OPERATIVE DENTISTRY

november/december 2012 • volume 37 • number 6 • 561-676

ISSN 0361-7734
e-ISSN 1559-2863

OPERATIVE DENTISTRY

NOVEMBER/DECEMBER 2012 • VOLUME 37 • NUMBER 6 • 561-676

Aim and Scope

Operative Dentistry publishes articles that advance the practice of operative dentistry. The scope of the journal includes conservation and restoration of teeth; the scientific foundation of operative dental therapy; dental materials; dental education; and the social, political, and economic aspects of dental practice. Review papers, book reviews, letters and classified ads for faculty positions are also published.

Operative Dentistry (ISSN 0361-7734) is published bimonthly by Operative Dentistry, Indiana University School of Dentistry, Room S411, 1121 West Michigan Street, Indianapolis, IN 46202-5186. Periodicals postage paid at Indianapolis, IN and additional mailing offices. Postmaster: Send address changes to: Operative Dentistry, Indiana University School of Dentistry, Room S411, 1121 West Michigan Street, Indianapolis, IN 46202-5186.

Subscriptions: Fax (317) 852-3162

Current pricing for individual, institutional and dental student subscriptions (both USA and all other countries) can be found at our website: www.jopdent.org, or by contacting our subscription manager: E-mail: editor@jopdent.org

Payment must be in US dollars only and accompany orders. Online payment is available on our website. American Express, Discover, MasterCard and Visa accepted.

Contributions

Contributors should study submission requirements found on our website at www.jopdent.org and follow them carefully.

Permission

For permission to reproduce material from Operative Dentistry please apply to Operative Dentistry at the above address.

The views expressed in Operative Dentistry do not necessarily represent those of the academies or the editors.

Editorial Office

Operative Dentistry
Indiana University School of Dentistry, Room S411
1121 West Michigan Street, Indianapolis, IN 46202-5186
Telephone: (317) 278-4800, Fax: (317) 278-4900
URL: http://www.jopdent.org/

Editorial Staff

Editorial Board

For the names of our Ad Hoc reviewers, please go to: https://www.jopdent.com/journal/editorial_board.html

We thank all our reviewers for their time and dedication to Operative Dentistry.

How to Register as an Individual or Institutional User
It's easy to register. Simply choose a unique user name and password and complete our registration form at http://www.jopdentonline.org/action/registration. You will receive an e-mail confirming your user name and password shortly after registering. Registration is not required for individuals to use this site but is required to see the full text of a non-public access article.

Using the User Profile to Customize Your Online Experience
Once you have registered, you can customize your profile in the User Profile section of the site.
Within the User Profile section you can:
Update your profile information, including your user name and password, address details and account preferences, manage your favorite articles, sign up for journal e-mail alerts, view saved searches, and manage your subscriptions and online access (for institutional subscribers only).
For additional assistance with your profile, complete our feedback form at http://www.jopdentonline.org/feedback/show
Your user name and password can be sent to the e-mail address provided during the registration process at any time. Follow the instructions at http://www.jopdentonline.org/action/requestResetPassword to obtain your user name and password by e-mail.

Editorial

In response to reader and author feedback, a couple of minor format changes are included in this issue of Operative Dentistry. First, the clinical technique, case report and clinical research articles are placed immediately after any invited manuscripts. Second, the e-publication only articles are identified with their clinical relevance statements. These manuscripts can be read in their entirety at www.jopdentonline.org. If your subscription includes online access and you are having any problems getting to the information, do not hesitate to contact our support desk at helpdesk@allenpress.com.

This month's issue includes an invited paper from Dr. David T. Wong covering the material that was presented during the Buonocore Lecture at the 2012 Academy of Operative Dentistry Annual Meeting in Chicago. I extend my sincere gratitude to Dr. Wong for his contribution to the Academy and this journal. I hope that those of you who missed an excellent presentation will benefit from this paper. The rest of the issue includes information on the treatment of dentin prior to bonding, issues surrounding zirconia, the impact of instrumentation of composite restoration margins and the interaction of bleach with tooth structure.

And I would like to say a special thank you to each individual who has provided reviews over the last year. Reviewers, alongside of authors, are the lifeblood of the journal. These individuals are listed on the journal cover or linked online as ad-hoc reviewers. Some have provided as many as eight manuscript reviews over the last twelve months. Kudos to you!

I hope that each of you finds something worthwhile in this issue. Happy reading!

Jeffrey A. Platt D.D.S., M.S., Editor

Staff Letter

Open Letter to All Submitting Authors of Operative Dentistry

Dear Authors,

We are so grateful to each of you for continuing to provide Operative Dentistry with such outstanding manuscripts to consider. We have seen a steady increase each year in the number of manuscripts that are sent to us for publication consideration. For example 20 years ago, during the 1992 volume year, we received 68 manuscripts for consideration, and printed 33 an acceptance rate of 48%; 10 years ago, during the 2002 volume year, we received 212 manuscripts—a 311% increase, 93 were printed putting our acceptance rate at 43%; last year we received 505—a 238% increase over 2002 and a 742% increase over 1992, 80 were printed (more pages than in 2002) for a 15% acceptance rate. As you can see, we are having to become more and more selective in the articles we publish, not only that, but because of the costs of maintaining a manuscript submission system that can deal with this kind of traffic, our costs are exponentially increasing as well. Unfortunately, it has come time that we need to pass a small portion of these costs on to our submitting authors. Operative Dentistry is charged by our submission vendor 25.00USD per manuscript that goes through our system, whether it is accepted for publication or not. Beginning with the first submission of 2013, that cost will now be charged to our authors to submit a manuscript into our system. This 25.00USD fee will be required for a manuscript to be considered in any way. Please understand that this fee is charged to us by our vendor, and will be non-refundable. Paying the submission fee will have no bearing on whether or not your manuscript will be accepted either for review, or for publication. We thank you for understanding the necessity of this step. Should you have any questions about this new policy, please contact our offices at editor@jopdent.org.

Sincerely,

Operative Dentistry Office Staff

©Operative Dentistry, 2012, **37-6,** 562-570

Buonocore Lecture

Salivary Diagnostics

DTW Wong

Clinical Relevance

The past 10 years have witnessed significant advances in the science and technology of salivary diagnostics. The integration of salivary diagnostics into the clinical practice of dentistry is on the horizon to enable chair-side detection of oral and systemic disorders in the dental office.

SUMMARY

Saliva is a noninvasive and accessible biofluid that permits early detection of oral and systemic diseases. Recent scientific and technologic advances have uncovered specific salivary biomarkers for a number of clinical conditions, including cancers, autoimmune diseases, and cardiovascular disorders. The availability of highly sensitive and high-throughput assays such as microarray, mass spectrometry, reverse transcriptase quantitative polymerase chain reaction (RT-qPCR) and nano-scale sensors that can measure proteins and nucleic acids are poising saliva as an emerging biofluid for translational and clinical applications. This paper will discuss development of salivary biomarkers for the detection of oral and systemic diseases and the translational application of these markers for clinical applications.

*David T.W. Wong, DMD, DMSc, professor, University of California, Los Angeles, School of Dentistry, Los Angeles, CA, USA

*Corresponding author: 10833 Le Conte Avenue, 73-017 CHS, Los Angeles, CA 90095; e-mail: dtww@ucla.edu, dwong@dentistry.ucla.edu

DOI: 10.2341/12-143-BL

INTRODUCTION

Current accurate diagnostic procedures available for most diseases require invasive sampling performed by trained professionals and the use of expensive, specialized machinery and equipment. The discovery that saliva contains molecular profiles that reflect diseases in the body has opened the doors to a new noninvasive diagnostic methodology: salivary diagnostics. Using saliva in early detection of diseases is quickly proving to be not only practical, noninvasive, and safe, at times it is proving more accurate than available alternatives.

Salivary diagnostics is a late comer; however, in the last decade there has been significant progress in the field. In this article, we will review the recent advances made in salivary biomarker-based diagnostics via genomic and proteomic approaches and their implications for dentistry and medicine.

SALIVA: THE BIOFLUID

Saliva is the secretions by the three major salivary glands (parotid, submandibular, and sublingual), hundreds of minor salivary glands, and gingival crevice fluid. The functions of saliva are many and, among others, include "regular" functions such as food digestion, bolus formation, lubrication, and taste facilitation, and immune functions through antimi-

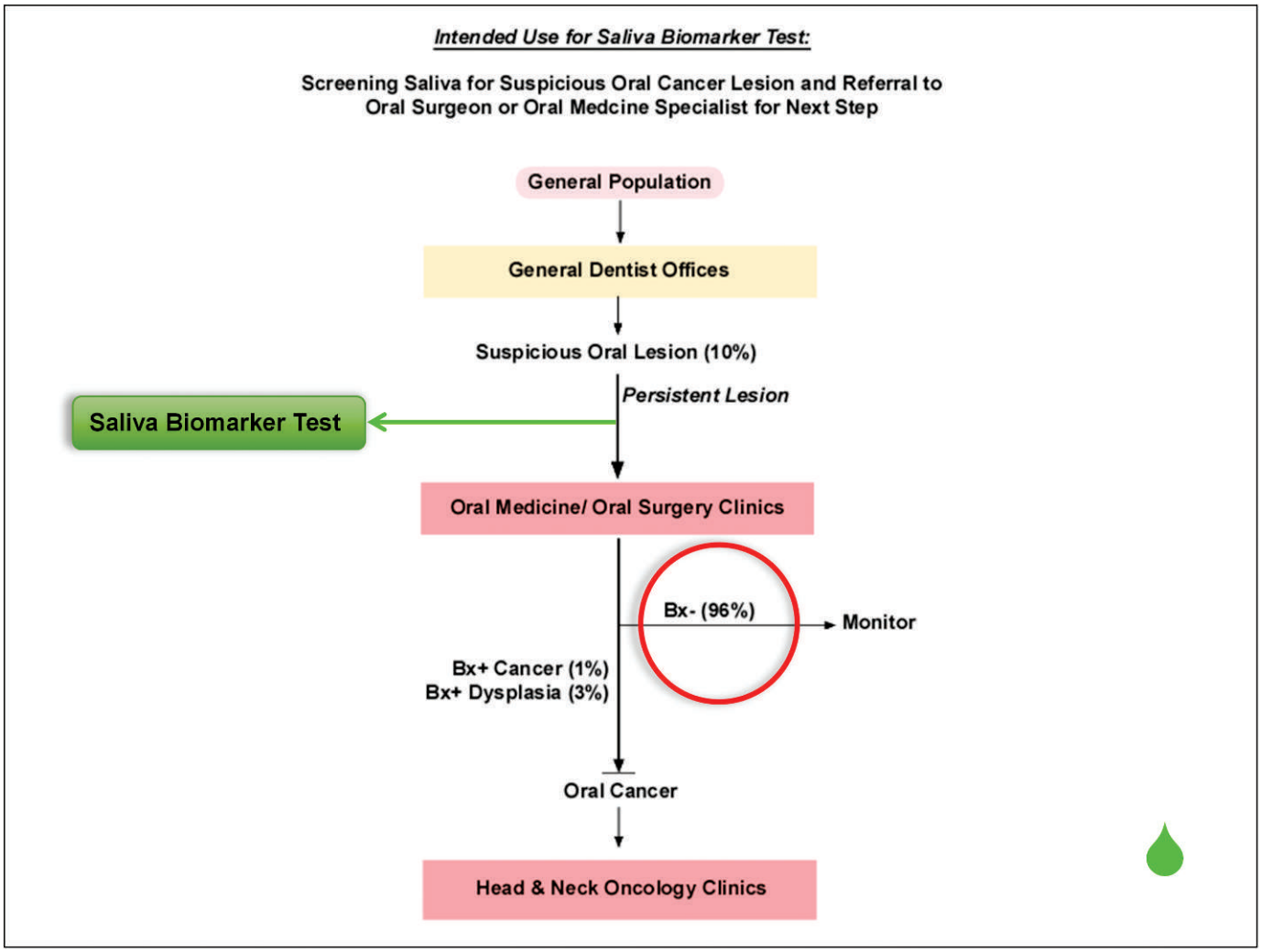


Figure 1. *Intended clinical context of salivary detection of oral cancer in dental office. Salivary diagnostics will reduce the number of unnecessary referrals to hospital for invasive, time-consuming, and expensive diagnostic procedures. Nine-six percent of biopsies of suspicious oral mucosal lesions are not cancer.*

crobial peptides[1] and immunoglobulins.[2] However, the mentioned saliva components only constitute a small part of secreted saliva. Considering the multitude of molecular species found and the unknown reason for their presence in the oral cavity (and subsequent gastrointestinal tract), saliva could play yet undetermined roles in maintaining systemic homeostasis.

