# OPERATIVE DENTISTRY

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Academy of Operative Dentistry • American Academy of Gold Foil Operators • Academy of R.V. Tucker Study Clubs

# OPERATIVE DENTISTRY

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Operative Dentistry
Indiana University School of Dentistry, Room S411
1121 West Michigan Street, Indianapolis, IN 46202-5186
Phone 317-278-4800, Fax: 317-278-4900
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## Buonocore Memorial Lecture

# How to Bridge Research Results to Everyday Clinical Care?

*Michael Buonocore*

VV Gordan



### Clinical Relevance

Partnering with health providers on studies that address everyday clinical research questions through practice-based research is a potential solution to speed up the translation of research findings.


## SUMMARY

**Laboratory and clinical studies are essential to the advancement of sciences. However, a significant gap exists between the research findings and clinical practice. Therefore, research findings can be of little importance if their outcome cannot be directly or indirectly applied to everyday clinical care or readily translated. This paper focuses on how we can shorten the gap between the generation of new knowledge and their implementation into everyday clinical care. A new model is discussed where clinicians are the ones generating the research idea are paired with researchers. They collaborate on studies whose results are readily applicable to everyday practice. Partnering with health providers on studies that address everyday clinical research questions is a potential solution to speed up the translation of the research findings. Generating clinically applicable results can better improve the health of the public. Quoting Dr. Lawrence W. Green: "If we want more evidence-based practice, we need more practice-based evidence." This paper presents the practice-based research model as a solution to address this knowledge gap.**


*Valeria V. Gordan, DDS, MS, MSc, Department of Restorative Dental Sciences, University of Florida, Gainesville, FL, USA

*Corresponding author: PO Box 100415, Gainesville, FL 32610, USA; e-mail: vgordan@dental.ufl.edu

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## INTRODUCTION

It is an honor to be the recipient of the Buonocore Memorial Lecture and to once more celebrate his findings at the Academy of Operative Dentistry annual meeting. Dr Michael Buonocore believed in making a difference in patients' lives, challenged existing paradigms, and was a forward thinker who just over 50 years ago revolutionized how we think today about prevention and restoration.[1] With the concept of adhesive dentistry, he gave another dimension to how we restore teeth today, and as a

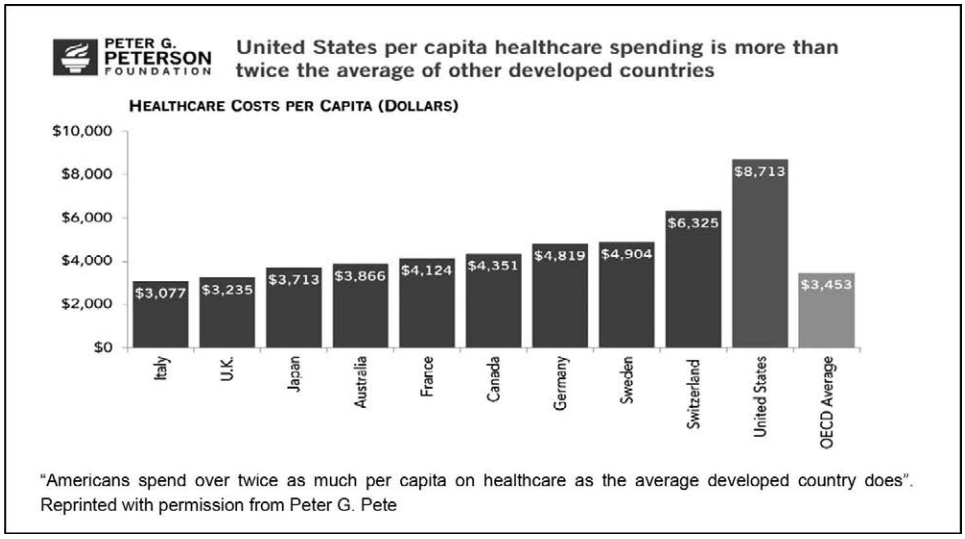


Figure 1. *Bar graph illustrating the per capita health care costs in the United States and several leading countries* for November 2015.

result, dentists can propose minimal intervention dentistry that ultimately benefits patient's health. That is where I would like to get started: on patient's health.

The cost of health care in the United States has been consistently higher than other developed countries, and it has dramatically increased in the last decade.[2] The Peter G. Peterson Foundation is an American foundation established in 2008 by Peter G. Peterson, former US Secretary of Commerce. It works to find fiscal solutions to help secure the country's economic growth. According to government projections, health care expenditures are projected to climb to 22% by 2039. Americans currently pay about twice as much per capita on health care as other leading nations. The annual cost per capita has remained high, approaching $9000 for the last four years (Figure 1). Therefore, one would expect that because the United States spends significant funds on health care that the life expectancy would be higher. Unfortunately, that is not the case, and the life expectancy in the United States is still below several leading countries.[3]

Significant time and money has been spent in biomedical research in the United States[4] and worldwide. We have accumulated a significant number of clinical and *in vitro* study results. Despite the immense amount of new knowledge, health in the United States is still below our expectation and below several industrialized countries. Several reasons need to be considered; however, we can focus on the following three. 1) A significant delay exists between the generation of new knowledge and its application into the medical/dental practicing community where it is delivered to patients. It takes an average of between 17 and 24 years to translate study findings to routine clinical practice.[5] 2) Too often, the study results are not immediately applied to everyday clinical care (ie, the results cannot be applied to benefit the patient's health).[5-7] 3) There is insufficient research that is evidence based on clinical practice for clinicians to make the correct choice during their decision-making process.[8] It is estimated that as little as 8% of clinical practice is based on peer-reviewed and critically appraised evidence.[9,10]

One example that has been my research interest is the treatment of defective restorations. It is one of the most frequent problems encountered by general practitioners today and accounts for more than 50% of all the treatment performed in general dental practice.[11-13] Several *in vitro* and clinical studies have shown that removal of the existing restoration will significantly reduce sound tooth structure, resulting in subsequently larger dental restorations.[14-17] Additionally, the removal of existing restorations may cause further trauma on the tooth with possible dentinal/pulpal response to thermal, chemical, or mechanical stimuli, depending on the size and depth of the existing restored site.[18-20] The consequence of replacing existing restorations could alter the outcome of the tooth and result in additional cost and time of treatment. All of these issues have a negative effect on patients. Two separate groups of investigators[21-26] through clinical studies have concluded that the repair of defective restorations is a viable treatment option that increased the longevity of the original restoration. Despite the results of the clinical studies involving the repair of restorations and several schools

including the teaching of repair of restorations in their curriculum,[27-30] most clinicians still do not routinely consider the repair or sealing of defective restorations as a viable treatment option,[31-34] and patients who may be eligible for repair of restorations are not offered this alternative treatment. One study published by the Dental Practice-Based Research Network (PBRN) in the United States involving close to 10,000 restorations (9875 restorations in 7502 patients) concluded that 75% of clinicians chose replacement over repair of defective restorations.[35-37] We also learned that dentists' decisions and bias will actually affect the restoration longevity.[35] A survival analysis of posterior restorations using an insurance claims database concludes that patients who change dentists are far more likely to have restorations replaced than if they do not.[38] An interesting finding is that most patients accept the repair of defective restorations. In another practice-based study involving close to 10,000 restorations and 200 clinicians, we assessed the behavioral aspect of patient satisfaction and found out that overall patient satisfaction was higher when the defective restoration was repaired compared with replaced.[39,40] Another study by the Network assessed the outcome of almost 6000 restorations that had been repaired vs replaced after 12 months by 195 dentists. The results showed that repaired restorations were less likely to need an aggressive treatment than restorations that had been replaced. Overall, the failure rate was low (n=378 [–6%]). When the restoration required additional treatment after the one-year follow-up, it was less likely to need a replacement, a root canal treatment, or an extraction if the restoration had been repaired (74%) rather than if it had been replaced (85%). In other words, although some repaired restorations failed, the failure was not catastrophic, it was a "friendly failure": a failure that could be repaired.[37]

## PARADIGM SHIFT

Despite all the efforts to study the treatment of defective restorations and ways to improve the longevity of the tooth and existing restoration through clinical studies, clinicians still do not routinely consider the repair of defective restorations as a viable treatment option. Therefore, a knowledge gap exists between the generation of new knowledge and its application to routine clinical care. How do we bridge the gap between research and clinical practice? How do we make sure that the topics being researched are of interest or will benefit the majority of the public at large? How do we make sure that once research findings become available that they will actually be implemented in dental and medical practices? These questions must be addressed if we hope to improve health while reducing costs.

Taxpayers and the public are very interested in immediate benefits from research investment.[41] Search engines have been created an easy way for the public to access the results generated from federally funded research (eg, http://www.ncbi.nlm.nih.gov/pubmed; http://www.nidcr.nih.gov/oralhealth/; http://www.webmd.com/; http://www.healthline.com/; and http://www.nidcr.nih.gov/research/ResearchResults/NewsReleases/). The committee for economic development concluded that "increased public access accelerates progress in science by speeding up and broadening diffusion of knowledge" and "increased public-access policies should be judged by their impact on the society and the development of high-quality scientific research." This opportunity for patients to access new results may lead patients to choose providers who rapidly implement research findings. A way to speed up the implementation of the research findings into clinical practice is to involve practitioners in the research process. That opportunity now exists with the creation of practice-based research (PBR). The commitment from the National Institute of Health (NIH) to fund PBR consolidates and attests where research efforts are headed (http://www.nidcr.nih.gov/research/ResearchResults/NewsReleases/CurrentNewsReleases/NDPBRN.htm and https://www.dentistry.ucla.edu/events/research-symposium-0).[42-44]

PBR is done by a teamwork approach: an effort in which clinicians and investigators work together to address clinical research questions that will ultimately benefit patient's health.[45] Dental practice-based research is conducted by dentists who are affiliated to investigate research questions and to share experiences and expertise. The dentists provide dental care to the public and are affiliated with an academic center that serves as the administrative base. The research is done by practitioners in and about the "real world" of dental practice, where the majority of the population receives its dental care. Practice-based research is not a new concept. It was introduced by the medical field back in 1970s. In 2012, AHRQ (Agency for Healthcare Research and Quality), US Department of Health & Human Services, identified more than 150 primary care PBRNs operating across the United States with more than 55,000 clinicians in more than 17,000

locations, serving approximately 46 million patients. Today, there are more than 170 networks registered at the AHRQ website.[43]

A handicap in most *in vitro* and some clinical studies is the translation of the research findings to everyday clinical practice. One limiting factor of traditional institutional-based clinical study is the lack of generalizability and external validity.[46] The result findings may not be readily applicable to everyday patients. Even if the findings are applicable to the everyday patient care, it takes time to translate the research results to everyday clinical practice. PBR addresses these obstacles in two ways: 1) it generates evidence-based knowledge with good external validity (the results apply to populations involved in the study (ie, the evidence comes directly from the end user, "the everyday patient"); and 2) PBR speeds up the adoption of the research findings by dentists who participated in the study. Passive absorption of knowledge usually does not work or works slowly.[5] In PBR, clinicians are involved in the entire research process from its inception: asking the clinical questions, gathering the research findings, and being involved in its dissemination. As the practitioner is involved in the research process, it is more likely that he or she will implement the research findings into their routine delivery of clinical care.

Although a well-conducted randomized clinical trial (RCT) is typically the most scientifically rigorous design for clinical studies, it is not always the best design to help move scientific evidence promptly into routine clinical practice. A key advantage of most PBRN studies is that they intentionally do not use highly selected samples, but instead enroll consecutive patients for whom certain treatment options would be appropriate. In that manner, they maximize the generalizability of conclusions made about treatment effectiveness. They also allow for an analysis of the process of care, such as determining which patients are offered treatment by clinicians and which patients choose to accept it, a possibility precluded in a RCT design.

The benefit of PBR supersedes the notion of access to large number of patients. Besides the diversity of patient population, it is in PBR where "effectiveness can be measured, where new clinical questions arise, and where readiness to change and adopt new treatments can be studied and addressed."[45] It is also "where the interface between patients and their physicians can be explored and medical care improved."[45] Two main points are critical for the success of PBR: 1) it needs to address questions that practicing clinicians judge to be important with the potential the results could improve clinical practice; and 2) the research must be feasible in most busy clinical practices.

What drives clinicians to participate in PBR? According to multiple testimonies over the 11 years of the existence of the Dental PBRN in the United States, participants reported that they 1) desire interaction with other colleagues in the dental field; 2) want to belong to a community or entity, 3) have a desire to give back to the profession, and 4) want to know the answers to everyday clinical questions.[47] Participants seek evidence-based answers to clinical questions and do not want to rely on biased opinions. Most information that is directed to clinicians is manufacturer driven, and bias is a major concern. The desire to be a part of a community that values answers informed by high-quality research drives most clinicians to join PBR.

Because networking with colleagues is important to practitioners, it is important that PBR allow an environment that fosters these interactions. One strategy is annual or semiannual meetings in which interaction with fellow practitioners is promoted. In those meetings, time is set aside for discussion among colleagues about the research results, including how to best implement the results into practice.

We discovered at the end of one of our interactive meetings that a significant number of practitioners actually changed how they practice as a result of the interactive discussion with their fellow clinicians. The improvement was toward using more prevention to treat dental caries and delaying the surgical treatment process in certain instances, according to the latest evidence-based research results.[48,49] We learned that a highly interactive meeting with fellow practitioner-investigators could be an effective mean to apply scientific findings into clinical practice, as clinicians reported that they would change how they treat patients as a result of being engaged in the scientific process. This "change in intention" is consistent with the health change theory, which suggests that this step is a prelude to the subsequent next step, which is the actual implementation of change in the practice.[50,51]

Another benefit to the clinician's career of participating in a PBR network is that it provides clinicians an opportunity to present the research findings at national and international meetings. This is a benefit not only to the clinicians, but also for the research community in which the paper is being presented, as researchers and academicians

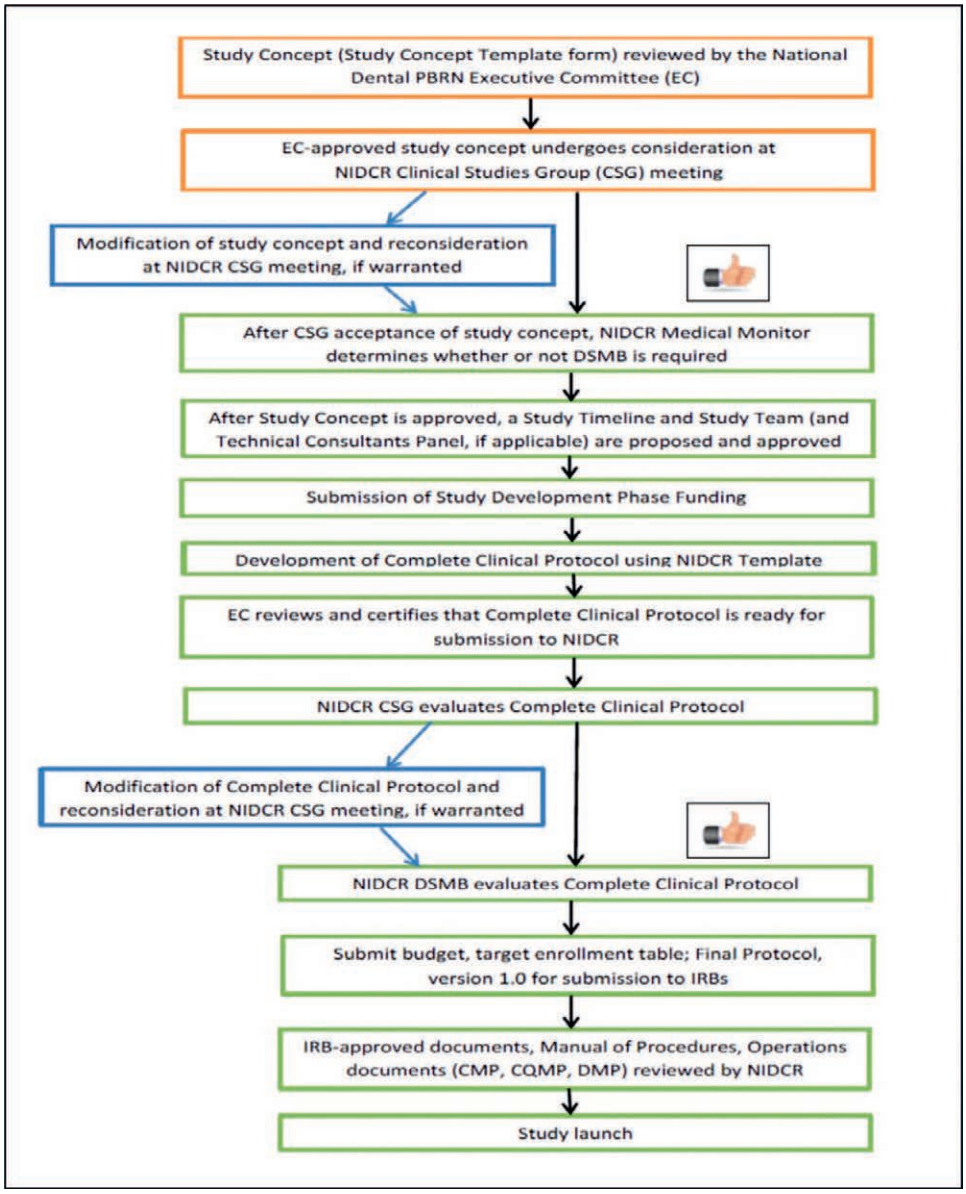


Figure 2. *Example of a diagram illustrating the steps from study concept to study launch by the National Dental PBRN funded by the NIH-NIDCR (http://www.nationaldentalpbrn.org/study%20development.php).*

get to hear from a clinician working in everyday practice about their experiences. PBR promotes interaction of researchers and academicians with their fellow practitioners and another opportunity to be a part of the research and educational processes.

One of the biggest challenges of practice-based research is to coordinate all the parts involved in the research process. We have learned that a lot can be accomplished when we have an organized teamwork approach. The academic institution provides a framework for the development of practice-based research, creating opportunities and resources for all those involved to be a part of the research process, but this would be meaningless if it did not have the involvement of the teams in the offices as the gathering of the research data takes place by the clinician and the dental team.

There are various steps involved in the PBR research process, from idea generation by the clinician (through the development of the study concept) all the way to the study launching (Figure 2). After a study concept is approved by both the executive committee and National Institute of Dental and Craniofacial Research (NIDCR) Clinical Studies Group, a study team is formed composed of statistician, one or more research investigators, private practitioner(s), research coordinator, a data manager staff, and a principal investigator. The study team will then develop and submit a complete clinical protocol using the NIDCR clinical study

protocol template.[52] After the approval of the complete protocol, the Coordinating Center will support the study team in the development of all required study startup documents, such as the Operations documents, Statistical Analysis Plan, Manual of Procedures, and Case Report Forms, using the NIDCR Toolkit templates.[53] Once all the forms are completed and approved, the study is implemented in the offices that agreed to participate and that had completed the training requirement for participation in in-office research.

Throughout the 11 years of existence of the dental PBR in the United States, we learned that patients' attitudes toward participation in dental research and experience with the delivery of care were valuable. We generated valuable results in the studies that had patient participation, ie, not only the attrition rate on survey studies was low, but also the overall response was positive. One behavioral science study involving 8000 patients asked patients about their dental office experience and their satisfaction with the dental procedure received.[39,40] Because we wanted to make sure that the anesthesia had worn off when patients responded the questionnaire (so that the report would be most unbiased), patients had to respond no sooner than 24 hours, which meant that they would have left the office when they responded. The research group had some concern if patients would remember to respond to the questionnaire and mail it accordingly 24 hours later. We were pleasantly surprised with a 78% patient response rate. According to dentists' reports, patients enjoyed being a part of the research process and appreciated the fact that the dental office was involved in research. Although there was some compensation for patients to participate (a $10 gift card), some patients returned the card back to the research as they felt it was "their responsibility to contribute to science."

We also learned that it is fundamental for clinicians to be a part of the dissemination of the research findings. In fact, we learned that clinicians may respond more positively to findings presented by other clinicians rather than by academic researchers. Therefore, close to 70% of publications and presentations from the National Dental Network include at least one full-time practicing clinician as a coauthor.[54,55] The roles of the clinicians have ranged from presenters, to coauthors, to lead authors. It is our impression that when a clinician working in the field presents the data, there seems to have a more positive interaction between the presenter and the audience. There is a higher sense of ownership and experience that is shared as opposed to a researcher who may understand the clinician's experiences, but is not participating in the daily routine of the dental office.

Another approach to communicate with clinicians about research findings, particularly those who participate in studies is to summarize their individual study results and to compare them with other practitioners working in their region and network-wide (ie, nationally). The careful analysis of their individual study results creates opportunities for them to reflect on their decision-making process and quality of care and if applicable consider a change in their practice pattern. Therefore, the Network will provide a summary of the research findings to those participating in the research process. The findings have bar graphs and/or tables illustrating the research results and a sentence summarizing it.

PBR is an excellent venue to foster international collaboration. Besides the exchange of information among clinicians and researchers, it explores the unique aspect that diversity of patient population and culture brings to the scientific process and discovery. The National Dental Network in the Unites States (http://www.nationaldentalpbrn.org/) favors global collaboration. At the request of the NIDCR Director, Dr Martha Somerman, the International Association of Dental Research Network workshop in Boston in 2015 included global/international collaboration as a discussion topic. The symposium had 20 guests from various countries with representation from most continents on the globe. Some discussion has already been initiated among some countries and ongoing work anticipates a fruitful collaboration.[56-60]

## FUTURE PLANS AND CONCLUSIONS

In conclusion, what does the future hold for clinicians and researchers? Three important key points to consider before formulating new research ideas: 1) research approaches and methods must be timely, relevant, nontraditional, and practical[61]; 2) traditional federally funded or corporate-funded research in academic institutions has significant value that can complement the studies that are conducted in PBRNs, but they must be innovative and readily applicable to survive in today's research climate; and 3) engaging clinicians in the research process will increase the potential for research that is relevant to daily practice and it will speed up the translation and dissemination of research findings that is fundamental to advancing population health. PBRNs can be an effective avenue for translation of

research findings as participants serve as change agents.

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#### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the University of Florida IRB 01. The approval code for this study is 161-2005.

#### Conflict of Interest

The author of this manuscript certifies that she has 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 Technique/Case Report

# Clinical/Photographic/Scanning Electron Microscopy Analysis of Pit and Fissure Sealants After 22 Years: A Case Series

D Sundfeld • LS Machado • LM Franco • FM Salomão
NIP Pini • MLMM Sundefeld • CS Pfeifer • RH Sundfeld

### Clinical Relevance

Resin-modified glass ionomer cements and polyacid-modified resin composite had similar clinical performance as pit and fissure sealants and successfully prevented dental caries lesions after 22 years.

### SUMMARY

**Pit and fissure sealant is a clinical technique adopted to prevent caries lesion development. Ionomeric and/or resin-based materials are commonly used for this purpose. This article presents a case series of sealed teeth with 22-year follow-up evaluated by clinical, photographic, and microscopic analysis. In 1992, sixteen patients (9-14 years of age) had at least three teeth sealed with one of the following materials: resin-modified glass ionomer cement (RMGIC, Vitrebond or Fuji II LC) or polyacid-modified resin composite (PMRC, VariGlass VLC), totaling 86 sealed permanent teeth. After 22 years, 10 patients were recalled, representing 41 teeth. The retention of sealants was assessed by three methods: clinical analysis by visual inspection; photography;**

Daniel Sundfeld, DDS, MDS, PhD student, Department of Restorative Dentistry, Piracicaba Dental School, University of Campinas, Piracicaba, Brazil

Lucas Silveira Machado, DDS, MDS, PhD, professor, Department of Conservative Dentistry, School of Dentistry, Federal University of Rio Grande do Sul, Porto Alegre, Brazil

Laura Molinar Franco, DDS, MDS, PhD student, Department of Restorative Dentistry, Araçatuba Dental School, State University of São Paulo, Araçatuba, Brazil

Fabio Martins Salomão, DDS, MDS, PhD student, Department of Restorative Dentistry, Araçatuba Dental School, State University of São Paulo, Araçatuba, Brazil

Núbia Inocência Pavesi Pini, DDS, MDS, PhD, temporary professor, Department of Restorative Dentistry, Araçatuba Dental School, State University of São Paulo, Araçatuba, Brazil

Maria Lúcia Marçal Mazza Sundefeld, DDS, MDS, PhD, professor, Department of Pediatric and Community Dentistry, Araçatuba Dental School, State University of São Paulo, Araçatuba, Brazil

Carmem Silvia Pfeifer, DDS, PhD, professor, Department of Restorative Dentistry, Division of Biomaterials and Biomechanics, Oregon Health and Science University, Portland, OR, USA

*Renato Herman Sundfeld, DDS, MDS, PhD, professor, Department of Restorative Dentistry, Araçatuba Dental School, State University of São Paulo, Araçatuba, Brazil

*Corresponding author: 1193 José Bonifácio St, Araçatuba, SP, Brazil; e-mail: sundfeld@foa.unesp.br

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**and scanning electron microscope (SEM) images and classified as retained (pits and fissures filled by sealant material); partially retained (pits and fissures partially filled by sealant material); or totally lost (no material was found in pits and fissures). The SEM images provided a higher number of retained sealants when compared with the clinical and photographic evaluations. Also, no totally lost scores were found with SEM analysis, regardless of the sealing material. No caries lesions were found. A fully or partially retained sealant in pits and fissures was capable of preventing caries lesions after 22 years within the patient pool analyzed.**

## INTRODUCTION

Pit and fissure sealants were introduced in the 1960s as a clinical technique for preventing dental caries,[1,2] the most prevalent oral disease among children.[3] Sealants act as a physical barrier that prevents food/biofilm from accumulating in pits and fissures and therefore prevents the growth of bacteria that can lead to dental decay.[4] Since the first clinical report[5] on the application of pit and fissure sealants by Cueto and Buonocore in 1967, this extraordinary preventive technique has been applied all over the world, and hundreds of reports have demonstrated its effectiveness in hindering dental caries lesion development.

The most susceptible site for developing a caries lesion is the occlusal surface—in about 67%-90% of children 5-17 years old[3,6]—due to the shape, depth, and narrowness of fissures.[7,8] This anatomical configuration increases the difficulty of self-cleaning by the food bolus, tongue, cheeks, and lips while also making cleaning by other methods difficult.[7,8] Although dental caries is a preventable disease,[9] some populations all over the world still experience a high incidence,[10,11] in contrast to others where the prevalence of caries lesions is declining.[11,12] However, because oral hygiene is highly behavior-dependent, dental caries is still a problem, even in populations where its prevalence is low.[4]

Three main types of materials are available as pit and fissure sealants: glass ionomer cements (GICs), polyacid-modified resin composite (PMRCs), and resins.[2] GICs can chemically bond to calcified tissues and are able to release and take up fluoride.[13,14] Due to the properties of GICs, many clinical research studies have applied them as pit and fissure sealants but with poor retention rates through the years,[15,16] which has been explained by their inherent brittle characteristics, with low mechanical strength and wear resistance.[17-19] To overcome this condition, resin-modified glass ionomer cements (RMGICs) and PMRCs were developed in the late 1980s, combining components of GICs and composite resins,[2,20] in order to provide better esthetic properties, less sensitivity to the application technique, and a decrease in imbibition and synerisis.[21] On the basis of previous findings,[15,16] lower retention rates have been reported after clinical evaluation of RMGICs. However, these studies showed clinical analyses of no longer than one year. PMRCs, also known as "compomers," have been reported to perform as well as composites,[22] with even better resistance to wear than composite resin[23] but still with controversial retention rates for fissure sealants.[20,21,24]

Since the 1980s, the ability of pit and fissure sealants to protect pits and fissures has been assessed using sealant retention as the relevant end point, which also provides a quantification of the longevity of various sealing materials.[25] A well-retained material in the pits and fissures exercises its preventive effect for much longer than any other intervention.[26] The retention can be evaluated by clinical, digital photographic, replica, and microscopic analysis.[8,27]

Little scientific literature data are available regarding the use of compomers as pit and fissure sealants.[2] Thus, the aim of this case series study is to report the retention rate and characteristics of pit and fissure sealants performed with two RMGICs and one PMRC after 22 years of clinical use.

## CASE SERIES

The patients had their teeth sealed in 1992 at the Araçatuba Dental School–São Paulo State University (UNESP), Brazil. Sixteen patients (9-14 years old; eight girls, eight boys) participated and had 86 permanent posterior teeth sealed (upper and lower molars and premolars)——on average, five to six teeth were sealed in each patient. All patients were given an initial clinical evaluation using visual inspection with adequate light, a dental mirror/probe, and bitewing radiographs to assess the presence or absence of caries lesions. The patients received oral hygiene instructions regarding tooth brushing and routine flossing. The general population of 9- to 14-year-olds in Araçatuba was previously deemed as being "moderate to high caries-risk" according to the 1994 decayed-missing-filled index. Fluoride was available in the city's water supply. The patients presented at least three sound, unsealed permanent premolars and molars. Teeth with

Table 1: *Composition of the Materials Used in This Study*

| Commercial Sealant | Clinical Indication | Composition | Powder to Liquid Ratio |
|---|---|---|---|
| Vitrebond (3M ESPE) Resin-modified glass ionomer | Liner/base | Powder: fluoroaluminosilicate glass. Liquid: copolymer of acrylic and maleic acid; HEMA; photoactivator, water. | 1:1 |
| Fuji II LC (GC) Resin-modified glass ionomer | Restorative material | Powder: fluoroaluminosilicate glass. Liquid: aqueous solution of polycarboxylic acid, TEGDMA, and HEMA. | 1:2 |
| VariGlass VLC (Dentsply) Polyacid-modified resin composite | Restorative material | Powder: fluoroaluminosilicate glass. Liquid: polyacrylic acid, HEMA, TEGDMA, 2,6-Di-tert-butyl-4-methylphenol. | 1:2 |

*Abbreviations: HEMA, 2-hydroxyethylmethacrylate; TEGDMA, triethyleneglycol dimethylmethacrylate.*

active decay or previously placed restorations were not included in the study. All the procedures were explained to the parents, who gave consent for the study.

### Clinical Procedures

One operator (last author) performed all of the occlusal sealing procedures. First, all teeth subjected to the sealing procedures were isolated with a rubber dam and were cleaned with pumice and water. Then, acid etching using 37% phosphoric acid was performed for one minute on the entire occlusal surface, under vibration, inside the pits and fissures with a tapered-end explorer probe. After washing and drying, one of the following sealing materials was applied: RMGIC (Vitrebond, 3M ESPE, St Paul, MN, USA); RMGIC (Fuji II LC, GC, Tokyo, Japan); PMRC (VariGlass VLC, Dentsply, Milford, DE, USA) (Table 1). Each patient had at least one type of each sealant material. The materials were hand-mixed and inserted into pits and fissures using a No. 5 explorer probe, also under constant vibration. Each material was light-cured for 40 seconds using a halogen light-curing device (Fibralux, Dabi Atlante, Ribeirão Preto, Brazil). Occlusal contacts were checked and adjusted when necessary using fine-tapered diamond finishing burs. No sealants required replacement.

### Clinical, Photographic, and SEM Analysis

After 22 years, 10 patients attended the recall appointment (five women, five men), representing 50 teeth subjected to the present analysis. Four sealed teeth were extracted due to orthodontic reasons, and five were restored: three due to interproximal caries and two to be used as retainers for fixed partial dentures. Therefore, 41 teeth (18 lower premolars, 11 upper premolars, seven lower molars, and five upper molars) were evaluated for sealant retention through clinical analysis, photography, and SEM images.

Sealed teeth were evaluated by three methods in order to assess the sealant retention: 1) clinical analysis: visual assessment using adequate light, drying the occlusal surface and dental mirror; 2) photographic images: sealed teeth were recorded using a digital photographic camera (Nikon D 300 Digital Camera–Macro Lens 105 mm f/2.8G, Nikon Corp, Japan); and 3) SEM images: impressions of the sealed teeth were made using a silicone impression material (Express XT Putty and Light body, 3M ESPE). Then, epoxy resin (Epo-Thin Resin, Buehler Lake Bluff, IL, USA) was used to cast the impressions and produce replicas. The replicas were positioned on aluminum stubs and sputter-coated with gold and subjected to SEM analysis (JSM 5600LV, JEOL, Tokyo, Japan) operated under 15 kV. Both photographic and SEM images were shown on an LED screen that had a resolution of $1366 \times 768$ pixels. The clinical analysis and photographic/SEM images were assessed by two double-blind authors who were previously calibrated and unaware of the type of sealant material used (only the last author knew which material was being evaluated). Intraexaminer reproducibility was assessed by the Cohen $\kappa$ test with a reliability of 81% for clinical analysis, 96% for photographic images, and 100% for SEM images.

The criteria adopted to rank the three methods of evaluation were 1) retained (R): pits and fissures filled by sealant; 2) partially retained (PR): pits and fissures partially filled by sealant; and 3) totally lost (T): no sealant material was found in pits and fissures.

### Clinical, Photographic, and SEM Analysis Results

The retention rate according to the clinical analysis and photographic/SEM images is represented in

Table 2: *Retention Rate (% in Parentheses) According to the Evaluation Method and Sealant Material After 22 Years*

| Sealant Materials | Clinical Analysis | | | Photographic Images | | | SEM Images | | |
|---|---|---|---|---|---|---|---|---|---|
| | R (%) | PR (%) | T (%) | R (%) | PR (%) | T (%) | R (%) | PR (%) | T |
| Vitrebond | 0 | 5 (38.5) | 8 (61.5) | 1 (7.7) | 6 (46.15) | 6 (46.2) | 10 (76.9) | 3 (23) | 0 |
| Fuji II LC | 1 (7.14) | 9 (64.3) | 4 (28.6) | 2 (14.3) | 7 (50) | 5 (35.7) | 11 (78.6) | 3 (21.5) | 0 |
| VariGlass VLC | 0 | 11 (78.6) | 3 (21.4) | 3 (21.4) | 9 (64.3) | 2 (14.3) | 10 (71.4) | 4 (28.5) | 0 |

*Abbreviations: R, retained; PR, partially retained; T, totally lost.*

Table 2. Fuji II LC and VariGlass VLC had similar retention behavior when clinical and photographic analysis was compared; however, the SEM analysis showed the same clinical behavior for all three sealing materials. The SEM images provided a higher number of retained sealants when compared with the clinical and photographic evaluations (Figure 1), regardless of the sealing material. Also, no "totally lost" scores were found when using SEM analysis.

Figures 2-7 represent clinical cases in which sealed teeth were photographed and subjected to SEM analysis. It was not possible to recover all initial photographic images; however, retained images between 1992 and 2014 are presented as representative. No caries lesions were found on sealed teeth.

## DISCUSSION

In 1992, when the present study began, the materials used were not primarily indicated to be pit and fissure sealant materials. Therefore, in an effort to find available ionomeric materials with better retention and mechanical properties, VariGlass VLC, Fuji II LC, and Vitrebond were chosen. In general, the three materials underwent chemical/physical degradation after 22 years of analysis.

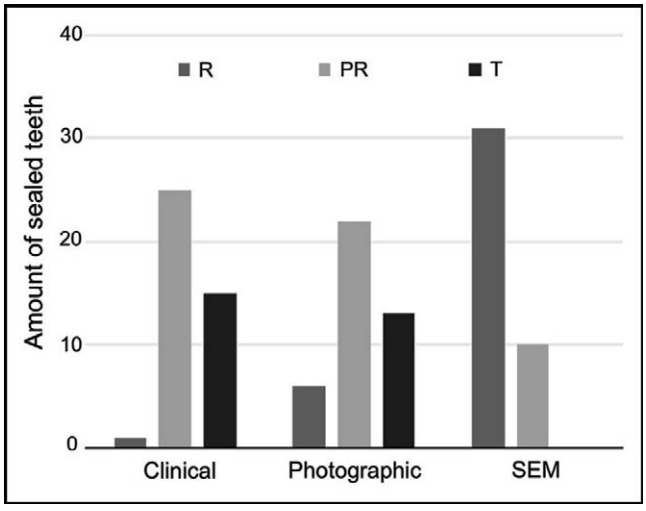


Figure 1. *Comparison between methods of evaluation and retention rate scores of sealants after 22 years. R, retained; RP, partially retained; T, totally lost.*

The three methods applied in this study successfully accomplished the objective of evaluating sealant retention. Clinical analysis resulted in predominantly partially retained scores, considerable totally lost scores, and low retained scores due to the difficulty of the naked eye to properly distinguish the anatomic form of sealants in deeper parts of pits and fissures. With photographic image analysis, a slight decrease in partially retained and totally lost and an increase in retained was found when compared with clinical analysis because it was possible to observe the images on an LED screen with higher magnification and better definition. Although the most common method for evaluating the clinical performance of a pit and fissure sealant is by visual clinical examination, the assessment of sealant retention from photographic images has a higher reproducibility[27] and enables retrospective analysis of the effectiveness in everyday dental practice.[8,28] However, it was not possible to retrieve all baseline images; therefore, precise comparison of the baseline (right after sealants were applied) with the present analysis was not possible.

SEM is considered a reference standard of sealant retention[27] because clinical analysis and photographic images do not present the same magnification and definition as SEM. This difference was quite evident, given that SEM analysis presented elevated retained scores and eliminated the totally lost scores for pit and fissure sealants, given that remnants of sealant material were clearly observed at deeper regions that were scored as totally lost when using clinical and photographic analyses (Figure 6).[26,27] Although some reports have found low retention rates for compomers and resin-modified glass ionomers applied in pits and fissures,[20,21,24,29] the assessment methods used in those studies was visual clinical analysis, which could not properly describe the presence of the sealant material at deeper parts of pits and fissures.

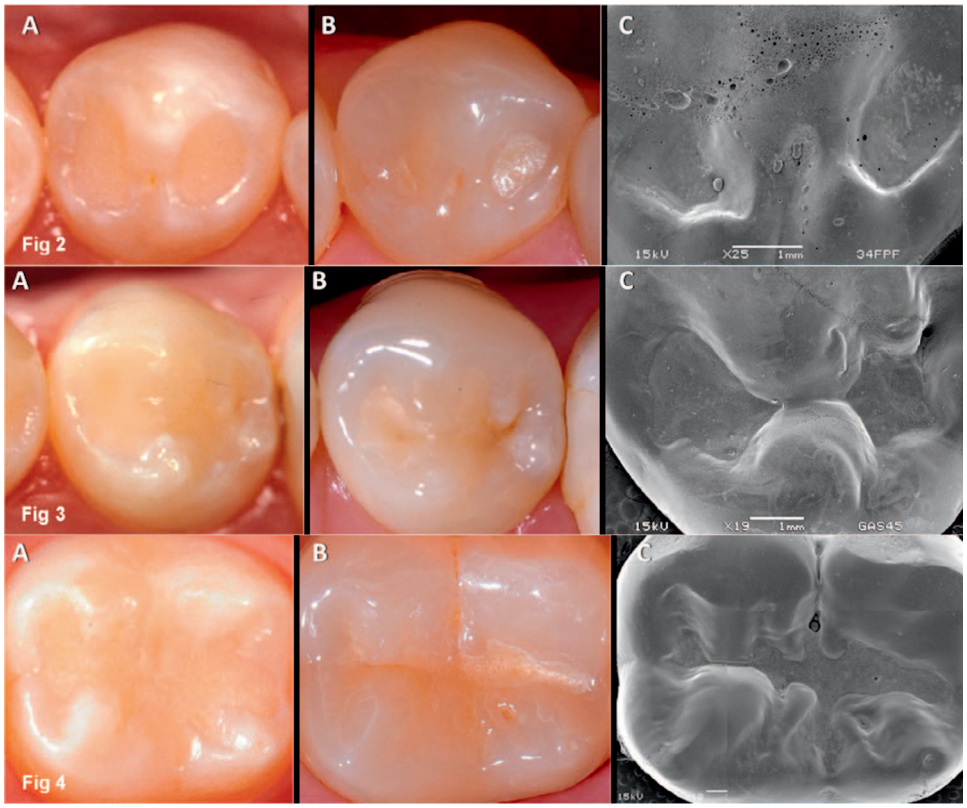


Figure 2. *(A): Photo six months after sealing with VariGlass VLC. (B): Photo after 22 years. (C): SEM showing the presence of sealing material. Evaluation scores: clinical analysis: partially retained; photographic image: retained; SEM image: retained.*

Figure 3. *(A): Photo two years after sealing with Fuji II LC. (B): Photo after 22 years. (C): SEM showing the presence of sealing material. Evaluation scores: clinical analysis: partially retained; photographic image: partially retained; SEM image: retained.*

Figure 4. *(A): Photo six months after sealing with VariGlass VLC. (B): Photo after 22 years. (C): SEM showing the presence of sealing material. Evaluation scores: clinical analysis: partially retained; photographic image: partially retained; SEM image: retained.*

Better wear resistance of the compomer was expected due to its improved mechanical properties over RMGICs[30] and because they contain no water and have components similar to composite resins.[31] However, compomers contain hydrophilic components that cause water to be drawn into the material following cure and inevitably lead to a decline in certain physical properties.[31] This might explain why VariGlass VLC presented a similar wear behavior when compared with Fuji II LC and Vitrebond. Even though the wear was not measured in the replicas at baseline and at 22 years, photographic evidence suggests that VariGlass presented clinically comparable wear patterns in relation to Fuji II LC and Vitrebond. Cehreli and others[32] reported marked occlusal wear for PMRCs, albeit with a good retention after three years. It is noteworthy that no resin material would present the same baseline shape/volume after 22 years of clinical performance because they all inevitably undergo physical/chemical degradation over time due to masticatory forces, articular accommodation, movements of eruption, and abrasion/erosion processes.

To achieve long clinical success with dental sealants, the maintenance of a satisfactory retention to enamel is primary.[33] In the case of teeth with partially retained sealants, the risk of developing caries lesions is higher than in a fully covered area[27] because food/biofilm may still accumulate in pits and fissures. However, the present case series report did not find any carious lesions on sealed teeth, regardless of their scores. Even if small portions of the sealant material are found in deeper parts of pits and fissures (Figures 5-7), their protective/preventive effects against caries lesions still goes on as a physical barrier, even as resin tags embedded in the etched enamel.[20] In 1992, the manufacturer did not recommend phosphoric acid etching before placing a resin-modified glass ionomer. However, the acid-etching procedure was adopted in the current study because it was hypothesized at that time that it would benefit the retention rate of the sealing material. Later reports have shown that etching with phosphoric acid improves the bond strength of sealing materials to enamel[34-36] by creating tags,[36] which may have greatly extended the retention and protection of pit and fissure materials against caries lesions. Horowitz and others[37] reported that teeth with partially retained sealants had lower incidences of caries lesions when compared with an unsealed group, confirming the positive effect of partially retained sealants.

Fluoride released from the tested materials may have played an important role as a protective/

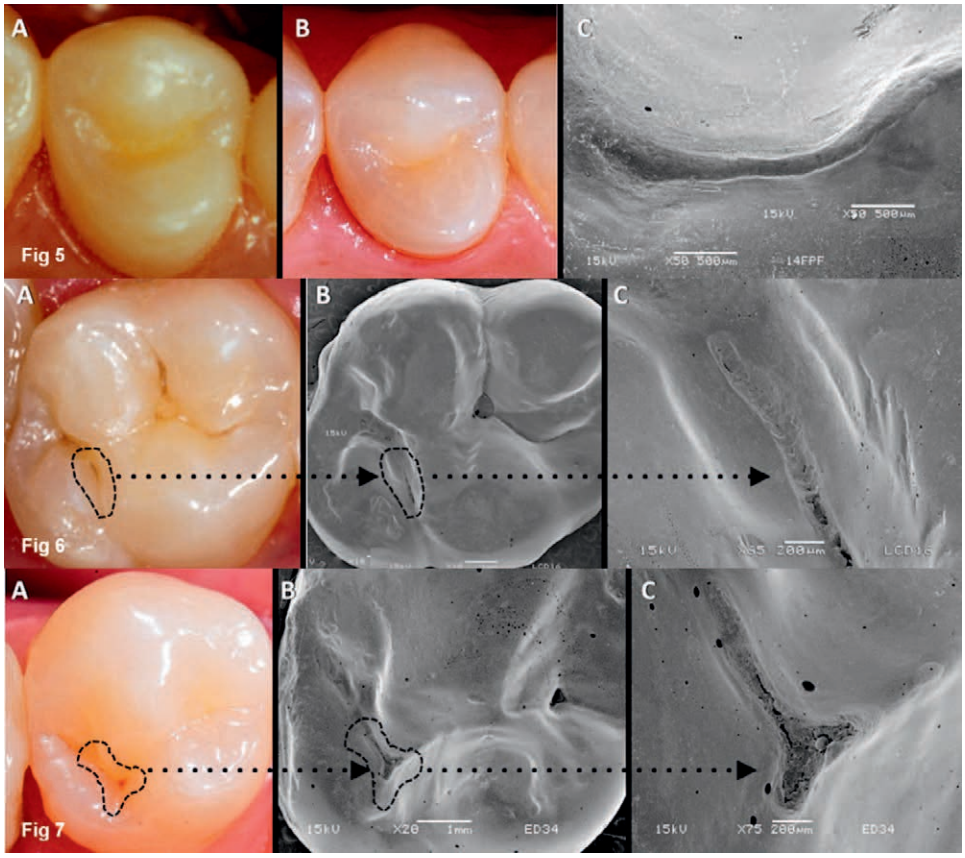


Figure 5. *(A): Photo five years after sealing with Vitrebond. (B): Photo after 22 years. (C): SEM showing the presence of sealing material. Evaluation scores: clinical analysis: totally lost; photographic image: retained; SEM image: retained.*
Figure 6. *(A): Photo 22 years after sealing with Vitrebond. (B): SEM showing all occlusal surfaces. (C): SEM showing the presence of sealing material in the deep part of pits. Evaluation scores: clinical analysis: totally lost; photographic image: totally lost; SEM image: retained.*
Figure 7. *(A): Photo 22 years after sealing with VariGlass VLC. (B): SEM showing all occlusal surface. (C): SEM showing the presence of sealing material in the deep part of pits. Evaluation scores: Clinical analysis: partially retained; photographic image: partially retained; SEM image: retained.*

preventive agent against caries lesions. Fluoride acts as a cariostatic agent, inhibiting demineralization and favoring remineralization.[38] Furthermore, it has been indicated that fluoride-releasing materials reduce the amount of enamel demineralization adjacent to the material.[39] However, polyacid-modified resin materials have no fluoride recharging capability[40] and release less fluoride when compared with conventional cements and RMGICs,[32,41-45] which may affect its caries preventive abilities.[41] It is not possible to state that fluoride is still being released from the sealing materials after 22 years for modified-glass ionomer materials that present a high initial fluoride release; on the contrary, a decrease during the aging period must have certainly occurred.[42]

Due to the multifactorial nature of caries lesions, it is challenging to determine the most effective manner of preventing tooth decay: sealants, proper dental hygiene, proper diet, fluoridated mouth rinses, supply water, toothpaste, or sealant. Definitely, when the four strategies are combined, the possibility of teeth to decay considerably decreases. Given that it is not possible to ensure that all people around the world are supplied with fluoridated water and/or toothpastes[46] and an anticariogenic diet may not be practiced by all levels of the population, sealants remain as a clinical technique that may greatly prevent tooth decay (Figure 8). One other important aspect that may have influenced the absence of caries lesions is that permanent enamel

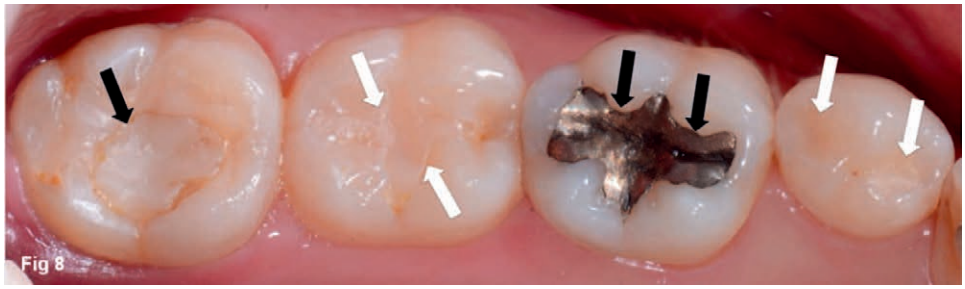


Figure 8. *Sealed No. 18 and No. 20 showing nondecayed teeth after 22 years (VariGlass VLC) (white arrows). In contrast, No. 19 was already restored when the sealing procedures were performed (black arrows). No. 17 was not sealed, and at the recall the tooth was restored, probably due to caries (black arrows).*

undergoes posteruptive maturation, accumulating fluoride, becoming harder, less porous, and less caries-prone[47] when compared with recently erupted teeth, which are known to be at higher risk for decay than old teeth.[48]

The present report corroborates previous research, with a minimum of 11 years of follow-up,[8,49-52] that sealed teeth had a reduction in caries lesions and restorations, associated with a long-lasting caries-prevention effect. There were no clinical failures of any sealant after 22 years, given that no occlusal restoration was performed and no cavitated caries lesions were found at any clinical examination.[53]

## CONCLUSIONS

All sealing materials suffered similar physical degradation after 22 years. Thus, the full retention of sealants is not of crucial concern because pit and fissure sealants showed remnants of material in deeper parts. Sealing pits and fissures continues to be a suitable and cost-effective clinical technique for preventing dental caries throughout life.

### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of Araçatuba School of Dentistry–UNESP, Brazil.

### Conflict of Interest

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

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# Clinical Effectiveness of Different Polishing Systems and Self-Etch Adhesives in Class V Composite Resin Restorations: Two-Year Randomized Controlled Clinical Trial

J-H Jang • H-Y Kim • S-M Shin • C-O Lee • DS Kim • K-K Choi • S-Y Kim

### Clinical Relevance

A multi-step polishing system provides more desirable clinical results compared to simplified abrasive-impregnated rubber instruments. One-step and two-step self-etch adhesives show clinically equivalent performance.


## SUMMARY

**The aim of this randomized controlled clinical trial was to compare the clinical effectiveness of different polishing systems and self-etch adhesives in class V composite resin restorations. A total of 164 noncarious cervical lesions (NCCLs) from 35 patients were randomly allocated to one of four experimental groups, each of which used a combination of polishing systems and adhesives. The two polishing systems used were Sof-Lex XT (Sof), a multi-step abrasive disc, and Enhance/Pogo (EP), a simplified abrasive-impregnated rubber instrument. The adhesive systems were Clearfil**



Ji-Hyun Jang, DDS, PhD, Department of Conservative Dentistry, Kyung Hee University Dental Hospital at Gangdong, Seoul, Korea

Hae-Young Kim, DDS, PhD, Department of Public Health Sciences, Graduate School & Department of Health Policy and Management, College of Health Science, Korea University, Seoul, Korea

Su-Mi Shin, DDS, MSD, Department of Conservative Dentistry, Graduate School, Kyung Hee University, Seoul, Korea

Chung-Ok Lee, DDS, Department of Conservative Dentistry, Graduate School, Kyung Hee University, Seoul, Korea

Duck Su Kim, DDS, PhD, Department of Conservative Dentistry, School of Dentistry, Kyung Hee University, Seoul Korea

Kyoung-Kyu Choi, DDS, PhD, Department of Conservative Dentistry, School of Dentistry, Kyung Hee University, Seoul Korea

*Sun-Young Kim, DDS, PhD, Department of Conservative Dentistry, School of Dentistry, Kyung Hee University, Seoul Korea

*Corresponding author: Department of Conservative Dentistry, School of Dentistry, Kyung Hee University, 1 Hoegi-dong, Dongdaemun-gu, Seoul, Korea, 130-701; e-mail: kimsunyoung@khu.ac.kr


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**SE bond (CS), a two-step self-etch adhesive, and Xeno V (XE), a one-step self-etch adhesive. All NCCLs were restored with light-cured microhybrid resin composites (Z250). Restorations were evaluated at baseline and at 6, 12, 18, and 24 months by two blinded independent examiners using modified FDI criteria. The Fisher exact test and generalized estimating equation analysis considering repeated measurements were performed to compare the outcomes between the polishing systems and adhesives. Three restorations were dislodged: two in CS/Sof and one in CS/EP. None of the restorations required any repair or retreatment except those showing retention loss. Sof was superior to EP with regard to surface luster, staining, and marginal adaptation ($p<0.05$). CS and XE did not show differences in any criteria ($p>0.05$). Sof is clinically superior to EP for polishing performance in class V composite resin restoration. XE demonstrates clinically equivalent bonding performance to CS.**

## INTRODUCTION

Despite tremendous improvements in the material properties of adhesives and composite resin over the past several decades, the procedures of adhesive restorations are still technique sensitive and need to be meticulously performed to achieve an ideal restoration. Of the many procedures involved in adhesive restoration, finishing and polishing require special care and attention to achieve the desired anatomical contour and lustrous surface. Roughly polished restorations can cause a number of clinical problems, including plaque retention, chronic gingival inflammation, marginal discoloration, and secondary caries.[1] Furthermore, finishing and polishing can influence the long-term clinical performance of composite resin restorations, affecting marginal integrity, wear, and the durability of adhesive restorations.[1-3] Thus, appropriate polishing is a critical clinical procedure to enhance both esthetics and longevity of composite resin restorations.

The various types of tools commercially available for polishing composite resin restorations include abrasive polishing pastes, fluted carbide burs, diamond burs, stones, abrasive discs, and abrasive-impregnated rubber cups, points, and wheels.[4] Abrasive discs have traditionally been used for planar surfaces, such as anterior tooth restorations and cervical restorations with composite resins. Typically, polishing with abrasive discs is composed of a four-step progression from coarse to superfine, following the traditional polishing procedures.[5] Recently, abrasive-impregnated rubber instruments have emerged on the dental market. The abrasive-impregnated rubber instruments have a distinct advantage in areas where disc-type polishing instruments do not work properly, such as on the occlusal surface of posterior teeth and on the lingual surface of anterior teeth. These instruments are easy to use because of their availability in various forms and also because they require fewer steps. Thus, since abrasive-impregnated instruments simplify the polishing process, clinicians may prefer to use them even on planar surfaces.[6]

Many studies have reported *in vitro* comparisons of polishing outcomes, such as surface roughness, gloss, marginal irregularities, and staining susceptibility between traditional multistep abrasive discs and abrasive-impregnated rubber instruments.[7-14] A number of these studies have demonstrated no significant differences between the two polishing systems.[7,8] However, contradictory results (ie, better performance of one or the other method, depending on the experimental design) have also been reported in several other studies.[9-14] Even though the *in vitro* studies have discussed the importance of performing clinical studies to confirm the clinical efficacy of polishing instruments, to the best of our knowledge, there have been no clinical trials to compare the efficacy of polishing methods on composite resin restorations. Since a clinical trial is considered to provide the most reliable evidence of the effectiveness of clinical materials and methods,[15] a clinical evaluation of the effectiveness of different polishing methods through a randomized controlled trial would be of great worth.

Noncarious cervical lesions (NCCLs) are considered an ideal model for evaluation of the clinical performance of adhesive restorations since NCCLs offer good access for operative procedures and evaluation, operator variability is reduced since there are relatively minimal restorative procedures, and the lesions themselves are widely available in multiple teeth, facilitating patient selection and study design.[12,15] Many clinical trials have compared the clinical effectiveness of adhesives, composites, and operative procedures in composite resin restorations of NCCLs.[16-19] Both multistep abrasive discs and simplified abrasive-impregnated rubber instruments are used for polishing the convex surface in composite restorations of NCCLs. It would be of great interest and significance to compare the effectiveness of these two popular polishing systems

Table 1: *Materials Used in This Study*

| Finishing/Polishing System | Type | Abrasive (Particle Size) |
|---|---|---|
| Sof-Lex XT (3M ESPE, St Paul, MN) | Dark orange (coarse) | Aluminum oxide (100 μm) |
| | Orange (medium) | Aluminum oxide (40 μm) |
| | Light orange (fine) | Aluminum oxide (24 μm) |
| | Yellow (superfine) | Aluminum oxide (8 μm) |
| Enhance/Pogo | Enhance | Aluminum oxide (40 μm) |
| (Dentsply Caulk, Milford, DE) | EP | Diamond micropolisher (10-15 μm) |
| **Adhesives** | **Chemical composition** | **Instruction for use** |
| Clearfil SE Bond<br>Two-step self-etch (Kuraray, Osaka, Japan) | Primer: MDP, HEMA, photoinitiator, water hydrophilic dimethacrylate<br>Bond: MDP, HEMA, Bis-GMA, hydrophobic dimethacrylate, colloidal silica, photoinitiators | 1. Dry surface.<br>2. Apply primer for 20 s.<br>3. Gentle air stream.<br>4. Apply adhesive.<br>5. Light cure for 10 s. |
| Xeno V<br>One-step self-etch (Dentsply Caulk) | Bifunctional acrylic amides, acrylamido alkylsulfonic acid, phosphoric acid ester, acrylic acid, water, tertiary butanol, butylated benzenediol, CQ, initiator, stabilizer | 1. Apple adhesive.<br>2. Gently agitate 20 s.<br>3. Air blow at least 5 s.<br>4. Light cure for 20 s. |
| **Composite resin** | **Chemical composition** | **Classification<br>filler wt%, filler size** |
| Filtek Z250 (3M ESPE) | Matrix: Bis-GMA,UDMA, Bis-EMA, TEGDMA<br>Filler: Zirconia, silica | Microhybrid<br>78 wt%, 0.01-3.5 μm |

*Abbreviations: MDP, methacryloyloxydecyl dihydrogen phosphate; HEMA, hydroxyethyl methacrylate; Bis-GMA, bisphenol A diglycidylether methacrylate; CQ, camphorquinone; UDMA, urethane dimethacrylate; BisEMA, bisphenol A polyethylene glycol diether dimethacrylate; TEGDMA, triethylene glycol dimethacrylate.*

in composite resin restorations of NCCLs. Therefore, the objective of this study was to compare the clinical effectiveness of abrasive disc–type and abrasive-impregnated rubber–type polishing systems in class V composite resin restorations. In addition, we have compared the clinical performance of a two-step self-etch adhesive and a one-step self-etch adhesive. Hence, composite resin restorations were placed in NCCLs using two different self-etch adhesives and then polished using either a multistep abrasive disc or a simplified abrasive-impregnated rubber instrument. The clinical performance of the restorations was then evaluated over 24 months using modified FDI criteria.

## METHODS AND MATERIALS

### Recruitment of Patients and Inclusion/ Exclusion Criteria

This study was a single-center prospective randomized controlled clinical trial. Thirty-five patients with at least two NCCLs who visited K-H University Dental Hospital (KHUDH) from September 2011 to February 2012 participated in this study. Their mean age was 55 years (ranged from 30 to 73 years). The participants were apparently healthy patients with good oral hygiene. Patients who had severe chronic periodontitis, rampant caries, xerostomia, or orthodontic appliances or who were pregnant or nursing were excluded. Each patient was informed of the study and signed a consent form.

### Operating Procedure

Four experimental groups combining two polishing systems and two adhesives were compared. The polishing systems used in this study were Sof-Lex XT (Sof; 3M ESPE, St Paul, MN) for the multistep abrasive disc and Enhance/Pogo (EP; Dentsply Caulk, Milford, DE) for the simplified abrasive-impregnated rubber instrument. The adhesives used were Clearfil SE bond (CS; Kuraray, Osaka, Japan) for the two-step self-etch adhesive and Xeno V (XE; Dentsply, DeTrey, Germany) for the one-step self-etch adhesive (Table 1). All NCCLs were restored with light-cured microhybrid resin composites (Z250, 3M ESPE). A total of 164 NCCLs from 35 patients were randomly allocated to one of four experimental groups using a randomization table. Approximately 40 restorations per group were placed in NCCLs of incisors, canines, premolars, and first molars. To minimize patient-related effects that may bias the results, no more than three restorations for one group were allowed in a patient.

Tooth shade was evaluated prior to the operative procedures by using Vita shade guide (Vita Zahnfabrik, Bad Säckingen, Germany). The lesion was cleaned with plain pumice slurry in a rubber cup and then washed and dried. No surface grinding or

enamel beveling were performed for the lesion. The lesion was isolated with a cotton roll and gingival retraction cord (Ultrapak, Ultradent, South Jordan, UT) during the procedure.

Adhesive, either CS or XE, was applied to the lesion according to the manufacturer's instructions listed in Table 1. Composite resin was then built up and light cured with an LED light-curing unit (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein). The intensity of light was measured by a portable LED radiometer (Bluephase Meter II, Ivoclar Vivadent) prior to each restoration procedure to check for any drop in intensity. For large lesions, two or three increments of resin composite were separately applied. Great care was taken to avoid significant overhanging in the gingival margin during the composite resin buildup. The light curing was performed using a pulse delay cure technique with an initial cure for 2 seconds and final cure for 20 seconds following a waiting period of 3 minutes.[20]

Excess composite resin on the gingival margin of all restorations was trimmed with a #12 blade during the waiting period for the pulse delay cure. Gross reduction and finishing were performed with a fine diamond point (Mani, Tochigi, Japan) after a final light curing. Further finishing and polishing were then performed using either the Sof system or the EP system. In Sof groups, polishing was performed in a sequence of grit from coarse to superfine with 3/8-inch discs and a mandrel. Each disc step was performed in a dry field for 15 to 20 seconds with a low-speed hand piece. Rinsing and drying were performed before proceeding to the next grit sequence. In EP groups, polishing was sequentially performed with the point shape of Enhance and Pogo using a buffing motion with moderate to light intermittent pressure. Each step was carried out without water for 15 to 20 seconds with a low-speed hand piece. A rinse-and-dry procedure was performed between the use of Enhance and Pogo. One experienced operator performed all the procedures for all the restorations.

### Clinical Outcome Evaluation

The class V restorations were evaluated at baseline, 6, 12, 18, and 24 months by two blinded independent examiners. A flow diagram of clinical evaluation is shown in Figure 1. Modified FDI criteria were used to evaluate the clinical performance with respect to the esthetic, functional, and biological properties of the restorations (Table 2). We chose two of the original FDI criteria for each property to evaluate the class V restorations: surface luster and staining for esthetic properties, fracture and retention and marginal adaptation for functional properties, and postoperative sensitivity and recurrence of caries, erosion, and abfraction for biologic properties. Each criterion was evaluated with five scores: all scores ranged from 5 to 1 (with a score of 1 being the poorest). For interexaminer and intraexaminer calibration, the examiners were trained for each criteria using representative sample photographs obtained from previously published literature and from a Web-based tool called "e-calib" (www.e-calib.info).[21,22] If a discrepancy between examiners occurred during evaluation, it was resolved by consensus. All missing cases, including cases involving unannounced nonattendance of the subject at recall, were withdrawn from the study. A restoration with a fracture and a retention score of 1 was counted as missing in the other evaluation criteria. The interexaminer agreement rate ranged from 95% to 100% depending on the criteria.

