods of evaluation ($p<0.05$). No statistically significant difference between the retention rates for premolars and molars was found at each evaluation period ($p>0.05$). There was no new caries formation throughout the 48-month recall period.**

Conclusion: **Fissure sealants placed with etch-and-rinse adhesive showed better retention rates than those placed with self-etch adhesive.**

INTRODUCTION

Dental caries is a global public health problem that can be effectively prevented and controlled through a combination of individual, community, and professional efforts.[1] Although only 12.5 percent of all tooth surfaces are occlusal, most of the total caries experienced by children and adolescents proceeds from occlusal caries.[2] As a result of the morphology of the pit and fissure surfaces there are stagnation areas, where the plaque formed is anatomically protected from even a single toothbrush filament by the dimensions of the fissure.[3] Because preventive approaches, such as control of bacterial plaque and topical applications of fluoride, have little effect on pits and fissures, more effective treatments are necessary.[4] Application of pit-and-fissure sealants are one of the treatment modalities that has been shown to be very effective in preventing occlusal caries by physical obstruction of the pits and fissures.[5,6] Therefore, the clinical effectiveness and success of sealants have been equated with their retention.[7] If the sealant is fully retained, then recurrent caries or progression of caries beneath the restoration is negligible.[5] To enhance the longevity of pit-and-fissure sealants, several materials and techniques have been developed, including the use of adhesive systems under sealants.[8,9]

Bonding to enamel with the etch-and-rinse system is a reliable technique. Phosphoric acid etching removes contaminants and creates an irregular microporous enamel surface that is infiltrated by the resin-based sealant material. It has been reported that the highest bond strengths to human enamel were obtained using phosphoric acid etching and adhesives underneath the sealants.[10,11] However, the taste, rinsing, and suction associated with the phosphoric acid etching stage may be unpleasant for patients.[12]

Self-etch adhesive systems have been developed to simplify the bonding procedures, which significantly reduces the clinical application time and technique sensitivity because the enamel/dentin acid etching, rinsing, and drying steps are eliminated.[13,14] Because they have fewer operative steps and a shorter chairtime, self-etch adhesives may also be advantageous for treating pediatric patients. However, previous studies[15,16] have reported that their adhesive performance with unground enamel is challenging. The literature includes limited documentation comparing the effects of etch-and-rinse and self-etch adhesive systems on clinical performance of pit-and-fissure sealants, and the results are contradictory. Although some studies[16–18] recommended the use of etch-and-rinse systems, one study reported that etch-and-rinse and self-etch systems cause similar results in terms of retention *in vivo*.[12]

In 2009, we reported detailed information about the clinical performance of fissure sealants placed with different adhesives for a 24-month period.[18] As there is little information about the effectiveness of a self-etch adhesive application before fissure sealant placement, the aim of this clinical study was to compare the retention rates of a nanofilled occlusal fissure sealant placed with the use of an etch-and-rinse adhesive or a self-etch adhesive after a longer follow-up period.

MATERIALS AND METHODS

The protocol and consent form for this study were reviewed and approved by the Hacettepe University Human Ethics Committee. Written informed consent for involvement in the study was obtained from all patients.

A total of 16 patients (15 women and 1 man) who were seeking routine dental care at the conservative dentistry clinics at the Hacettepe University, Faculty of Dentistry, were selected. Patients who participated in the current study had good general and oral health and hygiene. They also had no detecatable caries, bruxism, malocclusion, previously placed restorations or sealants on the fissures, or allergies to resins. The mean age of the patients was 20 years, and the patients ranged in age from 18 to 21 years.

Bitewing radiographs were taken. The fissures of teeth were then cleaned with a slurry of pumice applied with a bristle brush in a slow-speed

Table 1: *Distribution of Sealant Retention Rates*

Evaluation[a]	12 Months		24 Months		36 Months		48 Months	
	Solobond M	Futurabond NR	Solobond M	Futurabond NR	Solobond M	Futurabond NR	Solobond M	Futurabond NR
1 (No. [%])	109 (89.3)	25 (20.5)	93 (81.6)	18 (15.8)	86 (75.4)	13 (11.4)	82 (71.9)	10 (8.7)
2 (No. [%])	8 (6.6)	16 (13.1)	12 (10.5)	12 (10.5)	19 (16.6)	12 (10.5)	20 (17.5)	11 (9.6)
3 (No. [%])	5 (4.1)	81 (66.4)	9 (7.9)	84 (73.7)	9 (7.8)	89 (78)	12 (10.5)	93 (81.5)
Total No.	122	122	114	114	114	114	114	114
P value	<0.001		<0.001		<0.001		<0.001	

[a] *1, Completely retained; 2, partial loss; 3, total loss.*

handpiece to remove salivary pellicle and any remaining plaque. By using a table of random numbers, four previously calibrated dentists placed a total of 244 sealants on the permanent premolars and molars with either Solobond M (Voco, Cuxhaven, Germany), a two-step etch-and-rinse adhesive, or Futurabond NR (Voco), a one-step self-etch adhesive. After completion of the adhesive application, a nanofilled sealant, Grandio Seal (Voco), was applied and gently teased through the fissure with the tip of a periodontal probe to prevent voids and air entrapment. Then, the applied fissure sealants were polymerized using a quartz-tungsten-halogen light (Hilux, Benlioglu, Ankara, Turkey). Light output of the curing unit was found to exceed 550 mW/cm^2 before and after the study, as verified with a radiometer. The occlusion was checked with articulation paper. Finishing and polishing were performed using fine-grit diamond burs (Diatech, Swiss Dental, Heerbrugg, Switzerland) and rubber cups (Edenta AG, AU SG, Switzerland). All of the materials were used according to the manufacturers' instructions, and moisture control was maintained by use of adapted cotton-roll isolation procedures and a chairside assistant.

Two other calibrated examiners (ARY, MB), who were unaware of which adhesive had been used, independently evaluated the sealants with the aid of a dental explorer and an introral mirror. At the beginning of the study, Kappa values were calculated to test the intra- and interexaminer reproducibility. The Kappa values were high (0.95) and showed powerful intra- and interexaminer agreement. Each sealant was evaluated in terms of caries formation as present or absent, and retention was evaluated using the following criteria:

1) Completely retained (CR)
2) Partial loss (PL)
3) Total loss (TL)

The Pearson χ^2 test was used to evaluate differences in the retention rates of the sealants used with different adhesives for each evaluation period at a 5% level of significance.

RESULTS

Sixteen patients participated in this clinical study. The distrubution of sealant retention rates are displayed in Table 1. Differences between the retention rates of fissure sealants placed with Solobond M and Futurabond NR were statistically significant for all periods of evaluation ($p<0.05$).

After 12 months, 134 fissure sealants of the 16 patients were completely retained. The retention rates of sealants placed with Solobond M and Futurabond NR were 89.3% and 20.5%, respectively.

At the 24-month recall, one patient with 16 sealants could not be evaluated because of relocation to another city. Therefore, 228 sealants of 15 patients were available for evaluation. 111 teeth were fully sealed with the fissure sealant. The retention rate of the Solobond M group was found to be 81.6%, whereas it was 15.8% for the Futurabond NR group, as previously reported.[18]

After 36 months, the retention rates of sealants placed with Solobond M and Futurabond NR were

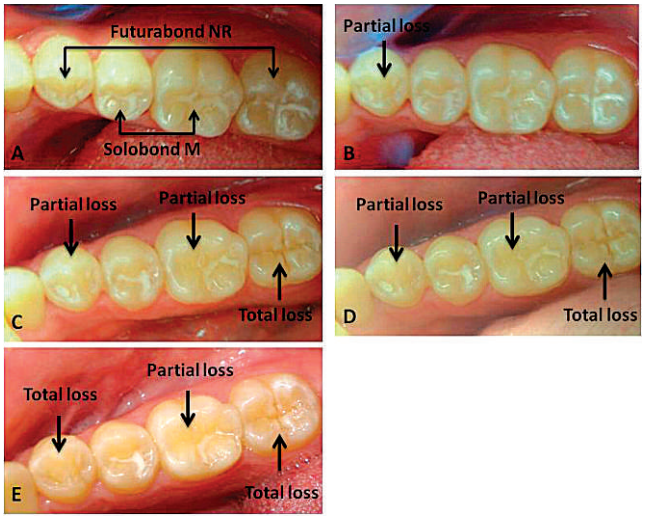

Figure 1. *Fissure sealants at baseline (A), at 12 months (B), at 24 months (C), at 36 months (D), and at 48 months (E).*

75.4% and 11.4%, respectively. There were nine total losses with Solobond M and 89 total losses with Futurabond NR.

At the end of 48 months, 82 sealants from Solobond M group and 10 sealants from Futurabond NR group were evaluated as completely retained; the retention rates for the groups were 71.9% and 8.7%, respectively. Twelve sealants from the Solobond M group were totally lost throughout the study; 93 were totally lost in the Futurabond NR group. Twenty sealants from the Solobond M group and 11 sealants from the Futurabond NR group were partially lost (Figure 1).

Distrubution of retention rates of premolars and molars are shown in Tables 2 and 3. No statistically significant differences were found between the retention rates of premolars and molars at each evaluation period. We did not observe caries development on any of the teeth during the 48 months.

DISCUSSION

Fissure sealants can be used for caries prevention in at-risk, caries-free teeth and as therapy for carious lesions limited to enamel (incipient caries). They are also appropriate as a conservative restoration in many situations in which caries extends into the dentin. This means that not only children, on which most sealant studies have focused, but also adults with appropriate indications can benefit from their use.[19,20] Nevertheless, clinical trials involving children are hard to perform, as sealants are very technique sensitive. Success of the follow-up is also dependent on parent cooperation and motivation to bring the child for follow-up. Therefore, we evaluated sealant retention rates in adults.

The extent and depth of the etching pattern logically should influence the bonding performance of an adhesive, as enamel bonding is primarily based on micromechanical interlocking of a low-viscosity resin into microporosities.[21] The depth of the enamel surface removed during the etching procedure can be affected by a number of factors, such as the type and concentration of acid, the duration of etching, and the chemical composition of the surface.[22,23] It has been demonstrated that the application of a one-step self-etch adhesive did not create a deep enamel

Table 2: *Distribution of Sealant Retention Rates of the Solobond M Group for Premolars and Molars*

Evaluation[a]	Solobond M							
	12 Months		24 Months		36 Months		48 Months	
	Premolar	Molar	Premolar	Molar	Premolar	Molar	Premolar	Molar
1 (No. [%])	54 (49)	56 (51)	47 (50.3)	46 (49.7)	40 (46.5)	46 (53.4)	37 (45.1)	45 (54.8)
2 (No. [%])	1 (12.5)	7 (87.5)	2 (16.7)	10 (83.3)	8 (42.1)	11 (57.8)	8 (4)	12 (6)
3 (No. [%])	3 (75)	1 (25)	5 (55.5)	4 (44.5)	6 (66.6)	3 (33.3)	9 (75)	3 (25)
Total no.	58	64	54	60	54	60	54	60
P value	>0.05		>0.05		>0.05		>0.05	

[a] *1, completely retained; 2, partial loss; 3, total loss.*

Table 3: *Distribution of Sealant Retention Rates of the Futurabond NR Group for Premolars and Molars*

Evaluation[a]	Futurabond NR							
	12 Months		24 Months		36 Months		48 Months	
	Premolar	Molar	Premolar	Molar	Premolar	Molar	Premolar	Molar
1 (No. [%])	14 (56)	11 (44)	10 (55.5)	8 (44.5)	5 (38.5)	8 (61.5)	4 (40.0)	6 (60.0)
2 (No. [%])	5 (31.2)	11 (68.7)	4 (33.3)	8 (66.7)	5 (41.7)	7 (58.3)	3 (27.3)	8 (72.7)
3 (No. [%])	39 (48.1)	42 (51.9)	40 (47.7)	44 (52.3)	44 (49.4)	45 (50.6)	47 (50.5)	46 (49.5)
Total No.	58	64	54	60	54	60	54	60
P value	>0.05		>0.05		>0.05		>0.05	

[a] *1, completely retained; 2, partial loss; 3, total loss.*

etching pattern compared to those of phosphoric acid.[24,25] Dos Santos and others[26] evaluated the penetration of adhesive materials into enamel before the application of a pit-and-fissure sealant and reported that etching with phosphoric acid exhibited significantly greater penetration than enamel treated with a self-etch adhesive. Beloica and others[27] have reported that the microshear and microtensile bond strength to intact enamel of the recently introduced all-in-one adhesives was inferior to that of an etch-and-rinse system. Various studies have also indicated the potential benefit of additional phosphoric acid etching of enamel before application of a self-etch adhesive.[28,29] Luhrs and others[30] showed significantly increased shear bond strength values to enamel with the addition of phosphoric acid etching to self-etch adhesives. Another study also reported that pre-etching the intact enamel with 37% phosphoric acid resulted in the formation of longer resin tags and a higher depth of penetration of the resin tags of the self-etch adhesive (Clearfil SE bond); it also attained a higher bond strength to intact enamel.[31]

Consistent with our 24-month results, the sealant retention rates were higher for the Solobond M group at the 36- and 48-month recalls. In accordance with these findings, Venker and others[32] reported that at the end of their 12-month clinical study, sealants placed with self-etch adhesives had lower retention rates compared with sealants placed with phosphoric acid etching. In another clinical study, the effects of a self-etch adhesive system and a conventional acid etching on retention of a fissure sealant were compared.[17] It has been found that at the end of a 12-month period, the retention of the acid-etch group was significantly superior to that of the self-etch group. They concluded that the best practice for placement of sealants remains enamel preparation with acid etch and use of an intermediate bonding layer.[17]

Contrary to the findings of the current study, Feigal and Quelhas[12] reported similar sealant retention rates using Prompt-L-Pop adhesive and conventional phosphoric acid etching without the use of any bonding agent *in vivo*. However, the results cannot be directly compared with our results, as no adhesive system was used in conjunction with the phosphoric acid. Moreover, it has been reported that the pH of Prompt-L-Pop was approximately one and was almost as aggressive as conventional phosphoric acid etching.[24] Moura and others[33] demonstrated a correlation between the pH of the adhesive systems and the level of morphological alterations of the enamel surface. Recently, it has been demonstrated that self-etch systems with higher pH values (AdheSE and Clearfil SE Bond) can have increased bond strength values when the application time is doubled.[34] They found a significant correlation between pH and mean bond strengths. The low retention rates observed with Futurabond NR at each evaluation period of the current study may be related to the pH of the adhesive (pH=1.4), which is considered to be a mild self-etch primer. This may have caused insufficient etching and deficient resin penetration of the self-etching priming agents into the fissure enamel.

Nevertheless, achieving a sufficient etching pattern on unground enamel remains a problem for self-etching adhesives.[15] The intact enamel surface is prismless, is hypermineralized, and contains more inorganic material than the inner enamel layer.[35] By using etch-and-rinse adhesive systems, the prismless enamel surface layer is removed because of the phosphoric acid etching and subsequent water rinsing of the etched enamel. Therefore, sufficient microretentive bonding of the fissure sealant can be provided by the exposure of the prismatic structured enamel. In contrast, treatment with self-etching priming agents does not remove a significant amount of the prismless enamel surface layer, as no rinsing takes place after application of the primer.[15,21] It is possible that the prismless enamel surface layer prevents the permeation of self-etching primers, thus leaving some areas partially unetched.[15] It has recently been shown that self-etching primers produce high-tensile bond strengths when enamel is roughened but lower tensile bond strengths when enamel is left unprepared.[15,16]

Before acid etching and sealant application, it is important to make sure that the fissures are free from plaque and debris, which may influence the etching and sealing pattern.[36] In the current study, prophylaxis was performed with pumice before the sealant placement. The remaining pumice and debris in the fissures could be another reason for the lower retantion rates of the Futurabond NR group, as its etching capacity is not high enough to remove remnants from the fissures. However, the etching capacity of phosphoric acid has been reported to be high enough to remove those remnants.[36]

Studies on sealant retention by tooth type report that premolars have the highest sealant retention rates and second molars have the lowest.[37–39] However, no statistically significant difference was found between the retention rates for premolars and molars in the current study.

CONCLUSION

This clinical study has demonstrated that, over a 48-month period, fissure sealants placed with an etch-and-rinse adhesive showed significantly higher retention rates than those placed with a self-etch adhesive. Further clinical studies are needed to confirm the reproducibility of these findings.

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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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A Retrospective Clinical Study of Cervical Restorations: Longevity and Failure-Prognostic Variables

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Clinical Relevance

The longevities of resin composite (RC) and glass ionomer (GI) used for cervical restorations were statistically not different, but the clinical performance of the RC was superior to GI in retention, marginal adaptation, and marginal discoloration.

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SUMMARY

The aim of this retrospective clinical study was to compare the longevity of cervical restorations between resin composite (RC) and glass ionomer (GI) and to investigate variables predictive of their outcome. The clinical performance of the two restorative materials in function was compared using the ratings of the modified United States Public Health Service (USPHS) criteria. A total of 479 cervical restorations were included in the study. Ninety-one already-replaced restorations were reviewed from dental records. The other 388 restorations still in function were evaluated according to the modified USPHS criteria by two investigators. Longevity and prognostic variables were analyzed with the Kaplan-Meier survival analysis and multivariate Cox proportional hazard model. The clinical performances of the two materials were evaluated according to the ratings of the USPHS criteria and com-

pared using the Pearson chi-square test and Fisher exact test. The longevity was not significantly different between RC and GI (median survival time, 10.4 ± 0.7 and 11.5 ± 1.1 years, respectively). The main reasons for failure were loss of retention (82.2%) and secondary caries (17.8%). The longevity of cervical restoration was significantly influenced by tooth group and operator group (Wald test, $p<0.05$), while material, gender, presence or absence of systemic diseases, arch, and reason for treatment did not affect the longevity. Contrary to the longevity, the clinical performance of RC was superior to GI in the criteria of retention, marginal discoloration, and marginal adaptation, but similar in secondary caries, wear, and postoperative sensitivity.

INTRODUCTION

In the clinic, dentists usually select restorative materials based on properties such as esthetics, physical strength, handling characteristics, biocompatibility, and wear resistance. As cervical lesions seem to be more frequently observed on the buccal surfaces of premolars and molars, tooth-colored restoratives should be considered as the materials of choice for restoration of cervical lesions.[1] Accordingly, those materials typically include resin composite (RC) and glass ionomer (GI, in this study includes conventional glass ionomer cement and resin-modified glass ionomer).[2] In general, RC has been the material of choice for cervical lesions due to superior esthetics, adequate strength, and versatility.[3] However, restoring cervical lesions with RC has several technical difficulties that can affect the clinical results. Difficulties in isolation, difficulties in adhesion to dentin margin, and polymerization shrinkage stress of RC make the restorative procedures very sensitive to the operator's technique.[4-6] Compared to RC, glass ionomers have been selected by virtue of adhesion to the tooth structure and fluoride release.[2,7,8] The GI restorative technique is relatively easy compared to that of RC restoration. However, esthetic results and mechanical properties of GI restorations are inferior to those of RC restorations.[2,9]

Even with the elastic bonding concept based on laboratory studies, the clinical longevity of cervical RC restorations was not affected by the stiffness of adhesive and RC.[10-12] Higher GI retention rates have also been attributed to laboratory observation of diffusion-based adhesion to calcium ions in dentin, as well as the low modulus of elasticity.[2] In non-prepared noncarious cervical lesions where direct occlusal force was not applied, the retention rate of restorations filled with various GIs was not shorter than those filled with RC, based on the results of short-term prospective studies within three years of restoration.[3,13-16] However, according to the United States Public Health Service (USPHS) criteria, the clinical performance of RC was superior to resin-modified glass ionomer cement and polyacid-modified resin composite.[3,13-15] Since the prognosis of cervical restoration may be greatly affected by various factors related to the material, patient, and the environment, it is difficult to predict the prognosis of restorations with laboratory results only. Clinical studies are needed to provide clinicians with predictive information on restorative materials and their prognostic variables.

With this retrospective clinical study, we investigated the longevity and prognostic variables of cervical restorations filled with RC and GI, which were retained with hybrid layer mechanical adhesion and chelating chemical adhesion, respectively. The clinical performance of the restorations in function was also compared between the two materials. The null hypothesis investigated was that there were no differences in the longevity and clinical performance between the cervical restorations filled with RC and GI. In order to investigate the hypothesis, the lifespan of the already-replaced restorations was determined from evaluation of dental records. The other restorations in use were clinically evaluated by two investigators according to modified USPHS criteria.

MATERIALS AND METHODS

Participants

Patients who had received restorative treatments in the Department of Conservative Dentistry, Seoul National University Dental Hospital before July 1, 2008, that is, who had restorations more than one year prior to initiation of the study, and revisited the department from July 6, 2009 to August 28, 2009, were enrolled in this study. Patients with systemic diseases that could affect the longevity of restorations were excluded. These included dry mouth, severe disability, wasting diseases such as uncontrolled diabetes mellitus, and impaired immune function. Permanent teeth in patients over 20 years of age were selected, and primary teeth with prolonged retention were excluded. The oldest restoration observed in this study was delivered in 1986. Since the 1980s, various restorative products and techniques were used in the department.

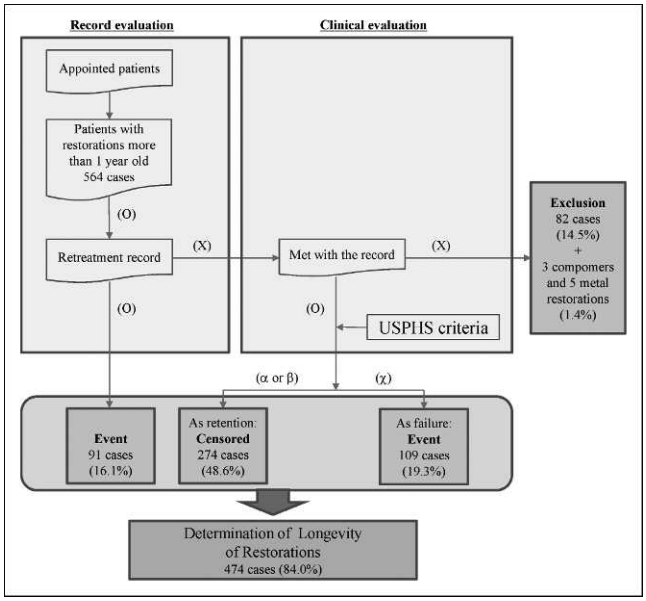

Figure 1. *Schematic representation of the survey procedure. First, if there was a record on retreatment or further treatment, the longevity of the restorations was defined as the time from the initial treatment to the retreatment. Second, if a patient had restorations with no records of retreatment or further treatment, the patient was clinically evaluated. Restorations confirmed to have not been replaced or treated further were evaluated according to the modified USPHS criteria. For clinically acceptable restorations rated as Alpha or Bravo, the lifespan was defined as the period from the initial treatment to the date of examination. When a restoration was rated as Charlie, clinically unacceptable, even in just one criterion, it was regarded as a failure and recommended to be retreated. Its longevity was determined as the period from the initial treatment to the date of examination.*

Because the aim of this study was to compare the longevity and clinical performance of the representative restorative materials used in the cervical restorations, that is, RC and GI, the restorative materials were divided into the two materials without considering restorative techniques in this study.

Survey Procedures

Under the approval of the Institutional Review Board of Seoul National University Dental Hospital, dental records were evaluated prior to the patients' visits. Based on the presence or absence of records on retreatment or further treatment of each restoration, including replacement, prosthetic treatment, endodontic treatment, and extraction, the survey procedure was divided into two pathways (Figure 1).

First, if there was a record on retreatment or further treatment, we concluded that an event had happened with the restoration. The longevity of the further-treated restoration was determined as the period from the initial treatment to the retreatment or further treatment. Information, including the date and details of and the reasons for retreatment, was collected from the records. Patient information included year of birth, gender, and premedical and predental history. Treatment information included tooth number, date of treatment, restorative material, operator, and diagnosis (reason for treatment).

Table 1: *Distribution of Restorations by Position of Teeth*

Restorative Materials	Anterior	Premolar	Molar	Total
Resin composite	155 (83.3%)	159 (80.3%)	63 (70.0%)	377 (79.5%)
Glass ionomers	31 (16.7%)	39 (19.7%)	27 (30.0%)	97 (20.5%)
Total	186	198	90	474

Second, if a patient had restorations that had no record of retreatment or further treatment, the patient was clinically evaluated after informed consent. For the existing restorations, two trained observers independently determined whether the characteristics of each restoration were consistent with the treatment record and whether the restoration had been replaced or further treated. In cases where it was unclear if there had been no further treatment of the existing restoration or whether the characteristics of the restoration agreed with the medical record, the case was excluded from the study. The two observers then independently evaluated the restorations in function according to the modified USPHS criteria (Table 1). If there was a disagreement between the observers, it was resolved by consensus. When the restoration was rated as Alpha or Bravo, the restoration was considered censored. Its censored lifespan was defined as the period from the initial treatment to the date of examination. Related information was also collected from the records.

Third, when a restoration remained in the oral cavity but was rated as "clinically unacceptable" Charlie even in a single criterion of the modified USPHS criteria, it was regarded as a failure and recommended to be retreated. For the clinically unacceptable restorations, longevity was defined as the period from the initial treatment to the date of examination.

Statistical Analysis

To evaluate the longevity of the cervical restorations filled with RC and GI, survival analysis was performed using Kaplan-Meier survival estimates. The effect of the assumed variables related to patients and teeth on the survival of restorations was analyzed using a multivariate Cox proportional hazard model by entering all variables simultaneously, and the relative risks were obtained. Patient age, gender, presence of systemic disease, type of tooth, restorative material, operator groups, and reasons for treatment were evaluated as potential prognostic variables. The operator groups were divided into three categories: professors, residents, and students. The reasons for treatment were subdivided into three categories: restoration of carious lesion, restoration of noncarious lesion, and replacement of previous restoration. Pearson chi-square test was performed on the numbers of restorations with acceptable (Alpha and Bravo) and unacceptable (Charlie) ratings according to the modified USPHS criteria to compare the clinical performance of the two restorative materials.

RESULTS

Surveyed Group and Case Distribution

Based on the date of treatment, the subjects were 23 through 81 years of age with a mean age and standard deviation of 63.9 ± 10.8 years. Based on the date of evaluation, the ages ranged from 20 to 80 years with a mean age of 57.4 ± 10.4 years. The lifespan of the restorations was from 0.1 to 22.9 years. The restoration with the longest service time was the one restored with conventional glass ionomer. Sixty-nine patients (52.7%) were male and 62 patients (47.3%) were female. Forty-seven (35.9%) patients had various systemic diseases. Hypertension was the most common (22 patients), followed by controlled diabetes (10 patients).

Data for 564 cervical restorations were collected from 131 patients during the survey. According to patient records, 91 (16.1%) restorations had been retreated or further treated. Among the restorations in function, 274 (48.6%) were rated as Alpha or Bravo according to the modified USPHS criteria and, as a result, were regarded as being censored. There were 109 restorations (19.3%) that were rated as Charlie and regarded as failure. Eighty-two restorations (14.5%), which were in function but did not agree with the medical records, were excluded from the study because their longevity was uncertain. Five metal restorations and three compomer restorations (1.4%) were also excluded from the survival analysis. Therefore, a total of 474 (84.0%) of 564 cases were included in this study (Figure 1). The main reasons for cervical restoration failure were loss of retention (82.2%) and secondary caries (17.8%).

Table 2: *Survival Time of the Cervical Restorations According to the Materials*

Variables	Survival Quartiles, y		
	75% ± SE	50% ± SE	25% ± SE
Materials			
Resin composite	15.1 ± 1.0	10.4 ± 0.7	5.1 ± 0.4
Glass ionomers	12.9 ± 0.1	11.5 ± 1.1	3.5 ± 0.4
Total	13.0 ± 0.7	11.0 ± 0.6	4.5 ± 0.4

Among the restoratives used for the cervical restorations, RC (n=377, 79.5%) was the most frequently used for all teeth in the maxillary and mandibular arches, followed by conventional glass ionomer cement (n=74, 15.6%) and resin-modified glass ionomer cement (n=23, 4.9%). Due to insufficient frequency of resin-modified glass ionomer and its similarity with conventional glass ionomer in the properties and the adhesion procedures, both materials were grouped as GIs in order to increase the statistical power (Table 2). RC was used more frequently as the restorative of choice in the anterior region (83.3%) than in the posterior region (premolar, 80.3%; molar, 70.0%). The other restorative materials used were compomer (n=3, 0.5%), amalgam (n=4, 0.7%), and gold inlay (n=1, 0.2%), which were not included in the survival analysis due to lack of cases. As a result, the survival estimates for the restorations were compared between RC and GI using Kaplan-Meier survival analysis.

Comparison of Survival Estimates According to Prognostic Variables

Although GI (63.7 ± 5.2%, cumulative survival rate ± standard error) showed a lower cumulative survival rate after five years than RC (74.7 ± 2.6%), the survival estimates of GI and RC were not significantly different (Breslow test, $p>0.05$; Figure 2a). The median survival times of RC and GI were 10.4 ± 0.7 and 11.5 ± 1.1 years (median ± standard error), respectively (Table 2). Among the

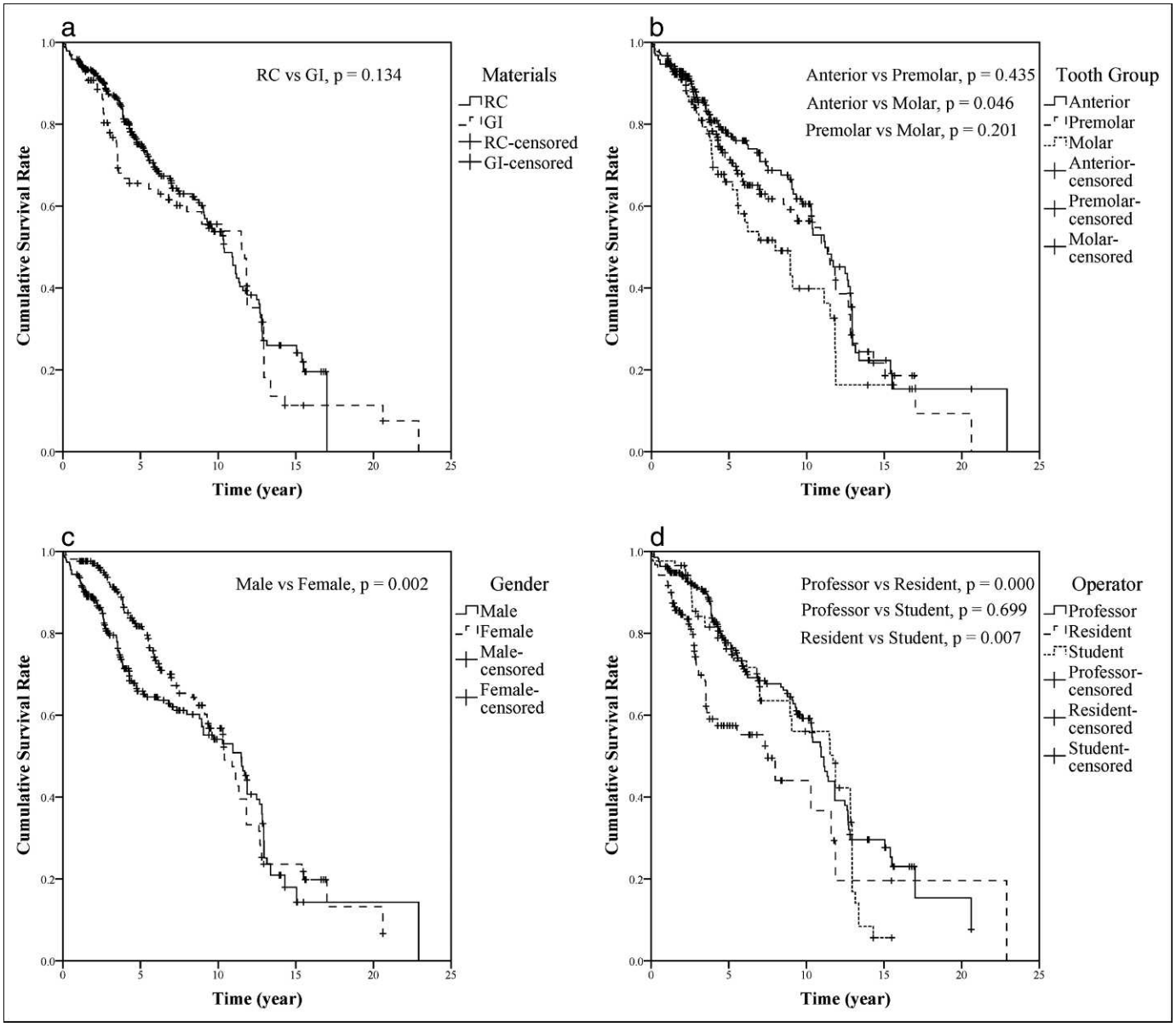

Figure 2. *Comparison of survival estimates according to prognostic variables using Kaplan-Meier survival analysis. (a): Materials. There was no significant difference in the survival estimates between resin composite and glass ionomers. (b): Tooth groups. The survival estimates showed significant differences among anterior teeth, premolars and molars. (c): Gender. The survival estimates showed significant difference between male and female patients. (d) Operators. Among the groups of professors, residents, and students, the survival estimate of the restorations practiced by residents was significantly lower than those performed by professors and students.*

tooth groups, the median survival times of anterior, premolar, and molar teeth were 11.2 ± 0.9 years, 11.0 ± 0.9 years, and 8.0 ± 1.5 years, respectively. The longevity of anterior teeth was significantly different from that of molar teeth (Breslow test, p=0.046; Figure 2b). However, within each tooth group, the longevity of RC and GI was significantly different only in the anterior teeth (Breslow test, p=0.016), in contrast to the premolars and molars (p=0.733 and p=0.532, respectively). Although GI was used relatively more frequently in molars (Table 1), GI did not show any difference in the longevity among tooth groups (Breslow test, p>0.05). RC in the anterior teeth only showed significant difference from those in the molar teeth (Breslow test, p=0.009). The median survival times of the male and female groups were 11.5 ± 1.1 years and 10.4 ± 0.4 years, respectively. The survival estimates of both genders were significantly different (Breslow test, p=0.002; Figure 2c). The difference in the

Table 3: *Contributions and Odds Ratios of Prognostic Variables*

Variables	Groups	Wald[a]	Odds Ratio[b]	95% CI		p Value
Operator group		9.322				0.009
	Professor		0.89	0.61	1.32	0.572
	Resident		1.60	1.04	2.46	0.032
	Student		1.00			
Tooth group		3.742				0.154
	Anterior		0.68	0.47	1.01	0.045
	Premolar		0.79	0.54	1.16	0.234
	Molar		1.00			
Gender		0.510				0.475
	Male		1.13	0.81	1.32	0.475
	Female		1.00			

[a] *Contributions of prognostic variables were estimated by the Wald test of the Cox proportional hazard model.*
[b] *The odds ratio of each group for each prognostic variable was evaluated using Exp(B) of the Cox proportional hazard model.*

survival estimates between the presence and absence of systemic disease was not significant (Breslow test, $p=0.143$). The median survival times of the two groups were 11.2 ± 1.0 years and 11.0 ± 0.5 years, respectively. The longevity of the restorations in the upper and lower arch was statistically not different (Breslow test, $p=0.657$). The median survival times of the restorations in the upper and lower arches were 11.5 ± 0.5 years and 10.9 ± 0.9 years, respectively. The survival estimates of the restorations placed by residents were significantly lower than those performed by professors and students (Breslow test, $p=0.000$ and $p=0.007$, respectively; Figure 2d). There were no significant differences in survival estimates among the three categories for treatment (restoration of carious lesion, restoration of noncarious lesion, and replacement of previous restoration).