Cell-free saliva has been found to contain over a thousand proteins involved in a wide range of biologic functions,[3] as well as messenger RNA (mRNA) and micro RNA (miRNA) transcripts[4-6] and metabolites.[7,8] Saliva proteins, mRNA, miRNA, and metabolites are proving useful for diagnostic purposes, as their increased and decreased occurrence in saliva is being found to reflect oral as well as systemic disease. The complex mixture that forms secreted saliva has called for developments in genomic and proteomic approaches to allow for its analysis and its use as a diagnostic medium.

ADVANCES IN SALIVA GENOMICS, PROTEOMICS, AND STUDY DESIGN

Saliva Analysis Technology

Microarray has allowed for a high throughput analysis of saliva and is the current gold standard for identifying salivary transcripts. Currently, the salivary transcriptome is profiled using microarray technology and validated with quantitative polymerase chain reaction (qPCR). Due to the low concentration of some biomarkers and the small volume of sample that can be collected from some subjects, salivary biomarker analysis has called for innova-

tions in technology to allow for detection of specific low-concentration transcripts.

Work conducted by Hu and others[9] has overcome the limitations of microarray and qPCR by two developments: a universal mRNA-amplification method for the microarray biomarker discovery phase, and in the qPCR validation step, a multiplex preamplification method has been developed and implicated. Thus, the performances of these technologies are not hindered by the low concentration of biomarker transcripts in saliva. Moreover, the multiplex preamplification method can perform simultaneously for many different targets and therefore provides a cost-effective screening with decreased workload as well as allowing for quantitative measurement using a relatively small amount of preamplification product.

Saliva Proteomics Analysis

Disease detection based on salivary protein biomarkers provides its own challenges. Proteins do not generally have long half-lives, although there is much difference from protein to protein. Not only does the nature of peptides cause special requirements for processing and storage of saliva protein analytes, the milieu of the oral cavity also imposes protein degrading factors. Saliva is a complex fluid that contains, among others, proteolytic enzymes that affect the stability of saliva protein analytes over time. Thus, protein-based salivary diagnostics requires immediate processing and/or analysis of saliva samples or the use of freezers and costly protease inhibitors for storage until processing.

In research and laboratory settings, requirements for protein-based detection of disease can be met easily. However, in a clinical setting, protein stabilization without need for freezers and other specialized machinery may be required for the clinical reality of salivary diagnostics. Our laboratory is in the forefront of development of protein stabilization methods that allow for storage of protein-containing saliva samples at room temperature and without expensive reagents and machines. We believe such development will greatly benefit salivary diagnostics and facilitate its implication in clinical practices.

Saliva Biomarker Study Design

The use of salivary biomarkers for detection of breast cancer has been explored since the early 2000s. Such studies[10-14] assessed the use of c-erbB-2, VEGF, EGF, and CEA for saliva biomarker-based breast cancer diagnostics. Thus, these studies looked for known

Figure 2. *The oral fluid nanosensor test of the University of California, Los Angeles: a portable device for point-of-care detection of salivary biomarkers.*

serum biomarkers in saliva. Zhang and others[5] were the first to use *de novo* transcriptomic and proteomic approaches to discovery and validation of a salivary biomarker profile for breast cancer detection.

Prospective-specimen-collection and retrospective-blinded-evaluation (PRoBE) Clinical Design—The use of biomarker for clinical decision-making obviously requires stringent testing of the biomarker's performance. Advances in biotechnology and the biomarker research field have increased dramatically in the last decade. However, the validation of discovered biomarkers has fallen behind.

Guidelines for reporting study results and a phased approach to biomarker development have been around for a while. However, not until recently has a comprehensive guide to biomarker study design been available. A notable advance in salivary biomarker clinical study design is PRoBE (prospective specimen collection and retrospective blinded evaluation, described by Pepe and others[15]). PRoBE design incorporates prospective specimen collection from the target population, collected in a blinded fashion with no knowledge about the patient's outcome. After outcome has been determined, patients with the outcome and control subjects without the outcome are selected randomly, and their specimens are tested in a blinded to case-control status fashion. The incorporation of the PRoBE design in biomarker development (discovery and validation) is critical to ensure the eventual validation of biomarkers in a specific clinical setting.

Salivary Transcriptome

mRNA and miRNA—mRNA and miRNA are transcribed by active cell machinery in response to

normal input in order to perform normal cellular functions and maintain cellular and systemic homeostasis. Transcription of RNA is governed by complex molecular pathways and associated molecules of both intracellular and extracellular origin. mRNA as well as miRNA can be found secreted in association with microvesicular structures in the extracellular milieu of transcribing cells and in biofluids (including blood, urine, and saliva) distant to the transcribing cell.[6,16,17] In the diseased state, transcription of specific mRNAs and miRNAs is altered (suppressed or induced) either in normal immunologic attempts to fight the disease (eg, immune-cell signaling) or by the pathology itself (eg, cancer cell transcripts). Although the exact cellular origin of salivary mRNA is unclear, characterization of mRNA profiles in bodily fluids provides insight into the state of the systems gene transcription network and therefore a reflection of the systems status.

There have been several translational advances in transcriptomic salivary biomarker characterization in the last past few years. Transcriptomic biomarkers for primary Sjögren syndrome have undergone preclinical validation,[18] and mRNA biomarkers for oral squamous cell carcinoma (OSCC) previously discovered in a US cohort[19] have been found to detect disease in a cohort of different ethnicity (Serbian).[20] Zhang and others[5] performed discovery and validation of transcriptomic salivary biomarkers for breast cancer, using a *de novo* approach involving mRNA profiling of matched patients and controls using Human Genome U133 Plus 2.0 Array (Affymetrix, Santa Clara, CA, USA) and then preclinical validation in an independent cohort using quantitative (RT)-qPCR.

Applying PRoBE design, Zhang and others[21] identified salivary mRNA biomarkers for pancreatic cancer with 93.3% sensitivity and 100% specificity in distinguishing patients with early stage resectable pancreatic cancer from healthy patients. Importantly, these biomarkers were found to distinguish pancreatic cancer from chronic pancreatitis with high sensitivity and specificity (both 96.7%). Finally, the ovarian cancer salivary transcriptome profile was discovered in a clinical case control study using HG U133 Plus-2.0 Array (Affymetrix) and then validated with qPCR by Lee and others,[22] achieving 85.7% sensitivity and 91.4% specificity.

miRNA) are short (19–25 nucleotides in length) RNA transcripts that function as posttranscriptional regulators as part of the RNA induced silencing complex of protein synthesis by binding to complementary sequences on the target mRNA transcripts (for an extensive review, see Bartel[23,24]). These novel RNAs have been well-characterized and found to play roles in cell growth, differentiation, apoptosis, pathogen-host interactions, as well as stress responses and immune function and are found in saliva[25] (for extensive review see Lu and others,[25] Zeng,[26] and Stadler and Ruohola-Baker[27]).

miRNAs in several cancer cell types have been demonstrated to be differentially expressed compared with normal cells with expression fold changes in the tens to hundreds. Cancer cell changes in mRNA are comparatively small, and cancer miRNAs also appear to cluster solid tumor-types more truly than mRNA.[25,28,29] These properties make miRNA cancer biomarkers very powerful, and it is likely that miRNA will become of great clinical use in salivary diagnostics. Park and others[6] found two miRNAs (miR-125a and miR-200a) that had significantly reduced levels in the saliva of oral cancer patients compared to controls and found that these two markers could be used for detecting OSCC. Liu and others[30] recently discovered that salivary miRNA-31 (miR-31) was significantly elevated in patients with oral cancer at all stages of disease and tumor size. This study also determined that salivary miR-31 was more abundant than blood miR-31, indicating oral tumor origin of this biomarker.

Salivary Proteome

The proteome is the protein complement of the transcriptome and genome and thus a link between cell biology and genetics. The discovery and study of proteins, their amino acid sequences, and their mRNA precursors have been invaluable to the life sciences. Although mRNA expression profiling may provide a more direct representation of gene-transcription, protein expression profiling provides a more direct representation of cellular function because expression of proteins is not only controlled at the transcription level but also at the level of translation.[31,32] Proteomic analysis of body fluids is therefore an accurate reflection of the life and function, disease and death of cells, organs, and the organism.

The human salivary proteome has been well characterized. Several different classes of salivary protein biomarkers that can assist with diagnosis have been reported. Endothelin-1, a vasoconstrictor, was reported as a potential biomarker for OSCC development in oral lichen planus patients.[33] Interleukin-8 (IL-8), interleukin-1"β (IL-1"β), and glycoprotein M2BP have been reported as salivary

biomarkers for oral cancer,[34-36] and immunoglobulins have long since been described as salivary biomarkers for HIV.[37,38]

Salivary Metabolome

The metabolome is the complement of small-molecule metabolites (metabolic intermediates, signalling molecules, and secondary metabolites) that can be measured in a bio-sample. Just as the transcriptome and proteome, the metabolome changes continually and is a dynamic picture of cellular and organ functions, reflecting gene and protein expression as well as environment. Metabolomic investigations generate quantitative data for many metabolites to elucidate metabolic dynamics related to disease state and drug exposure.[39]

The Salivary Metabolome in Periodontal Disease and Systemic Oncology—Using capillary electrophoresis time-of-flight mass spectrometry, our laboratory identified 57 principal metabolites that can accurately predict the probability of being affected by oral cancer, breast cancer, pancreatic cancer, and periodontal disease. Multiple logistic regression models yielded area under the receiver operating characteristic curves (AUCs) of 0.865 for oral cancer, 0.973 for breast cancer, 0.993 for pancreatic cancer, and 0.969 for periodontal diseases, and cross-validation accuracy AUCs of 0.810, 0.881, 0.994, and 0.954, respectively.[7] Wei and others[8] demonstrated, using ultra-performance liquid chromatography coupled with quadruple time-of-flight mass spectrometry, that a combination of three salivary metabolic biomarkers (valine, lactic acid, and phenylalanine) could discriminate OSCC from controls and oral leukoplakia, with accuracy of 0.89 and 0.97, sensitivity of 86.5% and 94.6%, specificity of 82.4% and 84.4%, and positive predictive values of 81.6% and 87.5%, respectively. These reports clearly demonstrate the utility of salivary metabolomic biomarkers for disease detection and the potential for clinical implementation.