### Statistical Analysis

The frequencies and percentages of below-excellent outcomes (score of $<5$) at 24 months were displayed and tested according to groups (combinations of polishing methods and adhesives used) using the Fisher exact test. We also used the generalized estimating equations (GEE) approach with unstructured covariance to compare the possibility of outcomes under considering the repeated measures.[23] Models for marginal staining and marginal adaptation were estimated to compare the four groups (combinations of polishing methods and adhesives) and subsequently to compare multistep abrasive discs vs the simplified abrasive-impregnated rubber polishing instrument and two-step vs one-step adhesives. The interaction term was omitted as insignificant (in marginal staining, $p=0.615$, and in marginal adaptation, $p=0.157$). Models for surface luster, staining surface, fracture and retention, postoperative sensitivity, and recurrent caries could not be estimated because of the limited number of cases with below-excellent outcomes (score of $<5$). The same statistical process was also performed for the frequencies and percentages of below-good outcomes (score of $<4$). A level of 0.05 was adopted to determine the statistical significance of all differences. All statistical analyses were performed using SAS statistical software, version 9.1.3 (SAS Institute, Cary, NC), and models were calculated using the GENMOD procedure.[24]

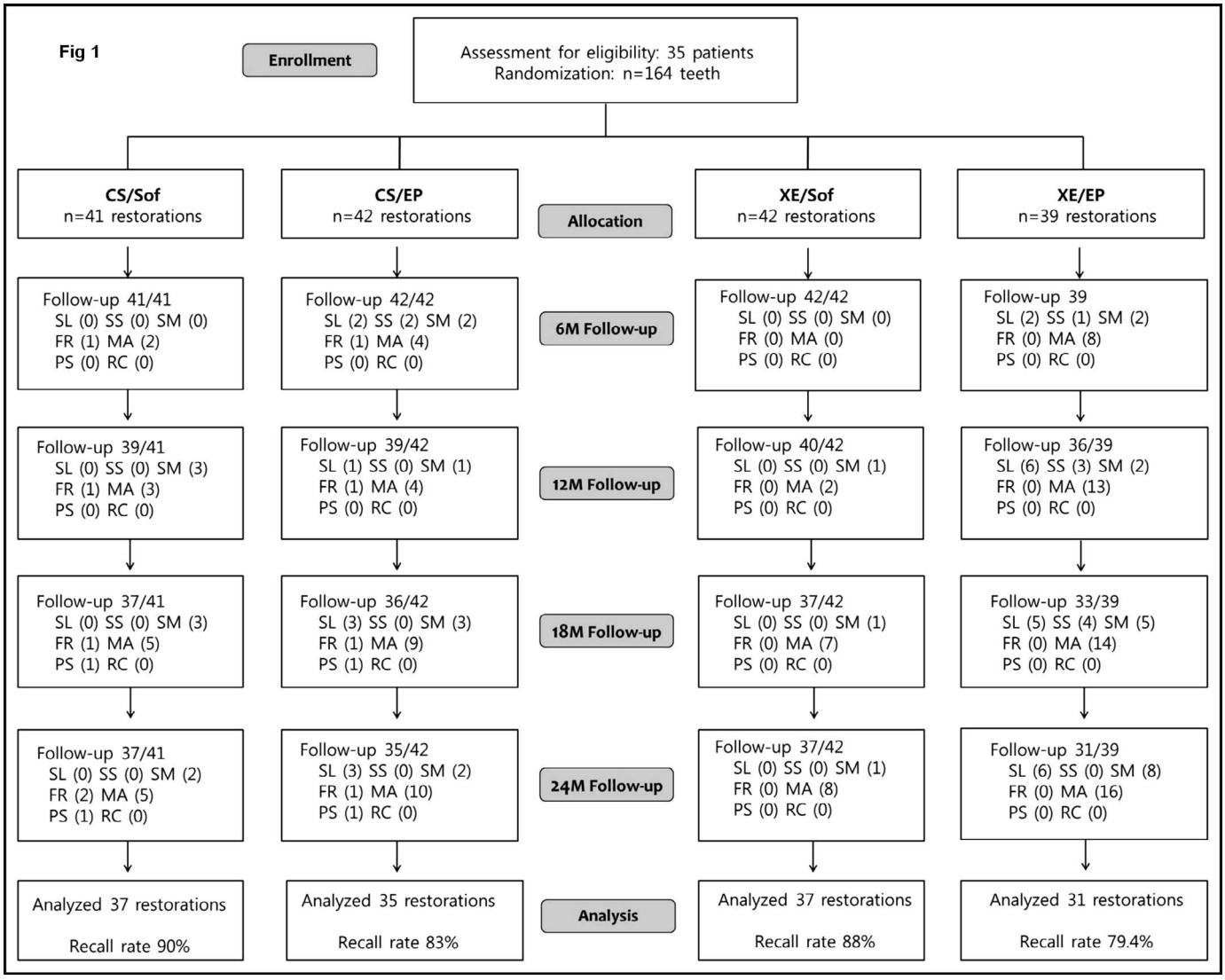


Figure 1. *Flow diagram for evaluation. Abbreviations: n, number of restorations; Sof, Sof-Lex; EP, Enhance/Pogo; CS, Clearfil SE bond; XE, Xeno V; SL, surface luster; SS, staining surface; SM, staining margin; FR, fracture and retention; MA, marginal adaptation; PS, postoperative sensitivity; RC, recurrent caries.*

## RESULTS

Of the 164 restorations at baseline, 164 (100%), 154 (93.9%), 143 (87.2%), and 140 (85.3%) were observed at 6, 12, 18, and 24 months, respectively. Descriptive data expressed as percentages of outcomes for the four groups are shown in Table 3. A total of three restorations were dislodged over 24 months: two in the CS/Sof group and one in the CS/EP group. Except for those retention losses, none of the restorations required any repair or retreatment in all criteria over 24 months. The frequencies of below-excellent outcomes (score of <5) at 24 months are shown in Table 4. Significant differences in the proportions of below-excellent outcomes among the four groups were observed for surface luster, surface staining, marginal staining, and marginal adaptation ($p<0.05$). Polishing with multistep abrasive discs (Sof) was superior to that with simplified abrasive-impregnated rubbers (EP) with regard to surface luster, surface staining, marginal staining, and marginal adaptation ($p<0.05$). Two-step (CS) and one-step (XE) adhesives did not show any statistically significant differences in any criteria ($p>0.05$). Representative photographs for clinical evaluation with a score of <5 at 24 months are presented in Figure 2.

GEE analysis considering the five repeated measurements from baseline to 24 months is shown in Table 5. In the evaluation of marginal staining, there was no significant difference among the four groups,

Table 2: *Modified FDI Criteria Used in This Study*

| Category | Rating | Description |
|---|---|---|
| A. Esthetic properties | | |
| 1. Surface luster | 5 | Luster comparable to enamel |
| | 4 | Slightly dull, not noticeable from speaking distance, some isolated pores |
| | 3 | Dull surface but acceptable if covered with film of saliva, multiple pores on more than one-third of the surface |
| | 2 | Rough surface, cannot be masked by saliva film, simple polishing not sufficient |
| | 1 | Very rough, unacceptable plaque-retentive surface |
| 2.1 Staining (surface) | 5 | No staining |
| | 4 | Minor staining, easily removable by polishing |
| | 3 | Moderate surface staining that may also be present on other teeth, not esthetically unacceptable |
| | 2 | Unacceptable surface staining; major intervention necessary |
| | 1 | Severe surface and/or subsurface staining, not acceptable for intervention |
| 2.2 Staining (margin) | 5 | No staining |
| | 4 | Minor staining, easily removable by polishing |
| | 3 | Moderate marginal staining, not esthetically unacceptable |
| | 2 | Pronounced marginal staining; major intervention necessary |
| | 1 | Deep marginal staining, not accessible for intervention |
| B. Functional properties | | |
| 3. Fractures and retention | 5 | Keep complete retention |
| | 4 | Small hairline crack |
| | 3 | Material chip fracture not affecting marginal integrity |
| | 2 | Material chip fracture that damages marginal quality, bulk fractures with partial loss (less than half of the restoration) |
| | 1 | Complete loss of restoration or multiple fractures |
| 4. Marginal adaptation | 5 | Harmonious outline, no gaps |
| | 4 | Slight ditching, slight step/flashes, minor irregularities |
| | 3 | Major irregularities, ditching or flash, steps |
| | 2 | Severe ditching or marginal fractures, larger irregularities or steps (repair necessary) |
| | 1 | Generalized major gaps or irregularities |
| C. Biologic properties | | |
| 5. Postoperative sensitivity | 5 | No hypersensitivity |
| | 4 | Minor hypersensitivity for a limited period |
| | 3 | Moderate hypersensitivity (no treatment needed) |
| | 2 | Intense hypersensitivity with subjective symptoms |
| | 1 | Intense, acute pulpitis or nonvital tooth |
| 6. Recurrence of caries, erosion, abfraction | 5 | No secondary or primary caries |
| | 4 | Small and localized erosion or abfraction |
| | 3 | Larger areas of erosion or abfraction, dentin not exposed |
| | 2 | Caries with cavitation and suspected undermining caries |
| | | Erosion, abfraction in dentin |
| | 1 | Deep caries |

*Score designation: 5, clinically excellent/very good; 4, clinically good; 3, clinically sufficient/ satisfactory (minor shortcomings, no unacceptable effects but not adjustable without damage to the tooth); 2, clinically unsatisfactory (but reparable); 1, clinically poor (replacement necessary).*

though the XE-EP group showed the highest percentage (6.5%) of worse outcomes. There was no significant difference both between the two polishing systems and between the two adhesives. In the evaluation of marginal adaptation, the XE-EP group showed a significant difference in the odds ratio (OR=4.07, $p$=0.001) compared to the CS-Sof group and also showed the highest percentage (24.6%) of worse outcome over 24 months. Use of the simplified abrasive-impregnated rubber polishing system re-

Table 3: *Descriptive Data (Percentage) of Clinical Outcomes Evaluated at Baseline and 6, 12, 18, and 24 Months According to the Modified FDI Criteria*[a]

| Group | | CS/Sof | | | | | CS/EP | | | | | XE/Sof | | | | | XE/EP | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Criterion** | **Time** | **5** | **4** | **3** | **2** | **1** | **5** | **4** | **3** | **2** | **1** | **5** | **4** | **3** | **2** | **1** | **5** | **4** | **3** | **2** | **1** |
| Esthetic properties | | | | | | | | | | | | | | | | | | | | | |
| SL | 0 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 |
| | 6 mo | 100 | 0 | 0 | 0 | 0 | 95.1 | 4.9 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 94.8 | 2.6 | 2.6 | 0 | 0 |
| | 12 mo | 100 | 0 | 0 | 0 | 0 | 97.4 | 2.6 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 83.3 | 13.9 | 2.8 | 0 | 0 |
| | 18 mo | 100 | 0 | 0 | 0 | 0 | 91.4 | 8.6 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 84.8 | 12.1 | 3.1 | 0 | 0 |
| | 24 mo | 100 | 0 | 0 | 0 | 0 | 91.2 | 8.8 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 80.6 | 16.2 | 3.2 | 0 | 0 |
| SS | 0 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 |
| | 6 mo | 100 | 0 | 0 | 0 | 0 | 95.1 | 4.9 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 97.4 | 2.6 | 0 | 0 | 0 |
| | 12 mo | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 91.7 | 8.3 | 0 | 0 | 0 |
| | 18 mo | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 87.9 | 12.1 | 0 | 0 | 0 |
| | 24 mo | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 83.9 | 16.1 | 0 | 0 | 0 |
| SM | 0 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 |
| | 6 mo | 100 | 0 | 0 | 0 | 0 | 95.1 | 4.9 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 94.9 | 5.1 | 0 | 0 | 0 |
| | 12 mo | 92.1 | 7.9 | 0 | 0 | 0 | 97.4 | 2.6 | 0 | 0 | 0 | 97.5 | 2.5 | 0 | 0 | 0 | 94.4 | 5.6 | 0 | 0 | 0 |
| | 18 mo | 91.7 | 8.3 | 0 | 0 | 0 | 91.4 | 8.6 | 0 | 0 | 0 | 97.3 | 2.7 | 0 | 0 | 0 | 84.8 | 15.2 | 0 | 0 | 0 |
| | 24 mo | 94.3 | 5.7 | 0 | 0 | 0 | 94.1 | 5.9 | 0 | 0 | 0 | 97.3 | 2.7 | 0 | 0 | 0 | 74.2 | 25.8 | 0 | 0 | 0 |
| Functional properties | | | | | | | | | | | | | | | | | | | | | |
| FR | 0 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 |
| | 6 mo | 97.6 | 0 | 0 | 0 | 2.4 | 97.6 | 0 | 0 | 0 | 2.4 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 |
| | 12 mo | 97.4 | 0 | 0 | 0 | 2.6 | 97.4 | 0 | 0 | 0 | 2.6 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 |
| | 18 mo | 97.2 | 0 | 0 | 0 | 2.8 | 97.2 | 0 | 0 | 0 | 2.8 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 |
| | 24 mo | 94.6 | 0 | 0 | 0 | 5.4 | 97.1 | 0 | 0 | 0 | 2.9 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 |
| MA | 0 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 |
| | 6 mo | 95 | 5 | 0 | 0 | 0 | 90.5 | 9.5 | 0 | 0 | 0 | 100 | 0 | 0 | 0 | 0 | 79.5 | 20.5 | 0 | 0 | 0 |
| | 12 mo | 92.1 | 7.9 | 0 | 0 | 0 | 89.5 | 10.5 | 0 | 0 | 0 | 95 | 5 | 0 | 0 | 0 | 63.9 | 33.3 | 2.8 | 0 | 0 |
| | 18 mo | 86.1 | 13.9 | 0 | 0 | 0 | 74.3 | 25.7 | 0 | 0 | 0 | 81.1 | 18.9 | 0 | 0 | 0 | 57.6 | 36.4 | 6 | 0 | 0 |
| | 24 mo | 85.7 | 14.3 | 0 | 0 | 0 | 70.6 | 29.4 | 0 | 0 | 0 | 78.4 | 18.9 | 2.7 | 0 | 0 | 48.4 | 41.9 | 9.7 | 0 | 0 |

*Abbreviations: SL, surface luster; SS, staining surface; SM, staining margin; FR, fracture and retention; MA, marginal adaptation; PS, postoperative sensitivity; RC, recurrent caries; Sof, Sof-Lex; EP, Enhance/Pogo; CS, Clearfil SE bond; XE, Xeno V.*

[a] *The data of biologic properties including postoperative sensitivity (PS) and recurrent caries (RC) were omitted because those criteria showed the highest score (score of 5) in all experimental groups and evaluation time points, except one restoration of each CS/Sof (score of 3) and CS/EP (score of 4) for PS criteria in 18 months, respectively.*

sulted in a significantly inferior performance compared to the multistep abrasive disc polishing system (OR=3.22, $p<0.001$).

In the analysis for the below-good outcomes (score of <4), frequencies at 24 months and GEE analysis considering the repeated measurements did not show any significant differences both between polishing systems and between adhesives.

## DISCUSSION

To the best of our knowledge, this is the first study employing a randomized controlled clinical trial to clinically compare multistep abrasive discs with simplified abrasive-impregnated rubber instruments in terms of polishing effectiveness in class V composite resin restorations. The main finding of the present study is that Sof, a multistep abrasive disc system, showed better clinical performance than EP, a simplified abrasive-impregnated rubber system, in all the esthetic properties (surface luster, surface staining, and marginal staining) and one functional property (marginal adaptation) in composite resin restorations at 24 months. According to GEE analysis considering repeated observations over 24 months, polishing with multistep abrasive discs also presented a superior outcome in marginal adaptation compared to that with simplified abrasive-impregnated rubber instruments.

The polishing effectiveness of abrasive-coated discs and abrasive-impregnated rubber instruments has been previously compared in many *in vitro*

Table 4: *Frequencies (Percentage) of Below-Excellent Outcomes (Score of <5) for Each Criterion at 24 Months According to the Groups, Polishing System, and Adhesives*[a]

| Criteria | Esthetic Properties | | | Functional Properties | | Biologic Properties | |
|---|---|---|---|---|---|---|---|
| | SL | SS | SM | FR | MA | PS | RC |
| Group | | | | | | | |
| CS/Sof | 0 (0.0) A | 0 (0.0) A | 2 (5.7) A | 2 (5.4) | 5 (14.3) A | 1 (2.9) | 0 (0.0) |
| CS/EP | 3 (8.8) A | 0 (0.0) A | 2 (5.9) A | 1 (2.9) | 10 (29.4) B | 1 (2.9) | 0 (0.0) |
| XE/Sof | 0 (0.0) A | 0 (0.0) A | 1 (2.7) A | 0 (0.0) | 8 (21.6) B | 0 (0.0) | 0 (0.0) |
| XE/EP | 6 (19.4) B | 5 (16.1) B | 8 (25.8) B | 0 (0.0) | 16 (51.6) B | 0 (0.0) | 0 (0.0) |
| *p*-value[b] | 0.001 | 0.001 | 0.011 | 0.509 | 0.008 | 0.726 | — |
| Polishing system | | | | | | | |
| Sof | 0 (0.0) | 0 (0.0) | 3 (4.2) | 2 (2.7) | 13 (18.1) | 1 (1.4) | 0 (0.0) |
| EP | 9 (13.9) | 5 (7.69) | 10 (15.4) | 1 (1.5) | 26 (40.0) | 1 (1.5) | 0 (0.0) |
| *p*-value | <0.001 | 0.022 | 0.039 | 1.000 | 0.005 | 1.000 | — |
| Dentin adhesive | | | | | | | |
| CS | 3 (4.4) | 0 (0.0) | 4 (5.8) | 3 (4.2) | 15 (21.7) | 2 (2.9) | 0 (0.0) |
| XE | 6 (8.8) | 5 (7.4) | 9 (13.2) | 0 (0.0) | 24 (35.3) | 0 (0.0) | 0 (0.0) |
| *p*-value | 0.325 | 0.208 | 0.159 | 0.245 | 0.079 | 0.496 | — |

*Abbreviations: SL, surface luster; SS, staining surface; SM, staining margin; FR, fracture and retention; MA, marginal adaptation; PS, postoperative sensitivity; RC, recurrent caries; Sof, Sof-Lex; EP, Enhance/Pogo; CS, Clearfil SE bond; XE, Xeno V.*

[a] *Different letters (A and B) represent significant differences among groups at an alpha level of 0.05.*

[b] p-*values by the Fisher exact test.*

studies. Among the various polishing systems, abrasive-coated discs have been reported to be the most effective polishing instruments available and are reported to produce the highest gloss and smoothest surface with composite resins.[9-11] On the other hand, in a number of recent studies, abrasive-impregnated rubber instruments showed comparable or even better performance in reducing the surface roughness when compared to abrasive discs.[12-14] These contradictory results from *in vitro* studies might be due to differences in the experimental conditions, such as initial roughness state, combinations of finishing and polishing protocols, and polishing time and pressure. Despite the use of a variety of *in vitro* experimental conditions, *in vitro* studies might not completely reflect the clinical

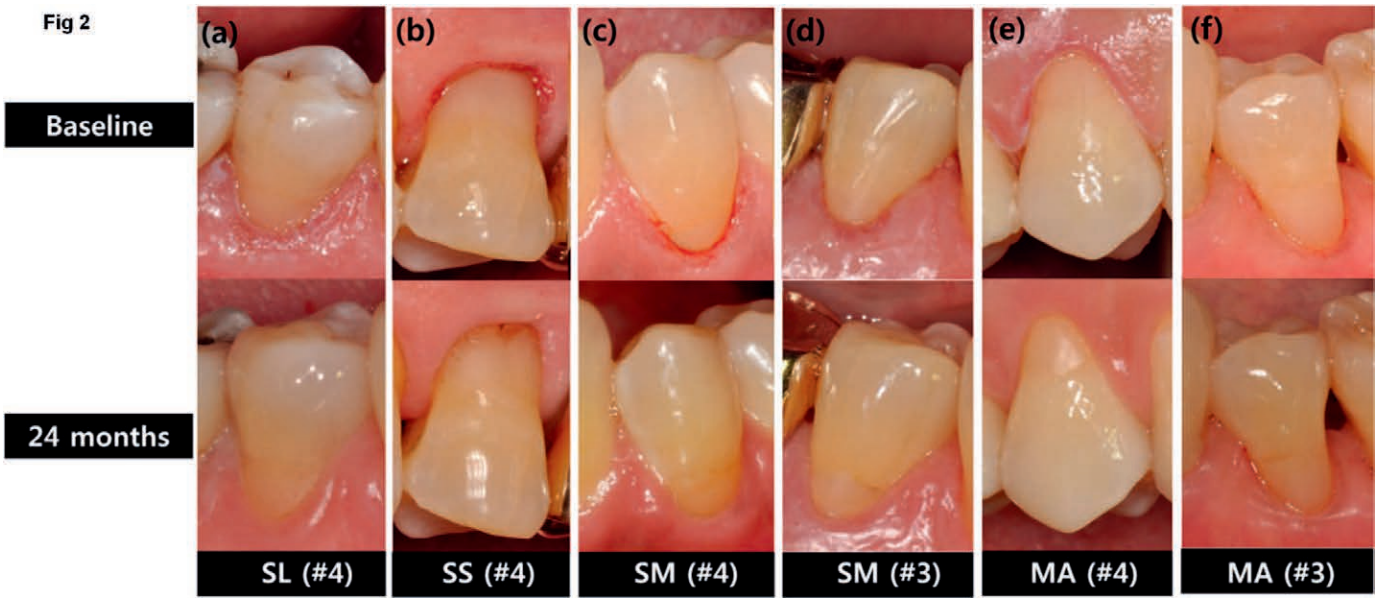


Figure 2. *Representative photographs for clinical evaluations with score <5 at 24 months. (a): Score 4 for surface luster. (b): Score 4 for surface staining. (c) and (d): Scores 4 and 3 for margin staining, respectively. (e) and (f): Scores 4 and 3 for marginal adaptation, respectively.*

**Table 5:** *Odds Ratio and Adjusted Proportion of Below-Excellent Outcomes (Score of <5) Under Consideration of Repeated Observation for 24 Months*[a]

| Criteria | Group | Odds Ratio (95% Confidence Interval) | *p*-Value | Adjusted Log Odds | Adjusted Percentage |
|---|---|---|---|---|---|
| SM | CS/Sof | Reference | — | −3.89 | 2.0% |
| | CS/EP | 1.22 (0.28-5.28) | 0.786 | −3.43 | 3.1% |
| | XE/Sof | 0.45 (0.07-2.73) | 0.382 | −4.46 | 1.1% |
| | XE/EP | 2.61 (0.68-9.99) | 0.162 | −2.67 | 6.5% |
| | EP[b] | 2.60 (0.92-7.33) | 0.072 | | |
| | XE[b] | 1.21 (0.44-3.28) | 0.714 | | |
| MA | CS/Sof | Reference | — | −2.79 | 5.8% A |
| | CS/EP | 1.76 (0.71-4.34) | 0.219 | −2.06 | 11.3% A |
| | XE/Sof | 1.04 (0.39-2.76) | 0.936 | −2.67 | 6.5% A |
| | XE/EP | 4.07 (1.75-9.44) | 0.001[c] | −1.12 | 24.6% B |
| | EP[b] | 3.22 (1.63-6.35) | <0.001[c] | | |
| | XE[b] | 1.88 (0.97-3.66) | 0.061 | | |

*Abbreviations: SM, staining margin; MA, marginal adaptation; Sof, Sof-Lex; EP, Enhance/Pogo; CS, Clearfil SE bond; XE, Xeno V.*
[a] *Different letters (A and B) represent significant difference among groups at an alpha level of 0.05.*
[b] *Interaction term was omitted as insignificant in SM (p=0.615) and MA (p=0.157).*
[c] *p<0.05 by generalized estimating equation considering repeated observations for 24 months.*

efficacy of polishing instruments. Most *in vitro* studies evaluate the polishing performance on a flat and homogeneously finished composite specimen for experimental standardization, while, in clinical practice, the desired surface of composite restorations is rather convex or concave and the finishing state not as homogeneous as that observed in the experimental setting. Finishing with diamond point, the procedure implemented in the present study, delivers a comparatively good finish on curved surfaces and feather edges but has also been reported to leave a relatively irregular surface.[25,26] Finishing using abrasive disc instruments begins with a coarse disc before polishing proceeds with gradually finer-grained abrasive discs. Therefore, abrasive discs may have been less affected by the irregularity of the finished surface and hence may have produced a smoother final polishing surface than the abrasive-impregnated rubber instruments. This may explain the better clinical performance score in surface luster, surface staining, and marginal staining at 24 months.

In the present study, Sof also showed better marginal adaptation than EP at 24 months. An earlier *in vitro* study reported that the hardness of the aluminum oxide abrasive used in abrasive discs is higher than that of most filler particles in the composite resin formulation; thus, this abrasive cuts filler and resin matrix equally to produce a smooth surface.[27] Because of the hardness of the abrasive particle and the thin flexible backing, Sof appears to work better than EP in convex cervical composite restorations by blending smoothly with the contours of the tooth being restored. Moreover, EP also appears to be too bulky to reach the interproximal and cervical margin of class V composite restorations, regardless of their shape.

Although analysis of the percentages below score 5 (the clinically excellent outcome) indicated that the polishing performance of Sof is better in class V composite restorations, EP did not show any significant differences from Sof, in any criteria, in the analysis of the percentages below score 4 (the clinically good outcome). Accordingly, EP is also considered to be an effective instrument to obtain a clinically good polishing performance in class V composite restorations. The use of EP, which is an abrasive-impregnated rubber polishing system, is more advantageous when access to the anatomically contoured occlusal surface is required since the various forms of the instrument (cup, point, and wheel) offset the access limitations of the abrasive disc.[13,28] Additional significant advantages of abrasive-impregnated rubber instruments over multistep abrasive discs include the relative ease of handling and the significant time savings.

It is not clearly understood why the XE-EP group showed a significantly inferior outcome to the CS-Sof group with regard to marginal adaptation. The observed difference between the polishing systems presented a high odds ratio showing significance, while the observed difference between the adhesives did not (Table 5). Therefore, the polishing system is assumed to be a variable with significantly more effect than the adhesive system. In previous studies, one-step self-etch adhesives have been reported to

show lower bond strength to enamel relative to two-step self-etch adhesives.[29-31] The lower bond strength of one-step self-etch adhesives is likely to be more evident for uncut enamel without additional acid etching, as in the present study.[32,33] The possibility of leaving a thin overhang of composite resin on the uncut enamel is greater when polishing with EP than when polishing with Sof, which shows superior finishing ability by starting the procedure with a 100-μm-grain-sized coarse disc. This thin overhang might be more easily fractured out from restorations bonded with XE due to the relatively weak bond strength in comparison to the bond strength with CS. As a consequence, fracturing of a thin overhang may be reflected as inferior performance in the marginal adaptation in the XE-EP group in comparison with the CS-Sof group.

The FDI criteria that were recently introduced for clinical evaluation of dental restorations were slightly modified in the present study. From the 16 categories of original FDI criteria for evaluation of class V restorations, we appropriately chose representative categories that we could easily assess. For the sake of convenience, each criterion was scored from a high of 5 (clinically excellent) to a low of 1. This was in contrast to the original FDI criteria, where 1 was best and 5 was worst.[15] In addition, in several criteria, the original subscores were integrated to present one score for simplification of the evaluation. The FDI criteria appear to be more sensitive and more precise in the evaluation of composite resin restorations in comparison with the US Public Health Service criteria system, which had been generally used in many clinical trials, because of the use of a detailed scoring system.

The present study does not address all clinical aspects of polishing systems utilized in composite resin restorations. A number of studies have reported that the polishing outcome depends on the filler size, shape, and loading of composite resin.[32,33] The present study used only one type of microhybrid composite resin. The dependence of clinical polishing effectiveness on the different filler types of composite resin would be an intriguing issue for future studies. Another limitation of this study is that the observation period of 24 months may be relatively short to confirm the clinical polishing performance over the whole service span of composite resin restorations. Further observations with longer follow-up are required.

## CONCLUSIONS

In conclusion, multistep abrasive discs (Sof) were clinically superior in clinical polishing performance to simplified abrasive-impregnated rubber instruments (EP) with regard to criteria such as surface luster, surface staining, marginal staining, and marginal adaptation for class V composite resin restorations over a 24-month follow-up period. Although EP showed inferior performance to Sof, this instrument still presented clinically good outcomes. Considering the advantage of EP in ease of handling and time saving, abrasive-impregnated rubber instruments may also be recommended for the polishing of class V composite resin restorations. XE, one-step self-etch adhesives showed clinically equivalent performance to CS, two-step self-etch adhesives.

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### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Institutional Review Board of KHUDH. The approval code for this study is KHD-IRB 1603-5.

### 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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# Efficacy of Home-use Bleaching Agents Delivered in Customized or Prefilled Disposable Trays: A Randomized Clinical Trial

NR Carlos • EC Bridi • FLB Amaral • FMG França • CP Turssi • RT Basting

### Clinical Relevance

Home-use bleaching agents delivered in customized or prefilled disposable trays are equally effective in promoting tooth color change but may cause tooth sensitivity that may intensify during treatment. Users seem to find customized trays more comfortable.


## SUMMARY

**The purpose of this study was to evaluate bleaching methods containing hydrogen peroxide (HP) or carbamide peroxide (CP), dispensed in customized or prefilled trays, in terms of color change, tooth sensitivity, gingival irritation, acceptance, and comfort. Seventy-five volunteers were randomly selected and distributed according to the whitening agent (n=25): 10% HP dispensed in prefilled trays (Opalescence Go 10%) and 9.5% HP (Pola Day) and 10% CP both delivered in customized trays (Opalescence PF 10%). HP was applied for 30 min/d for 14 days (d), and CP for 8 h/d for 14 days. Evaluations were performed at baseline and at 7 days and 14 days of treatment. Color change was measured with Commission internationale de l'éclairage color coordinates (L*, a*, b*), Vita Classical, and 3D Master scales. A visual analog scale was used to assess tooth sensitivity, acceptance of the method and degree of comfort of the tray. Gingival irritation was evaluated as present or absent and localized or generalized. Regarding gingival irritation, tray acceptance, and tooth sensitivity, no differences were observed among the groups at any time ($p>0.05$). As for degree of comfort, 10% HP showed lower scores (comfortable) than 10% CP, with significant differences ($p<0.05$) from the other groups (comfortable to very comfortable). In terms of**



Natália R Carlos, DDS, São Leopoldo Mandic Institute and Dental Research Center, Restorative Dentistry, Campinas, Brazil

Enrico C Bridi, MS, São Leopoldo Mandic Institute and Dental Research Center, Restorative Dentistry, Campinas, Brazil

Flávia L Botelho do Amaral, PhD, professor, São Leopoldo Mandic Institute and Dental Research Center, Restorative Dentistry, Campinas, Brazil

Fabiana M Gomes França, PhD, professor, São Leopoldo Mandic Institute and Dental Research Center, Restorative Dentistry, Campinas, Brazil

Cecilia P Turssi, PhD, professor, São Leopoldo Mandic Institute and Research Center, Campinas, Brazil

*Roberta T Basting, PhD, professor, São Leopoldo Mandic Institute and Dental Research Center, Restorative Dentistry, Campinas, Brazil

*Corresponding author: Rua José Rocha Junqueira, 13, Campinas, São Paulo 13045-755 Brazil; e-mail: rbasting@yahoo.com


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**ΔL, Δa, and ΔE, no difference was observed among the groups or between the time periods ($p>0.05$). The Δb average was higher at 14 days ($p<0.05$), and there was no difference among the groups ($p>0.05$). Localized gingival irritation was observed in both tray methods. Mild tooth sensitivity was observed with time, regardless of the bleaching agent concentration or the application time. Color change was similar for all the groups at 7 days and 14 days, but there was a greater reduction in the yellow hue at 14 days. All the bleaching methods were highly accepted and effective in promoting whitening. Although prefilled trays are generally comfortable, they proved less comfortable than customized trays.**

## INTRODUCTION

Supervised dental bleaching using dental trays is one of the most commonly used approaches to modify tooth color.[1-3] The main advantages of this technique are related to the patient's ease of use, reduced chair time, and a comparatively similar or lower prevalence of tooth sensitivity and gingival irritation during treatment in relation to chairside methods using high concentrations of peroxides.[4-11]

Ten percent carbamide peroxide has traditionally been the most suitable bleaching agent for this procedure;[1,10] however, to increase the efficacy of bleaching products, higher concentrations have been used,[12-14] and new bleaching agents containing 3% to 10% hydrogen peroxide have been released.[11,14,15]

Hydrogen peroxide in customized trays was proposed to reduce bleaching time, while maintaining effectiveness, compared with carbamide peroxide.[10,11,13,14,16] A carbamide peroxide gel containing desensitizing agents that could be applied in a shorter time has also been proposed to reduce the intensity of tooth sensitivity.[17]

Other home-use dental bleaching methods have become available, particularly those involving prefilled trays, such as Opalescence Go (Ultradent Products, South Jordan, UT), and containing a combination of carbamide and hydrogen peroxide, resulting in a total of 10% hydrogen peroxide. The use of prefilled trays can make application of the whitening treatment easier by eliminating the need for impressions, plaster casts, and tray customization, thus speeding up the bleaching treatment with proven effectiveness in tooth color change.[15] Prefilled trays may be adapted to the dental arches and resemble customized tray bleaching treatments, but they must be done under the supervision of a dentist.[15,18] Nevertheless, because prefilled trays do not provide adequate sealing, there may be overflow of the whitening product into the oral cavity, causing discomfort to the patient. In this regard, there have been no clinical studies showing a combination of effects, such as tooth whitening, tooth sensitivity, treatment acceptance, and level of comfort, when using this alternative prefilled approach, in comparison to other bleaching methods using carbamide peroxide or hydrogen peroxide in customized trays.

The objective of this study was, therefore, to compare the effects of whitening techniques using carbamide peroxide or hydrogen peroxide dispensed in customized or prefilled trays on color change, tooth sensitivity, gingival irritation, and treatment acceptance and comfort. The null hypothesis tested was that there would be no difference in terms of clinical parameters among the bleaching products containing 10% hydrogen peroxide in prefilled trays versus 9.5% hydrogen peroxide and 10% carbamide peroxide in customized trays.

## METHODS AND MATERIALS

### Experimental Design and Bleaching Agents Used

Three groups were investigated: 10% hydrogen peroxide prescribed in the form of prefilled trays (Opalescence Go 10% Mint [OPAGO], Ultradent Products), 9.5% hydrogen peroxide prescribed in the form of customized trays (Pola Day [POD], SDI), and 10% carbamide peroxide prescribed in the form of customized trays (Opalescence 10% PF Mint [OPA], Ultradent Products). The patients were evaluated at baseline before beginning treatment and after 7 days and 14 days of bleaching. The materials used, as well as their specifications and application protocols, are shown in Table 1. The pH value of the bleaching agents was measured in triplicate at different times using a benchtop pH meter (MS Tecnopon Special Equipment Ltd, Piracicaba, Brazil): baseline, 15 minutes, and 30 minutes (OPAGO and POD agents); and baseline, 15 and 30 minutes, and 1, 2, 4, 6, and 8 hours (OPA). All the measurements were made upon removal of the gel with a spatula, directly from the preloaded impression tray (OPAGO) or after being dispensed from a syringe (POD and OPA). The following pH values were obtained for the agents, according to the respective measurement periods: OPAGO (6:02; 6:01; 5.97); POD (6.25; 6.24; 6.14); OPA (6:53; 6:51; 6:52; 6:52; 6:56; 6:55; 6:55; 6:59).

Table 1: *Bleaching Agents, Compositions, and Manufacturers*

| Bleaching Treatments | Bleaching agents/ Manufacturer (City, State, Country)/Lot Number | Composition (Percentage in Weight) | Daily Time of Use/Total Number of Treatment Days |
|---|---|---|---|
| OPAGO (10% hydrogen peroxide delivered in prefilled disposable trays) | Opalescence Go Mint/ Ultradent Products (South Jordan, UT, USA)/D005R, D002U | 7.9% hydrogen peroxide (<13), 7.5% carbamide peroxide (<8), sodium fluoride (<0,3), sodium hydroxide (<5), glycerin (<39), potassium nitrate (≤3) | 30 minutes a day/14 days |
| POD (9.5% hydrogen peroxide delivered in custom-made trays) | Pola Day 9.5%/ SDI (Melbourne, Victoria, Australia)/P130308Z | 9.5% hydrogen peroxide, < 47% additives, 30% glycerol, 20% water, 0.1% flavoring, potassium nitrate | 30 minutes a day/14 days |
| OPA (10% carbamide peroxide delivered in custom-made trays) | Opalescence PF 10% Mint/ Ultradent Products/D003K,D00LT | 10% carbamide peroxide (<25), polyacrylic acid (<10), 0.3% sodium fluoride (<0.25), 3% sodium hydroxide (<5) | 8 hours at night/14 days |

## Patient Selection

Seventy-five participants of both sexes were selected. The minimum sample size was established at 60 participants for the experiment, 20 participants per group, according to a previous study.[9] Twenty-five participants were recruited per group (n=25) to compensate for possible subject withdrawals or loss.

Participants were included or excluded from the study based on history-taking and clinical examination considering the following inclusion criteria:[9] age between 18 and 30 years, presence of at least 20 sound teeth, and presence of central and lateral incisors, or maxillary and mandibular canines, with at most 1/6 of the buccal surface restored. Exclusion criteria were[9] teeth with an initial color of B1 assessed using a shade scale (Vitapan Classical, VITA Zahnfabrik, Bad Sackingen, Germany) or spectrophotometer (VITA Easyshade Advance, (VITA Zahnfabrik, Bad Säckingen, Germany), people wearing dentures or fixed/removable orthodontic appliances, pregnant or breast-feeding women, smokers, history of dentin sensitivity, presence of active caries in enamel or dentin, periodontal or other oral disease, tetracycline-pigmented teeth, and previous tooth bleaching.

## Tooth Color Shade Evaluation

The bleaching treatment was randomly assigned to each participant using a sequence of random software generated numbers. Tooth color evaluation was performed in a dental office using natural light (light from a window), in addition to artificial lighting from the fluorescent lamps in the dental office. The same examiner was responsible for assessing tooth color at all times.

A spectrophotometer (VITA Easyshade Advance) was used to measure the color at the middle third of the labial surface of the maxillary right central incisor at baseline. Color evaluation was performed using a probe tip supported and seated at a right angle with the tooth surface. This measurement was immediately duplicated to improve accuracy. When the two readings were the same for the Vita Classical scale, the value measured was noted after obtaining the second reading and the other parameters. If the two readings did not match, a new measurement was taken until agreement was reached between two readings. In sum, tooth color was verified using the Vita Classical shade guide, the Vita 3D Master, and Commission internationale de l'éclairage color coordinates where L represents the lightness, a represents the point on a red-green scale, and b the point on a yellow-blue scale (CIELab). Since all the evaluations for the Vita Classical shade guide, the Vita 3D Master, and the CIELab system parameters were performed using a spectrophotometer, no color-matching competency of the examiner was applied.

A week before starting the bleaching treatment, the participants underwent a run-in period to standardize the study toothbrush (Oral B Indicator Plus, Procter & Gamble, São Paulo, Brazil) and 1500 ppm fluoride toothpaste (Colgate Maximum Anticaries Protection, Colgate-Palmolive, São Bernardo do Campo, Brazil).

## Bleaching Treatments

In terms of the OPAGO bleaching agent, the participants were instructed on how and when to use the tray correctly; basically, 30 minutes a day for 14 consecutive days, following the manufacturer's recommendations.

In terms of the POD and OPA bleaching agents, alginate impressions (Jeltrate, Dentsply International, Milford, DE, USA) were taken from both dental arches and study models fabricated in dental stone (Type 3, Gesso Pedra, Rio de Janeiro, Brazil). No relief or reservoirs were made on the models, since there is no evidence of any added benefit of these maneuvers on bleaching effectiveness[19] or gingival inflammation.[20] All the teeth in the oral cavity were whitened and included in the manufactured tray. The trays were made in a vacuum laminator (P7, Bio-Art Dental Equipment Ltd. São Carlos, Brazil) using a 1-mm-thick ethylene vinyl acetate (EVA) rubber plate (Soft, Bio-Art Dental Equipment Ltd). Afterward, the trays were trimmed at 3 to 5 mm over the gingiva to provide greater retention and stability without risking gingival irritation.[21] Instructions were given to the participants regarding placement of the gel in the tray and the tray over the teeth. In regard to the POD agent, the participants were instructed to apply this bleach to their teeth for 30 minutes a day, for 14 consecutive days, following the manufacturer's recommendations. In regard to the OPA agent, the participants were instructed to perform this bleaching treatment for 8 hours at night (while sleeping), for 14 consecutive days, following the manufacturer's recommendations.

The subjects in all of the groups were instructed to rinse with water[18] and brush their teeth with the provided toothbrush and toothpaste after wearing the trays. They were instructed to return for follow-up appointments after 7 and 14 days from the start of treatment to monitor the bleaching process and to evaluate tooth color change and other clinical parameters evaluated in this study.

The tooth color obtained from the Vita Classical scale was converted into numeric values, as previously established in the literature,[4,8,9,12] according to an arrangement of colors from number 1 (shade B1) to 16 (shade C4), in order of brightness or value. Thus, the lower the numeric value, the higher the brightness and the whiter the tooth. The conversion was also performed according to degree of brightness from number 1 (OM1) to 29 (5M3) for the Vita Master D shade scale.

After obtaining the values of $\Delta L$, $\Delta a$, and $\Delta b$ for each treatment and time period, the $\Delta E$ was calculated using the following mathematical formula: $\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2}$ where $\Delta E$ is the color change; $\Delta L = L_{final} - L_{initial}$; $\Delta a = a_{final} - a_{initial}$; $\Delta b = b_{final} - b_{initial}$. The value of $\Delta E > 3.3$ was considered clinically noticeable.[23,24]

### Tooth Sensitivity, Gingival Irritation, Treatment Acceptance, and Comfort

An evaluation questionnaire was administered after 7 and 14 days from onset of the whitening treatment. A visual analog scale (VAS) was used to assess tooth sensitivity.[8,22] The patients were asked to draw a vertical line cutting a horizontal line. The scores for the sensitivity levels were 0-1 = no sensitivity; 2-3 = mild sensitivity; 4-6 = moderate sensitivity, 7-8 = severe sensitivity; 9-10 = unbearable sensitivity. The scores to evaluate gingival irritation were absent, localized, or generalized. A VAS was also used to evaluate the degree of acceptance of the bleaching technique and the degree of comfort of the whitening tray used: patients were asked to draw a vertical mark on a horizontal line. The scores used to assess the degree of acceptance to the technique were 0-2 = totally unacceptable; 3-4 = difficult to tolerate; 5-7 = acceptable; and 8-10 = totally acceptable. The scores assessing the degree of comfort were 0-1 = very uncomfortable; 2-4 = uncomfortable; 5-8 = comfortable; and 9-10 = very comfortable. Any other relevant information given by the patients regarding the bleaching treatment for any of the techniques was reported weekly in the questionnaire in a blank space where the patients' exact words were recorded. The information regarding tray loosening/movement and experiences with bleach overflow were collected as a free recording.

### Statistical Analysis

The comparison among groups to determine loss from the study, gender, and gingival irritation was performed using Fisher's exact test. Generalized linear models for repeated measures were used, since the color variable using the Vita Classical shade guide unit and Vita 3D Master shade guide unit ($\Delta$SGU),[4] the acceptance of the technique, the degree of tray comfort, and the $\Delta L$, $\Delta a$, and $\Delta b$ parameters did not meet the assumptions of parametric analyses. There were significant differences in the color shade at the baseline time among the bleaching treatment groups ($p>0.05$), as measured against the Vita Classical and Vita 3D Master scales and verified by generalized linear models for repeated measures. Therefore, comparisons were made by adjustments against the baseline value, which was considered a covariate. Multiple comparisons were performed using the likelihood ratio for the DIFF option of the GENMOD on the SAS program (Release 9.2, 2010, SAS Institute Inc, Cary, NC, USA). The degree of sensitivity and $\Delta E$ were analyzed using mixed models for repeated measures

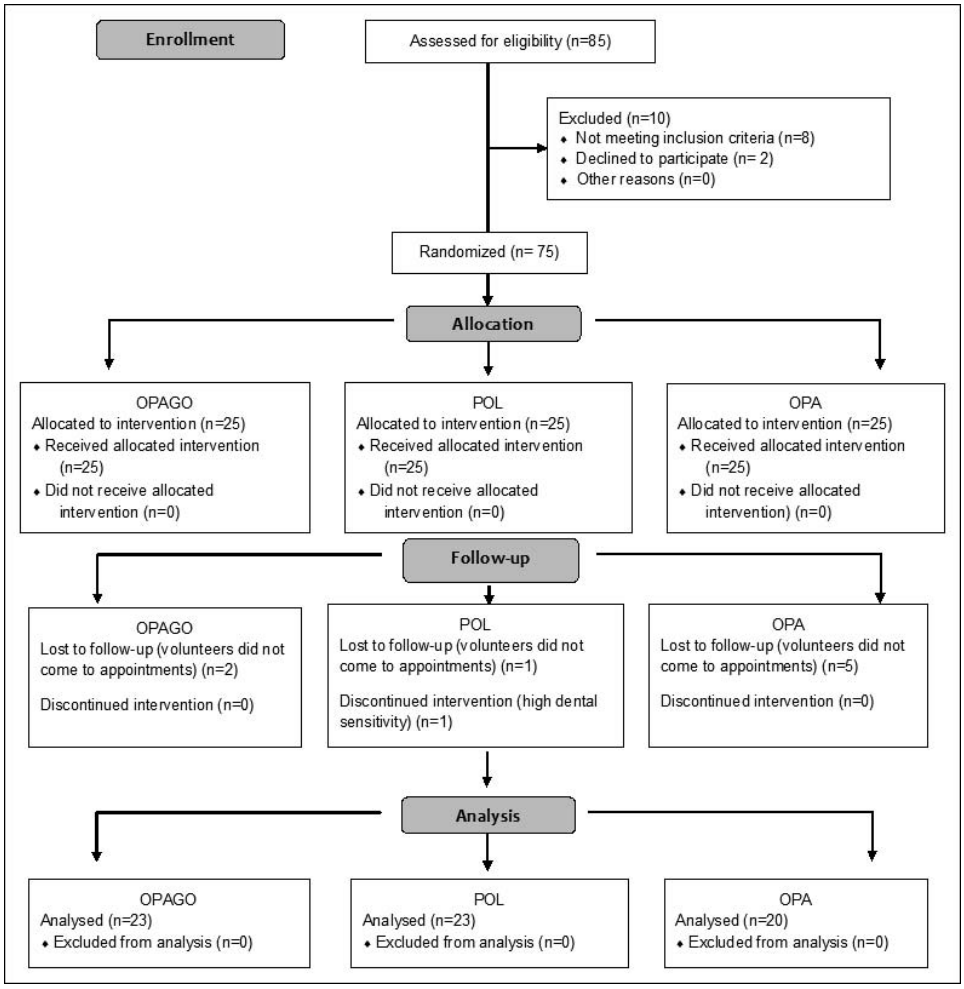


Figure 1. *CONSORT flow diagram of the experiment.*

(PROC MIXED on SAS), and multiple comparisons were performed using the Tukey Kramer test. All the analyses were performed on the SAS software at a significance level of 5%.

## RESULTS

Nine patients were lost similarly among the groups, according to Fisher's exact test ($p=0.6173$). The test showed no significant difference among the groups regarding gender distribution ($p=0.1087$). The causes for loss were related to noncompletion of treatment (withdrawal) or missed appointments for evaluation. Only one patient from the POD group gave up treatment due to very strong tooth sensitivity. The flow chart shows the distribution of patients among the groups and gives the reasons for allocation and dropout (Figure 1).

Regarding gingival irritation, there was no difference between the groups at any time period ($p>0.05$). Most of the patients experienced no gingival irritation during the treatment; however, when it did occur, it was revealed soon after the treatment began (Table 2).

In Table 3, a significant increase in tooth sensitivity was observed ($p<0.05$) over time in all groups, but mild sensitivity prevailed with no significant difference among the groups ($p>0.05$). The frequency of patients having any degree of sensitivity for each group and the level of sensitivity over time are shown by group in Table 4; 85% of the patients from the OPA group reported some degree of sensitivity in the 14 days of treatment.

There was no significant difference between the treatments regarding acceptance of the bleaching technique ($p>0.05$) (Figure 2). The patients reported that the approach used in each group was "completely acceptable," according to the mean values of the scores. The lowest average score regarding degree of comfort was observed for the OPAGO group, which evaluated the prefilled tray as "comfortable," unlike the other groups ($p<0.05$), which indicated the customized tray as "comfortable" to

Table 2: *Frequency and Percentage of Gingival Irritation According to Group and Time Period*

| Time | Group[a] | Gingival Irritation | | | Total | *p* Value |
|---|---|---|---|---|---|---|
| | | Absent | Localized | Generalized | | |
| Baseline | OPAGO | 23 (100.0%) | 0 (0.0%) | 0 (0.0%) | 23 (100.0%) | - |
| | POD | 23 (100.0%) | 0 (0.0%) | 0 (0.0%) | 23 (100.0%) | |
| | OPA | 20 (100.0%) | 0 (0.0%) | 0 (0.0%) | 20 (100.0%) | |
| 7 days | OPAGO | 11 (47.8%) | 11 (47.8%) | 1 (4.4%) | 23 (100.0%) | 0.2540 |
| | POD | 15 (65.2%) | 8 (34.8%) | 0 (0.0%) | 23 (100.0%) | |
| | OPA | 15 (75.0%) | 5 (25.0%) | 0 (0.0%) | 20 (100.0%) | |
| 14 days | OPAGO | 15 (65.2%) | 8 (34.8%) | 0 (0.0%) | 23 (100.0%) | 0.2402 |
| | POD | 16 (69.6%) | 5 (21.7%) | 2 (8.7%) | 23 (100.0%) | |
| | OPA | 17 (85.0%) | 3 (18.8%) | 0 (0.0%) | 20 (100.0%) | |

[a] *The OPAGO was treated with 10% hydrogen peroxide delivered in prefilled disposable trays; the POD group was treated with 9.5% hydrogen peroxide delivered in custom-made trays; the OPA group was treated with 10% carbamide peroxide delivered in custom-made trays.*

"very comfortable" (Table 5). In regard to the OPAGO group, 18 of the 23 patients (78.26%) reported that the gel overflowed from the tray; 10 of the 23 patients (43.47%) also reported that the tray sometimes became loose during the bleaching treatment. No reports of tray loosening or gel overflow were recorded for the other groups.

The mean color score (ΔSGU) from the Vita Classical scale decreased significantly over time ($p<0.05$) for all three treatments (Table 6), showing that there was an increase in brightness with the whitening treatment. At days 7 and 14, the lowest average was observed in the OPA group, which was the group that obtained the highest brightness using this scale; there was no significant difference between the OPAGO and POD groups ($p>0.05$). In terms of the analysis of the mean shade score using the Vita 3D Master scale, the color shade score dropped significantly over time for all three treatments ($p<0.05$), showing that there was an increase in brightness with the treatment. At days 7 and 14, the lowest average score was observed for the OPA group, which was significantly different from the POD group (Table 6).

The results for ΔL at days 7 and 14, compared with baseline, are shown in Table 7, where no significant differences were observed among the groups or between the time periods ($p>0.05$). Likewise, no significant difference for Δa was verified among the groups or between the time periods (Table 7). The Δb average was significantly higher at 14 days ($p<0.05$), but there was no significant difference among the groups ($p>0.05$) (Table 7). There was also no difference among the groups or between the time periods ($p>0.05$) for ΔE (Table 7).

Table 3: *Mean (Standard Deviation) Values of Tooth Sensitivity Score According to Group and Time Period[a]*

| Group[b] | Time | | |
|---|---|---|---|
| | Baseline | 7 Days | 14 Days |
| OPAGO | 0.00 (0.00) Ca | 2.05 (2.34) Ba | 2.87 (2.61) Aa |
| POD | 0.02 (0.08) Ca | 2.62 (2.38) Ba | 2.84 (2.52) Aa |
| OPA | 0.01 (0.04) Ca | 2.34 (2.20) Ba | 3.96 (2.82) Aa |

[a] *Means followed by different letters (capital letters in lines and lower cases in columns) are statistically different ($p\leq0.05$).*
[b] *The OPAGO was treated with 10% hydrogen peroxide delivered in prefilled disposable trays; the POD group was treated with 9.5% hydrogen peroxide delivered in custom-made trays; the OPA group was treated with 10% carbamide peroxide delivered in custom-made trays.*

## DISCUSSION

Opalescence Go bleaching gel is indicated for use with a prescription and under the supervision of a

Table 4: *Frequency and Percentage According to Absence or Presence of Any Type of Tooth Sensitivity Intensity Among Group and Time Period*

| Time | Group[a] | Tooth Sensitivity | | Total |
|---|---|---|---|---|
| | | Absence | Presence | |
| Baseline | OPAGO | 23 (100.0%) | 0 (0.0%) | 23 (100.0%) |
| | POD | 23 (100.0%) | 0 (0.0%) | 23 (100.0%) |
| | OPA | 20 (100.0%) | 0 (0.0%) | 20 (100.0%) |
| 7 days | OPAGO | 11 (47.8%) | 12 (52.2%) | 23 (100.0%) |
| | POD | 5 (21.7%) | 18 (78.3%) | 23 (100.0%) |
| | OPA | 7 (35.0%) | 13 (65.0%) | 20 (100.0%) |
| 14 days | OPAGO | 7 (30.5%) | 16 (65.5%) | 23 (100.0%) |
| | POD | 6 (27.0%) | 17 (73.0%) | 23 (100.0%) |
| | OPA | 3 (15.0%) | 17 (85.0%) | 20 (100.0%) |

[a] *The OPAGO was treated with 10% hydrogen peroxide delivered in prefilled disposable trays; the POD group was treated with 9.5% hydrogen peroxide delivered in custom-made trays; the OPA group was treated with 10% carbamide peroxide delivered in custom-made trays.*

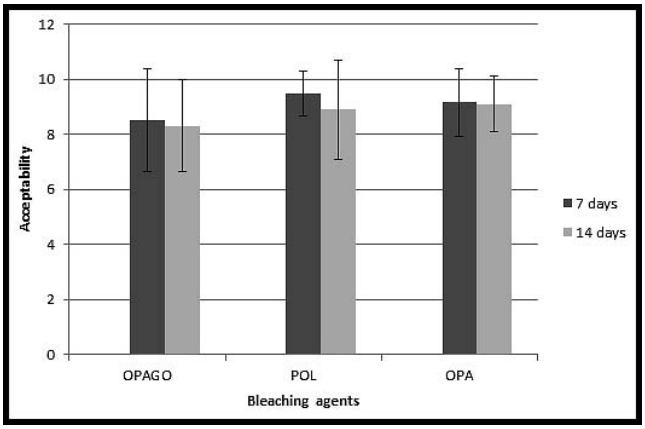


Figure 2. *Mean and standard deviation values of technique acceptability according to group and time periods.*

dentist. This product requires no impressions to be made for customized trays, an advantageous feature that saves customizing time. However, it is important to evaluate how well this alternative approach is accepted in terms of tray comfort before recommending it.

In this study, the null hypothesis tested was accepted. There was no significant difference among the treatments regarding acceptance of the methods by the patients or between the time periods (7 and 14 days). According to the VAS applied, where a value of 10 was considered "totally acceptable," the average acceptance was high (above 8.31), suggesting that the bleaching application methods, regardless of duration (8 hours every night for the OPA or 30 minutes daily for POD and OPAGO), were well accepted by the patients over the number of days needed to accomplish the treatment. The degree of comfort provided by the bleaching tray, however, proved significantly lower for patients in the OPAGO group than in the other groups for both 7 and 14 days of use (average scores of 6.48 and 6.97, respectively, considering that the patients scored this tray as "comfortable"). This degree of comfort could be attributed to the material used to manufacture the tray, which does not allow for tailor fitting or perfect retention to the teeth, thus permitting some movement in the patient's mouth. The whitening gel inside the tray plays a major role in retention of the appliance to the teeth; therefore, detachment of the tray and solubility of the bleaching agent in the saliva may have reduced tray retention, causing the patient some discomfort even for a short period of use (30 minutes). A similar finding was reported by Auschill and others,[25] who observed lower tolerance to the technique when whitening strips were used rather than customized trays. Da Costa and others[26]

Table 5: *Mean (Standard Deviation) Values of Tray Comfort Score According to Group and Time Period*[a]

| Group[b] | Time | | |
|---|---|---|---|
| | Baseline | 7 Days | 14 Days |
| OPAGO | - | 6.48 (2.53) Ab | 6.97 (2.03) Ab |
| POD | - | 8.45 (1.74) Aa | 8.60 (1.81) Aa |
| OPA | - | 8.14 (1.55) Aa | 7.96 (1.90) Aa |

[a] *Means followed by different letters (capital letters in lines and lower cases in columns) are statistically different ($p \leq 0.05$).*
[b] *The OPAGO was treated with 10% hydrogen peroxide delivered in prefilled disposable trays; the POD group was treated with 9.5% hydrogen peroxide delivered in custom-made trays; the OPA group was treated with 10% carbamide peroxide delivered in custom-made trays.*

also reported that patients had problems maintaining the bleaching strip in place, compared with trays, despite reporting that the strips seemed less harmful to the gums than the trays. Despite the trays being thin and fitting well around the teeth, they still became loose, triggered by even the slightest movements of the lips and salivary flow, corroborating the findings by Sundfeld and others[5] who also evaluated the effects of OPAGO. Furthermore, loosening of the tray within the oral cavity and bleach overflow, as experienced by 43.47% and 78.26% of the patients, respectively, at some time in this study, may have contributed to a lesser degree of comfort with this technique, since no reports of these problems were experienced by the patients from the other groups. It should be highlighted, however, that the assigned scores did not discourage the patients from continuing with this method, since none of them withdrew from the treatment. Although the use of a prefilled tray containing a bleaching agent could pose risks to the patient due to the release of peroxide in the oral cavity,[18] the risks may be minimized by the expectorating effect of increased salivary flow induced by the tray in the mouth, and further reduced by rinsing with water after removing the impression tray from the mouth at the end of the session.

Despite tray detachment and bleach overflow—a situation that was not reported by any patient from the OPA and POD groups—no significant difference in gingival irritation was observed among the groups. Localized gingival irritation was observed in all the groups after 7 and 14 days of bleaching, as opposed to its nonoccurrence at onset. It can be suggested that a patient may develop gingival irritation during treatment, irrespective of the tray type (prefilled or customized), agent (carbamide and/or hydrogen peroxide), or application time (30 minutes for OPAGO and POD; 8 hours for OPA).

Table 6: *Mean (Standard Deviation) Values of Color (ΔSGU) Regarding Vita Classical and 3D Master Scales According to Group and Time Period*[a]

| Shade Guide | Group[b] | Time | | |
|---|---|---|---|---|
| | | Baseline (Covariable) | 7 Days | 14 Days |
| Vita Classical | OPAGO | 4.13 (3.21) | 2.30 (1.14) Aa | 1.74 (1.05) Ba |
| | POD | 3.39 (2.29) | 2.09 (0.85) Aa | 1.61 (0.50) Ba |
| | OPA | 2.60 (0.99) | 1.65 (0.49) Ab | 1.15 (0.37) Bb |
| Vita 3D Master | OPAGO | 7.83 (4.21) | 5.35 (2.08) Aab | 4.65 (1.40) Bab |
| | POD | 8.04 (2.62) | 6.00 (1.98) Aa | 5.30 (1.87) Ba |
| | OPA | 7.40 (1.60) | 4.85 (1.63) Ab | 4.25 (0.91) Bb |

*Abbreviation: ΔSGU, Vita Classical shade guide unit and Vita 3D Master shade guide unit.*
[a] *Means followed by different letters (capital letters in lines and lower cases in columns) are statistically different ($p \leq 0.05$) according to each shade guide.*
[b] *The OPAGO was treated with 10% hydrogen peroxide delivered in prefilled disposable trays; the POD group was treated with 9.5% hydrogen peroxide delivered in custom-made trays; the OPA group was treated with 10% carbamide peroxide delivered in custom-made trays.*

Auschill and others[25] also observed no difference in gingival irritation when comparing whitening strips vs trays containing hydrogen peroxide. Leonard and others,[14] however, found that products containing 7% hydrogen peroxide led to higher gingival irritation than those containing carbamide peroxide. One must consider that even a placebo gel in a customized tray may cause gingival irritation. This could be attributed to manufacturing and maladaptation of the tray,[26,27] regardless of whether or not the appliance extended over the gingival tissue.[21] In the present study, localized gingival irritation appeared to be related more to trauma caused by maladaptation of the tray than to the bleaching product itself, which would have caused widespread gingival irritation,[1] or the fact that patients were provided with new toothbrushes at the beginning of the trial, which, despite being soft, may have also contributed to localized gingival irritation.[14,15]

Tooth sensitivity resulting from the bleaching treatment was caused by the penetration and diffusion of peroxides and their by-products, which may have led to inflammatory reactions in the pulp.[28] Between 20% and 60% of patients who undergo home bleaching report this type of symptom.[2,5,9,11,26]. It appears, however, that most people who undergo teeth whitening are able to tolerate the sensitivity caused by this procedure. In this study, only one patient from the POD group gave up treatment due to intense sensitivity. An increase in tooth sensitivity was observed over time for all the groups, despite being described as mild, regardless of the bleaching agent concentration and treatment application time. This finding was also observed by Alonso de la Peña and López Ratón[11] when using various concentrations of hydrogen or carbamide peroxide in customized trays. Some authors[7,12,27] also showed that no difference in tooth sensitivity was reported by participants for 10% and 15%-16% carbamide peroxide agents. It must be mentioned that the bleaching agents assessed had desensitizing components in their composition, such as potassium nitrate (OPAGO and POD) or sodium fluoride (OPA), which would have led to lower levels of sensitivity. In this regard, a meta-analysis carried out by Wang and others[28] reported that the addition of potassium nitrate or sodium fluoride to the composition of bleaching products for home use led to a decrease in tooth sensitivity. Although sensitivity to bleaching agents cannot be prevented altogether, Browning and others[6] and Navarra and others[29] also observed no less intense sensitivity when using a carbamide peroxide product containing sodium fluoride and potassium nitrate compared with the agent without these components. Sundfeld and others[15] found that no patients reported sensitivity when using the OPAGO bleaching agent for a period of 8 days. One should also consider that, although there were some differences in the hydrogen and carbamide peroxide concentrations in the agents studied, tooth sensitivity is a symptom that may vary from person to person.[5,9] While there are studies showing that higher sensitivity may be expected when using agents with higher concentrations,[7-9,14] it is difficult to predict the occurrence and intensity during treatment.

Regarding color change, various methods may be used to assess changes in tooth color upon completion of whitening treatments. The shade scales routinely used by professionals in the office, such as the Vita Classical and Vita 3D Master, are considered subjective assessment methods. Nonetheless, they are frequently used by dentists to show their patients the color of their teeth at the beginning of the treatment and to compare it against the final color at the end of the treatment. This

comparison facilitates understanding and communication between patient and professional. Shade scales have also been used in studies that evaluate different color shifts when performing whitening treatments.[4,8,9,11] The use of objective assessment methods, such as a spectrophotometer, in which the measurements can be obtained by the CIELab system, are important not only to evaluate color change in general but also to evaluate how the bleaching treatment may influence the color of the teeth within each light spectra (L), green-red axis (a) and blue-yellow axis (b), as used in this study.

Both the spectrophotometer and the Vita Classical scale revealed that all the bleaching agents were effective in whitening the teeth, with enhanced brightness from 7 to 14 days. A smaller tooth color change was observed for the Vita Classical shade guide in the present study than in other clinical trials[4,8,9,12] due to the lighter color shades at baseline observed for our volunteers (aged 18 to 30 years). Nevertheless, whiter teeth (higher brightness) were obtained when using the OPA bleaching agent in both treatment periods, probably due to the longer application time (8 hours) compared with the other techniques. On the other hand, both the OPA and the OPAGO systems showed similar outcomes in terms of brightness at days 7 and 14, according to the Vita 3D Master scale, despite the effectiveness of various peroxide-based agents in increasing brightness. Any differences could have been related to characteristics inherent to the evaluation method, since the Vita 3D Master scale has a greater number of shades (29 shades vs 16 for the Vita Classical scale), as well as to standardization of brightness by the groups in terms of chroma and hue, which makes Vita 3D Master more suitable for use due to greater uniformity between shades and proximity to reality.[11,17]

Conversely, no difference was observed among ΔL, Δa and ΔE values, between times or bleaching products, when using the objective evaluation by the CIELab system. Thus, it may be inferred that all bleaching agents had the same effect on tooth brightness (L), according to the red-green axis (a) and shade change (E), despite differences in bleach concentration, active agent (carbamide peroxide, hydrogen peroxide, or both) and time of use, with no differences among the results at each time period. The ΔE value obtained from the different products was greater than 3.3 at both evaluation periods; and ΔE values higher than 3.3 are accepted as clinically noticeable, demonstrating the effectiveness of the bleaching procedures,[23,24] especially at day 7. Other studies have also shown that the result for different bleaching agents containing carbamide or hydrogen peroxide in different concentrations for home use did not differ in ΔL or ΔE.[11,22,26]

Differences between time periods were observed only for Δb, which represents changes in the blue-yellow axis, showing a more significant decrease in yellow hues at day 14 than at day 7 for all the products studied. A reduction in parameter b has been reported as the most important indicator of shade change during bleaching, since it occurs more rapidly and to a greater extent than the other components of the CIELab system.[7,30] Therefore, combining this with the fact that no difference was observed in color change (ΔE) compared with baseline, it may be suggested that the bleaching agents evaluated may be indicated for only 7 days of use, thus preventing other signs and symptoms resulting from long-term use, such as gingival irritation and tooth sensitivity.

## CONCLUSIONS

The bleaching methods using carbamide peroxide or hydrogen peroxide dispensed in customized or prefilled trays had high acceptance by patients. The prefilled trays were less comfortable than customized trays. Gingival irritation was localized and similar across all the bleaching methods. Tooth sensitivity increased over time in all groups; however, it was reported as mild, regardless of the bleaching agent concentration and the application time. The whitening effect was similar for all bleaching agents at 7 and 14 days, except for parameter b, for which the highest reduction in yellow hue occurred at day 14.

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#### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of Centro de Pós Graduação São Leopoldo Mandic. The approval code for this study is 32499014.1.0000.5374.

#### 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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# Longevity, Esthetic Perception, and Psychosocial Impact of Teeth Bleaching by Low (6%) Hydrogen Peroxide Concentration for In-office Treatment: A Randomized Clinical Trial

E Fernández • C Bersezio • J Bottner • F Avalos • I Godoy • D Inda
P Vildósola • JRC Saad • OB Oliveira Jr • J Martín

### Clinical Relevance

In office bleaching with 6% hydrogen peroxide catalyzed by titanium dioxide that is activated with a hybrid light (blue LED/infrared laser) achieves clinical effectiveness at nine months and has a positive dental confidence and psychosocial impact on patients.