Among the variables evaluated with the Kaplan-Meier analysis, those demonstrating statistically significant differences in the survival estimates between groups were selected as covariates. These were tooth group, gender, and operator group. Their contribution and relative risks were compared with the Wald test and the Cox proportional hazard model, respectively (Table 3). The operator group was the most influential prognostic variable, followed by tooth group. Within each variable, the restorations placed by residents and in molar teeth showed significantly higher relative risks than those placed by professors and students and in anterior teeth, respectively ($p<0.05$). However, gender failed to be confirmed as a difference statistically ($p=0.457$; Table 3).

Comparison of Clinical Performance Between RC and GI

The number of RC restorations that were evaluated as clinically acceptable (Alpha or Bravo) according to modified USPHS criteria was significantly higher than the number of clinically acceptable GI restorations, including retention, marginal discoloration, and marginal adaptation (relative risks of GI/RC, 3.255, 7.649, and 6.784, respectively; $p<0.05$; Table 4). Between the two materials, the incidences of secondary caries, wear, and postoperative sensitivity were not significantly different (Fisher exact test, $p=0.512$, $p=1.000$, and $p=0.598$, respectively). In the criteria of retention, marginal discoloration, and marginal adaptation, RC demonstrated superior clinical performance in the oral cavity when compared to GI. With regard to color match, no comparison was available due to a lack of unacceptable cases with either material.

DISCUSSION

In this study, the number of groups in each variable was minimized as much as possible because too many groups would produce higher-order interactions and complicate the interpretation of the results. A small sample size may also increase type II errors and decrease statistical power.[17] In order to reduce the number of groups, resin-modified glass ionomer was included in the GI group, together with conventional glass ionomer cement. A variety of glass ionomer-derived materials use the advantage of fluoride release and of the combined setting reaction of acid-base reaction of the glass ionomer component and the chain-reaction polymerization of the resin component.[7,8,18,19] By the same token, all of the samples were divided into two groups based on the presence or absence of systemic disease because there were too many types of diseases and only small sample numbers for each disease. As in prior studies,

Table 4: *Comparison of the Clinical Performance Between the Restorations Filled With Resin Composite (RC) and Glass Ionomers (GI) Evaluated Based on the Ratings of the Modified USPHS Criteria*

Criteria	Chi-Square Test/ Fisher Exact Test[a]		Odds Ratio	
	χ^2	*p*	GI/RC	95% CI
Retention	19.058	0.000	3.255	1.884-5.625
Color match[b]	NA	NA	NA	
Marginal discoloration	—	0.005[a]	7.649	1.974-29.642
Secondary caries	—	0.512[a]	1.488	0.465-4.756
Wear (anatomic form)	—	1.000[a]	1.113	0.127–9.747
Marginal adaptation	21.558	0.000	6.784	2.747-16.756
Postoperative sensitivity	—	0.598[a]	NA	

[a] *When the expected incidence in more than one cell was less than 5, the result of Fisher exact test was selected.*
[b] *The χ^2 value and odds ratio were not calculated because more than one cell had no incidence in the 2 × 2 tables.*
Abbreviation: NA, not available.

the reasons for treatment were divided into treatments for carious lesions, noncarious lesions including abrasions and erosions, and replacement of old restorations.[3,20]

In total, RC (79.5%) was used approximately four times more frequently than GI. RC was used especially in anterior (83.3%) and premolar teeth (80.3%), but in posterior teeth the relative frequency of GI restorations increased (30.0%) compared to anterior teeth. As this study was confined to cervical restorations and two restorative materials, the proportion of RC (79.5%) was higher than in prior studies (Mjör,[20] 52.7%; Forss,[21] 74.9%), in which metal restorations and posterior occlusal and proximal restorations were included. This means that RC was the most frequently selected material for cervical restorations due to its esthetic excellence and adequate mechanical properties. However, in the posterior teeth, selection of GI was increased due to the characteristic adhesion capability to tooth structure and the relative ease of use.[2,22,23]

According to the Kaplan-Meier survival analysis and multivariate Cox proportional hazard model, there were no significant differences in materials, gender, presence, or absence of systemic disease, arch, and reason for treatment. However, with regard to the tooth group and operator group, there were significant differences in the longevity between groups. We were unable to find any previous reports on the effects of systemic diseases on the survival estimates of dental restorations. Within the limitations of the current study, we did not attempt to associate individual systemic diseases with the survival estimates of restorations due to the small number of samples for each disease. However, the presence or absence of systemic diseases did not affect the survival estimates of cervical restorations. Additional studies with larger sample sizes for specific diseases such as diabetes mellitus, hypertension, heart disease, liver, and renal disease are needed.

There were no significant differences between the upper and lower arches or between genders, but there was between the anterior and molar teeth ($p = 0.045$, Table 3). Generally, abfraction had a similar prevalence in maxillary and mandibular teeth.[24] The occurrence of abfraction from tooth flexure did not differ by gender.[24,25] Although the failure rate of extensive restorations in posterior teeth was reported to be higher in male than in female patients,[26,27] no previous literature that reported significant differences in the longevity of cervical restorations between genders, was found. The relative risk between anterior and molar teeth may be attributed to occlusal forces inducing tooth flexure.

The clinical outcomes of dental restorations are known to be affected by operator technique, even when the same restorative material and protocol are used. The technique sensitivity is especially high in adhesive procedures and with esthetic materials.[28,29] In this study, the relative risk for the restorations performed by residents was significantly higher than for those performed by professors and students. The reason for this observation may be that the students were strictly supervised by instructors, but residents may have practiced relatively freely with a wide range of materials. Although Folwaczny and others[3] and Mjör and others[20] divided the reasons for treatment into carious lesions, noncarious lesions, and replacement of old restorations, they only reported the proportions of each treatment reason out of the total cases. They did not report the

survival estimates according to the treatment reasons. In the current study, there were no significant differences in the longevity among the three treatment reason groups.

Most studies have reported no difference in the retention of cervical restorations among RC and GIs.[3,13,14,30-32] Other studies, with prospective longitudinal designs for relatively short durations, reported that glass ionomer-derived materials, especially resin-modified glass ionomer, had better retention than RC.[3,15,33,34] Reports demonstrating longer retention of RC than GI were not found. In this study, we divided the cervical restorative materials into two groups, RC and GI. The longevity of the two material groups was evaluated in a retrospective cross-sectional design and, as a result, the data included many cases with longer service duration (maximum lifetime, 22.9 years) than those in a prospective design. Although retrospective cross-sectional studies have limitations to differentiate important factors such as individual restorative materials, a large number of restorations with relatively long lifetime can be assessed in a short time.[35] By assessing such long-lasting restorations, the factors affecting late failure of the restorations such as fractures, secondary caries, and wear and deterioration of the materials, and their clinically relevant problems can be suggested.[35] Such practice-based research can be a source for further well-controlled prospective longitudinal study. The survival estimates were not different between RC and GI, similar to the majority of studies. Compared with previous studies, we may expect that the retention of GI is not inferior to that of RC. Further prospective and longitudinal studies are needed on the longevity of both materials in a well-controlled design.

Although there was no significant difference in the longevity between the two restorative materials, significant differences were observed in the ratings of USPHS criteria. These ratings represent the clinical performance of the existing restorations in the oral cavity. When both materials were compared with taking into account the location, the retention was significantly different in anterior teeth (χ^2 test, $p=0.001$, odds ratio=5.420) and in premolar teeth (χ^2 test, $p=0.006$, odds ratio=3.067) and the marginal adaptation was also different in premolar teeth (Fisher exact test, $p=0.04$) and in molar teeth (Fisher exact test, $p=0.03$). In the other criteria, there was no significant difference in the clinical status between both materials at each location. However, when both materials were compared without considering their locations, RC demonstrated superior clinical performance than GI in retention, marginal discoloration, and marginal adaptation among the criteria ($p<0.05$; Table 4), in contrast to the Cox proportional hazard model. These data suggest that although the longevity of both materials was not different, the clinical performance of existing RC restorations is superior to that of existing GI restorations while in function. In many other studies, the superior clinical performance of existing RC restorations has been reported, in addition to the survival analysis.[3,13-15]

Most laboratory studies suggested GI as the restorative material of choice for cervical lesions because of clinically acceptable interfacial gaps, its capacity for absorbing occlusal load, and the low polymerization shrinkage stress of slowly-setting glass ionomers.[2,7,36-38] According to the ratings of the modified USPHS criteria, secondary caries, anatomic form (wear), and hypersensitivity did not differ between the two materials. Contrary to the occlusal wear, the wear of the cervical restorations due to abrasion and erosion, for example, was not different between RC and GI. The results of the current study agree with prior observations.[16,39,40] When the sealing ability of adhesive systems was not enough to prevent postoperative sensitivity, conventional glass ionomer was frequently used to reduce the discomfort. With the advent of the concept of hybrid layer formation using total-etch three-step adhesives, the incidence of postoperative sensitivity decreased greatly, so that there were no differences between direct and indirect restorations, total-etch and self-etch, and GI and RC.[41,42] There was no significant difference between the two materials in regard to secondary caries. Glass ionomer cement was recommended as the material of choice for high caries risk patients due to its *in vitro* fluoride release.[18,43] However, under anticariogenic and fluoride dentifrice exposure conditions, the GI restorations were reported not to provide additional protection against secondary caries.[10,44,45] The clinical effectiveness of fluoride release from these materials and the relevance of the *in vitro* data in the context of caries prevention should be evaluated in further studies.

CONCLUSIONS

The survival estimates of resin composite and glass ionomers as cervical restorative materials were not statistically different. However, the longevity of cervical restorations was significantly influenced by the tooth group and operator group. During function

in the mouth, RC demonstrated superior clinical performance to GIs in the criteria of retention, marginal adaptation, and marginal discoloration.

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Conflict of Interest

The authors of this article 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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Efficacy of and Effect on Tooth Sensitivity of In-office Bleaching Gel Concentrations: A Randomized Clinical Trial

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Clinical Relevance

Faster bleaching was observed with Whiteness HP Blue 35 (35% hydrogen peroxide) and tooth sensitivity was similar to that found with the lower concentration Whiteness HP Blue 20 (20% hydrogen peroxide). These results cannot be extrapolated to other in-office 35% hydrogen peroxide gels.

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SUMMARY

With the aim of reducing the side effects of in-office bleaching agents, less-concentrated hydrogen peroxide (HP) gels have been released by manufacturers. We evaluated the tooth sensitivity (TS) and bleaching efficacy (BE) of two HP concentrations in this study. Gels containing 35% and 20% HP (HP35 and HP20, respectively) were applied on teeth of 60 caries-free patients. Color was recorded at baseline and one week after the first and second bleaching sessions using the Vita Classical shade guide. TS was recorded on a 0-4 scale. BE at each weekly recall was evaluated by Kruskall-Wallis and Mann-Whitney tests (α=0.05). Absolute risk of TS and its intensity was evaluated by Fisher exact and Mann-Whitney tests, respectively (α=0.05). After two

bleaching sessions, color change of approximately eight tabs was obtained with HP35; whereas, with HP20 it was six tabs ($p<0.05$). Only 26.7% (HP35) and 16.7% (HP20) of the participants reported TS, and no statistical differences were detected among them. Both in-office bleaching gels showed similar TS intensity, but the 35% HP agent produced faster bleaching.

INTRODUCTION

The at-home bleaching system is by far the most frequently recommended treatment for discolored vital teeth. However, some patients prefer not to use a bleaching tray or wish to obtain a faster whitening result. In this case, in-office bleaching procedures, using high hydrogen peroxide (HP) concentrations (30%-35%), are appropriate alternatives to the at-home bleaching techniques. The effectiveness of in-office bleaching is well documented in the literature. An overall color change of five to eight shade guide units (SGUs) is usually obtained after two bleaching sessions.[1-5]

In spite of this, most clinical studies have demonstrated that more than 70% of the patients who undergo in-office bleaching complain of tooth sensitivity (TS),[1,5,6] and this is the main deterrent to patients successfully completing their whitening treatment. Although the results of *in vitro* studies do not necessarily correlate to events that occur in an *in vivo* condition, one may hypothesize that the rapid transenamel and transdentinal diffusion of HP to the pulp, or other toxic components released with the degradation of the bleaching gels,[7,8] may be responsible for the high prevalence of TS. These by-products released from the bleaching gel act as free radicals and may cause oxidative stress in the pulp cells due to the imbalance between reactive oxygen species and endogenous and exogenous antioxidants.[9,10] In some cases, the oxidative stress produced by high HP concentrations applied for 45 minutes is so intense that it causes irreversible pulp damage by coagulation necrosis in the mandibular incisors.[11]

It is known that the diffusion of HP through dentin depends on the original concentration of the bleaching agent[7,12] and the length of time the agent is in contact with the dentin. This fact has led some manufacturers to release in-office bleaching gels with lower HP concentrations, with the aim of minimizing the side effects produced by bleaching products while retaining the same bleaching efficacy. Although empirically, it could be anticipated that the higher the concentration of the bleaching gel, the more likely it is to produce TS, this was not proved when 10% and 16% carbamide peroxide gels were compared with each other in a clinical trial.[13]

To the extent of the authors' knowledge, no clinical study has so far addressed the bleaching efficacy and TS produced by low-concentration bleaching gels. Therefore, the aim of the present investigation was to compare the bleaching efficacy and TS of 20% and 35% HP in-office bleaching gels. The null hypothesis to be tested was that both HP gels can yield similar bleaching efficacy and TS.

MATERIAL AND METHODS

The local university's Ethics Research Committee approved this clinical investigation (Protocol no. 05530/09). The experimental design was in accordance with the Consolidated Standards of Reporting Trials statement.[14] Based on pre-established criteria, 60 volunteers from the city of Ponta Grossa, Paraná, Brazil, were selected for this study. Two weeks before the bleaching procedures, all the volunteers received dental screening and dental prophylaxis with pumice and water in a rubber cup and signed a form of free and informed consent.

Study Design

This was a randomized, double-blind, parallel-group clinical trial with an equal allocation rate to receive either of two treatments. The study was conducted in the clinic of the School of Dentistry of the State University of Ponta Grossa from January 2010 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 local advertisement. A total of 270 participants were examined, seated in a dental chair, to check whether they met the inclusion and exclusion criteria (Figure 1). The participants were required to have caries-free maxillary and mandibular anterior teeth, without restorations on the labial surfaces. The central incisors had to be shade C2 or darker, judged by comparison with a value-oriented shade guide (Vita Lumin, Vita Zahnfabrik, Bad Säckingen, Germany). Participants who had undergone previous tooth-whitening procedures, presented anterior restorations, were pregnant or lactating, and/or had severe internal tooth discoloration (tetracycline stains, fluorosis, pulpless teeth), bruxism habits, or any other pathology that could cause sensitivity

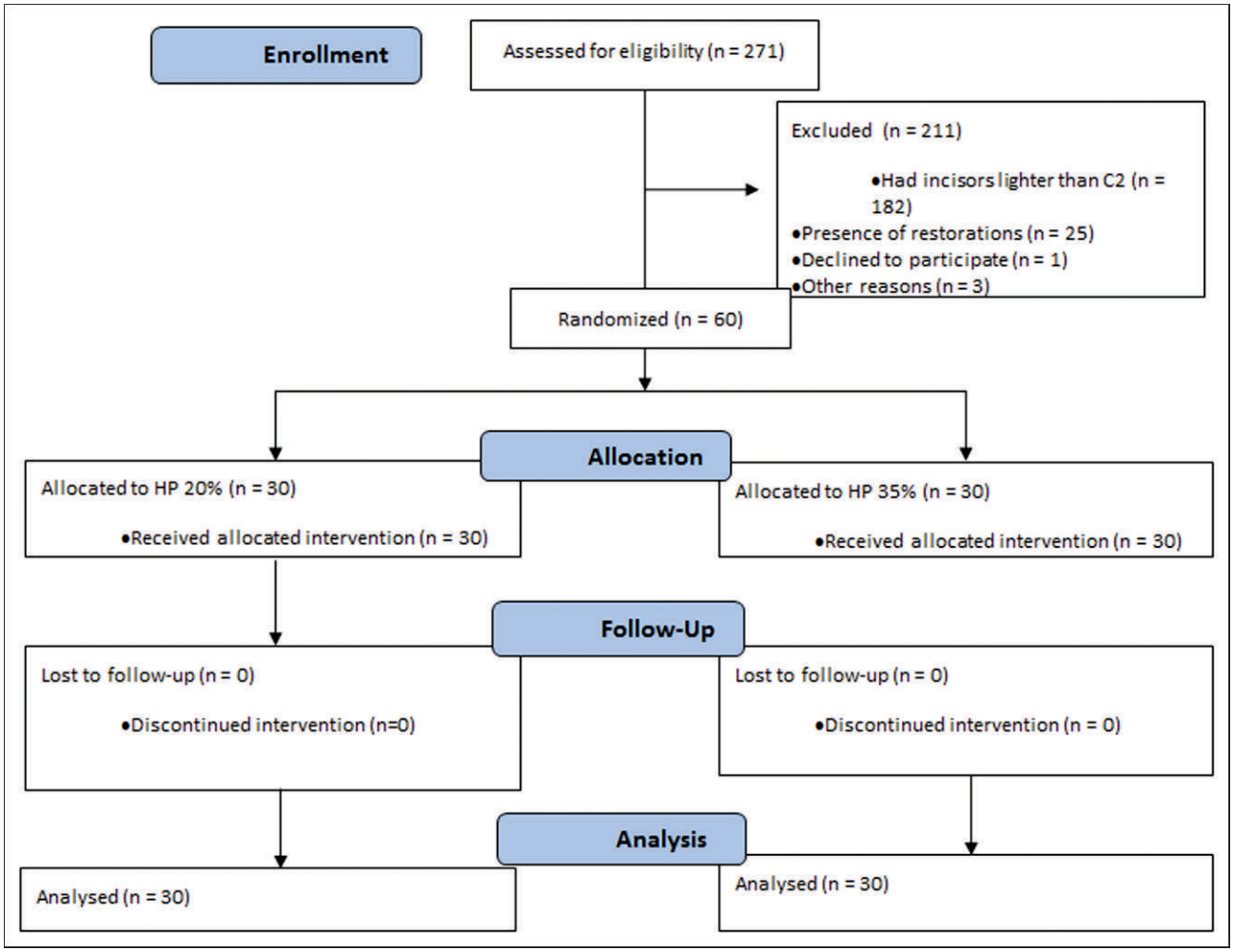

Figure 1. *Flow diagram of the clinical trial, including detailed information on the excluded participants.*

(such as recession, dentin exposure) were excluded from the study. This was because they would not be immediately eligible for a cosmetic treatment such as bleaching, for the other restorative needs would need priority attention. The patients were asked about previous experience of TS the week before the bleaching therapy began, using the criteria described in the "Tooth Sensitivity Evaluation" section. Patients with TS equal to or greater than mild were also excluded from the study.

Sample Size Calculation

The primary outcome of this study was the absolute risk of TS. Given that no study has so far reported the risk of TS for the products used in this study, the sample size calculation was based on the absolute risk of TS of another 35% HP gel from the same company (Whiteness HP Maxx, FGM Dental Products, Joinville, Brazil), which was reported to be approximately 87%.[1] Thus, 60 patients were required to have an 80% chance of detecting a significant decrease in the primary outcome measure, from 87% in the control group to 57% in the experimental group, at the level of 5%.

Study Intervention

The randomization process was performed by computer-generated tables prepared by a third person not involved in the research protocol. Details of the allocated group were recorded on cards contained in sequentially numbered, opaque, sealed envelopes. These 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 had completed all baseline assessments, the allocation assignment was revealed by this envelope being opened by the aforementioned third person. Neither the participant nor the operator knew the group allocation, both being blinded to the protocol.

Table 1: *Products, Composition, and Application Regimens*

Products	Composition	Application Mode
Whiteness HP Blue 35	35% hydrogen peroxide, thickeners, inert violet, neutralizing agent, calcium gluconate, glycol, and deionized water.	1. Attach the two syringes to each other.
		2. Mix the contents of both phases by alternately pressing the plungers of the syringes in opposite directions up to 8 times.
		3. Press the entire mixture content into one of the syringes.
		4. The bleaching gel should be applied by means of the syringe on the teeth surfaces and left on for 40 min. Then the material should be removed with an aspirator.
Whiteness HP Blue 20	20% hydrogen peroxide, thickeners, inert violet, neutralizing agent, calcium gluconate, glycol, and deionized water.	1. Attach the two syringes to each other.
		2. Mix the contents of both phases by alternately pressing the plungers of the syringes in opposite directions up to 8 times.
		3. Press the entire mixture content into one of the syringes.
		4. The bleaching gel should be applied by means of the syringe on the teeth surfaces and left on for 50 min. Then the material should be removed with an aspirator.

The gingival tissue of the teeth to be bleached was isolated using a light-polymerized resin dam (Top Dam, FGM Dental Products). Depending on the randomization, participants received either the 35% or 20% HP gels (Whiteness HP Blue 35 and Whiteness HP Blue 20, FGM Dental Products), which were used according to the manufacturer's instructions (Table 1). Two bleaching sessions were performed with a one-week interval between them. All participants were instructed to brush their teeth regularly using fluoridated toothpaste (Sorriso Fresh, Colgate-Palmolive, São Paulo, Brazil).

Shade Evaluation

The color was recorded at baseline and one week after the first and second bleaching sessions using a Vita shade guide. The shade evaluation was done in a single room with artificial lighting. The 16 tabs of the Vita Classical shade guide were arranged from highest (B1) to lowest (C4) value, as can be seen in Table 2. 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. Two calibrated evaluators recorded the shade of each participant's teeth at baseline and weekly. The measurement area of interest for shade matching was the middle third of the facial surface of one of the central incisors, which was chosen by tossing a coin. The two examiners were required to have an agreement of at least 85% (κ statistic) before beginning the study evaluation.

Table 2: *Ordering of Vita Shade Guide by Value*[a]

Tab	B1	A1	B2	D2	A2	C1	C2	D4	A3	D3	B3	A3.5	B4	C3	A4	C4
Rank	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16

[a] *Light-to-dark ranking by manufacturer.*

Tooth Sensitivity Evaluation

The patients recorded their perception of TS on a 5-point verbal rating scale during bleaching and up to 24 hours after each session. Subjects were asked to keep a daily record of whether they experienced sensitivity, using the following criteria: 0 = *none*, 1 = *mild*, 2 = *moderate*, 3 = *considerable*, and 4 = *severe*. Given that two bleaching sessions were performed, the worst scores obtained in the two bleaching sessions were considered for statistical purposes. The values were arranged into two categories: overall percentage of patients who reported TS at least once during treatment (absolute risk of TS) and TS intensity at each of the assessment points.

Statistical Analysis

The analysis followed the intention-to-treat protocol and involved all participants who were randomly assigned.[14] The statistician was blinded to the study groups. The agreement between the examiners' objective evaluation was assessed using the κ statistic. The primary outcome absolute risk of TS was compared by using the Fisher exact test (α=0.05, a test for comparison of independent proportions data. The relative risk as well as the confidence interval (CI) for the effect size was calculated.

TS intensity (secondary outcome) was also analyzed statistically. The median and interquartile ranges of the pain scale were calculated. The data sets of TS intensity were plotted on histograms and inspected for normal distributions. Data did not appear to be normally distributed, and therefore the two groups were compared using the Mann-Whitney U-test (α=0.05).

Color change, another secondary endpoint, was used to assess the efficacy of the bleaching treatment. Means and standard deviations of the SGU at each assessment point were calculated. In order to evaluate whether the bleaching therapies were effective over time, the data of each group were submitted to Kruskall-Wallis test and the Mann-Whitney test for pairwise comparisons (α=0.05). At each assessment point, color changes of the two groups were compared with each other by the Mann-Whitney test (α=0.05).

RESULTS

Of the 60 participants who took part in this investigation, all completed the study. The mean age of the participants in this study were similar between the groups (HP35: 25.0 ± 6.8 years and HP20: 29 ± 9.9 years). Of the participants from the HP35 and HP20 groups, 45% and 43%, respectively, were men. Figure 1 depicts the participant flow in the different phases of the study design.

Tooth Sensitivity

The data from 60 patients were used in this study, following the intention-to-treat analysis.[14] With regard to the absolute risk of TS (primary outcome), no significant difference was observed between groups (Fisher exact test, p=0.53). The absolute risk of TS was 26.7% (95% CI, 14.2%-44.5%) and 16.7 (95% CI, 7.0%-33.6%) for the HP35 and HP20 groups, respectively.

The medians (25 and 75 percentiles) of TS intensity were shown to be 0 (0-0) for HP20 and 0 (0-1) for the HP35 groups. Similarly, no significant difference was detected between groups (Mann-Whitney test, p=0.36). Figure 2 depicts the distribution of TS scores between the two groups.

Color Change

The level of agreement between the two evaluators by means of the κ statistic was 82%. The means and standard deviations of SGUs are shown in Table 3. The mean color of the teeth in SGUs before the treatment began was 9.4 (between A3 and D3 in the Vita Classical shade guide) for the HP35 group and 9.3 (between A3 and D3 in the Vita Classical shade guide) for the HP20 group. The mean tooth color at baseline was similar for the two groups (p>0.05).

After two bleaching sessions, a change of approximately eight tabs occurred for the HP35 group (1.6 [between B1 and A1 in Vita Classical shade guide]), whereas a change of six tabs occurred for the HP20 group (3.5 [between D2 and B2 in Vita Classical shade guide]. This difference is statistically significant (p<0.05) (Table 3).

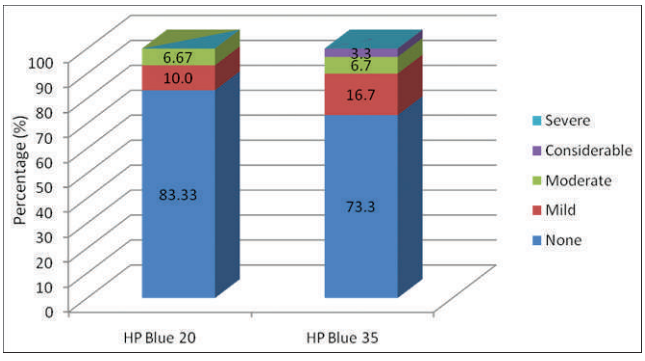

Figure 2. *Levels of sensitivity (%) perceived by the participants in both groups immediately after the bleaching protocol.*

Table 3: *Means and Standard Deviations of Shade Guide Units Between Assessment Points for the Two Treatment Groups*[a]

Assessment Point	HP35	HP20
Baseline	9.4 ± 1.3 aA	9.5 ± 1.4 aA
1 week after the first session	4.0 ± 1.2 bB	5.3 ± 0.9 bB
1 week after the second session	1.6 ± 0.7 dC	3.5 ± 1.0 cC

[a] *Comparisons between groups at each assessment point are represented by lowercase letters (Mann-Whitney test). Comparisons between assessment points in each group (Kruskall Wallis and Mann-Whitney tests) are represented by uppercase letters. Similar lower and uppercase letters indicate statistically similar means.*

DISCUSSION

The results of this study indicate that both groups demonstrated significant tooth color enhancement when compared with baseline (Table 3). Bleaching of eight and six SGUs was detected for the HP35 and HP20 groups, respectively. When the two materials were compared with each other, the 35% HP gel was capable of faster whitening than the 20% HP after two bleaching sessions.

HP is a thermally unstable chemical agent with a high oxidative power, which may dissociate into water, oxygen, and some free-radical species.[15] These species are responsible for the whitening process, and therefore one may assume that the bleaching effectiveness depends, among other factors, on the amount of free radicals produced by the bleaching gel when in contact with the dental structure. It was recently demonstrated that the concentration of HP in a proprietary bleaching gel had a marked effect on the number of applications required to produce an optimal shade outcome.[16] This means that bleaching gels with a higher peroxide concentration needed fewer applications to produce a similar bleaching effect.[16] Therefore, it was hypothesized that similar bleaching with HP20 and HP35 would have been achieved if three, instead of two, bleaching sessions had been performed for the former.

In spite of the lower whitening effect after two bleaching sessions, the overall color change produced by HP20 was within the range reported in the literature. A variation of five to nine SGUs after bleaching with 35% HP gels has been shown.[1-5,17] This means that other factors apart from the HP concentration play an important role in the bleaching effectiveness.

With regard to TS, the present study is not in agreement with previous findings in the literature. Although lower TS rates (16.7% to 26.7%) were reported in this study, TS rates three to four times higher were reported in earlier studies. For instance, Marson and others[2] reported that 63% of the participants in their clinical study reported TS. Higher TS rates such as 70%[6] and 80%[1,5] have also been reported.

One important difference between the two bleaching gels used in this study and other bleaching gels available on the market is that they contain 2% calcium gluconate, which is extensively used in the pharmaceutical industry as a source of calcium replacement in the body. Although calcium gluconate was added to the bleaching gel with the aim of preventing enamel demineralization, the fact cannot be ruled out that this addition might have played a role on the lower TS reported by the calcium-containing products. Perhaps the 2% of calcium gluconate dissolved in the HP gel was able to decrease dentin permeability and block enamel surface defects, similarly to that which is believed to occur with bleaching gels containing amorphous calcium phosphate.[18]

In addition, according to the manufacturer, the two bleaching gels used in this study maintain a high and stable pH (8.0-9.0) throughout the bleaching procedure, which allows them to be used in a single 40- or 50-minute application protocol. These pH values claimed by the manufacturer were also confirmed by the authors of this study by means of pH measurements in triplicate. Most of the in-office bleaching gels are delivered in a low pH (2.4 to 6.5)[19,20] to allow them to be stored for prolonged periods.[21] The decomposition kinetics and the by-products produced by HP depend on the pH of the media. It has been reported that HP delivered in an alkaline medium increases the bleaching effectiveness.[22] For instance, in a pH of 9, the dissociation rate of the HP was shown to be 2.7 times higher than in an acidic solution[22] (pH=4.4), most probably due to the fact that the dissociation constant (pKa) of the HP is around 11.5.

The free radicals released from HP depend on the pH of the media, and they may play a role in the prevalence and intensity of TS. In an acidic solution, higher concentrations of hydroxyl anions are produced; however, in an alkaline media there is a higher concentration of perhydroxyl ions.[23] Little is known about the deleterious effects of these different oxidizing agents on the dental-pulp complex, and therefore future studies should be encouraged in

order to elucidate whether this has any correlation with the lower TS observed for the calcium-containing products used in this study. Ongoing studies are being conducted to evaluate the effects of these products on the dental-pulp complex, and this will certainly partially clarify these issues.

It should be pointed out that the comparison between 35% HP and 20% HP products was performed as per manufacturer's instructions, and for this reason the 20% HP product was applied for an additional 10 minutes each session. However, even under this "improved" condition, the bleaching speed of this product was not equivalent to that of the 35% HP gel, which probably means that this additional application time was not enough to provide equivalent bleaching in the same time interval of two weeks.

In summary, the 35% HP gel tested in this study produced whiter teeth after two bleaching sessions, with TS rates similar to those produced by the 20% concentration. It is worth mentioning that this may not be true for other bleaching gel compositions.

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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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Laboratory Research

Effect of Postoperative Peroxide Bleaching on the Stability of Composite to Enamel and Dentin Bonds

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Clinical Relevance

The increased risk of adhesive bond degradation by bleaching should be considered when simplified self-etching adhesives are used. This should be taken into consideration while planning bleaching of adhesively reconstructed teeth.

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SUMMARY

This study investigated the effect of peroxide bleaching gel on the durability of the adhesive bond between composite material, enamel, and dentin created with the etch-and-rinse adhesive Gluma Comfort Bond (GLU) and with the self-etch adhesives Clearfil SE Bond (CLE), Adper Prompt (ADP), and iBond (IBO). The adhesives were applied to flattened enamel and dentin of extracted human molars and built up with a microhybrid composite (Charisma). After 25 eight-hour cycles of bleaching with a 20% carbamide peroxide bleaching gel (Opalescence PF 20), the shear bond strength was measured and compared with one-day and two-month control specimens stored in water. The data were analyzed using nonparametric Mann-Whitney and Kruskal-Wallis statistics ($p<0.05$). Detailed fractographic analysis was performed using scanning electron microscopy. The bleaching gel significantly decreased the bond strength on both enamel and dentin for the simplified single-step self-etch adhesives ADP and IBO and markedly affected a

fracture pattern of ADP specimens at the periphery of their bonded area. The results of our study indicate that the durability of adhesive restorations can be detrimentally influenced by carbamide peroxide bleaching and that different adhesives show varying sensitivity levels to the bleaching gel.

INTRODUCTION

The stability of the adhesive bond between composite restorations and tooth tissue is one of the main factors determining restoration longevity. This bond is challenged by mechanical stresses; water penetration, which causes plasticization and hydrolysis of its resinous components; and degradation of collagen fibers in the dentin hybrid layer.[1] In addition, chemical agents, such as those contained in foodstuffs, drinks, mouth rinses, disinfectants,[2,3] and peroxide bleaching gels, may affect bond longevity. Vital bleaching is now widely demanded by the public and ranks among the most frequent cosmetic treatments in dentistry. Office-administered and self-administered bleaching gels contain hydrogen peroxide or its precursor, carbamide peroxide, as the active ingredient in concentrations ranging from 3% to 35% of hydrogen peroxide equivalent. It is supposed that hydroxyl radicals (HO·), perhydroxyl radicals (HOO·), perhydroxyl anions (HOO–), and superoxide anions (OO·–) released from peroxides attack double bonds of chromophore molecules captured within tooth tissues.[4-6] The change in double-bond conjugation causes a shift in the absorption spectrum of chromophore molecules and thus bleaching of tooth tissues. Because of the high reactivity and nonspecific nature of these oxygen radicals, they may have a side effect on tooth tissues,[7,8] reconstruction materials,[9-11] and the bond between them, which is usually the most susceptible to degradation.