Salivary Microbiome

The human oral microbe identification microarray (HOMIM) is a recent development, where an oligonucleotide- microarray, based on the 16S rRNA, has allowed for the profiling and monitoring of changes in the oral microbiota.[40] Alterations in the bacterial profile of the oral cavity have been found to be associated with several diseases: pancreatic cancer,[41] oral cancer,[42] lung cancer,[43,44] colonic neoplasia and extracolonic malignancy,[45] cardiovascular disease and cerebrovascular disease,[46-48] and preterm birth.[49] These changes are reflections of systemic diseases and therefore offer another salivary diagnostic alphabet.

SALIVARY DIAGNOSTICS

The discovery of salivary biomarkers and their reflection of the system's health status, has naturally led to their exploration as tools for disease detection and diagnostics. Detecting disease at an early stage is imperative to success of therapy in most cancers. In the future, salivary diagnostics will aid in rapid and easily accessible clinical diagnosis, thus potentially allowing for more cases being detected at early stages and thereby decreasing mortality caused by cancers.

Oral Disease Detection

Since the 1990s, salivary diagnostics has been developed for oral disease to monitor periodontal disease and caries risk assessment.[50-52] In 2009, Gursoy and others[53] compared the concentration of a select subset of salivary proteins (elastase, lactate dehydrogenase, IL-1"β, interleukin-6 [IL-6], and tumor necrosis factor-α) and the presence of five pathogens (*Aggregatibacter actinomycetemcomitans*, *Porphyromonas gingivalis*, *Prevotella intermedia*, *Tannerella forsythia*, and *Treponema denticola*) in patients with advanced periodontal disease and healthy controls. This study reported an association of salivary IL-1"β and multiple oral pathogens with periodontitis. In a study on dysplastic oral leukoplakia in relation to tobacco habits and periodontitis, Sharma and others[54] found that increasing IL-6 levels correlated with increasing severity of dysplasia, indicating a potential for not only salivary biomarker-based detection of disease, but also determination of disease stage.

Discovery and validation of salivary diagnostic markers for oral cancer detection have been confirmed to be applicable across ethnic backgrounds to discriminate oral cancer from cancer-free subjects.[20] The potential of many different types of salivary biomarkers (metabolomic, transcriptomic [miRNA and mRNA], proteomic, and microbiome) have been described for oral cancer.[6,7,30,33,42]

Systemic Disease Detection

More recently, the advances in biotechnology, salivary diagnostics, genomics, and proteomics have extended the range of salivary diagnostics to systemic disease monitoring. Possibly the most attractive attribute of salivary diagnostics is that of its implica-

tion in systemic disease detection, due to the invasive nature of current clinical practice and in some cases poor accuracy of current standard methods.[5]

Currently, available tests for systemic cancers include a multitude of invasive and/or expensive procedures (magnetic resonance imaging, biopsy, X-ray, computed tomography, exfoliated cytology, positron emission tomography, barium swallow and endoscopy). This, combined with severely decreased prognosis associated with late diagnosis, makes the prospect of salivary diagnostics particularly valuable in oncology.

Pancreatic Cancer

A significant milestone in salivary diagnostics was reached by Zhang and others[21] with the characterization of a salivary transcriptome profile that discriminates patients with early stage resectable pancreatic cancer from cancer-free subjects. The use of the salivary transcriptome for detecting pancreatic cancer was also found to outperform currently used blood-based tests in terms of sensitivity and specificity. Farrell and others[41] further showed that variations in oral microbiota could be used to detect pancreatic cancer as well as pancreatitis.

Lung Cancer

The most frequent cause of cancer-related death in men and the second most common cause in women is lung cancer. Currently, lung cancer may be detected with chest X-ray and computed tomography, and diagnosis is confirmed via biopsy. As such, the current ability to detect lung cancer is limited by stage of disease progression, and more than 75% of cases are diagnosed in the late stages, significantly reducing survival rate. Lung cancer, an often asymptomatic or nonspecific symptom presenting disease, represents one of the greatest needs for salivary biomarkers-based diagnostics today.

Using two-dimensional difference gel electrophoresis and mass spectrometry, Hua and others[55] performed proteomic analysis of saliva of patients with lung cancer. Sixteen candidate lung cancer biomarkers were discovered and further verified in the discovery sample, a prevalidation sample set, and in a lung cancer cell line. Three candidate markers achieved 88.5% sensitivity and 92.3% specificity with an AUC of 0.90.

Breast and Ovarian Cancers

Today, breast cancer detection relies on physical examination and imaging techniques. Emerging technologies, such as molecular analysis of nipple fluid aspirate and ductal lavage,[56,57] may provide improved accuracy and potentially earlier diagnosis but are invasive and therefore limited to high-risk patients. Recently, our laboratory reported the discovery and preclinical validation of transcriptomic and proteomic salivary biomarkers with diagnostic power for breast cancer[5] as well saliva biomarkers for ovarian cancer, the most deadly gynecologic cancer.[22]

Other Systemic Diseases

Not only is salivary diagnostics proving useful in detecting systemic cancers, salivary biomarkers for autoimmune diseases, microbial systemic infections, and diabetes have also been described recently,[2,18,37,58] expanding the potential clinical spectrum of salivary diagnostics.

CONCLUSION: SALIVA DIAGNOSTICS IN THE DENTAL OFFICE FOR CHAIR-SIDE DETECTION OF ORAL AND SYSTEMIC DISEASES

Currently, the decision to use available accurate diagnostic methodology for many diseases is made based on symptoms reported by the patient and the clinical observations made by the physician. As such, the final diagnosis is often negative, thus imposing unnecessary burdens on hospitals and increased waiting time for patients. Salivary diagnostics is intended to provide accessible noninvasive primary testing for diseases and will greatly reduce the burden on hospitals, especially relating to complex, invasive diagnostic procedures yielding negative diagnosis and will significantly reduce the number of "unnecessary" invasive procedures being performed today. These technologies will enable the chair-side detection of oral and systemic disease in the dental office[59] with the potential to advance dentistry into primary healthcare (Figure 1).

Any diagnostic fluid requires proper processing, storage, and transport conditions especially suited to the analytes of interest and the milieu that they are found in to maintain the integrity of the analyte(s). The noninvasive and easily collected nature of saliva will allow for wide use and ultimate ease of access, only if analytes can be cost efficiently and easily stabilized for storage until analysis or transport to laboratory. With the expanding reports of diagnostic-value salivary biomarker profiles, it is timely that technologies for saliva sampling and stabilization as well as point-of-care technology are being developed. Our laboratory has developed methods of stabilizing whole saliva at room temperature without centrifu-

gation by simple addition of a stabilizing reagent as previously reported,[60,61] which will extend the usefulness of salivary diagnostics. A potential of saliva-based diagnostics is noninvasive detection of disease without specially trained professionals by use of point-of-care technology that can allow for sensitive and specific detection of diagnostic biomarkers without any processing of the saliva sample. Developed by our laboratory and supported by the National Institute of Dental and Craniofacial Research, the oral fluid nanosensor test (Figure 2) is a robust portable electrochemical multiplex sensor platform, which is able to detect nucleic acids without amplification of analyte transcripts.[62] Such technology will provide easier access to disease detection in remote and impoverished regions and reduce burdens on health systems worldwide.

Conflict of Interest

The author of this manuscript certifies that he 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 except for the following: Dr Wong reports proprietary interests in RNAmeTRIX Inc, a molecular diagnostic company, and personal interests in intellectual properties in salivary diagnostics technologies.

(Accepted 16 April 2012)

REFERENCES

1. Gorr SU (2012) Antimicrobial peptides in periodontal innate defense *Frontiers of Oral Biology* **15** 84-98.
2. Zaka-Ur-Rab Z, Abqari S, Shahab T, Islam N, & Shukla I (2012) Evaluation of salivary anti-Salmonella typhi lipopolysaccharide IgA ELISA for serodiagnosis of typhoid fever in children *Archives of Disease in Childhood* **97(3)** 236-238.
3. Denny P, Hagen FK, Hardt M, Liao L, Yan W, Arellanno M, Bassilian S, Bedi GS, Boontheung P, Cociorva D, Delahunty CM, Denny T, Dunsmore J, Faull KF, Gilligan J, Gonzalez-Begne M, Halgand F, Hall SC, Han X, Henson B, Hewel J, Hu S, Jeffrey S, Jiang J, Loo JA, Ogorzalek Loo RR, Malamud D, Melvin JE, Miroshnychenko O, Navazesh M, Niles R, Park SK, Prakobphol A, Ramachandran P, Richert M, Robinson S, Sondej M, Souda P, Sullivan MA, Takashima J, Than S, Wang J, Whitelegge JP, Witkowska HE, Wolinsky L, Xie Y, Xu T, Yu W, Ytterberg J, Wong DT, Yates JR 3rd, & Fisher SJ (2008) The proteomes of human parotid and submandibular/sublingual gland salivas collected as the ductal secretions *Journal of Proteome Research* **7(5)** 1994-2006.
4. Li Y, Zhou X, St. John MAR, & Wong DTW (2004) RNA Profiling of cell-free saliva using microarray technology *Journal of Dental Research* **83(3)** 199-203.
5. Zhang L, Xiao H, Karlan S, Zhou H, Gross J, Elashoff D, Akin D, Yan X, Chia D, Karlan B, & Wong DT (2010) Discovery and preclinical validation of salivary transcriptomic and proteomic biomarkers for the non-invasive detection of breast cancer *PLoS ONE* **5(12)** e15573.
6. Park NJ, Zhou H, Elashoff D, Henson BS, Kastratovic DA, Abemayor E, & Wong DT (2009) Salivary microRNA: Discovery, characterization, and clinical utility for oral cancer detection *Clinical Cancer Research* **15(17)** 5473-5477.
7. Sugimoto M, Wong DT, Hirayama A, Soga T, & Tomita M (2010) Capillary electrophoresis mass spectrometry-based saliva metabolomics identified oral, breast and pancreatic cancer-specific profiles *Metabolomics: Official Journal of the Metabolomic Society* **6(1)** 78-95.
8. Wei J, Xie G, Zhou Z, Shi P, Qiu Y, Zheng X, Chen T, Su M, Zhao A, & Jia W (2010) Salivary metabolite signatures of oral cancer and leukoplakia *International Journal of Cancer. Journal International du Cancer* Dec 28 [Epub ahead of print].
9. Hu Z, Zimmermann BG, Zhou H, Wang J, Henson BS, Yu W, Elashoff D, Krupp G, & Wong DT (2008) Exon-level expression profiling: A comprehensive transcriptome analysis of oral fluids *Clinical Chemistry* **54(5)** 824-832.
10. Streckfus C, & Bigler L (2005) The use of soluble, salivary c-erbB-2 for the detection and post-operative follow-up of breast cancer in women: The results of a five-year translational research study *Advances in Dental Research* **18(1)** 17-24.
11. Streckfus C, Bigler L, Dellinger T, Dai X, Kingman A, & Thigpen JT (2000) The presence of soluble c-erbB-2 in saliva and serum among women with breast carcinoma: A preliminary study *Clinical Cancer Research* **6(6)** 2363-2370.
12. Navarro MA, Mesia R, Diez-Gibert O, Rueda A, Ojeda B, & Alonso MC (1997) Epidermal growth factor in plasma and saliva of patients with active breast cancer and breast cancer patients in follow-up compared with healthy women *Breast Cancer Research and Treatment* **42(1)** 83-86.
13. Brooks MN, Wang J, Li Y, Zhang R, Elashoff D, & Wong DT (2008) Salivary protein factors are elevated in breast cancer patients *Molecular Medicine Reports* **1(3)** 375-378.
14. Bigler LR, Streckfus CF, Copeland L, Burns R, Dai X, Kuhn M, Martin P, & Bigler SA (2002) The potential use of saliva to detect recurrence of disease in women with breast carcinoma *Journal of Oral Pathology & Medicine* **31(7)** 421-431.
15. Pepe MS, Feng Z, Janes H, Bossuyt PM, & Potter JD (2008) Pivotal evaluation of the accuracy of a biomarker used for classification or prediction: Standards for study design *Journal of the National Cancer Institute* **100(20)** 1432-1438.
16. Garcia JM, Garcia V, Peña C, Domínguez G, Silva J, Diaz R, Espinosa P, Citores MJ, Collado M, & Bonilla F (2008) Extracellular plasma RNA from colon cancer patients is confined in a vesicle-like structure and is mRNA-enriched *RNA* **14(7)** 1424-1432.
17. Tinzl M, Marberger M, Horvath S, & Chypre C (2004) DD3PCA3 RNA analysis in urine—A new perspective for detecting prostate cancer *European Urology* **46(2)** 182-186; discussion 87.