## SUMMARY

**Objective: The aim was to evaluate the color longevity after nine months of in-office bleaching with gel (6% hydrogen peroxide), to compare this to a control concentration of 35% in a split-mouth study model, and to assess the dental confidence and psychosocial impact on patients.**

**Methods and Materials: Twenty-seven patients were assessed at the nine-month recall. The bleaching procedure with 6% or 35% hydrogen**

Cristian Bersezio, postgraduate student, Restorative Dentistry, University of Chile, Santiago, Chile and Restorative Dentistry, São Paulo State University, Araraquara, Brazil

Jessica Bottner, Restorative Dentistry, University of Chile, Santiago, Chile

Francisca Avalos, Restorative Dentistry, University of Chile, Santiago, Chile

Isidora Godoy, Restorative Dentistry, University of Chile, Santiago, Chile

Diego Inda, Restorative Dentistry, University of Chile, Santiago, Chile

Patricio Vildósola, Restorative Dentistry, University of Chile, Santiago, Chile

José RC Saad, Restorative Dentistry, São Paulo State University, Araraquara, Brazil

Osmir Batista de Oliveira Jr, Restorative Dentistry, São Paulo State University, Araraquara, Brazil

Javier Martín, DDS, Odontología Restauradora, Universidad de Chile, Santiago, Chile

*Eduardo Fernández, DDS, DS, PhD, Restorative Dentistry, University of Chile, Santiago, Chile

*Corresponding author: 11 de Septiembre 1881 of 2108, Santiago, Chile, 7500505. e-mail: edofdez@yahoo.com

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**peroxide gel was performed randomly in the upper hemi-arch of each patient. The color was measured at baseline and at one week, one month, and nine months after the procedure, using the Vita Easyshade spectrophotometer, the Vita classical shade guide organized by value, and Vita Bleach Guide 3DMaster. Moreover, two surveys, OHIP-Esthetics and PIDAQ, were used to assess the esthetic self-perception and psychosocial impact of the bleaching procedure. During the nine-month recall, the color was assessed before and after dental prophylaxis.**

**Results: Twenty-seven patients participated in the nine-month recall. There was a significant difference in ΔE between the two groups at all times assessed ($p<0.011$). The ΔL, Δa, and Δb showed a difference between the two groups at all times assessed ($p<0.038$), except for ΔL from the baseline vs nine-month after prophylaxis value ($p>0.20$). There was no significant difference in ΔSGU at all times ($p>0.05$). There was a significant difference in OHIP-Esthetics and PIDAQ sums compared with baseline scores ($p<0.03$).**

**Conclusion: The two compounds remained effective at nine months, with a slight rebound of color, and maintained their objective color difference but not the subjective color difference. Patients were satisfied with the bleaching procedure, and this had a positive impact on esthetic perception and a positive psychosocial impact at the nine-month recall.**

## INTRODUCTION

Dental bleaching is currently the treatment of choice for extrinsic discoloration pigmentation because it is quick, minimally invasive, and relatively inexpensive.[1] Recently, several studies have reported the effectiveness of bleaching gels with lower concentrations,[2] and there are *in vitro* studies that support lower cell damage at these low concentrations of peroxide.[3] There has been some research into bleaching gels catalyzed by agents such as titanium dioxide nanoparticles activated by hybrid light (laser/LED) with different concentrations (15%).[4] These concentrations show similar effectiveness, and in some cases, much lower adverse post-procedure effects.[4]

Information about the longevity of bleaching in the literature is somewhat controversial. Some studies have shown a marked rebound of color; others show only a slight difference.[5-7] Moreover, the regression continues with the passage of time. All of these reports are related to concentrations of gels higher than 10% hydrogen peroxide, with only one report[8] at 6%. This report by Vano and others indicates that the patients did not achieve a change of at least five units of ΔE intially and showed a color rebound near 50% at nine months.[8] It is important for clinicians to know about the new in-office concentrations and to correlate these with patients.

Patient expectations regarding dental bleaching are very important and poorly described in the literature. This is specifically true for effects on esthetic perception and other factors such as the psychosocial impact. A recent study by Martin and others indicated a positive effect on esthetic perception and psychosocial discomfort factors by the Oral Health Impact Profile (OHIP-Esthetics) at the one-month recall after bleaching.[9] It would be interesting for clinicians to know whether this effect is stable over time.

The objective of this trial was to show the longevity and effect on esthetic perception and psychosocial impact of bleaching by 6% hydrogen peroxide gel catalyzed by titanium dioxide nanoparticles and activated by hybrid light. The longevity of the color change was compared with that of a control concentration of 35% in a split-mouth study model. The first null hypothesis of this study was that the longevity of the color compared before and after dental prophylaxis at nine months after tooth bleaching is the same between the two gel methods. The second null hypothesis was that there is no effect on the esthetic perception or psychosocial impact on patients at nine months vs baseline.

## METHODS AND MATERIALS

This clinical study was approved by the Ethics Committee of the local Faculty of Dentistry where the study took place between July 2014 and September 2015. It is registered on the site of the Clinical Trials Registry (NCT02353611) and was conducted according to the Consolidated Standards of Reporting Trials Statement and Helsinki Declaration of 1975 revised in 2000.

Thirty-two volunteers were selected and received a dental prophylaxis and oral hygiene instructions one week prior to the beginning of this study to achieve similar oral conditions. They also freely signed an informed consent.

### Study Design

This was a randomized, double-blind (patients and evaluator), and split-mouth design (half of the dental arch, either left or right). One site was treated by compound 1 and the other by compound 2: these were randomly assigned. The patients were invited to participate in the study through posters put up around the city or recruited from participants in other studies in the same department who were contacted by email or phone.

A total of 131 patients were screened according to the inclusion and exclusion criteria. All subjects were over 18 years of age. Participants received a dental prophylaxis with pumice and water to determine whether they met the eligibility criteria: two central incisors with at least shade A2 or darker assessed by comparison with a value-oriented shade guide (Vita classical, Vita Zahnfabrik, Bad Sackingen, Germany), as well as anterior teeth without restorations, previous bleaching procedures, cervical lesions, or dental pain. Patients were excluded if they were pregnant or lactating; had moderate or severe fluorosis, tetracycline stains, orthodontic treatment, periodontal disease, orofacial tumors, trauma, or tooth malformation; or were taking analgesic, anti-inflammatory, or antibiotic drugs. There were 31 patients; one patient was excluded from the analysis due to missed appointments. Twenty-seven patients were assessed at nine months (Figure 1).

Two trained operators (restorative dentistry professors) administered the bleaching treatments. A third participant who did not have contact with the patients was responsible for conducting the randomization. The allocation of the groups was performed by random drawing using Microsoft Excel 2010 (Microsoft) from codes assigned to each participant.

There were two experimental groups: group A was the experimental group treated with a 6% hydrogen peroxide compound (HP6) catalyzed by titanium oxide nanoparticles that were activated with a blue hybrid light with an infrared laser. Group B acted as a control, and a 35% hydrogen peroxide bleaching compound was applied to the maxillary group of teeth. (The design of the lamp [whole mouth] did not isolate the radiation of each group.)

The following procedures were adopted to ensure double blinding: 1) labels, logos, packaging, and any other factors that could identify the products were removed, and procedures and instruments were standardized; 2) the bleaching protocol was performed in a different room from where the evaluator examined the patients; 3) the randomization was alpha-numerically coded to ensure blinding of the research team; and 4) a statistician received data tabulated in code that did not allow for identification of the treatment applied to each group.

### Sample Size Calculation

The primary outcome of this study was the efficacy determined by color alteration (ΔE). Previous studies showed that the use of in-office bleaching agents containing 35% hydrogen peroxide (HP35) with or without LED/laser light leads to a ΔE value of 2.0-7.0 after two bleaching sessions.[2,4,10] At least 28 subjects were needed for 80% power in detecting significance at the 5% level and a (1-β) of 0.90, and considering a change in the primary outcome measure from seven in the control group to five in the experimental group. Due to a higher dropout rate in the last two clinical studies of our research group (5% and 10%), we decided to add more patients for a total of 31 subjects.

### Bleaching Protocol

In each session, volunteers received prophylaxis with pumice powder and water. Next, gingival tissue was protected using a light-cured resin gum barrier applied according to the manufacturer's instructions (Lase Protect, DMC, São Carlos, SP, Brazil). Both bleaching agents (Lase Peroxide Flex 6% and Lase Peroxide Sensy 35%, DMC) were prepared by mixing hydrogen peroxide and thickening compounds according to the manufacturer's instructions (with three peroxide drops to one drop of thickener). The resulting gels (high viscosity allowing excellent control and avoiding contact with the neighboring tooth) were distributed uniformly on the surfaces of the teeth. Eight teeth between the second premolars were bleached for each patient. In each bleaching session, the bleaching gels were applied twice for 12 minutes each. In each application, the surface of the gel was light activated with continuous irradiance for 12 minutes using a LED/laser hybrid cold-light with a total power of 1500 mW (Bleaching Lase Plus, DMC). Three bleaching sessions were completed for the patients; the interval between sessions was seven days. The total contact time was 72 minutes for the bleaching treatments.

### Objective Evaluation

Two evaluators measured the tooth color at baseline and again at one week, one month, and nine months. The color evaluation was obtained from an area of 6 mm located in the middle third of the labial surface

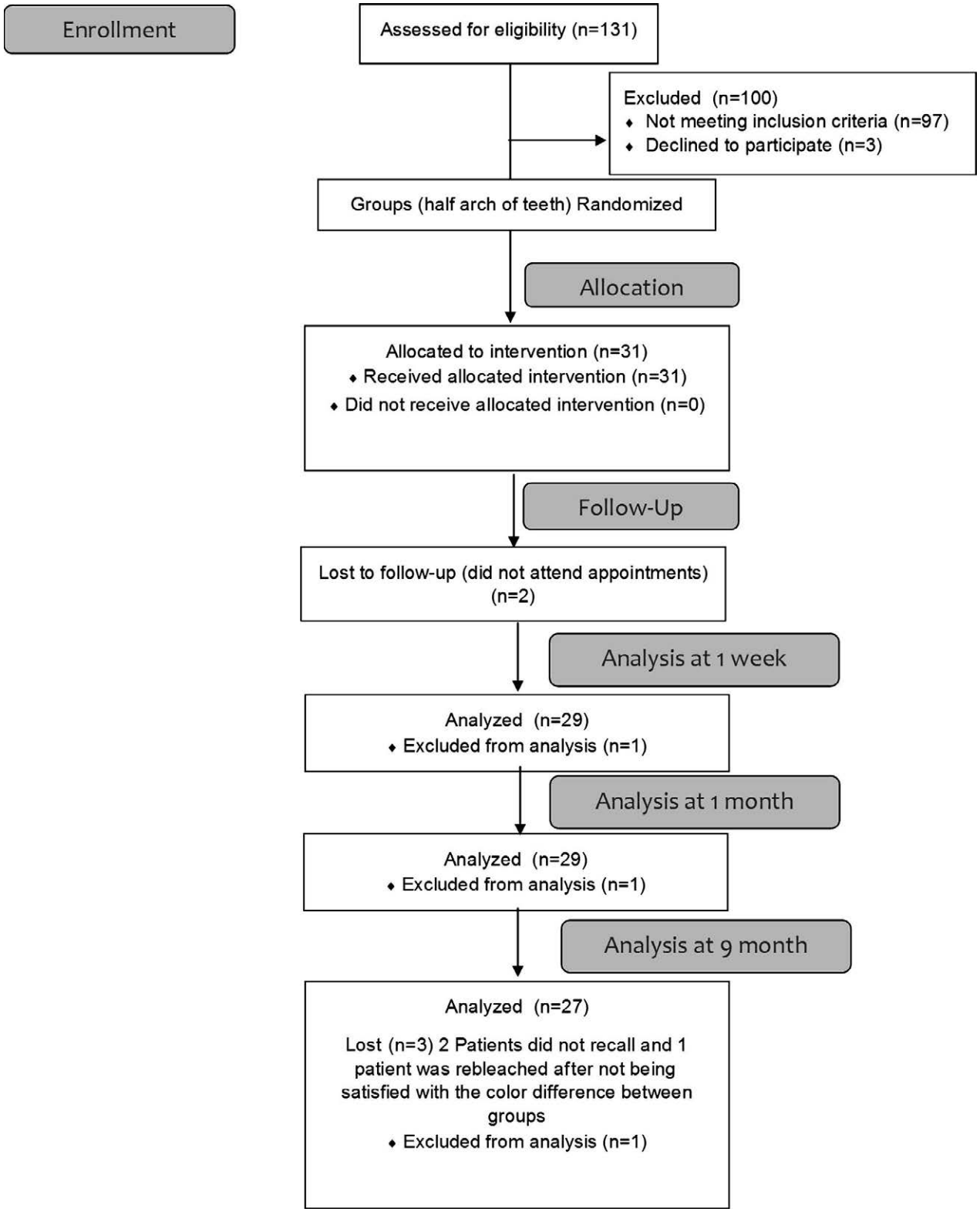


Figure 1. *CONSORT flow diagram.*

of the left and right central incisors. To standardize this evaluation, an impression of the maxillary arch was taken to make a guide using high-viscosity silicone putty (Zetaplus, Zhermack, Badia Polesine, Rovigo, Italy). A window was created on the labial surface in the middle third of the central incisor using a device with well-formed borders and a 3-mm radius corresponding to the diameter of the active part of the spectrophotometer (Vita EasyShade Compact, Vita Zahnfabrik). This device has 96% reliability.[11] The shade was determined using L*, a*, and b*. The color alteration after each session was given by the differences between the values obtained at the session and baseline. The ΔE was calculated using the following formula: $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$.

### Subjective Evaluation

For the subjective evaluation, two calibrated evaluators ($\kappa$=0.85) under standardized light conditions (same place, hour, patient position, natural light source, assessed between 10 am and 3 pm) used the 16 tabs of the Vita classical shade guide (Vita Zahnfabrik) and 15 tabs of the Vita Bleach shade guide (Vita Zahnfabrik). Although the Vita classical scale is not linear in the truest sense, we treated the changes as continuous with a linear ranking. This was done similar to several previous clinical trials on dental bleaching.[12] The evaluators recorded the shade of the maxillary left and right central incisors at baseline during the same period as the objective evaluation.

We checked the color in the middle third area of the labial surface of the central incisors according to the American Dental Association guidelines.[13] We calculated the color changes from the beginning of the active phase through the individual recall times by changing the number of shade guide units (ΔSGU) that occurred toward the lighter end of the value-oriented list of shade tabs. In the event that the operators disagreed on color matching, a consensus was reached prior to dismissing the patient. At nine months, the evaluation was performed before and after dental prophylaxis with a Robinson brush and prophylaxis paste (Herjos, Vigodent Coltene SA Indústria e Comércio, Rio de Janeiro, Brazil). After dental prophylaxis, the treated teeth were rehydrated in the patient's mouth for 15 minutes before color assessment.

### Oral Health Impact Profile (OHIP-Esthetics) Questionnaire

Satisfaction was measured using the OHIP-Esthetics questionnaire validated in Chilean Spanish (Table 1).[14] The questionnaire was administered by a research operator at baseline, at one week, one month, and nine months (before prophylaxis) after bleaching. Each statement was accompanied by a Likert-type scale that generated a score ranging from 0 to 4 (never = 0, hardly ever = 1, occasionally = 2, fairly often = 3, and very often = 4). These individual scores were added to give a summary score ranging from 0 (minimum) to 56 (maximum). The outcomes were defined as the sum of the OHIP-Esthetics and dimension scores. The internal consistency was evaluated using the Cronbach $\alpha$ test.

### The Psychosocial Impact of Dental Esthetics Questionnaire (PIDAQ)

The questionnaire consisted of 23 items (Table 1) grouped into four components using factor analysis: 1) dental self-confidence; 2) social impact; 3) psychosocial impact; and 4) esthetic concern.[15] The first factor, dental self-confidence, consisted of six items from the self-confidence scale. The second factor, social impact, contained eight items from the social aspects scale of the quality-of-life questionnaire. The third factor, psychosocial impact, was derived from six formulated items relating mainly to the psychosocial impact of dental esthetics. The fourth factor was the esthetics. The patient was asked to evaluate the items using a five-point Likert scale with numerical values 0 = not at all, 1 = a little, 2 = somewhat, 3 = strongly, and 4 = very strongly. The questionnaire was administered by a research operator at baseline, one week, one month, and nine months (before prophylaxis) after bleaching. This was validated in Spanish, with a confidence reported by a Cronbach $\alpha$ of 0.90.[16] The outcomes were defined as the sum of the PIDAQ questionnaire and factor scores; the internal consistency was evaluated using the Cronbach $\alpha$ test.

### Habits and Dietary Survey

A brief survey of habits was conducted. This review included questions regarding the use of toothpastes with whitening agents, drinks that could generate stains, and smoking.

### Statistical Analysis

After verifying the normality of the data distribution and the homogeneity of the variance-covariance matrix, the efficacy of the treatments was evaluated with respect to color alteration (ΔE and ΔSGU) and analyzed by the Wilcoxon test for within-group comparisons and the Mann-Whitney test for be-

Table 1: *OHIP-Esthetics and PIDAQ Questionnaires*

| | |
|---|---|
| | **OHIP-Esthetics Questionnaire** |
| Q1 | Have you noticed a tooth which doesn't look right?[1] |
| Q2 | Have you felt that your appearance has been affected by problems with your teeth?[1] |
| Q3 | Have you had sensitive teeth for example to heat or to cold food or drinks?[2] |
| Q4 | Have you had painful areas in your mouth?[2] |
| Q5 | Have you been self-conscious because of your teeth?[3] |
| Q6 | Have you felt uncomfortable about the appearance of your teeth?[3] |
| Q7 | Have you felt that your food is less tasty because of problems with your teeth?[4] |
| Q8 | Have you avoided smiling because of problems with your teeth?[4] |
| Q9 | Have you found it difficult to relax because of problems with your teeth?[5] |
| Q10 | Have you been a bit embarrassed because of problems with your teeth?[5] |
| Q11 | Have you been less tolerant of your spouse or family because of problems with your teeth?[6] |
| Q12 | Have you had difficulties doing your usual job because of problems with your teeth?[6] |
| Q13 | Have you been unable to enjoy the company of other people very much because of problems with your teeth?[7] |
| Q14 | Have you felt that life in general was less satisfying because of problems with your teeth?[7] |
| | **PIDAQ Questionnaire** |
| | *Dental Self-Confidence* |
| 1 | I am proud of my teeth. |
| 2 | I like to show my teeth when I smile. |
| 3 | I am pleased when I see my teeth in the mirror. |
| 4 | My teeth are attractive to others. |
| 5 | I am satisfied with the appearance of my teeth. |
| 6 | I find my tooth position to be very nice. |
| | *Social Impact* |
| 7 | I hold myself back when I smile so my teeth don't show so much. |
| 8 | If I don't know people well I am sometimes concerned what they might think about my teeth. |
| 9 | I'm afraid other people could make offensive remarks about my teeth. |
| 10 | I am somewhat inhibited in social contacts because of my teeth. |
| 11 | I sometimes catch myself holding my hand in front of my mouth to hide my teeth. |
| 12 | Sometimes I think people are staring at my teeth. |
| 13 | Remarks about my teeth irritate me even when they are meant jokingly. |
| 14 | I sometimes worry about what members of the opposite sex think about my teeth. |
| | *Psychological Impact* |
| 15 | I envy the nice teeth of other people. |
| 16 | I am somewhat distressed when I see other people's teeth. |
| 17 | Sometimes I am somewhat unhappy about the appearance of my teeth. |
| 18 | I think most people I know have nicer teeth than I do. |
| 18 | I feel bad when I think about what my teeth look like. |
| 20 | I wish my teeth looked better. |
| | *Aesthetic Concern* |
| 21 | I don't like to see my teeth in the mirror. |
| 22 | I don't like to see my teeth in photographs. |
| 23 | I don't like to see my teeth when I look at a video of myself. |

*In the OHIP-Esthetics questionnaire, numbers correspond to the dimensions (1 = functional limitation, 2 = physical pain, 3 = psychological discomfort, 4 = physical disability, 5 = psychological disability, 6 = social disability, 7 = handicap).*

tween-group comparisons. The statistical analyses were performed using SPSS 23.0 (SPSS Inc, Chicago, IL, USA) with $\alpha$=0.05. For comparison of OHIP-Esthetics and PIDAQ questionnaires scores, the Wilcoxon test was used.[17]

## RESULTS

### Baseline Characteristics

Of 131 patients examined, 31 patients were enrolled. Three patients did not continue, and one patient was

Table 2: *Baseline Color Features of Volunteers*

| | L value (mean ± SD) | 95% confidence interval | | a* value (mean ± SD) | 95% confidence interval | | b* value (mean ± SD) | 95% confidence interval | | SGU value (mean ± SD) | 95% confidence interval | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Upper limit | Lower limit | | Upper limit | Lower limit | | Upper limit | Lower limit | | Upper limit | Lower limit |
| Group A | 84.68 ± 4.29 | 86.38 | 82.98 | −0.39 ± 1.53 | −0.22 | −0.99 | 24.20 ± 4.17 | 25.85 | 22.56 | 6.81 ± 2.22 | 7.69 | 5.94 |
| Group B | 84.44 ± 4.59 | 86.25 | 82.62 | −0.38 ± 1.26 | −0.12 | 0.88 | 24.09 ± 3.69 | 25.56 | 22.64 | 6.93 ± 2.25 | 7.82 | 6.04 |

excluded from analysis in the monitoring at nine months (for not coming at the right time for evaluations). The sample consisted of 10 women (37.04%) and 17 men (62.96%), with average ages of 24.7 ± 5.86 years for men and 23.1 ± 2.81 years for women. The entire cohort was 24.1 ± 4.95 years of age. Features of color at baseline are shown in Table 2.

## Per-protocol Versus Intention-to-Treat Analysis

All statistical analyses were performed with data imputation for missing outcomes (intention to treat) and without data imputation (per protocol). The same overall conclusions were reached (data not shown) in all analyses. To avoid data repetition, we describe only the results obtained per-protocol analysis.

## Spectrophotometer Data

Color changes measured by units of ΔE, ΔL, Δa, and Δb from baseline are shown in Table 3. There was a significant ΔE difference according to the Mann-Whitney test between the two groups at all times assessed ($p<0.011$). There was also a color difference between the groups after one week and one month, with a noticeable difference greater than two units of ΔE suggesting a difference at the nine-month point. The ΔL, Δa, and Δb were different according to the Mann-Whitney test between the two groups at all times assessed ($p<0.038$) except for ΔL from baseline vs nine-month preprophylaxis value ($p>0.20$). To corroborate the statistical power and size effect, this

Table 3: *Changes of Color by ΔE, ΔL, Δa, and Δb (Δ Calculated From the Baseline Value) by Group in Different Periods Expressed by Mean, SD, Statistical Significance (p<0.05 in bold), Effect Size, and Statistical Power*

| | Group A | Group B | Mann-Whitney (*p*) | Effect size d | Power (1 − β) |
|---|---|---|---|---|---|
| ΔE | | | | | |
| Baseline vs week | 7.88 ± 10.49 | 8.19 ± 2.73 | **0.004** | 0.04 | 0.07 |
| Baseline vs month | 5.86 ± 3.79 | 8.24 ± 2.45 | **0.000** | 0.75 | **0.84** |
| Baseline vs 9-month PrePr | 6.03 ± 4.05 | 7.64 ± 2.64 | **0.011** | 0.47 | 0.51 |
| Baseline vs 9-month PostPr | 5.14 ± 3.49 | 7.81 ± 2.32 | **0.000** | 0.90 | **0.94** |
| ΔL | | | | | |
| Baseline vs week | 2.00 ± 3.51 | 3.75 ± 3.21 | **0.016** | 0.52 | 0.58 |
| Baseline vs month | 2.62 ± 4.06 | 4.10 ± 3.12 | **0.033** | 0.41 | 0.42 |
| Baseline vs 9-month PrePr | 3.06 ± 4.53 | 3.83 ± 3.71 | 0.279 | 0.19 | 0.16 |
| Baseline vs 9-month PostPr | 2.71 ± 3.16 | 4.50 ± 2.78 | **0.010** | 0.60 | 0.69 |
| Δa | | | | | |
| Baseline vs week | −0.82 ± 1.07 | −1.38 ± 0.74 | **0.038** | 0.61 | 0.70 |
| Baseline vs month | −0.77 ± 1.10 | −1.33 ± 0.81 | **0.015** | 0.58 | 0.66 |
| Baseline vs 9-month PrePr | −0.39 ± 1.28 | −1.00 ± 0.74 | **0.007** | 0.58 | 0.66 |
| Baseline vs 9-month PostPr | −0.50 ± 1.11 | −1.08 ± 0.74 | **0.019** | 0.62 | 0.70 |
| Δb | | | | | |
| Baseline vs week | −1.48 ± 12.41 | −6.14 ± 3.25 | **0.005** | 0.51 | 0.57 |
| Baseline vs month | −3.48 ± 3.48 | −6.14 ± 2.76 | **0.001** | 0.85 | **0.91** |
| Baseline vs 9-month PrePr | −2.82 ± 3.72 | −5.23 ± 2.92 | **0.002** | 0.72 | **0.82** |
| Baseline vs 9-month PostPr | −2.78 ± 3.53 | −5.44 ± 2.74 | **0.001** | 0.84 | **0.91** |

Table 4: *Changes of Color by ΔSGU (Vita classical and Vita Bleach Guide 3D-Master ) by Group in Different Time Frames Expressed by Median (Minimum/Maximum Value), Statistical Significance, Effect Size, and Statistical Power*

| | Group A | Group B | Mann-Whitney (*p*) | Effect size | Power (1 − β) |
|---|---|---|---|---|---|
| Vita Classic | | | | | |
| Baseline vs week | 4 (Min 2/Max 9) | 4 (Min 2/Max 9) | 0.655 | 0.10 | 0.10 |
| Baseline vs month | 4 (Min 2/Max 9) | 4 (Min 2/Max 9) | 0.672 | 0.10 | 0.10 |
| Baseline vs 9-month PrePr | 3 (Min 0/Max 9) | 4 (Min 2/Max 10) | 0.128 | 0.33 | 0.32 |
| Baseline vs 9-month PostPr | 4 (Min 0/Max 9) | 4 (Min 2/Max 10) | 0.186 | 0.33 | 0.31 |
| Vita Bleach Guide 3D-Master | | | | | |
| Baseline vs week | 3 (Min 1/Max 6) | 4 (Min 1/Max 6) | 0.253 | 0.32 | 0.30 |
| Baseline vs month | 3 (Min −1/Max 5) | 3 (Min 0/Max 6) | 0.136 | 0.42 | 0.45 |
| Baseline vs 9-month PrePr | 2 (Min −2/Max 4) | 3 (Min −1/Max 5) | 0.047 | 0.55 | 0.64 |
| Baseline vs 9-month PostPr | 2 (Min −2/Max 4) | 3 (Min −1/Max 5) | 0.020 | 0.64 | 0.75 |

outcome was calculated post hoc with the ΔE values by G-Power software.[18] All values showed a statistically significant difference vs baseline ($p<0.05$) by Wilcoxon test.

### Shade Guide Data

Color changes measured subjectively as expressed by ΔSGU are shown in Table 4. For Vita classical, there was no significant difference between the different evaluations ($p>0.10$). In contrast, there were significant differences by Vita Bleach Guide 3D Master ($p<0.02$) at the nine-month recall.

### OHIP-Esthetics

The OHIP-Esthetics survey scores (Table 5) were significant at different times when comparing the initial baseline survey prior to the treatment and a week after bleaching ($p=0.006$). This was replicated after one and nine months ($p<0.001$) to obtain more reliable data. The results were significant ($p<0.03$). Specifically, there was a statistically significant difference after one week in dimensions. The functional limitation and psychosocial discomfort were statistically significant at one month in the psychosocial discomfort dimension. The ninth month showed statistically significant differences in all factors ($p<0.03$) except physical pain and handicap factors as shown in Table 5. There was good internal consistency as shown by the Cronbach α (0.803).

### PIDAQ

The overall score on the PIDAQ was statistically significant at all times ($p<0.001$; Table 6).

### Habits and Diet Survey

Of the 27 patients, 11 (40.74%) were smokers, with a mean of 4.23 cigarettes per day; 22 patients (81.48%) consumed tea, coffee, or cola (mean of 1.95 times per day); 10 patients (37.04%) used bleaching toothpastes (mean of 2.80 times per day).

Table 5: *Distribution of Scores by Dimension and for the Total OHIP-14 (Oral Health Impact Profile in Spanish) Expressed in Mean and SD, Repeatability, and Internal Consistency*

| | Baseline | 1 week after bleaching | 1 month after bleaching | 9 months after bleaching | Corrected item total correlation of sum | Cronbach α if item deleted | Cronbach α | Number of items |
|---|---|---|---|---|---|---|---|---|
| OHIP-EE-14 | 26.33 ± 7.30 | 24.77 ± 6.57* | 24.57 ± 6.90* | 23.87 ± 6,31* | | | 0.803 | 7 |
| Functional limitation | 4.90 ± 1.83 | 4.37 ± 1.56* | 4.53 ± 1.85 | 4.17 ± 1.78* | 0.704 | 0.742 | | |
| Physical pain | 4.27 ± 1.53 | 4.20 ± 1.61 | 4.27 ± 1.68 | 4.70 ± 1.62 | 0.098 | 0.859 | | |
| Psychological discomfort | 5.70 ± 1.37 | 5.20 ± 1.37* | 4.93 ± 1.53* | 5.13 ± 1.38* | 0.513 | 0.781 | | |
| Physical disability | 2.83 ± 1.12 | 2.73 ± 0.98 | 2.67 ± 0.92 | 2.50 ± 0.86* | 0.746 | 0.757 | | |
| Psychological disability | 3.37 ± 1.85 | 3.20 ± 1.73 | 3.23 ± 1.55 | 2.87 ± 1.50* | 0.748 | 0.732 | | |
| Social disability | 2.60 ± 1.28 | 2.53 ± 1.22 | 2.50 ± 1.28 | 2.13 ± 0.43* | 0.470 | 0.789 | | |
| Handicap | 2.67 ± 1.47 | 2.53 ± 1.38 | 2.43 ± 1.07 | 2.37 ± 1.19 | 0.688 | 0.752 | | |

** p≤0.03 compared with baseline by Wilcoxon test.*

Table 6: *Distribution of Scores by Dimension and for the Total PIDAQ (Psychosocial Impact of Dental Aesthetics Questionnaire in Spanish) Expressed in Mean and SD*

| | Baseline | 1 week after bleaching | 1 month after bleaching | 9 months after bleaching | Cronbach α |
|---|---|---|---|---|---|
| PIDAQ | 59.61 ± 12.24 | 56.26 ± 10.92* | 56.61 ± 11.56* | 54.67 ± 10.19* | 0.808 |
| Dental self-confidence | 20.77 ± 5.61 | 23.52 ± 5.32* | 23.35 ± 5.33* | 24 ± 4.68* | |
| Social impact | 16.45 ± 7.78 | 14.23 ± 7.10* | 15.13 ± 7.23* | 13.5 ± 5.36* | |
| Psychological impact | 15.97 ± 5.57 | 12.77 ± 5.23* | 12.55 ± 5.55* | 12.25 ± 5.795* | |
| Esthetic concern | 6.42 ± 3.38 | 5.74 ± 3.29* | 5.58 ± 2.92* | 4.92 ± 2.96* | |
| Dental self-confidence | 20.77 ± 5.61 | 23.52 ± 5.32* | 23.35 ± 5.33* | 24 ± 4.68* | |

* $p \leq 0.001$ *compared with baseline by t-test.*

## DISCUSSION

This randomized clinical study used a novel treatment in an uncertain (split-mouth) design.[19,20] This was done to show the longevity and probable rebound of color of a protocol that has not been greatly explored using a low concentration of hydrogen peroxide (6%) vs a conventional high concentration peroxide control (35%). A previous report from this cohort of patients[9] showed that no patients were dissatisfied with the color difference between groups. However, after evaluating at one month, one patient requested that the research team match the colors of the groups; two patients did not attend the recall, and one was excluded for not coming at the right time for evaluations.

The results of the longevity of bleaching based on ΔE are quite interesting. There is a significant change that was maintained at nine months with a slight rebound of color. At nine months, the objective difference after prophylaxis was similar to previous controls. The color rebound was not significant vs baseline values; therefore, this accepts the first null hypothesis. The results based on ΔL, Δa, and Δb were very similar except in the preprophylaxis period. This was explained by stains that mainly affect the luminosity of the color, which occurred in both groups. A recent trial by de Geus and others[7] showed that the color had a slight change with prophylaxis. This coincides with our results. The longevity and rebound in color bleaching studies are controversial in the literature. Some studies report stable color at one year,[21,22] two years,[21,23] or longer,[24,25] but others reported stable color only for one to two years.[6,7,23,26-30] The only study with an in-office 6% hydrogen peroxide concentration technique reports 50% color rebound.[8] However, measurement methodologies in these studies are not completely standardized, and the comparison is difficult.

The blue light (cold lamp) could be a real catalyst for the chemical reaction, although there is evidence that the use of light does not improve the effectiveness of bleaching.[31] Modulating this light would be an interesting future investigation. Titanium oxide is a semiconductor under blue light and theoretically catalyzes the formation of hydroxyl radicals from hydrogen peroxide.[32] The exact role by which light or titanium oxide nanoparticles catalyze the mechanism of action remains unclear. In the literature, 6% hydrogen peroxide gels are applied for at least 120 minutes for effective whitening.[33] Here, there was a contact of 72 minutes, which assumes that this is the catalyst for the chemical reaction.

The infrared laser offers immediate control of the sensitivity produced by bleaching because it creates a temporary depolarization of nerve fibers.[4] Thus, it has no relevance to these data.

Teeth exposed to dietary coloring agents definitely have a greater potential to stain. It is important to note that diet was not considered relevant primary data because the study design was split-mouth and both sides were exposed to colorants. Smoking patients were included in this study because there is no significant difference on the effectiveness and longevity of color in patients who smoke fewer than 10 cigarettes a day according to de Geus and others.[7,34] In addition, the quantity of coffee, according to Rezende and others,[35] had no influence on the effectiveness of bleaching.

There are no reports on how paste with no bleaching agents may affect the bleaching in the medium or long term. Hopefully, this report will shed some light on that subject and may have some influence on future studies. However, the use of a prophylaxis protocol reported by de Geus and others[7] showed a difference—particularly in the light parameter specified in Table 3. This shows that the presence of accumulated pigments and/or plaque could be a factor that slightly influences color changes. This

could be solved with a prophylaxis. Therefore, evaluation of the longevity of color outcomes in long-term recalls requires color assessment before and after removal of extrinsic staining by mechanical cleaning and dental prophylaxis.[36] Many clinical studies assessed the color rebound of at-home bleaching and did not report the dietary habits during and after tooth bleaching. Only a few studies have associated diet with the longevity of at-home bleaching; the findings were inconclusive.[5,21,28]

The 6% compound was effective at nine months. According to Bizhang and others, bleaching is considered effective when there is at least a difference of five units of $\Delta E$.[37] Subjective outcomes measured by the variation of SGU by Vita classical and Vita Bleach scales units remain inconsistent with the objective results. The data at nine months by the Vita Bleach scale are nearest to objective data, which could be because it is a more symmetrical and appropriate scale to measure color changes in bleaching;[38] however, the immediate results by Vita Bleach (Table 4) are also inconsistent with the objective measurement. This may be explained by the high bias that exists in the measurements of two neighboring central incisors belonging to different groups because the human eye cannot discriminate between color changes below two units. Subjective scales are only complementary aides and might guide the clinician regarding the effectiveness of whitening.[39] The effect of subjective similarity could be a key point in explaining the effect on esthetics perception and psychosocial impact on patients. The two study groups had stable color at nine months. This means that the color stability is not dependent on the concentration, unlike effectiveness that is directly related to the concentration of the gel.[40]

The OHIP-Esthetics questionnaire results at the nine-month point are controversial. The questionnaires were administered before and after bleaching at one week, one month, and nine months of recall to increase the reliability of the data. The esthetic component measured by OHIP-Esthetics probably influenced the significant difference in the scores after one week, one month, and nine months for bleaching effectiveness. The positive change was evident in the self-perception of dental esthetics at the end of bleaching and one month later. This supports the notion that self-perception of dental esthetics is positively modified by teeth bleaching. The results of OHIP-Esthetics in the ninth month are striking because they show a positive effect vs baseline in terms of functional limitation factors, psychosocial discomfort, physical disability, psychosocial disability, and social disability. This might indicate that the medium-term effect of bleaching generates an esthetic perception that is sharper and deeper than a period of one month. This documents that the psychosocial effects are not immediate and interventions could have an effect in the medium term.[41,42] This might be captured by our OHIP-Esthetics results.

The PIDAQ questionnaire was originally developed to be applied in patients receiving orthodontic treatment;[15] however, isolated factors can also be applied to a patient who experiences dental esthetics through bleaching. In the first factor, "auto dental confidence," there was a positive effect of bleaching in this group of patients until nine months. This impacted dental esthetics on the emotional state of individuals, and maintaining the effect correlated with the maintenance of color. The second factor (social impact) also showed a positive effect at nine months, referring to potential problems in social situations due to a subjective perception of an unfavorable dental appearance. The effects persisted throughout the month. The third factor of psychosocial impact is composed of items dealing with feelings of inferiority and unhappiness when the affected individual compares him/herself with others who have superior dental esthetics. This was positively influenced at nine months of recall. It is known that comparison processes play an important role in psychosocial well-being and that upward comparisons might provoke dysphoric moods.[43]

There was a positive impact of bleaching in patients until the ninth month, similar to what was reported by Martin and others.[9] Clearly the PIDAQ questionnaire is a good tool to substantiate the effects of bleaching. This has been poorly reported in the literature, and additional tools are needed for successful clinical treatments.

According to our results, the second null hypothesis was rejected because there was a positive effect on esthetic perception and psychosocial impact measured by OHIP-Esthetic and PIDAQ by the bleaching procedure.

Methodologically, the blinded nature of the operators, evaluators, and all of the equipment was very strict. Two new evaluators were included to avoid the "probable recognition" bias, and thus all aspects of the study were completely blind. To assess the esthetic perception and psychosocial impact, it would have been appropriate to have a nonbleached group. However, both surveys were designed and validated to measure perception in

patients. Another limitation mentioned in the literature is problems that arise from answering the questionnaire, ie, the alertness of the patient or simply their interest in answering something that may not be pleasing. However, instruments such as the OHIP-Esthetics and PIDAQ are widely used and have been validated by the scientific community. These assessments have been used in many medical studies and could be a beneficial tool for future research in dentistry.

## CONCLUSIONS

Within the limitations and protocols of this study it can be concluded that there was a significant difference between the objective color. Both groups had a similar longevity of color. The two compounds maintained effectiveness at nine months with a slight rebound in color. Patients had a positive impact on their dental confidence and psychosocial well-being at nine months post teeth bleaching.

### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Faculty of Dentistry of the University of Chile PRI-ODO 15/01. The approval code for this study is: FIOUCH 13/18.

### 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**

# Effects of Er,Cr:YSGG Laser Pulse Frequency on Microtensile Bond Strength to Enamel

MK Ayar • T Yildirim

### Clinical Relevance

This study revealed that bonding to Er,Cr:YSGG laser–irradiated enamel depends on pulse frequency. Six watt–50 Hz parameters might be safe options for enamel ablation. However, 3 W–50 Hz parameters might improve resin-bond strength significantly when laser conditioning.


## SUMMARY

**Literature regarding the influence of Er, Cr:YSGG laser pulse frequency with different output power levels on adhesion properties of adhesive resin to lased enamel is limited. Therefore, the aim of the present study was to evaluate the effects of laser pulse frequency (20, 35, and 50 Hz) at two different output power settings (3 and 6 W) of Er,Cr:YSGG on the microtensile bond strength (μTBS) of adhesive resin to enamel. Crowns of 35 intact bovine incisors were embedded into self-cure acrylic resin individually, and then flat enamel surfaces were prepared with 600-grit silicon carbide papers under water cooling. Teeth were divided randomly into seven groups. Enamel surfaces were irradiated with Er, Cr:YSGG laser operated at one of six output power–pulse frequency combinations (6 W20 Hz, 6 W–35 Hz, 6 W–50 Hz, 3 W–20 Hz, 3 W–35 Hz, and 3 W–50 Hz) in groups 1-6, respectively. Bur-treated surfaces served as a control in group 7. After surface treatments and bonding procedures, composite build-ups were done in three layers up to a height of 4 mm. Next, all bonded teeth were sectioned into the resin-enamel sticks to be tested in a μTBS testing machine. The μTBS data were analyzed with univariate analysis of variance under a general linear model with the factor 'tooth' added as a random effect to the design. Resin-enamel interfaces were evaluated with scanning electron microscopy (SEM). The μTBS to laser-irradiated enamel in group 1 (6 W–20 Hz) was significantly lower than those of bur-treated enamel ($p<0.05$). However, group 6 (3 W–50 Hz) showed significantly higher μTBS values than did bur-treated teeth ($p<0.05$). SEM evaluation revealed enormous morphological alterations of laser-irradiated specimens, such as extensive vertical and horizontal microcracks and gaps, with the exception of group 6. The bonding effectiveness of adhesive resin to laser-irradiated enamel was affected by the pulse frequen-**


*Muhammet Kerim Ayar, DDS, PhD, Department of Restorative Dentistry, Faculty of Dentistry, Biruni University, Istanbul, Turkey

Tahsin Yildirim, PhD, Department of Restorative Dentistry, Faculty of Dentistry, Karadeniz Technical University, Trabzon, Turkey

*Corresponding author: Istanbul, 34010, Turkey; e-mail: kerimayar@biruni.edu.tr

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**cy of the Er,Cr:YSGG laser. Although the parameters recommended by the manufacturer lowered μTBS, increasing the pulse rate may maintain optimum μTBS.**

## INTRODUCTION

The Er,Cr:YSGG laser device has been considered one of the most efficient and safe dental hard tissue laser systems for dental hard tissue procedures, including cavity preparation[1] and laser conditioning.[2] The Er,Cr:YSGG laser has the noteworthy ability to remove dental hard tissues, in addition to providing minimum side effects to the pulp and surrounding tissues.[3,4] Additionally, in comparison with conventional mechanical drilling systems, Er,Cr:YSGG laser did not significantly alter the composition and microhardness of dentin tissue.[5] In addition, if Er,Cr:YSGG laser preparation was combined with self-etching adhesive, there was no difference in the composite fillings' microleakage among the laser and conventional bur preparations.[6] Therefore, the Er,Cr:YSGG laser device has been accepted as a safe and effective hard tissue laser.

The extensive interest and usage of erbium lasers in operative dentistry have caused an increase in the numbers of studies covering the effects of erbium lasers on resin bonding to dental hard tissues.[7] Although the effects of the Er,Cr:YSGG laser on the effectiveness of resin adhesives applied to enamel and dentin were investigated primarily, the effects of Er,Cr:YSGG laser parameters were assessed in a much more limited fashion. Currently, the bonding to Er,Cr:YSGG laser–irradiated enamel remains a controversial issue. A discrepancy exists among the findings of previous studies[8-11] on the influence of Er,Cr:YSGG laser irradiation on resin-enamel bonding. Lin and others[11] reported that Er,Cr:YSGG laser irradiation with a 5 W–20 Hz parameter showed no adverse effect on the resin-enamel bond strength of the etch-and-rinse adhesive tested. Despite that finding, several researchers[10,12] have stated that Er,Cr:YSGG laser irradiation with 5.5 W–20 Hz parameters significantly reduced resin bond strength to enamel. Cardoso and others[8] also reported that Er,Cr:YSGG laser irradiation with 6 W–20 Hz parameters resulted in a significant reduction in the bond strength of etch-and-rinse adhesive to enamel. These controversial findings may lead to open-ended discussions regarding the benefits of laser usage in conjunction with current adhesive systems.

One of the unfavorable features of the dental hard tissue lasers, including Er,Cr:YSGG and Er:YAG lasers, is the potential for thermomechanical damage to the surface and subsurface of enamel and dentin during laser irradiation, yielding significantly reduced bond strength.[8,12] Therefore, assessment of the precise laser parameters with which to decrease peripheral thermomechanical damage to a minimum is particularly crucial for long-lasting resin composite restorations. However, it has been suggested[13] that by correctly matching laser parameters (ie, output power and pulse frequency) these thermomechanical side effects can be minimized.

The Er,Cr:YSGG laser device used in the present study is able to emit laser irradiation at different pulse frequencies up to 50 Hz. To the authors' best knowledge, the most common Er,Cr:YSGG laser pulse frequency is 20 Hz, which is recommended by the Er,Cr:YSGG laser device manufacturer in the studies covering the effects of laser irradiation on resin-enamel bond strength. However, this pulse frequency commonly reduced bond strength to enamel.[8,10,12] In addition, a study on the optimization of output power and pulse frequency combinations in terms of bond strength to enamel does not exist in the literature. Thus, the aim of the present study was to evaluate the influence of Er,Cr:YSGG laser irradiation with different pulse frequency (20, 35, and 50 Hz) and output power (6 and 3 W) settings on microtensile bond strength (μTBS) to enamel. Subsurface damage of enamel due to laser irradiation was also assessed by scanning electron microscopy (SEM). The null hypothesis tested was that there is no difference in μTBS to enamel regardless of the different surface treatments used.

## METHODS AND MATERIALS

### Specimen Preparation

Thirty-five permanent bovine incisors were used in this study. The teeth were cleaned to remove any tissue remnants. All teeth were embedded in self-curing acrylic resin with labial surfaces parallel to the floor using double-faced adhesive tape. Labial surfaces of teeth were ground using 320-grit abrasive paper. Then surfaces were further ground with 600-grit abrasive paper for 60 seconds to obtain standardized smear layers. An area of 1 $cm^2$ on the flattened surface was demarcated by means of a marker pen to determine an exact area to be irradiated. Then teeth were divided into seven groups (n=5) randomly according to the study design (Table 1), as follows:

- Group 1 (G1): Enamel irradiated using Er, Cr:YSGG laser with 6 W–20 Hz power–pulse

Table 1: *Study Design of the Present Study*

| Groups (G) | Output Power, W | Pulse Frequency, Hz | Energy per Pulse, mJ/pulse |
|---|---|---|---|
| G1 (Er,Cr:YSGG laser+acid-etching) | 6 | 20 | 300 |
| G2 (Er,Cr:YSGG laser+acid-etching) | 6 | 35 | 200 |
| G3 (Er,Cr:YSGG laser+acid-etching) | 6 | 50 | 120 |
| G4 (Er,Cr:YSGG laser+acid-etching) | 3 | 20 | 150 |
| G5 (Er,Cr:YSGG laser+acid-etching) | 3 | 30 | 100 |
| G6 (Er,Cr:YSGG laser+acid-etching) | 3 | 50 | 60 |
| G7 (Bur+acid-etching) | — | — | — |

frequency combination (300 mJ per pulse, 90 J/cm$^2$ of energy density).

- Group 2 (G2): Enamel irradiated using Er, Cr:YSGG laser with 6 W–35 Hz power–pulse frequency combination (171 mJ per pulse, 90 J/cm$^2$ of energy density).
- Group 3 (G3): Enamel irradiated using Er, Cr:YSGG laser with 6 W–50 Hz power–pulse frequency combination (120 mJ per pulse, 90 J/cm$^2$ of energy density).
- Group 4 (G4): Enamel irradiated using Er, Cr:YSGG laser with 3 W–20 Hz power–pulse frequency combination (150 mJ per pulse, 45 J/cm$^2$ of energy density).
- Group 5 (G5): Enamel irradiated using Er, Cr:YSGG laser with 3 W–35 Hz power–pulse frequency combination (86 mJ per pulse, 45 J/cm$^2$ of energy density).
- Group 6 (G6): Enamel irradiated using Er, Cr:YSGG laser with 3 W–50 Hz power–pulse frequency combination (60 mJ per pulse, 45 J/cm$^2$ of energy density).
- Group 7 (G7; control group): Enamel treated with coarse diamond bur (Microdont ISO 806.314.001.524.012, Sao Paulo, Brazil) using high-speed air turbine (W&H Synea TA98, Bürmoos, Austria).

## Laser Treatments

The Er,Cr:YSGG laser device (Waterlase MD, Biolase Technology, San Clemente, CA, USA) was used for enamel specimens to be irradiated according to the following parameters: wavelength of 2.78 μm; pulse duration of 140 μs; spot size of 600 μm; air pressure setting of 90%; water pressure setting of 65%; irradiation time was 15 seconds; irradiation area of 1 cm$^2$; power of 3-6 W; and pulse frequency of 20, 35, or 50 Hz. Demarcated areas on teeth were irradiated at a 45° angle to the flattened surface in noncontact mode by hand.[14] A bur with a marker was adapted to the hand piece using a custom-made acrylic device to fix the working distance at 1 mm (Figure 1). All laser groups were treated in this manner (G1-G6). In the bur-treated control group (G7), coarse diamond bur using a high-speed air turbine was used by hand with almost no pressure for 15 seconds across the marked area.

## Adhesive Procedures

Following acid-etching of all enamel surfaces in each group using 37% phosphoric acid gel for 20 seconds (All Etch, Bisco, Schaumburg, IL, USA), two consecutive coats of resin adhesive (Single Bond 2, 3M ESPE, St Paul, MN, USA) were applied to dried enamel surfaces with a cotton applicator and air-dried gently for two to five seconds. Light was activated for 20 seconds using a halogen lamp with an output intensity of 400 mW/cm$^2$ (Ivoclar, Astralis 3 Ivoclar Vivadent AG, Schaan, Liechtenstein). After adhesive procedures, resin composite (Valux Plus, 3M ESPE) build-ups in three layers up to a height of 4 mm were done on the surfaces. Each increment layer was cured for 40 seconds using a halogen lamp (Ivoclar, Astralis 3).

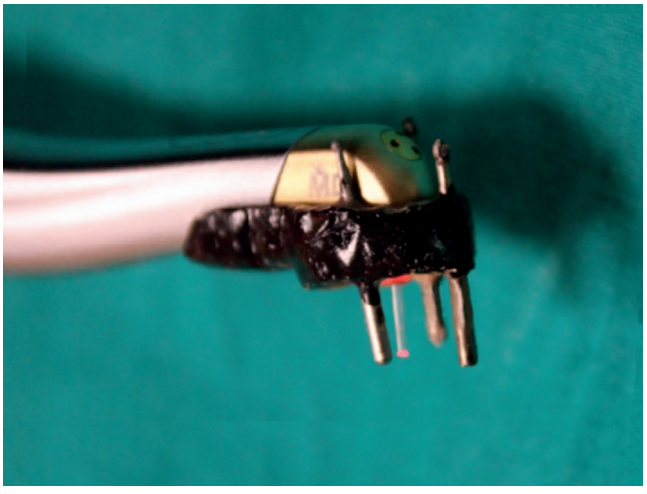

Figure 1. *A simple custom-made acrylic apparatus mounted on a laser hand piece to maintain working distance from enamel.*

Table 2: *Microtensile Bond Strength Means (μTBS; MPa), Standard Deviations (n=20), Percentage of Pretest Failures (%), and Failure Patterns (FPs) of μTBS Samples, as Analyzed Through Stereomicroscopy.* [a]

| Groups (G) | μTBS (%) | FP |
|---|---|---|
| G1 (6 W–20 Hz) | 12.99 ± 9.25 A (48) | M > C > A |
| G2 (6 W–35 Hz) | 21.17 ± 5.39 B (30) | M > C = A |
| G3 (6 W–50 Hz) | 27.16 ± 8.64 C (12.5) | M > C = A |
| G4 (3 W–20 Hz) | 25.63 ± 11.34 BC (13.7) | A > M > C |
| G5 (3 W–35 Hz) | 22.28 ± 7.9 BC (11.3) | M > A > C |
| G6 (3 W–50 Hz) | 36.22 ± 5.98 D (17.5) | C > A > M |
| G7 (bur) | 26.60 ± 8.50 C (11.3) | C > A > M |

*Abbreviations: A, adhesive failure; C, cohesive failure in enamel or composite; M, mix failure.*
[a] *Different superscripts indicate significant differences (p<0.05).*

### Microtensile Bond Test

All bonded teeth were stored in distilled water at 37°C for 24 hours before μTBS tests were conducted. Resin-enamel sticks with approximately 0.9 × 0.9 $mm^2$ dimensions were obtained using a diamond saw under copious water (Micracut 125, Metkon, Bursa, Turkey) running at 300 rpm. Four of the obtained resin-enamel sticks were selected from the center of each bonded tooth to test randomly, yielding 20 sticks for each group. Next, the specimens were fixed to jig with cyanoacrylate glue (Pattex, Henkel, Dusseldorf, Germany) and loaded in tension at a crosshead speed of 1 mm/min using a Bisco microtensile testing machine (Bisco Inc, Schaumburg, IL, USA). Exact dimensions of the interface area were measured with a digital calliper (Mitutoyo, Tokyo, Japan). The μTBS was derived by dividing the load at the time of fracture by the bond area ($mm^2$). Occurrence of failure prior to the actual testing was not included in the analysis, but numbers of such pretest failures were noted. The mode of failure was determined by stereomicroscope under 40× magnification (Meade Bresser Biolux, Meade Bresser, Rhede, Germany) and was recorded as "adhesive" or "cohesive," neither enamel nor resin, and "mix" failures included more than one of the enamel and resin parts.

### Statistical Analysis

The μTBS data were statistically analyzed with univariate analysis of variance in a general linear model with the factor 'tooth' added as a random effect to the design, in order to minimize the effect of using different teeth.[15] Multiple comparisons were made with a least significant difference test. Statistical analysis was performed with the Statistical Package for the Social Sciences (SPSS), version 13 software for Windows (SPSS Inc, Chicago, IL, USA). All tests were done at the 0.05 level of significance.

### SEM Analysis Resin-lased Enamel Interfaces

In order to evaluate interfaces between resin and lased enamel, one stick from the center of each bonded tooth, a total of five sticks for each group, was embedded into self-curing acrylic resin using a two-sided adhesive band. After that, surfaces of sticks were polished using 1000-grit, 1500-grit, and 2000-grit silicon carbide abrasive papers consecutively. Polished surfaces were then etched by 37% phosphoric acid for one minute. All specimens were stored at room temperature prior to SEM evaluation. Specimens were fixed on the metal stubs using two-sided carbon bands and were covered by gold. Morphological evidence regarding large vertical and horizontal resin extensions indicating subsurface cracks, regular resin tag formation, resin-enamel interface completeness, cracks among enamel rods, and vitrification signs were evaluated. SEM evaluation was conducted using a JEOL 6400 SEM (JEOL Ltd, Tokyo, Japan) at 10 kV and at different magnifications (100×-1500×).

## RESULTS

### Microtensile Bond Strength Test

The mean μTBS values, standard deviations, and percentages of pretest failures for each group are shown in Table 2. The mean μTBS values ranged from 12.99 MPa (group 1) to 36.21 MPa (group 6). Group 6 showed the highest μTBS value among all groups, while group 1 showed the lowest μTBS value. The use of different teeth had no significant effect in the statistical model for the enamel specimens ($p=0.656$). A univariate general linear analysis of means revealed that there were significant differences among test groups ($p=0.000$). The highest percentage of pretest failure was seen in group 1, followed by group 2. Percentages of pretest failures of the other groups were similar to each other (Table 2).

Group 1 (6 W–20 Hz) and group 2 (6 W–35 Hz) showed significantly lower bond strengths when compared with the control group (group 7) ($p=0.000$). Group 6 (3 W–50 Hz) showed significantly higher bond strength when compared with the control group ($p=0.003$). No significant differences existed among the bond strengths of other groups and those of the control group. The bond strength of group 1 (6 W–20 Hz) was found to be the lowest, with

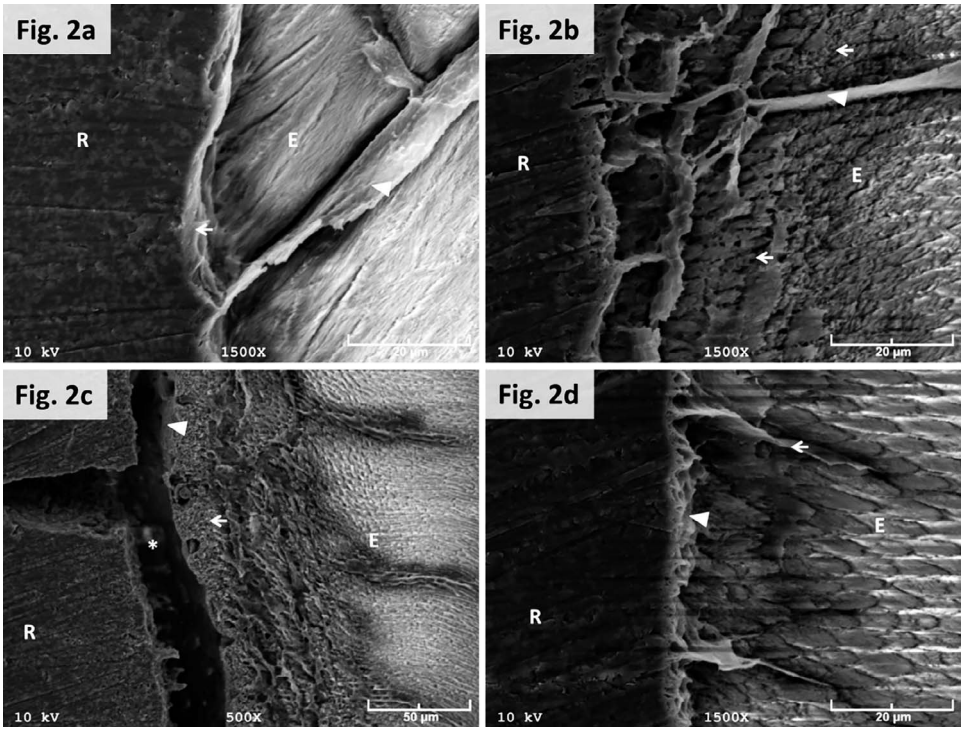


Figure 2. *(a) SEM image of an acid-etched cross section of resin-enamel interface, using acid-etching. There was no horizontal crack in the subsurface of the enamel. However, a tremendous vertical crack was seen as a result of the mechanical stress that occurred during the use of a high-speed abrading mechanism of the diamond bur (arrowhead). Adhesion occurs mainly through microretention as a result of infiltration of adhesive resin into microporosities left after partial dissolution by acid of enamel rods (arrow). (E=enamel; R=resin). (b) SEM image of acid-etched cross section of resin-enamel interface, using laser irradiation with 6 W–20 Hz parameters. Large vertical and horizontal resin extensions were evident (arrowhead). Occurrence of widening interprismatic rods indicates minor cracks throughout enamel rod interfaces (arrows). Extensive minor cracks occurred throughout enamel rods, along with horizontal subsurface cracks, possibly weakening the structure. (E=enamel; R=resin). (c) SEM image of acid-etched cross section of resin-enamel interface, using laser irradiation with 6 W–20 Hz parameters. Huge interface gap due to ineffective bonding to large vitrificated enamel surface was seen (*). Rounded enamel crystals due to vitrification were seen (white arrow). In addition, vitrificated enamel surface was evident, with characteristic recrystallized enamel surface (arrowhead). Extensive vitrification areas may play an important role in reduction of bond strength. (E=enamel; R=resin). (d) SEM image of acid-etched cross section of resin-enamel interface, using laser irradiation with 6 W–35 Hz parameters. Resin-enamel interface was intact. Typical resin tag formations were seen at the interface (arrowhead). However, large resin extensions with approximately 30-µm lengths were evident. Despite group 1, minor cracks throughout enamel rods were not extensive (arrow). (E=enamel; R=resin).*

statistical significance, when compared to those of all other groups. In the 6 W groups, increasing pulse frequency resulted in significant increments in bond strength. In the 3 W groups, improvement in bond strength was obtained when the frequency was increased from 35 Hz to 50 Hz (Table 2).

### Fracture Patterns

The distribution of fracture patterns is presented in Table 2. In all groups, with the exception of groups 6 and 7, the predominant type of fracture was mix. In contrast, in groups 6 and 7, the most frequent fracture type was cohesive.

### SEM Examination of Interface Cross Sections

In the present study, four types of micromorphological changes within the resin-enamel interface and subsurface enamel due to laser irradiations with different power and pulse rate parameters were observed by SEM. These were 1) large vertical cracks presented by infiltration of adhesive resin into cracks, which served as an infiltration highway for adhesive resin; 2) large horizontal cracks presented by infiltration of adhesive resin into macrocracks, which served as an infiltration highway for adhesive resin; 3) minor horizontal cracks, which occurred among sound enamel rods; and 4) localized vitrification areas.

1) Large vertical microcracks were rarely seen in cross sections of resin-enamel sticks in the bur-treated control group (Figure 2a) and the laser group with 3 W–50 Hz parameters (Figure 3d). In contrast, large vertical microcracks were always evident in all other laser groups (Figures 2b–3c). 2) Large horizontal microcracks were not seen in cross sections of resin-enamel sticks in the bur-treated control group.

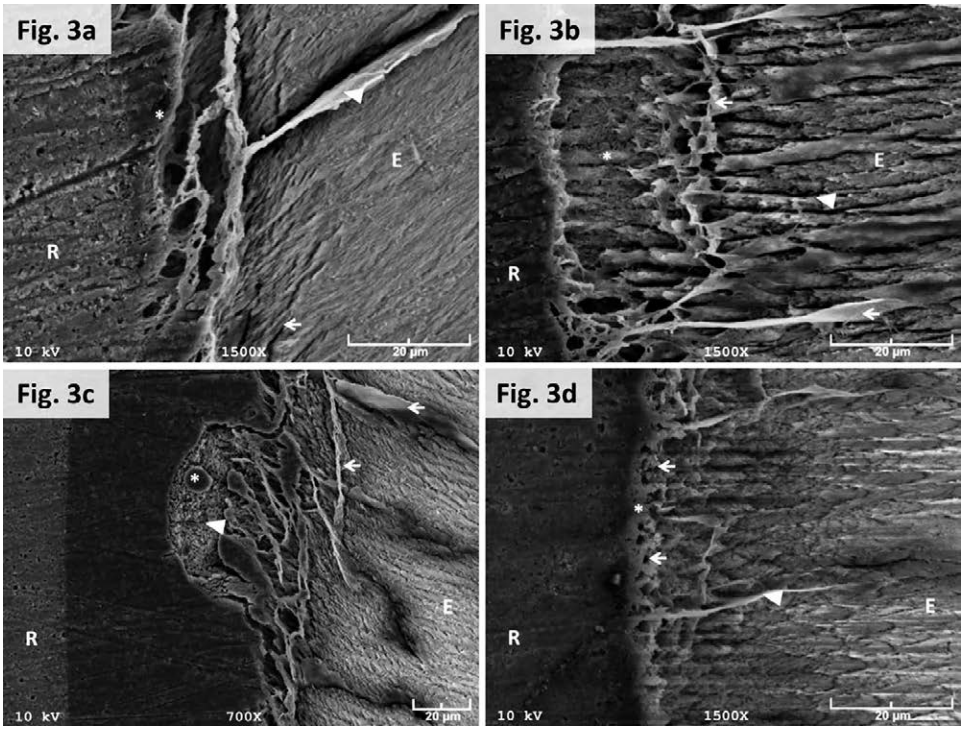


Figure 3. *(a) SEM image of acid-etched cross section of resin-enamel interface, using laser irradiation with 6 W–50 Hz parameters. Although the resin-enamel interface was intact (*), extensive subsurface fissuring resulting in large vertical resin extensions was evident. In addition, large vertical resin extensions exist (arrowhead). Minor cracks among enamel rods were seen (arrows). (E=enamel; R=resin). (b) SEM image of acid-etched cross section of resin-enamel interface, using laser irradiation with 3 W–20 Hz parameters. Subsurface cracks resulting in vertical and horizontal large resin extensions were evident (arrows). Widening interprismatic areas indicating minor cracks among enamel rods were seen (arrowhead). A cavity at the interface due to defragmented surface enamel fragment is indicated by an asterisk. Note that crystallinity of enamel was normal. No vitrification sign was evident. (E=enamel; R=resin). (c) SEM image of acid-etched cross section of resin-enamel interface, using laser irradiation with 3 W–35 Hz parameters. An irregular resin-enamel interface due to a smaller, partially defragmented vitrification area that was encapsulated by adhesive resin was seen (*). Different surface texture indicates vitrification of enamel surface exposed to laser irradiation with 3 W–35 Hz parameters. Texture of vitrification area seemed to comprise rounded atypical apatite crystals (arrowhead). Micro- and macroporosities (Resin Island was indicated by white asterisk) were evident within vitrification area. In addition, large vertical and horizontal cracks were seen. (d) SEM image of acid-etched cross section of resin-enamel interface, using laser irradiation with 3 W–50 Hz parameters. The resin-enamel interface was intact (*). Although large vertical resin extensions were evident, they were narrower than those of other groups (arrowhead). Thinner horizontal resin extensions were positioned at 10 µm below the interface (arrows). Cracks among enamel rods were absent (E=enamel; R=resin).*

However, they were seen in the laser group with 3 W–50 Hz parameters very rarely and in thinner form (Figure 3d). However, these were unexceptionally evident in all other laser groups (Figures 2b–3c). 3) Minor horizontal microcracks were commonly seen in all laser groups, with the exception of the group with 3 W–50 Hz parameters (Figures 2b and 3c). Locations of these cracks were approximately 20 µm below the interface and seemed to be independent of power and pulse rate. 4) Vitrification areas were noticed in the high- and low-power groups. However, the area affected from vitrification seems larger in the high-power group (Figure 2c) than in the low-power group (Figure 3c).