To date, most studies have focused on the preoperative influence of bleaching gels on adhesion of composites to enamel. It has been shown that freshly bleached enamel cannot establish a full-value bond to composites because residues of peroxide and oxygen in the enamel structure inhibit free radical polymerization of acrylate or methacrylate monomers.[6] Fewer studies have dealt with the influence of bleaching gels on the adhesive bond of previously prepared composite restorations. This effect was analyzed using different methods, including measurement of bond strength,[12,13] fracture toughness,[14] and microleakage.[15] The results of these studies are ambiguous, however. Some authors reported compromised adhesion to enamel, but not to dentin,[12] while others described a decreased adhesion to dentin[14] or to both tissues[13] after bleaching.

The aim of our study was to investigate the effect of repeated application of carbamide peroxide bleaching gel on the enamel-composite and dentin-composite shear bond strength and fractographic analysis of four contemporary etch-and-rinse and self-etch adhesive systems. The null hypothesis tested was that repeated bleaching has no effect on enamel-composite and dentin-composite bond strength.

METHODS AND MATERIALS

Materials

Four adhesive systems were chosen: 1) etch-and-rinse two-step Gluma Comfort Bond with Gluma Etch 20 Gel (GLU, Heraeus Kulzer GmbH, Hanau, Germany), 2) self-etch two-step Clearfil SE Bond (CLE, Kuraray Medical Inc, Okayama, Japan), 3) self-etch one-step Adper Prompt (ADP, 3M ESPE AG, Seefeld, Germany), and 4) self-etch all-in-one iBond (IBO, Heraeus Kulzer GmbH), all combined with a Charisma microhybrid composite (Heraeus Kulzer GmbH). Their composition and working protocols are summarized in Table 1. The adhesive systems and the composite material were applied strictly according to the manufacturer's instructions and light-cured with a halogen lamp (Elipar Trilight, 3M ESPE AG). The lamp output power of 800 mW/cm^2 was monitored at regular intervals by an integrated radiometer. Bleaching was performed with a 20% carbamide peroxide bleaching gel (Opalescence PF 20, Ultradent Products Inc, South Jordan, UT, USA), which is routinely used for home bleaching. The gel, which has an equivalent concentration of 6.6% of hydrogen peroxide, was stored at 4°C and used in the first quarter of its shelf life to minimize its decomposition and the risk of decreased carbamide peroxide concentration.

Design of the Study

Sound human third molars extracted for orthodontic reasons were used in this study. After extraction, the teeth were cleaned and stored for one week in a 0.5% chloramine-T aqueous solution and then in distilled water at 4°C as recommended by International Organization for Standards Technical Specification 11405:2003. The teeth were used within six months after extraction. The tooth roots were cut off by an Isomet low-speed saw using a diamond wafering blade in water spray (Buehler Ltd, Lake Bluff, IL,

Table 1: *Materials 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%) *Bond:* HEMA, 4-META, polyacid, ethanol, photoinitiators, polyacrylic acids	e (20 s), r, d (1–2 s), 3× b (15 s), w (15 s), d, c (20 s)
Clearfil SE Bond (CLE)	Kuraray Mediacal Inc, Okayama, Japan	*Primer:* MDP, HEMA, hydrophilic dimethacrylate, camphorquinone, N,N-diethanol-p-toluidine, water *Bond:* MDP, bis-GMA, HEMA, hydrophobic dimethacrylate, camphorquinone, N,N-diethanol-p-toluidine, silanated colloidal silica	p (20 s), d, b, d, c (10 s)
Adper Prompt (ADP)	3M ESPE AG, Seefeld, Germany	*A liquid:* methacrylated phosphoric esters, bis-GMA, initiators based on camphorquinone, stabilizers *B liquid:* water, HEMA, polyalkenoic acid, stabilizers	m (A+B), a (15 s), d, a, d, c (10 s)
iBond (IBO)	Heraeus Kulzer GmbH	4-META, UDMA, glutaraldehyde, acetone, water, photoinitiators, stabilizers	3× a, w (30 s), d, c (20 s)
Composite			
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 percentage, sodium fluoride 0.25 weight percentage potassium nitrate	25× 8 hours

Abbreviations: bis-GMA, bisphenol A diglycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; 4-META, 4-methacryloxyethyl trimellitic anhydride; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

[a] *Application protocol: a, application; b, bonding; c, curing; d, drying/spreading; e, etching; m, mixing; p, priming; r, rinsing; w waiting.*

USA), and the teeth were fixed in a stainless steel ring using the Spofacryl self-curing resin (SpofaDental as, Jicin, Czech Republic).

The 240 teeth were randomly divided into enamel and dentin groups (n=120) for shear bond strength testing. In the enamel group, the buccal or lingual enamel was ground with 600 grit P1200 SiC paper (Buehler Ltd) to yield a flat enamel surface of approximately 3–4 mm in diameter. In the dentin group, enamel was removed by a low-speed saw to expose dentin. A standard smear layer was created by grinding dentin with SiC paper P1200. The dentin and enamel groups were each divided into four subgroups (n=30) to test the four adhesive systems. These subgroups were further divided into two control groups (n=10), one group stored for one day and one group stored for two months in distilled water, and one experimental group (n=10) that was exposed to the bleaching gel 25 times for eight hours daily over two months.

Twelve more specimens with exposed enamel and dentin were prepared to characterize the conditioning ability of the adhesives.

Shear Bond Strength Test

After the adhesive system was applied on enamel or dentin and light-cured, composite build-ups were prepared using a cylindrical polyethylene mould (3.5 mm in diameter, 2 mm high) placed on the tooth surface and filled with one portion of composite and light-cured for 20 seconds. All the specimens were immersed in distilled water for one day, after which the mold was carefully removed. With the experimental groups, approximately 0.1 g of the bleaching gel was applied around the adhesive interface, and

Table 2: *Enamel Shear Bond Strength Results (Mpa) Expressed as Mean and (SD)*

Adhesive	Water				Bleaching Gel	
	1 Day		2 Months			
GLU	24.3	(4.0)	23.3	(3.8)	20.2	(2.9)
CLE	18.3	(5.4)	19.7	(3.6)	22.0	(1.5)
ADP	20.4	(2.0)	16.9	(2.6)	11.2	(3.3)
IBO	13.0	(1.9)	14.3	(4.3)	8.9	(3.1)

the specimens were then wrapped in Parafilm foil (Parafilm M, Alcan Packaging, Chicago, IL, USA) with a small piece of wet absorbent cotton around the stainless steel ring to keep the standard level of moisture. After eight hours, the bleaching gel was carefully removed using distilled water and a soft toothbrush, and the specimens were left in distilled water until the next application of bleaching gel. The two control groups were stored in distilled water for one day and for two months, respectively. The specimens were kept in an incubator at 37°C under all storage conditions. After being exposed, the specimens were fixed in a shear bond testing device (Bencor-Multi-T, Danville Engineering Co, San Ramon, CA, USA) and stressed with a universal testing machine (AGS-G, Shimadzu, Kyoto, Japan) at a crosshead speed of 0.75 mm/min. Shear bond strength (MPa) was calculated by dividing the force at the fracture by the area of the bonded interface.

Microscopy of Fractured Surfaces

Fractographic analysis was performed using a stereomicroscope (SMZ 2T, Nikon, Tokyo, Japan) at magnifications from 10× to 60×. A detailed analysis was made by a scanning electron microscope (SEM, JSM 5500-LV, Jeol, Tokyo, Japan) at magnifications from 20× to 6000×. Before SEM observation, the specimens were left to dry for one week at room temperature and then sputter-coated with gold (JFC-1200 Fine coater, Jeol). According to the location of fracture line, the fractures were assigned to one of the following categories: 1) interfacial fractures located between the substrates at the dentin/enamel-adhesive resin interface plus fractures in the adhesive resin and the hybrid layer; 2) cohesive fractures in the substrates where fractures propagated through the tooth tissues or composite material; 3) mixed mode of fracture, including interfacial and cohesive fractures. The area fraction of interfacial and the cohesive fracture category on the dentin side were measured using image analysis software (SigmaScan 5, SPSS Inc, Chicago, IL, USA) on SEM images at 25× magnification.

Conditioning Properties of Adhesive Systems

The priming components of each adhesive system were applied onto enamel and dentin according to the manufacturer's recommendation (Table 1). After rinsing with water (GLU) or acetone (CLE, ADP, IBO) to remove the adhesive monomers, the specimens were air-dried and analyzed using an SEM.

Statistical Analysis

To compare the initial bonding performance of the adhesives, statistical analysis of the shear bond strengths of one-day control groups was carried out using nonparametric Kruskal-Wallis multiple comparisons of mean ranks. To analyze the effect of bleaching gel on enamel and dentin bond strength of each adhesive system, the difference between control groups stored in distilled water for one day and for two months was first determined using the nonparametric Mann-Whitney U test. If no significant time effect was revealed, the bond strength data measured in water were pooled and compared with those of the experimental bleached group using the same statistics. In contrast, the bleached group was compared with the specimens stored in distilled water for two months. All the statistical analyses were performed at a confidence level of 95% and calculated using statistical software (Statistica 7.1, StatSoft Inc, Tulsa, OK, USA).

RESULTS

Shear Bond Strength

The mean shear bond strength values and standard deviations (SDs), together with nonparametric characteristics represented by median, first and third quartiles, and minimum and maximum values on enamel and dentin for each group, are shown in Tables 2 and 3 and Figures 1 and 2. As shown, the one-day bond strength on enamel decreased as follows: GLU > ADP > CLE > IBO, with the GLU ($p<0.00001$) and ADP ($p<0.002$) significantly exceeding the IBO bond strength. On dentin, the bond strength decreased as follows: CLE > GLU > ADP > IBO, and the IBO bond strength was significantly lower ($p<0.03$) than the CLE bond strength. After two months in water, the adhesives showed low

Table 3: *Dentin Shear Bond Strength Results (Mpa) Expressed as Mean and (SD)*

Adhesive	Water				Bleaching Gel	
	1 Day		2 Months			
GLU	22.3	(2.1)	24.1	(2.7)	19.6	(4.8)
CLE	25.0	(4.2)	26.9	(3.9)	22.0	(1.8)
ADP	20.7	(2.9)	17.2	(4.4)	14.5	(2.1)
IBO	17.5	(6.7)	20.0	(3.9)	13.4	(2.3)

decrease or increase in bond strength compared with the one-day groups (Tables 2 and 3). Statistical evaluation (Figures 1 and 2) showed that, except for ADP on dentin, the differences between the two-month and one-day groups were not significant. Therefore, the bond strengths of these systems measured in water were pooled for the subsequent statistical evaluation of the bleaching gel effect. With ADP on dentin, however, the two-month group was used as a control. Statistical analysis showed that GLU and CLE bond strength on enamel and dentin changed significantly after bleaching compared with water ($p = 0.02$-0.03) (Figures 1 and 2). With ADP and IBO, the bond strength on enamel and dentin dropped significantly after exposure to bleaching gel with lower values of p=0.003–0.0005, which unambiguously indicated adhesive joint degradation.

Fractographic Analysis

Enamel—After exposure to water and bleaching gel, the fractured surfaces of GLU, CLE, and IBO showed a typical prismatic texture with grooves resulting from abrasive particles of SiC papers and small areas of adhesive resin remnants (Figure 3), suggesting failure predominantly at the enamel-adhesive resin interface. With ADP groups in water, large areas of cohesively fractured adhesive resin with cracks perpendicular to the load direction were found (Figure 4A). After bleaching, however, there were two irregular zones of failure: an outer rim and a central zone (Figure 4C). Although the fracture character of the central zone was not different from that of the control groups, at the outer rim debris of composite, filler particles and small voids were found after particle debonding (Figure 4D), which indicated a failure between the top of the adhesive resin and the composite. Similar voids were observed on the composite side of the outer rim and on the composite build-up areas directly exposed to the bleaching gel, indicating filler particle debonding from the composite matrix.

Dentin—With the GLU and CLE groups, a high number of mixed fractures were found within composite or dentin after water storage. Although the extent of cohesive fractures within the substrate in CLE was retained after bleaching, it decreased

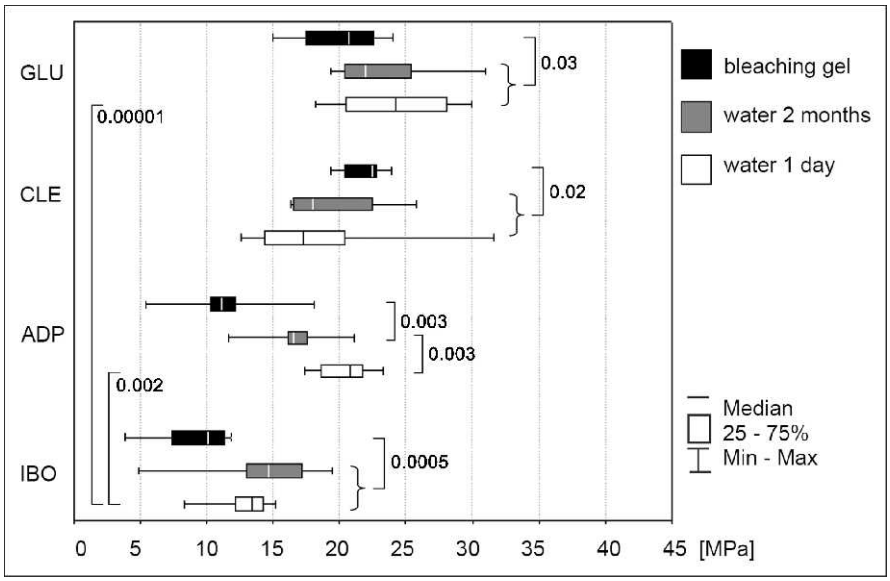

Figure 1. Enamel shear bond strength of the adhesive systems in the tested environments. Braces indicate pooled data. Statistically significant differences are marked by vertical lines and corresponding p values.

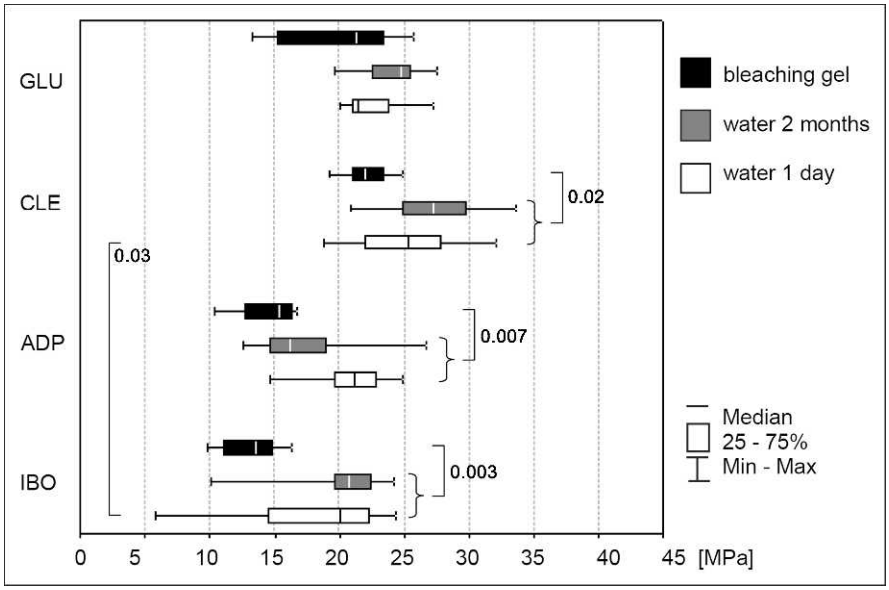

Figure 2. Dentin shear bond strength of the adhesive systems in the tested environments. Braces indicate pooled data. Statistically significant differences are marked by vertical lines and corresponding p values.

with GLU in favor of the interfacial type (Fig 7). Different behavior was found with self-etch adhesives ADP and IBO. With ADP, cohesive fractures within the adhesive resin or interfacial fractures between adhesive resin and composite occurred after water storage. After exposure to bleaching gel, the outer rim and central zone were observed on the fractured surface as on enamel, but with different fracture characteristics (Figure 5A). The central zone (Figure 5B) showed a similar fracture character as it did after water storage. In the outer rim, however, the fracture line passed through the adhesive resin but it had smooth fractured surfaces and no pronounced morphologic features (Figure 5C). With IBO in water, we observed areas of fractures between dentin and adhesive resin with resin tags in tubuli orifices (Figure 6A) and areas of cohesive fractures within adhesive resin with composite filler particles on top (Figure 6B). There were voids at the composite-adhesive resin interface on some specimens (Figure 6C), as frequently found in similar adhesives.[16] After the bleaching, the fractures between dentin and adhesive resin prevailed (Figure 6D).

Conditioning Properties of Adhesive Systems

The enamel and dentin surfaces after priming are shown in Figure 8. Gluma Etch 20 Gel etched the dental tissues most aggressively as the typical prismatic enamel structure and opened dentinal tubuli were clearly visible on the etched surfaces. An aggressive etching was also found for ADP, where wide open tubuli and areas of etched enamel with prismatic structure resembled the pattern created with phosphoric acid. However, less distinguishable enamel prisms in some areas suggested sensitivity of its conditioning properties on the enamel structure. On the other hand, enamel covered with the smear layer with grooves originating from the abrasive particles of SiC paper were typical of the lower etching ability of mild CLE and IBO adhesives. Even though the smear layer on dentin was removed and the dentin tubuli opened, deep grooves on its surface confirm their lower demineralization properties.

DISCUSSION

Many studies have shown that long-term exposure to water decreases the bond strength between tooth tissues and composite materials. The extent and mechanism of bond degradation largely depends on the composition of the adhesive system, its application protocol, the substrate to be bonded, and the length of water exposure.[1, 17-19] Much less information, however, is available showing how chemical substances or products intended for use in the oral cavity affect adhesive bond durability. Such products include, among others, peroxide bleaching gels for vital bleaching, which are one of the most frequently used cosmetic oral cavity treatments today. Three studies[12-14] dealing with the postoperative effect of bleaching gels on bond strength provide controver-

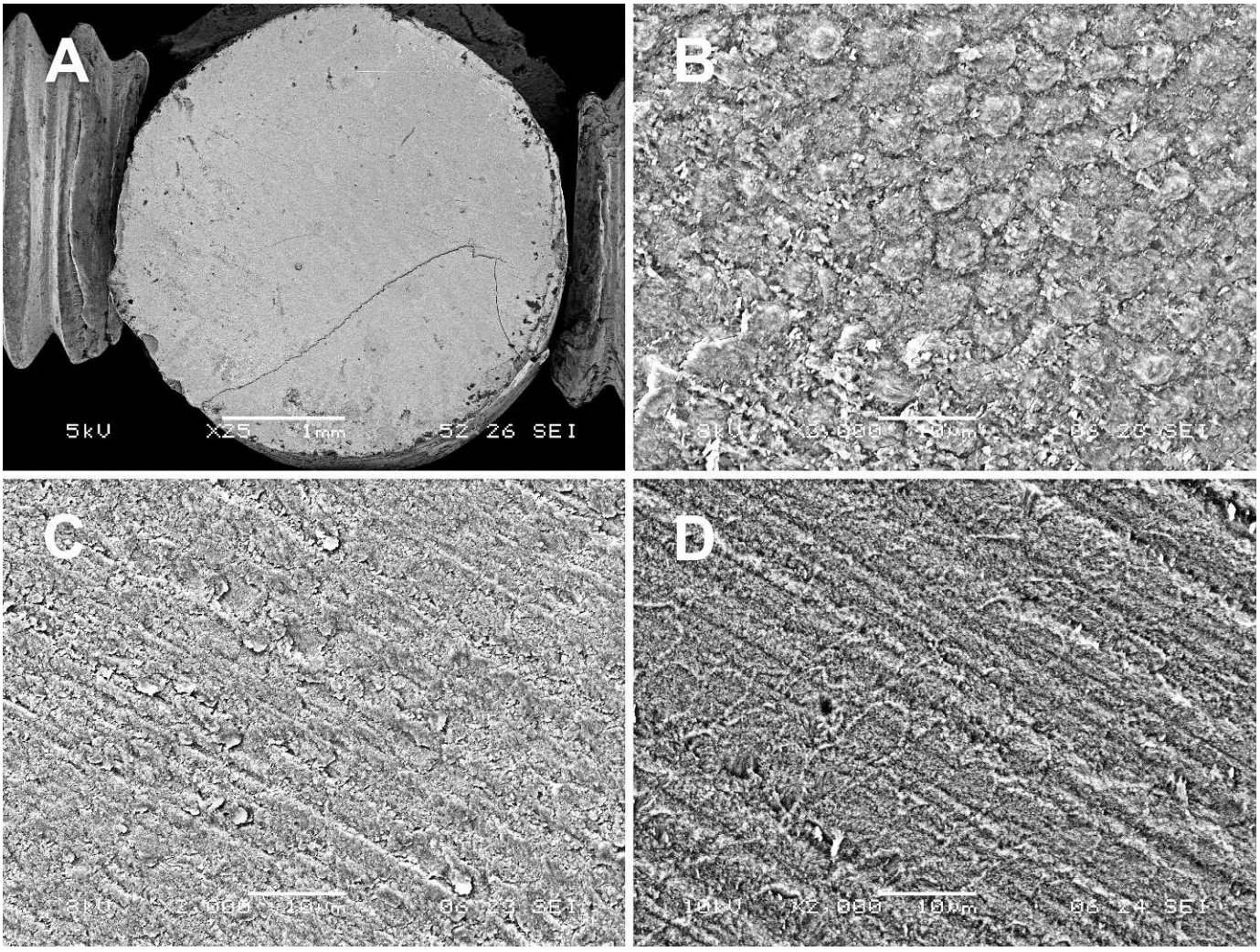

Figure 3. Representative enamel-composite fracture surfaces of GLU, CLE, and IBO specimens after water and bleaching gel expositions. (A): GLU, composite side, with fracture between the adhesive layer and enamel (25×). (B): GLU, enamel side, with enamel prisms clearly visible (2000×). (C): CLE, enamel side, surface covered with the remnants of smear layer with grinding grooves and shallow depressions resembling prismatic structure (2000×). (D): IBO, enamel side, prism borders on the grooved enamel surface (2000×).

sial results. Cavalli and others[12] used a microtensile bond strength method to study the resistance of enamel-composite and dentin-composite bonds created with the two-step etch-and-rinse Single Bond and the two-step self-etch Clearfil SE Bond. After bleaching with a 10% carbamide peroxide gel for six hours daily over two weeks, a decrease in the enamel-composite bond strength for Clearfil SE Bond was found. No significant influence of the bleaching gel on the dentin-composite bond strength was reported. On the other hand, Barcellos and others,[13] using a two-step etch-and-rinse Adper Single Bond 2, observed a decrease in the microtensile bond strength to bovine enamel and dentin after treatment with bleaching gel containing 15% and 20% carbamide peroxide for six hours daily for two weeks. Degradation of the dentin-composite bond created with the two-step etch-and-rinse Single Bond was reported by Far and Ruse,[14] who tested its resistance to bleaching gels of increasing carbamide peroxide concentrations by measuring fracture toughness. Their results show that carbamide peroxide gels at concentrations higher than 11% compromised fracture toughness of the dentin-composite joint after 70 hours of cumulative exposure. A change in fracture mode from cohesive fracture within the adhesive resin to fracture between dentin and adhesive resin interface suggested degradation at the dentin-adhesive interface.

As bond strength and its susceptibility to degradation depend on the adhesive system,[20-22] a few typical adhesives with different working protocols

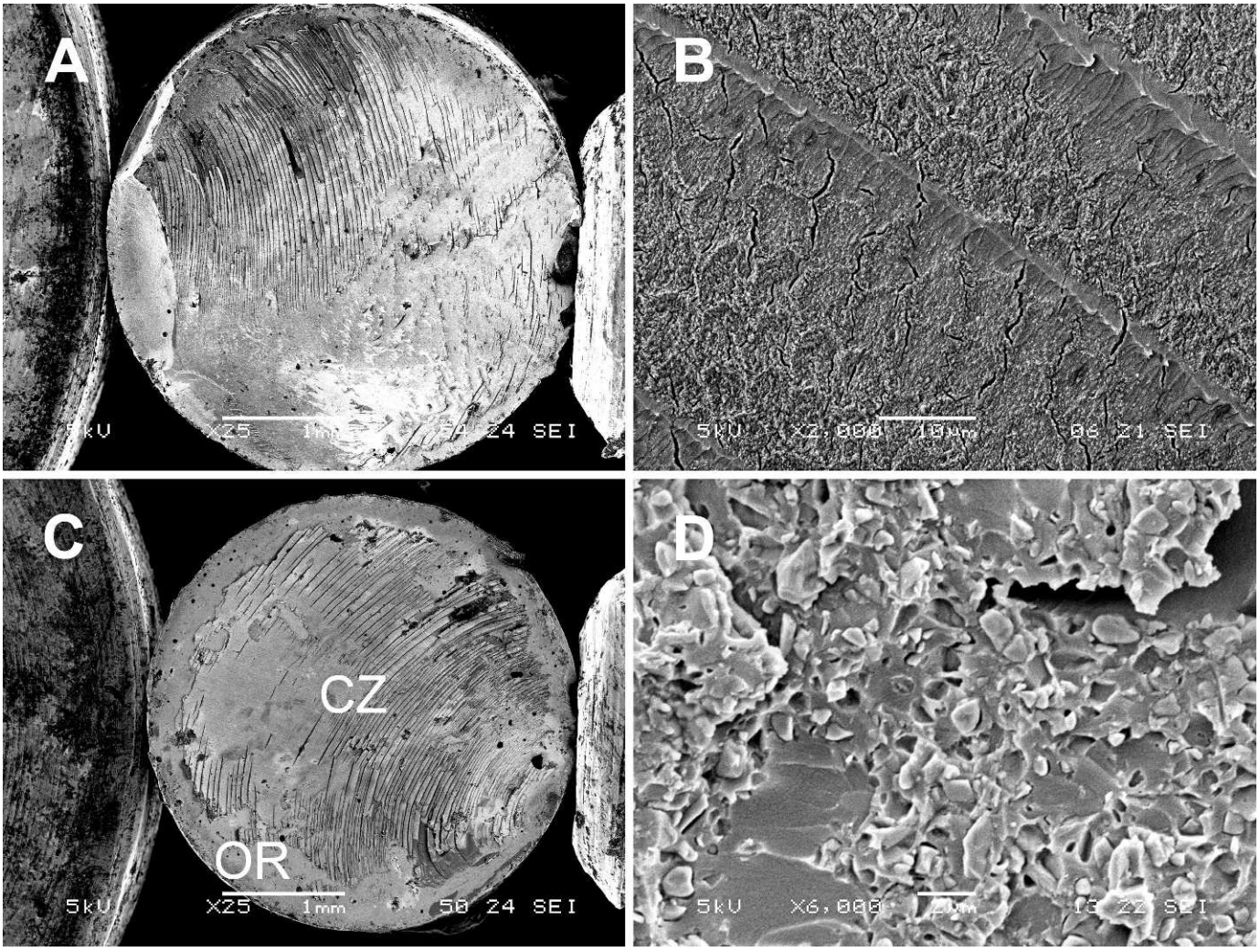

Figure 4. Representative enamel-composite fracture surfaces of ADP specimens. (A): Water, two months, composite side, fracture between adhesive resin and enamel (25×). (B): Enamel side corresponding to (A), enamel prisms and remnants of adhesive resin (2000×). (C): Bleaching gel, composite side, the outer rim and the central zone (25×). (D): Bleaching gel, enamel side, detail of the outer rim, top of the adhesive resin layer covered with remnants of composite material and voids after debonded composite filler particles (6000×). OR, the outer rim; CZ, central zone.

representing the state-of-the-art of current adhesive technology were chosen in our study. The bleaching gel containing 20% of carbamide peroxide as a representative of safe in-office and home systems[23] was applied in 25 cycles each for eight hours as recommended by the manufacturer for night bleaching. A statistical analysis revealed differences in the resistance of the bond strength of individual adhesive systems to the bleaching gel (Figures 1 and 2). Etch-and-rinse adhesive GLU and self-etch CLE, which rank among the most reliable adhesive systems proven *in vitro* and in clinical tests,[24,25] showed slight but significant changes in bond strength on enamel and, in the case of CLE, on dentin (Figures 1 and 2). Fractographic analysis revealed predominantly interfacial fractures between enamel and adhesive resin and mixed fractures within dentin or within composite on dentin groups; however, there was no pronounced change after bleaching gel application (Figure 7). Mixed fractures, including substrates, occur frequently in the shear bond strength measurement, resulting from uneven stress distribution in this experimental setup and differences in the mechanical properties of dentin and a composite.[26,27] In these cases, bond strength should be evaluated with care as the basic requirements on interfacial fracture are not met and the calculated bond strength will therefore not truly reflect the actual bond strength. With these adhesive systems,

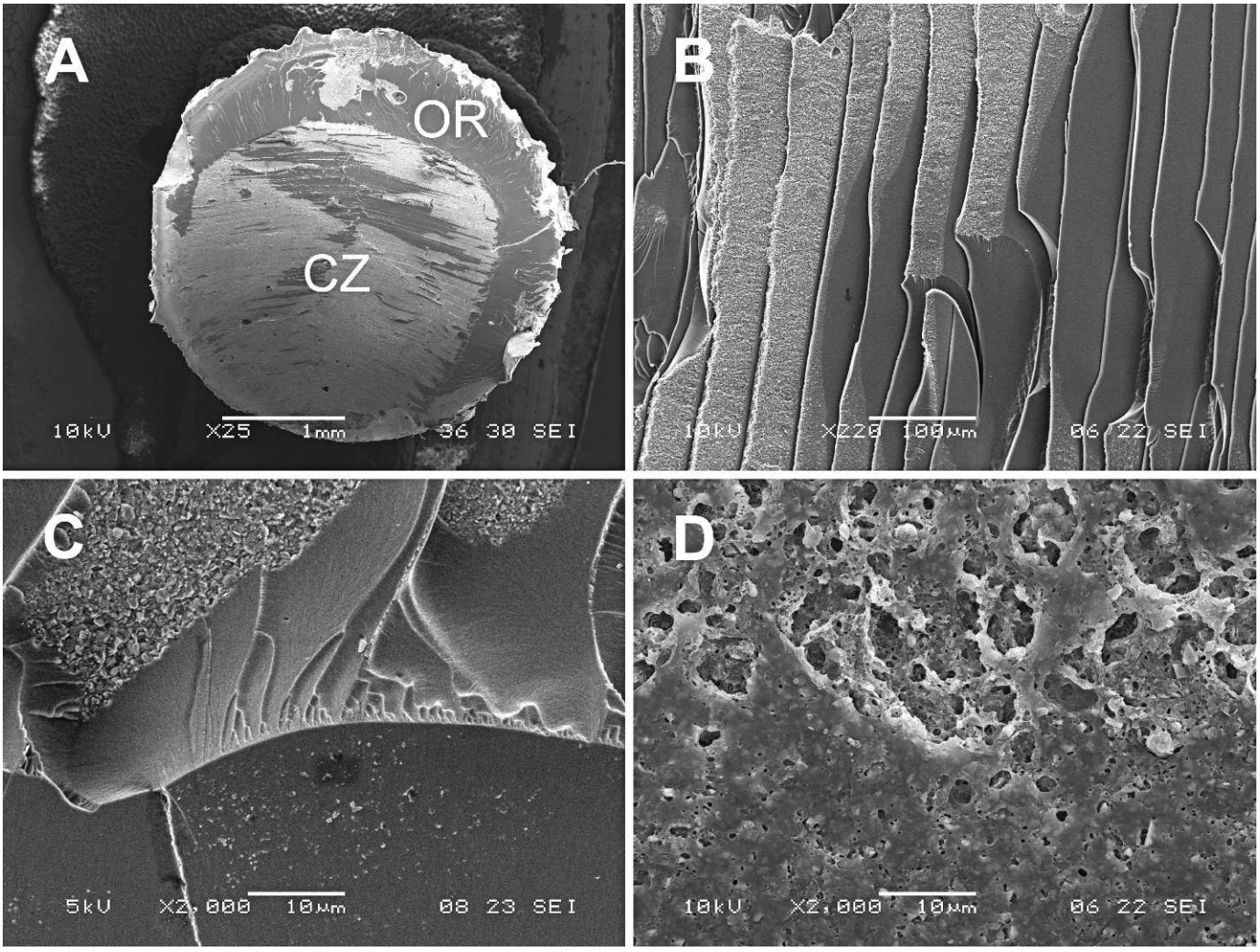

Figure 5. Representative dentin-composite fracture surfaces of ADP specimens after bleaching. (A): Composite side, the outer rim (OR) and the central zone (CZ) (25×). (B): Dentin side, detail of the CZ, cohesive fracture within the adhesive layer, and composite filler particles on its top (220×). (C): composite side, border line between the OR and the CZ, cohesive fracture in adhesive layer and fracture extending into composite material (2000×). (D): composite side, small region of voids in the OR (2000×).

the statistical significance p=0.02–0.03, close to the critical value p=0.05, and a considerable number of mixed fractures within both substrates do not allow for a reliable and unambiguous conclusion concerning the effect of a bleaching gel on their bond strength.

On the other hand, a significant decrease in the bond strength of ADP and IBO after bleaching with a low statistical significance p=0.007–0.0005 (Figures 1 and 2) indicates bond degradation that was more pronounced than in water. With ADP, bond strength degradation was also reflected in the pronounced change of fracture pattern at the periphery of specimens in close contact with the bleaching gel. In this area of irregular depth, fractures located at the adhesive resin-composite interface on enamel or within adhesive resin on dentin were found (Figure 4). This outer rim, which resembled the fractured surfaces of adhesive joints exposed to degradation in NaOCl solutions,[28] indicated gradual penetration of the bleaching gel reactive components into the adhesive joint and its degradation. The irregular depth of the outer rim might have resulted from the different thickness of the adhesive layer or an imperfectly defined adhesive edge during the preparation of composite build-ups onto tooth tissue surfaces.