18. Hu S, Gao K, Pollard R, Arellano-Garcia M, Zhou H, Zhang L, Elashoff D, Kallenberg CG, Vissink A, & Wong DT (2010) Preclinical validation of salivary biomarkers for primary Sjögren's syndrome *Arthritis Care & Research* **62(11)** 1633-1638.

19. Li Y (2004) Salivary transcriptome diagnostics for oral cancer detection *Clinical Cancer Research* **10(24)** 8442-8450.

20. Brinkmann O, Kastratovic DA, Dimitrijevic MV, Konstantinovic VS, Jelovac DB, Antic J, Nesic VS, Markovic SZ, Martinovic ZR, Akin D, Spielmann N, Zhou H, & Wong DT (2011) Oral squamous cell carcinoma detection by salivary biomarkers in a Serbian population *Oral Oncology* **47(1)** 51-55.

21. Zhang L, Farrell JJ, Zhou H, Elashoff D, Akin D, Park NH, Chia D, & Wong DT (2010) Salivary transcriptomic biomarkers for detection of resectable pancreatic cancer *Gastroenterology* **138(3)** 949-957.e7.

22. Lee Y-H, Kim JH, Zhou H, Kim BW, & Wong DT (2012) Salivary transcriptomic biomarkers for detection of ovarian cancer: For serous papillary adenocarcinoma *Journal of Molecular Medicine* **90(4)** 427-434.

23. Bartel DP (2009) MicroRNAs: Target recognition and regulatory functions *Cell* **136(2)** 215-233.

24. Bartel DP (2004) MicroRNAs: Genomics, biogenesis, mechanism, and function *Cell* **116(2)** 281-297.

25. Lu J, Getz G, Miska EA, Alvarez-Saavedra E, Lamb J, Peck D, Sweet-Cordero A, Ebert BL, Mak RH, Ferrando AA, Downing JR, Jacks T, Horvitz HR, & Golub TR (2005) MicroRNA expression profiles classify human cancers *Nature* **435(7043)** 834-838.

26. Zeng Y (2006) Principles of micro-RNA production and maturation *Oncogene* **25(46)** 6156-6162.

27. Stadler BM, & Ruohola-Baker H (2008) Small RNAs: Keeping stem cells in line *Cell* **132(4)** 563-566.

28. Mi S, Lu J, Sun M, Li Z, Zhang H, Neilly MB, Wang Y, Qian Z, Jin J, Zhang Y, Bohlander SK, Le Beau MM, Larson RA, Golub TR, Rowley JD, & Chen J (2007) MicroRNA expression signatures accurately discriminate acute lymphoblastic leukemia from acute myeloid leukemia *Proceedings of the National Academy of Sciences of the United States of America* **104(50)** 19971-19976.

29. Jiang J, Lee EJ, Gusev Y, & Schmittgen TD (2005) Real-time expression profiling of microRNA precursors in human cancer cell lines *Nucleic Acids Research* **33(17)** 5394-5403.

30. Liu CJ, Lin SC, Yang CC, Cheng HW, & Chang KW (2012) Exploiting salivary miR-31 as a clinical biomarker of oral squamous cell carcinoma *Head & Neck* **34(2)** 219-224.

31. Wilkie GS, Dickson KS, & Gray NK (2003) Regulation of mRNA translation by 5′- and 3′-UTR-binding factors *Trends in Biochemical Sciences* **28(4)** 182-188.

32. Padmanabhan K, & Richter JD (2006) Regulated Pumilio-2 binding controls RINGO/Spy mRNA translation and CPEB activation *Genes & Development* **20(2)** 199-209.

33. Cheng YS, Rees T, Jordan L, Oxford L, O'Brien J, Chen HS, & Wong D (2011) Salivary endothelin-1 potential for detecting oral cancer in patients with oral lichen planus or oral cancer in remission *Oral Oncology* **47(12)** 1122-1126.

34. St John MA, Li Y, Zhou X, Denny P, Ho CM, Montemagno C, Shi W, Qi F, Wu B, Sinha U, Jordan R, Wolinsky L, Park NH, Liu H, Abemayor E, & Wong DT (2004) Interleukin 6 and interleukin 8 as potential biomarkers for oral cavity and oropharyngeal squamous cell carcinoma *Archives of Otolaryngology–Head & Neck Surgery* **130(8)** 929-935.

35. Wei F, Patel P, Liao W, Chaudhry K, Zhang L, Arellano-Garcia M, Hu S, Elashoff D, Zhou H, Shukla S, Shah F, Ho CM, & Wong DT (2009) Electrochemical sensor for multiplex biomarkers detection *Clinical Cancer Research* **15(13)** 4446-4452.

36. Elashoff D, Zhou H, Reiss JK, Wang J, Xiao H, Henson B, Hu S, Arellano M, Sinha U, Le A, Messadi D, Wang M, Nabili V, Lingen M, Morris D, Randolph T, Feng Z, Akin D, Kastratovic DA, Chia D, Abemayor E, & Wong DT (2012) Pre-validation of salivary biomarkers for oral cancer detection *Cancer Epidemiology, Biomarkers and Prevention* **21(4)** 664-667.

37. Emmons W (1997) Accuracy of oral specimen testing for human immunodeficiency virus *American Journal of Medicine* **102(4A)** 15-20.

38. Malamud D (1997) Oral diagnostic testing for detecting human immunodeficiency virus-1 antibodies: A technology whose time has come *American Journal of Medicine* **102(4A)** 9-14.

39. Spielmann N, & Wong DT (2011) Saliva: Diagnostics and therapeutic perspectives *Oral Diseases* **17(4)** 345-354.

40. Huyghe A, Francois P, Charbonnier Y, Tangomo-Bento M, Bonetti EJ, Paster BJ, Bolivar I, Baratti-Mayer D, Pittet D, & Schrenzel J; Geneva Study Group on Noma (GESNOMA) (2008) Novel microarray design strategy to study complex bacterial communities *Applied and Environmental Microbiology* **74(6)** 1876-1885.

41. Farrell JJ, Zhang L, Zhou H, Chia D, Elashoff D, Akin D, Paster BJ, Joshipura K, & Wong DT (2012) Variations of oral microbiota are associated with pancreatic diseases including pancreatic cancer *Gut* **61(4)** 582-588.

42. Mager DL, Haffajee AD, Devlin PM, Norris CM, Posner MR, & Goodson JM (2005) The salivary microbiota as a diagnostic indicator of oral cancer: A descriptive, non-randomized study of cancer-free and oral squamous cell carcinoma subjects *Journal of Translational Medicine* **3** 27.

43. Anttila T, Koskela P, Leinonen M, Laukkanen P, Hakulinen T, Lehtinen M, Pukkala E, Paavonen J, & Saikku P (2003) *Chlamydia pneumoniae* infection and the risk of female early-onset lung cancer *International Journal of Cancer. Journal International du Cancer* **107(4)** 681-682.

44. Koyi H, Branden E, Gnarpe J, Gnarpe H, & Steen B (2001) An association between chronic infection with *Chlamydia pneumoniae* and lung cancer. A prospective 2-year study *APMIS: Acta Pathologica, Microbiologica, et Immunologica Scandinavica* **109(9)** 572-580.

45. Gold JS, Bayar S, & Salem RR (2004) Association of *Streptococcus bovis* bacteremia with colonic neoplasia and extracolonic malignancy *Archives of Surgery* **139(7)** 760-765.

46. Joshipura KJ, Douglass CW, & Willett WC (1998) Possible explanations for the tooth loss and cardiovascular disease relationship *Annals of Periodontology / the American Academy of Periodontology* **3(1)** 175-183.

47. Meurman JH, Sanz M, & Janket SJ (2004) Oral health, atherosclerosis, and cardiovascular disease *Critical Reviews in Oral Biology and Medicine* **15(6)** 403-413.

48. Morrison HI, Ellison LF, & Taylor GW (1999) Periodontal disease and risk of fatal coronary heart and cerebrovascular diseases *Journal of Cardiovascular Risk* **6(1)** 7-11.

49. Goldenberg RL, & Culhane JF (2006) Preterm birth and periodontal disease *New England Journal of Medicine* **355(18)** 1925-1927.

50. Kornman KS, Crane A, Wang HY, di Giovine FS, Newman MG, Pirk FW, Wilson TG Jr, Higginbottom FL, & Duff GW (1997) The interleukin-1 genotype as a severity factor in adult periodontal disease *Journal of Clinical Periodontology* **24(1)** 72-77.

51. Socransky SS, Haffajee AD, Smith C, & Duff GW (2000) Microbiological parameters associated with IL-1 gene polymorphisms in periodontitis patients *Journal of Clinical Periodontology* **27(11)** 810-818.