## DISCUSSION

The Er,Cr:YSGG laser is approved by the United States of America Food and Drug Administration for dental hard tissue operations, including cavity preparation, caries removal, and laser conditioning, because of its safety with respect to pulpal responses and thermal damage.[16] Cavities prepared by the Er,Cr:YSGG laser are generally restored by adhesive resin restorative materials. It is known that the adhesion of resin adhesive systems to lased dental hard tissues may be affected by several laser irradiation parameters, including laser irradiation distance,[17] pulse duration,[18] output power,[19] pulse frequency,[20] and water flow rate.[21] However, to the knowledge of the authors there is no study covering the effects of different output power–pulse frequency combinations of Er,Cr:YSGG laser on resin bond strength to enamel in the literature. Moreover, the conflicting results obtained from previous studies on the effects of Er,Cr:YSGG laser irradiation on resin-enamel bonding using the pulse frequency that is suggested by the Er,Cr:YSGG laser manufacturer

(20 Hz) are evident.[8,10,11] Therefore, pulse frequency–output power combinations of the Er,Cr:YSGG laser that could yield optimum resin-enamel bond strength were sought in the present study.

According to μTBS findings in the present study, the output power and pulse frequency combination of 6 W–20 Hz, which is close to those (5.5 W–20 Hz) suggested by the Er,Cr:YSGG laser manufacturer for cavity preparation on enamel, significantly decreased the μTBS of resin adhesive to enamel when compared to findings in the control group. Therefore, the null hypothesis cannot be accepted. Nevertheless, only a few studies covering the effects of high-power Er,Cr:YSGG laser irradiation on resin-enamel bond strength exist in the literature. The finding in the present study is correlated with the findings of previous studies. Cardoso and others[8] and Ansari and others[10] revealed that the 6 W–20 Hz and 5.5 W–20 Hz output power and pulse frequency parameters of the Er,Cr:YSGG reduced μTBS values, respectively. A possible explanation for the significant reduction effect of the 6 W–20 Hz combination on μTBS may be that it resulted in extensive vitrification areas and subsurface vertical and horizontal microcracks within the resin-enamel interfaces (Figure 2c).

When Er,Cr:YSGG laser irradiation is focused on enamel, it causes different surface morphology compared with that associated with bur-abraded enamel surfaces and morphological alterations on the surface and within the subsurface of enamel.[11,22,23] It was discovered that enamel lased with the Er,Cr:YSGG laser was smear-layer free, with microretentive characteristics. These observations led researchers[11] to suggest that lased enamel was more favorable with respect to resin bonding. Nevertheless, studies[8,23,24] indicated that high-power laser irradiation affected resin-enamel bond strength in an unfavorable way, revealing that corresponding laser parameters resulted in subsurface damage to enamel. In addition, although laser irradiation was applied by coupling with water spray, vitrifications were observed in the previous studies.[8] It was presumed that these subsurface morphological alterations might affect resin-enamel bond strength unfavorably.[7,8,24]

Other laser groups, including the 6 W–50 Hz, 3 W–20 Hz, and 3 W–35 Hz groups, yielded statistically similar bond strength values compared with the control group. These findings may imply that when the Er,Cr:YSGG laser is needed to cut enamel more quickly, increasing pulse frequency at a high power level may maintain resin-enamel bond strength in a manner similar to that associated with bur treating. However, it was found that the 3 W–50 Hz combination significantly increased μTBS when compared to the bur-treated group in the present study. SEM images showed tight adaptation of adhesive resin with lased enamel. However, no vitrification signs were seen. Nevertheless, subsurface microcracks were rarely observed (Figure 3d).

"Pulse frequency" represents the number of pulses that have equal pulse energy with each other delivered to target tissue per second. Increasing pulse frequency does not affect the total energy delivered to target tissue, while it does decrease the amount of energy that is carried by each pulse. The findings of the present study indicate that 300 mJ per pulse (6 W–20 Hz) and 171 mJ per pulse (6 W–35 Hz) rates are detrimental to resin enamel bond strength, whereas 120 mJ per pulse (6 W–50 Hz), 150 mJ per pulse (3 W–20 Hz), and 86 mJ per pulse (3 W–35 Hz) rates did not affect resin-enamel bonding in comparison to bur treating, respectively. However, further increasing pulse frequency to 50 Hz at a low output power level, yielding 60 mJ per pulse, significantly increased the bond strength of resin to lased enamel.

A microtensile bond strength test, which uses specimens of smaller diameter, was selected as a bond strength test in the present study to evaluate the effects of test variables on bond strength values. The higher means obtained from specimens of smaller diameter enable the microtensile bond strength test to be discriminative enough for detecting differences arising from treatment variables with the use of a smaller number of actual tooth specimens.[25]

In the present study, bovine incisors were used as enamel specimens. Bovine enamel is an accepted substitute for human dental hard tissue for bond strength testing studies. In addition, the use of bovine incisors provides the ready availability and increased enamel surface for enamel bond strength tests.[26,27]

## CONCLUSIONS

Within the limitations of the present *in vitro* study, it can be concluded that

1) Er,Cr:YSGG laser pulse frequency and output power are important parameters that might have significant effects on resin bond strength to irradiated enamel. A power–pulse frequency parameter combination of 6 W–20 Hz, which is

one of the commonly used output power–pulse frequency combinations of Er,Cr:YSGG laser irradiation for enamel preparation, significantly reduced μTBS when compared to the control (bur-treated) group.

2) The power–pulse frequency parameter combination of 6 W–50 Hz yielded similar μTBS when compared to the control. Therefore, high power with high pulse rate might be suggested for faster enamel ablation.
3) The power–pulse frequency parameter combination of 3 W–50 Hz significantly increased μTBS when comparing all groups. The use of this power–pulse frequency combination can be recommended to increase the μTBS of adhesive resin to laser-irradiated enamel.

#### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Biruni University in Istanbul, Turkey.

#### Conflict of Interest

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

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# Effect of Nd:YAG Laser Irradiation Pretreatment on the Long-Term Bond Strength of Etch-and-Rinse Adhesive to Dentin

J Gan • S Liu • L Zhou • Y Wang • J Guo • C Huang

### Clinical Relevance

Computer-aided design/computer-aided manufacturing resin composites have different physical properties, and care should be taken when selecting one for clinical use.


## SUMMARY

**Purpose: To investigate the effect of neodymium-doped yttrium aluminum garnet (Nd:YAG) laser irradiation pretreatment on the long-term bond strength of an etch-and-rinse adhesive to dentin.**

**Methods: Fifty molars were sectioned parallel to the occlusal plane and randomly divided into two groups (n=25 per group): control group (no treatment) and laser group (dentin surfaces were treated with Nd:YAG laser at a setting of 100 mJ/10 Hz). Afterward, resin was bonded to the dentin surface using a two-step etch-and-rinse adhesive (Adper SingleBond 2), and then 150 beams of each group were produced. Each group was divided into three subgroups (n=50 each group): 24 hours of water storage, thermocycling, and NaOCl storage. The microtensile bond strength (MTBS), failure modes, nanoleakage expression, and Masson's trichrome staining were evaluated. An additional 20 molars were sectioned to obtain 2-mm-thick flat dentin slices. These slices were randomly divided into control and laser-treated groups as mentioned previously. Then slices of each group were examined by scanning electron microscopy, attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR), X-ray diffraction (XRD), and the Knoop hardness test.**

**Results: The results of ATR-FTIR and Masson's trichrome verified that laser irradiation partly removed collagen fibers from the dentin surface; however, no significant difference was found in the Knoop hardness ($p>0.05$). The XRD result showed similar crystalline struc-**


Jing Gan, MSD, School and Hospital of Stomatology, Wuhan University, State Key Laboratory Breeding Base of Basic Science of Stomatology (Hubei-MOST) and Key Laboratory for Oral Biomedical Ministry of Education, Wuhan, China

Siying Liu, MSD, School and Hospital of Stomatology, Wuhan University, Wuhan, China

Liqun Zhou, MSD, School and Hospital of Stomatology, Wuhan University, Wuhan, China

Yake Wang, MSD, School and Hospital of Stomatology, Wuhan University, Wuhan, China

Jingmei Guo, MSD, School and Hospital of Stomatology, Wuhan University, Wuhan, China

*Cui Huang, MS, MDS, PhD, professor, School and Hospital of Stomatology, Wuhan University, Wuhan, China

*Corresponding author: School and Hospital of Stomatology, Wuhan University, 237 Luoyu Rd., Wuhan 430079, China; e-mail: huangcui@whu.edu.cn

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**ture regardless of laser pretreatment. There is no significant difference in short-term MTBS between control and laser-treated groups ($p>0.05$); however, long-term MTBS differed between the groups ($p>0.05$). Furthermore, the laser-treated group showed less silver deposition than the control group after aging ($p<0.05$).**

**Conclusion: Pretreatment by Nd:YAG laser irradiation appeared to have a positive effect on the adhesive-dentin bonding *in vitro* test, and the bonding effectiveness could be preserved after aging.**

## INTRODUCTION

The promotion of adhesive techniques has become increasingly important with the rising public requirement on esthetics and "minimum intervention" in modern operative dentistry.[1] Dentin bonding is a process in which adhesive resin replaces the hydroxyapatite (HAP) mineral phase removed from the dentin tissue surface through acid etching and then micromechanically interlocks with a network of exposed collagen fibrils to form the so-called hybrid layer. Hybrid layer formation on the surface and within the subsurface of dentin depends on the permeability of dentin and the diffusion of applied monomers. These features determine the durability of the dentin bonding interface over time.[2,3] Compared with bonding to enamel, dentin bonding is more challenging and less predictable.[1] Various bonding strategies, such as laser irradiation, have been developed to maintain resin-dentin bond strength.

Laser technology has been widely applied in clinical trials since the 1960s. Advances in laser technology have led to various dental applications, such as periodontal soft tissue plastic surgery, cavity preparation and treatment of dentinal sensitivity, caries prevention, and bleaching.[4] Moreover, several aspects of lasers can enhance dentin bonding, such as opening dentin tubules without demineralization of peritubular and intertubular dentin, dentin surface sterilization, and a bonding surface with microirregularities without a smear layer.[5-7] Conducting acid etching after laser treatment produces equal or better dentin-resin bond strengths,[8,9] while other aspects, such as the fused denatured collagen fibrils, probably limit resin infiltration into the intertubular dentin and weaken dentin bond strength.[10] Studies on short-term bonding strength after laser irradiation obtained inconsistent results. This inconsistency may be attributed to the different characteristics of the dentin surface resulting from different laser types and laser energy parameters.[11,12] In addition, only a few studies have explored the effects of the properties of laser-irradiated dentin on bond strength.

Neodymium-doped yttrium aluminum garnet (Nd:YAG) laser, which emits a wavelength of 1064 nm, is frequently used in dentin bond studies.[13] It is usually used to melt and recrystallize dentin HAP to occlude dentinal tubules,[14] and collagen loss occurring in this melting process has been reported.[15] In the present study, Nd:YAG laser was applied to melt the dentin surface to remove the smear layer and dentin collagen. Acid etching was subsequently used to form a microscopically rough substrate surface without a demineralized collagen fiber network, possibly strengthening the dentin bond.

Contemporary dentin adhesives present favorable short-term bonding strengths,[16] but the durability of these materials remains limited. To date, resin monomers cannot displace water and infiltrate the collagen network completely. The biodegradation of nonencapsulated collagen fibrils causes hybrid layer degradation, which subsequently results in resin-dentin bond breakdown.[17] Several studies have explored the effects of laser on short-term dentin bond strength. However, the durability of dentin bond strength after laser treatment remains unknown. Laser irradiation vaporizing dentin collagen has been demonstrated by several studies,[10,15] and the loss of dentin collagen may maintain the dentin bond strength by avoiding *in vivo* collagen degradation. Therefore, laser irradiation probably is favorable to dentin bond durability, and the influence of this process warrants further investigation.

This study aimed to investigate the effects of Nd:YAG laser irradiation on short-term and long-term dentin bond strength and on the microstructure of the dentin surface. The null hypothesis was that treating the dentin surfaces with or without laser irradiation before acid etching exerts no significant effect on the microstructure of dentin substrate, microtensile bond strength (MTBS), failure modes, and nanoleakage.

## METHODS AND MATERIALS

### Specimen Preparation of Field-Emission Scanning Electron Microscopy, Thin-Film X-Ray Diffraction, Fourier Transform Infrared Spectroscopy, and the Knoop Hardness Test

Seventy freshly extracted noncarious human third molars were selected after the patients' informed consent was obtained. The collected teeth were

stored in 0.9% (w/v) NaCl containing 0.002% sodium azide at 4°C and used within one month. Twenty molars were chosen to prepare 2-mm-thick flat slices that were cut from the mid-coronal dentin using a water-cooled low-speed cutting saw (Isomet, Buehler Ltd, Evanston, IL, USA), and another 50 molars were reserved for subsequent microtensile bond strength testing and nanoleakage evaluation. The dentin surfaces were polished using 600-grit SiC paper to create a standardized smear layer and then cleaned ultrasonically in deionized water, dehydrated with ethanol, and dried in a critical evaporator.

### Treatment with Nd:YAG Laser

The Nd:YAG laser equipment used in this study was the Miracle Laser-3100 (Wuhan Miracle Laser Technologies Inc, Hubei, China) at a wavelength of 1064 nm. The laser irradiation parameters were as follows: 1 W power, 10 Hz repetition rate, within the energy parameters of 100 mJ, and an energy density of 85 J/cm$^2$ per pulse. Before laser irradiation, black ink was applied to the dentin surfaces to increase the absorption of energy from the pulse into the dentin tissue.[18] To avoid the influence of the measurement of removing the ink and the potential ink remnants, dentin surfaces of the control group also received the same ink, and all the ink was removed as thoroughly as possible before adhesion. The laser was fitted with a quartz fiber tip that was 400 μm in diameter and applied freehand, in noncontact mode, emitting for 60 s/cm$^2$ two times.[19] During laser application, the laser tip was always perpendicular to the specimen surface at a distance of 2 mm. A special working stage was used to maintain the distance between the laser tip and the dentin surfaces. Samples were moved with a fixed laser beam position using a motorized stage.

### Characterization of the Laser Irradiated Dentin Surface

*Field-emission scanning electron microscopy*—The surfaces and the transverse sections of the acid-etched dentin slices and laser-treated dentin slices followed by acid etching were dried in a drying vessel for 24 hours and then sputter coated with Au-Pd alloy and observed using field-emission scanning electron microscopy (FE-SEM) at 5 kV (Sigma, Zeiss, Jena Germany).

*Thin-film X-ray diffraction*—Six dentin slices were chosen and randomly divided into control and laser treated groups (n=3). After surface treatment with or without laser, all the slices were examined by thin-film X-ray diffraction (TF-XRD) using an X-ray diffractometer (Bruker AXS D8-XRD, Karlsruhe, Germany) operated under a 40-kV acceleration voltage and 40-mA current with the angle of the incident X-ray beam fixed at 25 degrees and a scanning time of 1 degree/min for a 2θ scan.

*Attenuated total reflection Fourier transform infrared spectroscopy*—For attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR) spectra collection, six dentin slices were prepared as previously described, and each half of every slice was irradiated by laser or left untreated, respectively; three different spots on the surface of each half were randomly chosen and tested. The spectral data were expressed as absorbance. FTIR and ATR spectra of these specimens' surfaces were carried out with a Nicolet 5700 spectrometer (Thermo Scientific Inc, Madison, WI, USA) and a single-reflection ATR setup with germanium (Ge) as an internal reflection element (Smart OMNI-Sampler, Thermo Scientific).

The effect of laser irradiation on collagen depletion was evaluated using the collagen:apatite ratio (the ratio of absorbance of amide I and II peak to phosphate $\nu_3$ peak) and analyzed by one-way analysis of variance (ANOVA) and Tukey's test at the 5% level of significance.

*Knoop hardness test*—Three dentin slices were divided in half; one-half was irradiated by laser, and the other was left untreated. Microhardness was evaluated using a hardness tester (E. Leitz GmbH, Wetzlar, Germany). A load force of 100 g was used at a hold time of 10 seconds. Six indentations were performed on each half of the sample, and the mean values were statistically analyzed by ANOVA and Tukey's test at the 5% level of significance.

### Specimen Preparation of MTBS, Nanoleakage Evaluation and Masson's Trichrome Staining

Fifty freshly extracted intact human third molars were used in this study. A flat surface was prepared using a water-cooled low-speed cutting saw (Isomet, Buehler) to expose the mid-coronal dentin. The dentin surface was polished using 600-grit SiC paper to prepare a standardized smear layer. The teeth were randomly assigned to two groups that were conditioned either with an Nd:YAG laser (experimental group) or with no treatment (control group): a two-step etch-and-rinse adhesive Adper Single-Bond 2 (3M ESPE, St Paul, MN, USA) was used in the current study. Briefly, the dentin surface was first etched with 35% phosphoric acid for 15 seconds, rinsed and blotted dry, coated with two layers of adhesive, gently air thinned for 5 seconds, and light

cured for 10 seconds. Afterward, resin composite was built up using 4-mm increments of resin composite (Charisma, Heraeus Kulzer, Hanau, Germany). Each tooth was first sectioned into 0.9-mm-thick slabs after storage in deionized water at 37°C for 24 hours, and three resin-dentin bonded slabs were respectively selected from three teeth of laser-treated and control groups for Masson's trichrome staining. Then the rest of the slabs were sectioned into 0.9 × 0.9-mm beams. Six beams were obtained from the central part of each specimen. In total, 150 beams were harvested for the laser treatment and control groups separately.

### Artificial Aging Treatment

The beams with each surface treatment were further divided into the following three subgroups (n=50):

Group 1 (control): Beams without any artificial aging treatment served as the baseline.
Group 2 (thermocycling): Beams underwent a thermocycling test from 5°C to 55°C for 10,000 cycles, with the dwell time set at 30 seconds.
Group 3 (NaOCl storage): Beams were immersed in 10% NaOCl solution for 1 hour at room temperature.

As a matter of convenience, we abbreviated groups 1 to 3 as CC, CT, and CN for no surface treatment and as LC, LT, and LN for laser surface treatment, respectively.

### MTBS Test

After different aging treatments, MTBS of 45 bonded beams from each group were tested. Each beam was attached to a universal testing apparatus (Microtensile Tester, Bisco, Schaumburg, IL, USA) with a cyanoacrylate adhesive (Zapit, Dental Ventures of America, Corona, CA, USA) and loaded until failure at a crosshead speed of 1 mm/min. The exact dimensions of fracture region were measured using a digital caliper. The final MTBS values (MPa) were calculated as the maximum load at failure divided by the cross-sectional area.

The MTBS data were analyzed using SPSS 16 (SPSS Inc, Chicago, IL, USA). The correlation between surface treatment and artificial aging methods was evaluated by two-way ANOVA factorial analysis, followed by Tukey's *post hoc* multiple comparison test ($\alpha$=0.05).

### Failure Mode Analysis

After the MTBS test, the dentin side of all the failed modes was evaluated with a stereomicroscope (Stemi 2000-C, Carl Zeiss Jena, Göttingen, Germany) at 50× magnification and classified as A (adhesive failure), M (mixed failure), CD (cohesive failure in dentin), and CC (cohesive failure in composite).[20] Three typical failed samples from each group were examined using FE-SEM (Sigma, Zeiss) after coating with Au-Pd alloy. The general conditions of the fractured surfaces and typical region were captured at 100× and 2500× magnifications.

### Nanoleakage Evaluation

Five beams from each group were chosen for nanoleakage evaluation using the method described by Tay and others.[21] The beams were coated with nail varnish applied to within 1 mm of the interfaces. Afterward, the beams were placed in 50 wt% ammoniacal $AgNO_3$ in darkness for 24 hours, thoroughly rinsed in distilled water, and placed in a photodeveloping solution for 8 hours under a fluorescent light to facilitate the reduction of $[Ag\ (NH_3)_2]^+$ ions into metallic silver grains. After dehydrating, the silver-stained specimens were embedded in epoxy resin and then polished with 600-, 800-, and 1200-grit SiC papers and a soft cloth under running water. The specimens were ultrasonically cleaned and dehydrated for 24 hours at room temperature. After the carbon coating, the resin-dentin interface was analyzed with FE-SEM in the backscattered electron mode (Quanta 450 FEG, FEI, Eindhoven, The Netherlands). Nanoleakage within the adhesive-dentin interfaces was investigated, and four areas of each sample's hybrid layer were randomly photographed at 1000× magnification for quantitative analysis. Thus, 20 images were obtained for each group. Image J (National Institutes of Health, Frederick, MD, USA) was used to calculate the total silver particle percentage in the hybrid and adhesive layers.[22] The data were analyzed using SPSS 16 (SPSS Inc). The correlation between different surface treatments and aging methods was evaluated by two-way ANOVA factorial analysis, followed by Tukey's *post hoc* multiple comparison test ($\alpha$=0.05).

### Masson's Trichrome Staining

Resin-dentin bonded slabs prepared previously of both laser-treated and control groups (n=3) were fixed in a glass holder with a photocuring adhesive (Technovit 7210 VLC, Heraeus Kulzer GmbH & Co, Werheim, Germany) and polished with SiC papers until their thicknesses were approximately 10 μm. Slices were treated with Masson's trichromic acid

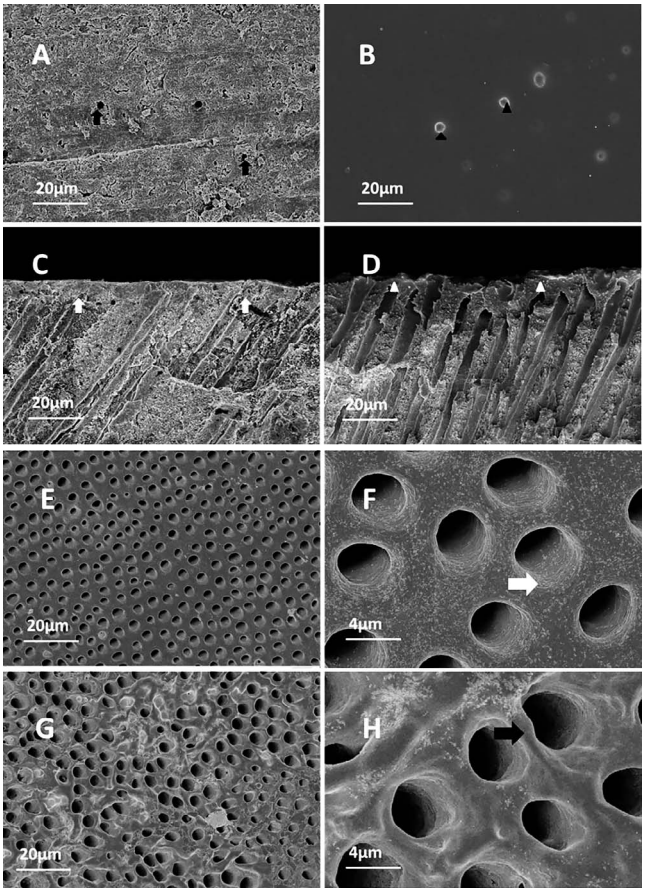


Figure 1. *FE-SEM surface and transverse examination of sound dentin (A and C) and laser-treated dentin (B and D). The sound dentin was covered by smear. The laser-treated dentin tubules were occluded by melting HAP crystallites. Black arrows: occluded dentin tubules by smear. Black triangles: occluded dentin tubules by laser irradiation. White arrows: smear layer. White triangles: laser-irradiated layer. FE-SEM surface examination of sound dentin after acid etching (E and F: the magnification of E) and laser treated dentin after acid-etching (G and H: the magnification of G). Black arrow: cufflike peritubular dentin. White arrow: funnel-shaped dentin tubules.*

staining technique.[23] A cover slip was placed and the specimens were examined by light microscopy (BH-2, Olympus, Tokyo, Japan) at 400× magnification.

## RESULTS

### Characteristics of Laser-Treated and Sound Dentin Surfaces

The surface and transverse section ultrastructure of sound dentin and Nd:YAG laser-irradiated dentin is shown in Figure 1A-D. The surface of sound dentin showed a widespread smear layer and several blocked dentin tubules (Figure 1A,C). By contrast, the laser-treated dentin slices displayed a flat, smooth, and integrated surface with no smear layer. Dentin tubules were occluded and integrated into the subsurface dentin structure and formed a new unbroken homogeneous modified layer. The transverse section showed that the upper dentin formed a melt layer barely containing collagen fiber and that the walls of dentin tubules became nonporous and smooth after laser treatment. No cracks appeared between the newly formed layer and the dentin substrate (Figure 1B,D).

Figures 1E-H show representative FE-SEM images (low magnification, 1000×; high magnification, 5000×) of sound dentin treated with or without Nd:YAG laser irradiation before acid etching. The characteristics of the dentin surface after acid etching are displayed in Figure 1E,F. The smear layer was completely moved, the surface was flat and regular, and dentin tubules became funnel shaped. As shown in Figure 1G,H, the laser-treated dentin surface became irregular and crater shaped after acid etching with 35% phosphoric acid for 15 seconds. Most of the occlusive dentin tubules were reopened with a cufflike peritubular dentin-protruding dentin surface. The dentin tubules also showed a smooth and nonporous wall in the laser-treated samples.

### Mechanical Property and Composition of the Laser-Irradiated and Sound Dentin Surfaces

Sound dentin and laser-modified dentin were characterized using XRD. The spectra are shown in Figure 2A. The peaks at 2θ degrees were 25.9688, 31.6998, 32.0798, and 32.7828 degrees, corresponding to the expected peaks for hydroxyapatite at 002, 211, 112, and 300 planes, respectively (JCPDS No. 09-0432).[24] There was no obvious difference between the two spectra.

The typical ATR-FTIR spectrum of sound dentin is shown in Figure 2B. The $HPO_4\nu_3$ band (997-1124 $cm^{-1}$) and the $HPO_4\nu_1$ band (964 $cm^{-1}$) indicated the presence of hydroxyapatite crystals,[25] and the bands at 1650 and 1453 $cm^{-1}$ corresponded to the collagen protein amide I and II bands.[15,26] After laser treatment, the collagen:apatite ratio decreased significantly compared with the control group ($p<0.05$) (Figure 2C).

The hardness of the dentin before and after laser irradiation was evaluated using the Knoop hardness test. The Tukey test showed that laser irradiation did not influence dentin hardness significantly ($72.91\pm12.45$ vs $77.06\pm7.38$, $p>0.05$).

### Optical Microscopy

Representative light micrographs of Masson's trichrome stained sections of the dentin/adhesive

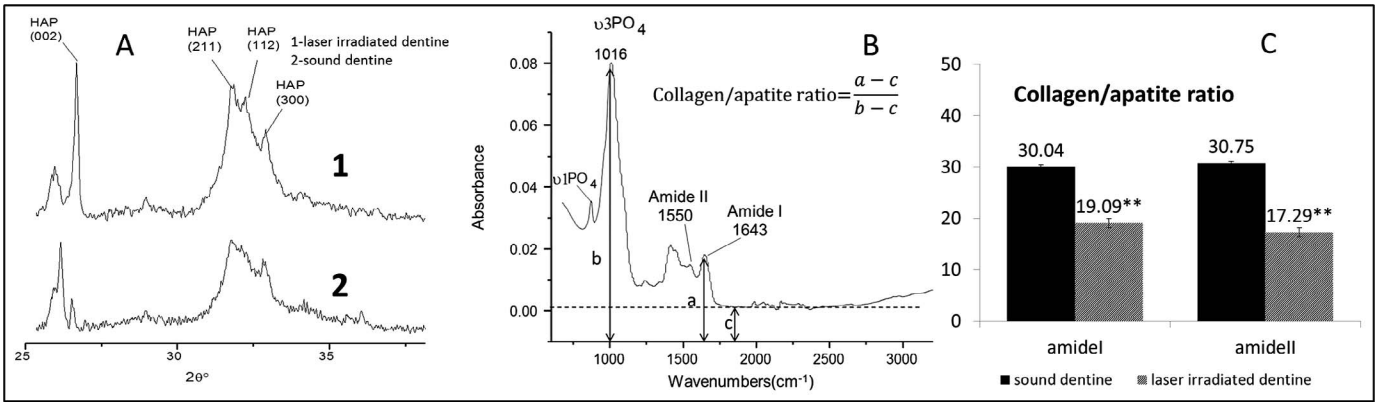


Figure 2. *(A): XRD spectra of dentin samples of the sound dentin surface and laser-irradiated dentin surface. (B): Schematic illustration of how the collagen:apatite ratio was derived from each FTIR spectrum. With the use of laser irradiation, the $v_3PO^4$ peak of apatite could not be used as a reference for examining changes in height of the amide I and II peaks of intact collagen. Hence, the collagen:apatite ratio was used as the substitute. (C): Column graph of the collagen:apatite ratio of laser-irradiated and control dentin surfaces. The collagen:apatite ratio of laser-treated dentin surfaces decreased significantly compared with the control samples (*p*<0.05).*

interface are presented in Figure 3. With these trichrome stains, mineralized dentin stained green, exposed demineralized collagen not encapsulated by the adhesive stained red, and adhesive stained beige.[27] Specimens from the control group presented a distinct red zone at the base of the hybrid layer, while a barely red zone was observed in laser-treated specimens.

### MTBS

The mean MTBS and standard deviation are summarized per experimental condition in Table 1. The variables dentin surface treatment and aging treatment did not have a significant effect on the bond strength ($p>0.05$). Laser treatment before acid etching had no significant difference on the MTBS ($p>0.05$). Specimens bonded with laser-treated surfaces could preserve the MTBS in both the thermocycling and the NaOCl aging groups. However, the MTBS of specimens only acid etched were significantly decreased ($p<0.05$) after thermocycling and NaOCl treatment (Table 1).

### Failure Mode Analysis

The failure modes were analyzed by frequency distribution (Table 1). For all groups, adhesive failure was the predominant fracture pattern. Mixed failure and CD failure was evident in the CN group. Mixed failure percentage also relatively increased in the LN group, but few CD failures were observed. The representative FE-SEM images are shown in Figure 4.

### Nanoleakage Observations

SEM interfacial analysis showed that both laser-treated and control samples presented silver uptake along the base of the hybrid layer after 24 hours of aging (Figure 5). Laser treatment had no influence

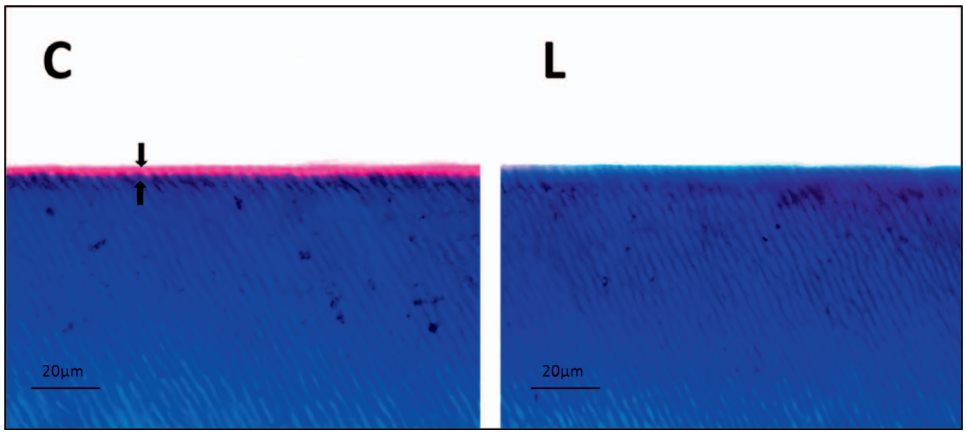


Figure 3. *Representative light micrographs of dentin specimens stained with Masson's trichrome (magnification: 400×). C: Specimen from the control group; the wide red band corresponds to the extent of exposed collagen matrix unprotected by the adhesive resin (space between black arrows). L: Specimen from laser irradiated group; the barely red band was observed in the laser-treated specimen.*

Table 1: *Microtensile Bond Strength (MTBS), Fracture Frequency, and Nanoleakage Expression of Each Group*[a]

| Groups | MTBS (MPa) | Failure Frequency (%) | | | | Nanoleakage Expression (%) |
|---|---|---|---|---|---|---|
| | | A | CC | CD | M | |
| CC | 20.8 ± 6.4 A | 71.1 | 0 | 2.2 | 26.7 | 9.2(0.8) A |
| CT | 15.1 ± 5.8 B | 84.1 | 2.3 | 2.3 | 11.3 | 24.3(4.5) B |
| CN | 16.1 ± 4.7 B | 55.6 | 0 | 20 | 24.4 | 18.9(1.35) B |
| LC | 21.0 ± 6.4 A | 86.7 | 0 | 2.2 | 11.1 | 9.1(0.8) A |
| LT | 20.6 ± 7.1 A | 81.8 | 4.5 | 2.3 | 11.4 | 12.2(1.5) A |
| LN | 20.8 ± 5.1 A | 67.4 | 0 | 4.7 | 27.9 | 12.4(3.4) A |

*Abbreviations: A, adhesive failure; CC, cohesive failure in composite; CD, cohesive failure in dentine; M, mixed failure.*
[a] *Values of MTBS are mean ± standard deviation. Groups with the same letters are not statistically significant (p>0.05).*

on silver deposition after 24 hours of water storage ($p>0.05$). Less silver deposition was observed in the laser-treated group than in the control group after aging treatments ($p<0.05$) (Table 1).

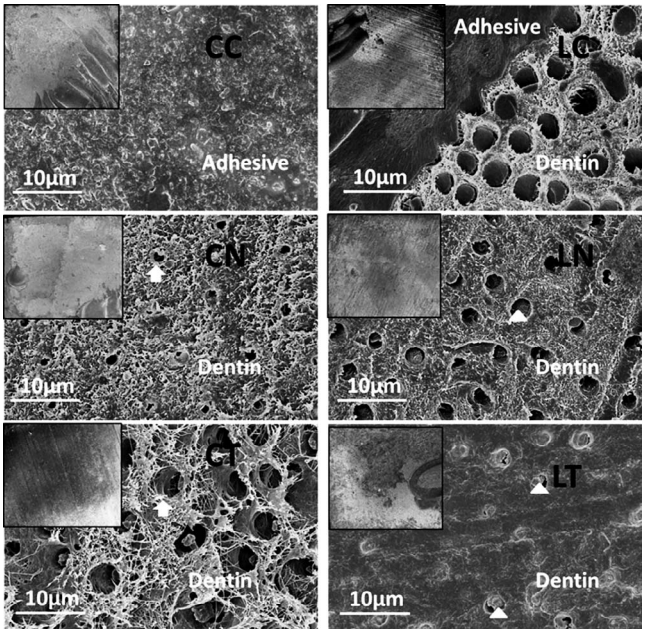


Figure 4. *Representative FE-SEM images (low magnification, 100×; high magnification, 2500×) of dentin sides of fractured specimens in both laser-treated and control groups. The insert indicates general condition of fractured surfaces. CC: Specimens of the CC group showed an adhesive failure. Much adhesive resin covered dentin surface. LC: Specimens of the LC group showed a mixed failure. Dentin tubules were sealed by resin tags, and remnant adhesive could be observed. CN: Specimens of the CN group showed a cohesive failure in dentin. The failure was mainly at the bottom of the hybrid layer. Dentin tubules and a large number of broken collagen fibers were observed. LN: Specimens of the LN group showed a cohesive failure in dentin. The presence of several open dentin tubules showed that the failure was mainly at the bottom of the hybrid layer. CT: Specimens of the CN group showed a cohesive failure in dentin. Wider tubules and the widespread broken collagen fibers showed that the failure was at the bottom of the hybrid layer. LT: Specimens of the LT group showed a cohesive failure in composite. Sealed tubules confirmed that the failure was at the top of the hybrid layer. Triangles, resin tags; arrows, open dentin tubules.*

## DISCUSSION

Dentin is a biological composite of ordered mineralization of apatite on the collagen fibril matrix with a fluid-filled tubular structure.[28] Because of this heterogeneous and intrinsically wet substrate, a strong bond between dentin and resin depends on the infiltration of adhesive into the exposed collagen matrix to create micromechanical interlocks.[29,30] The moist dentin environment can prevent intimate contact between the resin and the collagen fibrils, forming incompletely infiltrated zones along the bottom of the hybrid layer.[31] Biodegradation of the nonencapsulated collagen is a primary cause of the decrease in dentin-resin bond durability.[32]

Laser irradiation improves dentin bond strength by removing the smear layer and collagen fibrils of the dentin surface and occludes dentin tubules by melting and recrystallizing the HAP crystallites.[5,33] However, because of the deeper pigment of dentin and the higher penetration depth of the Nd:YAG laser, it is possible that exposure of dentin surfaces to Nd:YAG laser energy may cause pulp damage if the rise in temperature is sufficiently high, so a safe protocol is needed. It was reported that when laser parameters do not exceed 1 W and 10 Hz and the thickness of the dentin exposed to Nd:YAG laser irradiation for 10 seconds is at least 1 mm, no significant pulp damage can be expected.[34] Laser treatment was suggested to replace acid etching as a pretreatment for dentin bonding,[6,35] whereas other research has demonstrated that laser decreased dentin bond strength.[36,37] Instead of replacing the acid etching system, we applied laser irradiation as an adjunctive treatment to modify the dentin surface and subsequently performed acid etching to improve the reliability and durability of dentin bond strength.

In the present study, laser irradiation to the dentin surface did not affect immediate bond

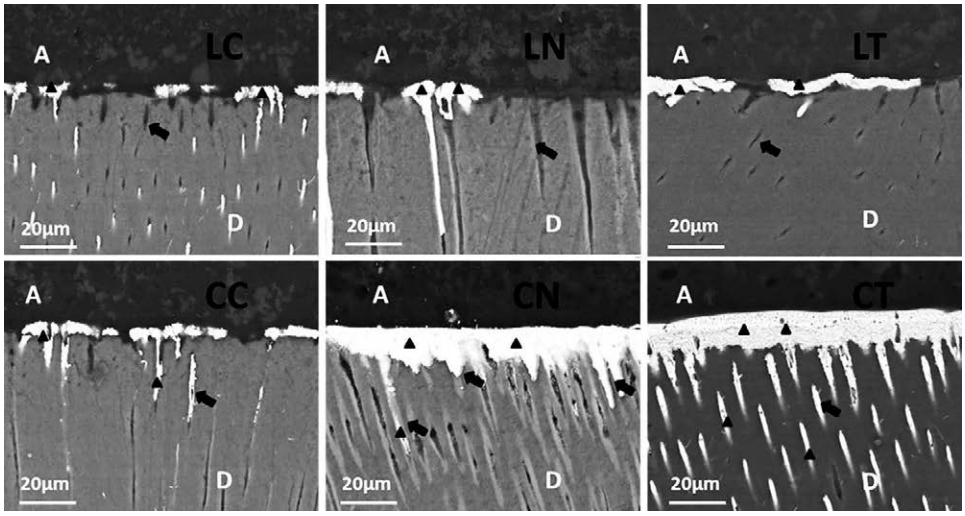


Figure 5. *Representative FE-SEM-BSE images (1000×) of interfacial nanoleakage expression of different groups. A, adhesive; D, dentin; arrows, dentin tubules; triangles, silver deposits; LC/LN/LT, laser-treated groups; CC/CN/CT, control groups. In the LC group, minimal silver deposition was detected along the hybrid layer. In artificial aging groups (LN/LT), more silver uptake occurred at the bottom of hybrid layer but not significantly. In particular, very little silver deposition was detected in the dentin tubules. In the CC group, sparse silver uptake could be observed along the hybrid layer within the adhesive layer. In the CN and CT groups, a large amount of silver uptake was continuously located along the bottom of hybrid layer and in the dentin tubules.*

strength. Aging bond strengths in the laser treatment groups were significantly higher than in the control groups (Table 1). The nanoleakage observations provided concordant results (Figure 5). Given the absence of collagen fibrils in the laser-irradiated dentin surface, resin polymers infiltrate the micropores of HAP created by acid etching instead of demineralized collagen matrices. This phenomenon leads to the formation of a stable dentin-resin bond interface that is insusceptible to the biodegradation *in vivo*. Methods of chemical and mechanical detection need to be further developed to explore the mechanism by which laser irradiation affects the dentin-resin bond strength.

XRD spectra indicated that the main components of both laser-treated and control dentin surfaces were HAP crystals and that the laser-irradiated dentin surface had similar crystallinity to the natural dentin HAP (Figure 2A). Although laser treatment increased the ratio of hydroxyapatite crystal to proteins and water, the microhardness test showed that it did not enhance the hardness of the dentin surface. These results demonstrated that laser irradiation did not influence the mechanical property and crystallographic features of dentin. SEM images showed that the laser-irradiated dentin integrated into the subsurface dentin structure and formed a homogeneous newly modified layer. Moreover, no cracks were observed between the newly formed layer and the dentin substrate (Figure 1). Laser irradiation can maintain the short-term dentin bond strength probably because of these characteristics.

The amide:phosphate ratio of ATR-FTIR spectra indicated that the relative collagen protein amount of the laser-irradiated dentin surface significantly decreased compared with that of the control group ($p<0.05$) (Figure 2C). Moreover, exposed collagen was hardly observed along the laser-pretreated dentin-resin interface as determined through Masson's trichrome staining. This result indicated that laser irradiation can remove the collagen from the dentin bonding interface. NaOCl is a nonspecific proteolytic agent that is widely used in various dental procedures.[38,39] In the present study, NaOCl was used to induce the collagen fiber degradation of the dentin-bond interface. Laser treatment maintained the MTBS and decreased the nanoleakage on the dentin-resin bond surface after immersion in 10% NaOCl solution. This behavior confirmed the speculation that laser irradiation may avoid dentin collagen degradation and increase dentin bond durability by removing the collagen fibers from the dentin-resin bond interface.

The application of acidic agents opens the pathway for the diffusion of monomers into the collagen network. It also facilitates the outward seepage of tubular fluid from the pulp to the dentin surface, deteriorating the bond of some of the current adhesives.[40,41] Wet dentin substrates also decrease the degree of polymerization. Therefore, the hydrophilic nature of the dentin matrix is another crucial factor that affects the durability of dentin bond strength.[42] The results of nanoleakage evaluation revealed less silver deposition in the laser-treated samples than in the control samples. The laser-treated samples barely had silver deposition in the dentin tubules after thermocycling or NaOCl aging (Figure 5). This result demonstrates that laser irradiation can effectively prevent water from in-

vading the dentin-resin bond interface, decrease water leakage on the dentin bond surface, and stabilize the dentin bond strength. These phenomena can be ascribed to three reasons. First, the diameter of the cufflike reopened dentin tubules on the laser-treated dentin surface was lower than that of the funnel-shaped sound dentin tubules after acid etching, confirmed via SEM. This surface morphology may increase dentin bond strength because dentin permeability depends on the size and patency of dentin tubules, and bond strength weakens with increasing dentin moisture content.[43,44] Second, the internal walls of laser-irradiated dentin tubules were smoother and denser, important because collagen fiber loss, porosity change, and denser structure can decrease the permeability of dentin fluid to the bond surface and facilitate the infiltration of bonding monomers into the substrate of dentin tubules. Finally, laser irradiation can vaporize water and other hydrated organic components of the dentin surface, subsequently forming a bond surface without the moist environment of the collagen matrix, and this likely promotes the penetration and polymerization of monomers.

Several approaches to improve long-term bonding have been developed and tested, including inhibition of dentinal endogenous proteases and improved penetration of the adhesive monomers into an ethanol-saturated exposed dentin matrix[3]. However, despite the promising results of *in vitro* research, clinically feasible and commonly accepted methods for improving long-term bonding are still lacking.[45] Lasers have beneficial and promising applications in dentistry. In the present study, laser irradiation combined with acid etching produced similar short-term bond strength and better long-term strength compared with acid etching alone. The pretreatment of the dentin surface with Nd:YAG laser irradiation provides a potential strategy for dentists to obtain the desired bond effectiveness during adhesion, resulting in a longer service life of restorations.

Further research should elucidate the mechanisms by which laser improves dentin bond durability, identify the optimal laser irradiation, and examine the effects of Nd:YAG laser irradiation and the structural transformations of teeth to establish Nd:YAG laser irradiation as a reliable operative technique that improves the durability of dentin bond strength. Most important, it was only demonstrated that this strategy was effective *in vitro*; whether it could achieve the same effect in *vivo* or whether it could be accepted for clinical application still needs to be confirmed in future studies.

## CONCLUSIONS

Laser irradiation can partially remove collagen fibers from the dentin surface but not influence its mechanical properties and crystallography. Laser treatment combined with acid etching as a pretreatment of the dentin-resin bond can maintain short-term dentin bond strength, reduce time-related nanoleakage, and increase dentin bond durability.

### Acknowledgements

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### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Ethics Committee for Human Studies, School of Stomatology, Wuhan University, China. The approval code for this study is 067.

### 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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# Microtensile Bond Strength of Composite Cement to Novel CAD/CAM Materials as a Function of Surface Treatment and Aging

DP Lise • A Van Ende • J De Munck • LCC Vieira • LN Baratieri • B Van Meerbeek

### Clinical Relevance

Novel composite and polymer-infiltrated ceramic CAD/CAM materials benefit from etching of the intaglio surface with hydrofluoric acid or sandblasting, both followed by silanization.


## SUMMARY

**Objectives: To evaluate the effect of different surface treatments on the bond strength to a composite and a polymer-infiltrated ceramic CAD/CAM block after six-month artificial aging.**

**Methods and Materials: Two types of CAD/CAM blocks (Cerasmart, GC; Enamic, Vita Zahnfabrik) were cut in slabs of 4-mm thickness, divided into six groups, and subjected to the following surface treatments: group 1: no treatment; group 2: sandblasting (SB); group 3: SB + silane (Si); group 4: SB + Si + flowable composite (see below); group 5: 5% hydrofluoric acid etching (HF) + Si; and group 6: 37% phosphoric acid etching ($H_3PO_4$) + Si. Sections of the same group were luted together (n=3: 3 sandwich specimens/group) using a dual-cure self-adhesive cement for all groups, except for the sections of group 4 that were luted using a light-curing flowable composite. After three weeks of storage in 0.5% chloramine at 37°C, the sandwich specimens were sectioned in rectangular microspecimens and trimmed at the interface to a dumbbell shape (1.1-mm diameter). One half of the specimens was subjected to a microtensile bond strength ($\mu$TBS) test, and the other half was tested after six months of water storage (aging). Data were**


Diogo Pedrollo Lise, DDS, MSD, PhD, Department of Operative Dentistry, Federal University of Santa Catarina (UFSC), Santa Catarina, Brazil

Annelies Van Ende, DDS, MSD, PhD, KU Leuven – BIOMAT Department of Oral Health Sciences, KU Leuven and Dentistry, University Hospitals Leuven, Leuven, Belgium

Jan De Munck, DDS, MSD, PhD, postdoctoral research fellow, KU Leuven – BIOMAT, Department of Oral Health Sciences, KU Leuven and Dentistry, University Hospitals Leuven, Leuven, Belgium

Luiz Clovis Cardoso Vieira, DDS, MSD, PhD, professor emeritus, Department of Operative Dentistry, Federal University of Santa Catarina (UFSC), Santa Catarina, Brazil

Luiz Narciso Baratieri, DDS, MSD, PhD, professor emeritus, Department of Operative Dentistry, Federal University of Santa Catarina (UFSC), Santa Catarina, Brazil

*Bart Van Meerbeek, DDS, MSD, PhD, full professor, KU Leuven – BIOMAT, Department of Oral Health Sciences, KU Leuven and Dentistry, University Hospitals Leuven, Leuven, Belgium

*Corresponding author: Kapucijnenvoer 7, Leuven 3000, Belgium; e-mail: bart.vanmeerbeek@kuleuven.be

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**statistically analyzed with a linear mixed-effects model for the factors surface treatment, material type, and aging, together with their first-degree interactions ($\alpha$=0.05).**

**Results: The lowest bond strengths were obtained in the absence of any surface treatment (group 1), while the highest $\mu$TBSs were obtained when the surface was roughened by either SB or HF, this in combination with chemical adhesion through Si. Loss in bond strength was observed after six-month aging when either surface roughening or silanization, or both, were omitted.**

**Conclusions: Both the composite and polymer-infiltrated ceramic CAD/CAM blocks appeared equally bonding-receptive regardless of the surface treatment used. Creating a microretentive surface by either SB or HF, followed by chemical adhesion using Si, is mandatory to maintain the bond strength after six months.**

## INTRODUCTION

Chairside CAD/CAM restorations have become increasingly popular in the latest decade thanks to recent improvements in CAD/CAM technology, which increased the ease of use and cost effectiveness of the restorations. Recently, new types of blocks containing either composite or both a polymeric and ceramic phase have been developed.[1] Cerasmart (GC, Tokyo, Japan) is a 'composite' block that consists of evenly dispersed ceramic nanoparticles (71 vol%) in a polymeric matrix,[2] providing a high flexural strength[3] and modulus of resilience.[1] Enamic (Vita Zahnfabrik, Bad Säckingen, Germany) is a so-called 'polymer-infiltrated ceramic' and presents a three-dimensionally interconnected pre-sintered ceramic network of 86 vol% that is infiltrated with a monomer mixture; thus, intertwined networks of polymers and ceramics are created.[4-6] The polymeric network is thought to render the material less brittle than classic ceramics, as inferred from its higher Weibull modulus.[7,8]

To be clinically successful, adequate adhesion of the restoration is very important. It has been shown[9-11] that surface treatment prior to cementation can enhance the bond strength to indirect restorations. Micromechanical retention can be provided through sandblasting or acid etching, while a silane coupling agent provides chemical bonding.[11] Different strategies are preferred depending on the material's characteristics.[12]

However, information about bonding protocols to new composite and polymer-infiltrated ceramic materials is scarce.[13-15] In a previous study,[14] it was suggested that bonding strategies were material-dependent. Therefore, the objective of this study was to evaluate the effect of different surface treatments on the bond strength of a self-adhesive composite cement to new CAD/CAM blocks. The null hypotheses were that 1) material type, 2) surface treatment, and 3) aging did not have an influence on the microtensile bond strength ($\mu$TBS) to either a composite or polymer-infiltrated ceramic CAD/CAM block.

## METHODS AND MATERIALS

The experimental procedure is schematically illustrated in Figure 1, and all of the materials that were used are listed in Table 1. Two types of CAD/CAM blocks (Cerasmart [CER], GC; and; Enamic [ENA], Vita Zahnfabrik) of 12 × 14 × 18 mm were sectioned using a diamond blade in slabs of 4-mm thickness and wet-polished with 600-grit silicon carbide paper for 30 seconds. Thirty-six slabs of each CAD/CAM material were selected and ultrasonically cleaned for five minutes in distilled water to remove surface contaminants. For each block type, the specimens were randomly divided into six groups of six 4-mm slabs in accordance with the surface treatment, as follows:

- Group 1: No surface treatment (NT).
- Group 2: Sandblasting (SB): the surface was sandblasted with 27-$\mu$m aluminum-oxide ($Al_2O_3$) particles perpendicular to the surface from a distance of 10 mm over the course of 20 seconds with a pressure of 0.28 MPa. Remaining particles were removed using a gentle air-blow for five seconds.
- Group 3: Sandblasting + silane (SB/Si): the surface was sandblasted following the same protocol as in group 2, and then a thin layer of a silane coupling agent (Ceramic Primer II, GC) was applied using a disposable microtip applicator. After 60 seconds, the surface was dried with an air syringe.
- Group 4: Sandblasting + silane + flowable composite (SB/Si/FLO): the surface treatment and silanization were carried out following the same protocol as in group 3; however, a flowable composite was used as luting agent.
- Group 5: Hydrofluoric acid etching + silane (HF/Si): the surface was etched with 5% hydrofluoric acid (HF; IPS Ceramic Etching Gel 5%, Ivoclar Vivadent, Schaan, Liechtenstein) for 60 seconds

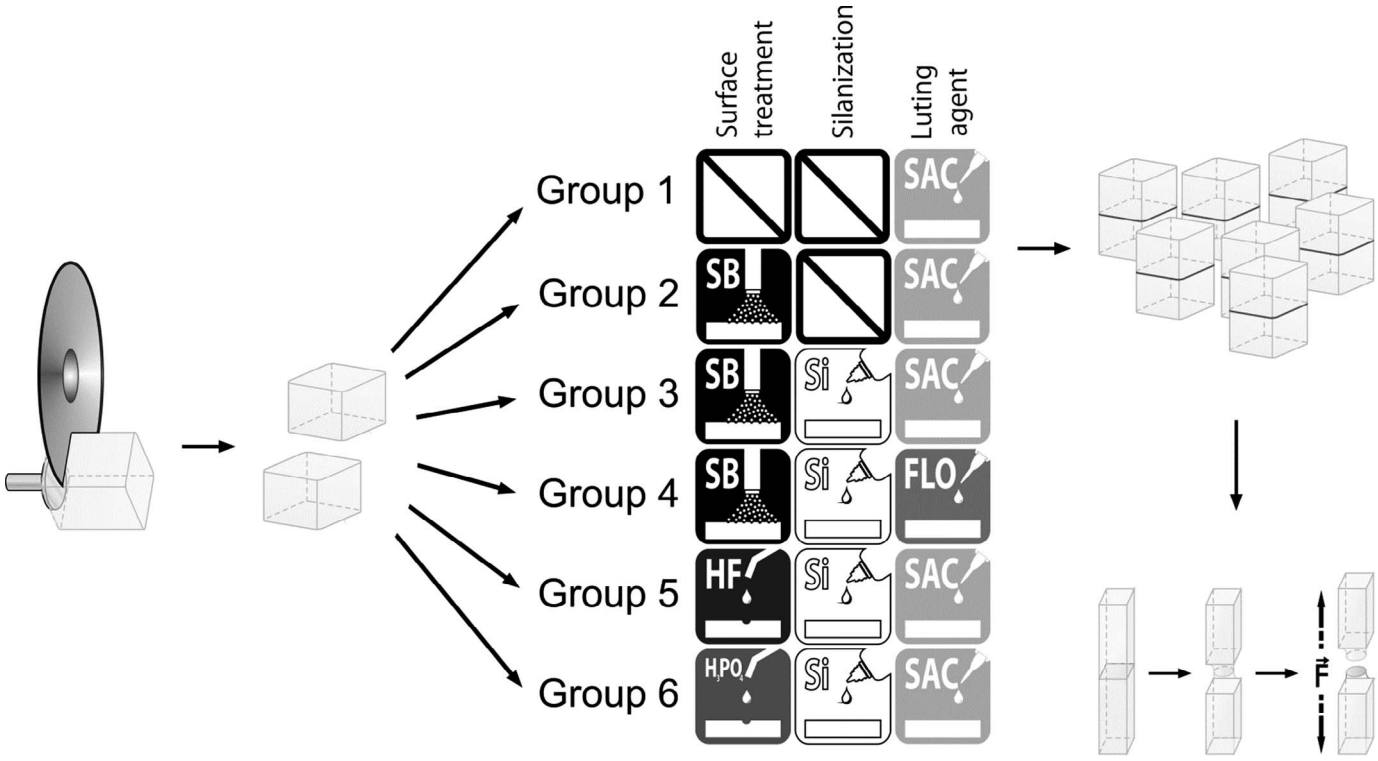


Figure 1. *Scheme explaining the study set. Standardized 4-mm slabs were cut from two types of CAD/CAM block materials and filled according to the respective experimental conditions, resulting in 2 (material) × 6 groups (surface treatment and silanization). Microspecimens were prepared and stressed until failure after three weeks and six months. SB: sandblasting with 27-μm $Al_2O_3$; Si: silane; HF: 5% hydrofluoric acid; $H_3PO_4$: 37% phosphoric acid; SAC: self-adhesive composite cement; FLO: flowable composite.*

and thoroughly rinsed by water spray for 60 seconds. Then the surface was cleaned ultrasonically in distilled water for five minutes and air-dried for 20 seconds. Silane was applied following the same protocol as in group 3.

- Group 6: Phosphoric acid etching + silane ($H_3PO_4$/Si): surface etching with 37% phosphoric acid (Total Etch, Ivoclar Vivadent) for 60 seconds, followed by water rinsing for 60 seconds and air-drying for 20 seconds. Silane was applied following the same protocol as in group 3.

Three pairs of 4-mm CAD/CAM slabs (12×14×4 mm) from the same CAD/CAM block material and the same group were luted together (3 sandwich specimens per group; n=3) using a self-adhesive composite cement (G-CEM LinkAce, GC), whereas a light-cured flowable composite (G-ænial Universal Flo, GC) was used to lute the slabs of group 4 (SB/Si/FLO). The cementation was performed under a constant weight of 1 kg over the course of 10 minutes. The excess luting agent was removed with a microtip applicator, and the cementation line was covered with a glycerin gel (Liquid Strip, Ivoclar Vivadent) in order to prevent the formation of an oxygen-inhibited layer. After the initial five minutes, the sandwich specimens were light-cured from each side for 40 seconds using a LED light-curing unit (Prima Light, GC) with an output of ~1600 mW/cm$^2$, as measured by the MARC Patient Simulator (Blue-Light Analytics, Halifax, NS, Canada). Specimens were stored in 0.5% chloramine solution for three weeks at 37°C. Next, each sandwich specimen was sectioned perpendicularly to the luting interface using an automated water-cooled diamond saw (Accutom-50, Struers, Ballerup, Denmark) to obtain up to six microspecimens (1.7×1.7×8 mm). Each microspecimen was trimmed at the interface to a dumbbell shape (1.1±0.1 mm diameter) with a cylindrical extrafine-grit (15 μm) diamond bur fixed in a water-cooled high-speed handpiece mounted in a computer-controlled lathe (MicroSpecimen Former, University of Iowa, Iowa City, IA, USA). Next, the cross-sectional diameters of the dumbbell-shaped specimens were measured with an X-Y multipurpose modular measuring microscope equipped with a digital readout (Leitz VRZ-U, Wetzlar, Germany) to an accuracy of 0.001 mm. One half of the microspecimens were stored in 0.5% chloramine at 37°C for three weeks and the other half during six months before testing. Upon testing, they were attached to a

Table 1: *Materials Used in This Study*

| Materials | | Composition | Batch No. |
|---|---|---|---|
| Composite CAD/CAM block | Cerasmart, GC, Tokyo, Japan | •Silica (20 nm) and barium glass (300 nm) nanoparticles (71 wt.%)<br>•Polymers (29%) of Bis-MEPP, UDMA, and DMA | 1403101 |
| Polymer-infiltrated CAD/CAM block | Enamic, Vita Zahnfabrik, Bad Säckingen, Germany | •Feldspar ceramic reinforced by oxides (86 wt.%)<br>•Polymers (14%) of UDMA and TEGDMA | 53570 |
| Luting agents | G-CEM LinkAce, GC | •Paste A: UDMA 10%-20%, Y-methacryloxypropyltrimethoxysilane >2.5%<br>•Paste B: UDMA 25%-50%, methacryloxypropyltrimethoxysilane >2.5-10%, A,α-dimethylbenzylhydroperoxide | 1401151 |
| | G-ænial Universal Flo, GC | •Matrix: UDMA, Bis-MEPP, TEGDMA<br>•Fillers (69 wt.%): silicon dioxide (16 nm) and strontium glass (200 nm)<br>•Others: pigments, photoinitiator | 1312072 |
| Silane | Ceramic Primer II, GC | 90%-100% ethanol, 1%-5% 2,2′-ethylene dioxydiethyl dimethacrylate, 1%-5% methacryloyloxydecyl dihydrogen phosphate, <1% (1-methylethylidene) bis[4,1-phenyleneoxy(2-hydroxy-3,1- propanediyl)] bismethacrylate | 14011222 |
| Hydrofluoric acid (HF) | IPS Ceramic Etching Gel 5%, Ivoclar Vivadent, Schaan, Liechtenstein | Aqueous solution of hydrofluoric acid (<5%) | S51072 |
| Phosphoric acid ($H_3PO_4$) | Total Etch 37%, Ivoclar Vivadent | Phosphoric acid (37%), water, | S29080 |
| Sandblasting particles | Aluminum-oxide 27-μm, Danville Materials, San Ramon, CA, USA | Aluminum-oxide 27-μm particles | 28482 |

*Abbreviations: Bis-MEPP, bisphenol A ethoxylate dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.*

notched BIOMAT jig[16] with cyanoacrylate glue (Model Repair II Blue, Dentsply-Sankin, Tochigiken, Japan) and stressed until failure in a universal testing device (Instron 5848 Micro Tester, High Wycombe, Bucks, UK) at a crosshead speed of 1 mm/min, using a load cell of 500 N. When a specimen broke during the trimming procedure, it was recorded as a pretesting failure (ptf). Microtensile bond strength data (μTBS per microspecimen in MPa) were statistically analyzed using a linear mixed-effects model. Fixed effects included in the model were the factors 'surface treatment' (groups 1 to 6), 'material type' (CER vs ENA), and 'aging' or storage period (three weeks vs six months), along with their first-degree interactions. The sandwich blocks were added to the model as a random factor to account for the multiple testings per block. In addition, to evaluate the six-month results more profoundly, specific contrasts, along with a $p$-value corrected for the amount of tests, were calculated in order to compare the six-month results to their respective negative (group 1: NT) and positive (group 3: SB/Si) control. Group 3 (SB/Si) was selected as the positive control as a result of the considerable polymer phase of both materials. All tests were performed at a significance level of $\alpha = 0.05$ using a software package.[17]

The mode of failure was assessed using scanning electron microscopy (SEM; JSM-6610LV SEM, Jeol, Tokyo, Japan) at a magnification of 70×, and failures were classified as either 'interfacial' (between the luting agent and the CAD/CAM block material and/or within the luting agent) or 'mixed' (involvement of both interfacial fracture and cohesive fracture within the CAD/CAM block substrate). Additional CAD/CAM block slabs were prepared following the previous surface treatment protocols (NT, SB, HF, and $H_3PO_4$) in order to morphologically analyze the surface topography using SEM. Specimens were mounted on aluminum stubs with adhesive carbon tape (PELCO Carbon Conductive Tape, Ted Pella Inc, Redding, CA, USA) and sputter-coated with gold-palladium by means of a sputter-coater (JFC-1300 Autofine Coater, Jeol) under a chamber pressure of 30 mA/Pa for 120 seconds. Specimens

Table 2: *μTBS Results*

| Group | Surface Treatment | Silane | Luting Agent | Material | 3-wk Storage | | | 6-mo Storage | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Mean (SD) | ptf/n | Mixed Failure/ Interfacial Failure, % | Mean (SD) | ptf/n | Mixed Failure/ Interfacial Failure, % |
| 1 | NT | No | SAC | Cerasmart | 40.5 (11.5) | 0/15 | 3/97 | 0.9 (3.0) | 16/18 | 3/97 |
| | | | | Enamic | 40.5 (13.7) | 0/15 | 17/83 | 10.4 (9.5) | 3/18 | 3/97 |
| 2 | SB | No | SAC | Cerasmart | 47.7 (20.2) | 0/17 | 12/88 | 32.6 (8.1) | 0/17 | 3/97 |
| | | | | Enamic | 56.8 (16.3) | 0/16 | 15/85 | 23.2 (13.8) | 2/18 | 22/78 |
| 3 | SB | Yes | SAC | Cerasmart | 53.3 (18.2) | 0/16 | 13/88 | 48.2 (9.6) | 0/17 | 17/83 |
| | | | | Enamic | 48.1 (14.9) | 0/17 | 38/62 | 58.8 (19.7) | 0/18 | 47/53 |
| 4 | SB | Yes | FLO | Cerasmart | 48.2 (20.5) | 0/18 | 6/94 | 48.4 (16.9) | 0/17 | 17/83 |
| | | | | Enamic | 52.8 (17.8) | 0/17 | 22/78 | 41.2 (13.2) | 0/13 | 31/69 |
| 5 | HF | Yes | SAC | Cerasmart | 50.8 (11.3) | 0/18 | 14/86 | 50.0 (18.4) | 0/15 | 17/83 |
| | | | | Enamic | 53.0 (21.6) | 0/18 | 33/67 | 52.9 (7.1) | 0/14 | 44/56 |
| 6 | $H_3PO_4$ | Yes | SAC | Cerasmart | 40.2 (16.4) | 0/17 | 9/91 | 30.0 (11.2) | 0/18 | 11/89 |
| | | | | Enamic | 48.7 (14.8) | 0/16 | 24/76 | 26.0 (15.6) | 2/14 | 22/78 |

*Abbreviations: FLO: flowable composite; HF: etching with 5% hydrofluoric acid; $H_3PO_4$: etching with 37% phosphoric acid; n: number of microspecimens; NT: no treatment; ptf: pre-testing failures; SB: sandblasting with 27-μm $Al_2O_3$; SAC: self-adhesive composite cement; SD: standard deviation.*

were observed under SEM at an accelerating voltage of 15 kV and a working distance of 11.0 mm.

## RESULTS

The lowest bond strengths were obtained in the absence of any surface treatment (Table 2). No effect was found for the 'material type' ($p=0.97$), while significant effects of the factors 'surface treatment' ($p<0.0001$) and 'aging' ($p<0.0001$) were found. A highly significant interaction between surface treatment and aging ($p<0.0001$) was found as well. After three-week storage, there were no significant differences in $\mu$TBS values between the experimental groups. After six months, any surface treatment (groups 2-6) resulted in significantly higher bond strengths than were noted in the negative control (group 1: NT), which didn't receive any treatment (Figure 2). However, when silanization was omitted (group 2: SB), the results were significantly lower than those of the positive control (group 3: SB/Si) when both sandblasting and silanization were applied. Substituting the sandblasting for etching with HF (group 5: HF/Si) or substituting the self-adhesive composite cement for a flowable composite (group 4: SB/Si/FLO) rendered results that were not significantly different from that of the positive control (group 3: SB/Si). In contrast, substituting the sandblasting for etching with $H_3PO_4$ (group 6: $H_3PO_4$/Si) was not sufficient and resulted in a significant decrease in bond strength in comparison with that of the positive control (group 3: SB/Si).