These results show that peroxide bleaching gels may induce degradation of adhesive bonds between the composite material and tooth tissues. Its extent, however, depends largely on the composition of the adhesive system. Hence, the null hypothesis on

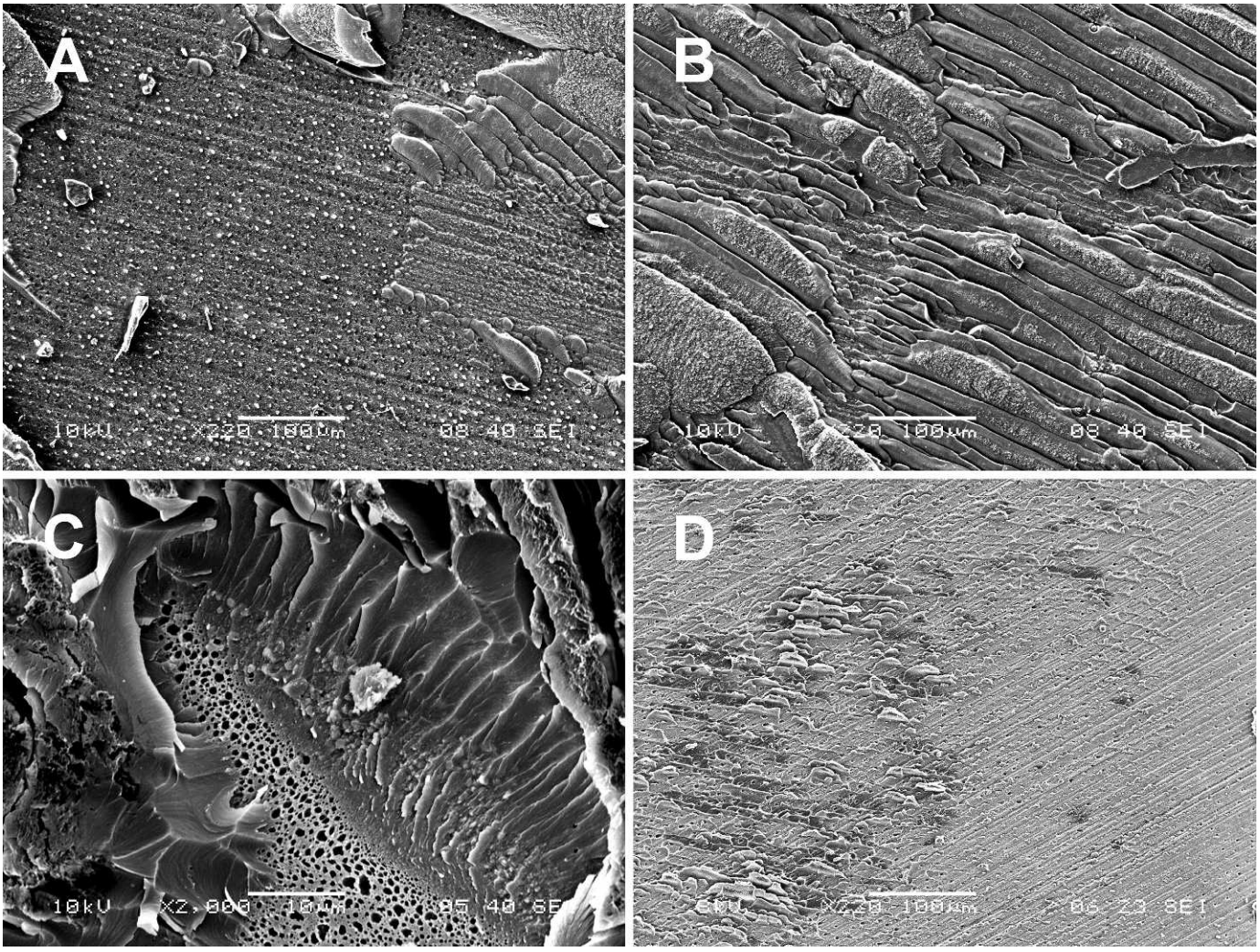

Figure 6. Representative dentin-composite fracture surfaces of IBO specimens. (A): Water, one day, dentin side, fracture between dentin and adhesive resin with grooves created by grinding particles and small areas of cohesively failed adhesive resin (220×). (B): Water, one day, dentin side, cohesive fracture within adhesive resin with composite filler particles on its top (220×). (C): Water, one day, composite side, small region of voids at composite-adhesive resin interface (2000×). (D): Bleaching gel, dentin side, fracture between dentin and adhesive resin with grooves from grinding and remnants of adhesive resin (220×).

resistance of adhesive joints to the peroxide bleaching gels can be rejected.

The bond strength studies revealed acceptable long-term stability of enamel-composite bond created with etch-and-rinse[29,30] and decreased stability of the bond created with self-etch adhesives.[31] The dentin-composite bond stability, on the other hand, is usually lower than that of the enamel-composite bond. The bottom of the dentin hybrid layer, particularly improperly impregnated collagen fibrils, is the most frequently attacked zone. Hydrolysis of collagen fibrils, their enzymatic decomposition, and adhesive resin hydrolysis are assumed to be the main mechanisms of dentin bond degradation[32] and occur after a few months of water storage.[30] Lower resistance of some self-etch adhesives arises not only from the presence of incompletely impregnated collagen fibrils,[33] as with etch-and-rinse adhesives, but also from their considerable hydrophilic properties, which allow water to penetrate into the adhesive joint,[34] causing water treeing,[35] swelling, and plasticization of the adhesive polymer matrix.[34] With these adhesives, defects that arise because of imperfect polymerization in the presence of water,[36] voids, blisters, and water droplets resulting from phase separation of nonmiscible components of some adhesives or from penetration of water from dentin to adhesive-composite interface[37,38] should be considered. Such defects may act as stress concentrators, accelerat-

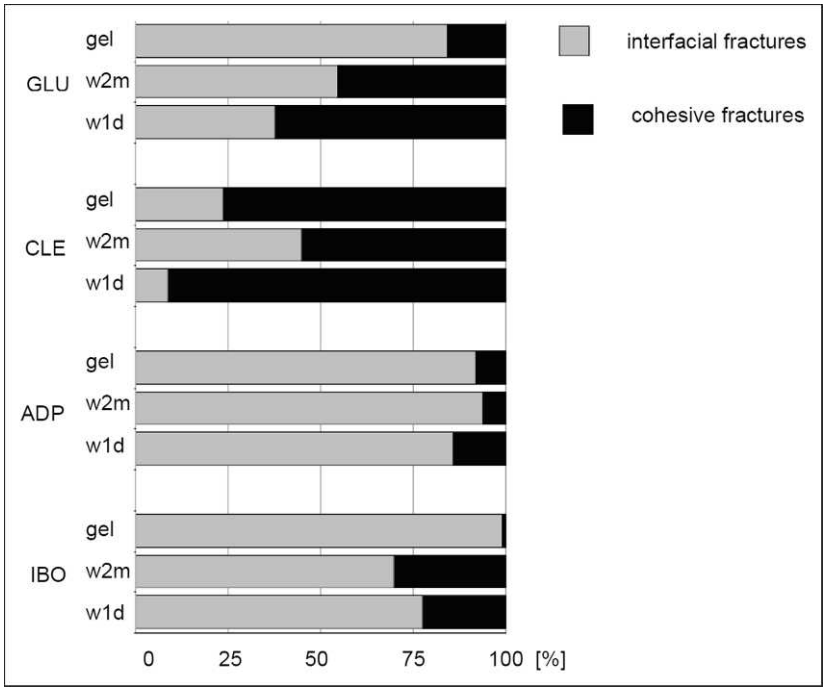

Figure 7. Distribution of fracture categories for dentin groups expressed as area percentage. Bleaching gel (gel); water, two months (w2m); water, one day (w1d).

ing water penetration and diminishing adhesive joint mechanical strength. In addition, the bond strength of adhesive systems also depends on their ability to remove and demineralize the smear layer and subsurface tooth tissues after tooth preparation. We assume that the GLU high bond strength and high bond resistance to bleaching and water resulted from optimally etched enamel and dentin surfaces with numerous microretentions created with a phosphoric acid etching (Figure 8). With self-etch CLE, which has weaker etching properties (Figure 8), a chemical bond between the MDP monomer and the hydroxyapatite of tooth tissues[39] may be a key factor contributing to its high bond strength and resistance to bleaching gel. In addition, a hydrophobic resin of this two-step system might seal primed tissues and form a barrier against water and active substances penetrating from the bleaching gel in the adhesive joint.[21] The lower bond resistance of one-step self-etch adhesive ADP to the bleaching gel is not surprising. This adhesive contains water necessary for the ionization of acidic monomers and 2-hydroxyethyl methacrylate (HEMA), which dissolves hydrophilic and hydrophobic components, enabling a homogeneous solution to be formed. Because of its high boiling point, HEMA is not as effective in removing water from the adhesive applied on dental tissues as the acetone or ethanol contained in other systems; thus, water retained in the adhesive structure might decrease its degree of polymerization[36,40] Such a system will be prone to water sorption and to degradation, as reflected in the decrease in ADP bond strength even after storage in water for two months. For the predecessor of ADP, the adhesive Prompt L-Pop (3M ESPE AG, Seefeld, Germany), which had similar composition, it was reported that because of the reaction of its acidic components with basic amines present in the composite photoinitiation system, composite polymerization may have been inhibited, resulting in decreases in bond strength.[41] Although such decreases did not occur until a few minutes after their contact, it might have started as soon as the uncured composite with a camphorquinone-amine photoinitiating system was applied on incompletely polymerized adhesive resin. In the enamel group, the outer rim surface covered with filler particles (Figure 4D) suggests that this region of the ADP resin-composite interface was the primary site of the bleaching gel penetration and attack. In contrast, a smooth fracture surface of the outer rim in the dentin group indicated a fracture inside the adhesive resin (Figure 5C). This may be due to a higher water content absorbed by ADP from wet dentin, easier penetration of the reactive bleaching gel compo-

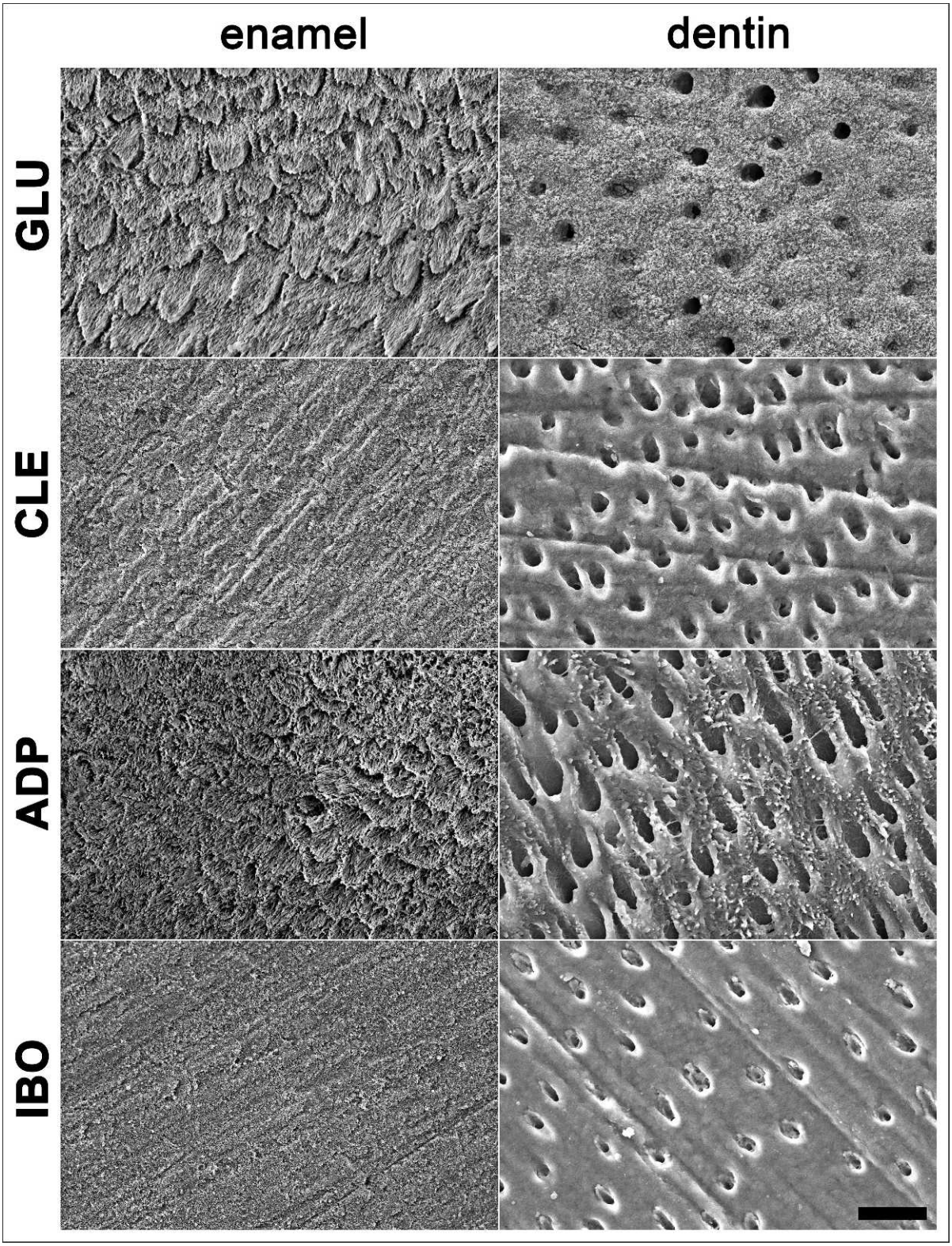

Figure 8. Morphology of enamel and dentin after conditioning. GLU Etch 20 was applied for 20 seconds. Note the prismatic structure of enamel. Dentin with open dentinal tubuli is covered with pyrogenic SiO_2 particles used as a thickening agent. CLE primer was applied for 20 seconds. SiC paper grinding grooves are visible on both enamel and dentin. Smear layer covers enamel but dentinal tubuli are opened. ADP was applied for 15 + 10 seconds. The smear layer is removed, and enamel prisms and open dentinal tubuli are visible. IBO was applied for 30 seconds. The smear layer is not removed from enamel, and dentinal tubuli with visible orifices and grinding grooves are clearly apparent. The bar length indicates 10 μm.

nents through the adhesive structure, and its fast oxidative degradation.[42] The question arises as to why similar behavior was not observed with IBO, too. It contains the less aggressive organic 4-methacryloxyethyl trimellitic anhydride (4-META) primer, which has demineralization properties that are not as efficient as those of ADP, which is obvious from its inability to remove the smear layer from highly mineralized enamel (Figure 8) and its initial shear bond strength being the lowest of all tested adhesives. Predominantly enamel or dentin-adhesive resin interface failure suggested that, as a result of the lower demineralization efficiency of IBO, this interface was the weakest region of the IBO adhesive joint and the most susceptible to degradation.

CONCLUSIONS

Our study has shown that carbamide peroxide bleaching gel may cause degradation of adhesive bonds. However, the type of the adhesive system used seems to be the factor determining the susceptibility of the adhesive bond to the influence of bleaching gel.

Acknowledgements

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Conflict of Interest

The authors of this article 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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Two-Year Interfacial Bond Durability and Nanoleakage of Repaired Silorane-Based Resin Composite

E Mobarak • H El-Deeb

Clinical Relevance

Silane application is not mandatory for repairing silorane-based resin composite, which can be successfully repaired even with different repair adhesive/materials. However, early signs of nanoleakage can be detected.

SUMMARY

Objectives: To investigate the effect of silane primer application, intermediate adhesive agent/repair composite, and storage period on the interfacial microtensile bond strength (μTBS) of repaired silorane-based resin composite compared with unrepaired composites and on the nanoleakage.

Methods: Forty-eight 1-month-old substrate specimens from Filtek P90 were roughened, etched, and distributed over two groups (n=24) based on receiving silane (Clearfil Ceramic Primer) or not. Then, half of the specimens (n=12) were repaired with P90 System Adhesive/Filtek P90 and the other half with Adper Scotchbond Multipurpose adhesive/Filtek Z250 resin composite. Within each repair category, repaired specimens were stored in artificial saliva at 37°C for either 24 hours (n=6) or two years before being serially sectioned into sticks (0.6 ± 0.01 mm^2). From each specimen, two sticks were prepared for nanoleakage determination and four sticks were used for μTBS testing. Additional unrepaired specimens from each composite (n=12) were made to determine the cohesive strength at 24 hours and two years. Mean μTBS were calculated and statistically analyzed. Modes of failure were also determined.

Results: General linear model analysis revealed no significant effect for the silane priming, intermediate adhesive agent/repair composite, and storage period or for their interactions on the μTBS values of the repaired specimens. There was no significant difference between the cohesive strength of Filtek P90 and Filtek Z250; both were significantly higher

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than all repaired categories. At 24 hours, nanoleakage was not detected when silorane-based composite was repaired with the same material. However, after two years, all repair categories showed nanoleakage.

Conclusions: Silane application has no effect on μTBS and nanoleakage. Durability of the interfacial bond of repaired silorane-based resin composite appeared successful regardless of the chemistry of the intermediate adhesive agent/composite used for repair. However, nanoleakage was detected early when a different repair intermediate adhesive agent/composite was used.

INTRODUCTION

The concept of minimally invasive dentistry opened the way for conservative tooth restorations as well as for repairing defective preexisting restorations rather than completely replacement them.[1] Researchers have shown that the repair of tooth-colored restorations is a conservative, time-saving, cost-reducing, effective method in which the intact part of the restoration can be maintained and unnecessary removal of dental hard tissues and pulp irritation is avoided.[1–3]

Currently, different tooth-colored resin composites are available, meaning that composite monomers other than dimethacrylates are being used. In 2005, a silorane-based composite was introduced. Because of its modified matrix consisting of siloxane and oxirane components, silorane-based composite exhibits a reduced shrinkage of approximately 1% by volume due to ring-opening cationic polymerization.[4] Because of its recent introduction, little is known about the repairing ability of this category of resin composites.

Different repair approaches of silorane composites were recently investigated.[5–11] The effect of intermediate adhesive agent/repair composite,[5,8] the impact of surface preparation,[7,9,11] or both[6,10] on silorane-based composite repair was tested. In these studies, some researchers tested the ability of methacrylate-based adhesive/composite to repair silorane restorations.[5,8–10] One study proved that fresh silorane composite can be equally repaired with the same material or with methacrylate-based composite.[9] Others[5,8,10] recommended the application of a silane-coupling agent to enhance the repair bond strength of methacrylate-based composites to aged silorane-based resin composite. However, separate application of a silane primer and a dentin adhesive can result in a thick, multiphase interfacial layer, which may introduce defects in each working step.[12] None of these studies evaluated nanoleakage with silver nitrate uptake. However, such analysis would provide good spatial resolution of submicron defects in such an interfacial layer.[13]

Although promising results were obtained from these studies, the results were about short-term bonding, which would not predict long-term clinical performance. Interfacial bond of the repaired composite may be impaired by mechanical, thermal, and chemical stresses in the intraoral environment,[14] mainly as a consequence of the limited hydrolytic stability of intermediate agents (ie, silanes and/or adhesives).[15] Thus, long-term water storage of specimens was recommended as a validated method for assessment of bond degradation.[16]

On reviewing the literature, no published data could be retrieved about the long-term repair bond durability of silorane-based resin composite. Thus, it would be of benefit to investigate the repair bond durability of silorane-based resin composite by means of microtensile bond strength (μTBS) and nanoleakage, when repaired with the same material or with methacrylate-based adhesive/composite with or without using silane primer as a repair bonding promoter. The null hypotheses tested were 1) the use of silane primer and the type of intermediate adhesive agents/repair composites do not affect the interfacial μTBS of repaired silorane-based resin composites; 2) there is no difference between silorane interfacial repair bond strength after short-term (24 hours) and after long-term (two years) storage in artificial saliva; 3) silane primer, different intermediate adhesive agent/repair composite, as well as different storage periods have no influence on interfacial nanoleakage of repaired silorane-based resin composites.

MATERIALS AND METHODS

The intermediate adhesive agents' brand names, manufacturers, chemical compositions, and batch numbers, as well as their steps of application, are listed in Table 1. Repair resin composite materials' brand names, manufacturers, batch numbers, and chemical compositions are listed in Table 2.

Mold Fabrication

A circular split Teflon mold (substrate mold) 30 mm in diameter and 4 mm in height was specially constructed. It had a central hole of 4 mm diameter in which the substrate resin composite specimen was

Table 1: *Silane Primer and Intermediate Adhesive Agents*

Brand Name (Manufacturer)	Chemical Composition (Batch Number)	Steps of Application
Scotchbond etchant gel (3M ESPE, St Paul, MN, USA)	35% by weight phosphoric acid, 60% water, and 5% synthetic amorphous silica as thickening agent (N105148)	Applied for 15 s, rinsed with oil-free air water syringe for 15 s, and dried with air for 5 s
Clearfil ceramic primer (Kuraray Medical Co Ltd, Osaka, Japan)	3-Trimethoxysilylpropylmethacrylate, MDP, ethanol (0007BA)	Applied for 60 s and gently air dried
P90 System Adhesive (3M ESPE, Seefeld, Germany)	Primer: Phosphoric acid methacryloxy-hexylesters mixture, 1,6-hexanediol dimethacrylate, copolymer of acrylic and itaconic acid, phosphine oxide, (dimethylamino) ethyl methacrylate, Bis-GMA and HEMA, water and ethanol, camphorquinone, silane-treated silica filler with primary particle size of about 7 nm; fillers % = 8-12wt% (8AY)	Primer bottle was shaking briefly before application; primer was applied with microbrush on the substrate surface and rubbed for 15 s, gently air-dried, and cured for 10 s
	Bond: TEGDMA, phosphoric acid methacryloxyhexylesters, 1,6-hexanediol dimethacrylate camphorquinone, silane-treated silica filler; fillers % = 5-10wt% (8AY)	The bottle of the bond was agitated first, then the bond was applied with a microbrush, exposed to a gentle air stream for 10 s, and cured for 10 s
Adper Scotch Bond Multipurpose Adhesive systems (3M ESPE, St. Paul, MN, USA)	Primer: HEMA, polalkenoic acid copolymer, water, ethanol (7BL)	Primer was applied on the substrate surface with a microbrush and left for 30 s, then gently air dried for 5 s
	Adhesive: Bis-GMA, HEMA (7PY)	Adhesive resin was applied with miocrobrush and cured for 10 s

Abbreviations: Bis-GMA, bisphenol A glycol dimethacrylate; HEMA, 2-hydroxyethyl-methacrylate; MDP, 10-methacryloxydecyl dihydrogen phosphate; TEGDMA, triethylene glycol dimethacrylate.

made. A second mold (repair mold) enclosed three split Teflon discs. The base disc (20 mm external diameter and 3.5 mm height) had a hole of 4 mm internal diameter and was used to hold the substrate specimen while applying the adhesive system. The other two discs were used on top of the base disc to build up the repair material. Both discs had external and hole diameters similar to the base disc. However, one was 2 mm in height and the other was 1.5 mm in height.[9] The repair mold is presented in Figure 1.

Substrate Specimen Preparation

A total of 48 Filtek P90 substrate specimens (4 mm diameter and 4 mm height) were made. The substrate mold was placed on a celluloid strip matrix (Dental Technologies Inc, Lincolnwood, IL, USA) and a glass slab. Filtek P90 shade A3 was packed in the mold in one increment of 4-mm thickness. The top surface of the substrate specimen was covered with a celluloid strip to prevent the oxygen inhibited layer. It was pressed using a glass slab to remove excess composite material before curing and to gain smooth surface of the specimen. The glass slab was removed, and each specimen was light cured using an LED light-curing unit (Blue Phase C5, Ivoclar Vivadent, Schaan, Lechtenstein) from its top and bottom for 40 seconds each. The specimen was then removed from the mold and cured from both of its sides for 40 seconds. Output light intensity ($\geq$500 mW/cm^2) was periodically checked using an LED radiometer (Kerr Dental Specialties, West Collins Orange, CA, USA). The specimens were left to stand for 15 minutes and then were immersed in artificial saliva[17] for one month at 37°C.

Repair of the Substrate Specimens and Their Storage

Following one month, each substrate surface was wet-ground flat with 320-grit silicon carbide grinding paper corresponding to the roughness obtained by diamond bur grinding.[9,18] Each specimen was then washed with tap water for 30 seconds and blotted dry. Then the surface was etched using the 37% phosphoric acid Scotchbond (3M ESPE, St. Paul, MN, USA) for 15 seconds. The etched surface

Table 2: *Repair Resin Composite Materials*

Brand Name	Manufacturer (Batch Number)	Chemical Composition
Filtek P90 Shades (A3 and B2)	3M ESPE, St Paul, MN, USA (9ET and 9BH)	Resin: 3,4-epoxycyclohexylethylcyclopolymethylsiloxane, bis-3,4-epoxycyclohexylethylphenylmethylsilane, camphorquinone; filler: silanized quartz/yttrium fluoride 0.1–2 μm; fillers loading: 53 vol% (76 wt%)
Filtek Z250 Shade (B2)	3M ESPE, St Paul, MN, USA (9AL)	Resin: Bis-GMA, UDMA, Bis-EMA; fillers: zirconia/silica 0.01–3.5 μm; fillers loading: 60 vol% (84 wt%)

Abbreviations: Bis-EMA, bisphenol A ethoxylated dimethacrylate; Bis-GMA, bisphenol A glycol dimethacrylate; UDMA, urethane dimethacrylate.

was rinsed with an oil-free air water syringe for 15 seconds and dried with air for 5 seconds from a distance of 1 cm.

Prepared substrate specimens were divided into two main groups (n=24) according to the silane primer application. Silane primer coupling agent (Clearfil Ceramic Primer Kuraray Medical Co, Osaka, Japan) was applied to one-half of the prepared substrate surfaces for 60 seconds and gently air-dried. The other half was not treated with silane. Then each group was further subdivided into two subgroups (n=12) according to the repair material, using either Filtek P90 (shade B2) with its corresponding adhesive system (P90 System Adhesive [SA]) or Filtek Z250 resin composite (shade B2) with Adper Scotchbond Multipurpose adhesive system (SBMP). Each substrate specimen was reinserted in the repair mold while the treated surface was directed upward. Adhesive systems were applied according to the manufacturers' instructions as presented in Table 1. The repairing resin composite was packed against the treated side of the Filtek P90 substrate specimen incrementally (1.5-mm thick followed by 2-mm thick). Each increment was cured for 40 seconds. A different shade was chosen for the repairing composite to enable visual identification and orientation of the repair interface during μTBS testing and failure mode analysis. Finally, each subgroup was subdivided into two categories (n=6) according to the storage periods of the repaired assembly for short-term (24 hours) and long-term (two years) in artificial saliva at 37°C. Twelve additional unrepaired substrate specimens of 4 mm diameter and 7.5 mm height were prepared from each resin composite (Filtek P90 and Filtek Z250) to test their cohesive strength[9,12] after 24 hours and two years of storage.

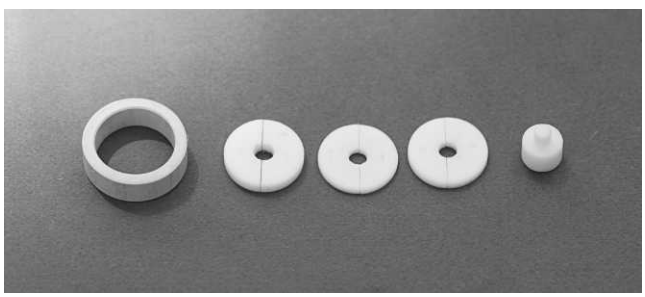

Figure 1. *Specially constructed repair mold.*

Microtensile Bond Strength Testing

After storage, specimens were fixed to the cutting machine (Isomet, low-speed saw, Lake Bluff, IL, USA) and serially sectioned to obtain multiple beam-shaped sticks. From each specimen, four sticks were tested, resulting in testing 24 sticks for each experimental category. The cross-sectional area (0.6 mm^2 ± 0.01 mm) was confirmed with a digital caliber (Mitutoyo digital caliber, Mitutoyo Corp, Kawasaki, Japan). For μTBS testing, each stick was fixed to the testing jig attached to the universal testing machine (Lloyd LRX, Lloyd Instruments Ltd, Fareham Hants, UK) using cyanoacrylate adhesive (Rocket, Dental Ventures of America Inc, Corona, CA, USA). The sticks were stressed in tension at a cross-head speed of 0.5 mm/min until failure. The load at failure was recorded in N, and the bond strength was calculated as MPa by dividing the load by the cross-sectional area at the bonded interface. The mean and standard deviation (SD) of each group were calculated. Comparison between groups was performed using the general linear model (GLM) with μTBS as the dependent variable and the surface priming, intermediate adhesive agents/repair composites and storage periods as independent variables while taking into consideration that every specimen had generated four stick values. A Bonferroni post hoc multiple-comparison test was used when indicated. The GLM was also used to test the interaction

Table 3: *Means and Standard Deviation (SD) of the Microtensile Bond Strength (μTBS) Values (MPa) of the Tested Categories*

Tested Variable	Repair Microtensile Bond Strength (MPa) Mean (SD)				Cohesive strength (MPa) Mean (SD)	
	SA/Filtek P90		SBMP/Filtek Z250		Filtek P90	Filtek Z250
	No Silane	With Silane	No Silane	With Silane		
24 hours	55.9 (8.5)[a]	56.5 (8.0)[a]	49.7 (8.6)[a]	51.8 (6.5)[a]	75.4 (8.8)[b]	65.1 (6.8)[b]
2 years	56.7 (10.6)[a]	53.7 (5.9)[a]	48.8 (5.6)[a]	54.3 (6.5)[a]	73.0 (8.3)[b]	64.5 (7.1)[b]

Abbreviations: SA, P90 System Adhesive; SBMP, Adper Scotchbond Multipurpose adhesive. Similar superscript letter indicate no significant difference (p>0.05).

between each of the two independent variables as well as the interaction among the three variables. A *t*-test was used to compare cohesive strength values of Filtek P90 and Filtek Z250 at each storage period as well as to compare the mean values of each repair category and cohesive strength values of Filtek P90 and Z250. $p<0.05$ was considered statistically significant. Data were analyzed using SPSS for Windows (Statistical Package for Social Sciences, release 15 for MS Windows, 2006, SPSS Inc, Chicago, IL, USA).

Failure Mode Analysis

Failed parts of the tested sticks were mounted on an aluminum stub, sputter coated with gold, and observed under a scanning electron microscope (SEM; 515; Philips, Einhoven, the Netherlands). Failure modes were evaluated at 100× and classified into cohesive in intermediate adhesive layer or resin composite, adhesive at the interface of the substrate side, or mixed (adhesive at the interface of the substrate side and cohesive in the adhesive layer and/or in resin composite).

Nanoleakage

Two bonded sticks from each prepared specimen at each storage period were used for nanoleakage determination. Sticks were immersed in 50% ammoniacal silver nitrate solution, which was prepared according to the protocol described by Tay and others.[19] The sticks were placed in the silver nitrate solution in darkness for 24 hours, rinsed thoroughly in distilled water, and immersed in photo-developing solution for eight hours under fluorescent light to reduce silver ions into metallic silver grains within voids along the bonded interface. Sticks were then polished using SiC paper of increasing grit size (1000, 1200, 2500, and 4000), rinsed with water for 30 seconds, and left to air-dry in a desiccator. Sticks were mounted on aluminum stubs, sputter coated with gold. The substrate composite-repair composite interface was analyzed using SEM (515; Philips) operated in the backscattered electron mode.

RESULTS

Microtensile Bond Strength

The descriptive statistics, means, and SDs for the μTBS (MPa) of the eight tested repair categories are presented in Table 3. Results of the cohesive strength values of Filtek P90 and Filtek Z250 are also shown in Table 3. GLM analysis revealed no significant effect for the silane priming application, intermediate adhesive agent/repair composite, and storage period as well as for their interactions on the μTBS values of the repaired specimens (Table 4). A *t*-test indicated no significant difference between the cohesive strength values of Filtek P90 and Filtek Z250 at each storage period, as presented in Table 3. All tested repair categories were statistically significantly lower than the cohesive strength of Filtek P90 and Z250 (Table 3).

Failure Mode Analysis

Failure mode analysis is shown in Figure 2. Cohesive fracture of the substrate and the repair materials were not seen. Overall, most failures were mainly adhesive at the substrate side followed by mixed failures. Few sticks showed cohesive failure in the intermediate layer. Representative SEM images of the fracture surfaces are shown in Figures 3 and 4.

Nanoleakage

Representative images of the tested groups can be seen in Figures 5 and 6. No silver nitrate deposits were seen for the SA/Filtek P90 repair categories either with or without silane application after 24-hour storage. However, few silver nitrate deposits

Table 4: F *and* p *Values for the Effect of the Studied Variables on Microtensile Bond Strength*

Variable	F Value	*P* Value
Surface priming	0.042	0.838
Repair material	2.366	0.126
Storage time	0.636	0.426
Surface priming * Repair material	0.923	0.338
Surface priming * Storage time	0.009	0.926
Repair material * Storage time	0.235	0.628
Surface priming * Repair material* Storage time	1.331	0.250

were seen in the SBMP/Filtek Z250 repair groups either with or without silane application after 24-hour storage. After two years of storage in artificial saliva, silver nitrate deposition was detected in all of the tested groups.