52. Baughan LW, Robertello FJ, Sarrett DC, Denny PA, & Denny PC (2000) Salivary mucin as related to oral *Streptococcus mutans* in elderly people *Oral Microbiology and Immunology* **15(1)** 10-14.

53. Gursoy UK, Könönen E, Uitto VJ, Pussinen PJ, Hyvärinen K, Suominen-Taipale L, & Knuuttila M (2009) Salivary interleukin-1beta concentration and the presence of multiple pathogens in periodontitis *Journal of Clinical Periodontology* **36(11)** 922-927.

54. Sharma M, Bairy I, Pai K, Satyamoorthy K, Prasad S, Berkovitz B, & Radhakrishnan R (2011) Salivary IL-6 levels in oral leukoplakia with dysplasia and its clinical relevance to tobacco habits and periodontitis *Clinical Oral Investigations* **15(5)** 705-714.

55. Hua X, Zhang L, Zhou H, Lee JM, Garon EB, & Wong DT (2012) Proteomic analysis of human saliva from lung cancer patients using two-dimentional difference gel elecrtophoresis and mass spectometry *Molecular and Cell Proteomics: MCP* **11(2)** M111.012112.

56. Sauter ER (2005) Analysis of nipple aspirate fluid for diagnosis of breast cancer: An alternative to invasive biopsy *Expert Review of Molecular Diagnostics* **5(6)** 873-881.

57. Li J, Zhao J, Yu X, Lange J, Kuerer H, Krishnamurthy S, Schilling E, Khan SA, Sukumar S, & Chan DW (2005) Identification of biomarkers for breast cancer in nipple aspiration and ductal lavage fluid *Clinical Cancer Research* **11(23)** 8312-8320.

58. Rao PV, Reddy AP, Lu X, Dasari S, Krishnaprasad A, Biggs E, Roberts CT, & Nagalla SR (2009) Proteomic identification of salivary biomarkers of type-2 diabetes *Journal of Proteome Research* **8(1)** 239-245.

59. Greenberg BL, Glick M, Frantsve-Hawley J, & Kantor ML (2010) Dentists' attitudes toward chairside screening for medical conditions *Journal of the American Dental Association* **141(1)** 52-62.

60. Jiang J, Park NJ, Hu S, & Wong DT (2009) A universal pre-analytic solution for concurrent stabilization of salivary proteins, RNA and DNA at ambient temperature *Archives of Oral Biology* **54(3)** 268-273.

61. Park NJ, Yu T, Nabili V, Brinkman BM, Henry S, Wang J, & Wong DT (2006) RNAprotect saliva: An optimal room-temperature stabilization reagent for the salivary transcriptome *Clinical Chemistry* **52(12)** 2303-2304.

62. Gau V, & Wong D (2007) Oral fluid nanosensor test (OFNASET) with advanced electrochemical-based molecular analysis platform *Annals of the New York Academy of Sciences* **1098** 401-410.

©Operative Dentistry, 2012, **37-6,** 571-575

Clinical Tech/Case Report

Periapical Healing After Direct Pulp Capping With Calcium-enriched Mixture Cement: A Case Report

S Asgary • A Nosrat • N Homayounfar

Clinical Relevance

This article shows the healing potential of pulp tissue in vital caries exposed mature permanent teeth; and also represents the "direct pulp capping" technique as a valuable treatment procedure to save pulp tissue.

SUMMARY

This article describes a successful direct pulp capping of a mature symptomatic mandibular second molar in a 14-year-old girl. The patient was referred with sensitivity to cold beverages and pain on chewing on the second left mandibular molar. Clinical examinations revealed extensive coronal caries and sensitivity to percussion. Radiographically, the tooth was mature and had a widened apical periodontal ligament (PDL) and a narrow periapical lesion. The concluding diagnosis was symptomatic irreversible pulpitis with symptomatic apical periodontitis. Treatment included caries removal under rubber dam isolation, capping of exposure sites with calcium-enriched mixture (CEM) cement, and permanent coronal restoration. At three-, 10-, and 15-month follow-up, the tooth was functional, had normal response to cold test, and did not have sensitivity to percussion. The PDL space regained its normal width, and the periapical lesion healed.

*Saeed Asgary, DDS, MS, professor, Iranian Center for Endodontic Research, Dental Research Center, School of Dentistry, Shahid Beheshti University of Medical Sciences, Tehran, Iran

Ali Nosrat, DDS, MS, assistant professor, Iranian Center for Endodontic Research, Shahid Beheshti University of Medical Sciences, Tehran, Iran

Negar Homayounfar, DDS, MS, graduate student, Department of Oral Biology, School of Dentistry, University of Washington, Seattle, WA, USA

*Corresponding author: Evin Blvd, Tehran, 1983963113, Iran; e-mail: ansrt2@yahoo.com

DOI: 10-2341/11-417-S

INTRODUCTION

Vital pulp therapies are biologically-based treatments in which the main objective is to save pulp health and vitality in carious or traumatic exposures.[1] These treatments include stepwise excavation and indirect pulp capping, direct pulp capping (DPC),[2] and pulpotomies.[3] Although vital pulp therapies are universally accepted in the treatment

of immature teeth, they are controversial in treating carious exposed mature teeth.[4]

DPC is defined as the treatment of an exposed vital pulp by sealing the pulpal wound with a pulp-capping material. The material is directly placed on a mechanical or traumatic exposure to promote pulp healing.[1] Several agents have been used for DPC; among them, calcium hydroxide (CH) and mineral trioxide aggregate (MTA) are the most studied ones. Histological studies on pulps capped with CH revealed that formation of dentinal bridges beneath CH is unpredictable. Besides, these bridges contain tunnel defects, and the underlying pulps are inflamed.[5,6] On the other hand, compared with CH, histological studies on DPC with MTA demonstrate favorable results, including continuous regular dentinal bridge formation along with less pulpal inflammation.[5,6] Dentinal bridges formed beneath MTA after DPC resemble tertiary dentin.[7] Properties such as biocompatibility, sealing ability,[8] and hard-tissue induction potential[5,7] make MTA an ideal biomaterial for vital pulp therapies.

Calcium-enriched mixture (CEM) cement is a water-based, tooth-colored cement containing lime (CaO), phosphorus oxide (P_2O_5), sulfur oxide (SO_3), and silica (SiO_2) as major elements.[9] Because of constant CH release during and after setting, CEM cement has antibacterial properties similar to CH and superior to MTA.[10] Several *in vitro* and *in vivo* studies reveal the similar sealing ability,[11] biocompatibility,[12] and hard-tissue induction potential[13] of CEM cement and MTA. Biological responses (the quality of dentinal bridges and the amount of pulp inflammation) after DPC with CEM cement and MTA were similar and better than CH in dogs' teeth.[13] CEM cement has been used successfully in pulpotomy of inflamed mature[14] and immature[15,16] human teeth. Two recent histological studies on DPC of healthy human teeth demonstrate similar biological responses to CEM cement and MTA.[17,18]

This article describes a successful direct pulp capping of a mature second mandibular molar with symptomatic pulpitis and symptomatic apical periodontitis using CEM cement.

CASE REPORT

A 14-year-old healthy girl was referred with a history of lingering pain on the left posterior mandible. The patient's chief complaint was severe lingering pain with cold beverages and pain on chewing. Clinical examinations showed extensive caries and a cavity in the left mandibular second molar. The tooth was sensitive to percussion but not to palpation. Cold test, using Endo-Frost cold spray (Roeko, Coltene Whaledent, Langenau, Germany), elicited a lingering, long-lasting pain. Radiographic examination demonstrated a mature tooth with an obvious widening of the apical periodontal ligament (PDL) space and a narrow apical radiolucent lesion (Figure 1a). Considering the clinical and radiographic findings, our concluding diagnosis was symptomatic irreversible pulpitis with symptomatic apical periodontitis. After explanation of possible risks of treatment, a written informed consent from the patient's legal guardians was obtained.

After local anesthesia with one vial (1.8 mL) of 2% lidocaine (36 mg) and 1:80,000 epinephrine (Darou Pakhsh, Tehran, Iran) and rubber dam isolation, caries were removed with a diamond fissure bur (Diatech, Heerbrugg, Switzerland) and high-speed hand piece with copious water spray. Two exposure spots were detected on mesiolingual and mesiobuccal pulp horns. Using the same bur and high-speed hand piece, the clinician gently extended the diameter of the exposures to 1–2 mm without entering the pulp. Hemostasis was achieved using NaOCl 5.25% for five minutes. CEM cement powder and liquid (Bionique-Dent, Tehran, Iran) were mixed according to the manufacturer's instructions. CEM cement was placed over the pulp wounds using an amalgam carrier and gently adapted to the pulp wounds and surrounding dentin. An ≈2-mm-thick layer of glass ionomer (Fuji II, Fuji Corporation, Japan) base was placed over the CEM cement, and the tooth was restored permanently with amalgam (SDI gs80, SDI limited, Australia; Figure 1b). The patient was recalled 2 days after treatment. Clinical examinations at this time showed that the tooth was not sensitive to either percussion or palpation, and the patient did not have any complaint about chewing with the tooth.

At three, 10-, and 15-month follow-up, the tooth was functional without any signs/symptoms. A cold test using Endo-Frost cold spray elicited normal response, and the tooth was not sensitive to percussion or palpation. The PDL space regained its normal width, and the apical radiolucent lesion healed (Figure 1c, d).

DISCUSSION

The success rate of DPC in carious pulp exposures in mature and immature human teeth is evaluated in some clinical studies using CH or MTA as pulp-capping agents. A retrospective study on 122 carious exposed teeth capped with either CH or MTA and

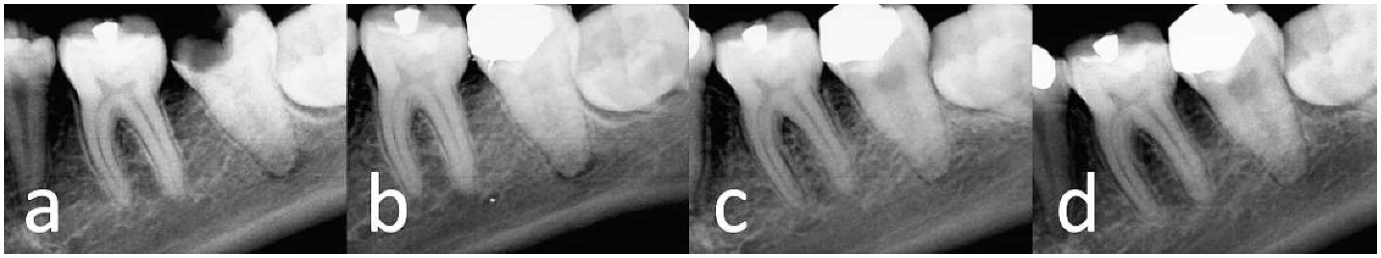


Figure 1. *(a) Preoperative periapical radiograph of a second mandibular left molar in a 14-year-old female patient. The tooth was sensitive to cold and percussion. Note the mesio-occlusal extensive caries and related coronal cavity, apical periodontal ligament (PDL) widening, and narrow radiolucent lesion. (b) Immediately after direct pulp capping with calcium-enriched mixture cement, placement of a glass ionomer base, and permanent coronal restoration. (c) Ten-month follow-up. (d) Fifteen-month follow-up. The tooth was functional without sensitivity to percussion. The PDL space regained its normal width, and the radiolucent lesion healed.*

follow-ups up to 80 months, excluding cases with symptoms of irreversible pulpitis, demonstrated significantly higher clinical and radiographic success rates for the MTA group.[19] A two-year clinical study on DPC with MTA in 30 young carious exposed asymptomatic permanent molars showed 93% vitality.[20] Moreover, a long-term (≈four-year) prospective study on DPC with MTA in 49 immature and mature carious exposed teeth with symptoms of reversible pulpitis demonstrated a 100% and 98% success rate, respectively.[21] The outcomes of these studies concur with histological evidences of favorable pulp responses to capping with MTA and unpredictable responses to capping with CH.[5,6,13]