SEM analysis of the surface treatments demonstrated that the untreated surface of CER had a smoother appearance than that of ENA. While roughening effects could be seen for both types of CAD/CAM blocks after SB and HF (Figure 3), surface treatment with $H_3PO_4$ did not result in a visible morphological difference. SB resulted in an irregular, rugged surface in both CAD/CAM block materials, while HF created porelike holes in the surface of CER, having dissolved the silica and barium-glass nanoparticles, but resulted in a more rugged appearance when dissolving the feldspathic ceramic network of ENA (Figure 3). Failure analysis demonstrated a higher prevalence of mixed fractures with ENA than with CER (Table 2; Figure 4).

## DISCUSSION

This *in vitro* study was designed to investigate the effect of various surface treatments on the adhesion of a dual-cure, self-adhesive composite cement to two novel CAD/CAM block materials after two storage times. Despite inherent differences in surfaces (Figure 3), no differences in bond strength to CER vs ENA were found (Table 2); thus, the first null hypothesis was accepted. Qualitative analysis using SEM showed that the untreated surface of ENA was rougher than the surface of CER (Figure 3); this might explain why there were fewer pretesting failures in ENA/NT than in CER/NT after six months (Table 2), although this difference was not significantly different ($p=0.084$).

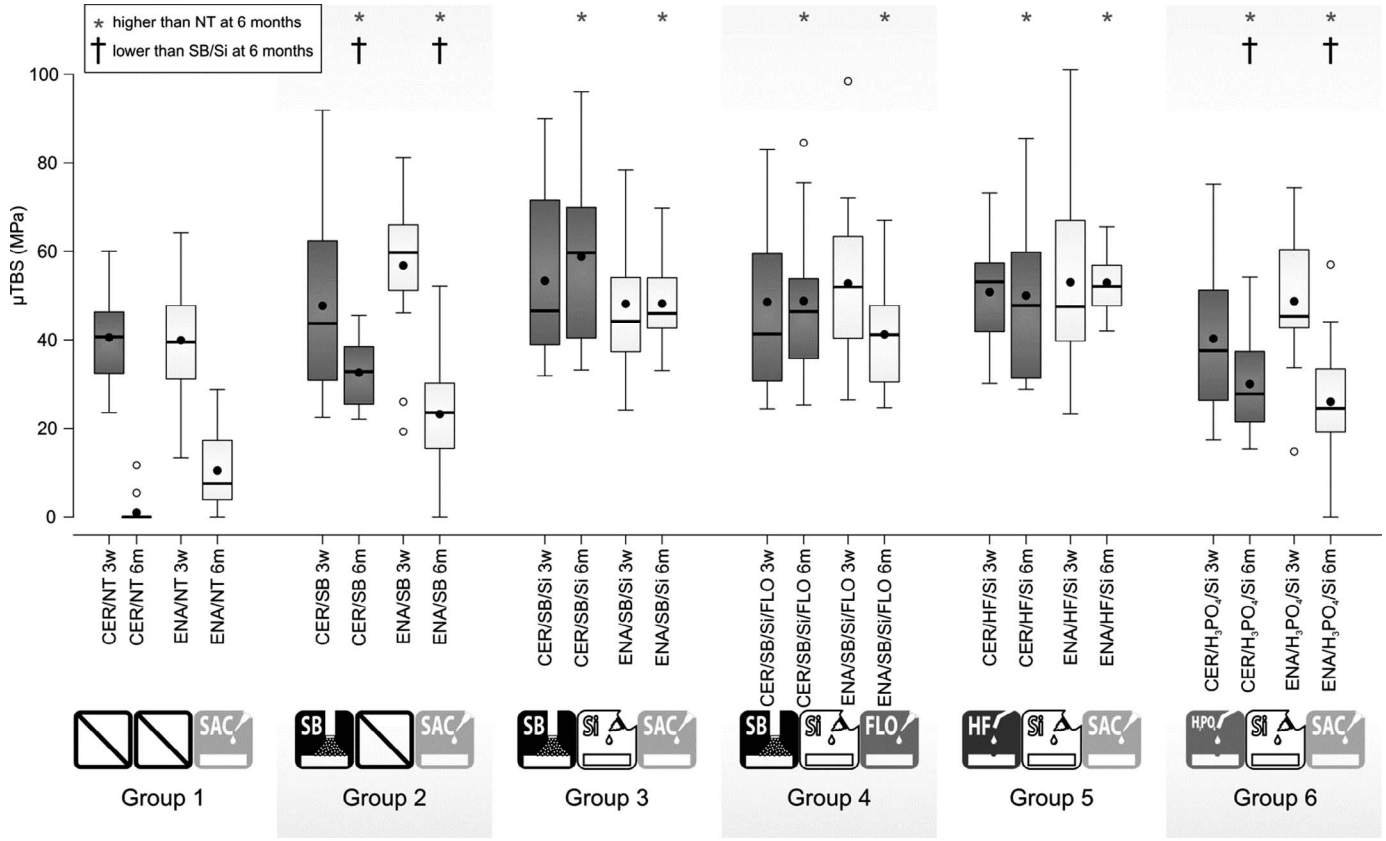


Figure 2. *Boxplots of the μTBS results. The box represents the spreading of the data between the first and third quartile. The central horizontal line and the black dot represent the median and mean, respectively. The whiskers extend to the minimum and maximum values measured, with the exception of the outliers that are represented with open dots (°). CER: Cerasmart; ENA: Enamic; NT: no treatment; SB: sandblasting with 27-μm $Al_2O_3$; Si: silane; HF: 5% hydrofluoric-acid etching; $H_3PO_4$: 37% phosphoric-acid etching; SAC: self-adhesive composite cement; FLO: flowable composite. Significant differences are based on linear mixed-effects models at a significance level of* $p=0.05$.

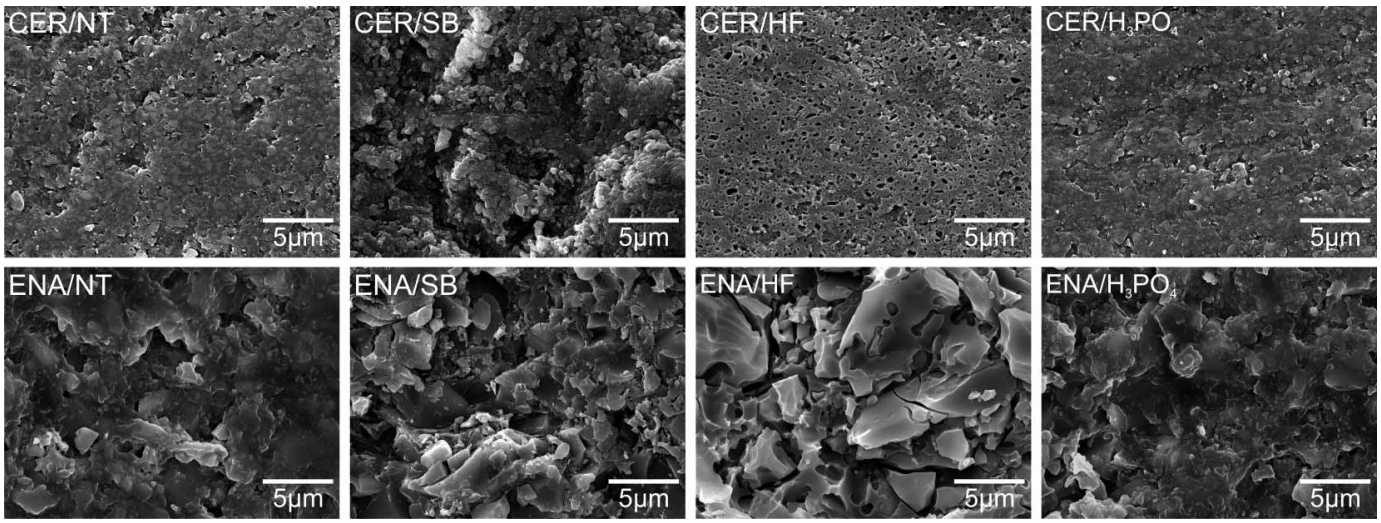


Figure 3. *SEM photomicrographs of pretreated surfaces. CER: Cerasmart (GC); ENA: Enamic (Vita Zahnfabrik); NT: no treatment; SB: sandblasting with 27-μm $Al_2O_3$; HF: 5% hydrofluoric-acid etching; $H_3PO_4$: 37% phosphoric-acid etching. Original magnification: 5000×.*

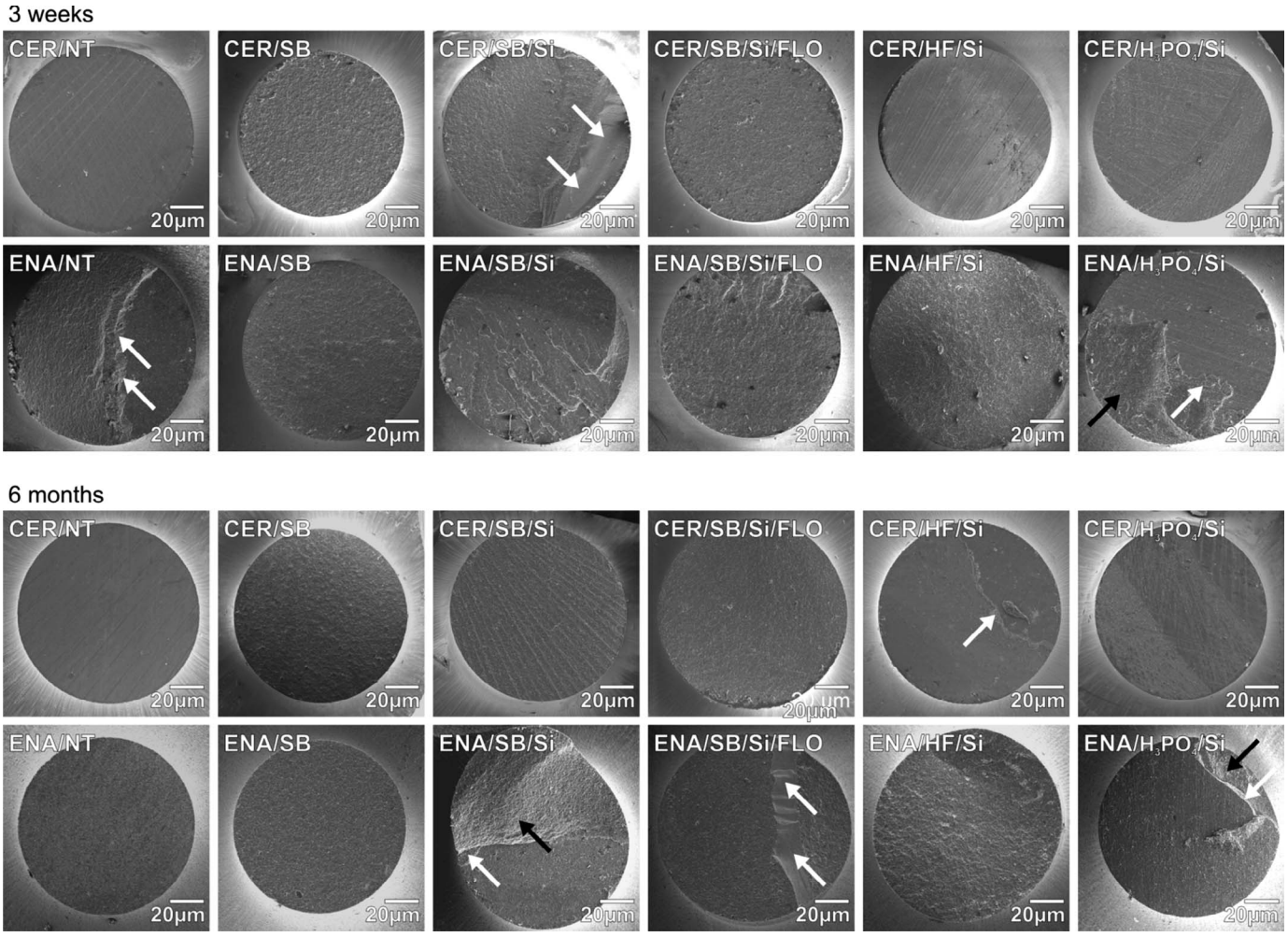


Figure 4. *SEM photomicrographs of μTBS-fractured surfaces. CER: Cerasmart (GC); ENA: Enamic (Vita Zahnfabrik); NT: no treatment; SB: sandblasting with 27-μm $Al_2O_3$; HF: 5% hydrofluoric-acid etching; $H_3PO_4$: 37% phosphoric-acid etching; Si: silane; FLO: flowable composite. White arrows: interfacial failure at the composite cement. Black arrows: mixed failure with involvement of CAD/CAM block substrate. Note that more mixed failures occurred within the CAD/CAM block substrate with ENA than with CER. Original magnification: 70×.*

It must be noted that the CER used in this study contains etchable barium-glass particles and has a lower hardness[3] as opposed to the zirconia-containing composite CAD/CAM block material used in previous studies.[14,15] As a consequence, CER is more susceptible to mechanical roughening and acid etching.[18] It is clear that differences within material classes must be interpreted with caution[19] and that those findings cannot be generalized to all composite CAD/CAM block materials.

Both surface treatment and storage period (aging) had a significant influence on bond strength, with a significant interaction between both factors ($p<0.001$); hence, the second and third null hypotheses were rejected. Overall, high levels of bond strength were found after only three weeks of water storage (Table 2). Differences between the surface treatments only manifested after a longer water storage period, which affected the bond strength, depending on the surface treatment. The lowest bond strengths were obtained in the absence of any surface treatment (group 1: NT), which became apparent after six-month storage. After this period of six months, any surface treatment resulted in a higher bond strength in comparison with the negative control NT (Figure 2). However, when compared to group 3 (SB/Si), which served as a positive control, SB alone (group 2) resulted in significantly lower bond strengths (Figure 2). This shows that the silanization step is crucial to maintain an optimal adhesion, which was also demonstrated for conventional ceramics[20,21] and

indirect composites.[22,23] Despite the presence of polymers in both CAD/CAM block materials, their inorganic content remained relatively high so that the extra coupling provided by the silane between the inorganic and polymer constituents of the luting agent enhanced the bond strength.[24] However, mechanical roughening is also necessary; in group 6 ($H_3PO_4$/Si), $H_3PO_4$ was not strong enough to induce visible surface roughening, and despite silanization lower bond strengths were obtained than in the positive control group 3 (SB/Si). Similar results have been found for conventional ceramics,[11,25] composites,[26] and polymer-infiltrated ceramics.[27] Surface roughness of the $H_3PO_4$-etched surface was similar to that of the untreated surface, but it is thought to have a cleaning effect.[18,27] $H_3PO_4$ might be preferred over HF as a surface treatment for intraoral repair because of the potential hazards of the latter material[28]; however, based on these results, it can be concluded that bonding in the long term might be compromised in the absence of sufficient micromechanical retention.

Fracture analysis revealed that the majority of specimens failed at the interface, which indicates that the stress was concentrated in this area during the tensile test.[29] More mixed failures, with large parts of cohesive fractures in the substrate, were seen for ENA; this might be a result of the inherent higher brittleness of this material in comparison with the more resilient CER. Indeed, it has been shown[1] that ENA has a lower flexural strength than CER. Flexural strength is closely related to tensile strength, and this might explain why failures propagated more often through the substrate in ENA.

A dual-cure, self-adhesive composite cement (G-CEM LinkAce, GC) was used to lute all of the specimens, except for those of group 4 (SB/Si/FLO), for which a light-curing flowable composite (G-ænial Universal Flo) was used as luting agent. Interestingly, the latter was found to be equally effective as the dual-cure composite cement with both CAD/CAM block materials (Figure 2). It must be kept in mind, however, that in this case the sandwich specimens were extensively light-cured from each side and that as a result of the flat interface of the specimens, a beneficial ratio between the circumference—which can be exposed directly to the light—and intaglio surface was created. To extrapolate this finding to clinical situations, however, more studies regarding the degree of conversion of these composites under composite/ceramic restorations of various thicknesses are necessary.

## CONCLUSIONS

Both the composite and polymer-infiltrated ceramic blocks performed equally well regardless of surface treatment. As previously shown for other indirect CAD/CAM materials,[13,20,30-33] creating a microretentive surface by either sandblasting or hydrofluoric-acid etching, followed by silanization for chemical adhesion, is mandatory to maintain the bond strength upon water storage for six months.

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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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# Degree of Conversion and Polymerization Shrinkage of Bulk-Fill Resin-Based Composites

P Yu • AUJ Yap • XY Wang

### Clinical Relevance

Bulk-fill resin-based composites (RBCs) are not a homogeneous group of materials. Their degree of conversion and polymerization shrinkage are product/depth dependent. Bulk-fill RBCs should generally not be cured in more than 4-mm increments. As the degree of conversion of some bulk-fill products is low, they should be used with caution clinically.


## SUMMARY

**This study evaluated the degree of conversion (DC) and polymerization shrinkage (PS) of contemporary bulk-fill resin-based composites (RBCs) including giomer materials. Two giomer bulk-fill (Beautifil Bulk Restorative [BBR], Beautifil Bulk Flowable [BBF]), two nongiomer bulk-fill (Tetric N-Ceram Bulk-fill [TNB], Smart Dentin Replacement [SDR]), and three conventional non–bulk-fill (Beautifil II [BT], Beautifil Flow Plus [BF], Tetric N-Ceram [TN]) RBCs were selected for the study. To evaluate the DC, disc-shaped specimens of 5-mm diameter and 2-mm, 4-mm, and 6-mm thickness were fabricated using customized Teflon molds. The molds were bulk filled with the various RBCs and cured for 20 seconds using a light-emitting diode curing light with an irradiance of 950 mW/cm$^2$. The DC (n=3) was determined by attenuated total reflectance Fourier transform infrared spectroscopy by computing the spectra of cured and uncured specimens. PS (n=3) was measured with the Acuvol volumetric shrinkage analyzer by calculating specimen volumes before and after light curing. The mean DC for the various materials ranged from 46.03% to 69.86%, 45.94% to 69.38%, and 30.65% to 67.85% for 2 mm, 4 mm, and 6 mm, respectively. For all depths, SDR had the highest DC. While no significant difference in DC was observed between depths of 2 mm and 4 mm for the bulk-fill RBCs, DC at 2 mm was significantly greater than 6 mm. For the conventional RBCs, DC at 2 mm was significantly higher than at 4 mm and 6 mm. Mean PS ranged from 1.48% to 4.26% for BBR and BF, respectively. The DC at 2 mm and PS of bulk-fill RBCs were lower than their conventional counterparts. At 4 mm, the DC of giomer bulk-fill RBCs was lower than that of nongiomer bulk-fill materials.**



Peng Yu, BDS, Peking University School and Hospital of Stomatology, Department of Cariology and Endodontology, Beijing, China

Adrian U J Yap, PhD, MSc, BDS, National University of Singapore, Faculty of Dentistry, Singapore

*Xiao-yan Wang, BDS, MD, PhD, Peking University School and Hospital of Stomatology, Department of Cariology and Endodontology, Beijing, China

*Corresponding author: 22 Zhongguancun South Street, Haidian District, Beijing 100081, China; e-mail: wangxiaoyan@pkuss.bjmu.edu.cn


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## INTRODUCTION

Chemically cured resin-based composites (RBCs) were first introduced as a replacement for silicate cements and autopolymerizing acrylic materials. Although they are esthetically superior, their poor clinical performance did not permit their use in posterior teeth.[1] With developments in dental RBC/adhesive technologies and trends toward more conservative cavity preparations, light-cured RBCs are now routinely used to restore posterior teeth and account for approximately half of all posterior direct restorations placed.[2] Posterior RBC restorations are, however, technically challenging and time consuming to perform as they require incremental 2 mm material placements and light curing due to depth of cure and polymerization shrinkage (PS) issues. Inadequately cured RBCs have reduced physicomechanical qualities and chemical stability and are potentially toxic to pulpal tissues.[3,4] Cited deleterious effects of polymerization-induced shrinkage stress include marginal leakage, gap formation, cuspal defection, tooth cracking, and reduced mechanical properties of RBC restorations.[5] In addition to increased clinical time and technical complexities, other disadvantages of the incremental filling technique include reduced bond strengths as well as voids, contamination, and bond failures between adjacent RBC layers.[6-9]

Innovative bulk-fill RBCs with claims of enhanced depth of cure and reduced PS have been introduced to the dental profession. These materials can apparently be placed in 4-mm increments, reducing the time and effort required for layering and adapting posterior RBCs. Strategies used to achieve bulk filling included the use of novel proprietary resins, special modulators, unique fillers, and filler control. At such increments, light transmission through RBCs may be compromised, leading to reduced monomer to polymer conversion.[10] The depth of cure and degree of conversion (DC) of bulk-fill RBCs have been investigated using a variety of methods. These include the ISO scraping test,[11] microhardness test, Fourier transform infrared (FTIR), and Raman spectroscopy. While some studies have conveyed adequate cure and DC at a depth of 4 mm,[12-15] others have reported contradictory outcomes.[16,17] The variances can be attributed to disparities in testing methodologies as well as materials evaluated. Bulk-fill RBCs are not a homogeneous group of materials, and differences in chemistry, viscosities, filler type, and quantity exist. PS studies showed that bulk-fill RBCs generally have lower shrinkage than conventional materials.[17-19]

DC and PS data on recently launched giomer bulk-fill materials are, however, scarce.[20,21] Giomers are based on prereacted glass ionomer (PRG) filler technology and are also known as PRG-RBCs. PRG fillers are obtained by reacting fluoride-containing glass with polyacids in the presence of water. The resultant product is freeze-dried, milled, silanized, and ground prior to being incorporated into silica-filled resins. Giomer RBCs are capable of fluoride release/recharge[22] and possess antibacterial properties.[23,24] Clinical qualities of most posterior giomer restorations were found to be acceptable even after 13 years of service.[25] Use of PRG fillers in giomer bulk-fill restoratives may, however, affect both DC and PS. The null hypotheses were as follows: 1) no significant differences exist in DC and PS between contemporary bulk-fill RBCs including giomer materials and 2) cavity depths do not influence DC of the various bulk-fill and conventional RBCs.

## METHODS AND MATERIALS

The materials selected for the study included two giomer bulk-fill (Beautifil Bulk Restorative [BBR], Beautifil Bulk Flowable [BBF]), two nongiomer bulk-fill (Tetric N-Ceram Bulk-fill [TNB], Smart Dentin Replacement [SDR]), and three conventional non–bulk-fill (Beautifil II [BT], Beautifil Flow Plus [BF], Tetric N-Ceram [TN]) RBCs. The technical profiles of the materials are reflected in Table 1.

Customized Teflon molds with cylindrical recesses of 5-mm diameter and 2-mm, 4-mm, and 6-mm depths were fabricated. A transparent polyester strip (Striproll, KerrHawe, Bioggio, Switzerland) was placed at the bottom of the molds. The molds were then bulk filled with the various RBCs in a single increment, and excess material was extruded by application of pressure through a glass slide. The materials were then light polymerized from the top surface using a light-emitting diode (LED) curing light (BluePhase, Ivoclar Vivadent, Shaan, Liechtenstein) with a curing tip diameter of 8 mm and an irradiance of 950 mW/cm$^2$ for 20 seconds. The irradiance of the curing light was assessed with an LED light tester (FB-M2000A, Fibop Medical Instrument, Foshan, China). Disc-shaped specimens (n=3) of 5 mm diameter and 2-mm, 4-mm, and 6-mm thickness were obtained. The DC of the bottom surfaces of the specimens was measured immediately after light polymerization using an FTIR spectrometer (Tensor 27, Bruker Optics, Ettlingen, Germany) with an attenuated total reflectance

Table 1: *Technical Profiles of the RBCs Evaluated in the Study*

| Materials | Abbreviation | Type | Composition | | Filler Content (wt%/vol%) | Filler size (μm) | Shade | Manufacturer | Lot No. |
|---|---|---|---|---|---|---|---|---|---|
| | | | Resin | Filler | | | | | |
| Beautifil Bulk Restorative | BBR | Bulk-fill restoratives | Bis-GMA, UDMA, Bis-MPEPP, TEGDMA | S-PRG filler based on fluoroboroalumino-silicate glass | 87.0/74.5 | N.A | U | Shofu Inc, Kyoto, Japan | 011510 |
| BeautifilII | BT | Conventional restoratives | Bis-GMA, TEGDMA | Multifunctional glass filler, S-PRG filler based on fluoro-boroaluminosilicate glass | 83.3/68.6 | 0.01-4, mean 0.8 | A1 | Shofu Inc, Kyoto, Japan | 041460 |
| Beautifil Bulk Flowable | BBF | Bulk-fill flowables | Bis-GMA, UDMA, Bis-MPEPP, TEGDMA | S-PRG filler based on fluoroboroalumino-silicate glass | 72.5/NA | NA | U | Shofu Inc, Kyoto, Japan | 011506 |
| Beautifil Flow Plus F00 | BF | Conventional flowables | Bis-GMA, TEGDMA | Multifunctional glass filler, S-PRG filler based on fluoroboro-aluminosilicate glass | 67.3/47.0 | 0.01-4, mean 0.8 | A1 | Shofu Inc, Kyoto, Japan | 011530 |
| Tetric N-Ceram Bulk Fill | TNB | Bulk-fill restoratives | Dimethacrylates | Barium glass, ytterbium trifluoride, mixed oxide, and copolymers | 80-81/55-57 | 0.04-3 | A1 | Ivoclar Vivadent AG, Schaan, Liechtenstein | S24104 |
| Tetric N-Ceram | TN | Conventional restoratives | Dimethacrylates | Barium glass, prepolymer, ytterbium trifluoride, and mixed oxide | 75-77/53-55 | 0.04-3, mean 0.6 | IVA | Ivoclar Vivadent AG, Schaan, Liechtenstein | T29061 |
| Smart Dentin Replacement | SDR | Bulk-fill flowables | Modified UDMA, EBPADMA, TEGDMA | Barium-alumino-fluoro-borosilicate glass, strontium alumino-fluoro-silicate glass | 68/45 | Mean 4.2 | U | Dentsply Caulk, Milford, DE, USA | 1410000524 |

*Abbreviation: Bis-GMA, bisphenol A glycidyl dimethacrylate; Bis-MPEPP, 2,2-bis[(4-methacryloxy polyethoxy)phenyl)propane; EBPADMA, ethoxylated bisphenol A dimethacrylate; NA, not available; S-PRG: surface prereacted glass ionomer; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.*

(ATR) accessory (MIRacle, PIKE Technologies, Madison, WI, USA). The ATR crystal was placed in close contact with the bottom surface, and FTIR spectra ranging from 600 to 4000 $cm^{-1}$ were documented by 32 scans at a resolution of 4 $cm^{-1}$. The FTIR spectra of uncured RBCs were recorded at the start of the experiment, and DC was calculated using the following formula:

$$DC\% = [1 - \text{Cured}\,(1638 cm^{-1}/\text{internal standard})/\text{Uncured}\,(1638\ cm^{-1}/\text{internal standard})] \times 100$$

The peak height around 1638 $cm^{-1}$, indicating the absorbance intensities of aliphatic C=C, was calibrated according to Rueggeberg and others.[26] The Peak of aromatic C=C around 1608 $cm^{-1}$ (1600 $cm^{-1}$ for SDR due to the lack of aromatic C=C) was taken as the internal standard and also calibrated.

PS (n=3) was measured by means of a video imaging device (Acuvol volumetric shrinkage analyzer, Bisco Inc, Schaumburg, IL, USA) in volumetric reconstruction mode. The RBC specimens were manually shaped into a hemisphere and placed on the rotational polytetrafluoroethylene pedestal inside the Acuvol chamber in front of the CCD camera. The specimens were imaged at a distance of 10 cm and subsequently irradiated for 20 seconds using the same LED curing light as for DC. The images were digitized and analyzed with the proprietary image-processing software. The volume of the specimens before and after curing was recorded as $V_1$ and $V_2$, respectively. The PS for the various RBCs was calculated as follows:

$$PS\% = [(V_1 - V_2)/V_1] \times 100$$

Statistical analysis was carried out using SPSS version 20.0 (IBM SPSS Inc, Chicago, IL, USA). DC

Table 2: *Mean Degree of Conversion (Standard Deviations) for the Various RBCs at the Various Depths*[a]

| Material | 2 mm | 4 mm | 6 mm |
|---|---|---|---|
| BBR | 46.03 (0.66)$^{Aa}$ | 45.94 (0.66)$^{Aa}$ | 41.26(0.19)$^{Ba}$ |
| BT | 62.78 (0.43)$^{Abc}$ | 56.38 (0.14)$^{Bb}$ | 35.24(0.79)$^{Cb}$ |
| BBF | 55.53 (2.03)$^{Adf}$ | 53.17 (0.81)$^{ABbc}$ | 49.04(3.20)$^{Bc}$ |
| BF | 66.11 (3.49)$^{Abe}$ | 50.80 (2.05)$^{Bc}$ | 30.65(0.56)$^{Cd}$ |
| TNB | 56.22 (0.24)$^{Adf}$ | 55.46 (2.02)$^{Ab}$ | 49.02(1.35)$^{Bc}$ |
| TN | 59.78 (0.17)$^{Acf}$ | 56.11 (0.32)$^{Bb}$ | 38.51(0.73)$^{Cab}$ |
| SDR | 69.86 (0.41)$^{Ae}$ | 69.38 (0.14)$^{Ad}$ | 67.85(0.93)$^{Be}$ |

[a] *Different uppercase letters in each row and different lowercase letters in each column indicate significant differences within the same materials and in the same increments, respectively ($p<0.05$; Tukey HSD test).*

and PS data were subjected to one-way analysis of variance and Tukey HSD test at a significance level of 0.05. Differences in DC and PS between RBCs as well as differences in DC between depths of 2 mm, 4 mm, and 6 mm for each material were examined. The relationship between DC at 2 mm and PS was also analyzed with Pearson's correlation at a significance level of 0.05.

## RESULTS

Mean DC and standard deviations of the various RBCs at the different depths are reflected in Table 2. Table 3 shows the DC obtained at 4 mm and 6 mm expressed as a percentage of DC at 2 mm. The latter was used as the reference as it offered the highest DC in our study and was the recommended depth of cure for most conventional RBCs. Mean PS and standard deviations of the various RBCs are shown in Table 4. Results of statistical analysis of DC and PS are displayed in Tables 2 and 4, respectively.

Mean DC for the various RBCs ranged from 46.03% to 69.86%, 45.94% to 69.38%, and 30.65% to 67.85% for 2 mm, 4 mm, and 6 mm, respectively. For all depths, SDR had the highest DC. For 2 mm and 4 mm depths, DC of BBR was the lowest. BF had the lowest DC values at 6 mm. At 4 mm, the decrease in DC ranged from 0.2% to 4.2% for bulk-fill RBCs and 6.1% to 23.2% for conventional materials. At 6 mm, the reduction in DC ranged from 2.9% to 12.8% and 35.6% to 53.6% for bulk-fill and conventional RBCs, respectively. While no significant difference in DC was observed between depths of 2 mm and 4 mm for the bulk-fill RBCs, DC at 2 mm was significantly greater than 6 mm. For the conventional RBCs, DC at 2 mm was significantly higher than at 4 mm and 6 mm. Significant differences in DC were also observed between 4 mm and 6 mm for the conventional materials.

Table 3: *DC at 4 and 6 mm Expressed as a Percentage of DC at 2 mm*

| Material | 2 mm | 4 mm | 6 mm |
|---|---|---|---|
| BBR | 100 | 99.8 | 89.6 |
| BT | 100 | 90.0 | 56.1 |
| BBF | 100 | 95.8 | 88.3 |
| BF | 100 | 76.8 | 46.4 |
| TNB | 100 | 98.6 | 87.2 |
| TN | 100 | 93.9 | 64.4 |
| SDR | 100 | 99.3 | 97.1 |

Table 4: *Mean Polymerization Shrinkage (Standard Deviation) for the Various RBCs*[a]

| Material | Volumetric Shrinkage (%) |
|---|---|
| BBR | 1.48 (0.01)$^{A}$ |
| BT | 2.21 (0.03)$^{B}$ |
| BBF | 3.02 (0.04)$^{C}$ |
| BF | 4.26 (0.02)$^{D}$ |
| TNB | 2.10 (0.01)$^{E}$ |
| TN | 2.11 (0.02)$^{E}$ |
| SDR | 3.38 (0.04)$^{F}$ |

[a] *Different uppercase letters in each row indicate significant differences within the same materials ($p<0.05$; Tukey HSD test).*

Differences in DC between RBCs were found to be product and depth dependent. At 2-mm depth, SDR had significantly higher DC than most other RBCs, with the exception of BF. BBR had significantly lower DC when compared with the other RBCs. The DC of giomer bulk-fill RBCs was significantly lower than that of their conventional giomer counterparts. No significant difference in DC was observed between TNB and TN. At 4 mm, SDR had significantly greater while BBR had significantly lower DC than all the other RBCs including BT. No significant difference in DC was observed between BBF and BF nor between TNB and TN. At 6-mm depth, significantly greater DC was observed for SDR when compared with the other materials. DC of the bulk-fill RBCs was higher than their conventional equivalents.

Mean PS ranged from 1.48% to 4.26% (Table 4; Figure 1). Ranking of PS from highest to lowest was as follows: BF > SDR > BBF > BT > TN = TNB > BBR. The PS of BF was significantly greater while that of BBR was significantly lower than all the other RBCs. The PS of the giomer bulk-fill RBCs was significantly lower than that of their conventional giomer counterparts. No significant difference in PS was observed between TNB and TN. A moderate,

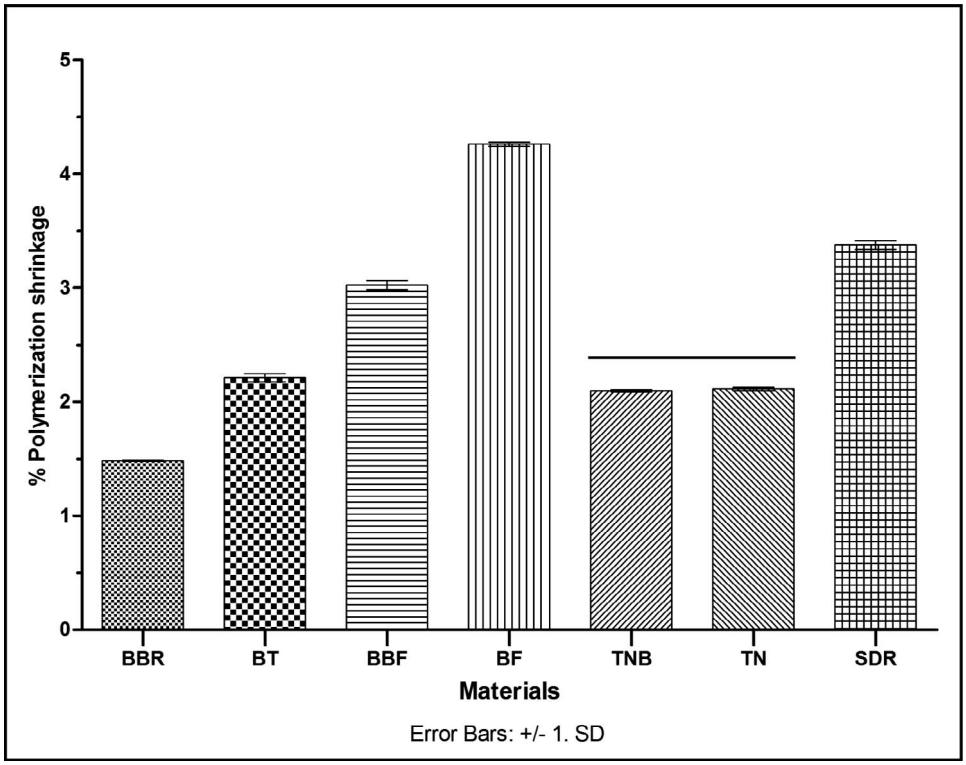


Figure 1. *Mean volumetric shrinkage (%) for the various RBCs. Horizontal line indicates homogeneous grouping. Results of Tukey HSD test at a significance level of 0.05.*

positive, and significant correlation was observed between DC and PS ($r$=0.72, $r^2$=0.52, $p$=0.0002) with Pearson correlation.

## DISCUSSION

We compared the DC and PS of contemporary bulk-fill RBCs including restorative and flowable giomer products. Based on the results of this study, the null hypotheses were rejected. FTIR and Raman spectroscopy were first used to study water sorption in dental resins and were subsequently employed to analyze DC in methacrylate polymerization.[27,28] Other techniques of measuring DC include electron paramagnetic resonance, nuclear magnetic resonance, differential scanning calorimetry, and differential thermal analysis.[29-32] FTIR, however, remains the most frequently used technique.[33] DC was determined by the proportion of remaining aliphatic C=C double bonds' concentration in the cured RBCs relative to the total number of C=C bonds in the uncured materials. DC was reported to be significantly influenced by light source and RBC materials.[34] Light source variables include type of light, power density, wave length, irradiation time, irradiation distance, and light-activation method, which were all standardized in our study. DC was found to be both product and depth dependent. The percentage decrease in DC with increased depth was RBC dependent, with bulk-fill materials exhibiting better conversion at increased depths when compared with their conventional counterparts (Table 3). Significant differences in DC were observed between 2 mm, 4 mm, and 6 mm depths for conventional materials but only between 2 mm and 6 mm depths for bulk-fill RBCs. Although results supported manufacturers' claims of bulk filling in 4 mm increments for the bulk-fill RBCs evaluated, the DC of bulk-fill giomer materials was relatively low (Table 2).

The DC of C=C double bond for RBCs generally ranges from 55% to 65%.[35] The DC of the bulk-fill restorative giomer BBR was less than 55%, even at the 2 mm depth, while the DC of the bulk-fill flowable giomer BBF was marginally greater than 55% at 2 mm. The DC of conventional giomer RBCs at 2 mm (62.8% for BT and 66.1% for BF) was significantly higher than that of BBF and BBR. The low DC of bulk-fill giomers (53.2% for BBF and 45.9% for BBR) at 4 mm in this study concurred with the findings of Al-Ahdal and coworkers.[20] They measured DC at a 4 mm depth after 20 seconds of light curing with an irradiance of 1200 mW/cm$^2$ and reported a DC of 56.3% for BBF and 38.9% for BBR. Ilie and Fleming[21] conducted a similar study with a higher irradiance of 1415 mW/cm$^2$ and also reported similar DC values of 57.7% and 40.0% for BBF and BBR, respectively. DC may not be optimized imme-

diately or 5 minutes after light curing, and some degree of postpolymerization conversion is anticipated for up to 24 hours. Even after 24 hours, the DCs of BBF and BBR were 65.7% and 49.7%, respectively. In comparison, the DC at 24 hours of other bulk-fill RBCs ranged from 54.5% to 71.9%.[20] As both BBF and BBR are similar in resin chemistry and filler types, differences in DC can be attributed to variation in filler quantity. BBF, which is flowable, has a lower filler content (72.5 wt%) than BBR (87.0 wt%). DC has been shown to decrease proportionally with increasing filler content and can be attributed to the light scattering at the resin-filler interfaces.[36] In addition to filler quantity, filler type, size, and shape can also influence the efficiency of light scattering.[37]

At all depths, SDR had the highest DC. At 4 mm and 6 mm, the decrease in DC was only 0.7% and 2.9% correspondingly. The high DC of SDR was in accordance with other works[38] and can be ascribed to its UDMA-based resin matrix and large filler particles. Sideridou and others[39] studied the effect of chemical structure on DC in light-cured dimethacrylate-based dental resins and found DC increased in the order Bis-GMA < Bis-EMA < UDMA < TEGDMA. UDMA has lower viscosity and higher molecular flexibility than BisGMA. It integrates an imino (-NH-) group for chain transfer reactions that offer another path for the continuation of polymerization. The large filler particles (mean 4.2 μm) used in SDR reduces filler-matrix interfaces. Incident light interference and scattering are reduced, increasing translucency, light penetration, and cure.[40] TNB contains a germanium-based photo-initiator (Ivocerin) that generates at least two free radicals for initiating polymerization initialization. Camphorquinone, the most widely used visible-light photo-initiator in RBCs, generates only one radical. The fore mentioned explains in part the significantly higher DC of TNB when compared with TN at 6 mm. According to the manufacturer, TNB is also more translucent (15%) than TN (10%), allowing for more light penetration and greater depth of cure.[15] The decrease in DC at 4 mm and 6 mm was only 1.4% and 12.8%, respectively, for TNB (Table 3).

PS of RBCs had been determined with a variety of techniques, including water/mercury dilatometry, cuspal deflection, specific gravity analysis, electrical strain gauges, and optical measurements.[41] The optical video-imaging technique was selected as it provided an easy method for measuring volumetric shrinkage. In addition, the AcuVol video-imaging technique had been shown to give reproducible results for volumetric shrinkage comparable with those measured by dilatometry.[42] Volumetric shrinkage for conventional methacrylate-based RBCs ranges from 2% to 6%.[43] Mean PS for the bulk-fill RBCs evaluated ranged from 1.5% to 3.4%, while that of conventional materials ranged from 2.1% to 4.3%. While no significant difference in PS was observed between TNB and TN, the bulk-fill giomers BBF and BBR had significantly lower shrinkage than their conventional counterparts BF and BT. Our findings corroborated those of studies using other shrinkage measurement techniques that reported generally lower shrinkage with bulk-fill materials.[17-19] PS of the flowable giomers (BBF and BF) were significantly greater than their restorative equivalents (BBR and BT). The difference in PS can be attributed to filler volume fraction and the DC of the RBCs.[44] Flowable giomers with their lower filler and higher resin content are anticipated to shrink more than their highly filled restorative counterparts. The correlation between DC and PS was moderate, positive, and significant. SDR, which had the highest DC, consequently had the highest shrinkage. The lower PS of bulk-fill giomers is also explained by their lower DC at 2 mm when compared with conventional equals. DC of the conventional giomers BT and BF was 62.8% and 66.1%, while that of the bulk-fill giomers BBR and BBF was only 46.0% and 55.5%. Further studies examining the effects of light sources including irradiation distance and light-activation method on DC and PS are warranted in view of inconsistencies during clinical light curing and the various light polymerization regimens/modes available.

## CONCLUSION

The DC and PS of contemporary bulk-fill RBCs including recently launched giomer materials were evaluated. Within the limitations of this study, the following conclusions can be drawn:

1. DC and PS of bulk-fill RBCs are product/depth dependent.
2. Bulk-fill RBCs should not be cured in more than 4-mm increments with the exception of SDR.
3. As DC of giomer bulk-fill RBCs at 4 mm depths was significantly lower than nongiomer bulk-fill materials, their clinical use at such depths should be exercised with caution.

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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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# Shear Bond Strength and Tooth-Composite Interaction With Self-Adhering Flowable Composites

C Brueckner • H Schneider • R Haak

### Clinical Relevance

The investigation of bonding performance and the assessment of tooth-composite interaction are helpful in the evaluation of restoration systems. Distinct differences between self-adhering flowables regarding the adhesive performance were observed, so that the clinical use must be pursued cautiously.


## SUMMARY

**Purpose: To evaluate the tooth-composite interaction (A) and shear bond strength (SBS; B) of self-adhering flowables.**

**Methods and Materials: (A) Thirty-two human molars with one Class V cavity were restored with Vertise Flow (VF), Fusio Liquid Dentin (FLD), an experimental self-adhering flowable (EF), or Adper Prompt-L-Pop/Filtek Supreme XT Flowable (PLP). Teeth were prepared according to laboratory standard and stored in water (24 hours, 37°C). Microleakage (ML; percentage interface length at enamel [E]/dentin [D]) and tooth-composite interaction were investigated. (B) The buccal surface of 160 embedded human molars was abraded to expose an enamel/dentin area of diameter ≥3 mm. Composite specimens were produced on enamel/dentin with VF, FLD, EF, or PLP. Prior to loading, 80 samples were water stored (24 hours, 37°C) and 80 thermocycled (5°C-55°C, 1500 cycles). The SBS was measured, and failure modes were classified by scanning electron microscopy.**

**Statistics: Kruskal-Wallis, Mann-Whitney U, and Fisher exact tests were performed ($\alpha$=0.05).**

**Results: (A) At enamel margins, EF and VF showed significantly lower ML than did FLD and PLP ($p_i \leq 0.009$; 81%-89%); in dentin, lower values resulted with FLD and VF compared with PLP and EF ($p_i \leq 0.01$; 77%-94%). Adhesive tags at E were consistently verifiable with EF and VF but irregularly with FLD and PLP. At D, tags were detectable with all systems. (B) In all groups, SBS decreased by up to 97% after thermocycling. It was generally diminished with self-adhering flowables (E: 50%-98%, D: 59%-98%; $p_i < 0.02$). More cohesive defects were observed with PLP ($p_i < 0.009$).**



†Christina Brueckner, University of Leipzig, Department of Cariology, Endodontology and Periodontology, Leipzig, Germany

†Hartmut Schneider, Dr. rer. nat., University of Leipzig, Department of Cariology, Endodontology and Periodontology, Leipzig, Germany

*†Rainer Haak, Prof. Dr. med. dent., MME, University of Leipzig, Department of Cariology, Endodontology and Periodontology, Leipzig, Germany

*Corresponding author: Liebigstraße 12, Leipzig 04103, Germany; e-mail: rainer.haak@medizin.uni-leipzig.de

†These authors contributed equally to this work.


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**Conclusion: Tooth-composite morphology and bond strength indicate that the clinical use of self-adhering flowables must be pursued cautiously.**

## INTRODUCTION

In the mid-1990s, flowable composites were developed wherein the low viscosity permits the composite to shape itself to fit cavity areas that are difficult to access.[1,2] These conventional flowables require a separate adhesive to bond to hard dental tissues. However, the interface between tooth and restoration remains the weak point of an adhesive filling.[3,4] One reason frequently discussed in this context is the technique sensitivity of current adhesive restoration approaches.[5] Simplification of clinical procedures is therefore one driving force in current material development and research.[6,7] While in the past, most attempts at optimization have been directed at reducing the time needed and the number of steps required for application, dentists are now increasingly calling for more user-friendly and less error-prone systems.

A particularly innovative route was to develop a product that combines the restorative material with the benefits of a bonding system.[6,7] Recently, flowable self-adhering composites have been introduced that appear to promise a combination of easy handling and simplified, time-saving procedures thanks to the absence of additional etching and bonding steps. The manufacturers claimed these composites were suitable for use as a filling material in small restorations and as a lining material with a bonding quality comparable to self-etching bonding systems.[8,9]

Since the commercial launch of self-adhering flowables, only a few studies investigating bonding performance have been published. None of these studies offer conclusive arguments about the clinical potential of this material group.[10-12] Whereas Poitevin and others[11] warned against routine clinical use, Bektas and others[13] judged Vertise Flow to be a useful material with acceptable bond strength and marginal seal. Stiffness was described as similar to packable composites,[14] whereas water sorption, hygroscopic expansion, and mass reduction were regarded as critical.[15,16] Furthermore, it remains unclear whether preliminary etching of the tooth substrate is beneficial.[11,12,17,18] Initiating bond strength evaluations found divergent values for self-adhering flowables compared with conventional ones.[19-21] Also, very few studies regarding the aging behavior of this material have been published.[22,23] Longevity in particular is a severe challenge for the tooth-composite interaction.[24] Against this background, we decided to examine this new material class in order to provide further information to help assess its adhesive potential.

This study aimed to assess the composite-tooth bond of an experimental self-adhering flowable composite in comparison to two established systems in terms of microleakage, tooth-composite interaction, and shear bond strength (SBS) to enamel and dentin both before and after thermocycling. As a control, a flowable composite in combination with a 1-step self-etch adhesive was used.

## METHODS AND MATERIALS

Noncarious human molars without cracks, stored in chloramine-t-trihydrate (0.5%, 4°C), were used in this study. Collection of the tooth specimens took place with informed consent and was approved by the ethics committee. The teeth were prepared according to ISO/TS 11405 within three months after extraction.[25] Three self-adhesive flowables (experimental flowable [EF], Vertise Flow [VF], Fusio Liquid Dentin [FLD]) and a flowable composite in combination with a self-etch adhesive (Filtek Supreme XT Flowable/Prompt-L-Pop [PLP]) were tested (Table 1).

The study was divided into evaluation of tooth-composite interaction (part A) and SBS to enamel and dentin (part B).

### Part A: Tooth-Composite Interaction

*Cavity Preparation and Restoration*—Thirty-two mixed, oval Class V cavities were prepared with a rounded cylindrical diamond bur (107 μm, 836KR.314.014, Komet/Gebr. Brasseler GmbH & Co. KG, Lemgo, Germany) at high speed with water cooling. Preparations had a standard size (approximately 1.5 mm deep, 3 mm wide in incisal-apical orientation, 4 mm wide in mesiodistal orientation) and were placed facially at the cemento-enamel junction with half of the cavity margin above (enamel) and half below this line (dentin). The enamel cavity margins were given a 0.5 mm bevel with a finishing diamond bur (46 μm, 8836KR.314.014, Komet/Gebr. Brasseler GmbH & Co. KG).

Immediately after preparation of each cavity, eight teeth were randomly assigned to one of four groups EF, VF, FLD, or PLP, and restorations were placed following the manufacturer's instructions (Table 1). Light curing of the composite

Table 1: *Used Materials*.

| Material (Group) | Type | Manufacturer | Composition[a] [Lot Number] | Bonding Procedure/Application |
|---|---|---|---|---|
| Experimental Flowable (EF) | Self-adhering flowable, experimental | DMG mbH, Hamburg, Germany | Unknown [F-142890] | Clean the tooth, remove all residuals with water spray, dry the tooth, apply product onto the cavity surface, massage a thin layer (~0.5 mm) into the entire surface for 20 s using the brush, light cure for 20 s, build up layers with a maximum of 2-mm thickness and light cure each for 20 s. |
| Vertise Flow (VF) | Self-adhering flowable | Kerr GmbH, Rastatt, Germany | GPDM, HEMA, prepolymerized filler, 1-μm barium glass filler, nano-sized colloidal silica, nano-sized ytterbium fluoride [3461596] | Wash the tooth thoroughly with water spray and air dry at maximum air pressure for 5 s, dispense product onto preparation with a dispensing tip, brush a thin layer (<0.5 mm) onto entire cavity surface with moderate pressure for 15-20 s, light cure for 20 s, build additional layers in increments of 2 mm or less, light cure each increment for 20 s. |
| Fusio Liquid Dentin (FLD) | Self-adhering flowable | Pentron Clinical Technologies LLC, Wallingford, CT, USA | UDMA, TEGDMA, HEMA, 4-MET, silane treated barium glass, amorphous silica, minor additives, photo curing system [201091] | Remove any potential debris with water and thoroughly clean the cavity, air dry briefly (2-3 s) to remove excess water and form a moist surface with no water pooling, dispense a 1-mm increment directly onto the tooth surface, gently rub the material into the preparation using the needle tip for 20 s, light cure initial layer for 10 s, add subsequent layers with a maximum thickness of 2 mm, and light cure each for 10 s; the final layer should be cured for additional 10 s. |
| Adper Prompt-L-Pop™/Filtek Supreme XT Flowable (PLP) | self-etch adhesive | 3M Espe AG, Seefeld, Germany | *first blister:* methacrylate phosphates, bis-GMA, phosphoric acid, photo initiators; *second blister:* water, HEMA, polyalkenoic acid polymer [405560] | Remove loose debris by spraying with water, use two to three brief blows of air to dry the cavity, mix PLP as stated in the manual, brush the adhesive onto the entire cavity surface, massage it in for 15 s applying pressure, use a gentle stream of air to thoroughly dry the adhesive to a thin film, rewet the brush tip with adhesive and apply a second coat, use a gentle stream of air again, light cure the adhesive for 10 s. |
| | flowable composite | 3M Espe AG, Seefeld, Germany | Bis-GMA, TEGDMA, functionalized dimethacrylate polymer, substituted dimethacrylate, silane treated ceramic, silane treated silica [N166300] | Dispense flowable, place maximum 2-mm-thick increments directly from the dispensing tip and light cure each for 20 s. |

*Abbreviations: 4-MET, 4-methacryloxyethyltrimetellitic acid; bis-GMA, bisphenol-glycidyl methacrylate; GPDM, glycerol phosphate dimethacrylate; HEMA, hydroxyethylmethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.*
[a] *As described by the corresponding manufacturer.*

layers was performed with a minimum intensity of 1000 mW/cm$^2$ at the minimum distance from the light source (HS LED 1200, Henry Schein Dental Deutschland GmbH, Langen, Germany). Restorations were finalized with a finishing diamond bur and silicone polishers (Politip F, 533602; Politip P, 533584; Ivoclar Vivadent AG, Schaan, Liechtenstein).

*Specimen Preparation: Evaluation of Microleakage*—The restored teeth were stored in double-distilled water (24 hours, 37°C) and immersion fixed in buffered glutaraldehyde (5%, 0.1 M sodium phosphate buffer, pH 7.2, 24 hours, 4°C). For microleakage assessment, the teeth were soaked three times in fresh buffer (0.1 M sodium phosphate buffer, pH 7.2, one hour each, 20°C). The apices of

the teeth were sealed with composite resin and the whole tooth surface was covered with two coats of nail polish, with the exception of a 1 mm window around the restoration. Microleakage was tested using a standardized tracer penetration method. The samples were immersed in basic ammoniacal silver nitrate ($AgNO_3/NH_4OH$, 3 M, pH 9.5, 24 hours, 37°C) in darkness and rinsed in distilled water (60 seconds, 20°C) before they were incubated in photo-developing solution (TETENAL Europe GmbH, Norderstedt, Germany) under fluorescent light (Konrad Benda Laborgeräte, Wiesloch, Germany, eight hours, 20°C). After rinsing in distilled water again (60 seconds, 20°C), the roots were cut 2 mm under the restoration margin and the crown was embedded in Stycast 1266 (Emerson & Cuming ICI Belgium N.V., Westerlo, Belgium). Restorations were then sectioned longitudinally under water cooling (n=3 each, 200 μm thickness, Leitz 1600 Sägemikrotom, Ernst Leitz Wetzlar GmbH, Wetzlar, Germany). Each section was examined stereomicroscopically with a digital microscope camera (20×, ProgRes CT3, JENOPTIK Laser, Optik, Systeme GmbH, Jena, Germany) with accessory operation and control software. Microleakage was measured separately for enamel and dentin in terms of length of $AgNO_3$ penetration and stated as the percentage of the interface length.

*Specimen Preparation: Tooth-Composite Interaction Features*—The sections were decalcified (2% HCl, 10 seconds), deproteinized (10% NaOCl, 30 seconds), rinsed with distilled water, dehydrated in ascending ethanol baths (30% to 100%), immersed in hexamethyldisilazane (Carl Roth GmbH & Co. KG, Karlsruhe, Germany), and air dried. All samples were mounted on specimen holders, gold sputtered (20 nm; Edwards Sputter Coater S150B, BOC Edwards Ltd., Crawley, UK) and examined by scanning electron microscopy (SEM; 30× to 2000×, 25 kV accelerating voltage, LEO 1430 vp, Carl Zeiss Microscopy GmbH, Oberkochen, Germany) in the backscattered and secondary electron mode.

The tooth-composite interaction was characterized by tag formation at enamel (yes/no) and dentin (intra, peritubular, and lateral resin penetration). For each of the three sections per tooth, the interfacial gap formation (adhesive failure) was scored for the interface between restoration and enamel as well as dentin (1 to 5):

- 1: no adhesive failure at enamel or dentin interface
- 2: <33% adhesive failures of the interface length
- 3: 33% to 50% adhesive failures of the interface length
- 4: >50% adhesive failures of the interface length
- 5: total adhesive failure (100%)

The mean score for each tooth was determined separately for enamel and dentin.

**Part B: SBS Measurements**

*Specimen Preparation: Evaluation of SBS*—One hundred sixty human molars were embedded in Stycast 1266 (Emerson & Cuming ICI Belgium N.V.) and randomly assigned to the groups EF, VF, FLD, or PLP (n=40). In each group, the buccal surfaces of the teeth were wet abraded with a 120-grit silicon-carbide paper until enamel or dentin (n=20) surfaces with a diameter greater than 3 mm were exposed (Struers DAV-P, Struers GmbH, Willich, Germany). The surfaces were polished for 60 seconds under water cooling with a 600-grit silicon carbide paper to standardize the smear layer. It had already been ascertained that the specimens had the right diameter of flat surface, and the dentin specimens were additionally assessed for the absence of enamel (25×, stereomicroscope SM 20, Carl Zeiss, Jena, Germany). In accordance with ISO, a split mold (polytetrafluoroethylene, 3 mm diameter, 3 mm high) was positioned on the exposed tooth surfaces with a strong clamp to define and limit the bonding area and to produce a standardized composite test specimen. Composite cylinders were bonded to enamel and dentin according the manufacturer's instructions (Table 1) and light cured as already mentioned. Specimens that failed immediately during the bonding procedure were documented as "pretesting failure" (PTF) and were considered statistically.

Test specimens were stored in distilled water (24 hours, 37°C). From each group, 20 samples ($n_{enamel}=10$, $n_{dentin}=10$) were thermocycled (TC, 5°C-55°C, 1500 cycles; Willytec Thermalcycler V 2.8, SD Mechatronik GmbH, Feldkirchen-Westerham, Germany). Specimens that debonded during this procedure were recorded as "testing failure" (TF) and were considered statistically.

Immediately after removal from water, the SBS was measured according to ISO. Each specimen was positioned in a Universal Testing Machine (Z 010, software testXpertII, version 2.2, Zwick GmbH & Co. KG, Ulm, Germany) and loaded at a cross-head speed of 0.75 ± 0.25 mm/min.

*Specimen Preparation: Evaluation of Failure Modes*—Debonded composite cylinders and enamel/

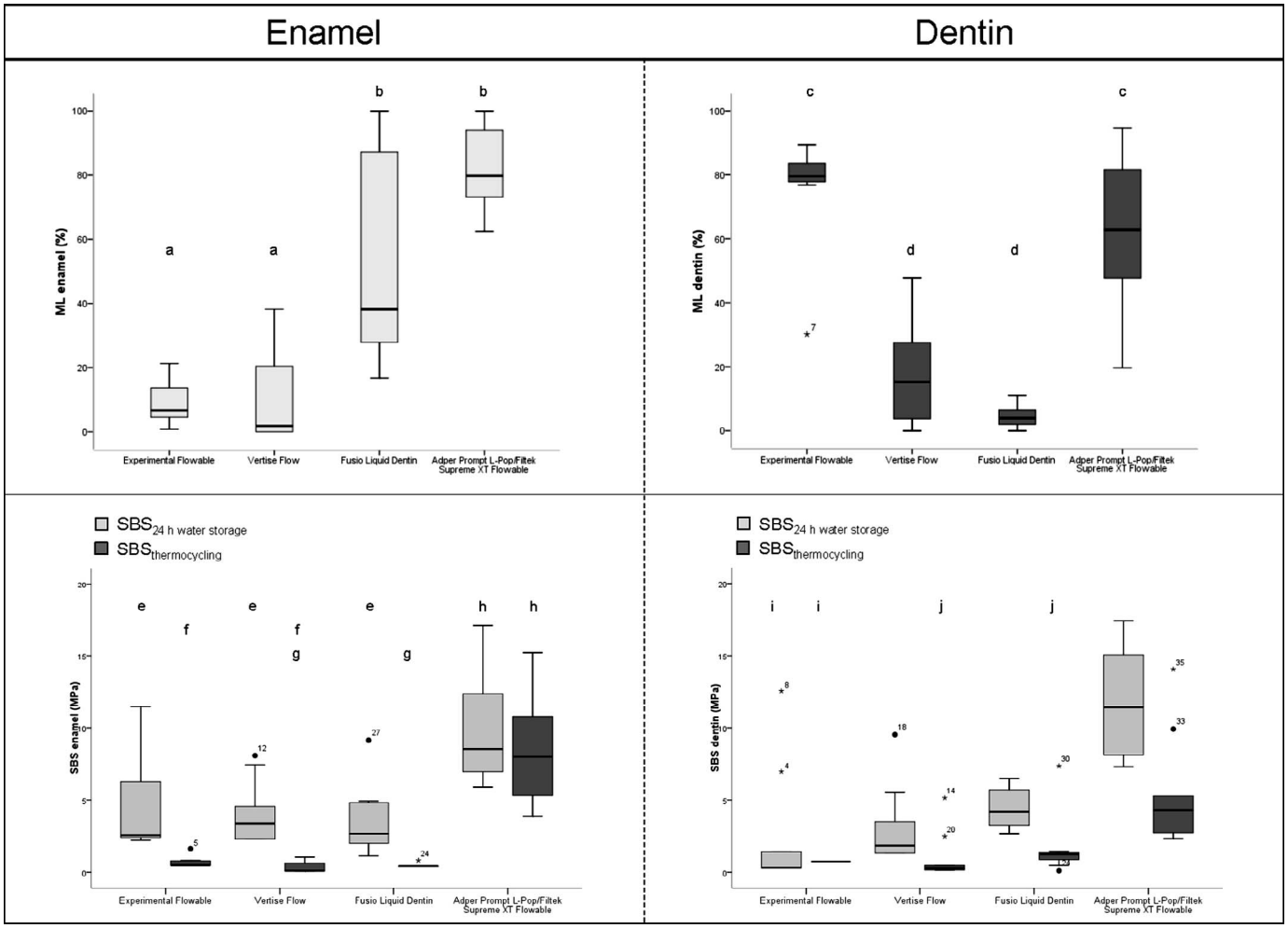


Figure 1. *Microleakage and shear bond strength on enamel and dentin. Groups with the same superscript letters are not significantly different ($p_i > 0.05$).*

dentin samples were mounted on specimen holders before being gold sputtered (20 nm, Edwards Sputter Coater S150B) and examined by SEM (30×, 20 kV, CamScan CS 24, Cambridge Scanning Corp Ltd, Cambridge, UK) in the backscattered and secondary electron mode. SEM images were digitized (EPSON Expression 1680 Pro, Seiko Epson Corp, Nagano, Japan) and stitched (Adobe Photoshop CS 4 Extended, Adobe Systems Inc, San José, CA, USA). Using the stitched SEM images, failure modes were scored by cohesive/adhesive failure percentages (1 to 6):

- 1: 100% cohesive failure at composite, adhesive, or enamel/dentin
- 2: 75% to <100% cohesive/1% to <25% adhesive
- 3: 50% to <75% cohesive/25% to <50% adhesive
- 4: 25% to <50% cohesive/50% to <75% adhesive
- 5: 1% to <25% cohesive/75% to <100% adhesive
- 6: 100% adhesive failure between composite and enamel/dentin

### Statistical Analysis

The data were subjected to statistical analysis using SPSS (PASW Statistics 18, SPSS Inc, Chicago, IL, USA). The significance level was set at $\alpha=0.05$.

*Part A*—Microleakage mean values ± standard deviations were determined; medians and percentiles were illustrated with boxplots (Figure 1). Differences between groups regarding microleakage formation and adhesive failures were analyzed using Kruskal-Wallis and 2-tailed Mann-Whitney U-test. Materials were ranked based on statistically significant differences with grades 1 to 4. Grade 1 corresponded to the most favorable of the four values (lowest mean value of microleakage) and grade 4 to the most unfavorable.

Table 2: *Microleakage/Adhesive Defects (Score) at Enamel and Dentin Interface*[a]

| | Enamel | | | | Dentin | | | |
|---|---|---|---|---|---|---|---|---|
| | **EF** | **VF** | **FLD** | **PLP** | **EF** | **VF** | **FLD** | **PLP** |
| Microleakage, % | 9.0 (6.7)$_a$ | 10.3 (16.1)$_a$ | 52.9 (33.8)$_b$ | 82.1 (13.4)$_b$ | 75.2 (18.6)$_c$ | 17.6 (16.3)$_d$ | 4.4 (3.6)$_d$ | 62.3 (24.5)$_c$ |
| Score | 2.0$_e$ | 2.2$_e$ | 4.5$_f$ | 3.9$_f$ | 4.5$_{g,h}$ | 3.6$_{g,i,j}$ | 3.3$_i$ | 4.3$_{h,j}$ |

[a] *Mean (standard deviation). Means with same subscript letters are not significantly different ($p>0.05$).*

*Part B*—SBS mean values ± standard deviations were determined; medians and percentiles were illustrated with boxplots (Figure 1).

PTFs and TFs were replaced by a singular imputation with the lowest measured SBS value in each group, differences between groups, and differences within a group (without vs with thermocycling), and differences regarding fracture mode were analyzed using Kruskal-Wallis and 2-tailed Mann-Whitney U-test. Differences in the appearance of PTFs and TFs were analyzed using a 2-tailed Fisher exact test. Materials were ranked based on statistically significant differences with grades 1 to 4. Grade 1 corresponded to the most favorable of the four values (highest mean value of SBS) and grade 4 to the most unfavorable.