DISCUSSION

Because the dentist often has no information about the chemical composition of the existing composite, the repair of silorane-based restorations is critical. Some researchers[5,6] found that the use of silane primer might be reasonable when the composite to be repaired cannot be identified, especially when the repair system is dimethacrylate based. However, the results of the present study revealed that the use of silane primer did not improve the short- or long-term repair μTBS when a silorane-based or methacrylate-based repair system was used. Composite repair with silane application is a point of debate in many studies.[20–22] Silanes are molecules with two main

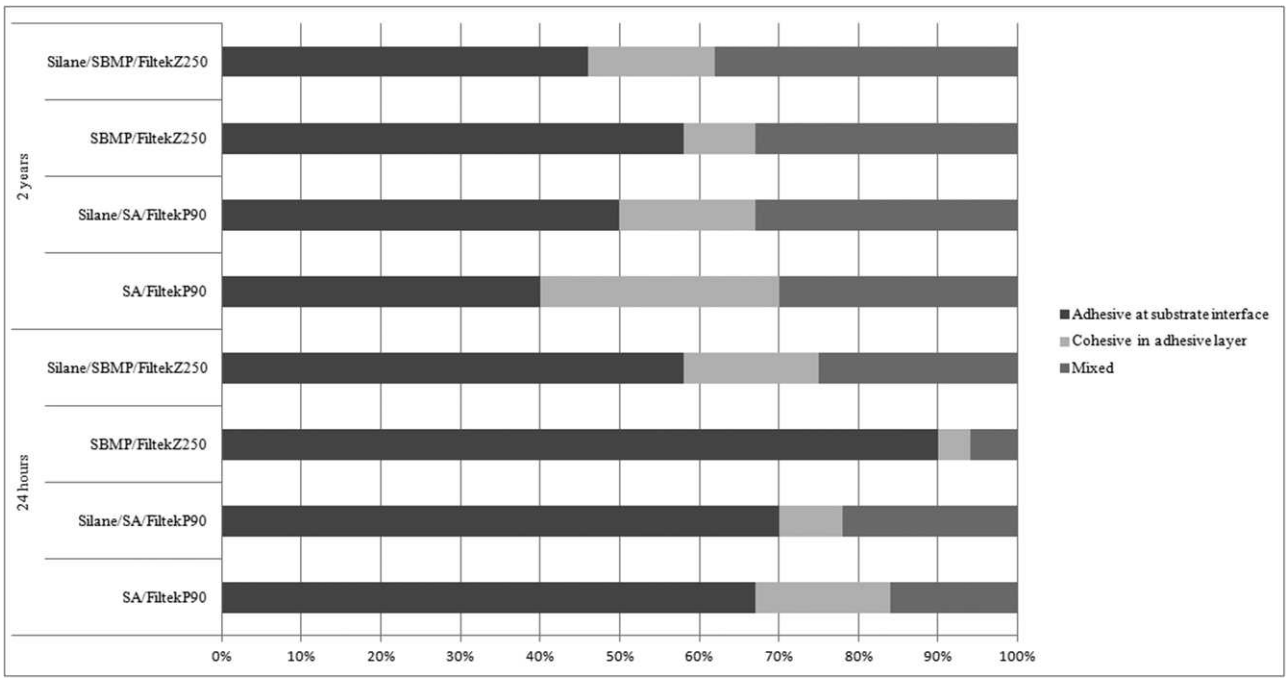

Figure 2. *Percentage distribution of failure modes obtained in the tested groups. SA, P90 System Adhesive; SBMP, Adper Scotchbond Multipurpose adhesive.*

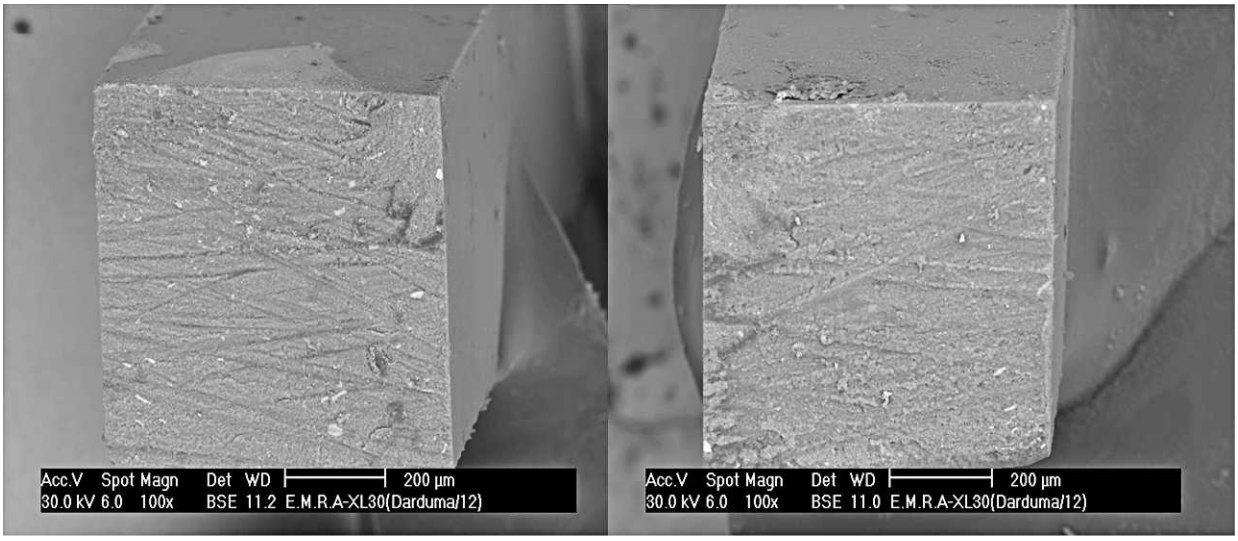

Figure 3. *SEM micrographs for representative two resulting surfaces of a stick after failure. Left: Adhesive failure at the substrate surface of Filtek P90; Right: The intermediate adhesive agent attached to the repair side.*

functional groups: the silanol, which bonds to the silica of the composite filler, and the organofunctional group, which copolymerizes to the methacrylate of the bonding agent.[15,23] The silane also enhances the wetting of the surface for the bonding agent, which is expected to infiltrate more easily through the irregularities created by the surface roughening. Previous researchers,[22,24–27] who used silane primer in repairing methacrylate-based resin composite, proved that it had no significant effect in the repair bond strength. On the contrary, some researchers[5,10] confirmed the important role of silane primer when silorane-based resin composite was repaired with methacrylate-based adhesive/repair composite but not with the silorane-based intermediate adhesive agent/repair composite. However, it should be emphasized that these studies used a shear mode of testing as well as different methacrylate-based intermediate adhesive agents. The risk of silane priming as a step in the repair protocol is tooth substrate contamination.[5] This is not yet clear and requires further research.

In this study, there was no significant difference between silorane-based composite repaired with a

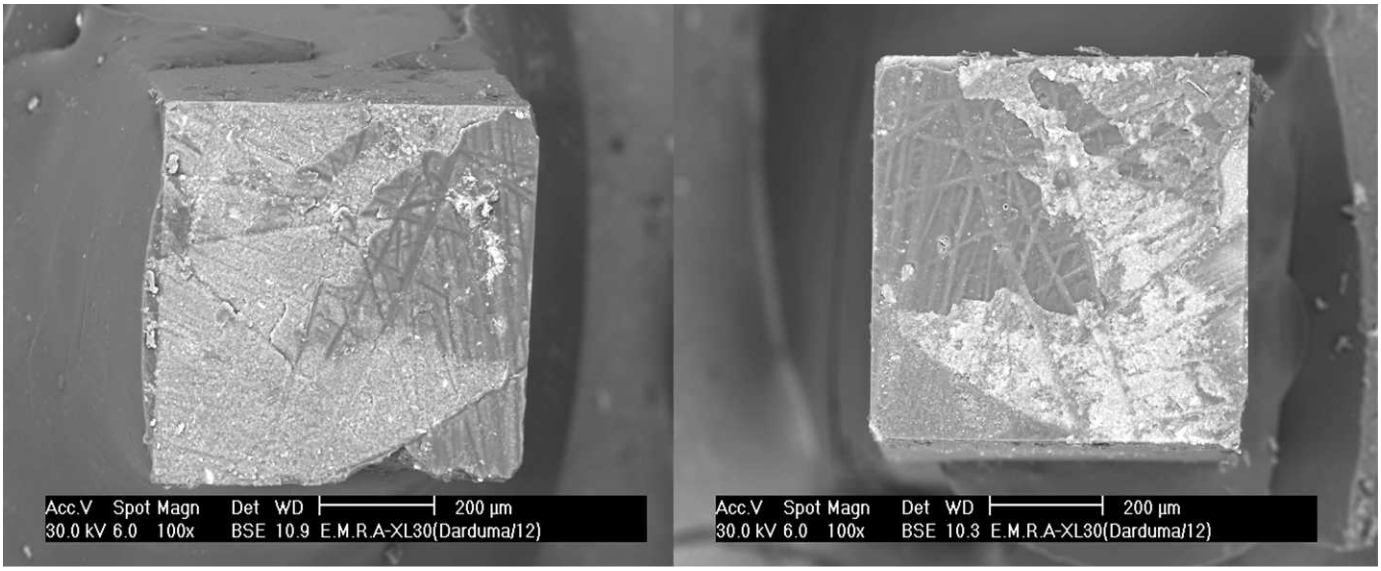

Figure 4. *SEM micrographs for representative stick after failure. Left: Mixed failure at the substrate surface of Filtek P90; Right: Mixed failure at intermediate adhesive agent and the repair side.*

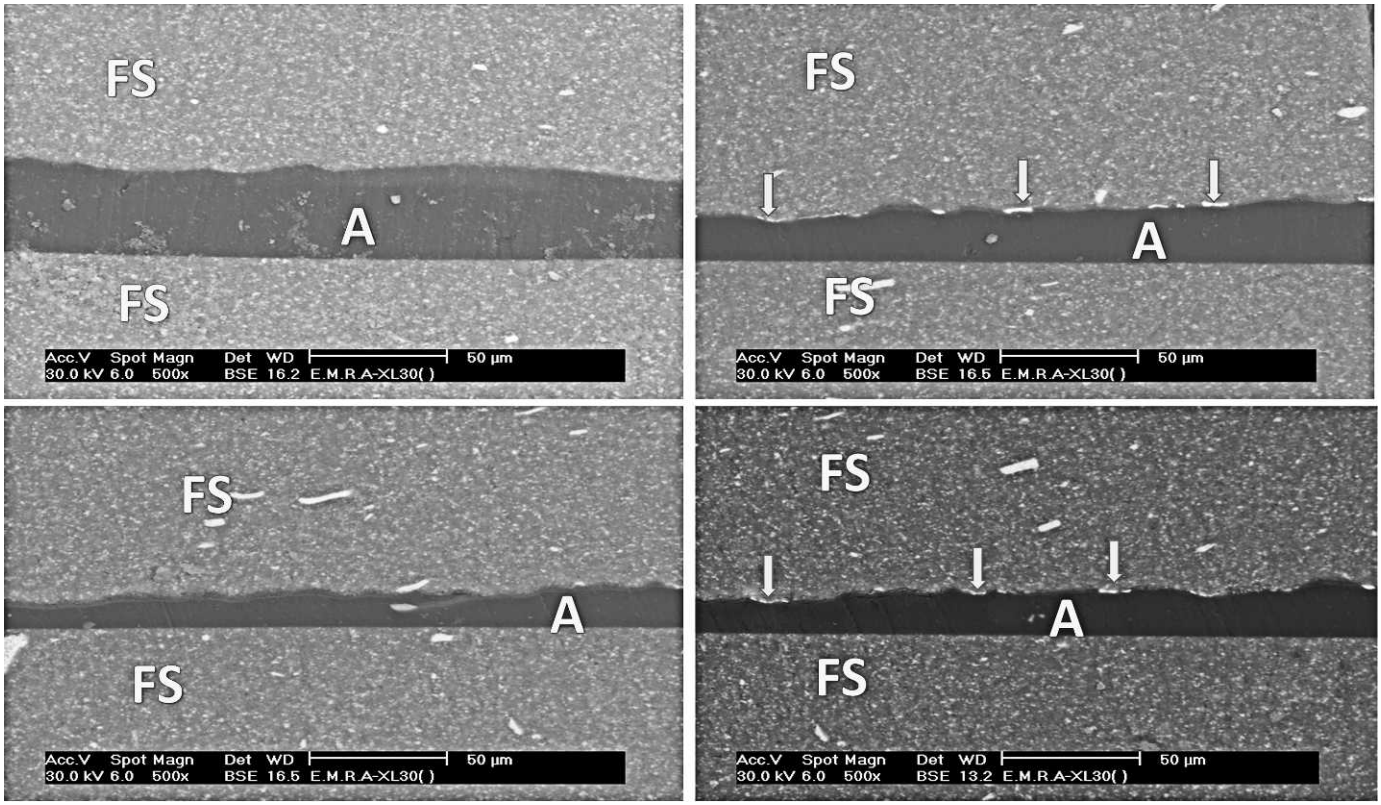

Figure 5. *Silver nitrate uptake of specimens repaired with P90 System Adhesive (SA)/Filtek P90 with/without silane primer as adhesion promoter after the 24-hour and two-year storage period. (a): Filtek P90 repaired with SA/Filtek P90 without silane primer and stored for 24 hours. (b): Filtek P90 repaired with SA/Filtek P90 without silane primer and stored for two years; arrows point to silver uptake. (c): Filtek P90 repaired with SA/Filtek P90 with silane primer and stored for 24 hours. (d): Filtek P90 repaired with SA/Filtek P90 with silane primer and stored for two years. FS, FiltekP90; A, intermediate adhesive layer.*

different intermediate adhesive agent/composite and that repaired with the same material. Based on these findings, the first null hypothesis was accepted. Previous studies proved that it is possible to repair a silorane-based composite with the same material[5,9,10] or even in combination with dimethacrylate-based adhesive/composite[9] if the appropriate repair technique is used. Quartz fillers in Filtek P90 are surface treated with an oxirane-functionalized silane. Therefore, there is an expected chemical affinity between the treated fillers and the P90 System Adhesive. However, in the present study, mechanical (with finishing or polishing) and chemical (after acid etching) treatments were applied to the surface of Filtek P90, which in turn removed the functional silane from the exposed fillers. This rendered them with no affinity to adhesives. Based on this, the micromechanical and/or the chemical coupling to the resin matrix is expected to be the cause of the obtained repair bond strength of Filtek P90 and its adhesive. For SBMP, there is no chemical affinity between its components and Filtek P90; thus, micromechanical retention may contribute to the repair mechanism.[9] Wiegand and others[10] reported that repair of aged silorane requires adequate mechanical surface treatment and application of the corresponding intermediate adhesive agent/composite. Researchers agreed that the bonding of new to old composite is mainly micromechanical,[3,9,28] although chemical bonding cannot be disregarded.[29]

There are no available data in the literature on the repair bond strength considered adequate enough to survive in occlusal function.[30] Therefore, many studies[20,30,31] have suggested that the cohesive strength of the intact unrepaired material should be taken into consideration as a control in the evaluation of the repair bond strength.[24] The results of this study clearly indicated that none of the repaired specimens was as strong as the mean cohesive strength of the control specimens. Nevertheless, the repair μTBS ranged from 66% to 77% of the cohesive strength of the intact unrepaired silorane-based specimens. This corresponds to previous findings, although the test materials and methodologies were different.[20,31,32]

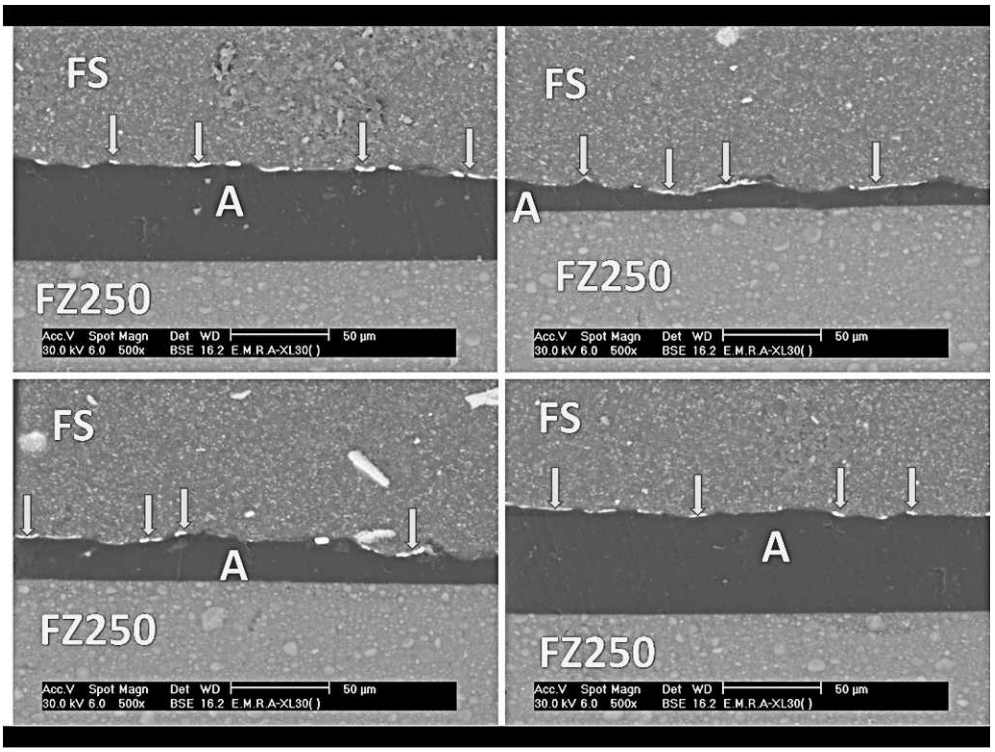

Figure 6. *Silver nitrate uptake of specimens repaired with Adper Scotchbond Multipurpose adhesive (SBMP)/Filtek Z250 with/without silane primer as adhesion promoter after the 24-hour and two-year storage period. (a): Filtek P90 repaired with SBMP/Filtek Z250 without silane primer and stored for 24 hours; arrows point to silver uptake. (b): Filtek P90 repaired with SBMP/Filtek Z250 without silane primer and stored for two years. (c): Filtek P90 repaired with SBMP/Filtek Z250 with silane primer and stored for 24 hours. (d): Filtek P90 repaired with SBMP/Filtek Z250 with silane primer and stored for two years. FS, FiltekP90; A, intermediate adhesive layer; FZ250, Filtek Z250 resin composite.*

Silorane repair μTBS of all stored repair categories were comparable and had no significant statistical difference. This result demonstrated that hydrophilicity of the intermediate adhesive agent had no effect on the silorane-based resin composite repair bond strength durability. This leads to the acceptance of the second hypothesis. There is no previously published work that investigates the long-term repair bond strength of silorane-based resin composite. Costa and others[33] found no significant difference when SBMP was used as the intermediate-adhesive agent to repair methacrylate-based resin composite. The repair bond strength did not change after 6 months of water storage.

Materials with equal bond strengths do not always fail in the same manner.[34] For all of the 24-hour stored repair categories, adhesive failure at the substrate side was the predominant mode of failure. After two-year storage, this percentage slightly decreased, while cohesive in the adhesive layer and mixed types of failure tended to increase. These results denote that the repair interface is still the weakest part, especially at the substrate side.[9] After two-year immersion in artificial saliva, different parts of the repair interface may become fatigued and degraded by time. One of the interesting findings that can be interpreted from the SEM pictures is that the surface topography of the repair side of the adhesively failed sticks was almost a positive duplicate to the negative depressions present on the roughened surface of the substrate side of the failed stick. This may emphasize the previously mentioned postulation in the Mobarak[9] study, in which resin-filled depressions go in the same line as the applied tensile forces. Hence, these depressions in the prepared surface may add to the surface area for adhesion rather than creating macromechanical retention.

The results of the present study suggest the partial acceptance of the third null hypothesis, whereby silane application did not influence the nanoleakage results. After 24 hours of storage, no silver nitrate deposition was detected when silorane-based resin composite was repaired with the same material. This finding could be explained by the relative hydrophobicity and the perfect coupling between the repaired biomaterial assembly. However, for the methacrylate-based (SBMP/Filtek Z250)

repair system, silver nitrate deposition was detected at 24 hours. Also, silver nitrate deposition increased relatively after two-year storage in all groups. Thus, nanoleakage evaluation supports the SEM observation of the mode of failure percentage. It seems that differences in the material polarity play a preponderant role in silver nitrate deposition.[33] No published study tested the nanoleakage of repaired silorane-based resin composite. However, Costa and others[33] reported that adhesive interfaces of repaired methacrylate-based resin composite absorb water after long-term water storage (six months). The amount of water sorption was reported to be positively correlated with the hydrophilicity of the adhesive system.[35,36] Absorbed water molecules occupy the free volume between polymer chains and crosslinks, causing swelling of the polymer structure and leading to plasticization and softening of the resin structure.[37] After this relaxation process, unreacted monomers trapped in the polymer network are released to their surroundings, resulting in a higher solubility.[36] This might create microvoids that were likely to be filled with silver nitrate. Although these findings did not result in any reduction in composite repair bond strength, it may represent signs of early degradation. Previous work[38] has shown that saliva enzymes could accelerate the adhesive degradation. Further research is required to elucidate the effect of these enzymes on repair bond durability.

The results of the study suggested that a mean bond strength value could not be taken as a sole indicator of the bond quality and that an interfacial morphological evaluation may provide additional information about interfacial bonding quality.[9] However, it has to be taken into account that nanoleakage is not necessarily associated with an impaired clinical performance. Thus, despite the importance of laboratory studies attempting to predict the performance of biomaterials, clinical trials remain the ultimate way to collect scientific evidence on the clinical effectiveness of a restorative treatment.

CONCLUSIONS

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

1. Silane application as a separate step in the repair process of the silorane-based resin composite has no effect on the short- or long-term bond durability and nanoleakage.
2. Repair bond strength of the silorane-based resin composite appeared successfully durable irrespective of the chemistry of the intermediate repair adhesive agent/composite material.
3. Usage of different adhesive/repair materials in repairing silorane-based composite tends to show early nanoleakage.

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Conflict of Interest

The authors of this article 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 Dentinal Tubule Occlusion by Desensitizing Agents: A Real-time Measurement of Dentinal Fluid Flow Rate and Scanning Electron Microscopy

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Clinical Relevance

Light-curing adhesive and oxalate-type desensitizing agents exhibited better reduction of dentinal fluid flow rate than did protein-precipitation and fluoride-type desensitizing agents based on measurements by a new fluid flow measuring device of subnanoliter scale.

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SUMMARY

The aims of this study were to examine changes in dentinal fluid flow (DFF) during the application of a desensitizing agent and to compare the permeability reduction levels among different types of desensitizing agents.

A cervical cavity was prepared for the exposure of cervical dentin on an extracted human premolar connected to a subnanoliter fluid flow measuring device under 20 cm of water pressure. The cavity was acid-etched with 32% phosphoric acid to make dentin highly permeable. The different types of desensitizing agents that were applied on the cavity were Seal&Protect as the light-curing adhesive type, SuperSeal and BisBlock as oxalate types,

Gluma Desensitizer as the protein-precipitation type, and Bi-Fluoride 12 as the fluoride type. DFF was measured from the time before the application of the desensitizing agent throughout the application procedure to five minutes after the application. The characteristics of dentinal tubule occlusion of each desensitizing agent were examined by scanning electron microscopy.

The DFF rate after each desensitizing agent application was significantly reduced when compared to the initial DFF rate before application for all of the desensitizing agents ($p<0.05$). Seal&Protect showed a greater reduction in the DFF rate when compared to Gluma Desensitizer and Bi-Fluoride 12 ($p<0.05$). SuperSeal and BisBlock exhibited a greater reduction in DFF rate when compared to Bi-Fluoride 12 ($p<0.05$).

The dentin hypersensitivity treatment effects of the employed desensitizing agents in this study were confirmed through real-time measurements of DFF changes. The light-curing adhesive and oxalate types showed greater reduction in the DFF rate than did the protein-precipitation and fluoride types.

INTRODUCTION

Among several theories that explain dentin hypersensitivity (DH), the hydrodynamic theory has been the most widely accepted. This theory proposes that a stimulus applied to an affected tooth causes the movement of dentinal tubular fluid in either an outward or inward direction. This movement stimulates the mechano-receptors of the sensory nerves in the dentin or pulp.[1] Based on the hydrodynamic theory, hypersensitive dentin exhibits open dentinal tubules and high permeability so that, in theory, if dentinal tubules are partially or completely occluded, DH symptoms decrease or vanish as a result.[2] Patients who have DH usually exhibit dentin exposure caused by microleakage of a restoration, cervical abrasion, or cementum loss. Hypersensitive dentin has wider and much more permeable dentinal tubules when compared to nonhypersensitive dentin.[3]

Cervical resin composites or glass ionomer restorations are typically performed if a tooth has moderate to severe cervical tooth loss. However, in cases of slight cervical abrasion or root exposure with DH, the application of a desensitizing agent to relieve symptoms is usually preferred over restoration treatment. Although the mechanism of desensitizing agent for hypersensitive dentin has not been clearly revealed, most of the currently employed desensitizing agents in clinics are intended to seal the dentin surface or to occlude dentinal tubules by protein precipitation or calcium complex formation so that the movement of dentinal tubular fluid can be suppressed.[4] A number of studies have reported the effects of the application of desensitizing agent on dentinal tubule occlusion. However, there exist no consistent conclusions regarding which product or which mechanism is superior.[5]

Evaluations of the effects of desensitizing agent on dentinal tubule occlusion are generally performed by the observation of the occlusion of the dentinal tubules using a scanning electron microscope (SEM) or by the observation of changes in permeability by measuring the hydraulic conductance of a dentin disc.[5] However, to date no report has investigated changes in the permeability of dentinal tubules in real time throughout the desensitizing agent application process for permeable dentin.

Recently, a study[6] reported the measurement of the dentinal fluid flow (DFF) in real time during a restorative procedure on an extracted tooth using a newly fabricated subnanoliter-scaled fluid flow measuring device (NFMD), which was capable of discriminating a volume change of 0.2 nL. In the current study, the DFF was measured in real time during the desensitizing agent application process for a cervical cavity. The immediate effects of dentinal tubule occlusion were compared with the different types of desensitizing agents. The dentin surfaces and subsurfaces onto which the desensitizing agents were applied were also examined by SEM to confirm the different aspects of dentinal tubule occlusion. The null hypothesis was that there would be no difference in the DFF rates before and after desensitizing agent application and that there would be no differences in the permeability reduction among the agents, despite the fact that the agents employed different mechanisms to occlude the dentinal tubules.

MATERIALS AND METHODS

Structure of the NFMD

The NFMD used in this study consisted of three parts: a glass capillary and photosensor to detect the fluid movement; a servomotor, lead screw, and ball nut to track the fluid movement; and a rotary encoder and computer software to record the data. The minimum measurable volume of water move-

ment was 0.196 nL. Details of the working mechanism of the NFMD were described in a previous study.[6]

Specimen Preparation

Upper and lower premolars that were extracted for orthodontic reasons were used in this study. The project was approved by the Institutional Review Board of the Seoul National University Dental Hospital (CRI 09005). Extracted teeth were stored in a 1% chloramine-T solution at 4°C and were used within three months following their extraction.

Each root was removed 5 mm below the cemento-enamel junction using a low-speed diamond saw (Isomet, Buehler, IL, USA). The pulp tissue in the pulp chamber was carefully removed without altering the pre-dentin surface using thin tissue forceps and endodontic files. A sandblasted Plexiglas square (10 mm per side and 2 mm thick) with a hole drilled at its center was used to mount each tooth. A metal tube with a diameter of 0.9 mm was inserted into the hole, and the Plexiglas was attached to the tooth using an adhesive (Adper Scotchbond MultiPurpose, 3M ESPE, St Paul, MN, USA) and a flowable composite (Denflow, Vericom, Anyang, Korea) to ensure that one end of the metal tube was located in the pulp chamber. The exposed root surface and outer surface of the bonded interface between the Plexiglas and the tooth on the top surface and between the Plexiglas and the metal tube on the bottom surface were covered with nail varnish.

The prepared specimen was stored in distilled water and was connected to a water reservoir containing distilled water. A hydrostatic pressure of 20 cm H_2O was applied to the specimen 24 hours before the experiment was conducted.[7]

Cervical Cavity Preparation and the Measurement of the DFF During Desensitizing Agent Application

The prepared specimen was connected to a glass capillary by silicone tubing filled with distilled water (Figure 1). A hydrostatic pressure of 20 cm H_2O was applied throughout all of the procedures with a water reservoir to simulate physiological pulp pressure. The temperature and relative humidity of the environment were 24°C ± 0.5°C and 30% ± 5%, respectively. Each specimen underwent a stabilizing time of 10 minutes after it was connected to the NFMD. After confirming that the fluctuation level of the DFF was within ±5 nL for another 10 minutes, the cavity preparation was conducted.

A V-shaped cervical cavity with a mesio-distal width of 5 mm, an occluso-cervical height of 3 mm, and a depth of 2 mm was prepared with a round-end tapered diamond bur of 106-125-μm grit size (Mani, Tochigi, Japan) using an air-driven, high-speed handpiece (MACH-QD, NSK, Tokyo, Japan) at 200,000–300,000 rpm under water coolant. Acid-etching for 15 seconds using 32% phosphoric acid was performed to remove the smear layer formed during cavity preparation and to make dentin highly permeable. The cavity was then rinsed with water and blot-dried with a wet cotton pellet. Different types of desensitizing agents were applied in the prepared cavity according to the manufacturer's instructions (Table 1).

DFF measurement was performed continuously from 60 to 100 seconds after blot-drying the cavity, throughout the desensitizing agent application, and five minutes after desensitizing agent application in real time.

The average DFF rate, as measured before the desensitizing agent application, was set as the baseline flow rate. This baseline flow rate referred to the permeability of each tooth specimen and served as an internal reference for comparison with the subsequent flow rate measurements after desensitizing agent application. To determine the DFF rate after desensitizing agent application, the average flow rate was calculated for five minutes after applying the desensitizing agent. Reductions in the flow rate were indicated as a percentage of the decreased flow rate after desensitizing agent application with respect to the baseline flow rate [% reduction in flow rate $= 100 \times (\text{flow rate}_{\text{baseline}} - \text{flow rate}_{\text{postapplication}})/\text{flow rate}_{\text{baseline}}$].

The number of specimens required for each desensitizing agent was six, which was determined by a power analysis (78% power, 0.05 type 1 error level). A paired *t*-test was conducted to analyze whether there were any differences in the flow rate before and after desensitizing agent application. One-way analysis of variance was conducted to analyze whether there were differences in the reduction of the flow rate among desensitizing agents. A multiple comparison test was conducted using the Duncan test. The level of significance was $\alpha = 0.05$.

SEM Analysis

Premolar teeth extracted for orthodontic reasons were prepared in the same manner as described above to simulate physiologic pulp pressure. One

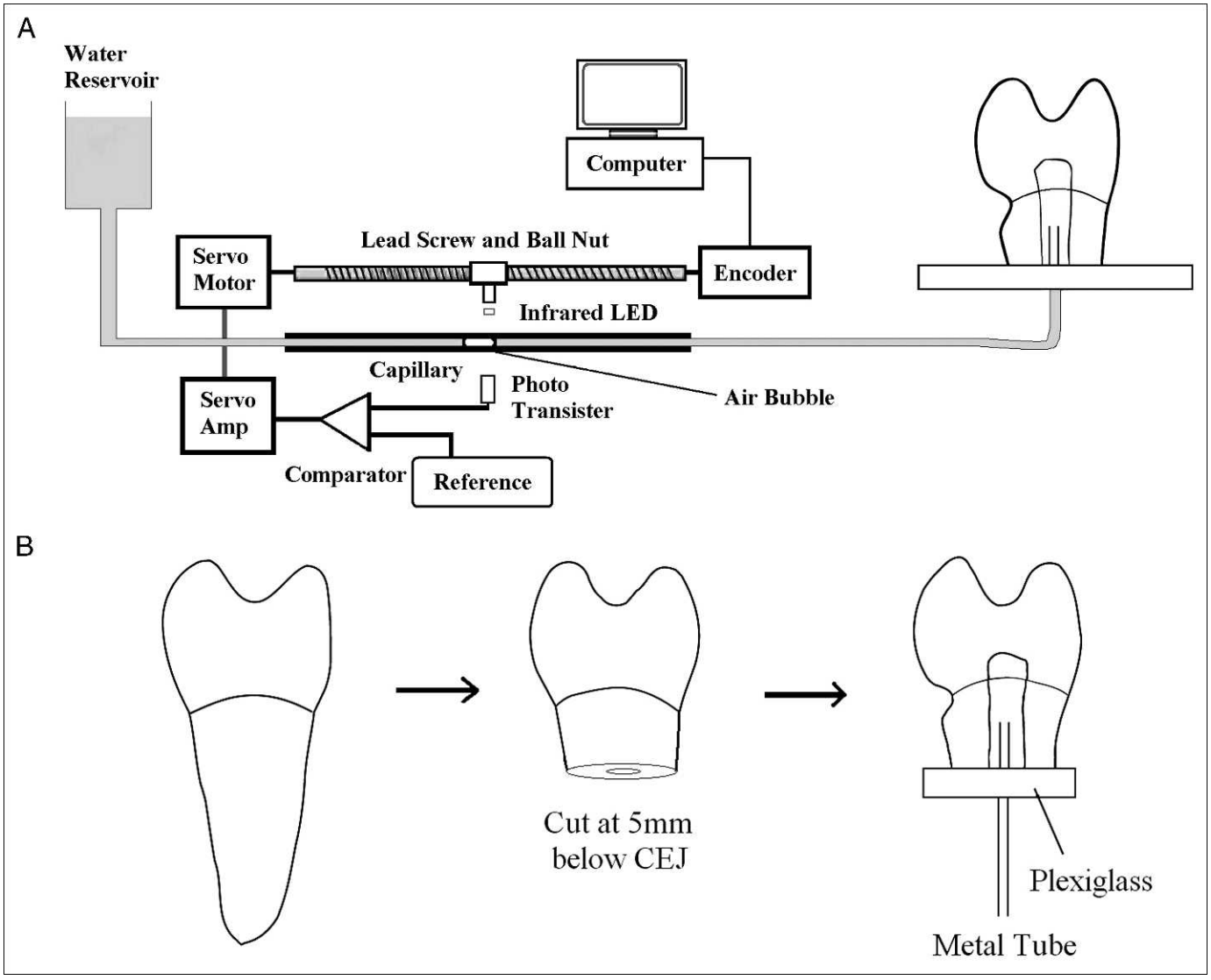

Figure 1. *(A) Schematic diagram of the subnanoliter-scaled dentinal tubular fluid flow measurement device (NFMD). (B) Specimen preparation.*

premolar tooth was representatively prepared for every individual desensitizing agent. After horizontal crown reduction was performed to expose the dentin surface, the specimen was connected to a water reservoir that contained distilled water at a hydrostatic pressure of 20 cm H_2O 24 hours before the experiment.

Acid-etching for 15 seconds using 32% phosphoric acid was performed to remove the smear layer that had formed during the preparation and to expose the highly permeable dentinal tubules. The dentin surface was then rinsed with water and blot-dried with a wet cotton pellet. Desensitizing agent was applied to half of the dentin surface, while the other half of the dentin surface was left exposed to observe the differences in both the areas *via* SEM. After the specimen was dried, a dentin disk was obtained by cutting 1–2 mm below the dentin surface using a high-speed handpiece. The dentin disk was fractured perpendicular to the border between the surface on which the desensitizing agent was applied and the surface on which the desensitizing agent was not applied. The specimens were then dried and sputter-coated with gold. Each specimen was subsequently examined using SEM (S-2300, Hitachi, Tokyo, Japan).