The biological properties of MTA, including sealing ability, biocompatibility, and hard-tissue induction potential, are attributed to bioactive reactions between calcium ion released from MTA and phosphorus ion in the surrounding tissue fluids. These reactions cause formation of hydroxyapatite crystals.[22] In addition, the sealing ability of MTA increases during storage in phosphate-buffered saline solution, a phenomenon that does not happen in normal saline.[23] However, CEM cement releases calcium and phosphorus ions from indigenous sources and, unlike white MTA, has the ability of inducing hydroxyapatite crystal formation in the absence of environmental phosphorus.[24] Therefore, the comparable features of CEM cement and MTA, such as biocompatibility,[12] sealing ability,[11] and hard-tissue induction potential,[13] and the superior bioactive properties of CEM cement (which is associated with its indigenous phosphorus reservoirs),[24] make CEM cement a potentially suitable biomaterial for DPC. These properties can partly explain the favorable results in the presented case.

Based on histological and clinical evidences, some recent studies show that carious exposed pulps with established irreversible pulpitis in mature and immature teeth maintain their healing potential.[3,14,16,25,26] However, the treatment technique in these studies is full pulpotomy, which often causes negative response to pulp vitality tests in follow-up sessions. By saving coronal pulp in the DPC technique, the clinician has the opportunity to perform pulp vitality tests in addition to periapical and radiographic examinations and to compare them with the baseline data and draw a distinct conclusion about pulp status. Besides, compared with pulpotomy and root canal therapy, DPC is a simpler, less expensive, and more conservative treatment.[27]

Although there are several reports of successful partial pulpotomy of carious/traumatic exposed pulps,[28,29] successful DPC in symptomatic carious exposed mature teeth is a rare finding. The authors believe that to reach this goal, some technical modifications are necessary. Gentle extension of exposure site to ≈1–2 mm diameter may eliminate infected dentinal chips from the pulp surface, increase the contact area between the pulp-capping agent and pulpal wound, and facilitate bioactive reactions in the pulp-pulp capping agent interface.[30] The antibacterial properties of CEM cement[10] and its sustained CH release[24,31] are also other important factors that can keep the pulpal wound bacteria free over time.

As demonstrated in long-term studies, an immediate permanent coronal seal plays an important role in the success rate of DPC treatment.[19,32] Therefore, performing immediate coronal restoration in this case can be another reason for favorable results.

Although studies have shown that the success rate of DPC with CH decreases with the passage of time,[19,32] the success rate of DPC with MTA remains almost constant over time.[19,21] Regarding the similar biological properties of CEM cement and MTA, it is

anticipated that the successful results in the presented case remain constant over time. However, yearly follow-up of the presented case is recommended.

CONCLUSION

Based on biological properties of CEM cement, especially its bioactive and antibacterial properties, this cement might be a suitable biomaterial in direct pulp capping of symptomatic carious exposed mature teeth. However, further clinical studies with longer follow-up periods and larger samples are recommended. While clinical studies are important, histological confirmation using human teeth will provide the ultimate proof of the success of calcium-enriched mixture cement.

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.

(Accepted 6 February 2012)

REFERENCES

1. Bogen G, & Chandler N. (2008) Vital Pulp Therapy. In: Ingle J, Bakland L, Baumgartner J (eds) *Ingle's Endodontics* 6th ed. BC Decker Inc, Hamilton, Canada 1310-1329.
2. Bjørndal L, Reit C, Bruun G, Markvart M, Kjaeldgaard M, Nasman P, Thordrup M, Dige I, Nyvad B, Fransson H, Lager A, Ericson D, Petersson K, Olsson J, Santimano E, Wennstrom A, Winkel P, Gluud C (2010) Treatment of deep caries lesions in adults: randomized clinical trials comparing stepwise vs. direct complete excavation, and direct pulp capping vs. partial pulpotomy *European Journal of Oral Science* **118(3)** 290-297.
3. Witherspoon D, Small J, & Harris H (2006) Mineral trioxide aggregate pulpotomies: a case series outcomes assessment *Journal of American Dental Association* **137(5)** 610-618.
4. Miyashita H, Worthington H, Qualtrogh A, & Plasschaert A (2007) Pulp management for caries in adults: maintaining pulp vitality *Cochrane Database of Systematic Reviews* **18** CD004484.
5. Nair P, Duncan H, Pitt Ford T, & Luder H (2008) Histological, ultrastructural and quantitative investigations on the response of healthy human pulps to experimental capping with mineral trioxide aggregate: a randomized controlled trial *International Endodontic Journal* **41(2)** 128-150.
6. Pitt Ford T, Torabinejad M, Abedi H, Bakland L, & Kariyawasam S (1996) Using mineral trioxide aggregate as a pulp-capping material. *Journal of American Dental Association* **127(10)** 1491-1494.
7. Andelin W, Shabahang S, Wright K, & Torabinejad M (2003) Identification of hard tissue after experimental pulp capping using Dentin Sialoprotein (DSP) as a marker *Journal of Endodontics* **29(10)** 646-650.
8. Torabinejad M, & Parirokh M (2010) Mineral trioxide aggregate: a comprehensive literature review—part II: leakage and biocompatibility investigations *Journal of Endodontics* **36(2)** 190-202.
9. Asgary S, Shahabi S, Jafarzadeh T, Amini S, & Kheirieh S (2008) The properties of a new endodontic material *Journal of Endodontics* **34(8)** 990-993.
10. Asgary S, & Kamrani F (2008) Antibacterial effects of five different root canal sealing materials *Journal of Oral Science* **50(4)** 469-474.
11. Asgary S, Eghbal M, & Parirokh M (2008) Sealing ability of a novel endodontic cement as a root-end filling material *Journal of Biomedical Materials Research* **87(3)** 706-709.
12. Parirokh M, Mirsoltani B, Raoof M, Tabrizchi H, & Haghdoost A (2011) Comparative study of subcutaneous tissue responses to a novel root-end filling material and white and grey mineral trioxide aggregate *International Endodontic Journal* **44(4)** 283-289.
13. Asgary S, Eghbal M, Parirokh M, Ghanavati F, & Rahimi H (2008) A comparative study of histologic response to different pulp capping materials and a novel endodontic cement. *Oral Surgery Oral Medicine Oral Pathology Oral Radiology Endodontology* **106(4)** 609-614.
14. Asgary S, & Ehsani S (2009) Permanent molar pulpotomy with a new endodontic cement: a case series *Journal of Conservative Dentistry* **12(1)** 31-36.
15. Nosrat A, & Asgary S (2010) Apexogenesis treatment with a new endodontic cement: a case report *Journal of Endodontics* **36(5)** 912-914.
16. Nosrat A, & Asgary S (2010) Apexogenesis of a symptomatic permanent molar with calcium enriched mixture. *International Endodontic Journal* **43(10)** 940-944.
17. Zarrabi M, Javidi M, Jafarian A, & Joushan B (2010) Histologic assessment of human pulp response to capping with mineral trioxide aggregate and a novel endodontic cement *Journal of Endodontics* **36(11)** 1778-1781.
18. Zarrabi MH, Javidi M, Jafarian AH, & Joushan B (2011) Immunohistochemical expression of fibronectin and tenascin in human tooth pulp capped with mineral trioxide aggregate and a novel endodontic cement *Journal of Endodontics* **37(12)** 1613-1618.
19. Mente J, Geletneky B, Ohle M, Koch M, Friedrich Ding P, Wolff D, Dreyhaupt J, Martin N, Staehle H, Pfefferle T (2010) Mineral trioxide aggregate or calcium hydroxide direct pulp capping: an analysis of the clinical treatment outcome *Journal of Endodontics* **36(5)** 806-813.
20. Farsi N, Alamoudi N, Balto K, & Al Mushayt A (2006) Clinical assessment of mineral trioxide aggregate (MTA) as direct pulp capping in young permanent teeth *Journal of Clinical Pediatric Dentistry* **31(2)** 72-76.
21. Bogen G, Kim J, & Bakland L (2008) Direct pulp capping with mineral trioxide aggregate: an observational study *Journal of American Dental Association* **139(3)** 305-315.
22. Sarkar N, Caicedo R, Ritwik P, Moiseyeva R, & Kawashima I (2005) Physicochemical basis of the biologic

properties of mineral trioxide aggregate. *Journal of Endodontics* **3192** 97-100.

23. Parirokh M, Askarifard S, Mansouri S, Haghdoost A, Raoof M, & Torabinejad M (2009) Effect of phosphate buffer saline on coronal leakage of mineral trioxide aggregate. *Journal of Oral Science* **51(2)** 187-191.
24. Asgary S, Eghbal M, Parirokh M, & Ghoddusi J (2009) Effect of two storage solutions on surface topography of two root-end fillings *Australian Endodontic Journal* **35(3)** 147-152.
25. Eghbal M, Asgary S, Baghlue R, Parirokh M, & Ghoddusi J (2009) MTA pulpotomy of human permanent molars with irreversible pulpitis. *Australian Endodontic Journal* **35(1)** 4-8.
26. Chueh L, & Chiang C (2010) Histology of Irreversible pulpitis premolars treated with mineral trioxide aggregate pulpotomy *Operative Dentistry* **35(3)** 370-374.
27. Komabayashi T, & Zhu Q (2010) Innovative endodontic therapy for anti-inflammatory direct pulp capping of permanent teeth with a mature apex. *Oral Surgery Oral Medicine Oral Pathology Oral Radiology Endodontology* **109(5)** e75-e81.
28. Cvek M (1978) A clinical report on partial pulpotomy and capping with calcium hydroxide in permanent incisors with complicated crown fracture *Journal of Endodontics* **4(8)** 232-237.
29. Mejare I, & Cvek M (1993) Partial pulpotomy in young permanent teeth with deep carious lesions *Endodontics and Dental Traumatology* **9(6)** 238-242.
30. Asgary S, & Ahmadyar M (2012) Can miniature pulpotomy procedure improve treatment outcomes of direct pulp capping? *Medical Hypotheses* **78(2)** 283-285.
31. Asgary S, Nosrat A, & Seifi A (2011) Management of inflammatory external root resorption by using calcium-enriched mixture cement: a case report. *Journal of Endodontics* **37(3)** 411-413.
32. Barthel C, Rozenkranz B, Leuenberg A, & Roulet J (2000) Pulp capping of carious exposures: treatment outcome after 5 and 10 years: a retrospective study *Journal of Endodontics* **26(9)** 525-528.