## RESULTS

### Part A

Microleakage was always observed in different occurrences (Table 2; Figure 1). At enamel interfaces, EF and VF showed significantly lower microleakage than did FLD and PLP (81%-89%) and were both ranked 1.5 compared with 3.5 for FLD and PLP (Table 3). Within dentin cavity segments, FLD and VF showed significantly lower microleakage than PLP and EF (77%-94%), which is consistent with the material ranking of 1.5 compared with PLP and EF, with rank 3.5.

The tooth-composite interaction was generally based on adhesive tag formation at enamel. The dentin tooth-composite interaction showed product-specific characteristics. Typical SEM images are shown in Figures 2 and 3. At enamel, the experimental flowable always showed distinct tags, whereas these arose frequently and lightly with VF and sparsely in the case of FLD and PLP. At dentin, in the PLP group, tags appeared as slightly peritubular anchored and regularly lateral branched. With EF, tags were thinner and partially branched; with FLD and VF, they were thin, sparse, and without branching.

Interfacial adhesive failures occurred with all systems at enamel and dentin (Table 2). At enamel, in the self-adhering flowables group, EF and VF showed significantly lower score values than FLD. At dentin, FLD exhibited the lowest score in the group, with a significant difference from EF.

### Part B

The SBSs are illustrated in boxplots in Figure 1. Tables 4 and 5 summarize the mean values and standard deviations, number of PTFs and TFs, and scores of failure modes after water storage and after thermocycling.

After 24 hours of water storage, higher values of SBS were found in the control group ($p_i<0.004$) irrespective of the tooth substrate. The experimental flowable did not have a significantly higher SBS to enamel ($p_i>0.237$) than the other two self-adhering flowables. For the self-adhering flowables, the highest bond strength to dentin was seen with FLD ($p_i<0.0029$) and the lowest value with EF ($p_i<0.043$). In the materials ranking, first rank was given to the control, followed in descending order by FLD, VF, and EF (Table 3).

Table 3: *Ranking of Materials by Specific Parameters*

| | Enamel | | | | Dentin | | | |
|---|---|---|---|---|---|---|---|---|
| | **EF** | **VF** | **FLD** | **PLP** | **EF** | **VF** | **FLD** | **PLP** |
| Microleakage | 1.5 | 1.5 | 3.5 | 3.5 | 3.5 | 1.5 | 1.5 | 3.5 |
| Adhesive defects | 1.5 | 1.5 | 3.5 | 3.5 | 3.5 | 1.5 | 1.5 | 3.5 |
| $SBS_{24h\ water}$ | 3 | 3 | 3 | 1 | 4 | 3 | 2 | 1 |
| $SBS_{thermocycling}$ | 2 | 3.5 | 3.5 | 1 | 4 | 2.5 | 2.5 | 1 |
| Tooth-composite interaction | Distinct tags | Frequent smooth tags | Sparse tags | Sparse tags | Thin, partly branched tags | Sparse tags | Sparse tags | Peritubular anchored, branched tags |

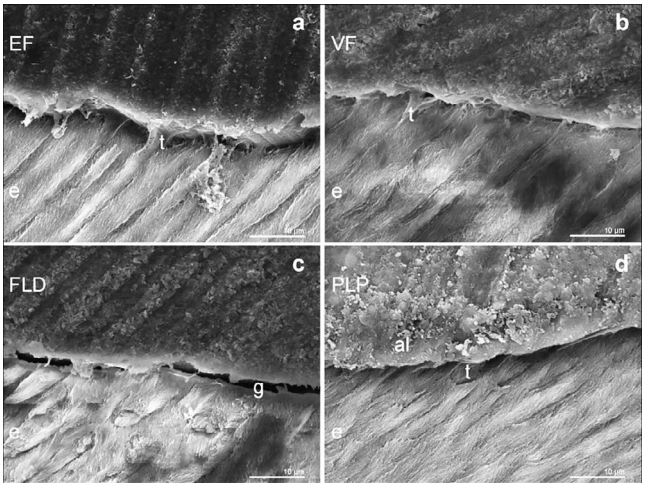


Figure 2. *Typical scanning electron microscope images of enamel-resin interface. (a): Distinct enamel tags with EF. (b): Light tags with VF. (c): A large gap can be observed between FLD and enamel. Tags were not verifiable in this image. (d): A thin adhesive layer between resin and enamel; sparse enamel tags were observed with PLP. e, enamel, t, tag; g, gap; al, adhesive layer.*

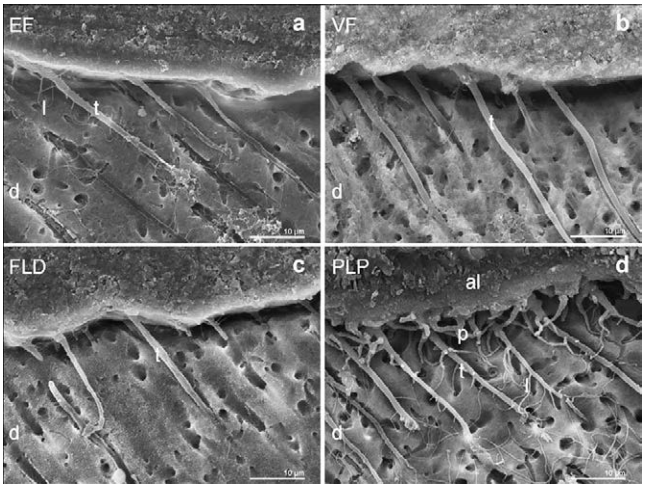


Figure 3. *Typical scanning electron microscopy images of dentin-resin interface. (a): Thin and partial branched tags produced by EP. (b, c): Tags are thin, sparse, and not laterally branched with VF/FLD. (d): Peritubular anchored and regularly lateral branched dentin tags produced by PLP are shown. d, dentin; t, tag; l, lateral branches; p, peritubular resin penetration; al, adhesive layer.*

After thermocycling, generally higher values of SBS were found in the control group ($p_i<0.001$). On enamel within the group of self-adhering flowables, EF showed higher SBS than FLD ($p_i<0.005$). At the dentin surfaces, only one specimen survived aging with EF, which is why statistical analysis was not performed for EF. FLD showed significantly higher SBS than VF. Irrespective of the tooth substrate, SBS values decreased after TC in all groups. Within the self-adhering flowables group, the reduction of SBS was up to 90%; with PLP, it was up to 53% after thermomechanical loading. After TC, the ranking of the systems was changed only marginally (Table 3).

The quantity of PTFs differed between the materials. While FLD showed no pretesting failure at all, this phenomenon occurred in all other groups. The flowables EF and VF presented more failed specimens in comparison with FLD and PLP ($p_i<0.02$).

No TF occurred within the control group, while failures were observed in the groups of flowables with varying frequencies of 3/10 to 8/10 (E) and 0/10 to 8/10 (D). The experimental flowable showed the highest rates of total failures (15 of 20) on enamel and dentin in comparison with all others ($p_i<0.033$).

Failure mode analysis revealed significantly more cohesive defects in the control group compared with the self-adhering flowables ($p_i<0.009$). Among the self-adhering flowables with VF on enamel, significantly more cohesive defects were observed compared with FLD ($p=0.033$). No other significant differences were found between the self-adhering flowables ($p_i>0.057$); for FLD without TC, the modes were all adhesive.

## DISCUSSION

Currently, only a few self-adhering flowable materials are commercially available, and in consequence, only a very few investigations regarding the bonding performance of the materials have been carried out. In this study, we tested the leading products in the market and one experimental material of the same class.

The literature might lead to the expectation that these simplified materials with no additional adhe-

Table 4: *Shear Bond Strength to Enamel and Dentin, After 24 Hours of Water Storage*[a]

| | Enamel | | | | Dentin | | | |
|---|---|---|---|---|---|---|---|---|
| | EF | VF | FLD | PLP | EF | VF | FLD | PLP |
| PTF, n | 2 | 2 | 0 | 0 | 5 | 3 | 0 | 1 |
| SBS, MPa | 4.4 (3.0)$_a$ | 4.0 (2.1)$_a$ | 3.5 (2.3)$_a$ | 9.8 (3.6) | 2.4 (4.1) | 3.0 (2.6) | 4.4 (1.3) | 11.6 (3.5) |
| Score | 5.8$_{b,c}$ | 5.5$_b$ | 6.0$_c$ | 3.8 | 6.0$_d$ | 5.6$_d$ | 6.0$_d$ | 3.4 |

[a] *Mean (standard deviation). Means with same subscript letters are not significantly different (p>0.05).*

Table 5: *Shear Bond Strength to Enamel and Dentin, After Thermocycling*[a]

| | Enamel | | | | Dentin | | | |
|---|---|---|---|---|---|---|---|---|
| | EF | VF | FLD | PLP | EF | VF | FLD | PLP |
| PTF/TF, n | 2/4 | 1/3 | 0/8 | 0/0 | 5/4 | 3/0 | 0/0 | 0/0 |
| SBS, MPa | 0.7 $(0.4)_a$ | 0.4 $(0.4)_{a,b}$ | 0.5 $(0.1)_b$ | 8.3 (3.7) | 0.7 (0.0) | 1.0 $(1.6)_c$ | 1.6 $(2.1)_c$ | 5.4 (3.7) |
| Score | $5.9_d$ | $5.4_d$ | $5.6_d$ | 3.7 | $5.9_e$ | $5.7_e$ | $5.8_e$ | 2.9 |

[a] *Mean (standard deviation). Means with same subscript letters are not significantly different ($p>0.05$).*

sive system would bond less efficiently with tooth substrate when compared with multistep etch-and-rinse or self-etch reference materials.[11,24] Nonetheless, hopes were pinned on the new self-adhering composites, with the expectation that their bonding effectiveness might be similar to the already simplified one-step self-etch adhesives combined with flowable composites. For this reason, we chose a restoration system consisting of a one-step self-etch adhesive with a flowable composite as control (Adper Prompt L-Pop, Filtek Supreme XT Flowable). Materials from the same manufacturer were combined to reduce the risk of side effects caused by unexamined interaction between substances.[5] An important element in assessing a restoration system's performance is evaluating the testing system itself by comparing it with a long-standing adhesive system as a control under the same laboratory conditions.[6,26,27]

The investigation of the tooth-composite bond and bond failures allows a complex assessment of restoration systems. Therefore, the evaluation of microleakage and interfacial bond failures in three-dimensional cavities was combined with the two-dimensional evaluation of SBS. With regard to silver nitrate penetration, the comparison between groups revealed significant differences. Regarding the ranking of the systems, VF achieved top positions (rank 1.5) for both tooth structures. The other tested self-adhering flowables achieved either a high rank in enamel and a low one in dentin (EF 1.5/3.5) or vice versa (FLD 3.5/1.5). These results are in contrast with others,[18] who found no significant differences between the microleakage of VF and FLD in enamel and a lower microleakage in dentin with VF compared with FLD. A possible reason for the deviation of the present results from previous ones is the study design; Celik and others[18] used thermocycling for artificial aging before microleakage measurement, which may enhance the effect of this parameter and influence the materials' ranking.

Regarding the relationship between microleakage and tooth-composite interaction at enamel, it is notable that the lowest amounts for microleakage and adhesive failure (EF and VF) corresponded with the most intensive interlocking of resin with enamel by tag formation and vice versa; high values of microleakage (FLD and PLP) were associated with poor micromechanical interlocking. For dentin, this correlation was not seen, as EF and PLP with an acceptable adhesive interlocking (tag formation, peritubular anchoring, lateral branches) showed the highest rates of microleakage. In contrast, low levels of microleakage generally corresponded with fewer adhesive failures both at enamel and dentin.

Thermocycling as a standard artificial aging method was used prior to the SBS measurements.[28] The thermocycling regimen of ISO TS 11405[25] was modified because there is a reference that the recommended number of 500 cycles is too low to achieve a realistic aging effect. Gale and Darvell stated that 10,000 cycles are equivalent to clinical use of one year.[29] Thus, the 1500 cycles applied in the present study may not simulate long-term clinical use, but they are sufficient to discriminate between materials that cannot withstand a wet environment on one hand and those that can on the other. In the current study, significant differences in SBS between the materials were found before and after artificial aging. For both tooth substrates, the self-adhering flowables showed significantly lower bond strength before and after thermal loading than the control. Furthermore, on dentin, FLD was more effective in bonding than the other two self-adhering materials. This was confirmed in a similar microtensile bond strength evaluation.[11] TC decreased SBS significantly in all cases, which is also in line with the findings of other working groups.[22,23]

Differences between the groups may be the result of a number of factors, but the largest influence is probably associated with the composition (Table 1), the rheological potential, and the types of the functional monomer.[30,31] FLD contains 4-methacryloxyethyltrimetellitic acid (4-MET) as a functional monomer, whereas VF contains glycerol phosphate dimethacrylate (GPDM). No information about the

chemical composition of the EF is given by the manufacturers.

4-MET is a well-known and frequently used acidic functional monomer with good adhesive durability on dentin.[32] It partially demineralizes dentin, leaving hydroxyapatite partially attached to collagen within a submicron hybrid layer. The residual hydroxyapatite interacts chemically with carboxyl groups of 4-MET.[31] At enamel, FLD showed higher microleakage, inferior micromechanical interlocking, but equivalent SBS compared with the other self-adhering materials. At the dentin surfaces, regarding microleakage and SBS, FLD performed better than the other flowables, although the micromechanical interlocking was poor. The fracture analysis also showed high rates of adhesive failures, which also confirms that FLD has weak micromechanical anchoring. On the basis of these observations, it can be speculated that FLD might be less acidic, resulting in a weaker enamel bonding and that the chemical bond between 4-MET and dentin is stronger compared with the other functional monomers.

Data regarding the bonding effectiveness and chemical analyses of the interfacial hybrid zone of the functional monomer in VF, GPDM, have been rare until now. The manufacturer points out that, on one hand, the phosphate functional group of the GPDM monomer will create a chemical bond with the calcium ions of the tooth. On the other hand, GPDM holds two methacrylate functional groups for copolymerization with other methacrylate monomers.[33] GPDM is also used by the manufacturer in the well-known etch-and-rinse adhesive OptiBond FL, which has performed excellently in numerous clinical and laboratory studies.[34,35] However, in a two-dimensional geometry (SBS measurements), VF did not perform as well as FLD. Looking at the microleakage values and adhesive defect formation in SEM images, the material achieved best sealing rates, independent of tooth structures. This could be the result of a chemical bond caused by the acidic phosphate group in the GPDM monomer.[11] Otherwise, looking at failure mode characterization, VF showed more cohesive defects than the other self-adhering composites regardless of the tooth substrate, which is also an indicator of a stronger micromechanical or chemical anchoring. The experimental system was able to seal the enamel sufficiently, supported both by microleakage values and tooth-composite interaction. Compared with the other self-adhering materials, EF showed a weaker sealing and bond at dentin. This may be an indication of a more acidic composition—with good bonding on enamel and less efficient performance on dentin. Whether the differences in efficiency are related to a potentially acidic mode of function remains speculation, because no information about the chemical composition is given by the manufacturers.

As a result, self-adhering composites should provide a strong chemical bond to the tooth on account of filler content and viscosity. One potential explanation of the lower SBS values with the self-adhering materials could be that the higher viscosity of the flowable resin composites compared with a separate adhesive system hinders deep penetration into the dentin tubules and between collagen fibers, which would improve sealing of the tooth structures and SBS values.

In accordance with several working groups, distinct differences in the bonding effectiveness of the materials were also found with the occurrence of PTFs.[11,36] The correct handling of this phenomenon is often discussed.[6,37,38] Excluding all PTFs from statistical analysis overestimates the mean bond strength and should be avoided because a high proportion of PTFs is typically associated with low SBS, measured for those specimens that resist debonding prior to testing.[39] Alternatively, researchers may assign an SBS value of 0 MPa or a value equivalent to the lowest measured for each PTF. Using 0 MPa for calculation in the event of PTF occurrence penalizes the tested materials severely, as there was low bond strength above 0 MPa. In case of a high incidence of PTFs, methods with data imputation will inevitably result in data distribution that is skewed. Because of this debate, we decided to assign the lowest measured SBS value within the respective group to a PTF.

In part, group differences regarding the evaluation of microleakage, adhesive defects, and degree of tooth-composite interaction and corresponding material ranking are in conflict with the ranking of SBS measurements. In contrast to the evaluation of tooth-composite interaction features in three-dimensional Class V restorations with flat specimens, a pure material science parameter is determined. C-factor, other physical outcomes resulting from three-dimensional geometry, and chemical effects remain unconsidered.

In general, the question can be raised as to whether short-term experimental studies can forecast clinical outcomes. Whereas some authors have questioned the value of experimental studies predicting clinical reliability in general,[40] the outcomes of prior laboratory investigations are often highlighted as indispensable and beneficial for characterizing adhesive

systems prior to product launch.[27,41-43] In addition, De Munck and others[4] and Peumans and others[35] have retrospectively concluded that the bonding effectiveness found *in vitro* correlates to a certain extent with their *in vivo* findings. In our opinion, clinical studies with self-adhering composites as the definitive filling material should be initiated only when more promising laboratory results are available and when these results are comparable with established reference systems.

## CONCLUSION

The study indicates that the self-adhering flowables exhibit significantly lower SBS to dental tissue than the self-etching control PLP. An individual evaluation of every newly launched self-adhering restorative appears necessary as these materials differ considerably in bonding failure mode and tooth-composite interaction. If microleakage and SBS values of clinically successful restoration materials are used as a benchmark, the self-adhering flowables tested in this study are currently not suitable as a permanent filling material *in vivo*.

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#### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Ethics Committee of the University of Leipzig, Germany. The approval code for this study is 299-10-04102010.

#### 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 Thermal Cycling on Flexural Properties and Simulated Wear of Computer-aided Design/ Computer-aided Manufacturing Resin Composites

A Tsujimoto • WW Barkmeier • T Takamizawa • MA Latta • M Miyazaki



### Clinical Relevance

Computer-aided design/computer-aided manufacturing resin composites have different physical properties, and care should be taken when selecting one for clinical use.


## SUMMARY

**Objective: The purpose of this study was to evaluate the influence of thermal cycling on the flexural properties and simulated wear of computer-aided design/computer-aided manufacturing (CAD/CAM) resin composites.**

**Methods: The six CAD/CAM resin composites used in this study were 1) Lava Ultimate CAD/CAM Restorative (LU); 2) Paradigm MZ100 (PM); 3) CERASMART (CS); 4) Shofu Block HC (SB); 5) KATANA AVENCIA Block (KA); and 6) VITA ENAMIC (VE). Specimens were divided randomly into two groups, one of which was stored in distilled water for 24 hours, and the other of which was subjected to 10,000 thermal cycles. For each material, 15 specimens from each group were used to determine the flexural strength and modulus according to ISO 6872, and 20 specimens from each group were used to examine wear using a localized wear simulation model. The test materials were subjected to a wear challenge of 400,000 cycles in a Leinfelder-Suzuki device (Alabama machine). The materials were placed in custom-cylinder stainless steel fixtures, and simulated localized wear was generated using a stainless steel ball bearing ($r$=2.387 mm) antagonist in a water slurry of polymethyl methacrylate beads. Simulated wear was determined using a noncon-**



*Akimasa Tsujimoto, DDS, PhD, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan; General Dentistry, Creighton University School of Dentistry, Omaha, NE, USA

Wayne W Barkmeier, DDS, MS, General Dentistry, Creighton University School of Dentistry, Omaha, NE, USA

Toshiki Takamizawa, DDS, PhD, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

Mark A Latta, DMD, MS, General Dentistry, Creighton University School of Dentistry, Omaha, NE, USA

Masashi Miyazaki, DDS, PhD, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

*Corresponding author: 1-8-13, Kanda-Surugadai, Chiyoda-Ku, Tokyo, 101-8310 Japan; e-mail: tsujimoto.akimasa@nihon-u.ac.jp


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**tact profilometer (Proscan 2100) with Proscan and AnSur 3D software.**

**Results: The two-way analysis of variance of flexural properties and simulated wear of CAD/CAM resin composites revealed that material type and thermal cycling had a significant influence ($p<0.05$), but there was no significant interaction ($p>0.05$) between the two factors. The flexural properties and maximum depth of wear facets of CAD/CAM resin composite were different ($p<0.05$) depending on the material, and their values were influenced ($p>0.05$) by thermal cycling, except in the case of VE. The volume losses in wear facets on LU, PM, and SB after 10,000 thermal cycles were significantly higher ($p<0.05$) than those after 24 hours of water storage, unlike CS, KA, and VE.**

**Conclusion: The results of this study indicate that the flexural properties and simulated wear of CAD/CAM resin composites are different depending on the material. In addition, the flexural properties and simulated wear of CAD/CAM resin composites are influenced by thermal cycling.**

## INTRODUCTION

Computer-aided design and computer-aided manufacturing (CAD/CAM) systems have developed in the field of dentistry over last two decades.[1] CAD/CAM materials available for restorations include glass ceramics, resin composites, aluminum-oxide, zirconia, and titanium.[2] Clinicians' interest in fabricating restorations using CAD/CAM systems continues to grow worldwide, and the demand for nonmetallic restorations from both clinicians and patients has encouraged researchers to seek alternative materials.[3]

Current resin composite technology has made considerable progress with the development of nanoparticle fillers.[4] It has been reported that nano-filled resin composites exhibit good mechanical properties,[5,6] improved surface characteristics and esthetics,[7] better gloss retention,[8] reduced polymerization shrinkage,[9] and reduced wear.[10] In conjunction with the improvement of resin composite technology, CAD/CAM resin composites that include nanoparticle fillers also have been introduced for clinical use.[11] CAD/CAM resin composites are fabricated by high pressure and temperature polymerization, resulting in improved physical properties that might make them more suitable as materials for applications from inlays to single crown restorations.[12] In addition, restorations produced from CAD/CAM resin composites can be more easily fabricated and repaired than restorations made from CAD/CAM ceramics.[13] However, there is a limited amount of independent research on CAD-CAM resin composites, creating a need for an evaluation of their physical properties. The measurement of parameters such as flexural properties and simulated wear will provide novel insight into the dynamic behavior of CAD/CAM resin composites under simulated occlusal stresses.

In clinical situations, occlusal stress is transmitted to resin composite restorations through the rigid and brittle fillers into the more flexible and ductile resin matrix during both function and parafunction.[14] Stress concentrations at the filler-resin matrix interface may result in filler dislodgement and exposure of the resin matrix leading to wear.[15] Such stress concentrations may also be generated by cyclic temperature changes.[16] During such temperature changes, differences in thermal expansion coefficients between fillers and resin matrices in resin composite restorations may lead to high interfacial stresses.[17] A thermal cycling test is the process of subjecting specimens to temperature changes that simulate intraoral conditions.[18] A previous study[19] established that 10,000 thermal cycles (TCs) correspond to one year of clinical function of restorations; this estimate is based on the hypothesis that such cycles might occur 20 to 50 times a day. Evaluation of the interaction effects between occlusal and thermal stresses on material properties is important because the stability of materials is related to the long-term clinical success of restorations. Thus, evaluation of the physical properties of CAD/CAM resin composites after thermal cycling may give valuable information about restoration longevity. However, there are few studies regarding the influence of thermal cycling on the flexural properties and simulated wear of CAD/CAM resin composites.

The purpose of this study was to investigate the influence of thermal cycling on the flexural properties and simulated wear of CAD/CAM resin composites. The null hypotheses to be tested were the following: 1) there is no significant difference in flexural properties and simulated wear of CAD/CAM resin composites; and 2) flexural properties and simulated wear of CAD/CAM resin composites are not influenced by thermal cycling.

## METHODS AND MATERIALS

The six CAD/CAM resin composites in this study were 1) Lava Ultimate CAD/CAM Restorative (LU;

Table 1: *CAD/CAM Resin Composites Used in This Study*

| CAD/CAM Resin Composite (Code, Shade) | Resin Matrix Composition | Inorganic Filler (Content) | Manufacturer (Lot No.) |
|---|---|---|---|
| Lava Ultimate CAD/CAM Restorative (LU, A2-LT) | Bis-GMA, UDMA, Bis-EMA, TEGDMA | $SiO_2$, $ZrO_2$, aggregated $ZrO_2$/$SiO_2$ cluster (80.0 wt%) | 3M ESPE, St Paul, MN, USA (N400900) |
| Paradigm MZ100 Block (PM, A2) | Bis-GMA, TEGDMA | zirconia-silica ceramic (85.0 wt%) | 3M ESPE, St Paul, MN, USA (N723583) |
| CERASMART (CS, A2-LT) | Bis-MEPP, UDMA, dimethacrylate | silica, barium glass (71.0 wt%) | GC, Tokyo, Japan (1407281) |
| Shofu Block HC (SB, A2-LT) | UDMA, TEGDMA | silica powder, microfused silica, zirconia silicate (61.0 wt%) | SHOFU, Kyoto, Japan (061401) |
| KATANA AVENCIA Block (KA, A2-LT) | UDMA, TEGDMA | aluminum filler, silica filler (62.0 wt%) | Kuraray Noritake Dental, Tokyo, Japan (000011) |
| VITA ENAMIC (VE, M2-T) | UDMA, TEGDMA | feldspar ceramic enriched with aluminum oxide (86.0 wt%) | Vita Zahnfabrik, Bad Säckingen, Germany (39440) |

*Abbreviations: Bis-EMA, ethoxylated bisphenol A-glycol dimethacrylate; Bis-GMA,bisphenol A-glycidyl methacrylate; Bis-MEPP, 2,2-Bis(4-methacryloxypolyethoxyphenyl)propane; CAD/CAM, computer-aided design/computer-aided manufacturing; $SiO_2$, silicon dioxide; TEGDMA, triethylene glycol dimethacrylate; UDMA urethane dimethacrylate; $ZrO_2$, zirconium dioxide.*

3M ESPE, St Paul, MN, USA); 2) Paradigm MZ100 block (PM; 3M ESPE); 3) CERASMART (CS; GC, Tokyo, Japan); 4) SHOFU Block HC (SB; SHOFU, Kyoto, Japan); 5) KATANA AVENCIA Block (KA; Kuraray Noritake Dental, Tokyo, Japan); and 6) VITA ENAMIC (VE; Vita Zahnfabrik, Bad Säckingen, Germany). The CAD/CAM resin composites are listed in Table 1 with the associated lot numbers and components.

### Flexural Strength and Elastic Modulus Measurement

Flexural properties were determined using a three-point bending test according to ISO 6872.[20] The bar-shaped specimens, 4.0 mm wide, 14.0 mm long, and 1.2 mm thick, were prepared using a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). All six sides of the specimen were wet ground with 1200-grit silicon carbide (SiC) paper to achieve the required dimensions of $4.0 \pm 0.2 \times 14.0 \pm 0.2 \times 1.2 \pm 0.2$ mm. The specimens were prepared under ambient conditions of 23° ± 2°C and 50% ± 10% relative humidity. Thirty specimens of each CAD/CAM resin composite were randomly divided into two groups (n=15 per group). Specimens from the first group were transferred to distilled water and stored at 37°C for 24 hours (24 h water storage). The other group was allocated 10,000 TCs between 5°C and 60°C (10,000 TCs). Thermal cycling was conducted using a custom-built machine. Each cycle consisted of water bath incubation lasting 30 seconds, with a transfer time of five seconds.

After the designated storage time, the specimens for the test group were subjected to the three-point bending test (span distance: 14 mm) using a universal testing machine (5500R, Instron Worldwide Headquaters, Norwood, MA, USA)at a cross-head speed of 1.0 mm/min until the specimen fractured. The peak breaking stress and the elastic modulus were determined from the stress-strain curve using a computer with custom software (Bluehill 2 Ver. 2.5, Instron Worldwide Headquaters) linked directly to the testing machine).

### Wear Simulation

Twenty specimens of each of the CAD/CAM resin composites after 24 h water storage and after 10,000 TCs were prepared for simulated localized wear (occlusal contact area [OCA] wear). The surfaces of the CAD/CAM resin composites were polished flat to 4000-grit using a sequence of SiC papers (Struers, Cleveland, OH, USA).

A Leinfelder-Suzuki device (Alabama machine) was used for this study. The simulator has a plastic water bath, and the custom-wear fixtures were mounted inside the four-station bath. A brass cylinder was then placed around each fixture in the bath to serve as a reservoir for the abrasive media (water slurry of unplasticized polymethyl methacrylate [PMMA] with an average particle size of 44 μm). The media was placed inside the brass cylinders to cover the surface of the resin composite in the custom fixtures. The water slurry of PMMA inside the brass cylinders was approximately 6 mm in height over the surface of the resin composite.

The antagonist for the localized (OCA) wear simulation was a stainless steel ball bearing ($r$ = 2.387 mm). The antagonist tips were mounted on spring-loaded pistons to deliver the wear challenges. During the application of the load, the antagonists

rotated approximately 30° as the maximum force was reached (maximum load of 78.5 N at a rate of 2 Hz), and then they counterrotated to the original starting position as the load was relaxed to complete the cycle. Each set of specimens was exposed to 400,000 cycles in the wear simulator.

### Wear Measurements

Prior to wear simulation, each resin composite specimen was profiled using a Proscan 2100 noncontact optical profilometer (Scantron Industrial Products Ltd, Taunton, England) with a $10 \times 10$ $\mu m^2$ resolution. These profiles provided the pretest digitized contours (20 specimens for each of the six CAD/CAM resin composites after 24 h water storage and 10,000 TCs for localized wear testing).

Following the 400,000 wear cycles, the specimens were ultrasonically cleaned (L&R Solid State Ultrasonic Cleaner T-14B, L&R Manufacturing Company, Kearny, NJ, USA) in distilled water for three minutes and then profiled again using the Proscan 2100 unit. The x-, y-, and z-coordinates of the before and after scans from the Proscan software were exported to another computer for analysis with AnSur 3D software (Minnesota Dental Research Center for Biomaterials and Biomechanics, University of Minnesota, Minneapolis, MN, USA).

Wear measurements were determined from the differences between the before and after data sets (before and after surface contours). A computerized fit was accomplished in AnSur 3E with the before and after data sets. Maximum depth (μm) and volume loss ($mm^3$) of the wear facets on the CAD/CAM resin composites were generated and recorded for each localized wear specimen.

### Statistical Analysis

The flexural strength, flexural modulus, maximum depth, and volume loss of the wear facets of the CAD/CAM resin composites were analyzed using a commercial statistical software package (SPSS Statistics Base, International Business Machines, Armonk, NY, USA). A two-way analysis of variance (ANOVA) and Tukey post hoc test were used for analysis of each data set with a significance level of 0.05.

### Scanning Electron Microscopy

Ultrastructural observations were completed on the polished surfaces of CAD/CAM resin composites with argon-ion etching using field-emission scanning electron microscopy (SEM; ERA 8800FE, Elionix, Tokyo, Japan). The surfaces were polished with 600-, 1200-, and 4000-grit SiC paper using a grinder-polisher (Ecomet 4/Automet 2, Buehler). The surfaces were polished with abrasive discs (Fuji Star Type DDC, Sankyo-Rikagaku, Saitama, Japan) followed by a series of diamond pastes down to 0.25 μm particle size (DP-Paste, Struers) to bring the surfaces to a high gloss. SEM specimens of the polished surfaces were dehydrated by immersion in ascending concentrations of aqueous tert-butanol (50% for 20 minutes, 75% for 20 minutes, 95% for 20 minutes, and 100% for 2 hours) and were then transferred to a critical-point dryer (Model ID-3, Elionix) for 30 minutes. These polished surfaces were etched for 30 seconds using an argon ion-beam (EIS-200ER; Elionix) directed perpendicularly to the surface at an accelerating voltage of 1.0 kV and an ion current density of 0.4 $mA/cm^2$. This treatment enhances the visibility of filler particles.[21] Surfaces were coated with a thin film of gold in a vacuum evaporator (Quick Coater Type SC-701, Sanyu Electron, Tokyo, Japan). SEM observations were carried out using an operating voltage of 10 kV.

SEM (TM3000 Tabletop Microscope, Hitachi-High Technologies, Tokyo, Japan) examinations were also accomplished on the wear facets of the CAD/CAM resin composites. Following the wear analysis, three representative specimens per group were coated with a gold-palladium thin film in a vacuum evaporator (Emitech SC7620 Mini Sputter Coater, Quorum Technologies, Ashford, UK). The SEM observations were carried out using an operating voltage of 15 kV.

## RESULTS

The results for the flexural properties of CAD/CAM resin composites are shown in Table 2. The two-way ANOVA revealed that material type and thermal cycling had a significant influence ($p<0.05$) on the flexural strength and modulus. In addition, there was no significant ($p>0.05$) interaction between the two factors. The flexural strength of CAD/CAM resin composites after 24 h water storage and 10,000 TCs ranged from 143.6 to 197.3 MPa and 140.3 to 178.7 MPa, respectively. The flexural modulus after 24 h water storage and 10,000 TCs ranged from 9.9 to 23.2 GPa and 8.2 to 22.2 GPa, respectively. Flexural properties after 24 h water storage and 10,000 TCs were significantly different ($p<0.05$) depending on the material. The flexural properties of CAD/CAM resin composites after 10,000 TCs were significantly lower ($p<0.05$) than those after 24 h water storage except for VE.

Table 2: *Flexural Properties of CAD/CAM Resin Composites*[a]

| CAD/CAM Resin Composite | 24 h Water Storage | | 10,000 TCs | |
|---|---|---|---|---|
| | Flexural Strength (MPa) | Elastic Modulus (GPa) | Flexural Strength (MPa) | Elastic Modulus (GPa) |
| LU | 180.3 (11.8) [a,A] | 13.8 (0.9) [a,A] | 159.3 (12.5) [a,B] | 12.5 (0.9) [a,B] |
| PM | 168.5 (14.5) [b,A] | 9.9 (1.3) [b,A] | 144.7 (14.0) [b,B] | 8.2 (0,7) [b,B] |
| CS | 197.3 (14.2) [a,A] | 12.2 (1.1) [c,A] | 178.7 (10.7) [c,B] | 11.1 (0.9) [c,B] |
| SB | 175.2 (12.3) [a,b,A] | 10.1 (0.7) [b,A] | 150.6 (13.5) [a,b,B] | 9.7 (0.7) [b,B] |
| KA | 189.8 (12.8) [a,A] | 12.4 (0.8) [c,A] | 170.4 (11.9) [c,B] | 10.4 (0.8) [b,c,B] |
| VE | 143.6 (9.7) [b,A] | 23.2 (1.4) [d,A] | 140.3 (10.7) [a,c,A] | 22.2 (1.4) [d,A] |

*Abbreviations: CAD/CAM, computer-aided design/computer-aided manufacturing; CS, CERASMART; KA, KATANA AVENCIA Block; LU, Lava Ultimate CAD/CAM; PM, Paradigm MZ100 Block; SB, SHOFU Block HC; VE, VITA ENAMIC.*
*[a] Values in parentheses are standard deviations (n = 15). Same small letter in columns indicates no significant difference (p > 0.05). Same capital letter in rows indicates no significant difference (p > 0.05).*

The results for the localized simulated wear of CAD/CAM resin composites are shown in Table 3. Two-way ANOVA revealed that material type and for thermal cycling had a significant influence ($p<0.05$) on the maximum depth or volume loss. In addition, there was no significant interaction ($p>0.05$) between the two factors. The maximum depth and volume loss of wear facets after 24 h water storage and 10,000 TCs were significantly different ($p<0.05$) depending on the material. The maximum depths of wear facets on CAD/CAM resin composites after 10,000 TCs were significantly higher ($p<0.05$) than those after 24 h water storage except for VE. The volume loss of wear facets on LU, PM, and SB after 10,000 TCs was significantly higher ($p<0.05$) than that after 24 h water storage, unlike for CS, KA, and VE.

Representative SEM images of polished CAD/CAM resin composite surfaces with argon-ion etching are shown in Figure 1. There were clear differences in filler-particle size, shape, and distribution. The resin composites exhibited a wide variety of filler-particle sizes and shapes. The particle size distribution of LU, PM, and SB appeared to include larger particles than either CS or KA. On the other hand, the polished VE surfaces showed a different structure compared with other CAD/CAM resin composites, and an irregular ceramic network was observed.

Representative SEM images of the localized wear facets after 10,000 TCs are shown in Figure 2. The worn surfaces showed evidence of particle loss (plucking). There did appear to be a qualitative morphological difference, in that LU, PM, and SB seemed to have more filler-particle plucking than CS, KA, and VE. This observation is consistent with the difference in volume loss of wear facets after 10,000 TCs.

## DISCUSSION

In the present study, the measurement of flexural properties of CAD/CAM resin composites was conducted according to ISO 6872, a standard normally used to measure the flexural properties of dental ceramics.[20,22] On the other hand, the flexural strength measurement using the three-point bending test specified by ISO 4049[23] is widely used to evaluate the flexural properties of polymer-based restorative materials.[11] Therefore, flexural strength measurement using the three-point bending test

Table 3: *Simulated Localized Wear of CAD/CAM Resin Composites*

| CAD/CAM Resin Composite | 24 h Water Storage | | 10,000 TCs | |
|---|---|---|---|---|
| | Maximum Depth (μm) | Volume Loss ($mm^3$) | Maximum Depth (μm) | Volume Loss ($mm^3$) |
| LU | 102.5 (14.9) [a,A] | 0.025 (0.008) [a,b,A] | 121.1 (14.9) [a,B] | 0.035 (0.009) [a,b,B] |
| PM | 133.7 (17.3) [b,A] | 0.035 (0.009) [b,A] | 150.7 (14.3) [b,B] | 0.047 (0.010) [b,B] |
| CS | 74.2 (11.4) [c,A] | 0.021 (0.009) [a,A] | 98.1 (12.1) [c,B] | 0.028 (0.009) [a,A] |
| SB | 127.1 (18.7) [b,A] | 0.032 (0.010) [a,b,A] | 147.8 (15.7) [b,B] | 0.046 (0.011) [b,B] |
| KA | 90.4 (12.8) [a,A] | 0.024 (0.008) [a,A] | 107.4 (12.8) [c,B] | 0.028 (0.010) [a,A] |
| VE | 69.2 (10.3) [c,A] | 0.019 (0.007) [a,A] | 74.2 (12.3) [d,A] | 0.021 (0.010) [a,A] |

*Abbreviations: CAD/CAM, computer-aided design/computer-aided manufacturing; CS, CERASMART; KA, KATANA AVENCIA Block; LU, Lava Ultimate CAD/CAM; PM, Paradigm MZ100 Block; SB, SHOFU Block HC; VE, VITA ENAMIC.*
*[a] Values in parentheses are standard deviations (n = 20). Same small letter in columns indicates no significant difference (p > 0.05). Same capital letter in rows indicates no significant difference (p > 0.05).*

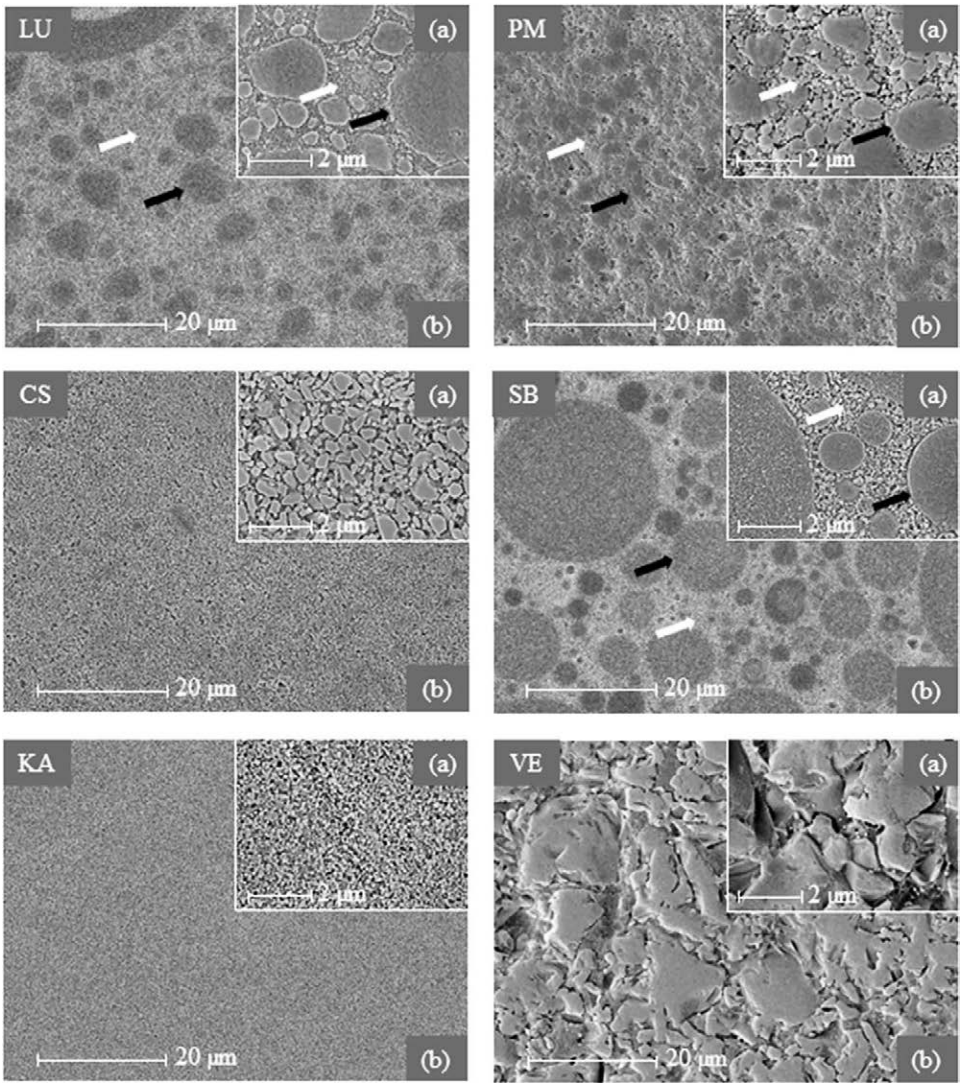


Figure 1. *Representative SEM images of the polished CAD/CAM resin composite surfaces at (a): 20,000× magnification and (b): 2500× magnification. LU, wide size range (1-20 μm) of irregular particles (black arrow) and small irregular particles (white arrow); PM, wide size range (1-15 μm) of irregular particles (black arrow) and small irregular particles (white arrow); CS, uniform small irregular particles; SB, wide size range (1-20 μm) of spherical particles (black arrow) and small spherical particles (white arrow); KB, uniform small irregular particles; VE, uniform irregular ceramic network. Size and distribution of fillers of CAD/CAM resin composite blocks were different, depending on the material.*

specified by ISO 4049 may appear to be a better way to evaluate the flexural properties of CAD/CAM resin composites. However, it has been pointed out that, whereas the ISO 4049 specifications for width and thickness are acceptable, the recommended length is clinically unrealistic.[24] In addition, it has been reported that although the flexural properties of resin composites measured by these test methods were not identical, the tests do provide similar results, even though the specifications use different specimen sizes.[25] The specimen size specified in ISO 6872 is as follows: length, 12.0 to 40.0 ± 0.5 mm; width, 4.0 ± 1.1 mm; thickness, 2.1 ± 1.1 mm . On the other hand, ISO 4049 specifies as follows: length, 25.0 ± 2.0 mm; width, 2.0 ± 0.1 mm; thickness, 2.0 ± 0.1 mm. Therefore, it is possible to use smaller specimens following ISO 6872. Due to the limited size of CAD/CAM resin composite blocks and tested materials, including the hybrid ceramic materials (VE), flexural strength measurement using the smaller specimens in this study.

The results showed that the flexural strength and modulus after 24 h water storage and after 10,000 TCs were different depending on the material. The results that are provided by the manufacturers of the inorganic filler contents show high variance (Table 1). Although increasing the filler load of resin composites purportedly increases the flexural strength and modulus,[26] there does not seem to be a clear relationship between flexural properties and inorganic filler contents in the present study. Therefore, it is speculated that the type of resin matrix, degree of conversion, and surface treatment of the filler might contribute to the flexural properties of CAD/CAM resin composites in the same manner as has been reported for other resin

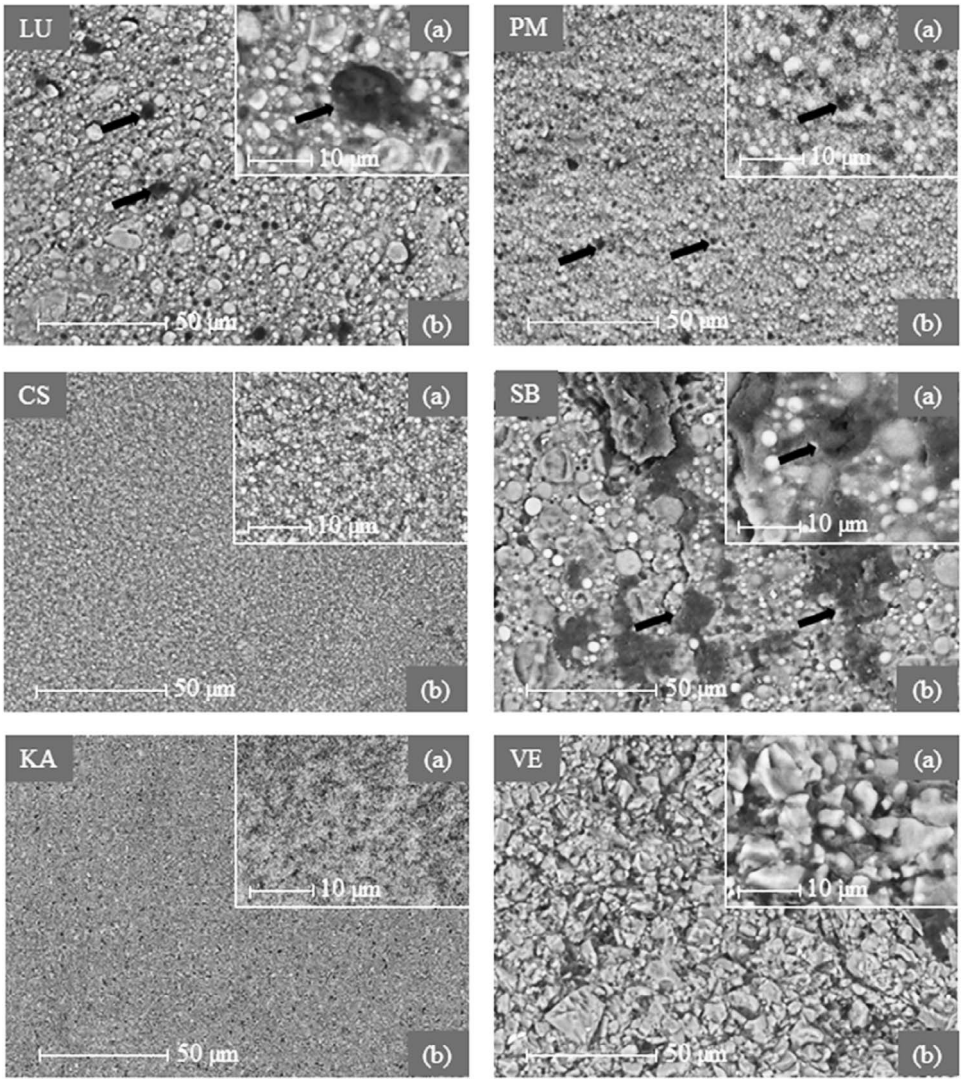


Figure 2. *Representative SEM images of the localized wear facets of CAD/CAM resin composites after 10,000 thermal cycles at (a): 20,000 × magnification and (b): 2500× magnification (b). The worn surfaces of CAD/CAM resin composites showed evidence of particle loss (plucking) from the simulated localized wear. There appeared to be a qualitative morphological difference: LU, PM, and SB seemed to have more filler-particle plucking than CS, KA, and VE.*

composites.[27] In addition, SEM observation of the polished CAD/CAM resin composite surfaces with argon-ion etching revealed that the size, shape, and distribution of fillers were different, depending on the material. A previous study[28] of resin composites revealed correlations between filler size, distribution of filler particles, and flexural properties. This may be one of the reasons why the flexural properties differed according to the materials used. It has also been reported that the type of polymerization has an effect on the physical properties of CAD/CAM resin composites.[14,29] Unfortunately, information about the polymerization methods from manufacturers is so limited that, in this respect, no further comparison can be made.

The flexural strength and modulus after 10,000 TCs were significantly lower than those after 24 h water storage but not those of VE. Immersion in water causes water penetration into the resin matrix of the resin composite, softening the polymer.[30] Furthermore, the absorbed water would cause hydrolysis of the interfacial silane coupling agent, especially in the case of zirconium dioxide, which cannot be effectively silanized due to its high crystalline content.[31] Accordingly, the flexural properties of CAD/CAM resin composites, especially LU, PM, and SB, which include zirconium dioxide, decreased after thermal cycling. However, VE, which is a polymer-infiltrated ceramic network enriched with aluminum, has unique characteristics as seen in the SEM observation of polished surfaces with argon-ion etching, and may be categorized as a CAD/CAM hybrid ceramic material.[32] It is thought that the amount of water absorption in this material is

lower than that of the others. Moreover, the ceramic component of the material forms an interconnected microstructure, which may explain the resistance of the resin-ceramic bond to hydrolytic degradation. Therefore, the flexural properties of the CAD/CAM hybrid ceramic material were not influenced by thermal cycling, unlike those of CAD/CAM resin composites.

Wear in OCA is caused by opposing tooth contact, which is considered a localized process mainly related to local microfracture.[33] Barkmeier and others[34] developed a laboratory simulated model capable of evaluating localized wear. This system transfers masticatory stresses to a composite specimen by means of a stainless steel conical stylus in the presence of a slurry of PMMA beads. This methodology has facilitated the development of *in vitro* studies capable of helping to predict *in vivo* performance. A previous study using clinical data from two study sites on two resin composites found a good relationship between simulated localized wear in the laboratory and OCA clinical wear.[35] Using this system, simulated localized wear of CAD/CAM resin composites was also evaluated in this study.

In the present study, the maximum depth and volume loss of wear facets on the CAD/CAM resin composites after 24 h water storage and 10,000 TCs were significantly different, depending on the material. If we consider the CAD/CAM resin composites (excluding VE), the maximum depth and volume loss of wear facets on CS and KA after 24 h water storage and 10,000 TCs were significantly smaller than those of LU, PM, and SB. The mean filler size has previously been reported to affect the wear properties of resin composites, and smaller-sized filler particles were associated with less wear.[27,36] From the SEM observations of the polished CAD/CAM resin composite surfaces with argon-ion etching, equally distributed nanoparticle fillers were observed on CS and KA, whereas a wide size range of larger particle fillers was observed on the polished surfaces of LU, PM, and SB. Therefore, it is speculated that the lower wear of some CAD/CAM resin composites can be attributed to the smaller particle of their fillers. Such improved wear resistance has been hypothesized to result from smaller inter-particle spacing between the fillers of small-particle composites.[37] The smaller particle fillers become more closely packed, so that the resin between them is protected from further abrasion from neighboring particles.[38] Furthermore, the flexural properties of resin composite have previously been correlated[39] with quantitative wear data and specifically seem to reflect wear performance. If little energy is needed to fatigue and fracture the materials, cracks appear to form more readily, resulting in an increased wear rate. Consequently, CS and KA, with relatively high flexural properties, demonstrated less wear than LU, PM, and SB, with lower flexural properties.

Although the maximum depth of wear facets after 10,000 TCs was significantly higher than those after 24 h water storage, except in VE, the volume loss of wear facets on LU, PM, and SB after 10,000 TCs was significantly higher than that after 24 h water storage, unlike in CS and KA. From the SEM observations of the localized wear facets after 10,000 TCs, the LU, PM, and SB materials seemed to have more filler-particle plucking than CS and KA. In addition, the flexural strengths of LU, PM, and SB after 10,000 TCs were significantly lower than those of CS and KA. This may be one reason why LU, PM, and SB showed higher volume loss of wear facets after 10,000 TCs than after 24 h water storage. Furthermore, the maximum depth and volume loss of wear facets on VE after 24 h water storage and after 10,000 TCs did not show any significant difference, whereas the flexural properties of VE were not influenced by thermal cycling. Overall, there may be a relationship between flexural properties and simulated wear in CAD/CAM resin composites. However, it is important to bear in mind that there are a number of different ways to simulate wear, and the method chosen may influence the results.[40] Therefore, different results may be obtained if the testing is performed using a different wear simulator. Further research is needed to confirm that these findings can be reproduced using different approaches to wear simulation.

According to the results of this study, the first null hypothesis (there are no significant differences in flexural properties and simulated wear of CAD/CAM resin composites) was rejected. The second null hypothesis (flexural properties and simulated wear of CAD/CAM resin composites are not influenced by thermal cycling) was also rejected.

## CONCLUSION

The results of this study indicate that the flexural properties and simulated wear of CAD/CAM resin composite are different, depending on the material. In addition, the flexural properties and simulated wear of CAD/CAM resin composites were influenced by thermal cycling. It appears that the selection of a CAD/CAM resin composite for clinical use should be carefully made with special considerations regarding

the physical properties of the material. Clinicians should put more weight on the difference in physical properties, as well as degradation by thermal stress and hydrolysis, between CAD/CAM resin composites and ceramics when making clinical decisions.

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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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**NEW YORK UNIVERSITY COLLEGE OF DENTISTRY**

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### Effect of Insufficient Light Exposure on Polymerization Kinetics of Conventional and Self-adhesive Dual-cure Resin Cements

**Y Jang • JL Ferracane • CS Pfeifer • JW Park • Y Shin • BD Roh**

Clinical Relevance: Insufficient light exposure could result in low degree of conversion of dual-cure resin cement, which is even lower than that of self-curing alone. Clinicians need to modify their curing strategies when sufficient light activation is difficult to achieve.

doi: http://dx.doi.org/10.2341/15-278-L

### A Conservative Technique for Repairing Class IV Composite Restorations

**VC Ruschel • SC Stolf • S Shibata • LN Baratieri**

Clinical Relevance: The repair of the facial surface of a class IV composite restoration with poor coloration is a minimally invasive treatment that allows satisfactory restoration of esthetics and function.

doi: http://dx.doi.org/10.2341/15-316-T

### Microcomputed Tomography Evaluation of Polymerization Shrinkage of Class I Flowable Resin Composite Restorations

**CS Sampaio • K-J Chiu • E Farrokhmanesh • M Janal • RM Puppin-Rontani • M Giannini • EA Bonfante • PG Coelho • R Hirata**

Clinical Relevance: Since polymerization shrinkage in a 2.5 mm deep class I cavity was, in general, not different in a self-adhesive, a bulk-fill, and a conventional flowable resin composite, endeavors to simplify clinical procedures and to reduce steps and treatment times are promising.

doi: http://dx.doi.org/10.2341/15-296-L

### The Silorane-based Resin Composites: A Review

**GA Maghaireh • NA Taha • H Alzraikat**

Clinical Relevance: Silorane-based resin composites (SBRC) have been found to exhibit a decrease in polymerization shrinkage stresses and properties at least as good as that associated with methacrylate-based resin composites. This review summarizes the current literature on the SBRC to help the dental practitioner trying to make evidence-based decisions.

doi: http://dx.doi.org/10.2341/15-311-LIT

### Dentin Protection of Different Desensitizing Varnishes During Stress Simulation: An In Vitro Study

**G Schmalz • F Hellwig • RF Mausberg • H Schneider • F Krause • R Haak • D Ziebolz**

Clinical Relevance: In treatment of dentin hypersensitivity, light-curing desensitizing varnishes might be able to avoid dentin loss. Consequently, these materials could be a promising preventive approach and may be preferred for clinical use.

doi: http://dx.doi.org/10.2341/16-068-L

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# Effect of Insufficient Light Exposure on Polymerization Kinetics of Conventional and Self-adhesive Dual-cure Resin Cements

Y Jang • JL Ferracane • CS Pfeifer • JW Park • Y Shin • BD Roh



### Clinical Relevance

Insufficient light exposure could result in low degree of conversion of dual-cure resin cement, which is even lower than that of self-curing alone. Clinicians need to modify their curing strategies when sufficient light activation is difficult to achieve.


## SUMMARY

**Objectives: The purpose of this study was to investigate the influence of insufficient light exposure on the polymerization of conventional and self-adhesive dual-cure resin cements under ceramic restorations.**

**Methods: Two conventional dual-cure resin cements (Rely-X ARC, Duolink) and two self-adhesive resin cements (Rely-X U200, Maxcem Elite) were polymerized under different curing modes (dual-cure or self-cure), curing times (20 and 120 seconds), and thickness of a ceramic overlay (2 and 4 mm). Polymerization kinetics was measured by Fourier transform infrared spectroscopy for the initial 10 minutes and after 24 hours. Data were analyzed using mixed model analysis of variance (ANOVA), one-way ANOVA/Student-Newman-Keuls post hoc test, and paired *t*-test ($\alpha$=0.05).**

**Results: When light-curing time was set to 20 seconds, the presence of the ceramic block significantly affected the degree of conversion (DC) of all resin cements. Especially, the DC of the groups with 20 seconds of light-curing time under 4 mm of ceramic thickness was even lower than that of the self-cured groups at 24 hours after polymerization ($p<0.05$). However, when light-curing time was set to 120 seconds,**



Youngjune Jang, DDS, PhD, Yonsei University, Department of Conservative Dentistry and Oral Science Research Center, College of Dentistry, Seoul, Republic of Korea

Jack L Ferracane, PhD, Oregon Health & Science University, Restorative Dentistry, Portland, OR, USA

Carmem Silvia Pfeifer, DDS, PhD, Oregon Health & Science University, Restorative Dentistry, Portland, OR, USA

Jeong-Won Park, DDS, MSD, PhD, Yonsei University, Department of Conservative Dentistry and Oral Science Research Center, College of Dentistry, Gangnam Severance Hospital, Seoul, Republic of Korea

Yooseok Shin, DDS, MSD, PhD, Yonsei University, Department of Conservative Dentistry and Oral Science Research Center, College of Dentistry, Seoul, Republic of Korea

*Byoung-Duck Roh, DDS, MSD, PhD, Yonsei University, Department of Conservative Dentistry and Oral Science Research Center, College of Dentistry, Seoul, Republic of Korea

*Corresponding author: 50-1 Yonsei-ro, Seodaemun-gu, Seoul 03722, Republic of Korea; e-mail: operatys16@yuhs.ac


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**a similar DC compared with the group with direct light exposure ($p>0.05$) was achieved in all dual-cure groups except Maxcem Elite, at 24 hours after polymerization.**

**Conclusions: For both conventional and self-adhesive dual-cure resin cements, insufficient light exposure (20 seconds of light-curing time) through thick ceramic restoration (4 mm thick) resulted in a DC even lower than that of self-curing alone.**

## INTRODUCTION

Nowadays, dual-cure resin cements have been the material of choice in bonding of ceramic restorations. They rely both on light-cure and chemical-cure mechanisms in an attempt to ensure sufficient polymerization throughout the material, even without proper light activation. However, it has been claimed that the two components do not always work synergistically.[1-3] Meng and others[4] reported poor microhardness of dual-cured resin cements, even lower than the self-cured group, when it was insufficiently light-activated through a 3-mm-thick ceramic restoration overlay. They suggested that the early vitrification induced by insufficient light activation could interfere with the subsequent self-polymerization, compromising the overall polymerization of dual-cure resin cements.

This is an important issue when considering that predictable outcomes of ceramic restorations are closely associated with sufficient polymerization of underlying resin cement.[5] However, there has been a lack of evidence regarding the characteristics of this phenomenon. It is essential to observe the initial polymerization kinetics to understand the polymerization characteristics of materials in different conditions; however, few studies have provided such information having only presented intermittent measurements such as microhardness at 24 hours after polymerization.[4] Therefore, the understanding of this issue remains unclear, and accordingly, the risk of insufficient light exposure on dual-cure resin cement has not been well recognized in the field of restorative dentistry. Most manufacturers do not consider ceramic thickness when recommending light-curing protocols for dual-cure resin cements, which is one of the major factors that determines the amount of light transmission.[6,7] Moreover, several manufacturers have recommended even further reduced light-curing times, ie, 20 seconds or below, for some recent materials. Therefore, there is a need to verify this issue and make guidelines that are clinically relevant.

The purpose of this study was to investigate the influence of insufficient light exposure on the polymerization of conventional and self-adhesive dual-cure resin cements under ceramic restorations. For this, several different light-curing conditions were simulated by combining the ceramic restoration thicknesses and the light-curing times, and polymerization kinetics were measured for the initial 10 minutes and then followed up after 24 hours. The null hypothesis tested was as follows: dual-cured resin cements always show higher degree of conversion (DC) compared to self-cured resin cements.

## METHODS AND MATERIALS

### Fabrication of Ceramic Blocks

Lithium disilicate ceramic blocks (10×10 mm, IPS e.max CAD/CAM, shade A3, low translucency, Ivoclar Vivadent, Schaan, Liechtenstein) were cut by low-speed diamond saw (Dk-2610, Struers Minitom, Rodovre, Denmark) and polished with 400-, 600-, and 800-grit silicon carbide papers, and their final thickness was adjusted to 2.00 ± 0.01 and 4.00 ± 0.01 mm. They were cleaned with ultrasonic cleaner (BioSonic UC50D, Coltène/Whaledent, Cuyahoga Falls, OH, USA) for 5 minutes and then subjected to the crystallization process in a ceramic furnace (Programat P300, P81 mode, Ivoclar Vivadent).

### Experimental Groups

Two conventional dual-cure resin cements and two self-adhesive resin cements were studied (Table 1). Each group of resin cement contained six subgroups (2 ceramic thicknesses × 2 different light-curing times + 1 positive control (direct light exposure [DLE]) + 1 negative control (self-cured [SC]). Five specimens were prepared for each subgroup ($n=5$). The descriptions of the tested groups are given in Table 2.

### Measurement of the Curing Light Intensity Through Ceramic Blocks

The light transmission value of an LED curing light (Bluephase, Ivoclar Vivadent) was measured with a laser power meter (Powermax 5200, Molectron, Portland, OR, USA) with and without a ceramic block. Measurements were repeated five times for each group, and the average value was calculated (Table 3).

### Specimen Preparation

Resin cements were mixed according to the manufacturer's instructions and placed on a glass slide.

Table 1: *Dual-cure Resin Cements and Self-Adhesive Resin Cements Used in This Study*

| Resin cement | Manufacturer | Shade | Recommended light exposure protocol | Batch |
|---|---|---|---|---|
| Rely-X ARC | 3M ESPE, St Paul, MN, USA | Transparent | Light cure 40 s/surface | N352609 |
| Duolink | Bisco, Schaumburg, IL, USA | Transparent | Light cure 40 s/surface | 1200003240 |
| Rely-X U200 (Self adhesive) | 3M ESPE, St Paul, MN, USA | Transparent | Single surface, from occlusal: 20 seconds | 491292 |
| | | | Any other surface: additional 20 seconds | |
| Maxcem Elite (Self adhesive) | Kerr, Orange, CA, USA | Clear | Light cure 10 seconds when irradiance is 1000 mW/cm$^2$ | 4720553 |
| | | | Light cure 20 seconds when irradiance is 600 mW/cm$^2$ | |

Adhesive tape (Scotch Tape, 3M, St. Paul, MN, USA) was applied to the glass slide before resin cement placement and served as a spacer (100±10 μm) to ensure a constant thickness of the resin cement layer. Then resin cement was covered with a Mylar strip and pressed with another slide glass to remove the excess cement. Then a ceramic block (2 or 4 mm thick) was placed above the Mylar strip. For the SC group, a 2-mm-thick ceramic block was placed on the specimen. For the DLE group, additional glass slides were placed over the specimen to control the distance between the light-curing unit tip and the specimen at 2 mm (Figure 1).

### Measurement of Degree of Conversion

The specimen was mounted in a plastic holder and placed in the Fourier transform infrared spectrometer (Nicolet 6700 FT-IR Spectrometer, Thermo, Madison, WI, USA) at a 45° vertical angle (Figure 1). Infrared spectra were recorded immediately after the placement, and then the specimen was illuminated with the LED curing light for 20 or 120 seconds, except for the SC group. During the illumination, the curing light was fixed with an additional holder to maintain a uniform position throughout the whole experiment. Spectra collection was set up between 6140 and 6200 cm$^{-1}$ on transmission enhanced synchronous protocol, near infrared (IR) mode, taking one spectrum per second (2 scans/spectrum) at 4 cm$^{-1}$ resolution. Data collection was continued for 600 seconds, and each test condition was replicated five times. DC was calculated by the following equation:

$$\mathrm{DC}(\%) = \{1 - (\text{Peak area [p]}/\text{Peak area [u]})\} \times 100(\%),$$

where u and p refer to the unpolymerized and polymerized cement, respectively, with the reaction peak set at 6165 cm$^{-1}$.[8-10] Peak area (u) was calculated by averaging values over the first 20 seconds before light exposure (40 data points), and peak area (p) was measured twice at 10 minutes and 24 hours after data collection. Peak area (p) at 10 minutes was calculated by averaging values of the last 50 seconds (100 data points), and peak area (p) at 24 hours was calculated by averaging values for 20 seconds (40 data points) after 24 hours.

The polymerization rate curve was obtained by taking the first derivative of the DC regarding the time. The maximum value of the derivative was taken as the maximum polymerization rate (Rp max, ΔDC%/s), and the time required to reach the Rp max was taken as the time of maximum polymerization rate (Time of Rp max, seconds).