RESULTS

The specific behaviors of DFF during each desensitizing agent application are shown in Figure 2. The average DFF rate before desensitizing agent application indicates the baseline flow rate, which was

Table 1: *The Components and Application Procedures of the Desensitizing Agents Used in This Study*

Desensitizing Agent	Components	Procedure	Manufacturer
Seal&Protect (Lot No. 0909002823)	Di-and trimethacrylate resin, PENTA, Ffunctionalized amorphous silica, photoinitiators, butylated hydroxytoluene, cetylamine hydrofluoride, triclosan, acetone	Apply (dwell for 20 s), gentle air, light-cure[a] (10 s), reapply, gentle air, light-cure (10 s)	Dentsply, Milford, DE, USA
SuperSeal (Lot No. 991583)	Oxalate, potassium salt	Apply 30 s, gentle air-dry	Phoenix Dental, Fenton, MI, USA
BisBlock (Lot No. 0900000453)	Ferric oxalate	E&R[b], apply (dwell for 30 s), rinse	Bisco, Schaumburg, IL, USA
Gluma Desensitizer (Lot No. 010082)	Glutaraldehyde, HEMA, purified water	Apply (dwell for 60 s), air-dry, rinse	Heraeus, Hanau, Germany
Bi-Fluoride 12 (Lot No. 0941489)	Sodium and calcium fluoride	Apply (dwell for 20 s), air-dry	Voco, Cuxhaven, Germany

Abbreviations: E&R, acid-etching and rinse; HEMA, hydroxyethyl methacrylate; PENTA, dipentaerythritol penta acrylate mono monophosphate.
[a] LED light-curing unit (Elipar FreeLight 2, 3M ESPE, St Paul, MN, USA) of 600 mW/cm^2 intensity was used.
[b] E&R were omitted from this study because it had already been performed during specimen preparation.

used as an internal reference to compare the changes in the flow rate throughout the desensitizing agent application process. The postapplication flow rate decreased significantly compared to the baseline flow rate with all of the desensitizing agents used in this study ($p<0.05$). The application procedure for each desensitizing agent was reflected by the specific curve of the DFF. Regarding the SuperSeal and Bi-Fluoride 12, which require a simple "application-and-dry" procedure, even though there was a single fluctuation due to a decrease in the flow rate through the application itself and an abrupt increase in the flow rate through drying, a gradual decrease in the flow rate was observed on average. For Gluma Desensitizer and BisBlock, which have similar application procedures to SuperSeal and Bi-Fluoride 12 except for the water rinsing step, the water rinsing step led to a transient negative flow rate. For Seal&Protect, which involved two "application-and-light-curing" steps, two negative slopes by light-curing were reflected in the DFF characteristics during the application.

Figure 3 shows the mean reductions in flow rate as a percentage after individual application of desensitizing agents, when compared to the baseline flow rate. Seal&Protect showed a greater reduction in flow rate when compared to either Gluma Desensitizer or Bi-Fluoride 12 ($p<0.05$). SuperSeal and BisBlock showed a greater reduction in the flow rate when compared to Bi-Fluoride 12 ($p<0.05$).

Typical SEM images of the surface and the subsurface on which each desensitizing agent was applied are shown in Figure 4. For Seal&Protect, a thick resinous layer covering the treated area was observed when compared to the nontreated area. Resin plugs of approximately 5-10 μm that had formed in the dentinal tubules were also observed in the subsurface view. In the SuperSeal-treated area, many tiny crystals were filled in the dentinal tubules, and the depth of crystal penetration was at a maximum of approximately 20 μm. For BisBlock, larger and rounder crystals were present in the dentinal tubules when compared to SuperSeal, and the depth of crystal penetration was at a maximum of approximately 40 μm. On the Gluma Desensitizer–treated area, amorphous particles of the precipitation were observed on the surface, but not as frequently in the dentinal tubules. It was difficult to determine the precipitation characteristics in the dentinal tubules in a subsurface view. On the Bi-Fluoride 12–treated area, although many resinous plugs in the dentinal tubules were observed, they appeared porous and did not appear to fill the dentinal tubules completely.

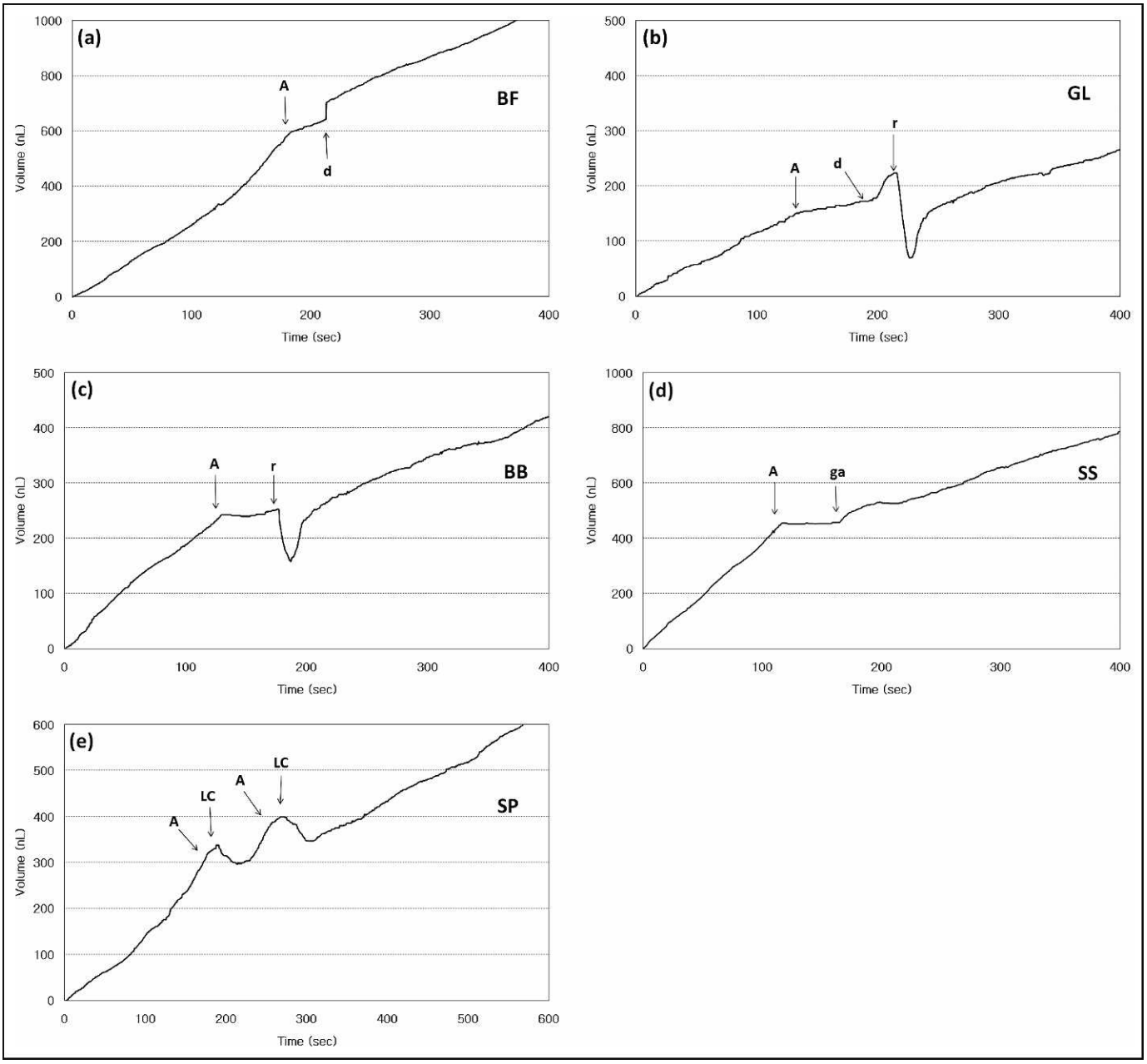

Figure 2. *A representative graph of consecutive dentinal fluid flow (DFF) during the application of each desensitizing agent. (a-e) The representative DFF graphs of Bi-Fluoride 12, Gluma Desensitizer, BisBlock, SuperSeal, and Seal&Protect, respectively. Upward (positive slope) movement vs the time on the graph indicates outward DFF, whereas downward (negative slope) movement indicates inward DFF. A, Application of a desensitizing agent; d, air dry; r, rinse; ga, gentle air-dry; LC, light-curing.*

DISCUSSION

There are two methods of treating DH: the first is to reduce the sensory nerve activity by increasing the K^+ ion concentration, and the second method involves reducing the dentinal tubular fluid flow by occluding the dentinal tubule.[2] Ever since the wide acceptance of Brannstrom's hydrodynamic theory, desensitizing agents that effectively occlude the dentinal tubule have been typically used in clinics. Numerous laboratory studies[5,8] have investigated the effects of desensitizing agent on dentinal tubule occlusion by comparing the permeability difference before and after the application of desensitizing agent on a dentin disk connected to a capillary in a split chamber. Although this method has been used because of the convenience of specimen preparation

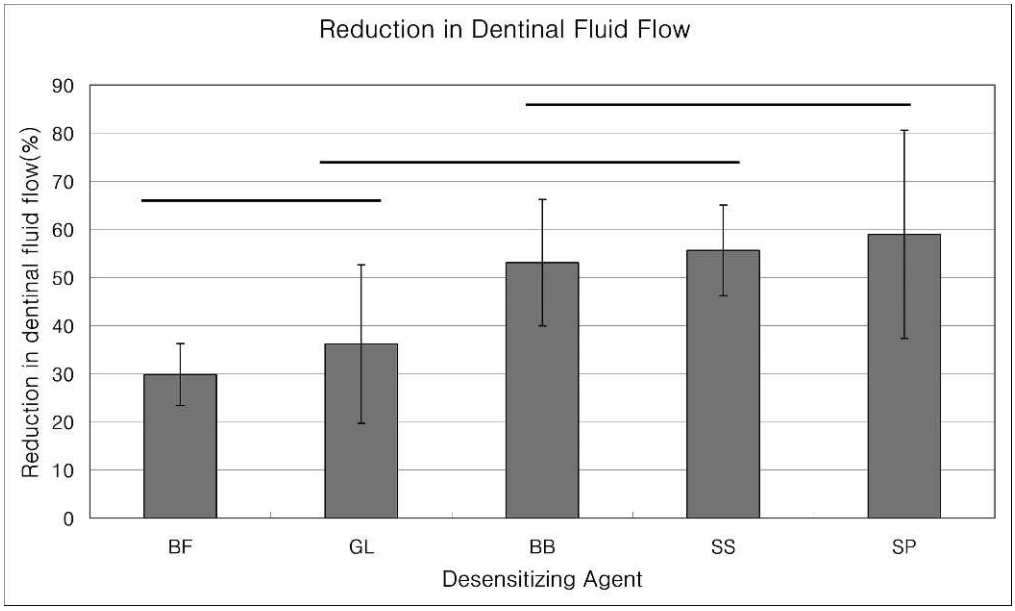

Figure 3. *Reduction in dentinal fluid flow by desensitizing agents (%). Desensitizing agents under the same bar did not show statistically significant differences (n=6).*

and desensitizing agent application, most of the studies loaded a much higher pressure than the physiologic pulp pressure to accelerate the movement of the air bubble in the capillary of the flow measurement device. The designs of previous studies were very different from clinical scenarios in terms of the location and shape of the exposed dentin. However, when compared to the preexisting and other reported studies, the design employed in the present study is very much similar to the clinical situation, because rather than employing a dentin disk, an original tooth form was used, a physiologic pulp pressure of 20 cm H_2O was applied, and the cervical dentin, which is the area that most frequently undergoes DH, was exposed. Moreover, this study is the first of its type to measure DFF change during desensitizing agent application in real time.

The change in DFF caused by each desensitizing agent during the application was reflected as a specific curve on the graph of the NFMD (Figure 2). For example, for SuperSeal and Bi-Fluoride 12, the simple treatment procedure of "application-and-dry" can be seen as a "step-like" figure on the graph. The desensitizing agent application itself caused a decrease in the DFF rate, and air-drying caused an abrupt increase in the DFF rate, even when it was performed gently. After the application period, the DFF graph eventually showed a decreased flow rate when compared to the baseline flow rate. Gluma Desensitizer and BisBlock reflect the water rinsing step as a transient negative flow rate on the DFF graph. In the case of Seal&Protect, which involved two light-curing steps, the DFF rate changed negatively when light-curing was performed. This indicates that dentinal tubular fluid was forced into the pulp as a result of the thermal expansion caused by the heat from the light-curing unit.[6] There was an abrupt increase in the DFF rate due to rebounding effect at the end of the light-curing process. Shortly after this rebounding increase in the flow rate, Seal&Protect returned to a consistent flow rate that was lower than the baseline flow rate.

All of the desensitizing agents used in this study resulted in significant reductions in flow rate following application when compared to the baseline, although all of the agents did not stop the DFF completely ($p<0.05$). Generally, the flow rate under the same pressure depends mainly on the radius of the dentinal tubules. Therefore, a reduction in the flow rate reflects the effect of dentinal tubule occlusion. As such, the desensitizing agents used in this study were expected to demonstrate the effect of treating DH to a certain degree based on the results that all the desensitizing agents occluded dentinal tubules.

However, significant differences were found among the desensitizing agents used in this study in terms of permeability reduction. Seal&Protect, SuperSeal, and BisBlock showed a greater dentinal tubule occlusion effect when compared to Bi-Fluoride

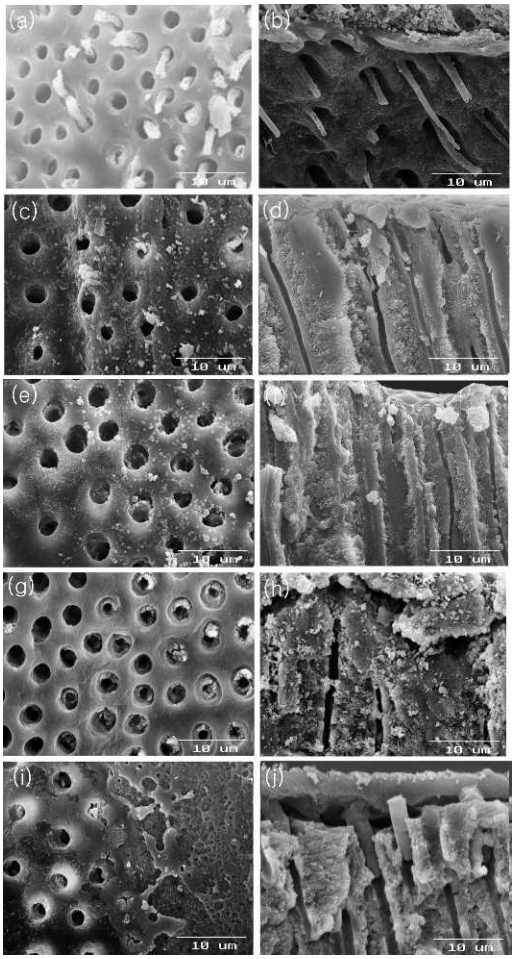

Figure 4. *Representative scanning electron microscopy (SEM) images of occlusal surface (left panels) and fractured subsurface (right panels) following application of different desensitizing agents, as follows: (a, b) Bi-Fluoride 12, (c, d) Gluma Desensitizer, (e, f) BisBlock, (g, h) SuperSeal, and (i, j) Seal&Protect. The occlusal surface images of desensitizing agents (a), (c), (e), (g), and (i) contain both the desensitizing agent-applied surface in the right side and an unapplied surface in the left side. The left side of each occlusal surface image showing wide-open dentinal tubules is an area in which desensitizing agent was not applied.*

12 and Gluma Desensitizer. Seal&Protect consists of methacrylate resins, photoinitiators, fillers, and dipentaerythritol penta acrylate monophosphate (PENTA), which is a partially acidic monomer. Therefore, the working mechanism of Seal&Protect is likely to occlude the entrance of the dentinal tubules by forming a hybrid layer in a manner similar to that of the all-in-one adhesive. The resin plug in the SEM images of Seal&Protect also indicates that dentinal tubule occlusion by Seal& Protect is similar to that of all-in-one adhesive. If an exposed dentin surface is given only one layer of Seal&Protect, it would be difficult to effectively occlude the dentinal tubules through which fluid flows out, considering that the thin all-in-one adhesive works as a permeable membrane, even after polymerization.[9] The second layer of Seal& Protect is considered to strengthen the sealing effect of the first layer. This mechanism may explain why the manufacturer suggests applying two layers of Seal&Protect. Seal&Protect showed good dentinal tubule occlusion, illustrating its feasibility for DH treatment in previous laboratory and clinical studies.[10,11]

SuperSeal may have two mechanisms in the treatment of DH. First, potassium ions inhibit pulp sensory nerve excitation, and second, oxalate binds with the calcium ions of the dentin surface or the dentinal tubules to form calcium oxalate crystals, which occlude the dentinal tubules. The manufacturer of SuperSeal claims that the formation of calcium oxalate crystal occurs within two minutes, which is proven to some extent by the reduction in the flow rate as measured during the application in this study. Potassium oxalate has long been used because it was known from previous studies[12,13] to have a great effect on dentinal tubule occlusion and is considered as a viable treatment for DH. Considering that the SuperSeal exhibited good dentinal tubule occlusion in this study, consistent with the results of previous studies, a high level of DH treating effect would be expected with SuperSeal, especially if it is used in conjunction with the inhibition mechanism of potassium ions on nerve excitation.

The clinical procedure for BisBlock, as suggested by the manufacturer, is to acid-etch, oxalate-apply, rinse, apply prime-and-adhesive, and light-cure. However, in this study, the adhesive application and light-curing process steps were not performed in order to examine the dentinal tubule occlusion effect of ferric oxalate itself and to exclude the adhesive effect. BisBlock had a substantial effect on the reduction in the flow rate and in causing a decrease in the dentinal tubule diameter through oxalate action without dentin sealing by a cured adhesive. Larger and rounder crystals were observed in the SEM images when compared to SuperSeal. Further studies need to be carried out to examine how the dentin tubule was occluded through double action by both the oxalate and adhesive and to determine whether it would last for a longer period of time when the adhesive application and light-curing were additionally performed according to the manufacturer's suggestion.

The Bi-Fluoride 12 of the fluoride agent exhibited the lowest reduction in flow rate in this study.

Fluoride is known to occlude the dentinal tubule by forming CaF_2 crystals.[14] A number of studies[2,12,15] reported that the dentinal tubule occlusion effect of fluoride is lower than that of oxalate agents and that the crystal structure cannot be observed in SEM images of fluoride-applied dentin specimens. Other clinical studies,[16,17] on the other hand, reported good DH treatment effects of using fluoride. Fluoride appears to be a subject of some debate with regard to the mechanism and effect of DH treatment. In this study, the Bi-Fluoride 12 specimen did not show a crystal structure but instead showed a resinous plug in the dentinal tubules.

Determination of the ingredients of a resinous plug could not be done solely on the basis of the manufacturer's disclosure of the chemical compositions. The crystal structure could not be detected, possibly because fluoride does not easily form crystals with calcium ions and/or because CaF_2 can be easily dissolved as a result of its chemical instability in a moisture-rich environment.

Gluma Desensitizer has the longest history of use as a desensitizing agent in clinical settings. The desensitizing mechanism of Gluma Desensitizer is based on the reaction of glutaraldehyde with a protein in the dentinal tubules, which in turn causes precipitation, which decreases the diameter of the dentinal tubule. Subsequently, this precipitation promotes hydroxyethyl methacrylate (HEMA) polymerization, which also causes dentinal tubule occlusion.[18,19] In contrast to previous studies[20-22] that have reported good DH treatment effects of Gluma Desensitizer, the present study showed a lower reduction in the permeability when compared to the other employed desensitizing agents. In the SEM images, decrease in the dentinal tubule diameter was not clearly observed. Moreover, only small dispersed particles that appeared to be polymerized HEMA particles were observed on the dentin surface. The lack of good dentinal tubule occlusion effects of Gluma Desensitizer, as observed in this study, was likely due to the use of distilled water as a dentinal tubular fluid (distilled water does not contain proteins). Such an application of distilled water would greatly limit the role of glutaraldehyde. In fact, previous studies[10,23] in which serum albumin was used as dentinal tubular fluid showed a highly effective reduction in permeability with Gluma Desensitizer.

A physiologic solution containing protein could not be used in this study because protein can cause sedimentation in the capillary tube. This change in the tubular diameter can reduce the consistency of the NFMD. Future studies are demanded to establish an elaborate method that will enable the NFMD to consistently measure fluid flow using physiologic fluid.

Occlusal dentin was employed to observe the occluding characterization of each desensitizing agent in SEM analysis because occlusal dentin is easier to use to standardize the specimen preparation than is cervical dentin. However, occlusal dentin and cervical dentin have different aspects with respect to direction and amount of occlusal force and may have different direction of dentinal tubules. Further study may be needed to investigate if the occluding effect of each desensitizing agent would have different aspects for the cervical dentin and occlusal dentin.

We investigated immediate dentinal tubule occlusion effects by measuring the DFF from the preapplication of desensitizing agent to postapplication in real time. In fact, it may be difficult for desensitizing agent to be retained on an exposed dentin surface because of interactions with saliva and cyclic brushing. Desensitizing agent containing a resin component may also not be retained on the dentin surface as a result of differences in thermal expansion from the dentin according to thermal changes in the oral cavity. It would be interesting to investigate how long desensitizing agent can maintain the dentinal tubule occlusion effect after application through measurements of DFF under different simulated oral cavity conditions.

CONCLUSION

Within the results of this study, based on measurements by a new fluid flow measuring device of subnanoliter scale, the following conclusions could be drawn. All of the desensitizing agents employed in this study led to a significant reduction in DFF rate following application of each desensitizing agent. Light-curing adhesive and oxalate-type desensitizing agents exhibited better reduction in dentinal fluid flow rate than did protein-precipitation and fluoride-type desensitizing agents.

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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Evaluating Dentin Surface Treatments for Resin-Modified Glass Ionomer Restorative Materials

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Clinical Relevance

A dentin bonding agent provided greater bond strength to dentin for RMGI than the manufacturers' recommended products.

SUMMARY

This *in vitro* study evaluated the effect of six surface treatments on the shear bond strength of three resin-modified glass ionomers (RMGIs) to dentin. Occlusal surfaces of caries-free third molars were reduced to expose only dentin. Surface treatments were smear layer intact (negative control), Cavity Conditioner, EDTA, Ketac Primer, Self Conditioner, and etching with 35% phosphoric acid followed by the application of Optibond Solo Plus. Filtek Z250 composite resin bonded with Optibond Solo Plus served as a positive control. Conditioning agents were used according to the manufacturers' instructions. After surface treatments, Fuji II LC, Riva LC, Ketac Nano, and Filtek Z250 were placed in copper-band matrices 5 mm in diameter and 2 mm in height and were light-cured for 20 seconds. Specimens were stored in 100% humidity for 24 hours, after which they were placed in deionized water for 24 hours at 37°C. They were then tested under shear forces in an Instron Universal Testing Machine at a crosshead speed of 0.5 mm/min. A two-way analysis of variance and Tukey honestly significant difference statistical analyses ($p<0.05$) indicated significant interaction between RMGIs and conditioning agents. Acid etching followed by Optibond Solo Plus provided highest bond strengths for all three RMGIs, which were not statistically different from the positive control.

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INTRODUCTION

Resin-modified glass ionomers (RMGIs) are the result of grafting water-soluble monomers such as 2-hydroxyethyl methacrylate (HEMA) to polyalkenoic acid chains of conventional glass ionomers (CGIs) and incorporating resins and photoinitators.[1,2] The fluoride release of RMGIs is comparable and often better than that of CGIs.[2-5] Advantages of RMGIs compared with CGIs include increased working time, decreased setting time, ease of handling, and improved physical properties and esthetics.[1-4] Resin-modified glass ionomers have been marketed as liners, bases, dentin bonding agents, sealants, luting agents, and core foundations, as well as direct restorative materials, particularly for class V lesions.[1-3]

There are two distinct bonding mechanisms for RMGIs to tooth structure: a chemical bonding between anions of polyalkenoic acid chains and calcium ions in hydroxyapatite[1-4] and a micromechanical bond similar to that which occurs between dentin bonding agents and dentin.[6-10] Several agents have been evaluated to condition dentin prior to application of CGIs and RMGIs. These have included citric, polyacrylic, phosphoric, tannic, and ethylenediaminetetraacetic (EDTA) acids.[11-13] Polyacrylic acid has been the mainstay for conditioning dentin prior to application of CGIs and RMGIs. Recently, manufacturers have recommended other conditioners to replace polyacrylic acid; they include Ketac Primer (3M ESPE, St Paul MN, USA) and Self Conditioner (GC, Tokyo, Japan).

Resin-modified glass ionomers are packaged in capsules as a powder and liquid or as two separate pastes. Examples of powder/liquid RMGIs include Fuji II LC (GC) and Riva LC (Southern Dental Industries, Victoria, Australia), which both require trituration. Ketac Nano (3M ESPE) contains two separate pastes that are mixed together when dispensed through an auto-mixing tip. In lieu of using polyacrylic acid to condition dentin, the manufacturer of Ketac Nano recommends conditioning with Ketac Primer. When using Fuji II LC, the manufacturer recommends dentin conditioning with either Cavity Conditioner (GC) or Self Conditioner. Cavity Conditioner contains 20% polyacrylic acid and 3% aluminum chloride. Self Conditioner contains HEMA and 4-methacryloxyethyl trimellitate anhydride (4-META) and is purported by the manufacturer to provide stronger and more stable bonding. In addition to containing hydrophilic resins, both Ketac Primer and Self Conditioner are acidic, with a pH of 3.0 and 1.8, respectively. Southern Dental Industries, manufacturer of Riva LC, recommends using either polyacrylic or phosphoric acid for dentin conditioning. Because RMGIs contain resin, it has been hypothesized that the bond strength of RMGIs can be improved by the application of dentin bonding agents. The purpose of this *in vitro* study was to compare the effect of five different conditioning protocols (polyacrylic acid, EDTA, Ketac Primer, Self Conditioner, and a dentin bonding agent) on the shear bond strength of three different RMGIs to dentin. In addition, a negative control was included that consisted of leaving the smear layer intact. A group consisting of Filtek Z250 composite resin (3M ESPE) bonded to dentin with Optibond Solo Plus (Kerr, Orange, CA, USA) after phosphoric acid etching was used as a positive control. The null hypothesis we tested was that there would be no difference in bond strengths as a result of surface treatments or RMGIs.

METHODS AND MATERIALS

Dental materials and their compositions are listed in Table 1. Recently extracted, caries-free third molars were cleaned of debris and disinfected in a 0.5% solution of sodium hypochlorite and sterile water for 30 minutes. Teeth were embedded in phenolic rings (Buehler Ltd, Lake Bluff, IL, USA) 2 mm apical to their cementoenamel junctions using Orthodontic Resin (LD Caulk/Dentsply, Milford, DE, USA). The occlusal surface of each tooth was carefully reduced using a conventional model trimmer (Handler Mfg Co, Garwood, NJ, USA) with water to produce a dentin surface. The surface was not treated with any other abrasive papers or discs. Conditioners, primers, and bonding agent were applied by one investigator according to the manufacturers' instructions as described in Table 2. Copper-band matrices 5 mm in diameter and 2 mm in height were held on the dentin surface by a second investigator by grasping the copper band with a cotton forceps and holding the band steady against the flat dentin surface. The first investigator activated, triturated, or mixed the RMGI according to the manufacturer's instructions. The first investigator placed the RMGI or Z250 resin into the copper band, placed a polyester strip on the coronal aspect of the copper band, and used finger pressure to compress the restorative materials against dentin. Using a SmartLite IQ (LD Caulk/Dentsply) with an intensity of 500 mW/cm^2, the first investigator photo-cured the RMGI or Z250 for 20 seconds while the second investigator held the copper band steady. Fifteen specimens were made for each experimental and control group (see Figure 1).

Table 1: *Composition of Materials*

Material	Composition
RMGI	
Fuji II LC	Powder
	Fluoro-alumino-silicate glass
	Liquid
	Polyacrylic acid, HEMA, trimethlyene dicarbonate, and other proprietary ingredients
Ketac Nano LC	Paste A
	Silanted glass, zirconia and silica HEMA, PEGDMA, Bis-GMA, TEGMA, fluoro-alumino-silicate glass
	Paste B
	Silanated ceramic, water, HEMA, and copolymer of acrylic and itaconic acids
Riva LC	Liquid
	Polyacrylic and tartaric acids, HEMA, DMA, and acidic monomer
	Powder
	Fluoro-alumino-silicate glass and polyacrylic acid
Composite	
Filtek Z250	Silane-treated ceramic filler, bisphenol A polyethylene glycol diether DMA, diurethane DMA, Bis-GMA, TEGDMA
Conditioning Agents	
EDTA	17% ethylene-diamine-tetracetic acid
Cavity Conditioner	20% polyacrylic acid and 3% aluminum chloride
Self Conditioner	HEMA, 4-META, ethanol, and water
Ketac Primer	HEMA, water, and copolymer of acrylic and itaconic acids
Optibond Solo Plus	Bisphenol glycidyl methacrylate, glycerol DMA, glycerol phosphate DMA, DMAs, ethanol silicone oxide, barium borosilicate, and sodium hexafluorosilicate$_6$

Abbreviations: Bis-GMA, bisphenol A glycidyl methacrylate; DMA, dimethacrylate; 4-META, 4- methacryloxyethyltrimellitate anhydride; HEMA, 2-hydroxyethyl methacrylate; PEGDMA, polyethylene glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate.

Table 2: *Conditioning Protocols*

Agent	Manufacturer Instructions
Cavity Conditioner	1. Apply for 10 seconds.
	2. Rinse thoroughly with air/water aerosol and gently dry with air but do not desiccate.
EDTA	1. Apply for 60 seconds.
	2. Rinse thoroughly with air/water aerosol and gently dry with air but do not desiccate.
Self Conditioner	1. Apply a thin layer on dentin and leave undisturbed for 10 seconds.
	2. Dry for five seconds with air but do not desiccate.
Ketac Primer	1. Apply for 15 seconds, keeping the surface wet with primer.
	2. Gently dry with air for 10 seconds.
	3. Photo-cure for 10 seconds.
Optibond Solo Plus	1. Apply 37% phosphoric acid for 10 seconds and thoroughly rinse with an air/water aerosol for 10 seconds.
	2. Apply bonding agent with a scrubbing motion for 10 seconds, and evaporate the solvent for 10 seconds using air.
	3. Repeat Step 2 and photo-cure for 20 seconds.

Excess restorative material was removed from the matrix/dentin interface with a sharp #25 Bard Parker blade (Miltex, York, PA, USA). The samples were stored for a total of 48 hours prior to testing. Initially, they were stored for 24 hours in 100% humidity at 37°C. They were then removed from the humidor and placed in 37°C deionized water for 24 hours. Samples were placed in an Instron Universal Testing Machine (Model TTC, Instron Corporation, Canton, MA, USA) and shear tested with a crosshead speed of 0.5 mm/min. A shearing bar beveled to a 1-mm-thick contact surface area was placed at the junction of dentin and copper band matrix (see Figure 2). The load required for failure was recorded

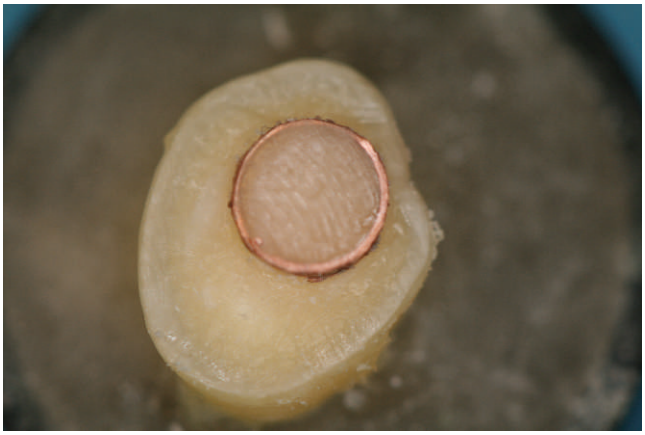

Figure 1. *Completed specimen.*

in pounds and converted to megapascals (MPa). To assess the effect of the independent variables (surface treatment and RMGI), the parametric data were analyzed with a two-way analysis of variance (ANOVA). A Tukey honestly significant difference multiple comparison procedure was used to assess significant differences among groups at a confidence level of $p < 0.05$. All analyses were performed using SAS software (SAS Institute Inc, Cary NC, USA). After testing, the specimens were visually inspected with 4.8× magnification to classify failures as cohesive, adhesive, or mixed.

RESULTS

The minimum, maximum, mean shear bond strengths, standard deviations, and failure modes are listed in Table 3. The data were skewed and therefore analyzed on a log-transformed scale. A two-way ANOVA indicated significant differences between groups ($p<0.0001$) and significant interac-

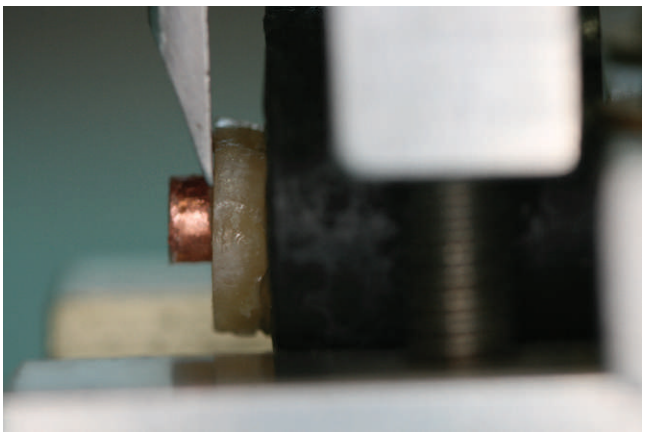

Figure 2. *Specimen in the Instron.*

Table 3: *Shear Bond Strength (MPa)*

RMGI and Conditioning Agent	Min	Max	Mean	SD	Failure Mode (Adhesive, Cohesive, Mixed)
Fuji II LC, Smear Layer	1.85	6.50	3.47	1.18	(15, 0, 0)
Fuji II LC, Cavity Conditioner[a]	1.81	8.79	4.99 DEF[b]	2.07	(15, 0, 0)
Fuji II LC, EDTA	5.14	10.68	7.90 BCD	1.75	(15, 0, 0)
Fuji II LC, Ketac Primer	6.60	12.20	9.36 ABC	1.74	(15, 0, 0)
Fuji II LC, Self Conditioner	7.24	12.20	9.37 ABC	1.49	(15, 0, 0)
Fuji II LC, OBSP	10.13	15.13	12.11 AB	1.50	(12, 0, 3)
Ketac Nano, Smear Layer	1.00	3.88	1.79	0.83	(15, 0, 0)
Ketac Nano, Cavity Conditioner	0.27	7.80	1.70 G	2.68	(15, 0, 0)
Ketac Nano, EDTA	2.00	9.90	4.76 DEF	2.73	(15, 0, 0)
Ketac Nano, Ketac Primer[a]	1.90	6.78	3.72 EF	1.44	(15, 0, 0)
Ketac Nano, Self Conditioner	5.10	10.32	7.66 BCD	1.61	(15, 0, 0)
Ketac Nano, OBSP	8.70	16.60	12.11 AB	2.23	(13, 0, 2)
Riva LC, Smear Layer	2.88	8.23	4.76	1.56	(15, 0, 0)
Riva LC, Cavity Conditioner[a]	1.70	7.08	3.63 F	1.56	(15, 0, 0)
Riva LC, EDTA	2.37	6.82	3.86 EF	1.56	(15, 0, 0)
Riva LC, Ketac Primer	2.40	6.40	3.59 F	1.20	(15, 0, 0)
Riva LC, Self Conditioner	4.89	7.08	6.15 CDE	0.75	(15, 0, 0)
Riva LC, OBSP	13.30	18.70	15.47 A	1.75	(12, 0, 3)
Filtek Z-250, OBSP	10.88	19.73	14.63 A	2.88	(12, 0, 3)

Abbreviations: Max, maximum; Min, minimum; OBSP, Optibond Solo Plus; RGMI, resin-modified glass ionomer; SD, standard deviations.
[a] *Indicates the manufacturer's recommended conditioner.*
[b] *Tukey multiple comparison procedure: Means not sharing the same letter are significantly different* ($p<0.05$).

tions between RMGIs and conditioning agents. The results for each RMGI and conditioning agent will be presented and discussed individually (see Figure 3). For Fuji II LC the five conditioning protocols were significantly different and higher than leaving the smear layer intact ($p<0.013$). Regarding Ketac Nano, all conditioning agents were significantly different and higher than the negative control except for Cavity Conditioner ($p=0.7335$). When bonding Riva LC, none of the conditioning agents were stronger than the negative control ($p<0.05$) except Optibond Solo Plus.