©Operative Dentistry, 2012, **37-6,** 576-583

Fiber-Reinforced Framework in Conjunction with Porcelain Veneers for the Esthetic Replacement of a Congenitally Missing Maxillary Lateral Incisor: A Case Study

PP Benito • RD Trushkowsky • KS Magid
SB David

Clinical Relevance

Fiber reinforced composite can allow for conservative, esthetic, biocompatible and functional restoration of a missing anterior tooth in selective clinical situations.

Pamela P. Benito, DDS, New York University College of Dentistry, resident, NYUCD International Program in Advanced Aesthetic Dentistry, New York, NY, USA

*Richard D Trushkowsky, DDS, FAGD, FICD, FACD, FADM, FPFA, associate clinical professor, Cariology and Comprehensive Care, associate director, International Program in Advanced Aesthetic Dentistry, New York University College of Dentistry, New York, NY, USA

Kenneth S. Magid, DDS, associate clinical professor, Cariology and Comprehensive Care, NYUCD, New York, NY, USA

Steven B. David, DMD, clinical professor, Cariology and Comprehensive Care, director, International Program in Advanced Aesthetic Dentistry, NYUCD, New York, NY, USA

*Corresponding author: Cariology and Comprehensive Care, International Aesthetic Dentistry, New York University 345 East 24th Street , 7W. New York,NY 10010-4086 USA; e-mail: rt587@nyu.edu

DOI: 10.2341/11-200-S

SUMMARY

This article presents a case of a congenitally missing right maxillary lateral incisor and the contralateral incisor with discolored composite resin restorations. The technique of fiber reinforcement in conjunction with porcelain veneers was used to provide a satisfactory outcome for the patient. The key learning points of the article are the following: proper diagnosis, treatment plan and appropriate utilization of materials are mandatory for a successful result.

INTRODUCTION

Replacement of a single tooth in the maxillary anterior region presents exacting esthetic demands.[1]

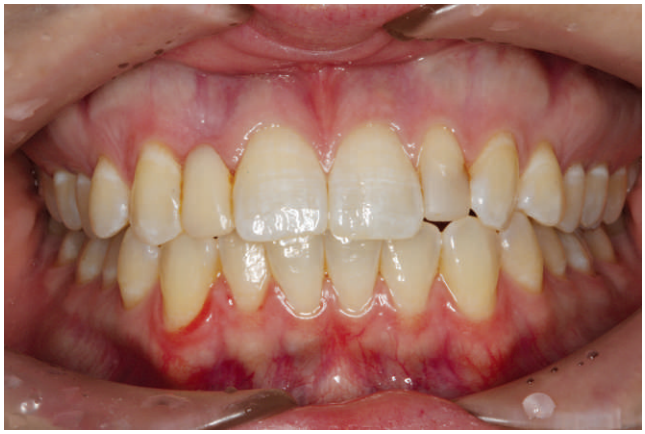


Figure 1. *Labial view prior to treatment.*

The loss of anterior teeth may be a result of trauma or congenital aplasia. The incidence of congenitally missing teeth in the Caucasian population is between 1% and 10%. The maxillary lateral incisor ranks after third molars and mandibular second premolars in frequency of tooth agenesis.[2] The resulting space may be closed by orthodontics, prosthetics, or a combination of both.[3] Orthodontic treatment may often not be the treatment of choice as it may not permit optimal esthetics and function.[3] If a space no longer exists, it can be opened for the fabrication of an implant or a bridge. If the canines are in the lateral position, they can be reshaped *via* additive composite techniques or porcelain veneers so that they will resemble lateral incisors. Prior to making a treatment modality decision, the molar relationship and the esthetic and the functional potential should be considered. Class I occlusion will generally permit treatment of the space by maintenance or opening. If a patient has an Angle Class II malocclusion and exhibits no crowding in the mandible, the molar relationship will be Class II and the premolars will be located in the canine position, and canine substitution can be used to replace the lateral incisor. If a Class I malocclusion exists with mandibular crowding sufficient to require extractions, canine substitution for the lateral incisor can again be used. Class III malocclusions contraindicate orthodontic space closure as the occlusal relationship would be worsened by the therapy.[3]

Also to be considered are the status of the adjacent anterior teeth; the height and width of the ridge; the condition of the buccal plate; periodontal esthetic considerations; the patient's openness to treatment alternatives; patient finances[4]; interdental spacing; treatment time; parafunctional habits[5]; predictability of treatment alternatives; and stability of treatment outcomes. The treatment options for replacing a missing anterior tooth are implant-supported crowns, resin-bonded fixed partial dentures (RBFPDs), conventional fixed partial dentures, and removable partial dentures. Implants and adhesive bridges are preferred as they represent a more conservative treatment option compared to fixed prosthodontics. This is especially true for young patients with large pulp chambers. Implants are often not the ideal choice as tooth loss and congenitally missing teeth frequently result in insufficient bone and soft tissue for esthetic placement of the implant. Guided bone regeneration and grafts may be able to solve these problems. Osseointegration is not the only process that needs to be accomplished. Esthetic integration of the implant-supported restoration with the surrounding hard and soft tissue must also be achieved.[1] That being said, the increased cost and the intricacy of these procedures often preclude this option.[3]

RBFPDs require a minimal amount of abutment modification. However, bridges with metal substructure bonded to the lingual of relatively thin anterior teeth can result in a "graying phenomenon." Opaque cements may reduce this effect but the esthetic result is compromised.[4] To overcome this problem a variety of metal-free bridges have been developed. Some have combined fiber-reinforced composite with ceramics and others are all ceramic, consisting of aluminum oxide, lithium disilicate, or zirconium oxide. This clinical report will describe an esthetic result achieved with the use of feldspathic porcelain veneers in conjunction with a highly filled composite resin (Sculpture, Pentron, Wallingford, CT, USA) that is lab processed utilizing light, heat, vacuum, and a unidirectional fiber (FibreKor®, Pentron).

CASE REPORT

A 28-year-old female patient presented at the Advanced Program for International Dentists in Aesthetic Dentistry at the New York University College of Dentistry. Her chief complaint was the discolored composite bonding on the maxillary left lateral incisor and the poor appearance of the existing three-unit fiber-reinforced bridge replacing the maxillary right lateral incisor. The maxillary right canine and right central incisor were the abutment teeth (Figures 1 through 3). The patient also expressed a desire to have her teeth bleached.

A comprehensive examination was conducted, including a full-mouth radiographic series, caries detection, periodontal probing, and intraoral and

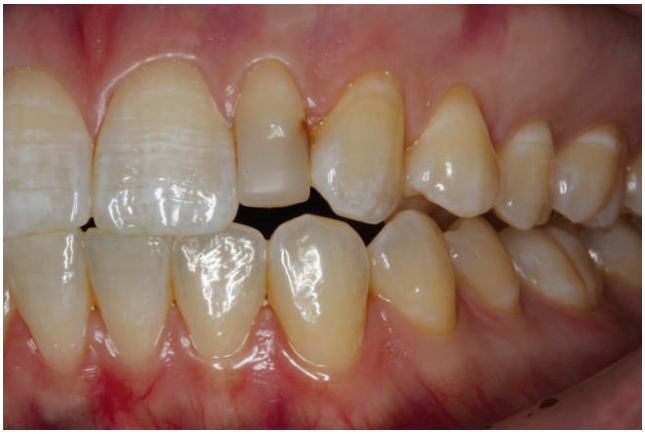


Figure 2. *Left lateral view.*

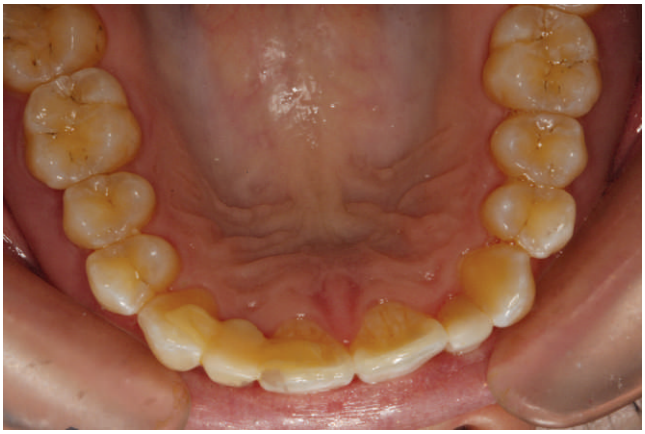


Figure 3. *Occlusal view of previous bridge.*

extraoral soft tissue and temporomandibular joint exam. Palpation directly over the joint while the patient opened and closed the mandible and the extent of mandibular condylar movement were assessed. Findings were within normal limits and therefore were considered noncontributory. The masticatory and cervical muscles were palpated and searched for areas of tenderness or sustained contraction. The palpation was initiated with the sternocleidomastoid, trapezius, and posterior cervical muscles. The masseter at its attachments to the zygomatic arch and angle of the mandible, the temporalis, both in the temporal fossa and intraorally along the ascending ramus of the mandible, were palpated. The medial pterygoid was palpated bimanually. The patient had a shift from centric relation (CR) to maximum intercuspation (MIP) and exhibited canine guidance with a mutually protected occlusion. Upon completion of the examination, a determination was made that the occlusion was physiologic and that the patient should be restored to a position of MIP.

Intraoral and extraoral photographs were taken in order to aid in the esthetic evaluation. Study models were obtained with Reprosil (Dentsply/Caulk, Milford, DE, USA). The Kois Dento-Facial Analyzer System (Panadent, Colton, CA, USA) was used to register and transfer the patient's occlusal plane to the PSH Panadent articulator. This enabled the transfer of the incisal edge position to the laboratory. After ascertaining CR with a leaf gauge, a CR bite was taken with Blu-Bite HP Rigid Fast Set (Henry Schein Inc, Melville, NY, USA). A bite registration was also taken in MIP with the Blu-Bite material.

Facial analysis revealed that the occlusal plane was parallel to the interpupillary line, that the midline relationship of the teeth to the face (Philtrum) was symmetric, and that the lips were also symmetrical to the face. Maxillary tooth exposure at rest was 3 mm and 1 mm in the mandibular arch (Figure 4). The patient also had a high smile line, with an incisal edge that was convex in relation to the lower lip. Upon smiling the patient exhibited 12 teeth.