Polymerized specimens were removed from the holder after 10 minutes, and resin cement thickness was measured with a digital micrometer (Absolute Digimatic Caliper, Mitutoyo Corp, Aurora, IL, USA)

Table 2: *Experimental Groups With Different Ceramic Thickness and Curing Methods*

| Ceramic thickness, mm | Curing time, s | Resin cements and code of experimental groups | | | |
|---|---|---|---|---|---|
| | | Rely-X ARC | Duolink | Rely-X U200 | Maxcem Elite |
| 0 | 120 | A DLE | D DLE | U DLE | M DLE |
| 2 | 120 | A 2-120 | D 2-120 | U 2-120 | M 2-120 |
| 2 | 20 | A 2-20 | D 2-20 | U 2-20 | M 2-20 |
| 4 | 120 | A 4-120 | D 4-120 | U 4-120 | M 4-120 |
| 4 | 20 | A 4-20 | D 4-20 | U 4-20 | M 4-20 |
| 2 | 0 | A SC | D SC | U SC | M SC |

Table 3: *Light Transmittance Mean Values (SD) for Each Experimental Condition (n=5)*

| | Positive control group | Experimental groups | |
|---|---|---|---|
| Ceramic thickness, mm | 0 | 2 | 4 |
| Distance from light-curing unit, mm | 2 | 2 | 4 |
| Irradiance, mW/cm$^2$ | 1085.4 (6.3)[a] | 128.2 (2.6)[b] | 25.6 (0.9)[c] |

*Significant differences are shown by different letters within row according to Student-Newman-Keuls post hoc test (p<0.05).*

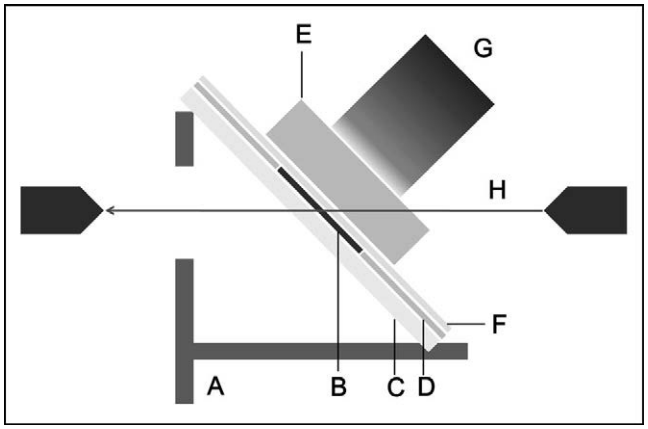


Figure 1. *Specimen positioning. (A) Holder; (B) resin cement; (C) slide glass; (D) adhesive tape; (E) ceramic block or slide glass; (F) mylar strip; (G) light-curing unit tip; and (H) IR path.*

to ensure uniform thickness among the specimens. Then both dual-cured and self-cured specimens were stored in light-proof, dry conditions at 25°C for 24 hours. An IR spectrum was collected again for each specimen after 24 hours, and the DC was calculated.

IR spectra data were extracted from the IR spectra analysis software package OMNIC 6.0 (Thermo Electron, Inc, Madison, WI, USA). Raw data were analyzed by PeakFit v4.12 (Systat Software, Inc, San Jose, CA, USA) and smoothed under the Loess algorithm, 2.0% level. A representative curve was obtained by averaging data of five specimens in each group.

### Statistical Analysis

Statistical analysis was performed using SPSS version 20 (SPSS Inc, Chicago, IL, USA). For DC of resin cements, one-way analysis of variance (ANOVA) was done between six groups (4 experimental groups + 2 control groups) to compare DC (%) at 10 minutes, DC (%) at 24 hours, Rp max (ΔDC %/s), and time of Rp max (seconds), and a Student-Newman-Keuls post hoc test was done for multiple comparisons. Then the paired *t*-test was done to compare DC (%) at 10 minutes and DC (%) at 24 hours for each group. For light transmission values of curing light, one-way ANOVA was done, followed with a Student-Newman-Keuls post hoc test for multiple comparisons ($\alpha$=0.05).

## RESULTS

Table 4 shows the mean and standard deviations (SD) of the DC (%), Rp max (ΔDC %/s), and time of Rp max (seconds) of the four resin cements. Light transmittance values for each experimental condition are given in Table 3.

Table 4: *DC (%) (SD) at 10 Minutes and 24 Hours After Polymerization, Maximum Polymerization Rate (ΔDC %/s), and Time of Maximum Polymerization Rate (Seconds) of Rely-X ARC, Duolink, Rely-X U200, and Maxcem Elite*

| **Rely-X ARC** | **A DLE** | **A 2-120** | **A 4-120** | **A 2-20** | **A 4-20** | **A SC** |
|---|---|---|---|---|---|---|
| DC 10 minutes | 80.65 (0.69)$^{A,b}$ | 78.89 (1.10)$^{B,b}$ | 75.09 (2.07)$^{C,b}$ | 71.92 (1.02)$^{D,b}$ | 63.46 (1.71)$^{E,b}$ | 56.25 (0.86)$^{F,b}$ |
| DC 24 hours | 86.97 (0.97)$^{A,a}$ | 86.48 (0.97)$^{A,a}$ | 85.29 (2.27)$^{A,a}$ | 82.82 (0.94)$^{B,a}$ | 75.78 (2.22)$^{D,a}$ | 78.98 (1.68)$^{C,a}$ |
| Rp max | 7.42 (0.82)$^{A}$ | 4.80 (0.40)$^{B}$ | 3.44 (0.60)$^{C}$ | 4.73 (0.38)$^{B}$ | 2.75 (0.41)$^{D}$ | 1.08 (0.18)$^{E}$ |
| Time of Rp max | 4.55 (0.73)$^{B}$ | 7.33 (1.23)$^{B}$ | 13.07 (1.97)$^{B}$ | 8.42 (0.93)$^{B}$ | 10.69 (2.23)$^{B}$ | 137.38 (33.14)$^{A}$ |
| **Duolink** | **D DLE** | **D 2-120** | **D 4-120** | **D 2-20** | **D 4-20** | **D SC** |
| DC 10 minutes | 69.27 (1.66)$^{A,b}$ | 69.62 (1.44)$^{A,b}$ | 69.69 (1.20)$^{A,b}$ | 62.95 (1.30)$^{B,b}$ | 60.23 (1.13)$^{C,b}$ | 53.79 (1.30)$^{D,b}$ |
| DC 24 hours | 74.55 (1.84)$^{A,a}$ | 74.74 (1.24)$^{A,a}$ | 75.56 (0.51)$^{A,a}$ | 69.65 (1.45)$^{B,C,a}$ | 68.05 (1.10)$^{C,a}$ | 70.91 (1.23)$^{B,a}$ |
| Rp max | 7.51 (1.22)$^{A}$ | 3.98 (0.61)$^{B}$ | 2.47 (0.22)$^{C}$ | 4.17 (0.36)$^{B}$ | 2.52 (0.27)$^{C}$ | 0.65 (0.13)$^{D}$ |
| Time of Rp max | 3.07 (0.65)$^{B}$ | 4.55 (0.89)$^{B}$ | 9.90 (1.26)$^{B}$ | 4.36 (0.73)$^{B}$ | 12.87 (0.78)$^{B}$ | 144.10 (42.04)$^{A}$ |
| **Rely-X U200** | **U DLE** | **U 2-120** | **U 4-120** | **U 2-20** | **U 4-20** | **U SC** |
| DC 10 minutes | 65.38 (1.21)$^{A,b}$ | 65.47 (1.70)$^{A,b}$ | 63.75 (1.27)$^{A,b}$ | 56.92 (1.53)$^{B,b}$ | 49.39 (2.44)$^{C,b}$ | 30.63 (2.81)$^{D,b}$ |
| DC 24 hours | 71.63 (1.28)$^{A,a}$ | 71.86 (1.88)$^{A,a}$ | 69.78 (0.65)$^{A,a}$ | 64.28 (1.60)$^{B,a}$ | 58.14 (1.69)$^{D,a}$ | 61.73 (2.17)$^{C,a}$ |
| Rp max | 5.04 (0.90)$^{A}$ | 2.80 (0.29)$^{B}$ | 2.59 (0.45)$^{B}$ | 3.00 (0.36)$^{B}$ | 2.32 (0.55)$^{B}$ | 0.61 (0.27)$^{C}$ |
| Time of Rp max | 4.55 (0.73)$^{B}$ | 4.85 (0.41)$^{B}$ | 9.60 (1.90)$^{B}$ | 4.65 (0.75)$^{B}$ | 7.03 (2.85)$^{B}$ | 170.68 (45.15)$^{A}$ |
| **Maxcem Elite** | **M DLE** | **M 2-120** | **M 4-120** | **M 2-20** | **M 4-20** | **M SC** |
| DC 10 minutes | 69.97 (2.03)$^{A,b}$ | 68.31 (1.41)$^{A,b}$ | 63.39 (2.96)$^{B,b}$ | 55.84 (1.25)$^{C,b}$ | 48.43 (3.11)$^{D,b}$ | 54.77 (1.21)$^{C,b}$ |
| DC 24 hours | 75.48 (2.18)$^{A,a}$ | 74.56 (1.69)$^{A,a}$ | 70.80 (3.49)$^{B,a}$ | 64.57 (1.07)$^{C,a}$ | 59.50 (2.33)$^{D,a}$ | 69.61 (1.14)$^{B,a}$ |
| Rp max | 3.22 (0.72)$^{A}$ | 1.78 (0.36)$^{B}$ | 1.88 (0.29)$^{B}$ | 1.58 (0.23)$^{B}$ | 1.67 (0.19)$^{B}$ | 0.96 (0.10)$^{C}$ |
| Time of Rp max | 4.36 (1.07)$^{B}$ | 7.33 (1.54)$^{B}$ | 10.89 (1.64)$^{B}$ | 7.72 (1.34)$^{B}$ | 10.79 (1.33)$^{B}$ | 94.50 (14.83)$^{A}$ |

*Significant differences are written by different letters (uppercase letters within row; lowercase letters within column) within each resin cement according to Student-Newman-Keuls post hoc test ($p<0.05$).*

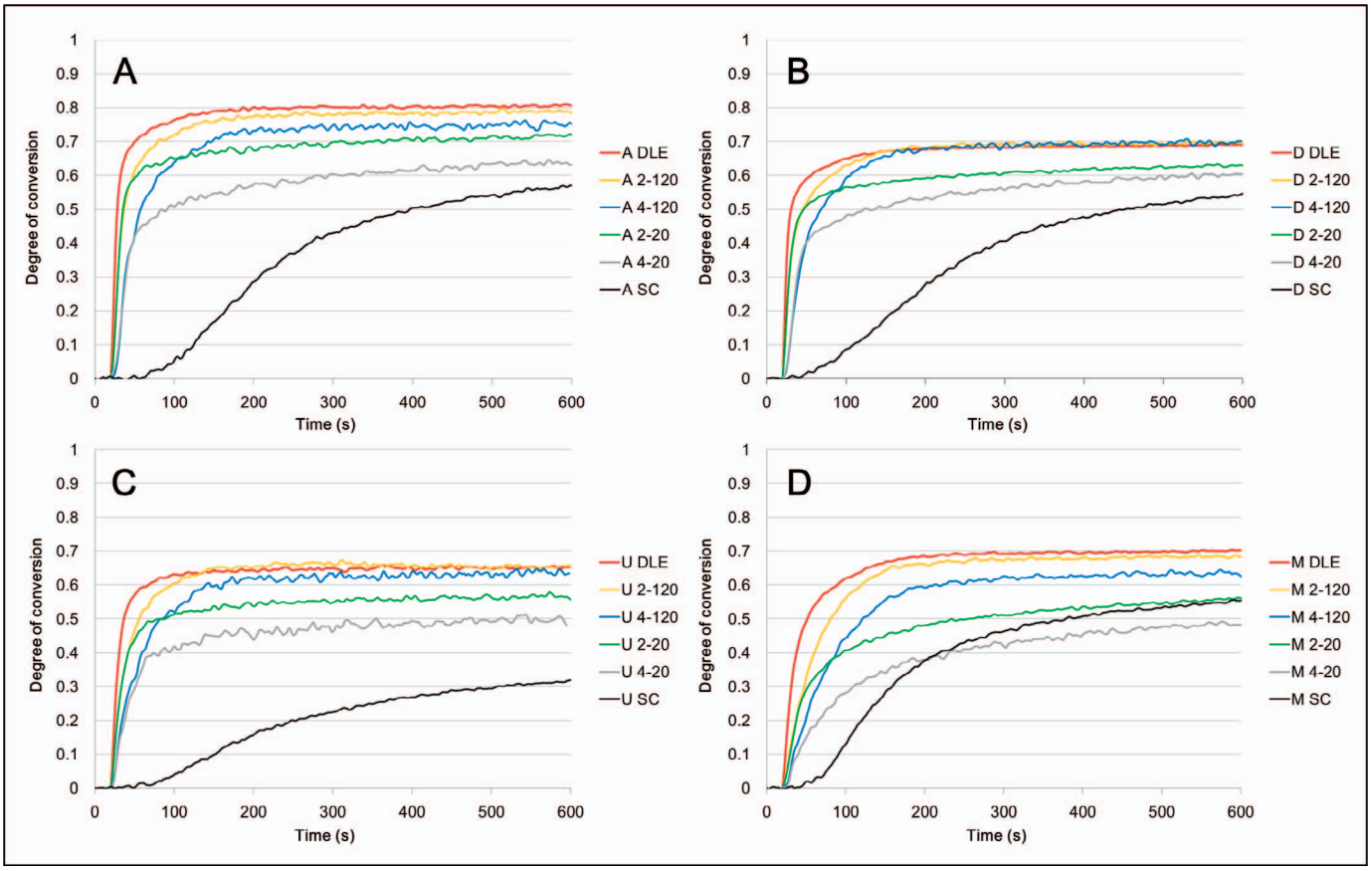


Figure 2. *Representative real-time polymerization profiles of (A) Rely-X ARC, (B) Duolink, (C) Rely-X U200, and (D) Maxcem Elite according to light-curing condition during the initial 10 minutes.*

### Polymerization Kinetics (0-10 Minutes)

Figure 2 shows the effects of light-curing condition on the real-time polymerization profile for each resin cement. Generally, the dual-cure groups showed a rapid DC increase immediately after the light exposure, whereas the self-cure groups showed a more gradual increase. Figure 3 describes polymerization rate as a function of DC. When assuming the point of DC with Rp max as the vitrification point,[11] dual-cure groups with a 4-mm-thick ceramic overlay revealed early vitrification in relation to other dual-cure groups, whereas the point of vitrification was not apparent in self-cure groups (Figure 3). The overlying ceramic thickness (0, 2, and 4 mm thick) significantly influenced Rp max and time of Rp max (Table 4), which is directly related to the polymerization profile before 20 seconds.

### DC (10 Minutes and 24 Hours)

At 10 minutes after polymerization, significantly higher DC was observed in all dual-cure groups compared with the self-cure group, except Maxcem Elite. However, at 24 hours after polymerization, the DC of the self-cure groups equaled or surpassed that of the 4-mm, 20-second groups regardless of the material, and the M SC group showed even higher DC compared with M 2-20 and M 4-20 groups ($p<0.05$), which means there was even more increase of DC in the self-cure group (Table 4; Figure 4).

The self-cured group revealed the highest DC increase for all resin cements when comparing 10 minutes and 24 hours (Table 4). For the dual-cured groups, the DC increase had a trend of inverse relationship with the amount of initial light energy received.

When light-curing time was set to 20 seconds, DC was significantly affected by the presence of the ceramic block, resulting in significantly lower values compared with the group with DLE at 24 hours after polymerization ($p<0.05$). However, when light-curing time was set to 120 seconds, a similar DC compared with the group with DLE ($p>0.05$) was achieved in all dual-cure groups at 24 hours,

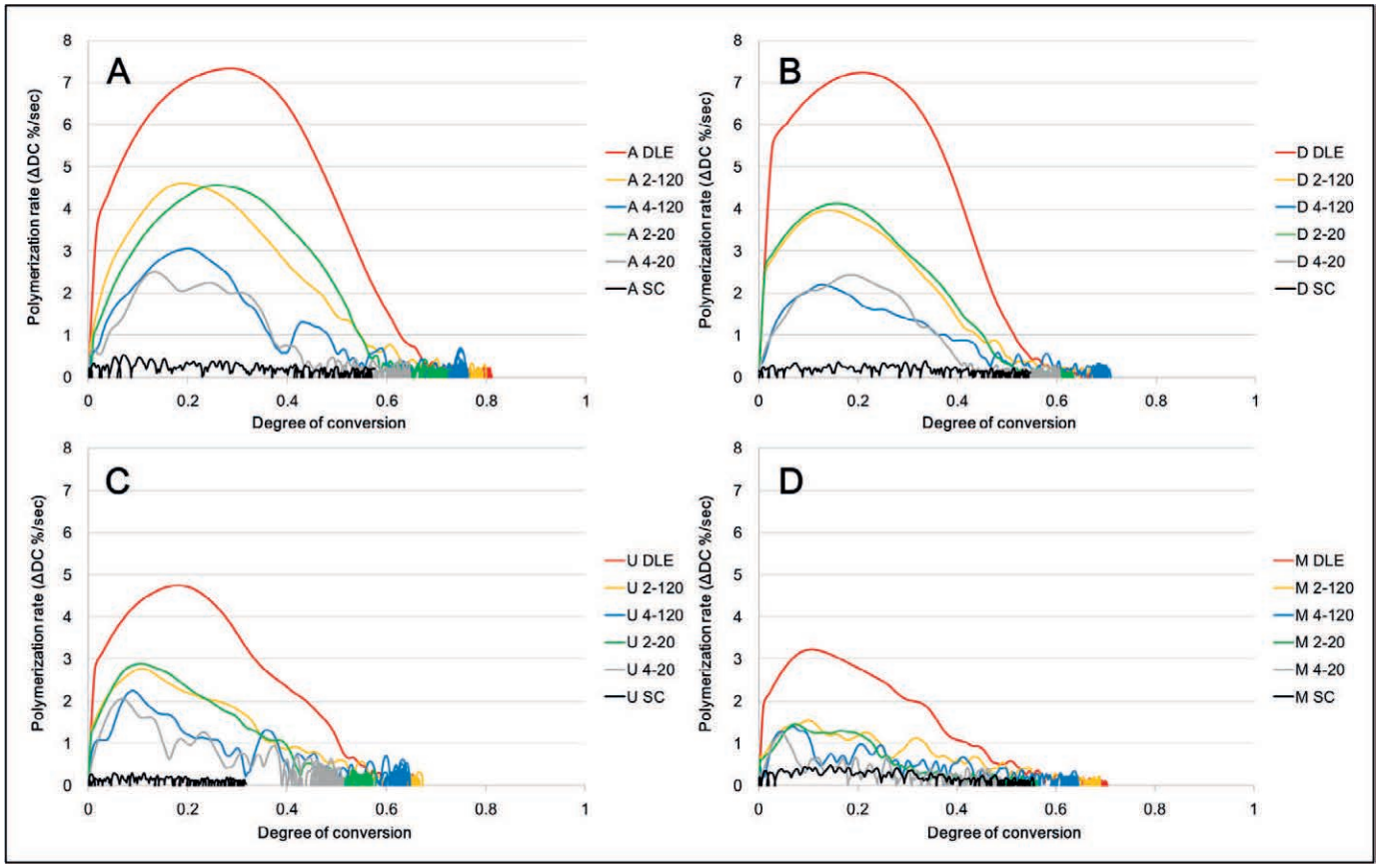


Figure 3. *Polymerization rate as a function of DC for (A) Rely-X ARC, (B) Duolink, (C) Rely-X U200, and (D) Maxcem Elite according to light-curing condition during the initial 10 minutes.*

regardless of the ceramic thickness, except Maxcem Elite at 4 mm (Table 4; Figure 4).

## DISCUSSION

The most notable point of this study is that all 4-mm, 20-second groups showed significantly lower DC compared with the SC groups at 24 hours after polymerization, rejecting the null hypothesis (Table 4; Figure 4). This confirms that insufficient light exposure of a self-adhesive resin cement could result in incomplete polymerization, even lower than that of self-curing alone, which is consistent with previous studies that investigated conventional dual-cure resin cements.[2,4]

As dimethacrylate-based dental composites are polymerized, mobility of radicals becomes restricted by the growing cross-linked polymer network, and radicals become essentially immobilized after the point of vitrification.[2] In the dual-cure mode, light activation induces rapid polymerization, with large amounts of free radicals becoming trapped within the organic matrix at the initial stage of polymerization, if insufficient curing energy is applied to drive the reaction further to completion.[1-3] Considering that the self-cure mechanism proceeds more slowly, the early vitrification brought on by the initial light activation minimizes the extent of the subsequent self-polymerization of the dual-cure resin cement, due to restricted molecular mobility. When a sufficient amount of light energy is delivered to a dual-cure resin cement, this kind of competitive reaction would not be apparent because the dual-cure resin cement has been polymerized well even if the self-cure component becomes partially impaired. In contrast, in the absence of light energy being delivered to the resin cement, a certain degree of polymerization could be attained solely due to the progress of the self-cure component. In this study, dual-cure groups with insufficient light exposures (4-mm, 20-second and 4-mm, 120-second groups) revealed early vitrification in relation to other dual-cure groups (Figure 3), which is known to be associated with the reduction in overall amount of polymerization.[11] Meanwhile, the point of vitrification was not apparent in self-cure groups (Figure 3),

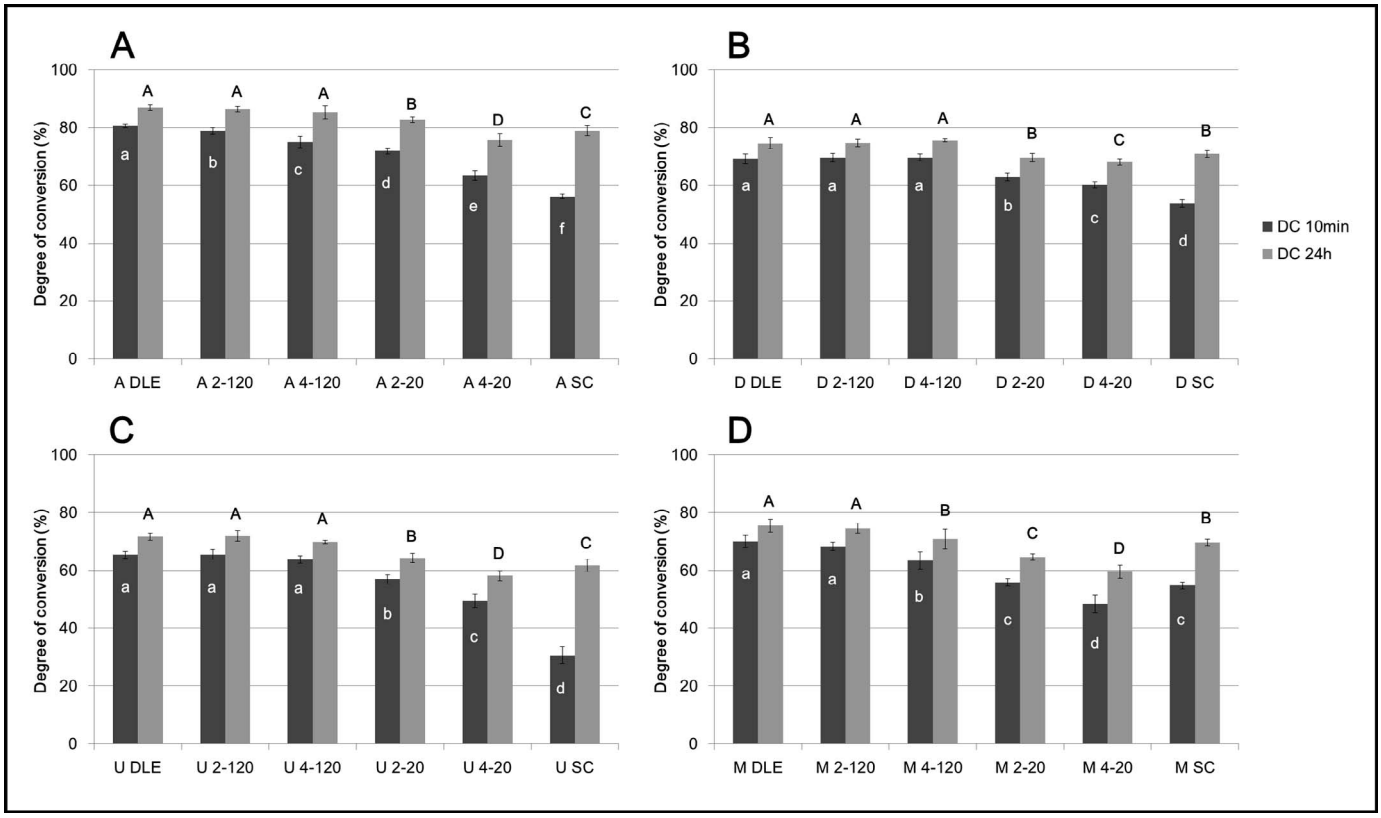


Figure 4. *DC (%) (SD) of Rely-X ARC, Duolink, Rely-X U200, and Maxcem Elite at 10 minutes and 24 hours after polymerization. Significant differences are written by different letters (uppercase letters for DC at 24 hours after polymerization; lowercase letters for DC at 10 minutes after polymerization) according to Student-Newman-Keuls post hoc test (*$p<0.05$*).*

which could be associated with more molecular mobility and large amounts of delayed polymerization as a consequence. Therefore, cases in which insufficient energy could be applied to the cement, it would be better, from the standpoint of DC, to allow the self-cure reaction to progress without additional light exposure.

When considering that the total light energy applied for the DLE group (130,248 mJ/cm$^2$) was about 40 times higher compared with the 4-mm, 120-second group (3072 mJ/cm$^2$) in this study, the similar DC of both groups cannot be explained based on the light energy–based polymerization concept. However, recent studies suggest that DC is not directly proportional to the total amount of energy applied to resin cement.[13-15] It should be noted that the light energy (mJ/cm$^2$) reveals simply the number of photons per unit area, and delivering more photons does not necessarily result in more activated photo-initiators and free radicals, because of rapid saturation of the photo-initiator system and restricted availability of monomers.[13,14] Therefore, the large discrepancy of received light energy between the two groups cannot be the key factor explaining the polymerization of the 4-mm, 120-second group. Musanje and others[15] reported that when sufficiently long light-curing time is provided, adequate polymerization can be achieved even at very low light irradiance (25 mW/cm$^2$). Although "sufficiently long" light-curing time would vary depending on the composition of the resin cement, 120 seconds of light-curing time was effective in three of the four resin cements tested in this study. Therefore, prolonged light-curing time could be an option to improve the polymerization level of dual-cure resin cement.

Rely-X U200 showed low DC in self-cure mode at 10 minutes after mixing, being less than half of that of U DLE group. However, it should be noted that the DC recovered to a level similar to that of U 2-20 ($p>0.05$) at 24 hours after polymerization, showing a large amount of delayed polymerization (Table 4; Figure 4). Such low initial polymerization rate and large delayed polymerization are general features of self-adhesive resin cements, and others have pointed out the presence of acidic functional monomers in the self-adhesive resin cements as the major cause.[16,17] Acidic functional monomers are believed to deactivate free radicals of methacrylate and

produce an acid-base setting reaction, inducing a low rate of co-polymerization and increased delayed polymerization.[17] As shown in this study, self-adhesive resin cements experienced large delayed polymerization that continued up to 24 hours, and probably 7 days according to recent studies.[18] Therefore, sufficient time is needed to properly evaluate the polymerization of self-adhesive resin cements. Also, the self-curing potential of self-adhesive resin cements might be understated if it is evaluated at 10-30 minutes after polymerization, as reported in previous studies.[16,19,20]

On the contrary, Maxcem Elite showed high Rp max in the self-cure mode, which resulted in higher DC compared with M 4-20 group at 10 minutes, and DC was even higher than the M 2-20 group at 24 hours after polymerization ($p<0.05$; Table 4; Figure 4). This high self-curing potential of Maxcem Elite has already been described in previous studies, and higher amounts of self-curing components than other self-adhesive resin cements and use of an amine-free redox initiator system were suggested as possible explanations.[16,19] Although a definitive explanation has not been given due to a lack of information from the manufacturers, such high self-curing potential of Maxcem Elite seems to be suitable to obtain rapid polymerization in cases with gold or zirconia crowns, where there is little chance for light activation of the cement.

An attenuated total reflectance (ATR) accessory has been used with a Fourier transform infrared spectrometer in most studies on the polymerization kinetics of resin composite.[12,16,20-28] However, only the surface layer of the resin specimen (approximately 1-2 μm) can be analyzed in the ATR setting. When considering that the polymerization level can vary depending on the location within a layer of dual-cure resin cement,[29] ATR may not be the best method to assess the overall quality of the resin cement layer. Therefore, in this study, the infrared beam path was designed to penetrate the full thickness of the resin cements. For this purpose, near-IR, with greater penetration, was selected instead of mid-IR, and the specimen was mounted on a holder at a 45° vertical angle to avoid blockage of the IR path by the light-curing tip (Figure 1). In this method, the IR beam penetrates the overlying ceramic block and the resin cement layer. However, it is well known that ceramic does not alter the infrared spectrum, although it does reduce the intensity,[30] and we could not find any disturbance of the IR spectrum by the ceramic interposition compared with the background spectrum.

## CONCLUSIONS

Within the limitations of this study, the following conclusions could be made. For both conventional and self-adhesive dual-cure resin cements, insufficient light exposure (20 seconds of light-curing time) through thick ceramic restoration (4 mm thick) resulted in a DC even lower than that of self-curing alone.

### 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 Conservative Technique for Repairing Class IV Composite Restorations

VC Ruschel • SC Stolf • S Shibata • LN Baratieri

### Clinical Relevance

The repair of the facial surface of a class IV composite restoration with poor coloration is a minimally invasive treatment that allows satisfactory restoration of esthetics and function.


## SUMMARY

**Composite resin may make a restoration noticeable as time passes, on account of its color instability. The repair technique is a minimally invasive treatment for class IV composite resin restorations that show unsatisfactory coloration. Thus, the objective of the present article was to report a clinical case involving a conservative technique used for repairing a class IV composite resin restoration in the left maxillary central incisor and the replacement of a class IV restoration in the right maxillary central incisor.**



*Vanessa Carla Ruschel, DDS, MSD, PHD, Federal University of Santa Catarina, Operative Dentistry, Florianopolis, Brazil

Sheila Cristina Stolf, DDS, MS, PhD, Federal University of Santa Catarina, Operative Dentistry, Florianopolis, Brazil

Shizuma Shibata, DDS, MSD, PhD, Federal University of Santa Catarina, Operative Dentistry, Florianópolis, Brazil

Luiz Narciso Baratieri, DDS, MSD, PhD, Federal University of Santa Catarina, Operative Dentistry, Florianopolis, Brazil

*Corresponding author: Disciplina de Dentística, CCS Campus Universitario Trindade, Florianopolis, SC 88040-900, Brazil; e-mail: ruschelvc@gmail.com


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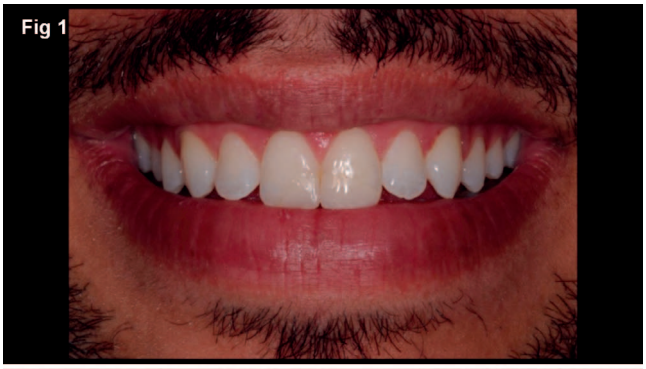
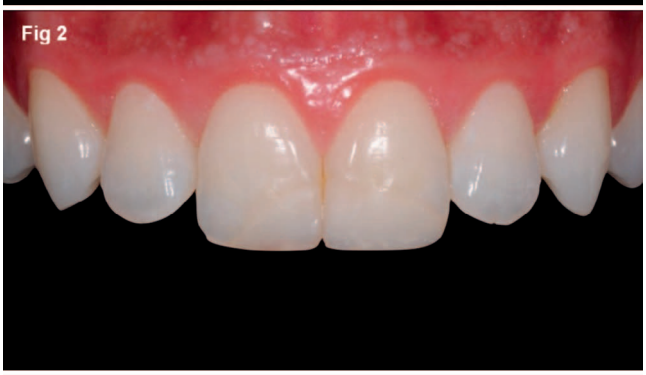
## INTRODUCTION

Restorative treatment with composite resin in a fractured anterior tooth is generally considered successful when there is optical integration between the tooth structure and the restoration. In this context, the concept of natural stratification proposes the combination of optical properties from different resin layers, with the objective of mimicking the natural color and translucency of dental tissues without needing a bevel.[1-4] Nevertheless, there are still difficulties in mimicking remaining tooth restorations in fractured anterior teeth using composite resin stratification. This difficulty occurs because of the variety of currently available shades, chroma, and translucency levels of composite resin. Hence, it is necessary to have a professional, detailed perception of natural optical tooth characteristics and knowledge of the optical behavior of the composite resin used to reproduce the restorations.

In addition, the color instability of composite resin[5-7] can make the restoration noticeable over time. Therefore, when an anterior composite resin restoration is considered clinically unacceptable, a decision should be made whether the best option is to repair or replace the entire restoration. Generally, replacement is the treatment of choice, mainly in situations of color incompatibility between the tooth and the restoration. The repeated replacement of the



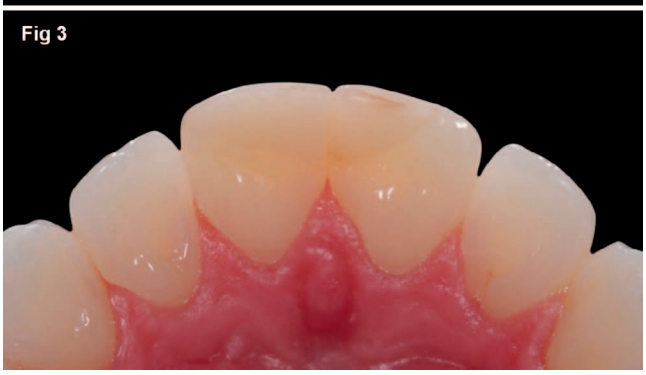




Figure 1. *Initial aspects of the patient's smile. Note the unsatisfactory color of the class IV restorations in the two maxillary central incisors.*
Figure 2. *Intraoral view of maxillary anterior teeth.*
Figure 3. *Palatal view of maxillary anterior teeth. Observe that the palatal surface of the restoration in tooth No. 9 has adequate marginal adaptation, unlike the restoration in tooth No. 8.*

same restoration causes wear of sound tooth structure, leading to the need for more extensive restoration, injuries to the dentin-pulp complex, or fractured tooth remnants.

Repair of a restoration is a conservative treatment, entailing the addition of restorative material after the preparation of the aged restoration.[8] Such a procedure favors restoration longevity and preserves healthy tooth structure.[9,10] There is growing scientific support in the literature for the repair of direct composite resin restorations.[8-16] A clinical study found that composite resin restorations in posterior teeth showed a clinical survival of 10 years after the repair procedure.[16] Opdam and others[10] reported that the repair of composite resin restorations in posterior teeth had a failure rate of only 5.7% four years after clinical evaluation.

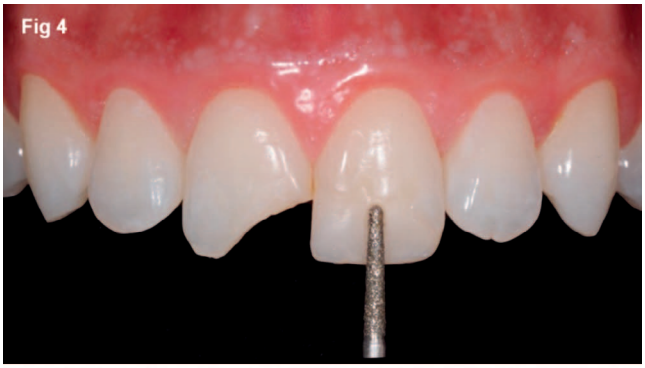
This technique can be an alternative to treating aged class IV composite resin restorations with unsatisfactory color, in which the original color and composition of the materials are unknown. A prerequisite for performing this technique is having optimal marginal fit on the palatal surface. Furthermore, the repair may be performed in cases of a fractured anterior tooth, where the restoration is initially performed with a composite resin corresponding to the basic color of the remaining tooth structure and without stratification. In the following session, the preparation of the facial surface is performed, and the repair with composite resin is stratified to reproduce the opalescent effect and enamel.

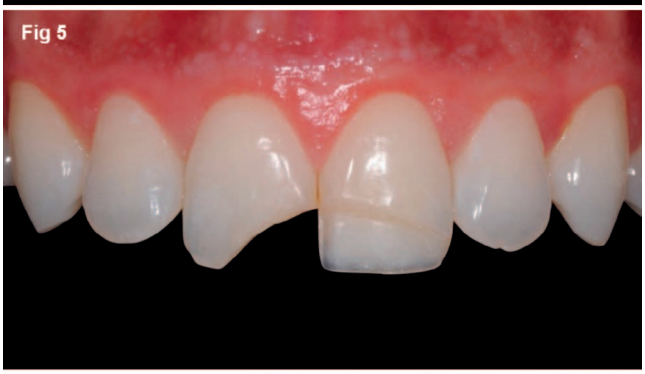
With this in mind, this article reports on a clinical case in which a conservative technique was used for repairing a class IV composite resin restoration in the left maxillary central incisor and a replacement was made of a Class IV restoration in the right maxillary central incisor.

## CASE REPORT

A 22-year-old patient came to the Federal University of Santa Catarina dissatisfied with the color of 2 class IV composite resin restorations, one in the right maxillary central incisor (No. 8) and the other in the left maxillary central incisor (No. 9; Figures 1 and 2). The radiographic examination showed that the patient's teeth had normal periapical and periodontal tissues. During the clinical examination, teeth Nos. 8 and 9 showed pulp vitality. The palatal surface of the restoration in tooth No. 8 was discontinuous, featuring marginal leakage. The restoration on the palatal surface of tooth No. 9 was complete and had adequate marginal adaptation (Figure 3). The replacement of class IV restoration in tooth No. 8 and the restoration repair in tooth No. 9 were proposed to the patient.

Prophylaxis was initiated in the restoration region with a nylon brush and prophylactic paste, followed by color selection. The restoration in tooth No. 8 was removed, and an elastomeric impression of the upper and lower jaw of the patient was taken (Express XT, 3M ESPE, St Paul, MN, USA) to make diagnostic wax-ups of tooth No. 8. A tapered diamond bur (2135 F, KG Sorensen, São Paulo, SP, Brazil) was used to





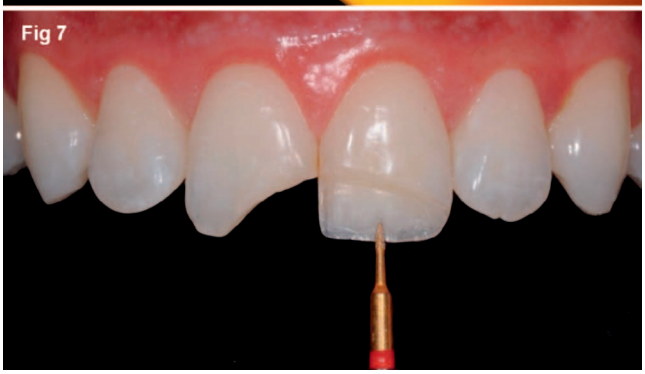



Figure 4. *Restoration in tooth No. 8 removed and preparation of the facial surface of the restoration in tooth No. 9 with a tapered diamond bur.*
Figure 5. *Aspect of the prepared facial surface of the restoration.*
Figure 6. *Lateral view of the silicone guide in position. Note that the space for insertion of the composite resin is 0.7 mm.*
Figure 7. *Design of the dentin mamelons with a sharp-ended diamond bur.*

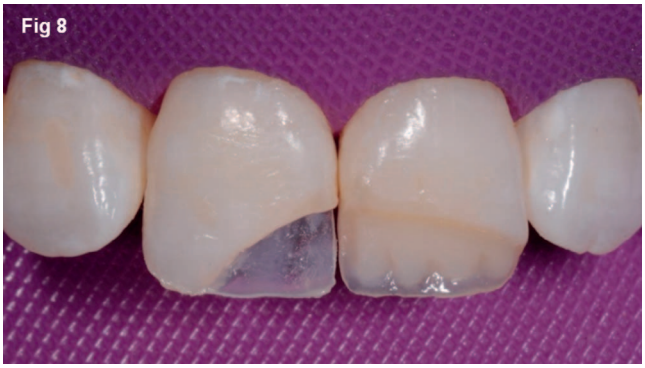
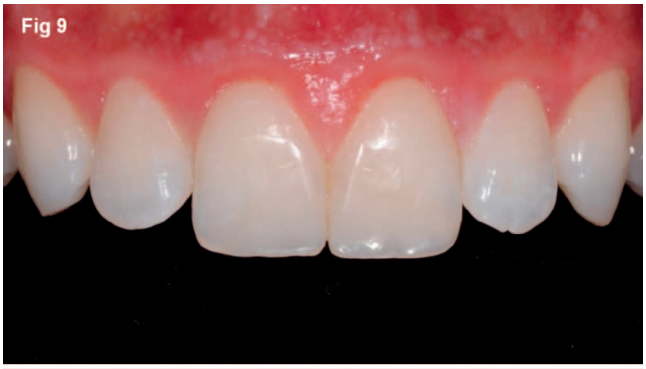


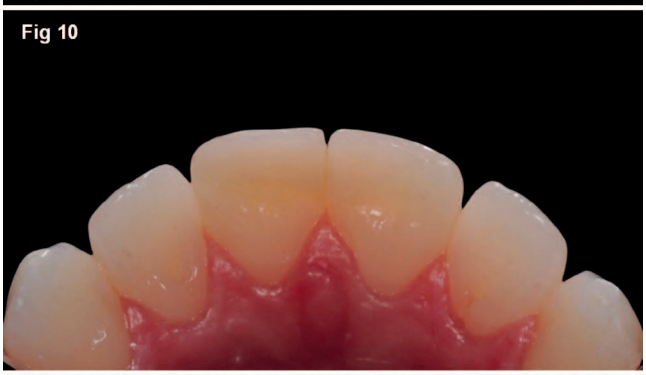

Figure 8. *Intraoral view of the palatal enamel on tooth No. 8 and the preparation of tooth No. 9 after adhesive procedures.*

remove the restoration in tooth No. 8 and to prepare the facial surface of the restoration in tooth No. 9 (Figure 4). The preparation was conducted to provide space for the composite resin stratification, across the entire facial surface of the restoration, respecting the inclination of the mesial and incisal thirds. In addition, the mesial surface was prepared to create space for insertion of proximal artificial enamel (Figures 5 and 6). A design of the mamelons was made in the incisal region, using a sharp-ended diamond bur (2137F, KG Sorensen), to obtain space for the reproduction of an opaque and opalescent halo (Figure 7). Afterward, a mock-up was conducted to verify the correct composite resin color selection and was left for 1 week as a temporary restoration.

During the next session, the operative field was isolated with a rubber dam, the surface of the old resin was sandblasted with aluminum oxide (50 μm, MicroJet Gold Line, Essence Dental VH, Araraquara, São Paulo, Brazil), and the enamel and resin were etched with phosphoric acid at 37% for 30 seconds (PowerEtching, BM4, Palhoça, SC, Brazil, Table 1). Afterward, silane was applied (Monobond Plus, Ivoclar Vivadent, Schaan, Liechtenstein) using a disposable brush (Microbrush, Coltène/Whaledent, Altstatten, Switzerland). The silane was gently air dried for 60 seconds. Adhesive was applied (Scotchbond Universal, 3M ESPE) with a disposable brush (Microbrush, Coltène/Whaledent). Care was taken to ensure adequate solvent evaporation prior to light curing (20 seconds), which was conducted using a light-emitting diode-based light-curing unit (Translux, Hereaus Kulzer, Hanau, Germany, intensity of 800 mW/cm$^2$).

The stratification of the composite resin on tooth No. 8 included high translucent resin EB1 (IPS Empress Direct, Ivoclar Vivadent) to reproduce the palatal enamel, using a silicone guide obtained from



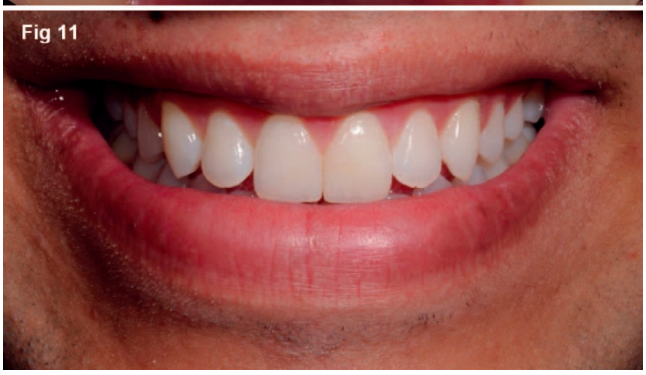




Figure 9. *Aspect of the upper arch after the restoration is finished and polished.*
Figure 10. *Palatal view of the finalized restorations.*
Figure 11. *Patient's smile after restorative treatment.*

the waxing (Figure 8). The incisal halo was reproduced with low translucent resin DB1 (IPS Empress Direct, Ivoclar Vivadent). Low translucent resin DA1 (IPS Empress Direct, Ivoclar Vivadent) was used to reproduce mesial thirds dentin, and low translucent resin DB1 (IPS Empress Direct, Ivoclar Vivadent) was used to reproduce incisal third dentin and dentin mamelons. A translucent resin was used (Trans 30, IPS Empress Direct, Ivoclar Vivadent) to reproduce the opalescent halo, which was lightly applied to the dentin mamelons. The facial enamel was finished with a thin layer of high translucent resin EB1 (IPS Empress Direct, Ivoclar Vivadent). The restoration in tooth No. 9 was performed with

Table 1: *Materials Used For the Patient Treatment*

| | |
|---|---|
| PowerBleaching 16% | BM4, Florianópolis, SC, Brazil |
| PowerEtching 37% | BM4, Florianópolis, SC, Brazil |
| Monobond Plus | Ivoclar Vivadent, Schaan, Liechtenstein |
| Scotchbond Universal | 3M ESPE, St. Paul, MN, USA |
| IPS Empress Direct (composite resins) | Ivoclar Vivadent, Schaan, Liechtenstein |
| Finishing strips | 3M ESPE, St. Paul, MN, USA |
| Sof-Lex (polishing discs) | 3M ESPE, St. Paul, MN, USA |
| Diamond Excel (polishing paste) | FGM, Joinville, SC, Brazil |
| Diamond Flex (felt disc) | FGM, Joinville, SC, Brazil |

low translucent resin DB1 (IPS Empress Direct, Ivoclar Vivadent), in order to reproduce the dentin body, and a translucent resin (Trans 30, IPS Empress Direct, Ivoclar Vivadent), to reproduce an opalescent halo. The facial enamel was reproduced with a thin layer of high translucent resin EB1 (IPS Empress Direct, Ivoclar Vivadent). Coarse removal of excess material from both restorations was done with a No. 12 sharp curved blade (Feather, Osaka, Japan).

In the next session, the finishing and polishing procedure was initiated with abrasive strips (3M ESPE) on the proximal surfaces. The facial surfaces were finished with sequential polishing discs of decreasing grit size (Sof-Lex Pop On, 3M ESPE). A carbide bur (FG 7664F, KG Sorensen) was used to remove excess resin from the preparation margin and to reproduce vertical texture. A felt disc (Diamond Felt Disc, FGM, Joinville, SC, Brazil) with diamond polishing paste (Diamond Excel, FGM; Figures 9-11) was used to perform the final polishing.

## Potential Problems

When assessing the three color dimensions, value is what most influences natural tooth appearance, followed by chroma and shade.[17-19] Greater translucency of artificial dentin can decrease the restoration value and make the restoration more noticeable than an error in shade selection.[4] In the case presented, the facial preparation depth of 0.7 mm on tooth No. 9 provided adequate space for insertion of dentin resins, opalescent effect, and facial enamel. This depth was necessary since the color of the aged composite resin was incompatible with the remaining tooth structure. Thus, the thickness of the artificial dentin provided adequate opacity without interfering with the restoration value.

In the clinical cases in which the professional restores a fractured tooth in its basic color and carries out the repair in another session, the reduction of the facial surface can be slight without requiring a chamfer at the adhesive interface. It is important to highlight that the thickness of the high translucent resin, corresponding to the enamel, must be smaller compared with the natural enamel in order to prevent a decrease in restoration value.[20] The repair technique for the class IV composite resin restoration may also be a conservative alternative in cases of permanent restorations that remain noticeable after treatment, due to errors in color selection. Once the facial surface has been repaired, the color can then be corrected, resulting in reduced clinical time and preservation of sound dental structure in comparison with replacement.

It is important to note that a restorative mock-up should be performed whenever possible to ensure greater outcome predictability, since the composite resin color changes during polymerization, as well as 24 and 48 hours after polymerization.[5]

With regard to the surface treatment of the aged resin, studies report that the most favorable results were obtained with the roughening and application of a silane agent. The roughening procedure can be performed using diamond burs, sandblasting with aluminum oxide, or applying a tribochemical silica coating. This creates irregularities on the surface of the aged resin, which increases the contact area for micromechanical retention of the adhesive and for chemical adhesion with the new composite resin.[8,21-23] In the case presented, the surface treatment was sandblasted with aluminum oxide (50 μm), enamel and resin were acid-etched, and silane and adhesive were applied.[24]

In this clinical case, the replacement of the class IV restoration in tooth No. 8 was necessary because of the presence of a defect in the interface between the palatal surface and the restoration. This condition contraindicates facial surface repair because of possible early restoration failure. The restoration in tooth No. 9 could be repaired because of the optimal marginal adaptation of the palatal surface. However, the final esthetic result between the repaired restoration and the replaced restoration was similar, and both were satisfactory.

### Advantages

- Greater preservation of sound tooth structure
- Decreased chance of pulp injury
- Increased restoration longevity
- Does not require anesthesia
- Less clinical procedure time
- Lower treatment cost for the patient
- Good acceptability by the patient

### Limitations

- Color difference between the restoration and the tooth, because of the difficulty in color selection and layering of the composite resin

## CONCLUSIONS

- The esthetic result of the new technique for repairing the facial surface of a class IV composite resin restoration is similar to that obtained by replacing the restoration.
- The repair of a class IV composite resin restoration with unsatisfactory color is a viable alternative treatment that preserves sound tooth structure, restoring function and esthetics satisfactorily.

#### Conflict of Interest

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

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# Microcomputed Tomography Evaluation of Polymerization Shrinkage of Class I Flowable Resin Composite Restorations

CS Sampaio • K-J Chiu • E Farrokhmanesh • M Janal • RM Puppin-Rontani
M Giannini • EA Bonfante • PG Coelho • R Hirata

### Clinical Relevance

Since polymerization shrinkage in a 2.5 mm deep class I cavity was, in general, not different in a self-adhesive, a bulk-fill, and a conventional flowable resin composite, endeavors to simplify clinical procedures and to reduce steps and treatment times are promising.

## SUMMARY

**The present study aimed to characterize the pattern and volume of polymerization shrinkage of flowable resin composites, including one conventional, two bulk fill, and one self-adhesive. Standardized class I preparations (2.5 mm depth × 4 mm length × 4 mm wide) were performed in 24 caries-free human third molars that were randomly divided in four groups, according to the resin composite and adhesive system used: group 1 = Permaflo + Peak Universal Bond (PP); group 2 = Filtek**

*Camila S Sampaio, DDS, MD, PhD student, Department of Restorative Dentistry, Piracicaba Dental School - UNICAMP, Piracicaba, Brazil; Department of Biomaterials and Biomimetics, New York University College of Dentistry. New York, NY, USA; Department of Biomaterials, Universidad de Los Andes, Chile

Kai-Jen Chiu, DDS, Department of Biomaterials and Biomimetics, New York University College of Dentistry, New York, NY, USA

Ehsan Farrokhmanesh, DDS, Department of Biomaterials and Biomimetics, New York University College of Dentistry, New York, NY, USA

Malvin Janal, DDS, MS, PhD, Department of Epidemiology and Health Promotion, New York University College of Dentistry, New York, NY, USA

Regina M Puppin-Rontani, DDS, MS, PhD, Department of Pediatric Dentistry, Piracicaba Dental School - UNICAMP, Piracicaba, Brazil

Marcelo Giannini, DDS, MS, PhD, Department of Restorative Dentistry, Piracicaba Dental School - UNICAMP, Piracicaba, Brazil

Estavam A Bonfante, DDS, MS, PhD, Department of Prosthodontics, Bauru College of Dentistry – University of São Paulo, Bauru, Brazil

Paulo G Coelho, DDS, MS, PhD, Department of Biomaterials and Biomimetics, New York University College of Dentistry, New York, NY, USA

Ronaldo Hirata, DDS, MS, PhD, Department of Biomaterials and Biomimetics, New York University College of Dentistry, New York, NY, USA

*Corresponding author: 433 First Avenue, New York, NY 10010; e-mail: camisobral@hotmail.com

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**Bulk Fill + Scotchbond Universal (FS); group 3 = Surefil SDR + XP Bond (SX); and group 4 = Vertise flow self-adhering (VE) (n=6). Each tooth was scanned three times using a micro-computed tomography (μCT) apparatus. The first scan was done after the cavity preparation, the second after cavity filling with the flowable resin composite before curing, and the third after it was cured. The μCT images were imported into three-dimensional rendering software, and volumetric polymerization shrinkage percentage was calculated for each sample. Data were submitted to one-way analysis of variance and post hoc comparisons. No significant difference was observed among PP, FS, and VE. SX bulk fill resin composite presented the lowest values of volumetric shrinkage. Shrinkage was mostly observed along the occlusal surface and part of the pulpal floor. In conclusion, polymerization shrinkage outcomes in a 2.5-mm deep class I cavity were material dependent, although most materials did not differ. The location of shrinkage was mainly at the occlusal surface.**

## INTRODUCTION

Polymerization shrinkage is an unavoidable by-product of resin composite restorations, mainly influenced by material formulation/properties, adhesion, flow on the free surface, and polymerization kinetics.[1,2] Shrinkage due to polymerization can result in postoperative sensitivity, marginal gaps and leakage, restoration debonding, tooth fractures, and resin composite fractures.[3]

Incremental resin composite techniques have been suggested to compensate for polymerization shrinkage by reducing the stresses developed within the tooth-restoration system.[4] Although an incremental technique may be important for polymerization stress control and to minimize light attenuation, its disadvantages are the possibility of void entrapment between layers and the extended time required to place restorations.[5]

Developments have been attempted in the field of adhesive systems, and the most recent efforts in resin composite technology have focused on reducing technique sensitivity and chair time while enhancing clinical longevity. Results of such endeavors have led to improvements in bulk fill and self-adhesive resin composites, which are both commercially available as flowable resin composites. In essence, flowable materials are low viscosity resin composites with less filler load or a greater portion of diluent monomers than regular resin composite.[2,6] An increasing number of flowable resin composites have been released in the market, with a proportional number of investigations centered on bulk filling and self-adhering resin composites.[1,7-11]

Bulk fill resin composites are intended for placement in increments up to 4 mm in thickness with reduced volumetric polymerization shrinkage[1] and stress levels.[12] Resin composite bulk application simplifies cavity filling, substantially reducing chair time and requiring fewer clinical steps.[10] According to the manufacturers, matrix and initiator chemistry, as well as filler technology, have been optimized in order to obtain specific properties required for the aforementioned applications.

Another new approach is the self-adhesive resin composite. Data concerning this resin composite are limited,[7,9,13] and the lack of volumetric shrinkage reports warrants investigation. According to manufacturers, self-adhesive resin composite simplifies the restorative procedure by incorporating an all-in-one bonding system into a flowable resin composite, eliminating the need for an additional adhesive application step, saving time and potentially minimizing handling errors.

Flowable resin composites have been regarded as materials that provide polymerization stress relief and improved adaptation.[14,15] Although studies show that this type of resin composite acts as a stress breaker,[2,15] some investigations indicate that volumetric polymerization shrinkage is higher than in conventional resin composites.[15,16] Flowable bulk fill resin composites have also been regarded as presenting less volumetric shrinkage than conventional flowable ones.[1,12]

Different methods have been described to evaluate volumetric shrinkage and/or gap formation, most of them based on destructive methods.[14,17-19] Micro-computed tomography (μCT) has demonstrated its efficacy in the assessment and visualization of resin composites regarding their polymerization shrinkage vectors,[20] volumetric shrinkage,[1] and leakage after curing,[21] among others. The use of μCT allows for nondestructive two-dimensional (2D) and three-dimensional (3D) imaging, and the possibility of analyzing the material behavior inside a given geometric configuration, such as a tooth cavity.[1,22]

This study sought to characterize the volumetric polymerization shrinkage of one conventional flowable resin composite, two bulk fill flowable resin composites, and one self-adhesive flowable resin

Table 1: *Composition and Composites Filler Loading, Manufacturers, and Batch Numbers of the Materials Studied*

| Material/ Manufacturer | Batch No. | Composition |
|---|---|---|
| PermaFlo Flowable / Ultradent Products, South Jordan, UT, USA | N114 | Bisphenol-A-glycidyl methacrylate, triethylene glycol dimethacrylate, sodium monofluorophosphate; zirconium filler. Filler loading: 68 wt% |
| Filtek Bulk fill Flowable / 3M ESPE, St Paul, MN, USA | 1506500564 | Silane-treated ceramics, diurethane dimethacrylate (UDMA), substituted dimethacrylate, bisphenol A polyethylene glycol dietherdimethacrylate (BISEMA-6), ytterbium fluoride (YbF3), bisphenol A diglycidyl ether dimethacrylate (BISGMA), benzotriazol, triethylene glycol dimethacrylate (TEGDMA), ethyl 4-dimethyl aminobenzoate. Filler loading: 64.5 wt% |
| Surefil SDR Flow / Dentsply Caulk, Milford, DE, USA | 110527 | Barium-alumino-fluoro-borosilicate glass, strontium alumino-fluoro-silicate glass, modified urethane dimethacrylate resin, ethoxylated bisphenol A dimethacrylate (EBPADMA), triethylene glycol dimethacrylate (TEGDMA), camphorquinone (CQ), photo-accelerator, butylated hydroxyl toluene; ultraviolet stabilizer; titanium dioxide; iron oxide pigments, fluorescing agent. Filler loading: 68 wt% |
| Vertise Flow Self-adhering flowable / Kerr Corp, Orange, CA, USA | 5353402 | Glycerol phosphate dimethacrylate, prepolymerized filler, 1-μ barium glass filler, nano-sized colloidal silica, nano-sized ytterbium fluoride Filler loading: 70 wt% |
| Peak Universal Bond / Ultradent Products | H024 | 2-hydroxyethyl methacrylate, methacrylic acid dehydrated alcohol, chlorhexidine diacetate |
| Scotchbond Universal Adhesive / 3M ESPE | 488169 | 10-methacryloyloxydecyl dihydrogen phosphate, dimethacrylate monomers, 2-hydroxyethyl methacrylate, polyalkenoic acid copolymer, silane, photoinitiators, filler, ethanol, water |
| XP Bond Universal Total-Etch Adhesive / Dentsply Caulk | 1104001218 | Carboxylic acid modified dimethacrylate, phosphoric acid modified acrylate resin, urethane dimethacrylate, triethylene glycol dimethacrylate, 2-hydroxy ethyl methacrylate, butylated benzenediol, ethyl-4-dimethyl aminobenzoate, camphorquinone, functionalized amorphous silica, t-butanol |

composite in standardized class I restorations using a μCT tomography technique.

## METHODS AND MATERIALS

Twenty-four sound, freshly extracted human third molars were obtained according to protocols approved by the NYU Medical School Institutional Review Board. Teeth were cleaned and kept in distilled water at 5°C until their use. A standardized class I preparation with a cavity design of 2.5 mm depth × 4 mm length × 4 mm width was performed in each tooth with a diamond bur (AD20 Occlusal Reduction Bur, Code 845-022, Strauss, Westport, CT, USA) that presents a standardized active head size and a vertical stop to deliver consistent cavity preparation depth. The bur was replaced after every five cavity preparations. Final cavity preparation was then checked for dimensional accuracy with a digital caliper. All teeth were maintained in distilled water at room temperature (25°C) before and after preparation procedures.

Study materials, manufacturers, and batch numbers are provided in Table 1. Teeth were cleaned with a pumice slurry and randomly divided in four groups (n=6 each): group 1 = PP (Permaflo + Peak Universal Bond); group 2 = FS (Filtek Bulk Fill + Scotchbond Universal); group 3 = SX, (Surefil SDR + XP Bond); and group 4 = VE (Vertise flow self-adhering without separate bonding agent).

### μCT Evaluation

Each tooth was scanned three times using a μCT apparatus (mCT40, Scanco Medical, AG, Basserdorf, Switzerland). The apparatus was calibrated using a phantom standard at 70 Kvp/BH 200 mgHA/cm. The

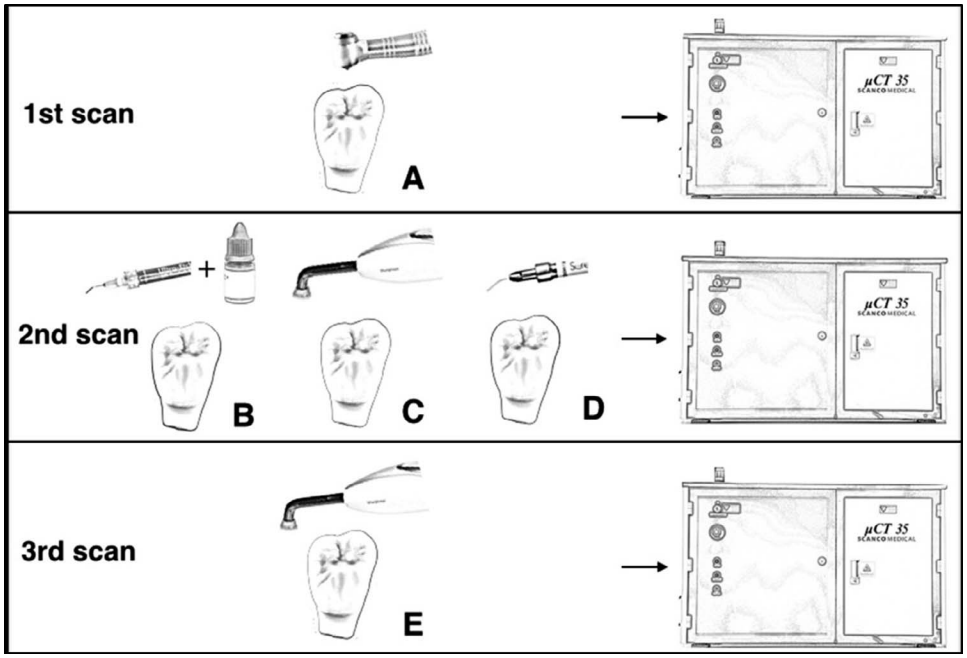


Figure 1. *Representative images describing the scanning and sample preparation workflow. (A): Cavity preparation. (B): Cavity acid-etching and bonding (when indicated by the manufacturer). (C): Light-curing. (D): Cavity filling with the flowable composite. (E): Light curing. The first micro-CT scan was taken after step A, the second after step D, and the third after E.*

operating condition for the μCT device used was 70 kVp–114 microamperes with a resolution giving 16 μm/slice. The average of the total number of slices was approximately 250, and the average scan time was 28 minutes.

Figure 1 shows a schematic of the workflow used for data collection. The first μCT scan was performed after cavity preparation for all teeth. Then, all teeth (except in the VE group) were etched with phosphoric-acid (Ultra-etch 35%, Ultradent Products) for 30 seconds in enamel and 15 seconds in dentin, followed by rinsing for 20 seconds and excess water removal with a thin absorbent paper. Restorative and bonding procedures were performed according to manufacturer's recommendations. Since VE is a self-adhering composite, no bonding agent was used.

After the first scan, cavities were filled in bulk using their assigned resin composites, left uncured, and immediately placed inside the μCT holder. To prevent unwanted curing, the μCT holder was first covered with a dark plastic, avoiding contact with any light source, and then placed inside the μCT apparatus for the second scan and volume quantification. Samples stayed in a dark environment until light curing. Subsequently, resin composites were light cured for 40 seconds with a Polywave light-curing unit (Bluephase 20i, Ivoclar Vivadent, Schaan, Liechtenstein) and inserted back into the holder for the third scan.

The μCT data were imported into a workstation and evaluated with Amira software (version 5.5.2, VSG, Burlington, MA). Superimposition of all three scan images was performed by the software, perfectly aligning them.[1] Due to the similar radiodensity of the tooth and the resin composite, this procedure was performed in order to avoid scattering and possible noise formation.[1] Registered μCT data of uncured and cured samples were subtracted from the cavity data, isolating the restoration (cavity filled with uncured resin composite minus cavity preparation, and cavity filled with cured resin composite minus cavity preparation), avoiding any scattering interference in the measurements. This procedure enabled both uncured and cured resin composite volumes to be isolated and quantified, allowing the volumetric polymerization shrinkage to be calculated as a percentage. Afterward, another subtraction was conducted for uncured minus the cured for imaging of the resin composite's shrinkage.