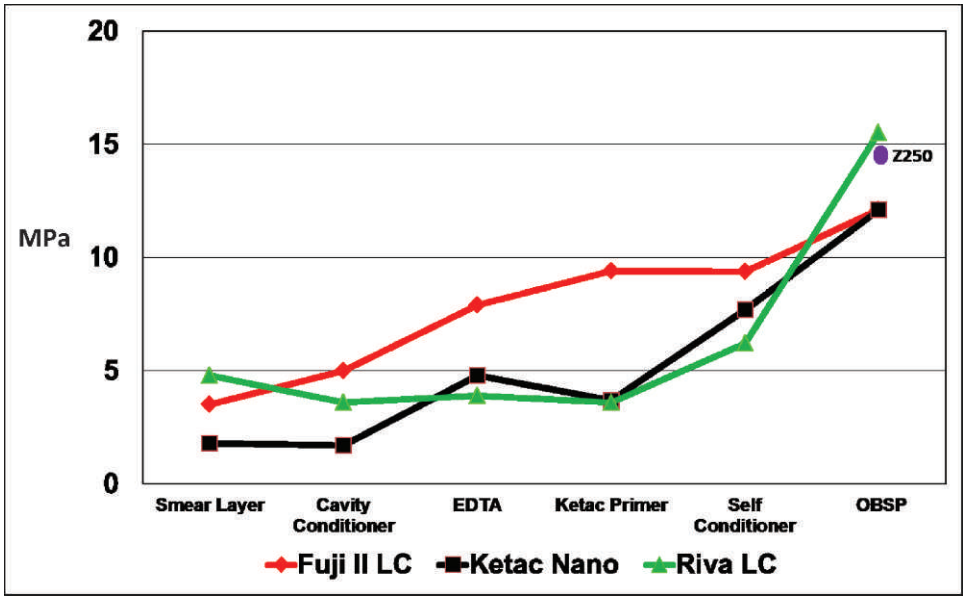

Figure 3. *Graph of the shear bond strengths of three RMGIs.*

Comparing the three RMGIs within each conditioning group, all RMGIs were significantly different from one another when the smear layer was intact ($p<0.031$), with Riva LC (4.8 MPa) significantly stronger than the others. Additionally, all three RMGIs were significantly different from one another when Cavity Conditioner was used ($p<0.0297$), with Fuji II LC (5.0 MPa) stronger than the others. When conditioning with EDTA, Ketac Nano (4.8 MPa) and Riva LC (3.9 MPa) were not different ($p>0.15$), but they were weaker than Fuji II LC (7.9 MPa) ($p<0.0006$). Within the Ketac Primer group, Fuji II LC (9.4 MPa) was significantly stronger than Ketac Nano (3.7 MPa) and Riva LC (3.6 MPa) ($p<0.031$), which were not significantly different from each other ($p>0.08$). When Self Conditioner was used, Fuji II LC (9.4 MPa) and Ketac Nano (7.7 MPa) were not different from each other ($p>0.13$), but both were stronger than Riva LC (6.2 MPa) ($p=0.0042$). There was no difference among the three RMGIs and Z250 when Optibond Solo Plus was used ($p=0.2083$).

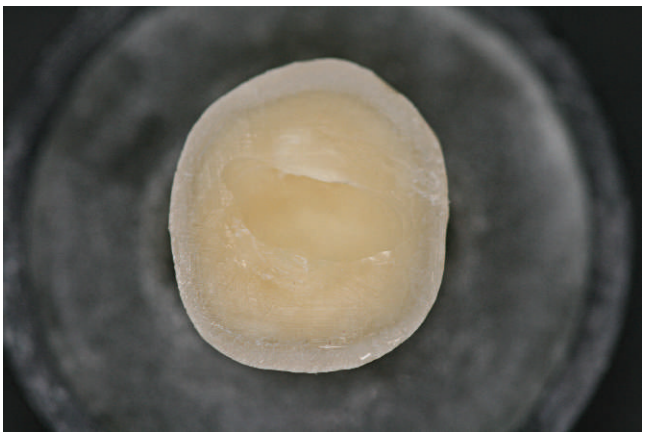

Figure 4. *Mixed failure in which dentin and RMGI fractured.*

DISCUSSION

The null hypothesis that surface treatments would not have an effect on the shear bond strength of three RMGIs to dentin was rejected. Using visual examination of the fractured specimens, we observed predominately adhesive failures for all groups except those that were bonded with Optibond Solo Plus. In these groups, including the positive control, two to three specimens from each group exhibited a mixed failure in which the dentin and RMGI fractured (see Figure 4). This would suggest these bonding configurations surpassed the inherent strength of the RMGI and dentin, or dentin and the RMGI may have been weakened by the presence of structural flaws. Therefore, the true maximum bond strength could not be measured when dentin or RMGI fractured during testing.

The overall question of which combination is best may be answered by comparing all of the experimental groups excluding the negative control (see Table 3). The Tukey multiple comparison procedure indicated that all three RMGIs when used with a total etch technique and Optibond Solo Plus were

statistically equal to the positive control or the resin-dentin bond strength. Additionally, Fuji II LC used with Ketac Primer or Self Conditioner was equal to the positive control ($p>0.05$).

The shear bond strength of the Fuji II LC with the smear layer intact (3.5 MPa) is nearly the same as that in a study by Hajizadeh and colleagues,[14] who reported shear bond strength of 3.1 MPa. There are several explanations why RMGIs are able to bond to the smear layer. First, RMGIs contain polyacrylic acid (polyalkenoic acid chains), which acts as a mild self-conditioner.[15] Second, the smear layer contains calcium ions that may provide bonding sites for chemical bonding with the polyalkenoic acid chains in the RMGI. Furthermore, the inherent dentin irregularities produce during specimen preparation provided micromechanical retention.

The purpose of Cavity Conditioner (polyacrylic acid) is to remove the smear layer without completely unplugging the dentin tubules. The exposed calcium ions within hydroxyapatite are available for chemical bonding with the carboxyl groups of the polyalkenoic acid. Any collagen that becomes exposed may provide additional micromechanical retention. The addition of aluminum chloride in Cavity Conditioner is thought to stabilize the collagen matrix during demineralization, allowing better penetration of the RMGI.[16] This study did not demonstrate any benefit of using Cavity Conditioner, which, ironically, two manufacturers recommend as the conditioning agent. This finding is supported by several other studies.[16-21]

Increased bond strengths with EDTA for Fuji II LC and Ketac Nano may be the result of enhanced micromechanical retention due to improved removal of the smear layer and dentinal plugs. Additionally, EDTA does not alter the fibrillar structure of collagen, allowing the mineral content of collagen to bond with the ionic component of RMGIs.[22] Fagundes and others[23] demonstrated increased microtensile bond strength of Fuji II LC and Vitremer (3M ESPE) following pretreatment with EDTA compared with polyacrylic acid. Additionally, Nakanuma and others[24] using Fuji II LC demonstrated greater tensile bond strength when EDTA was combined with experimental dentin primers and a bonding agent than when used with polyacrylic acid.

The purpose of primers such as Ketac Primer and Self Conditioner is to improve the wettability of dentin, allowing improved monomer penetration into the hydrophilic dentin substrate. Primers contain unsaturated carbon-carbon bonds that may lead to direct covalent bonding with resin constituents of RMGIs when polymerized. If the pH of the primer is low enough, it may also partially remove the smear layer, allowing the RMGI matrix to penetrate into the superficial layer of dentin, creating a cement-matrix-dentin interdiffusion zone.[25] Ketac Primer and Self Conditioner have a pH of 3.0 and 1.8, respectively. Self Conditioner, with a lower pH, may have more completely removed the smear layer than Ketac Primer, resulting in higher bond strengths for Ketac Nano and Riva LC. However, we did not observe the same effect with Fuji II LC because Ketac Primer (9.4 MPa) and Self Conditioner (9.4 MPa) produced the same bond strengths, suggesting that the RMGIs react differently with specific conditioners and primers. Other investigators support the fact that primers compared with polyacrylic acid increased bond strength of RMGI to dentin.[18,25-27]

Riva LC performed best only when used with Optibond Solo Plus after total etching. This suggests that Riva LC behaves more like a resin than a CGI. In this study all three RMGIs obtained their highest bond strengths when Optibond Solo Plus was applied after etching dentin with phosphoric acid. Obtaining improved bond strengths with bonding agents compared with other conditioning agents is supported by other investigations.[21,28,29] Micromechanical bonding through the hybrid layer for RMGIs has been demonstrated with different conditioners producing different degrees of demineralization and hybrid layer thickness.[16] Dentin bonding agents are able to form a chemical union with RMGIs due to the presence of HEMA and other resins in RMGIs. Why manufacturers continue to recommend the use of polyacrylic acid as a conditioner for RMGIs despite the knowledge that they contain resin is not fully understood. Development of dentin conditioners for CGIs occurred in the late 1970s, a decade before acid etching dentin was an accepted procedure. Many of the conditioning agents evaluated removed the smear layer, opened dentin tubules, and were considered detrimental.[17] The calcium-rich hydroxyapatite was thought to be unavailable for bonding with the polyalkenoic acid chains in conventional glass ionomers. The tradition of using polyacrylic acid was simply carried over for use with RMGIs.

Results of this study suggest the hybrid layer may be the major source of bond strength, whereas chemical bonding may play an important role in providing marginal integrity, bond durability, and extending the longevity of the restoration.[30,31] In addition to increased bond strength, an advantage of

using a dentin bonding agent is that the sandwich technique in which a resin is veneered over a RMGI is simplified by using a single conditioning step that works both for composites and RMGIs.

Clinicians may be reluctant to use dentin bonding agents with RMGIs because there may be a decrease in the amount of fluoride released. It has been demonstrated that a dentin bonding agent significantly reduced, but did not prevent, fluoride passage.[32,33] However, the minimal quantity of fluoride necessary to clinically inhibit the caries process has never been established. Using silver nitrate ions and scanning electron microscopy, Sano and others[34] demonstrated leakage within the hybrid layer. Given that silver nitrate ions used in their study have similar dimensions as fluoride ions, one may postulate that fluoride ions may also penetrate through the hybrid layer and into adjacent dentin to provide cariostatic properties.[34]

Setting reaction of RMGIs relies on two different mechanisms that depend and compete with each other.[35] Khoroushi and others[36] demonstrated that delaying light polymerization of Fuji II LC for two minutes, thus allowing the acid-base chemical reaction to proceed first, resulted in significantly decreased shear bond strength. Therefore, for maximum bond strength it is important to photo-cure RMGIs soon after placement. Any remaining RMGI not photo-cured may continue to undergo the acid-base setting reaction but at a reduced rate.

It is impossible to uniformly compare results across different studies. Slight differences in methodologies produce varying results. Some studies bond a column of RMGI; others bond the RMGI, then apply a column of composite resin on top of it. Resin-modified glass ionomers have been tested as dentin bonding agents, luting agents, liners/bases or restorative materials. It is unacceptable to uniformly compare results for all types of RMGI materials. Additionally, some studies have used human teeth and others bovine incisors. Research using microtensile and microshear bond methodologies have produced much greater bond strengths than what was reported in this study.[37,38] When the bonding surface can be kept small (1 mm^2 for microtensile and microshear methodologies), there is a more homogeneous stress distribution. The bonding surface area in this study was 19 mm^2. When larger surface areas are used the bonding material will contain larger flaws and voids resulting in higher stress concentrations in these areas that lead to lower bond strengths. Additionally, a 1-mm flat-edge chisel used in this study causes greater stress concentration at the area of load application.[39] However, having the RMGI surrounded by a copper band may have allowed the load to be applied more evenly due to copper having a higher modulus than RMGI.[40,41] We believe this to be similar to using a wire loop or notched bar for load application.[39,42] Promising laboratory results do not necessarily guarantee clinical success. Rather, *in vitro* studies serve as a good screening tool and guide for selection of dental materials and procedures that may be tested clinically.

CONCLUSIONS

1. The use of Cavity Conditioner did not significantly improve the bond strength of the three RMGIs compared with leaving the smear layer intact.
2. The effect of conditioning agent was dependent upon the RMGI.
3. All three RMGIs bonded with Optibond Solo Plus after acid etching produced shear bond strengths that were not statistically different than the composite-dentin bond.

Conflict of Interest

The authors of this article 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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Bond Durability of Self-etch Adhesive to Ethanol-Based Chlorhexidine Pretreated Dentin after Storage in Artificial Saliva and under Intrapulpal Pressure Simulation

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Clinical Relevance

Ethanol-based chlorhexidine pretreatment is not recommended as a generalized approach to enhance bonding durability.

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SUMMARY

Objective: **To evaluate the bond strength durability of a single-step self-etch adhesive to dentin pretreated with either ethanol-based chlorhexidine (ECHX) or water-based chlorhexidine (WCHX) after storage in artificial saliva and under intrapulpal pressure simulation (IPPS).**

Methods: **The occlusal enamel of 30 freshly extracted premolars was trimmed to expose midcoronal dentin. Roots were sectioned to expose the pulp chamber. Specimens were distributed over three groups (n=10) according to the dentin pretreatment used. In the first group, Adper Easy One (3M ESPE) was**

applied to the dentin surfaces according to the manufacturer's instructions (control group). In the second group, dentin was pretreated before bonding with 1 mL of 2% CHX diacetate dissolved in 100% ethanol (ECHX). The third group received the same pretreatment; however, CHX was dissolved in distilled water (WCHX). Pretreatment and bonding were carried out while the specimens were subjected to IPPS. Resin composite (Valux Plus, 3MESPE) buildups were made. After curing, specimens were stored in artificial saliva and under IPPS at 37°C in a specially constructed incubator (n=5/group) either for 24 hours or six months prior to testing. Thereafter, bonded specimens were sectioned into sticks with a cross section of 0.9 ± 0.01 mm^2 and subjected to microtensile bond strength (μTBS) testing (n=25/subgroup) using a universal testing machine. Data were statistically analyzed using two-way analysis of variance (ANOVA) with repeated measures, one-way ANOVA , and Bonferroni post hoc tests ($p \leq 0.05$). Failure modes were determined using a scanning electron microscope.

Results: **After 24 hours of storage, control and WCHX groups revealed significantly higher μTBS than the ECHX group. After six-month storage in artificial saliva and IPPS, only the WCHX group maintained its μTBS value. The predominant mode of failure was the mixed type, except for the ECHX group, which was mostly adhesive.**

Conclusion: **Pretreatment of the dentin with ECHX had a negative effect on bonding of the tested single-step self-etch adhesive; however, WCHX showed bond stability under IPPS.**

INTRODUCTION

Based on both *in vitro* and *in vivo* work done over the past decade, considerable evidence has been accumulated concerning the lack of bond durability of resin adhesives to dentin.[1] The tooth-restoration interface, which consists of dentin organic matrix, remaining hydroxyapatite, adhesive resin, and solvents, is still the weakest part for any adhesive restoration.[2] Hydrolytic degradation of denuded dentin collagen has been suggested as a possible mechanism responsible for adhesive bond degradation. Hence, some approaches were encountered to enhance bond durability.[3]

The wet-bonding technique introduced by Kanca[4] has been adopted as a standard protocol for the latest generation of dentin adhesive systems. It made use of the continued presence of water in demineralized dentin, which is critical to prevent collapse of the dentin matrix. However, this technique was found to cause phase separation of hydrophobic BISGMA monomer in the hybrid layer.[5] Furthermore, there is no general consensus on how wet the dentin should be. Recently, a new philosophical approach has been developed called ethanol wet bonding.[6] This philosophy embraces the concept of water replacement from interfibrillar and intrafibrillar collagen spaces by ethanol to create a comparatively hydrophobic, ethanol-suspended demineralized collagen matrix for infiltration by hydrophobic resin monomers.[7-9] This allows for more and deeper resin penetration and better collagen fiber encapsulation. Replacement of water by ethanol from the collagen intrafibrillar compartments also removes the hydrolytic medium necessary for the function of the collagen-bound matrix metalloproteinases (MMPs). MMPs are responsible for the degradation of resin-sparse collagen fibrils within the hybrid layer.[10,11]

Ethanol wet bonding differs conceptually from the use of chlorhexidine (CHX), a potent MMP inhibitor in preventing hybrid layer degradation.[11-13] Substantivity of CHX, or its ability to be retained in dentin matrices, could be the reason why CHX-treated acid-etched dentin may form hybrid layers that are more stable over time.[11-15] Once bound, CHX is relatively resistant to the displacing effects of ethanol but is more easily removed by water.[16] Thus, if CHX-treated acid-etched dentin is not rinsed with water, most of the CHX applied to the matrix will remain bound during the application of a solvated etch-and-rinse adhesive.[16]

Very few studies[17-19] examined the benefits of the adjunctive use of CHX and ethanol as an efficient simplification step on the durability of resin-dentin bonds. However, the authors tested this adjunct with an etch-and-rinse adhesive system at normal laboratory conditions. This study compared the effectiveness of using CHX dissolved in either water or ethanol on the prevention of bond degradation of recently simplified single-step self-etch adhesive under some clinical simulating conditions (artificial saliva immersion, intrapulpal pressure simulation at 37°C). The null hypothesis tested was that water-based CHX as well as ethanol-based CHX do not influence the microtensile bond strength of single-step self-etch adhesive after storage in artificial

saliva at 37°C and under intrapulpal pressure simulation for 24 hours and six months.

MATERIALS AND METHODS

Specimen Preparation

Sound single-rooted premolars, extracted from an age group of 18 to 20 years, were stored in phosphate buffer solution containing 0.2% sodium azide at 4°C. The teeth were collected from patients after the protocol had been approved by the Faculty of Dentistry's Ethics Committee at Cairo University, Egypt. All teeth were used within 1 month after extraction. Each tooth was trimmed perpendicular to its long axis, exposing the dentin using a slow-speed diamond saw sectioning machine (Buehler Isomet Low Speed Saw, Lake Bluff, IL, USA) under water coolant. Another cut was made parallel to the occlusal surface, 2 mm below the cementoenamel junction, exposing the pulp chamber. Remnants of pulp tissue in the pulp chamber were removed using a discoid excavator (Carl Martin GmbH, Solingen, Germany) without touching the walls of the pulp chamber.[20] Dentin surfaces were then wet polished with 600-grit SiC paper to create a standard surface roughness and smear layer. The specimens (n=30) were connected to the intrapulpal pressure assembly during bonding and storage following the same procedures described by Mobarak.[21]

Restorative Procedures

Prepared specimens were divided into three groups (n=10) according to the dentin pretreatment used: in the first group (control group), Adper Easy One (single-step self-etch adhesive system; 3M ESPE dental products, St Paul, MN, USA) was applied to the prepared dentin surface according to the manufacturer's instructions. It was applied using a disposable brush tip, left undisturbed for 20 seconds, dried with oil-free mild air flow for 5 seconds, then light cured for 20 seconds. In the second group, dentin was pretreated before bonding with 1 mL of 2% CHX diacetate (Sigma-Aldrich, St Louis, MO, USA) dissolved in 100% ethanol (ECHX). The third group received the same pretreatment; however, CHX was dissolved in distilled water (WCHX). CHX was left undisturbed for 60 seconds and dried with oil-free mild air flow for 30 seconds at a distance of 10 mm.[21]

Materials specifications, manufacturers, compositions, and batch numbers are listed in Table 1. Resin composite (Valux Plus, 3M ESPE dental products) of shade A1 was applied in two increments of 2 mm each. Each increment was polymerized for 40 seconds using Bluephase C5 (Ivoclar Vivadent, Schaan, Liechtenstein) with an intensity $\geq$500 mW/cm^2. Light intensity was checked using an LED radiometer (Kerr Dental Specialties, West Collins, Orange, CA, USA). The specimens were then immersed in artificial saliva[11] at 37°C in a specially constructed large incubator, to accommodate the intrapulpal pressure assembly. Specimens of each group (n=10) were divided into two subgroups (n=5) according to the storage duration, which was 24 hours or six months.

Microtensile Bond Strength Testing

After storage of the bonded teeth, each tooth was longitudinally sectioned in both mesiodistal and buccal-lingual directions across the bonded interface to obtain multiple sticks of approximately 0.9 $\pm$ 0.01 mm^2 for the microtensile bond strength (μTBS) test. From each tooth, the central sticks were collected. A digital caliber was used to check the cross-sectional area and length of the sticks. Sticks of similar length and remaining dentin thickness (average of five sticks from each tooth) were tested. This resulted in a total of 25 sticks for each subgroup to be tested. Each stick was fixed to the attachment with a cyanoacrylate adhesive (Rocket Heavy, Corona, City, CA, USA) and stressed in tension using a universal testing machine (Lloyd Instruments Ltd, Ametek Company, West Sussex, UK) at a cross-head speed of 0.5 mm/min until failure. The tensile force at failure was recorded and converted to tensile stress in MPa units using computer software (Nexygen-MT Lloyd Instruments). Sticks that failed before testing were counted as 0 MPa.[22,23] Cohesively failed specimens in the resin composite or the dentin were discarded and not included in the calculations.[24] Two-way analysis of variance (ANOVA) with repeated measures was used to compare the effect of dentin pretreatment, storage time, and their interaction. One-way ANOVA was used to test the effect of the difference in dentin pretreatment on the bond strength values for each storage time. This was followed by the Bonferroni post-hoc test for pairwise comparison. A *t*-test was used to compare between 24-hour and six-month μTBS mean values for each adhesive system. The significance level was set at $p \leq 0.05$. Data were analyzed using the SPSS program for windows (Statistical Package for the Social Sciences, release 15 for MS Windows, 2006, SPSS Inc., Chicago, IL, USA).

Fractured sides of all specimens were inspected under a scanning electron microscope (SEM; 515;

Table 1: *Materials Specifications, Manufacturers, Compositions, and Batch Numbers*

Material (Specification; Manufacturer)	Composition	Batch No.
Adper Easy One (single-step self-etch adhesive system; 3M ESPE dental products, St Paul, MN, USA)	HEMA, Bis-GMA, methacrylated phosphoric esters, 1.6 hexanediol dimethacrylate, methacrylate functionalized polyalkenoic acid (vitre bond copolymer), finely dispersed bonded silica filler with 7 nm primary particle size, ethanol, water, initiator based on campharquinone, stabilizers	D-82229
Valux Plus $A_{3.5}$ (light-cured microhybrid resin composite; 3M ESPE dental products, St Paul, MN, USA)	Monomer matrix: Bis-GMA and TEGDMA resins	$5540A_{3.5}$
	Inorganic fillers: zirconia/silica of a particle size range 3.5 to 0.01 μm (66% by volume)	
	Additional contents: catalysts, stabilizers, and pigments (0.8% by weight)	

Abbreviations: Bis-GMA, bisphenol-A-diglycidyl-dimethacrylate; HEMA, hydroxyethyl-methacrylate; TEGDMA, tri-ethylene-glycol dimethacrylate.

Philips, Einhoven, the Netherlands) at different magnifications. Failure modes were evaluated at 100× and classified into four types: type 1, adhesive failure at the dentin side; type 2, cohesive failure in the adhesive layer; type 3, mixed failure (adhesive failure at the dentin side and cohesive in the adhesive layer); type 4, mixed all (adhesive failure at the dentin side, cohesive in adhesive, and cohesive in resin composite). The frequency of each mode of failure was calculated for each subgroup.[25]

RESULTS

Two-way ANOVA with repeated measures revealed that there were statistically significant differences for dentin pretreatment ($p<0.0001$) and storage time ($p=0.04$) as well as for their interaction ($p<0.05$). The means and standard deviations (SD) of the μTBS values of all tested groups are presented in Table 2. One-way ANOVA indicated that there was a significant difference among the dentin pretreatments when tested after 24 hours ($p<0.001$) and after six-month storage ($p<0.0001$). After 24 hours of storage, the Bonferroni post hoc test revealed no statistically significant difference between the mean microtensile values of the control group and the WCHX group, which were significantly higher than ECHX (Table 2). After six months of storage, there was a statistically significant difference among all tested groups. The WCHX-pretreated group maintained its higher values. However, the control group and the ECHX group decreased significantly (Table 2). For the control group, after 24 hours of storage, the predominant mode of failure was the mixed type

Table 2: *Microtensile Bond Strength Values (in MPa) of Tested Adhesive Systems*[a]

Storage Period	Dentin Pretreatment						
	Control		Water-Based Chlorhxidine Pretreatment		Ethanol-Based Chlorhxidine Pretreatment		*p*-value
	Mean (SD)	ptf/tnt	Mean (SD)	ptf/tnt	Mean (SD)	ptf/tnt	
24 h	28.5 (1.4)^{Aa}	0/25	28.3 (1.8) ^{aA}	0/25	16.2 (2.2) ^{bA}	4/25	0.001
6 mo	18.1 (1.8) ^{aB}	0/25	32.2 (4.8) ^{bA}	0/25	5.8 (0.9)^{cB}	8/25	0.001
p value	0.001		1.02		0.00001		

Abbreviation: ptf/tnt, pretest failure/total number of tested sticks.
[a] *Within rows, means with different small letters are statistically significantly different (p≤0.05, Bonferroni test). Within columns, means with different capital letters are statistically significantly different (p≤0.05,* t*-test).*

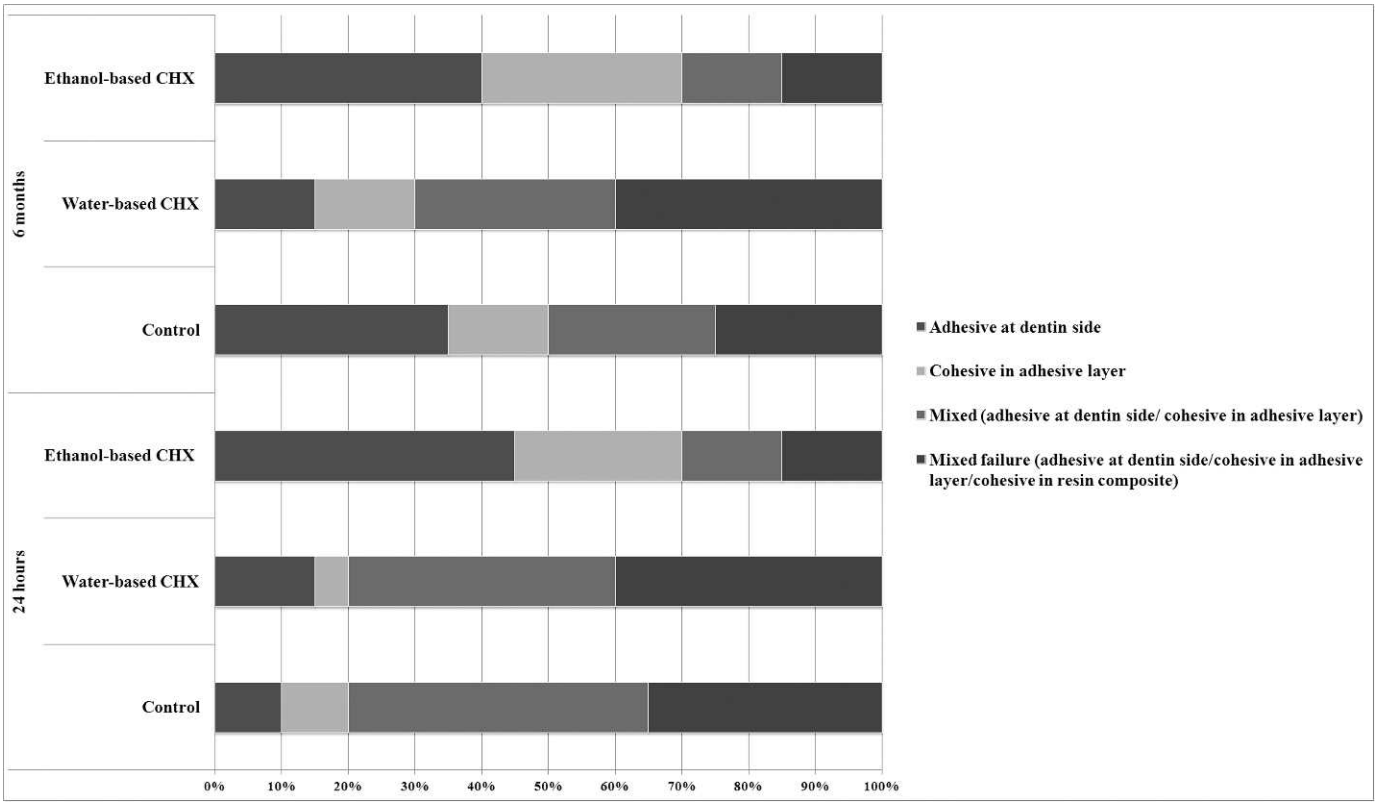

Figure 1. *Failure mode percentages of all tested groups.*

(type 3), which changed to adhesive failure at the dentin side after six months of storage. For the WCHX group, the major modes of failure were mixed types (types 3 and 4) after both aging periods. For the ECHX group, adhesive failure at the dentin side (type 1) was the predominant type after 24 hours and six months of storage (Figure 1). Representative SEM micrographs for some modes of failure are presented in Figure 2.

DISCUSSION

The results of the present study indicate the rejection of the null hypothesis, where there was a significant difference among tested groups at 24 hours and six months of storage in artificial saliva at 37°C and under simulated intrapulpal pressure.

After 24 hours of storage, the μTBS values of the control group and WCHX-pretreated group showed no statistically significant difference. Previous studies[13,15,21,26-28] revealed that 2% WCHX dentin pretreatment did not show a negative effect on the bond strength after 24 hours of storage. Surprisingly, the ECHX group recorded the least readings after 24 hours and six months of storage. The reason behind this finding is not yet clear. Previous studies[29,30] on etch-and-rinse adhesives revealed that the use of absolute ethanol (100%) for water replacement is a very sensitive technique compared with the use of ascending ethanol concentrations (50%, 70%, 80%, 95%, then 100% three times for 30 seconds each). The collapse of the collagen matrix caused by water evaporation during transition from the water to ethanol phase could result in stiffening and stabilization of the matrix in its collapsed state. Thus, reducing the application time by using a simplified absolute ethanol replacement protocol was not recommended.[29]

For this study, the concentration of CHX used with the ECHX group could be crucial. Nor and others, in 2011,[17] observed that when 2% or 3% CHX diacetate that was dissolved in absolute ethanol was used as dentin pretreatment before bonding, reduction in the initial μTBS was recorded. Nevertheless, this did not occur when 1% CHX was used. However, it should be noted that those concentrations were used with the etch-and-rinse technique. Further study is needed to investigate the most suitable CHX concentration to be dissolved in ethanol when used with single-step self-etch adhesives. Another possible reason for the loss of the μTBS values with ECHX groups is that, after its evaporation, the dentin was left dry to ionize

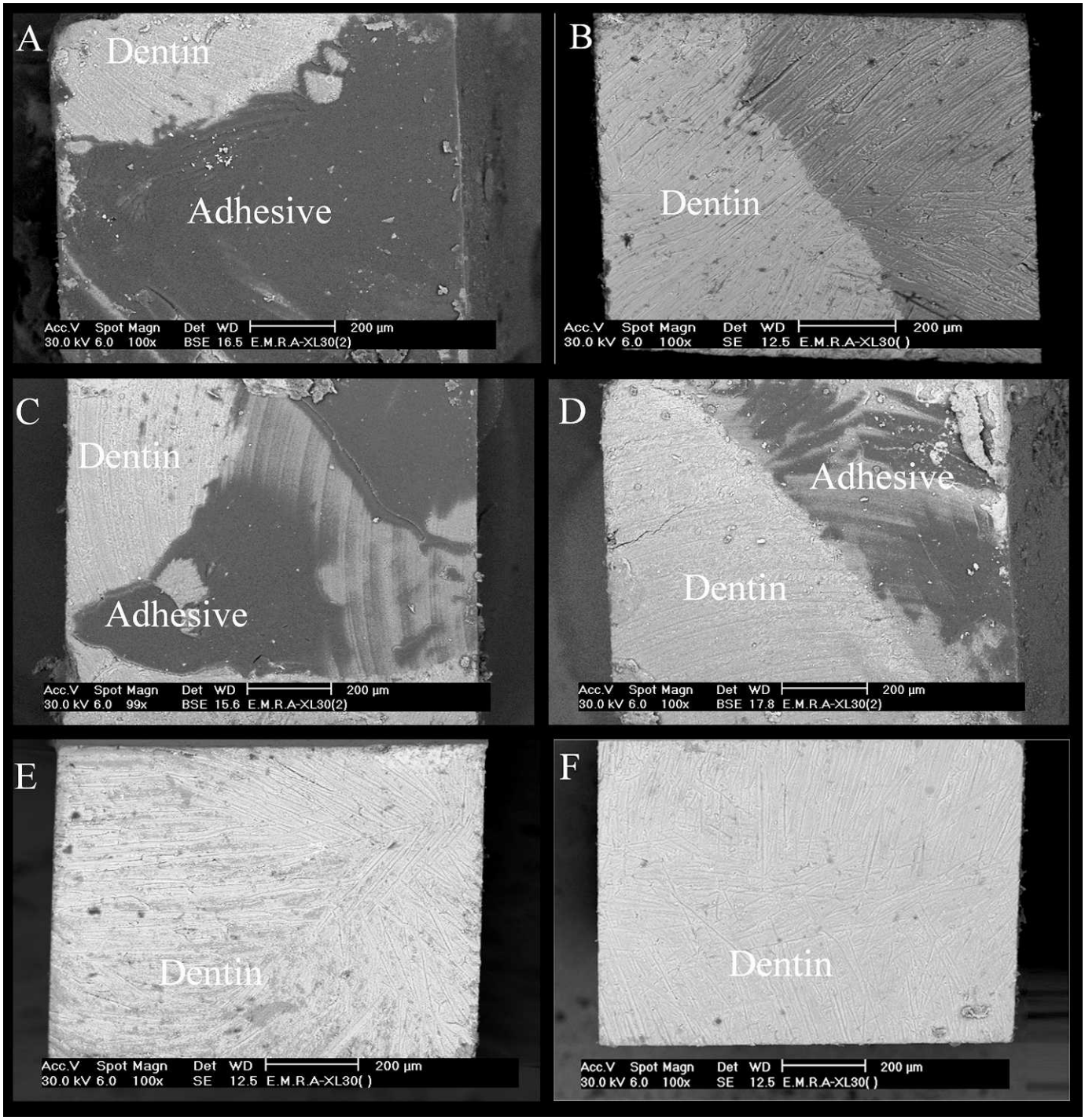

Figure 2. *Representative SEM photomicrographs of fractured surfaces of the control group showed (A) mixed failure (adhesive failure at dentin side and cohesive in adhesive layer) after 24 hours of storage, while (B) showed adhesive failure of the control group after six months. The fractured surface of the WCHX group showed a mixed type of failure (adhesive failure at the dentin side and cohesive in the adhesive layer) after 24 hours (C) and after six months (D) of storage. (E, F): Adhesive failure at the dentin side of the ECHX group after 24 hours and six months of storage.*

this single-step adhesive. For the self-etch adhesives, the water is required for the decalcification process and for preservation of the hydrated state of demineralized collagen and underlying dentin integrity.[31] However, we still need further investigation and chemical analysis to confirm this suggestion.

The mode of failure of the tested groups confirmed the μTBS results, as the control and WCHX groups

showed almost similar results, with an increased percentage of mixed type of failures. The results of the control group corroborated with the Mine and others[32] study, which showed high rates of mixed failure with Adper Easy One. This was explained by the micromechanical as well as chemical interaction of the functional monomer included in this adhesive with the hydroxyapatite crystals that remain at the surface. For the ECHX group, the most common mode of failure was adhesive failure at the dentin side (type 1), denoting that the defect was located at the dentin/adhesive interface.

After six months of storage under simulated intrapulpal pressure and in artificial saliva at 37°C, the WCHX pretreated group had a statistically higher value than the control group. Clearly, there was a drop in the bond strength of the control group, and this loss of bond strength can be due to the action of water (hydrolytic degradation) and dentin enzymes (enzymatic degradation). Regarding the enzymatic degradation, it is known that mild acids have a potential to activate MMPs[33]; pH values ranging from 2.3 to 5 are effective in activating gelatinases. The self-etch adhesive used in this study has a pH value of 2.4 and so is capable of enhancing dentin proteolytic activity without denaturation of the enzymes.

After 6-month storage under simulated pulpal pressure and in artificial saliva at 37°C, the μTBS of the groups pretreated with WCHX was significantly higher than that of the control group. This result was in accordance with the findings of other researchers.[13,15,26,27] The maintained mode of action of CHX over the six-month study period could be due to its substantivity. CHX is characterized as a strong base with cationic properties. The cationic part of the CHX molecule binds to the negative site of the substrate, which results in a cationic-anionic reaction. This involves an electrostatic attraction between the protonated amine groups of the CHX and the mineral phosphates and the carboxylic groups of collagen.[34] In the present study, the mode of failure confirmed the six-month μTBS results in which the WCHX-pretreated group maintained a higher percentage of mixed failures. On the other side, the control group showed a higher percentage of adhesive failures at the dentin side.

It seems that dentin pretreatment using CHX, as an antibacterial and antiproteolytic agent, is a beneficial way to enhance adhesive/dentin bond strength durability. However, the effect of ethanol as a solvent still needs further investigation to elucidate its advantages and its precautions for use.

CONCLUSIONS

Pretreatment of the dentin with ECHX had a negative effect on bonding of the tested single-step self-etch adhesive; however, WCHX showed bond stability under intrapulpal pressure simulation.

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 Nd:YAG Laser on the Bond Strength of Self-etching and Conventional Adhesive Systems to Dental Hard Tissues

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Clinical Relevance

The application of Nd:YAG laser prior to photopolymerization of adhesive systems in an attempt to create a new bonding layer by dentin/adhesive melting significantly increased the shear bond strength to the dentin substrate.

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SUMMARY

Purpose: The aim of this study was to investigate the influence of Nd:YAG laser on the shear bond strength to enamel and dentin of total and self-etch adhesives when the laser was applied over the adhesives, before they were

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photopolymerized, in an attempt to create a new bonding layer by dentin-adhesive melting.

Material and Methods: One-hundred twenty bovine incisors were ground to obtain flat surfaces. Specimens were divided into two substrate groups (n=60): substrate E (enamel) and substrate D (dentin). Each substrate group was subdivided into four groups (n=15), according to the surface treatment accomplished: X (Xeno III self-etching adhesive, control), XL (Xeno III + laser Nd:YAG irradiation at 140 mJ/10 Hz for 60 seconds + photopolymerization, experimental), S (acid etching + Single Bond conventional adhesive, Control), and SL (acid etching + Single Bond + laser Nd:YAG at 140 mJ/10 Hz for 60 seconds + photopolymerization, experimental). The bonding area was delimited with 3-mm-diameter adhesive tape for the bonding procedures. Cylinders of composite were fabricated on the bonding area using a Teflon matrix. The teeth were stored in water at 37°C/48 h and submitted to shear testing at a crosshead speed of 0.5 mm/min in a universal testing machine. Results were analyzed with three-way analysis of variance (ANOVA; substrate, adhesive, and treatment) and Tukey tests (α=0.05). ANOVA revealed significant differences for the substrate, adhesive system, and type of treatment: lased or unlased ($p<0.05$). The mean shear bond strength values (MPa) for the enamel groups were X=20.2 ± 5.61, XL=23.6 ± 4.92, S=20.8 ± 4.55, SL=22.1 ± 5.14 and for the dentin groups were X=14.1 ± 7.51, XL=22.2 ± 6.45, S=11.2 ± 5.77, SL=15.9 ± 3.61. For dentin, Xeno III self-etch adhesive showed significantly higher shear bond strength compared with Single Bond total-etch adhesive; Nd:YAG laser irradiation showed significantly higher shear bond strength compared with control (unlased).

Conclusion: Nd:YAG laser application prior to photopolymerization of adhesive systems significantly increased the bond strength to dentin.

INTRODUCTION

The basic mechanism of bonding to enamel and dentin is essentially an exchange process involving replacement of minerals removed from the hard dental tissue as a result of acid etching by resin monomers. When these set, they become micromechanically interlocked in the porosities thus created.[1]

Conventional adhesive systems are based on acid etching followed by a conditioning step with the primer and the application of the adhesive resin, or systems that combine the primer and the bonding agent into one application.[2] In 1994, Watanabe introduced the self-etching adhesive systems, which use nonrinse acidic monomers that simultaneously condition and prime dentin.[3] However, their low acid concentration and high hydrophilicity promote low bond strength values to enamel substrate, thin hybrid layers, and doubtful marginal sealing.[3]

Today, laser technology is being widely applied in clinical trial procedures. In 1999, Gonçalves and others[4] developed a technique for Nd:YAG laser application to dentin substrates. This technique consisted of irradiating the dentin substrate with Nd:YAG laser after etching it with phosphoric acid and applying the bonding agent. After this, the adhesive is polymerized. Gonçalves and others[4] explained that Nd:YAG laser promoted fusion and recrystallization of dentinal hydroxyapatite in the presence of resin monomers, thereby developing a new layer of dentin tissue and adhesive system joined by the action of the laser (ie, a mechanically intermingled substrate that was chemically receptive to bonding).[3]

Although most researchers have found significantly higher bond strength values for adhesive systems that received Nd:YAG irradiation prior to polymerization, most of the studies investigated total-etch adhesives bonded to dentin.[4-6] The influence of laser irradiation on self-etch adhesives has not yet been adequately evaluated, especially on enamel.

Thus, the aim of this study was to evaluate *in vitro* the influence of Nd:YAG laser on the shear bond strength to enamel and dentin of a two-step total-etch adhesive and one-step self-etch adhesives when the laser was applied over the adhesives. The null hypothesis tested was that Nd:YAG laser irradiation would not affect the bond strength of the adhesive systems to enamel and dentin substrates.

MATERIALS AND METHODS

Sample Preparation

One hundred twenty extracted bovine incisors were cleaned with a scalpel and water/pumice slurry in dental prophylactic cups. The teeth were stored in distilled water and frozen at −18°C until use, within a period of less than 28 days.[7] The roots were sectioned with a low-speed diamond saw, and the

Table 1: *Surface Conditioning Methods*

	Grit of Silicone Carbide Paper (/Time)			
	First Step	Second Step	Third Step	Fourth Step
Enamel substrate	400	600/15 s	—	—
Dentin substrate	80	320/15 s	400/15 s	600/15 s

pulp was removed using endodontic instruments. An opening was made on the lingual side of the teeth to promote exposure of the pulp chamber.

The teeth were mounted in a silicone matrix with self-curing acrylic resin, with their buccal surface kept above the surface of the mounted blocks. After polymerization, the lingual portions of the mounted teeth were ground in a trimmer using wet 80-grit sandpaper, until the wax was removed. The pulp chamber opening was used as access to measure the remaining dentin thickness with a caliper[8].

For the enamel specimens, the buccal portion was ground in a trimmer using wet 400-grit silicon carbide paper in a polishing machine (Politriz, Struers A/S, Copenhagen, Denmark), under water cooling, until the overlying enamel was removed. After this, the specimen was ground again using wet 600-grit silicon carbide paper for 15 seconds, under constant pressure, to obtain a uniform surface (Table 1).

For the dentin specimens, the buccal portion was ground in a trimmer using wet 80-grit sandpaper until dentin was exposed. Next, the dentin thickness was measured with a caliper, and this was considered the baseline measure. After this, the specimen was ground again until half of the baseline measure was obtained. This was done in a polishing machine using 320- to 600-grit silicon carbide paper for 15 seconds, under constant pressure, to obtain a uniform smear layer (Table 1).

Bonding Procedures

To delimit the area for adhesive system application, a special Scotchtape Mold (3M ESPE, St Paul, MN, USA) with a standard central hole, 3 mm in diameter, was placed on each specimen.

One-hundred twenty specimens were divided into two groups: substrate E (enamel) and substrate D (dentin). Each group was divided into four subgroups (n=15/subgroup), according to the surface treatment performed:

- Group X (control): The Xeno III self-etch adhesive (Dentsply De Trey GmbH D, Konstanz, Germany) was applied passively for 20 seconds, gently air-dried and polymerized for 10 seconds with a light unit (Curing Light XL 3000; 3M ESPE) with power density of 600 mW/cm^2 as measured by a radiometer (Curing Radiometer Model 100, Demetron Research Corporation, Danbury, CT, USA).
- Group XL (experimental): Specimens received the application of Xeno III self-etch adhesive, followed by irradiation with Nd:YAG laser in noncontact mode, scanning for 60 seconds, and light polymerization for 10 seconds.
- Group S (control): Specimens were etched for 15 seconds with 37% phosphoric acid gel, rinsed, and gently dried with absorbent paper to remove excess water. Two layers of Single Bond 2 total-etch adhesive (3M ESPE) were applied on the surface in a scrubbing motion for 15 seconds. The remaining solvent was evaporated with a brief, mild air blast, and the adhesive was polymerized for 10 seconds.
- Group SL (experimental): The Single Bond 2 total-etch adhesive was applied in the same way as in group S, and the specimens were irradiated with Nd:YAG laser in noncontact mode, scanning for 60 seconds, followed by light polymerization for 10 seconds.

Treatment with Nd:YAG Laser

The Nd:YAG laser equipment used in this study was the Laser Pulse Master 600 iQ (American Dental Technologies Inc, Corpus Christi, TX, USA) at a wavelength of 1.064 μm. The output energy of this laser device was 140 mJ per pulse, with a pulse repetition rate of 10 pulses per second (10 Hz) and total energy of 1.4 W. In this study, the laser was fitted with a noncontact tip 320 μm in diameter, and the energy density was 1200 J/cm^2 (Table 2). The laser was applied freehand, in noncontact mode, and scanning for 60 seconds. During laser application, the laser tip was at a 90° angle, perpendicular to the specimen surface, and at a distance of 5 mm from it.[4] Self-curing acrylic resin was used to make a device especially for the purpose of maintaining the distance between the laser tip and the enamel and dentin surfaces.

Restoration Placement

After the surface treatment, a split Teflon mold, with a 3-mm-diameter hole in the middle of it, was

Table 2: *Nd:YAG Laser and Irradiation Parameters Used*

Laser	Nd:YAG
Wavelength	1.064 μm
Mean power	1.4 W
Pulse frequency	10 Hz
Pulse duration	100 μs
Pulse energy	140 mJ
Energy density	1200 J/cm^2

adapted to the specimen in order to insert the composite resin. The composite resin (Filtek Z250, 3M ESPE) was inserted in two increments of about 1.5 mm each, using a spatula, and each increment was polymerized with a halogen light (Curing Light XL 3000; 3M ESPE) for 20 seconds. After removing the mold, the composite resin cylinder was light polymerized for an additional 60 seconds.

Specimens were immersed in distilled water at 37°C for 48 hours. Subsequently, the shear bond strength test was performed in a universal testing machine at a crosshead speed of 0.5 mm/min using a 100-kgf load cell (EMIC, São José dos Pinhais, Paraná, Brazil). The bond strength was determined from the highest point on the stress-strain curve measured by the load cell of the testing machine. Results obtained were expressed in MPa.

After the shear bond strength test, the specimens were analyzed under a stereomicroscope (Stemi 2000, Karl Zeiss, Gottingen, Germany) at 20× magnification. Failures were classified as cohesive failure in composite, cohesive failure in enamel/dentin, adhesive at the interface, or mixed.

Bond strength data were analyzed by three-way analysis of variance (ANOVA; substrate, adhesive system, and surface treatment—lased or unlased) followed by Tukey test (α=0.05).

Scanning Electron Microscopy (SEM) Examination

Two teeth from each group were sectioned perpendicularly to the bonding interface. The sections were polished with 2000 and 4000 mesh sheets. Phosphoric acid etchant was applied for 5 seconds and rinsed off with water for 10 seconds. Specimens were dehydrated, sputter-coated with gold-palladium, and examined using SEM.

Table 3: *Mean Bond Strength Values (MPa) and Standard Deviations (SD) for Each Group*

Substrate	Group	Mean (SD)
Enamel	X	20.29 (5.61)
Enamel	XL	23.61 (4.92)
Enamel	S	20.89 (4.55)
Enamel	SL	22.10 (5.14)
Dentin	X	14.14 (7.51)
Dentin	XL	22.23 (6.45)
Dentin	S	11.29 (5.77)
Dentin	SL	15.98 (3.61)

Abbreviations: group S, Single Bond; group SL, Single Bond/Nd:YAG laser; Group X, Xeno III; group XL, Xeno III/Nd:YAG laser.

RESULTS

The mean bond strength values in all experimental groups are presented in Table 3 and Figure 1.

ANOVA revealed that enamel substrate presented higher bond strength values compared with dentin substrate (p=0.000), Xeno III adhesive system presented higher bond strength values compared with the Single Bond adhesive system (p=0.0152), and the lased surface treatment presented higher bond strength values compared with unlased surface treatment (p=0.000).

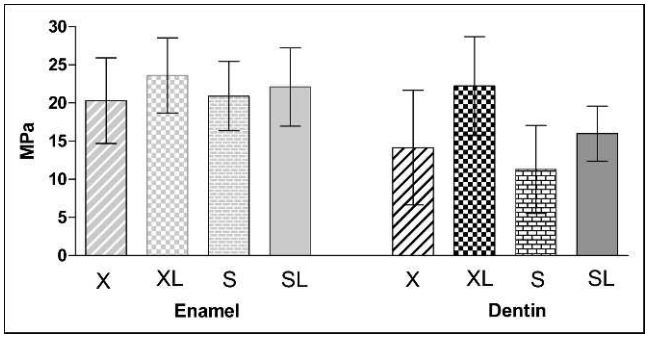

Figure 1. *Graph of means and standard deviation (MPa) of bond strength for each group. Group X, Xeno III; group XL, Xeno III/Nd:YAG laser; group S, Single Bond; group SL, Single Bond/Nd:YAG laser.*

Table 4: *Tukey Test (5%) Comparison of Bond Strength Means (MPa) for Adhesive and Substrate*[a]

Adhesive	Substrate	Mean
Xeno III	Enamel	21.953[a]
Single Bond	Enamel	21.501[ab]
Xeno III	Dentin	18.188[b]
Single Bond	Dentin	13.635[c]

[a] *Mean values with the same letters showed no statistically significant difference.*

Table 5: *Tukey Test (5%) Comparison of Bond Strength Means (MPa) for Substrate and Treatment*[a]

Laser	Substrate	Mean
Presence	Enamel	22.858[a]
Absence	Enamel	20.595[ab]
Presence	Dentin	19.107[b]
Absence	Dentin	12.716[c]

[a] *Mean values with the same letters showed no statistically significant difference.*

There was significant interaction between the independent variables of "substrate" and "treatment" (p=0.044). Enamel substrate with Xeno III adhesive system (21.95 MPa) presented higher bond strength values compared with dentin substrate, irrespective of the adhesive system tested, and dentin substrate with the Single Bond adhesive system (18.18 MPa) presented the lowest bond strength (Table 4). The Xeno III adhesive system presented higher bond strength values compared with the Single Bond adhesive system, irrespective of the substrate tested (Table 4).

There was significant interaction between the independent variables of "substrate" and "adhesive system" (p=0.045). Enamel substrate lased (22.85 MPa) presented higher bond strength values compared with dentin substrate, lased (19.10 MPa) or unlased (12.71 MPa), and irrespective of the adhesive system tested (Table 5). Treatment with Nd:YAG laser prior to photopolymerization of the adhesive systems significantly increased the bond strength to dentin substrate (Table 5).

Three-way ANOVA revealed that the interaction between the three variables was not statistically significant, so the relationship between laser and substrate for the adhesive Xeno III was similar to that for Single Bond (Figure 2).

Examination of specimens after failure indicated predominantly adhesive and mixed failure for all groups, except for group X (Xeno III) without laser (control) in enamel, which showed an almost equal distribution between the four types of failure (Table 6).

SEM micrographs of the bonded interface in dentin with Xeno III showed that the control group generally showed a well-developed resin-impregnated zone (hybrid layer; Figure 3). Figure 4 showed that there were more resin tags in the sample on which Nd:YAG laser was applied when compared with the sample in which no irradiation was applied (Figure 3) and the presence of resin tags with well-defined terminations in dentinal tubules.

SEM images showed that in the sample on which no irradiation was applied (Figure 5) and in the sample on which Nd:YAG laser was applied (Figure 6), the bond interface between enamel and Xeno III was similar in both treatments.

DISCUSSION

This study investigated the possible influence of Nd:YAG laser on the shear bond strength to enamel and dentin of adhesive systems (total etch and self-etch) when the laser was applied over the adhesives, before they were light polymerized, and on the formation of an optimized bonding layer.

Greater difficulty in promoting a satisfactory bond to dentin was observed in this study, in which the bond strength values were lower than those to enamel for both adhesive systems, independent of laser treatment. Although the bond to enamel is usually more effective than to dentin, evaluations in enamel continue to be relevant because the performance of new self-etch adhesive systems show lower bond strength values when compared with conventional systems because of the lower acid concentration.[9,10]

Peumans and others,[2] in a systematic review of contemporary clinical trials, found an inefficient clinical performance for the most commonly tested one-step self-etch adhesives, Prompt L-Pop (3M

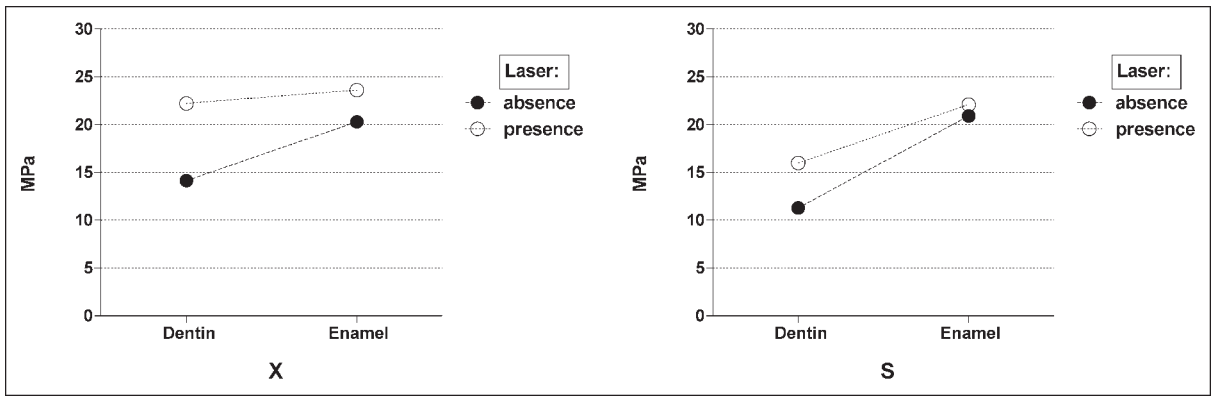

Figure 2. *Graph of means showing the interaction between the three variables. X, Xeno III self-etch adhesive; S, Single Bond total-etch adhesive.*

ESPE) and PSA (Dentsply-Detrey). They showed the highest average annual failure rate (8.1%), and most adhesives failed to meet the American Dental Association Acceptance requirements.[2]

The results of the present research showed the superiority of the self-etching adhesive Xeno III both in enamel and dentin. There was no significant difference between Xeno III and Single Bond in enamel, but it was significant in dentin. On the other hand, Faria-E-Silva and others[11] showed that the adhesive Xeno III presented lower bond strength to enamel than to dentin and that the best results for this adhesive were in dry substrates. In the study by Chaves and others,[12] Xeno III had a good performance in combination with the luting agent Variolink II, with regard to microtensile bond strength after 90 days of storage in water.

The null hypothesis was rejected for dentin substrate because the Nd:YAG laser irradiation significantly increased the bond strength to this substrate. For enamel substrate, the null hypothesis was accepted because the difference was not statistically significant.

The first applications of Nd:YAG lasers for dental surface treatment (before bond application) resulted in a reduction in bond strength when compared with

Table 6: *Failure Mode*

Substrate	Adhesive	Laser	Failure Mode[a]			
			A	M	CD	CR
Enamel	Single Bond	Absence	15	—	—	—
Dentin	Xeno III	Absence	11	2	2	—
Dentin	Xeno III	Presence	14	—	1	—
Enamel	Single Bond	Presence	15	—	—	—
Dentin	Single Bond	Presence	15	—	—	—
Enamel	Xeno III	Presence	4	5	6	—
Enamel	Xeno III	Absence	15	—	—	—
Dentin	Single Bond	Absence	12	3	—	—

[a] *Failure mode: A, adhesive failure; CD, cohesive failure in dentin; CR, cohesive failure in resin; M, mixed failure.*

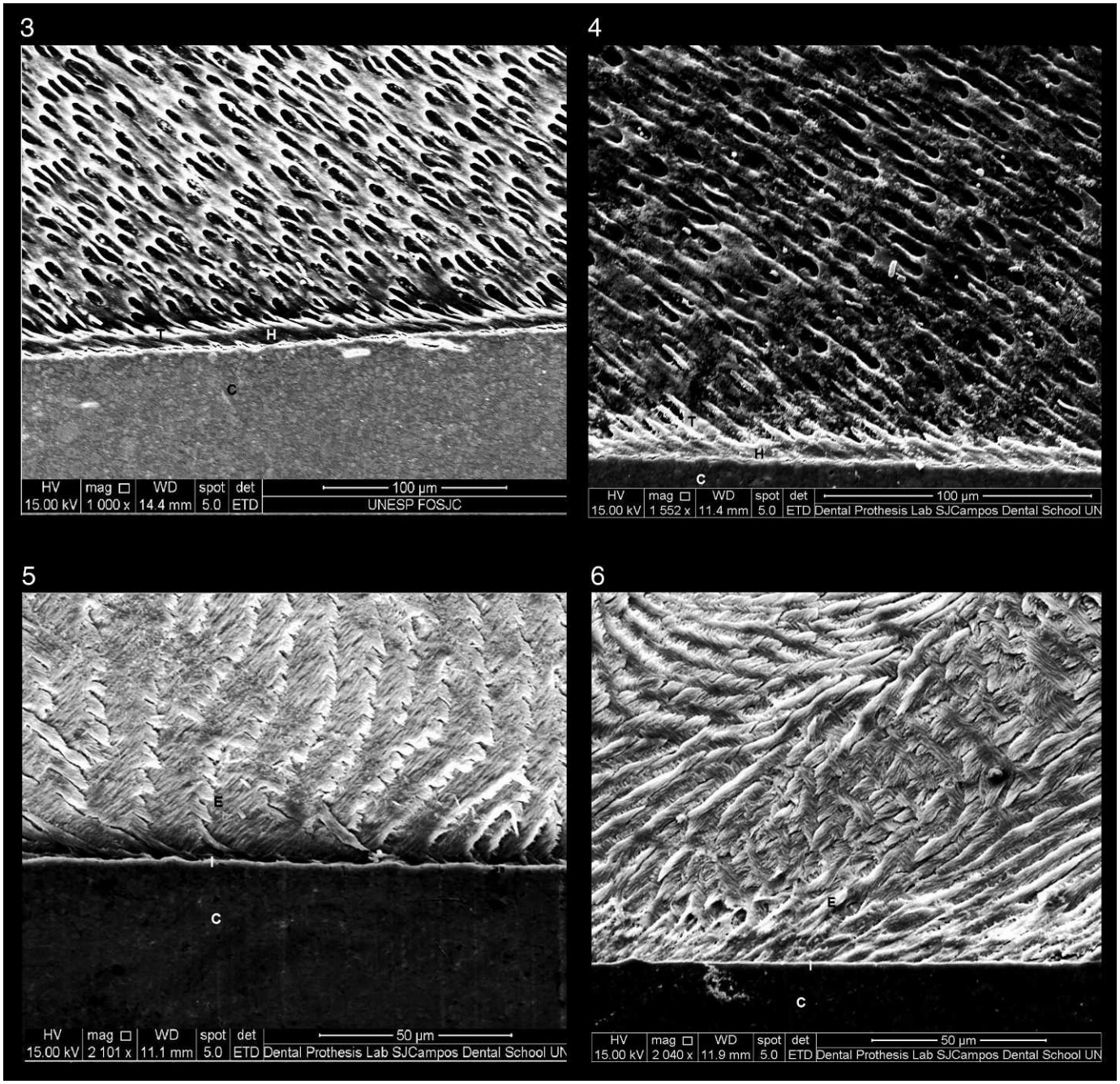

Figure 3. *Scanning electron micrograph of the bond interface of the specimen that received dentin treatment according to the manufacturer's instructions (Xeno III self-etch adhesive). C, composite; H, hybrid layer; T, resin tags.*

Figure 4. *Scanning electron micrograph of the bond interface of the specimen that received dentin treatment according to the manufacturer's instructions (Xeno III self-etch adhesive) + laser Nd:YAG. C, composite; H, hybrid layer; T, resin tags. It can be observed that there were more resin tags in the sample on which Nd:YAG laser was applied and the presence of resin tags with well-defined terminations in dentinal tubules.*

Figure 5. *Scanning electron micrograph of the bond interface of the specimen that received enamel treatment according to the manufacturer's instructions (Xeno III self-etch adhesive). C, composite; E, enamel; I, interface.*

Figure 6. *Scanning electron micrograph of the bond interface of the specimen that received enamel treatment according to the manufacturer's instructions (Xeno III self-etch adhesive) + Nd:YAG laser. C, composite; E, enamel; I, interface. It can be observed that there were no differences between the enamel-resin interface of the sample on which no irradiation was applied when compared with that of the sample that was irradiated.*

nonirradiated cavities,[13,14] because Nd:YAG lasers promote denaturation of the organic components of dentin by heat generation, fusion, and recrystallization of the dentin surface, obliterating some dentinal tubules.[13-16] These alterations in the morphology of the tooth substrate occur because of reduction in the percentage of calcium and phosphate in the dentin structure, causing changes in the organic composition of hydroxyapatite, leading to its recrystallization.[16,17]

Therefore, in the present study, the time when Nd:YAG laser was applied was changed to evaluate the influence of Nd:YAG laser after bond application and before light polymerization, a technique developed by Gonçalves and others.[4]

Gonçalves and others[4] recommended Nd:YAG laser irradiation on dentin previously conditioned and impregnated with the adhesive system but before light polymerization. This technique would promote the development of a new substrate, in which dentin substrate and adhesive would be fused by the action of the laser. The development of this new substrate explains why all of the groups irradiated by laser in this study showed a substantial increase in bond strength. This is in agreement with Matos and others[5] and Matos and others,[6] who evaluated Nd:YAG irradiation before and after adhesive application and concluded that the best results were observed when laser was used after the adhesive system application. According to Dayem and others,[18] treatment of the acid-etched dentin with Nd:YAG laser led to the significantly increased penetration depth of the adhesive bonding system into dentin.

After SEM analysis, the adhesive penetration in dentin showed fewer resinous tags in the specimen on which no irradiation was applied (Figure 3) compared with the sample that was irradiated with Nd:YAG laser (Figure 4). In addition, a new adhesive substrate composed of dentin tissue and adhesive system joined by the action of the laser was observed, as well as the presence of resin tags with well-defined terminations in dentinal tubules.[4,19] The Nd:YAG laser treatment promoted more numerous tags and improved adhesive infiltration, probably producing a resistant substrate contributing to better bond strength.[4,19]

The representative SEM micrographs obtained from the enamel substrate generally showed that there was no difference between the enamel-resin interface in the sample on which no irradiation was applied (Figure 5) when compared with that of the sample that was irradiated (Figure 6). The energy density of 140 mJ used in this study seems to contribute to better interaction between the adhesive and enamel substrate.

With regard to failure mode analysis, it should be noted that the fracture was predominantly initiated at the interface between the composite and dentin or enamel substrate (adhesive or mixed failure), except in group X (Xeno III) without laser (control) in enamel, which showed an almost equal distribution between the four types of failure. This group showed the best bond strength results. Therefore, the type of treatment (presence or absence of Nd:YAG laser irradiation) was not able to alter the failure mode of composites bonded to enamel and dentin substrates.

The energy density of 140 mJ used in this study followed instructions of previous studies. Ribeiro and others[20] observed the highest marginal microleakage results with this parameter, also using the technique of Nd:YAG laser irradiation after application of the adhesive However, future *in vivo* studies should be conducted to evaluate the thermal side effects of this technique, because Nd:YAG lasers have a high penetration depth that varies according to its parameters, so care should be taken not to overheat the pulp.

The results of the present study showed that the enamel samples subjected to laser irradiation presented the highest bond strength values, although the difference was not statistically significant when the enamel was not laser irradiated. In dentin, the irradiation with Nd:YAG laser significantly increased the bond strength values. Thus, further research is necessary to establish efficient and safe parameters.

CONCLUSION

Within the limitations of this study, it could be concluded that high bond strength values were found with enamel substrate both with Xeno III and Single Bond. It was also found that Nd:YAG laser significantly increased the bond strength to dentin substrate, although the difference in enamel was not statistically significant.

Conflict of Interest

The authors of this article 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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Award

Clinician of the Year Award 2012

Dr. Clyde Roggenkamp received the Clinician of the Year Award at the 2012 annual meeting of the American Academy of Gold Foil Operators. The following is excerpts from the award introduction, written by Mel Lund.

Following graduation from dental school at Loma Linda in 1970, he was active in the private practice of dentistry in Vermont and Massachusetts into 1976. For the next 21 years, he was a dental officer in the U.S. Air Force Dental Corps, retiring as a Lt. Colonel. In the early part of his Air Force career he was given the opportunity of advanced dental education at Indiana University. His major was in Graduate Operative with companion studies in Dental Materials and Preventive. Shortly after his program at I.U., Clyde was assigned to an Air Force base in the U.K. He just happened to own a Cessna 210 and he was allowed the option of flying himself to his appointed base.

At the conclusion of his Air Force career, Clyde joined Loma Linda University School of Dentistry as a full time faculty member in Restorative Dentistry. In 1990, Clyde successfully challenged the American Board of Operative Dentistry. In the years since, he has become active in the management of the Board and currently serves as President. Of great interest to the AAGFO is Clydes' involvement with the material we know as E-Z Gold. Historically, we know it exists as a development by Lloyd Baum. Now in the past two years, Clyde has inherited total responsibility of the manufacturing and marketing of E-Z gold.

For the most part, the forgoing scenario was accomplished while living a solo life style. However, in 2010, a friendship of many years ago was rekindled with Kirsten, otherwise known as Kirsty, being the major player. A marriage took place on Dec. 26, 2010. Clyde continues to lead a very active life at Loma Linda University. This includes current dental academics and support of research, which involves preservation of research activities provided by former mentors. In addition, his lifestyle includes various acts of significant philanthropy. At most the recent meeting of this Academy, Clyde has been a clinician or provided photographic results of the clinical activity.

Clyde Roggenkamp

For this and reasons beyond, it is our pleasure to present on behalf of the Academy of Gold Foil Operators, the award for 2012 Clinician of the Year.

Online Only

On occasion we receive manuscripts that we would like to publish, but do not have the page room to include in the print journal. Some of these articles are accepted for publication "online only". These article's clinical relevance statements will appear printed in this section. For the full article, please go to www.jopdentonline.org or enter the provided address into your address bar.

Pullout Bond Strength of Fiber Posts Luted to Different Depths and Submitted to Artificial Aging

VC Macedo • NAY Souza • AL Faria e Silva • C Cotes • C da Silva • M Martinelli • ET Kimpara

Clinical Relevance:

Increased depth of luting tended to improve the fiber post retention. The bond strength of the self-adhesive resin cement was less affected by aging than the conventional resin cement.

http://dx.doi.org/10.2341/12-321-L

Overview of Clinical Alternatives to Minimize the Degradation of the Resin-dentin Bonds

A Reis • M Carrilho • L Breschi • AD Loguercio

Clinical Relevance:

The use of several clinical approaches to improve resin impregnation and the strength of polymer formed by the adhesives as well as to reduce the activation of host-derived proteases can improve the longevity of the resin-dentin bonds.

http://dx.doi.org/10.2341/12-258-LIT

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