### Statistical Analysis

Data were analyzed using a one-way analysis of variance. Post hoc comparisons were carried out using 95% confidence intervals based on the pooled estimate of residual variability.

To determine sample size, we started with previous data suggesting a standard deviation (SD) of 1% shrinkage in each treatment group and posited a clinically important reduction of at least 1.5% in absolute terms, or 30% relative to a mean level of 5% shrinkage. Six samples per group provided 80% power to detect a reduction of 1.5 SD (or 1.5%

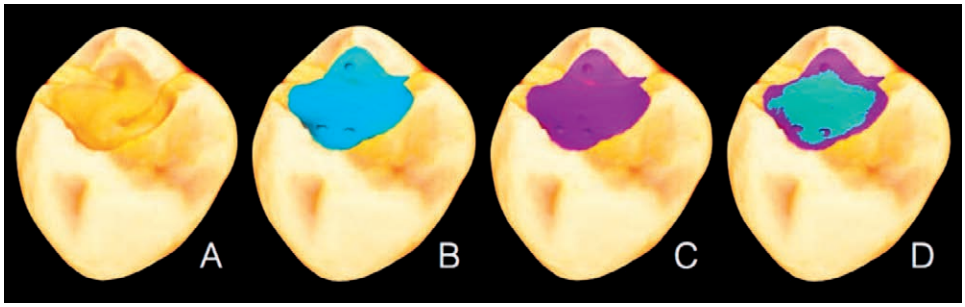


Figure 2. *Diagram of the cavity in different stages. (A): Preparation. (B): Uncured composite. (C): Cured composite. (D): Shrinkage.*

absolute) between any two groups in an independent samples $t$ test at $p<0.05$ (one-tailed, because we want to detect reduced shrinkage) (G*Power, v3).[1]

## RESULTS

Figure 2 shows representative images obtained from the μCT scans. The mean (95% confidence level) of the volumetric shrinkage of groups is presented in Table 2. Group 3 (SX), a bulk fill resin composite, showed the lowest shrinkage values and was significantly different from all other groups. Group 1 (PP), a conventional resin composite, Group 2 (FS), a bulk fill resin composite, and Group 4 (VE), a self-adhesive resin composite, showed higher shrinkage values, but there were no statistical differences between those groups ($p<0.05$).

Qualitative 3D reconstructions depicted voids within most of the resin composite fillings (Figure 3). However, these were present in qualitatively higher levels for the FS and VE groups relative to the other resin composites. For PP, voids were seldom observed. The SX samples showed voids in half of the samples and their absence in the other half.

For all groups, shrinkage was mostly seen along the occlusal surface and part of the pulpal floor of the class I restorations. For the PP group, samples showed shrinkage only on the occlusal surface, and there was no gap formation on pulpal and lateral walls. For the FS group, most samples presented shrinkage on the occlusal surface and pulpal floor. For the SX group, half of the samples presented shrinkage only on the occlusal surface, and half mainly on the occlusal surface and less so on the pulpal floor. For the VE group, most samples presented only occlusal shrinkage. Gaps and shrinkage were, in general, less frequently observed on the mesiodistal and buccolingual walls of restorations.

## DISCUSSION

The present study showed that volumetric polymerization shrinkage varied among the different types of flowable resin composite restorative materials. No conventional resin composites (ie, methacrylate based) were included in this study as controls because previous research from our group showed increased shrinkage for some conventional composites relative to bulk fill resin composites.[1] With the aim of understanding polymerization shrinkage values and patterns of currently available flowable resin composites, this study showed a smaller percentage of volumetric polymerization shrinkage for one bulk fill (SX), whereas no differences between the other bulk fill (FS), the conventional (PP), and a self-adhesive (VE) flowable were detected.

A recent study[10] reported similar results, when volumetric shrinkage of Surefil SDR was compared to a conventional flowable resin composite. However, in contrast to our results, significantly different polymerization shrinkage between groups SX and FS was not found. Other investigations[16,23] have also shown that Surefil SDR resin composite has lower shrinkage levels compared to other bulk fill and

Table 2: *Volumetric Polymerization Shrinkage (n=6) of Each Flowable Composite*

| Groups and Composites | Shrinkage[a] % (SD) |
|---|---|
| Group 1: Permaflo + Peak Universal (PP) | 4.81 (0.42) a |
| Group 2: Filtek Bulk Fill + Scotchbond Universal (FS) | 5.49 (1.84) a |
| Group 3: Surefil SDR + XP Bond (SX) | 3.31 (0.33) b |
| Group 4: Vertise (VE) | 5.79 (1.13) a |

[a] *Means followed by different letters differ from each other in the same column (p≤0.05).*

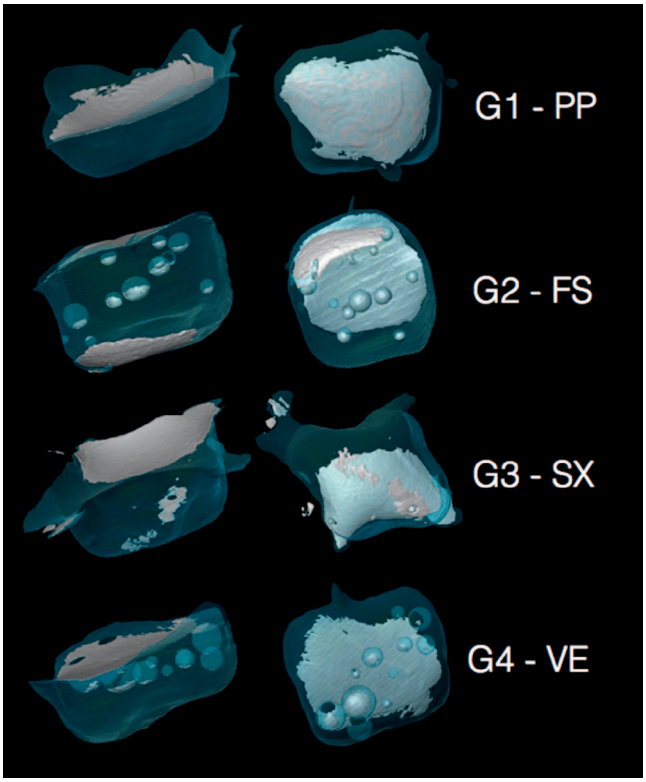


Figure 3. *3D renderings of the groups in distal (left) and pulpal (right) views.*

conventional flowable materials, which is in agreement with our findings.

Extensive efforts have been put toward the development of low-shrinkage dental restorative materials. Besides changes in filler amount, shape, or surface treatment, changes in monomer structure or chemistry and modification of polymerization dynamics have been regarded as promising approaches.[12] The lower values of volumetric shrinkage observed for the SX group may be explained by the monomer composition of the resin composite, a modified diurethanedimethacrylate (UDMA; 849 g/mol) that has a higher molecular weight than other monomers such as bisphenol a diglycidyl ether dimethacrylate (Bis-GMA; 512 g/mol), bisphenol A polyethylene glycol dietherdimethacrylate (Bis-EMA; 496 g/mol), ethoxylated bisphenol A dimethacrylate (EPBADMA; 452 g/mol), and conventional UDMA (470 g/mol). A higher molecular weight would decrease the number of reactive sites per unit volume, conversely reducing shrinkage.[2,24]

To the best of our knowledge, no bulk fill resin composites have been compared to self-adhesive resin composites when it comes to volumetric shrinkage in a restoration cavity. Although not significantly different from some bulk fill groups, the VE group presented higher numeric volumetric shrinkage compared to three materials studied. Earlier studies have shown that the dentin bond strength of VE is lower when compared to flowable resin composites used along with self-etching or total-etch adhesives.[8,9] The lower bond strength of VE to dentin can also explain the higher mean volumetric polymerization shrinkage values reported here, as this phenomenon may be related to the debonding of the resin composite from the cavity walls of the teeth, allowing higher volumetric shrinkage.[8,9]

Even though FS is a bulk fill material and should theoretically present lower degrees of polymerization shrinkage, our results showed that it did not show significantly different values compared to the conventional flowable resin composite group (PP). This finding may be related to the percentage of fillers contained in the materials, as Permaflo Flowable resin composite contains 68% by weight of fillers and Filtek Bulk fill flowable resin composite contains 64.5%. Previous literature has pointed out that resin composites with a lower percentage of fillers may have a higher shrinkage than those with higher percentages,[2,25] although this finding did not corroborate our study when VE was analyzed. The self-adhesive flowable composite presents 70% of inorganic fillers but still showed the highest polymerization shrinkage percentage among the groups.

Qualitatively, the shrinkage patterns were similar for all groups, which presented higher shrinkage on the occlusal surface relative to other surfaces of the cavity. Although pulpal gaps were frequently seen within the groups, the shrinkage in the free occlusal surface was more prominent, a finding that is in agreement with previous literature that depicts shrinkage occurring in unbonded free surfaces.[1,26] Based on our results, some of the materials, when used for bulk filling of cavities, generate gap formation on the pulpal floor, potentially indicating a technical limitation.

Another clinical concern regarding polymerization shrinkage is the resin composite's degree of conversion. The rate of conversion is a significant factor affecting the generation of contraction stress in dental composites. Previous studies suggested that conversion and its resultant volumetric shrinkage are the most important factors affecting the development of contraction stress in dental composites,[27] and the volumetric shrinkage of composites has been shown to be proportional to its degree of conversion.[28,29] The magnitude of volumetric shrinkage

experienced by a resin composite is determined by its filler volume fraction and the composition and degree of conversion of the resin matrix.[2] Therefore, it is suggested that additional in vitro and clinical studies should be conducted to determine degree of polymerization of such studied materials and to correlate it to their volumetric polymerization shrinkage and polymerization shrinkage stress.

## CONCLUSION

The bulk fill resin composite Surefil SDR showed less volumetric polymerization shrinkage compared with the other groups, where no difference was detected among the self-adhesive Vertise flow, the bulk-fill Filtek Flowable, and the conventional Permaflo Flowable resin composites. Results from this study suggest that bulk fill and self-adhesive flowable resin composites present at least comparable volumetric polymerization shrinkage performance relative to a conventional flowable resin composite in a 2.5 mm depth cavity, which is an important finding related to simplification of steps in clinical practice.

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### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of NYU Medical School Institutional Review Board.

### Conflict of Interest

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

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# The Silorane-based Resin Composites: A Review

GA Maghaireh • NA Taha • H Alzraikat



### Clinical Relevance

Silorane-based resin composites (SBRC) have been found to exhibit a decrease in polymerization shrinkage stresses and properties at least as good as that associated with methacrylate-based resin composites. This review summarizes the current literature on the SBRC to help the dental practitioner trying to make evidence-based decisions.


## SUMMARY

**This article aims to review the research done on the silorane-based resin composites (SBRC) regarding polymerization shrinkage and contraction stresses and their ability to improve the shortcomings of the methacrylate-based resin composites (MRBC). Special attention is given to their physical and mechanical properties, bond strength, marginal adaptation, and cusp deflection. The clinical significance of this material is critically appraised with a focus on the ability of SBRC to strengthen the tooth structure as a direct restorative material. A search of English peer-reviewed dental literature (2003-2015) from PubMed and MEDLINE databases was conducted with the terms "low shrinkage" and "silorane composites." The list was screened, and 70 articles that were relevant to the objectives of this work were included.**



*Ghada A Maghaireh, BDS, MS, ABOD, Department of Conservative Dentistry, Jordan University of Science and Technology, Irbid, Jordan

Nessrin A Taha, BDS, DClinDent Endo, MFDS, FRACDS, FRACDS (Endo), Department of Conservative Dentistry, Jordan University of Science and Technology, Irbid, Jordan

Hanan Alzraikat, PhD, Department of Conservative Dentistry, Jordan University of Science and Technology, Irbid, Jordan

*Corresponding author: PO Box 3030, Irbid, 22110, Jordan; e-mail: gmaghair@just.edu.jo


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## INTRODUCTION

Current resin-based composite (RBC) restorations have become an essential part of everyday dental practice. This is due to the increase in patients' demand for esthetic restorations, along with greater emphasis on the preservation of sound tooth structure and the improvement of adhesive dentistry as a result of bonding mechanisms that can reinforce the remaining tooth structure. Resin-based composites have also been proven to have enhanced mechanical properties and abrasion resistance that have continued to improve since their introduction as dental restorations. This allows expanded use for posterior teeth with good longevity.

Polymerization shrinkage and the associated stress transmitted to the adhesive bond and the remaining tooth structure are the most important and clinically relevant problems associated with the methacrylate-based resin composites (MBRC).[1] Depending on the bond strength of the interface between the RBC and tooth structure, these shrinkage stresses can lead to clinical consequences, such as marginal gap formation and leakage, debonding at the restoration/tooth interface, cusp deflection,

and microfractures of the tooth structure.[2,3] Consequently, several clinical strategies have been developed in order to overcome the problems associated with polymerization shrinkage, including the placement of low-modulus liners or bases and incremental placement of the RBC, in addition to modifications in light application, which is the so-called "soft-start" method.[4-6] However, conclusive data on the efficacy of these techniques and their relationship to increased technique sensitivity.

Consequently, research has focused on advances in material formulation that may improve the shortcomings of the MBRC, including reducing monomer percentage through the addition of inorganic fillers, utilizing multiple-sized filler particles, and utilizing prepolymerized filler particles.[7] However, changes in filler configuration offered only limited reduction in polymerization shrinkage.[8] Furthermore, researchers have begun to examine the ways in which the matrix and monomer chemistry can be modified. A novel monomer technology with unique polymerization characteristics to minimize polymerization shrinkage has been developed, the silorane-based resin composites (SBRC). The silorane matrix is formed by the cationic ring-opening polymerization of the silorane monomer. This is in contrast to the linear chain reaction of methacrylates, which is cross-linked via radicals. This change in composite chemistry and polymerization reaction resulted in a significant reduction in the polymerization shrinkage to a level less than 1.0% of the total volumetric shrinkage.[9] This is in comparison to the 2.6% to 7.1% associated with MBRC.[10] The reduction in polymerization shrinkage of SBRC often results in a significant decrease in polymerization shrinkage stresses,[11,12] lower microleakage scores,[13] improved marginal adaptation,[14] and reduced cusp deflection.[15] These benefits occur while maintaining comparable mechanical properties to those of MBRC.[16]

The objective of the present work is to review both experimental and clinical studies that were done on the SBRC in terms of polymerization shrinkage and contraction stresses, physical and mechanical properties, bond strength, cusp deflection, and fracture strength of teeth restored with SBRC. A search of English peer-reviewed dental literature (2003-2015) from PubMed and MEDLINE databases was conducted with the terms "low shrinkage composites" and "silorane composites." The list was screened, and 70 articles that were relevant to the objectives of this work were included.

## POLYMERIZATION SHRINKAGE AND CONTRACTION STRESSES

Modern MBRC exhibit excellent esthetics and physical properties. However, their major drawbacks are polymerization shrinkage and its related polymerization stress. Most MBRC undergo contraction, which ranges from 2.6% to 7.1% as a result of the polymerization reaction.[10]

The use of alternative chemistries has been at the forefront of research and development for dental RBC for many years. The silorane molecule in SBRC presents a siloxane core with four oxirane (oxygen-containing) rings attached. Thus, these rings are opened during polymerization to bond to other monomers. The hydrophobic properties of the material are attributed to the siloxane molecules. Therefore, exogenous discoloration and water absorption are minimized. The oxirane rings are responsible for the physical properties and the reduced polymerization shrinkage. Furthermore, the opening of the oxirane ring causes a volumetric expansion that may compensate, to some degree, for the shrinkage resulting from molecular bonding.[17]

Weinmann and others[9] found that the volumetric shrinkage of SBRC is less than 1% of the volume. This finding concurs with those of other studies,[11,12,18] which found that the SBRC exhibited significantly lower polymerization shrinkage and polymerization stress than did the MBRC. However, polymerization stress is not determined by volumetric shrinkage alone but also by a number of other factors, including properties that are intrinsic to the material, such as the modulus of elasticity, the degree of cure, the coefficient of thermal expansion, and the silanization characteristics at the resin-filler interface.[19] This is in addition to clinical factors, such as the rate of cure and polymerization kinetics, the cavity configuration factor, and the compliance of the remaining tooth structure. In this respect, recent studies have evaluated these new materials in terms of polymerization stress, as well as other aspects involved in its development, in addition to shrinkage.

Boaro and others[17] compared a SBRC to a MBRC in terms of polymerization stress, volumetric shrinkage, elastic modulus, and reaction rate. The SBRC had high polymerization stress value (4.3 MPa) in spite of the low volumetric shrinkage (1.4 %). Thus, the authors[17] speculated that the high initial flexural modulus shown by the SBRC may explain its high polymerization stress value, in spite of the low volumetric shrinkage. This observation was also found in a study by Marchesi and others,[20] who

reported that Filtek Silorane LS resulted in higher stresses (1.3 MPa) when compared to Tetric EvoCeram (0.95 MPa), which is a MBRC with lower elastic modulus. In addition, a low degree of conversion was observed in this study for the Filtek Silorane LS. This result has been hypothesized in other studies to result in slower polymerization that allows enough time for stress relaxation. However, Marcchesi and others[20] claimed that the cationic polymerization in the SBRC continues for extended periods of time at a slower rate. This may also lead to increased stresses over time.

Other studies[11,21] reported that the polymerization kinetics of SBRC are comparable to those of the MBRC. Moreover, Yamasaki and others[21] found that despite the different reaction mechanisms, P90 (SBRC) showed similar behavior to the MBRC regarding the kinetics of polymerization. However, it had lower polymerization stresses (2.6 MPa); this was explained by the ring-opening polymerization mechanism of silorane. In view of the above, the complexity of the interactions between factors that determine polymerization shrinkage and stress development should be highlighted more by future research. This is in a bid to quantify the polymerization shrinkage associated with the SBRC.

## BOND STRENGTH

Most of the shrinkage stresses generated during polymerization of MBRC are generated as a result of the entire composite material being strained during the polymerization reaction through its adherence to the cavity walls. Contraction stresses will appear as tensile forces at the adhesive-tooth interface because the composite will attempt to shrink toward the bonded surface. Nevertheless, it will be constrained by the rest of the composite mass, which is also bonded to the opposite side. To relieve those stresses, the polymer matrix will attempt to flow to any free surface. In addition, localized interfacial failures or weaker bonded areas will provide sites for stress relief. If the local contraction stresses exceed the local bond strength, stress-relieving gaps might be formed.[1]

Therefore, for successful composite-dentin bonding and less debonding, either the bond strength between the cavity wall and dental adhesive must be higher than the shrinkage stress or the composite should exhibit low shrinkage stresses.[22] A study by Cho and others,[22] using the acoustic emission analysis technique to detect the debonding at the tooth-composite interface during composite curing, found that composites with lower shrinkage and slower polymerization reactions demonstrated fewer interfacial debonds during cavity restoration.

The SBRC comes with a two-step self-etch adhesive known as silorane system adhesive (3M ESPE, Seefeld, Germany). Consequently, it still boasts features of conventional methacrylate adhesives, especially with regard to its bonding mechanism to tooth tissue. However, it should be compatible with the highly hydrophobic silorane matrix. Transmission electron microscopy analysis of the interface complex of a SBRC bonded to enamel and dentin found that the two-step self-etch adhesive effectively bridged the hydrophilic tooth substrate with the hydrophobic silorane composite.[23] Moreover, the silorane adhesive system formed a hybrid layer of comparable thickness with that of methacrylate-based adhesives in scanning electron microscopy (SEM) analysis.[24]

Several studies concluded that there was no significant difference in shear bond strength to dentin between the SBRC and MBRC[25.26] or in the microtensile bond strength.[27-29] On the contrary, Khosla and others[30] found that the total etch system that is used with MBRC has a significantly higher shear bond strength value (13.4 MPa) than does the self-etch system of the SBRC (9.5 MPa).

Fernandes and others[31] found that MBRC yielded the highest bond strength values (26.3 MPa), regardless of the type of adhesive system used, when compared to the SBRC. Furthermore, Almeida and others[32] found that the MBRC showed superior performance regardless of the placement technique. Duarte and others[33] also found that SBRC shows compatibility only with its dedicated adhesive. Pucci and others[34] measured the microtensile bond strength of a SBRC to dentin after artificial aging of the specimens. They found that the surface treatment of dentin (laser or phosphoric acid) and the use of primer agitation improved the bond strength of the silorane adhesive system.

Furthermore, increasing the cavity configuration factor (C-factor) has been associated with progressive decrease in bond strength.[35] Isaac and others[36] found that a MBRC with total etch adhesive obtained higher bond strength (32.4 MPa) than did the SBRC (24.4 MPa) on a flat surface. On the other hand, no significant difference was found between both restorative materials in a Class I cavity (high C-factor) model. It was suggested that the adhesive system of the SBRC, although leading to lower bond strength mean values, is not subjected to the same stress at the bond interface since there is a lower

degree of volumetric shrinkage of the composite.[36] Furthermore, El-Sahn and others[37] found that, unlike MBRC, the increase in the C-factor did not negatively affect the bond strength of the SBRC. Van Ende and others[38] found that the silorane two-step self-etch adhesive performed significantly better than the one-step self-etch adhesive in high–C-factor cavities regardless of the composite used.

It can be concluded that the reduction in the polymerization shrinkage associated with the SBRC is also associated with the improvement in bond strength values in cavities with high C-factor. Hence, this may be considered to be more clinically relevant than bonding to a flat surface.

## CUSP DEFLECTION

Cusp deflection of teeth restored with the MBRC was found to be highly correlated with polymerization shrinkage.[39] The decrease in polymerization shrinkage associated with the SBRC has been associated with a decrease in cusp deflection. Palin and others[40] found a significant reduction in cusp deflection and microleakage of maxillary premolars restored with two experimental SBRC (2.5 and 6.0 μm) when compared with two conventional MBRC (16.5 and 20.6 μm). Moreover, their results correspond with those obtained by Bouillaguet and others,[41] who showed that a SBRC induced the lowest tooth deformation (3.5 μm) when tested against four MBRC. On the other hand, Tantbirojn and others[42] found that the low-shrinkage composites did not necessarily reduce coronal deformation.

Several studies[13,14,43-46] found that the SBRC was more efficient for cavity sealing than was the MBRC. In a study by Papadogiannis and others,[14] the SBRC showed better behavior than the MBRC in setting shrinkage and marginal adaptation with dentin. Santos and others[47] found that while the SBRC and MBRC had no significant difference in immediate push-out bond strength (8.0 and 9.8 MPa, respectively) and in marginal adaptation, the SBRC presented an increase in the mean push-out bond strength after six months of water storage (12.5 MPa). However, it should be mentioned that there was a high incidence of adhesive failures in the silorane restorations; thus, this may indicate a weak adhesive interface of the silorane adhesive system. This finding of failure pattern was observed in other studies as well.[27,38] In addition, a micro–Raman spectroscopy study by Santini and Miletic[24] demonstrated an intermediate zone of approximately 1 μm between the silorane primer and the bond. According to the authors,[24] this may be the weakest link in the failure mechanism of silorane restorations, and it requires further investigation.

## MECHANICAL AND PHYSICAL PROPERTIES

The physical and the mechanical properties of the SBRC require further investigation. Some studies[16,48,49] showed inferior mechanical performance compared to the MBRC, while others[50-52] showed mixed or comparable mechanical properties. Generally, the physical and mechanical properties are directly influenced by the degree of conversion that is obtained during adequate polymerization.

The degree of conversion for the SBRC varies in the literature from 50% to 80%,[13,14,40,49] which may be explained by the distinct power densities and curing times selected in these studies. Boaro and others[48] reported a very low degree of conversion for a SBRC (30%), which explained its lower mechanical performance (flexural modulus, 9.1 GPa, and flexural strength, 111.0 MPa ) compared to that of a nanohybrid MBRC.

Similarly, Ilie and Hickel[16] found that the macroscopically measured strength of the SBRC was comparable to that of most of the analyzed MBRC but was statistically lower than that of the nanohybrid MBRC. In a study by Lien and Vandewalle,[50] the SBRC (Filtek LS) showed an overall mixed mechanical performance. It had relatively high flexural strength/modulus (120 MPa/9 GPa) and fracture toughness (0.7 MPa $m^{1/2}$) but a relatively lower compressive strength (250 MPa) and microhardness (43 Kg/$mm^2$). The authors[50] related the lower microhardness of the SBRC to the reduced filler-volumetric fraction (55%). This is also in agreement with the work of Torres and others,[49] who found a low Knoop Hardness Number (41.7 Kg/$mm^2$) for a SBRC compared to those found in the literature for MBRC (60 Kg/$mm^2$).

On the other hand, other studies[51,52] showed that the SBRC was comparable to that of the regular micro-hybrid MBRC in terms of mechanical properties. Moreover, Zakir and others[53] found that the mechanical properties (fracture toughness and compressive strength) of Filtek Silorane increased considerably from day 1 to day 90. This was after incorporating 5% and 10% nano-hydroxyapatite crystals into the composite resin. The increase in the fracture toughness was attributed to the possible interruptions in the crack propagation by the hydroxyapatite crystals.

Surface roughness, water sorption, and solubility are essential to predicting the behavior of RBC

restorations. The literature reports that the SBRC presents lower sorption, solubility values, and diffusion coefficient compared with MBRC; furthermore, SEM analysis showed no surface changes after one year of water storage.[48,54-56] The reduction in water sorption and solubility was attributed to the hydrophobic backbone of the silorane molecule. Siloranes were also found to be stable and insoluble in biological fluid simulants using aqueous solutions containing either epoxy hydrolase, porcine liver esterase, or dilute hydrochloric acid.[57] Moreover, Yesilyurt and others[58] found that the hardness and flexural strength of Filtek Silorane were not significantly affected by storage in food-simulating liquids compared to MBRC.

Several studies[59-63] have reported better color stability for SBRC compared to MBRC. However, Pires-de-Souza and others[64] found that the SBRC (P90) underwent greater alteration in color and higher surface degradation after accelerated artificial aging compared to MBRC.

## FRACTURE RESISTANCE OF TEETH RESTORED WITH SBRC

The use of adhesive restorations has been recommended for reinforcing the remaining tooth structure after cavity preparation.[65] The SBRC has been suggested as an alternative to the MBRC to overcome the polymerization shrinkage problem and its consequences.

The number of studies that assessed the fracture resistance of teeth restored with SBRC is limited. However, the SBRC (Filtek P90) was not able to restore the fracture resistance of teeth with MOD cavities compared to a MBRC (Filtek P60).[66] Akbarian and others[67] found that there was no significant difference between SBRC and MBRC in MOD cavities. Similarly, Taha and others[68] found that SBRC restorations had no superior strengthening effect on endodontically treated maxillary premolar teeth with MOD preparations compared to MBRC, although both restorative materials modestly increased the fracture strength. On the other hand, Shafiei and others[69] reported that the SBRC revealed significantly higher strength for restored endodontically treated premolar teeth compared to that of MBRC, regardless of fiber insertion.

## CLINICAL STUDIES

Silorane-based resin composites have been found by experimental studies[12,14,43,44,46,51,52] to exhibit properties that are at least as good as those of MBRC. However, these findings should be validated by clinical studies (Table 1).

In a randomized clinical trial, Schmidt and others[70] found better performance for MBRC (Ceram X) compared to Filtek Silorane. This is in terms of the marginal adaptation (occlusal and proximal) of 158 Class II restorations after one-year follow-up. They concluded that the reduction in polymerization shrinkage demonstrated in the laboratory was not clinically significant. The external validity of their study may be affected by the fact that it was conducted at a dental school by one dentist; therefore, the results of the study cannot by directly related to everyday dental practice.[70]

To overcome the subjectivity of the results obtained by one operator, Burke and others[71] conducted a practice-based cohort study to evaluate the performance of Filtek Silorane restorations. The two-year assessment of 100 Filtek Silorane restorations (30 Class I and 70 Class II) indicated satisfactory clinical performance with no complaints of postoperative sensitivity; this could be attributed to lower values of cusp deflection as a result of reduced polymerization stresses.

In a double-blind, randomized clinical trial, Goncalves and others[72] found that both SBRC and MBRC performed similarly. Both showed marginal discoloration and changes in surface texture at 18 months when compared with baseline. However, P60 (MBRC) performed better than Filtek LS (SBRC) in the marginal integrity criterion. This finding was in agreement with that of a randomized clinical trial[73] that reported no significant difference in the clinical performance of Filtek Silorane and Ceram X in Class I posterior restorations after two years.

In a three-year prospective randomized clinical study, Mahmoud and others[74] found insignificant differences in the overall clinical effectiveness of SBRC and MBRC in Class II restorations. Filtek Silorane showed excellent clinical performance that was comparable to that of Ceram X Mono when it was used to restore noncarious cervical lesions over a three-year period.[75] The findings from the previous studies were confirmed by a five-year randomized clinical trial,[76] in which the authors found no statistically significant differences between Filtek Silorane and Ceram X in terms of proximal contacts, anatomic form, fractures, or discoloration.

Yazici and others[77] found that P60 showed the best marginal adaptation compared with Filtek Supreme and Filtek Silorane. However, all restorative resins performed equally well in clinical

Table 1: *Summary of the Clinical Studies that Evaluate Silorane-based Resin Composites (SBRC)*

| Study | Year | Duration | Type of Restoration (No.) | No,. of Patients | Materials Tested/Adhesive |
|---|---|---|---|---|---|
| Burke and others[71] | 2011 | 2 y | Class I (30)<br>Class II (70) | 64 | Filtek Silorane/Silorane System Adhesive |
| Schmidt and others[70] | 2011 | 1 y | Class II (158) | 72 | Filtek Silorane/Silorane System Adhesive<br>Ceram X /Xeno III |
| Baracco and others[80] | 2012 | 1 y | Class I (38)<br>Class II (37) | 25 | Filtek Silorane/Silorane System Adhesive<br>Z250/Adper Scotchbond (XT)<br>Z250/Adper Scotchbond (SE) |
| Goncalves and others[79] | 2012 | 6 mo | Class II (100) | 33 | Filtek P90/Silorane Adhesive System<br>Filtek P60/Adper SE Plus |
| Goncalves and others[72] | 2013 | 18 mo | Class II (100) | 33 | Filtek P90/Silorane Adhesive System<br>Filtek P60/Adper SE Plus |
| Efes and others[73] | 2013 | 2 y | Class 1(100) | 50 | Filtek Silorane/Silorane System Adhesive<br>Ceram X Duo |
| Mahmoud and others[74] | 2014 | 3 y | Class II (156) | 78 | Filtek P90/P90 System Adhesive<br>Quixfil/Prime &Bond NT |
| Popoff and others[86] | 2014 | 2 y | Class I or II (100) | 34 | Filtek P90/P90 System Adhesive<br>Filtek P60/Adper SE Plus |
| Walter and others [78] | 2014 | 3 y | Class II (82) | 31 | Filtek LS/Filtek LS System Adhesive<br>Tetric EvoCeram/AdheSE |
| Yaman and others[75] | 2014 | 3 y | Class V noncarious (144) | 24 | Filtek Silorane/Silorane System Adhesive<br>Ceram X Mono/Clearfil SE<br>Ceram X Mono/XP Bond |
| Yazici and others[77] | 2014 | 3 y | Class I (84) | 28 | Filtek Silorane/Silorane Adhesive System<br>Filtek P60/Adper Single Bond 2<br>Filtek Supreme/Adper Single Bond 2 |
| Schmidt and others[76] | 2015 | 5 y | Class II (158) | 72 | Filtek Silorane/Silorane System Adhesive<br>Ceram X/Xeno III |

conditions during the three-year evaluation period. The SBRC Filtek LS also performed similarly to the MBRC (Tetric EvoCeram) in Class II posterior restorations over a three-year clinical service.[78]

Goncalves and others[79] found no significant difference in the short-term clinical performance of the proximal contacts of 100 Class II restorations restored with Filtek P90 compared with those restored with Filtek P60 over the course of six months. Baracco and others[80] found that Filtek LS performed similarly to Filtek Z250 (used with two different adhesive systems) over two years of clinical service; in addition, the marginal adaptation of restorations deteriorated over the evaluation period.

As a consequence of the progression of techniques and materials in adhesive dentistry, repair of preexisting restorations—instead of their complete replacement—has become part of everyday dental practice. *In vitro* studies[81-83] found that the repair methods used for MBRC can be applied for SBRC. For aged SBRC, repairs were considered successful after sandblasting ($Al_2O_3$) and adhesive application with either SBRC or MBRC.[84,85] A clinical trial by Popoff and others[86] showed that SBRC are clinically acceptable to repair failed conventional MBRC

Table 1: *Summary of the Clinical Studies that Evaluate Silorane-based Resin Composites (SBRC) (ext.)*

| Study | Assessment Parameters | Results |
|---|---|---|
| Burke and others[71] | Retention of the restoration, lack of fracture, marginal integrity, secondary caries, gingival health, color match, stain resistance, surface quality | All restorations were found intact, without secondary caries; 97% of the restorations were rated optimal for anatomic form, 84% optimal for marginal integrity, 77% optimal for marginal discoloration, 99% optimal for color match, and 93% optimal for surface quality |
| Schmidt and others[70] | Marginal adaptation | Better performance for CeramX™ both occlusally ($p$=0.01) and proximally ($p$<0.01) compared to Filtek Silorane |
| Baracco and others[80] | Color match, retention, marginal adaptation, anatomic form, surface roughness, marginal staining, sensitivity, secondary caries | Filtek Silorane and XT had similar performance at 1 yr, SE had marginal staining, While XT had the best performance |
| Goncalves and others[79] | Proximal contacts | No significant differences were found between the two restorative materials |
| Goncalves and others[72] | Proximal contacts, fracture, marginal integrity, marginal discoloration, surface texture | No significant differences were found between the two restorative materials |
| Efes and others[73] | Anatomic form, marginal adaptation, surface texture, secondary caries, and postoperative sensitivity | No significant differences were found between the two restorative materials; no reports of sensitivity or secondary caries |
| Mahmoud and others[74] | Anatomical form, marginal adaptation, color match, marginal discoloration, surface roughness, secondary caries | No significant difference in the annual failure rate between the two composites (1.7% for Filtek P90 vs 1.2% for Quixfil) |
| Popoff and others[86] | Marginal adaptation, anatomic form, surface roughness, marginal discoloration, postoperative sensitivity, secondary caries | No significant differences were found between the two restorative materials |
| Walter and others [78] | Surface luster, surface staining, color stability and translucency, anatomic form, fracture and retention, marginal adaptation, wear, contact area, radiographic appearance, postoperative sensitivity, tooth vitality, recurrent caries, tooth integrity, periodontal response, adjacent mucosa, oral and general health | No significant differences were found between the two restorative materials |
| Yaman and others[75] | Retention, color match, marginal discoloration, anatomic form, marginal adaptation, surface texture, secondary caries, postoperative sensitivity | No significant differences were found among the restorative materials |
| Yazici and others[77] | Retention of restoration, marginal adaptation, marginal discoloration, loss of anatomic form, postoperative sensitivity and recurrent caries | All restorative resins performed equally well in clinical conditions during the 3-yr evaluation, with no significant differences, except for marginal adaptation, for which P60 showed superior results |
| Schmidt and others[76] | Marginal adaptation, marginal discoloration, proximal contact, anatomic form, fracture, secondary caries, hypersensitivity | No significant differences were found between the two restorative materials |

restorations, but they did not demonstrate any advantage over MBRC.

Long-term clinical studies on the longevity of SBRC restorations are required. On the basis of the results of the previous studies, it seems reasonable to conclude that there is no evidence yet that the SBRC perform clinically better than do conventional MBRC.

## CONCLUSIONS

Resin manufacturers have already done much toward significantly lowering volumetric shrinkage through the introduction of the SBRC. However, this review found that the attempt to reduce shrinkage by ring-opening polymerization is not yet conclusive in terms of efficacy. The reduced volumetric shrinkage of the SBRC did not have an advantage over the conventional MBRC in terms of clinical performance. Physical and mechanical properties of the SBRC and its ability to bond to dentin have been found to be comparable to those of the MBRC *in vitro*. However, SBRC showed lower values of water sorption and solubility compared to MBRC. Therefore, long-term clinical evaluations are required to fully assess the performance of this material.

**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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# Dentin Protection of Different Desensitizing Varnishes During Stress Simulation: An *In Vitro* Study

G Schmalz • F Hellwig • RF Mausberg • H Schneider • F Krause • R Haak • D Ziebolz



### Clinical Relevence

In treatment of dentin hypersensitivity, light-curing desensitizing varnishes might be able to avoid dentin loss. Consequently, these materials could be a promising preventive approach and may be preferred for clinical use.


## SUMMARY

**Objective: The aim of this study was to investigate dentin protection of different desensitizing varnishes (light- and self-curing) during acid action/abrasion stress and thermocyclic loading *in vitro*.**

**Methods: Dentin discs of 2 mm thickness were cut from 120 human molars, embedded, and polished. Specimens were randomized into five groups (n=24): A, negative control; B, Gluma Desensitizer; C, Cervitec plus (self-curing); D, Seal&Protect; and E, Admira Protect (light-curing). In groups B-E, varnish was applied on two-thirds of the dentin surface, and one-third acted as internal control. Stress cycle (2 cycles/day) for specimens were as follows: 1, acid action (pH: 2.9: five minutes); 2, remineralization (synthetic saliva: 60 minutes); 3, brushing (100 strokes); 4, thermocycling (five cycles); and 5, remineralization (synthetic saliva: six hours) for each group (n=12) for 30 (15 days) or 60 times (30 days). Specimens were analyzed using an incident light microscope. Substance loss was measured in micrometers. Statistical analysis was performed with the multiple contrast test ($p<0.05$).**

**Results: Groups B and C had a significantly lower dentin loss than A ($p<0.01$). After 30 days, group A showed the highest dentin loss ($p<0.01$), whereas the other groups lacked a significant difference regarding their substance loss (dentin and/or varnish; $p>0.05$).**



Gerhard Schmalz, Department of Cariology, Endodontology and Periodontology, University Medical Center Leipzig, Leipzig, Germany

Fabian Hellwig, Dr. med. dent., Department of Preventive Dentistry, Periodontology and Cariology, University Medical Center Goettingen, Göttingen, Germany

Rainer F. Mausberg, Dr. med. dent., Department of Preventive Dentistry, Periodontology and Cariology, University Medical Center Goettingen, Göttingen, Germany

Hartmut Schneider, Dr. rer. nat., Department of Cariology, Endodontology and Periodontology, University Medical Center Leipzig, Leipzig, Germany

Felix Krause, PD Dr. med. dent., Department of Cariology, Endodontology and Periodontology, University Medical Center Leipzig, Leipzig, Germany

Rainer Haak, MME, Prof. Dr. med. dent., Department of Cariology, Endodontology and Periodontology, University Medical Center Leipzig, Leipzig, Germany

* Dirk Ziebolz, MSc, PD Dr. med. dent., Department of Cariology, Endodontology and Periodontology, University Medical Center Leipzig, Leipzig, Germany

* Corresponding author: Liebigstr. 10-14, D 04103 Leipzig, Germany; e-mail: dirk.ziebolz@medizin.uni-leipzig.de


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**Varnish layer loss was shown for groups D and E with a remaining protective layer; groups A-C showed dentin removal.**

**Conclusion: All four varnishes are protective compared with an untreated control. Light-curing varnishes might provide higher dentin protection than self-curing materials.**

## INTRODUCTION

In a current review, Splieth and others highlighted the importance of dentin hypersensitivity (DHS), with a prevalence range between 3% and 98%.[1] The crucial problem of DHS is the exposed dentin surface, whereby, based on the current scientific opinion, the short and sharp pain is explained by Brännström's hydrodynamic theory: nociception as a result of nerve stimulation induced by fluid movements in dentin tubules.[2]

Furthermore, the different etiologic factors including dentin exposure because of gingival recession during periodontal disease, traumatic loss of the tooth surface, and erosion and abrasion must be considered. In this context, erosive food and drink, as well as tooth-brushing using abrasive toothpaste, are important.[3]

Considering DHS as a painful and frequently occurring problem, several therapeutic approaches are available.[4] One is the use of fluoride-containing toothpastes and varnishes.[5,6] Other ingredients, such as potassium, could help manage the pain caused by hypersensitive dentin.[7] Varnishes in different application forms as self-curing and light-curing materials are also available to treat DHS. For self-curing varnishes like Gluma Desensitizer and Cervitec plus, a positive clinical effect was reported.[8,9] These clinical benefits are consistent for light-curing materials, such as Seal&Protect and Admira Protect.[10,11] Additionally, an intervention, such as laser irradiation and the combination of laser and desensitization varnishes, could be a possible approach.[12,13]

Although a clinical benefit was shown for both light- and self-curing varnishes, different results were found.[8-11] The light-curing materials appeared to show a higher effectiveness compared with self-curing materials, especially over a period of a few months.[11,14-17] An important point regarding this issue might be the wear resistance of the desensitizing varnishes. Therefore, their stability against erosive and abrasive stress could be a decisive factor for their ability to reduce DHS sufficiently over a prolonged time. Moreover, protection of exposed dentin appears to be preferable to avoid further dentin loss. Data regarding dentin protection of desensitizing varnishes are rare, showing higher protective potential of light-curing varnishes.[18]

Accordingly, the current study investigated the resistance of different self- and light-curing desensitizing varnishes during erosion, abrasion, and thermocyclic loading to draw conclusions on their potential of dentin protection. The aim of this study was to investigate the dentin protection with light-curing and self-curing varnishes *in vitro*. It was hypothesized that light-curing materials protect dentin better than self-curing varnishes.

## METHODS AND MATERIALS

### Study Design

A randomized, five-arm *in vitro* study was performed on extracted caries-free human molars. The teeth were not extracted for this study, but for periodontal or orthodontic reasons (third molars). The use of teeth for *in vitro* studies was approved by the ethics committee (number 16/6/09); patients were informed and gave written informed consent. The resistance of two light- and two self-curing dentin varnishes against abrasion and erosion was investigated compared with an untreated control (Figure 1).

### Test Specimen Preparation

A total of 120 freshly extracted caries-free human molars were cleaned and stored in physiologic saline solution. Discs of 2 mm thickness were cut from upper and middle dentin in the transverse direction (Exakt Apparatebau GmbH, Norderstedt, Germany). Discs were embedded (Palavit G, Heraeus Kulzer GmbH, Hanau, Germany) avoiding contamination of the upper dentin surface as well as possible. Finally, the test specimens were polished with water-cooled sandpaper discs at a grain size of 1200/4000 (Struers GmbH, Willich, Germany).

### Test Material and Group

Self-curing varnishes (groups B and C) included Gluma Desensitizer (Heraeus Kulzer GmbH, Hanau, Germany) and Cervitec Plus (Ivoclar Vivadent GmbH, Ellwangen, Germany). Light-curing materials (groups D and E) were Admira Protect (Voco GmbH, Cuxhaven, Germany) and Seal&Protect (Dentsply DeTrey GmbH, Konstanz, Germany). Group A without a varnish layer was used as a negative control (Table 1).

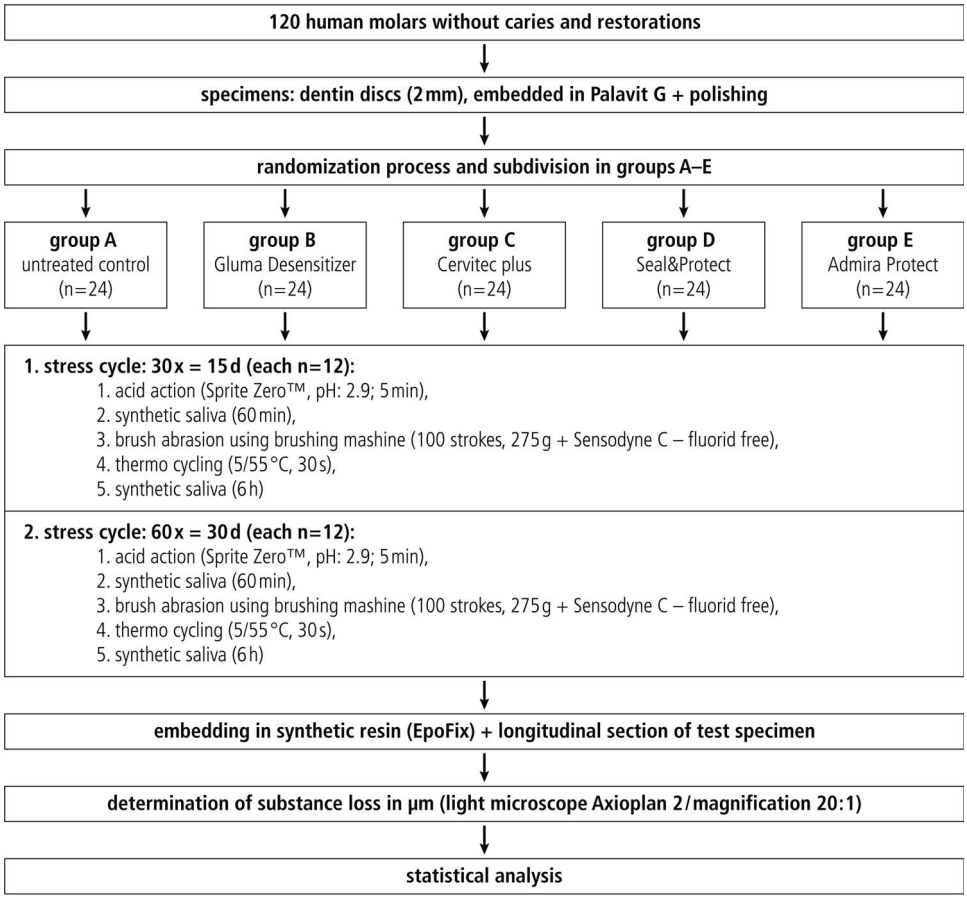


Figure 1. *Study design.*

### Randomization Procedure and Varnish Application

Test specimens were randomly divided into five groups (A-E) with 24 probes each. A further selection of the groups in A1-E1 and A2-E2, in accordance with the cycle length (30 or 60 cycles), was performed afterward (n=12). Test specimens from groups B1 and B2 to E1 and E2 were taped to cover one third of the dentin using sticky tape number 1 (thickness 0.15 mm, Coroplast, Wuppertal, Germany), protecting them from loading. Groups B1 and B2 to E1 and E2 were treated according to the manufacturer's instructions, with the corresponding varnishes (Table 1). Light-curing materials (groups D and E) were light cured with an LED polymerization lamp (Bluephase [1200 mW/cm$^2$], Ivoclar Vivadent, Schaan, Lichtenstein). Groups A1 and A2 remained untreated. Additionally, tape number 2 was applied to one half of the varnished dentin specimens to obtain a reference surface, which was also protected from loading. The specimens from groups A1 and A2 were only taped on one half because there was no varnish layer (Figure 2).

### Stress Cycles: Abrasion, Erosion, and Thermocyclic Loading

For stress simulation, a repetitious cycle was conducted, consisting of acid action, remineralization, brush abrasion,[18] and thermocycling (Figure 1). A soft drink with a pH of 2.9 (Sprite Zero, Coca Cola GmbH, Berlin, Germany) was used, followed by a remineralization step with synthetic saliva.[19] Abrasion was performed with slurry of synthetic saliva and fluoride-free toothpaste (Sensodyne C, GlaxoSmithKline Consumer Healthcare GmbH & Co. KG, Hamburg, Germany).

Acid action lasted for five minutes, followed by remineralization for 60 minutes in synthetic saliva.[18] Brush abrasion was performed with an automatic brushing machine (University Medical Centre Goettingen) with a loading mass for the brushes on specimens of 275 g. The brushing machine operated at 100 strokes/min for one minute and used 20 mL

Table 1. *Information About Used Materials: Ingredients and Method of Application*

| Group | Product name | Manufacturer | Mechanism of action | Ingredients | Application |
|---|---|---|---|---|---|
| A1/A2 | Untreated control | — | — | — | — |
| B1/B2 | Gluma Desensitizer | Heraeus Kulzer | Self-curing | (2-hydroxyethyl) methacrylate, glutaraldehyde, purified water | Clean the dentin surface with a pumice slurry.<br>Rinse off with water.<br>Apply the smallest possible amount of GLUMA Desensitizer required for treatment to the dentinal surface using brush and then leave for 30-60 seconds.<br>Dry the surface carefully by applying a stream of compressed air until the fluid film has disappeared and the surface is no longer shiny.<br>Then rinse thoroughly with water. |
| C1/C2 | Cervitec plus | Ivoclar Vivadent | Self-curing | Ethanol, water, acrylate copolymer, vinyl acetate copolymer, and chlorhexidine diacetate 1%, Thymol 1% | Clean the tooth surfaces thoroughly.<br>Dry with cotton rolls and an air syringe.<br>Apply a thin coat of varnish using a brush.<br>Let the varnish dry.<br>Dry again with compressed air for 30 seconds (no water rinse). |
| D1/D2 | Seal&Protect | Dentsply | Light-curing | Di- and Trimethacrylate-resins, PENTA (dipentaerythritol pentacrylate-phosphoric acid - monomer), functionalized amorphous silicium dioxide, photoinitiators, butylated hydroxytoluol, cetylaminhydrofluoride, Triclosan, acetone | Clean the dentin surface with a rubber cup and a prophy paste.<br>Remove prophylaxis paste with an air/water spray.<br>Dry clean the area with a two-second blow of air free of oil or water contamination; avoid desiccating the dentin, leave a moist, but not wet, glistening surface.<br>Two to three drops are required per surface to be treated.<br>Apply with an applicator tip.<br>Leave the dentin surface undisturbed for 20 seconds.<br>Remove excess solvent by blowing gently with air for a few seconds.<br>Cure Seal&Protect for 10 seconds using a curing light.<br>Apply a second layer of Seal&Protect.<br>Remove excess solvent from the second layer by blowing gently with air.<br>Cure Seal&Protect for 10 seconds using a curing light.<br>Remove oxygen-inhibited (soft surface) layer with a cotton pellet or cotton roll. |
| E1/E2 | Admira Protect | Voco | Light-curing | Mixture of different dimethacrylates, acetone, catalysts, ormocers, auxiliaries | Clean teeth with fluoride-free cleaning paste on a rubber cup.<br>Remove excess moisture with an oil-free airjet; the dentin surface should be slightly moist.<br>Apply Admira Protect with a disposable brush on dentin surfaces.<br>Disperse Admira Protect with a faint airjet.<br>Light cure for 10 seconds.<br>Apply a second layer of Admira Protect, disperse it with a faint airjet and light-cure for 10 seconds.<br>Remove the oxygen-inhibited layer (soft surface) with a cotton pellet. |

slurry for each brushing procedure.[18] Thermocycling was conducted with five cycles between 5°C and 55°C in tempered water for 30 seconds each with a changeover time of 15 seconds (Haake DC10, Thermo Fisher Scientific GmbH, Schwerte, Germany). This cycle was conducted 30 times for groups A1-E1 and 60 times for groups A2-E2 to simulate 15 or 30 days of loading with two cycles per day. Between cycles, specimens were stored in synthetic saliva for six hours.

### Microscopic Analysis

After air drying for 60 seconds and the removal of the sticky tapes, the specimens were embedded in epoxy resin (EpoFix, Struers GmbH, Willich, Germany) and separated vertically using a cutting disc. Thicknesses of the varnish layer and substance loss were imaged and measured in micrometers by an incident light microsope (Axioplan, Carl Zeiss Jena GmbH, Jena, Germany; magnification 20×) in combination with a digital camera (AxioCam HRc,

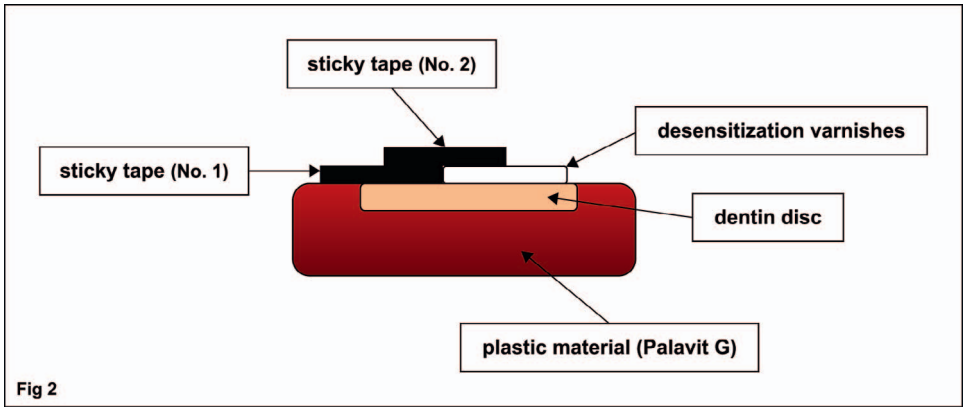


Figure 2. *Graphic presentation of test specimen with varnish application and placement of sticky tape.*

Software AxioVision 4.7, Carl Zeiss Jena GmbH). The measurement was executed every 50 μm using a digital ruler. The untreated, unstressed dentin surface served as an internal reference. Three parameters were measured: layer thickness, varnish layer loss, and/or dentin loss.

### Statistical Analysis

Mean values of layer thickness and varnish/dentin loss of both halves of each probe were summarized to a total value of the test specimen. Mean values of groups were generated out of the total values from the specimen. Group differences were assessed by the nonparametric multiple contrast test. Calculation was conducted with "nparcomp" with the help of the Software "R GUI" (www.r-project.org). The significance level was set at $\alpha=0.05$.

## RESULTS

Results are given in Table 2 and Figure 3.

### References and Negative Control

The untreated groups A1 and A2 lacked a varnish layer. Although at the reference surface (under tape no. 2; Figure 2) for self-curing materials (groups B1/B2 and C1/C2) no varnish layer could be measured, light-curing groups D1 and D2 and groups E1 and E2 showed a varnish layer of 65.27 (D1), 59.89 (D2), 42.28 (E1), and 41.07 μm (E2).

### Dentin and Material Losses After 15-day Simulation of Erosion/Abrasion and Thermocyclic Loading

There were significant differences of substance loss between groups A1-B1, B1-D1, B1-E1, C1-D1, and

Table 2. *Loss of Varnish Layer and Dentin (μm)*

| Group | Varnish layer: reference (MV ± SD in μm) | Varnish layer: postcycle (MV ± SD in μm) | Substance loss (MV ± SD in μm) |
|---|---|---|---|
| **Time 1 (after 15 days)** | | | |
| A: untreated control | — | −11.1 ± 2.8 | 11.1 ± 2.8 |
| B: Gluma Desensitizer | — | −7.9 ± 1.4 | 7.9 ± 1.4 |
| C: Cervitec plus | — | −8.4 ± 1.8 | 8.4 ± 1.8 |
| D: Seal&Protect | 65.3 ± 21.7 | 50.2 ± 21.1[a] | 15.1 ± 5.5 |
| E: Admira Protect | 42.3 ± 13.7 | 21.6 ± 7.3[a] | 20.7 ± 11.7 |
| **Time 2 (after 30 days)** | | | |
| A: untreated control | — | −63.2 ± 27.7 | 63.2 ± 27.7 |
| B: Gluma Desensitizer | — | −21.5 ± 3.7 | 21.5 ± 3.7 |
| C: Cervitec plus | — | −24.1 ± 6.0 | 24.1 ± 6.0 |
| D: Seal&Protect | 59.9 ± 19.8 | 41.2 ± 21.5[a] | 20.4 ± 7.6 |
| E: Admira Protect | 41.1 ± 13.8 | 12.0 ± 32.1[a] | 29.1 ± 24.6 |

*Substance loss for groups A-C is pure dentin removal, whereas the substance loss for D and E indicates a loss of varnish layer. The missing material layer is expressed by missing values. MV, mean value; SD, standard deviation.*
[a] *Remaining varnish layer on dentin.*

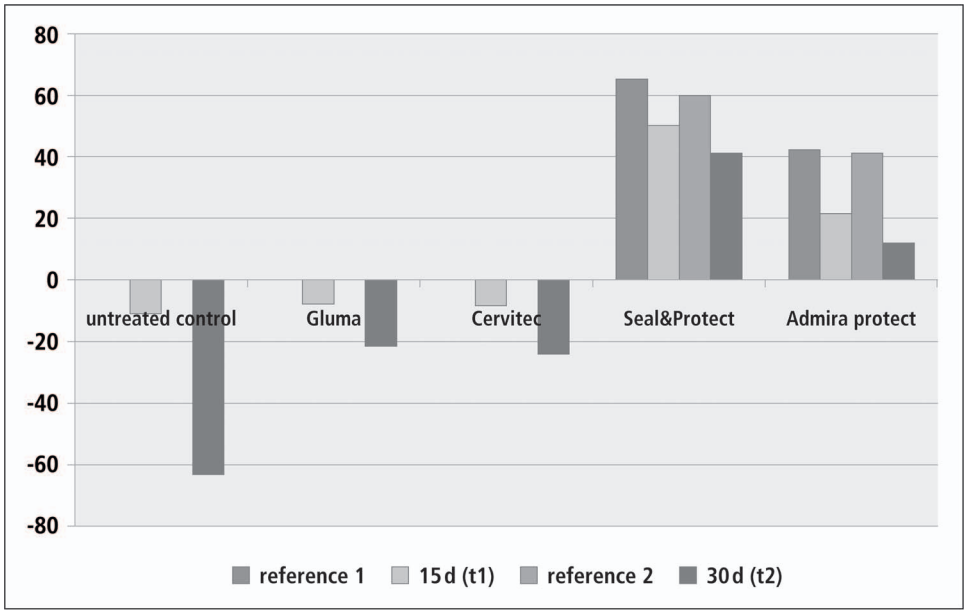


Figure 3. *Layer thickness reference, at t1/t2 and substance loss (dentin) at t1/t2. (The average values [in micrometers] are illustrated at baseline 1 and 2, which shows the reference values for each group. It is worth noting that the first three groups show no values at baseline because there was no detectable layer. At t1 [after 15 days] and t2 [after 30 days], the first three groups had negative values, which shows dentin loss, whereas the positive values for Seal&Protect and Admira Protect represent the presence of a varnish layer.)*

C1-E1 ($p_i<0.01$). Additionally, a trend was seen comparing groups A1 and C1 ($p=0.051$). After 15-day simulation of stress, a dentin loss was registered in groups A1, B1, and C1. The untreated group A1 had the highest dentin loss. Groups with self-curing varnishes showed less dentin loss compared with group A, with minor differences between groups B and C. In contrast, for the light-curing groups (D1 und E1), a limited varnish removal with a remaining layer was observed.

### Dentin and Material Losses After 30-day Simulation of Erosion/Abrasion and Thermocyclic Loading

After 30-day stress simulation, there were significant differences between the groups A2-B2, A2-C2, A2-D2, and A2-E2 ($p_i<0.01$). The significantly highest dentin loss was detected in group A2. Substance losses of the self-curing desensitizers were not significantly different ($p>0.05$). The light-curing varnishes had a remaining varnish layer with the highest remaining layer thickness in group D2.

Each specimen showed a substance loss at both times of measurement. Group A had a pure dentin loss, for groups B and C a complete varnish and dentin loss, and for groups D and E a pure varnish loss were registered. Accordingly, no dentin removal was measured for the light-curing varnishes.

## DISCUSSION

The aim of this *in vitro* study was to investigate dentin protection with light-curing and self-curing varnishes during abrasion, acid action, and thermocyclic loading under standardized conditions.

The main result of the study registered for self-curing varnishes a complete varnish and dentin loss, whereas for light-curing materials, a remaining varnish layer could generally be detected after 30 or 60 cycles simulating loading of 15 or 30 days. This suggests that light-curing materials are able to protect dentin, whereas self-curing varnishes showed no stable protective layer for the study period, which resulted in a measurable dentin loss. In this study setup, loading caused substance removal in every specimen. However, with light-curing materials, the dentin always remained undamaged.

Methods for stress simulation were chosen in accordance to Schneider and others.[18] Periods of acid action, storage in artificial saliva, and brush abrasion were standardized. The medium for erosive action in the earlier study mentioned above was Sprite Light with a pH of 2.9. This is almost identical to the measurements for Sprite Zero in the current study with a pH of 2.9. Other investigations with comparable issues also used Sprite Light.[20,21] Likewise, Schneider and others[18] explained the use of an automatic brushing machine; in the current study, the same number of brushing strokes and bearing mass was used (100 strokes/275 g). The chronology and times for exposure in the current study are similar to those of another investigation.[22] Erosion (0.3% citric acid, pH 3.2, five minutes), remineralization (artificial saliva, one hour, pH 7.0), and

abrasion (120 linear strokes with 300 g loading) varied only slightly between studies. Use of an automatic brushing machine is a common procedure; accordingly, other authors also chose comparable loadings and brushing strokes. Therefore, Yu and others[23] used 100 strokes with a load of 250*g*, whereas Vieira and others[24] performed 200 strokes with load attuned to 150*g*. Implementation of two brushing actions each day was among others introduced by Ganss and others.[25] In the current study, thermocyclic loading was additionally conducted to simulate thermal stress as thermal changes may cause defects on a dentin-adhesive surface.[26] Synthetic saliva pH varied between different studies with a range of 6.4 to 7.0.[19,22,25,27] Use of synthetic saliva is necessary for standardization of wet surroundings of specimen's surface quality. Light microscopy was already performed in another *in vitro* investigation.[26]

There was a significant difference between Gluma Desensitizer and control groups after simulation of 15 and 30 days of loading ($p<0.01$). Gluma Desensitizer causes dentinal tubule occlusion by reaction of glutaraldehyde with a dentinal tubule protein, resulting in reduced diameter of dentinal tubules and dentinal tubule occlusion.[28,29] This could explain why dentin with occluded tubules might be more wear resistant than untreated dentin and why no measurable varnish layer was found. With Cervitec plus, less dentin loss was also found ($p<0.051$) both after 15- and 30-day simulation ($p<0.01$). It was assumed that this material might reduce the hydraulic permeability of dentin.[8] This could contribute to the desensitizing effect, but it does not explain sufficiently the potential dentin protection.

After 15- and 30-day simulation of loading, Seal&Protect and Admira Protect showed a remaining varnish layer. In accordance with this, in a recent *in vitro* study, Seal&Protect showed more reductions in dentinal fluid flow rate than Gluma Desensitizer.[15] This also suggests a higher effectiveness and dentin protection of light-curing materials. There are no results available about remaining varnish layer thickness *in vivo*. However, *in vitro* studies have examined this topic. The investigation by Schneider and others[18] reported that Seal&Protect ensured best dentin protection. Additionally, Gluma Desensitizer treatment caused lower dentin loss than untreated controls.[18] This and other investigations confirm the current study results.[30-32] One study simulated erosive impact of intrinsic and extrinsic acids using hydrochloric and citric acid and reported that Seal&Protect significantly reduced enamel mineral loss.[32] In addition, Seal&Protect protected dentin from erosive wear *in situ*.[30] Furthermore, One Coat Bond and Optibond FL were more resistant to erosive stress from Coca Cola than Gluma Desensitizer.[31] This confirms the conclusion that light-curing varnishes might ensure better dentin protection than self-curing materials. However, the protection appears to be for short term, because a varnish layer loss is found. This is in accordance with Zhao and others, where a short-term protection of Seal&Protect was shown, but a repetitious application is necessary to ensure long term protection of dentin.[33] To the best of the authors' knowledge, no investigation has reported on the wear resistance of Cervitec plus and Admira Protect.

Clinical effectiveness of the investigated self-curing[8,14,34] and light-curing varnishes[11,14] has already been found in several studies. In this context, light-curing materials are repeatedly discussed to ensure a better reduction of DHS compared with self-curing varnishes, especially over a period of several months.[11,28-30,35] Based on the results of the current study, a potential reason for this benefit is the protective varnish layer on the dentin surface, which was more stress resistant, compared with the self-curing materials, and prevented dentin loss in the simulated observation period.

In summary, neither light-curing nor self-curing materials are completely resistant to erosive/abrasive wear, but they ensure a certain degree of dentin protection. Moreover, light-curing varnishes can ensure sufficient dentin protection despite substance removal of a detectable varnish layer. Self-curing materials were at least able to reduce the dentin loss. The remaining varnish layer in light-curing materials could be a reason for their high effectiveness in available clinical investigations. However, only detected substance loss could be assessed in the current study and therefore it is impossible to draw strong conclusions on clinical effectiveness.

There were limitations in the current investigation. Based on the results of this study, the clinical effectiveness of varnishes could only be anticipated. Furthermore, different test specimens were compared post-sectioning after 15 and 30 days, and destruction of the specimen is a possible criticism. Alternative methods, such as use of an optical profilometer[36] or scanning electron microscope (SEM) and micro–computed tomography (μCT)[6,37] might illustrate the substance loss more effectively, but they were not available for the current investigation. It must also be mentioned that the simula-

tion of protein precipitation induced by Gluma Desenitizer in extracted teeth might differ from *in vivo* conditions as to what influences the potential to protect dentin. However, for standardization, this experimental setup was necessary, and Gluma Desensitizer as a common therapeutic option was included in the current investigation. To conclude, methods that were used are reproducible and close to the clinical situation.

## CONCLUSION

Within the limitations of the study, all four varnishes protected the dentin surface compared with the untreated control. The results of the present study suggest that light-curing varnishes ensure higher dentin protection than self-curing materials and could therefore be recommended for clinical use. To verify these results *in vivo*, further clinical studies are needed.

### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of University medical center Goettingen, Germany. The approval code for this study is 16/6/09.

### 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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