The potential esthetic benefits of the recommended treatment were demonstrated to the patient using a putty stent obtained from the wax-up of the proposed treatment and Luxatemp® (DMG America, Englewood, NJ, USA) (Figure 5). The patient also expressed a desire to correct her buccal corridors and change the shape and shade of her upper canines and premolars. These comments/concerns were addressed in fabrication of the mock-up. Bleaching trays were also delivered to the patient, and 10% Opalescence bleach (Ultradent Products, South Jordon, UT, USA) was used for two weeks. The maxillary arch was bleached prior to the mandibular

Figure 4. *Incisal edge display.*

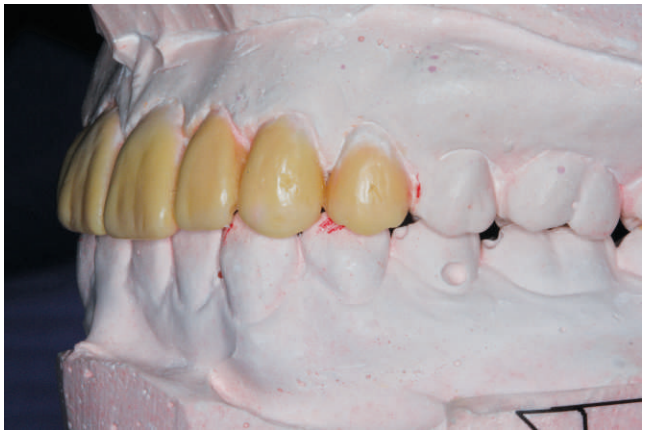


Figure 5. *Wax-up.*

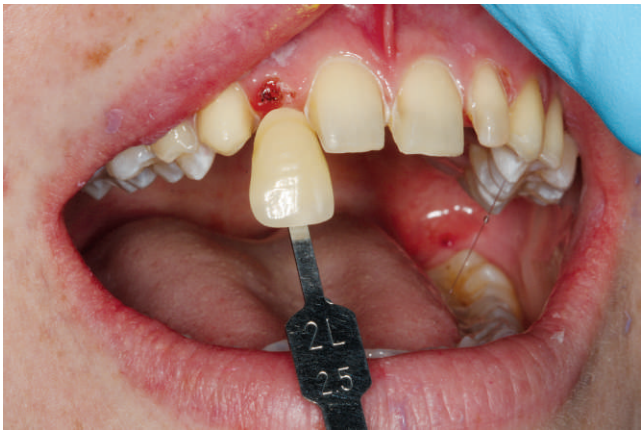


Figure 7. *Shade selection with Vident 3D shade guide and ovate pontic site creation after veneer preparation.*

arch. The patient was satisfied with the result of the tooth whitening, and the final treatment plan was confirmed. As institutional review board approval was not required by any of the procedures to be performed, none was requested.

A metal-free, two-component, resin-bonded bridge, the Encore Bridge®, was selected to replace the congenitally missing upper right lateral incisor and porcelain veneers on teeth 5-12. The framework consists of FibreKor, a fiber-reinforced composite that comes in three forms of fiber strips, and a 16K bar (16,000 individual fibers) that is formulated with a filled resin for increased strength. This is overlaid with Sculpture Plus composite. The resin composite is processed under heat (250°F), light, and pressure in the presence of nitrogen gas (80 psi) to improve the physical properties of the resin. The nitrogen gas excludes oxygen to reduce porosity and increase surface hardness. The lower modulus of elasticity of the framework decreases the stress on the wing/tooth interface so that abutment movement will not cause dislodgement or fracture at the pontic/wing juncture. The pontic is cut back with a veneer preparation to allow for the placement of a porcelain veneer to increase the esthetic potential.

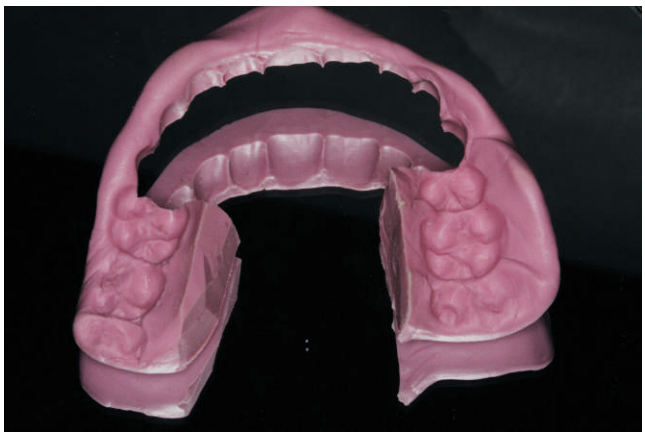


Figure 6. *Putty matrix reduction guide.*

The Encore Bridge, consisting of two components, allows for two paths of insertion. The first is for the lingual framework and the second is for the facial veneer. This is advantageous, as the proximal of the abutment teeth is not narrowed, nor is the appearance changed.[6]

The Encore Bridge can be used to replace any missing maxillary or mandibular anterior tooth if adequate clearance exists or can be created. Ideally, the abutments should not have greater than Class I mobility. There is a greater chance of debonding and fracture with increased tooth mobility. Additional contraindications would include teeth that are too thin to allow proper preparation; teeth that are too short for adequately sized connectors and resin wings; and parafunctional habits.

The teeth were anesthetized with 2 carpules of 1.7 mL of 2% lidocaine with epinephrine 1:100,000. The previous bridge (Nos. 6, 7, and 8) and composite bonding on No. 10 were removed. A Biolase Ezlase™ 940 (Irvine, CA, USA) was used to create an ovate pontic in the No. 7 site and to contour the tissue around No. 10. Keeping the teeth moist, the shade was selected (Figure 6 Aided by the use of a putty matrix reduction guide teeth (Figure 7 Nos. 5, 6, 8, 9, 10, 11, and 12 were prepared for porcelain veneers (Figure 6). GingaTrac™ (Centrix, Shelton, CT, USA) was used to obtain gingival retraction of the multiple preparations. The GingaTrac Matrix material was expressed into a stock plastic tray and placed over

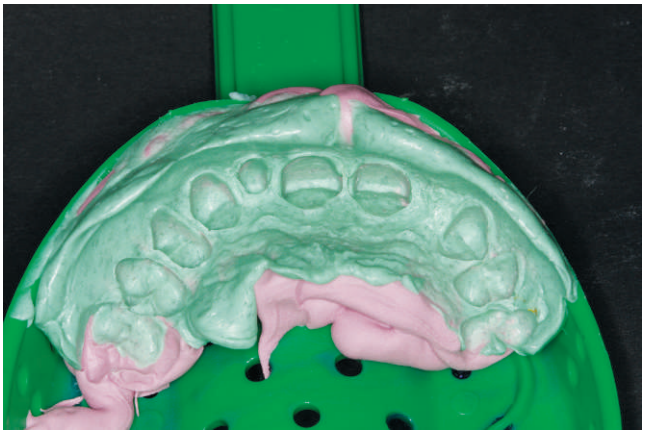


Figure 8. *GingiTrac*.

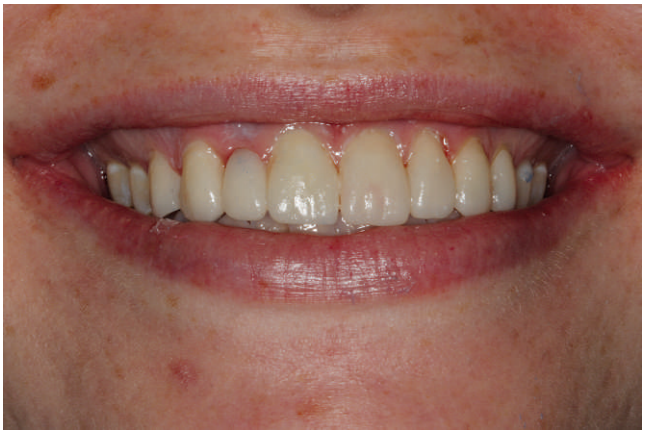


Figure 10. *Luxatemp provisional bridge with high smile*.

the prepared teeth. The now-custom tray was removed from the mouth. The GingaTrac retraction paste was injected around and over the prepared teeth. The customized GingaTrac Matrix was then filled with the GingaTrac retraction material and placed back over the preparations and held in place for three to five minutes with firm biting pressure. The GingaTrac material was removed and complete retraction was verified (Figure 8). Access® (Centrix) impression material was used to obtain the final impression (Figure 9). Luxatemp was used to fabricate a provisional bridge and provisional veneers (Figure 10). Photographs, final maxillary impression, counter impression, bite registration, model of the provisional restorations, and putty incisal guide derived from the provisional were sent to the laboratory. Final shade selection and stump shade were specified. The laboratory was instructed to duplicate the incisal edge position as indicated from the incisal guide and to proportion the teeth as in the provisional. The degree of incisal translucency and texture were also specified. From the materials sent to them, the laboratory was able to fabricate dies and solid models and articulated the case to construct the final restorations (Figure 11).

Upon return from the laboratory, the Encore Bridge and porcelain veneers were inspected on the models. The patient was appointed, the provisional bridge removed, and the Encore Bridge and the porcelain veneers were tried intraorally. The accuracy of the margins was confirmed (Figures 12 through 14). The bridge and the veneers were cemented utilizing Prime and Bond® NT™ (Dentsply/Caulk), a light-cure self-priming dental adhesive, and Variolink II® (Ivoclar/Vivadent, Amherst, NY, USA) translucent shade. The excess cement was removed and the occlusion was verified in MIP and in excursions. A follow-up visit was scheduled for two weeks later so that the gingival response could be evaluated. The final result provided the color,

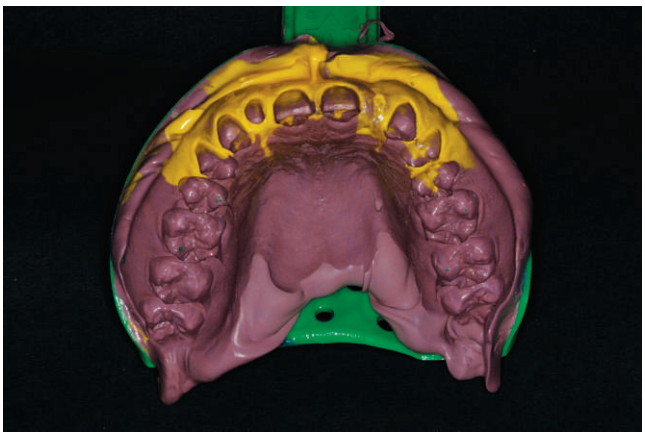


Figure 9. *Final impression*.

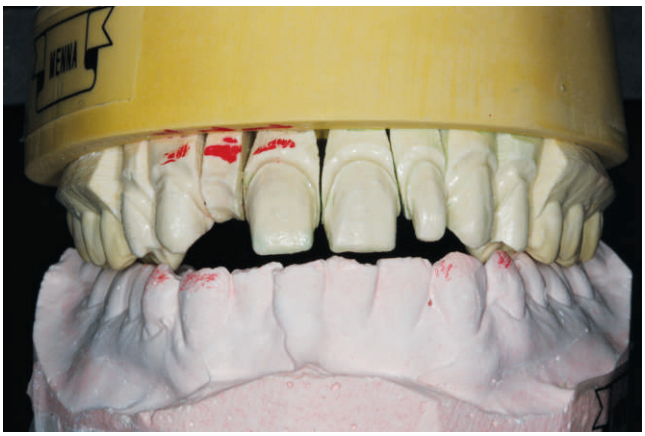


Figure 11. *Die model of prepared teeth*.

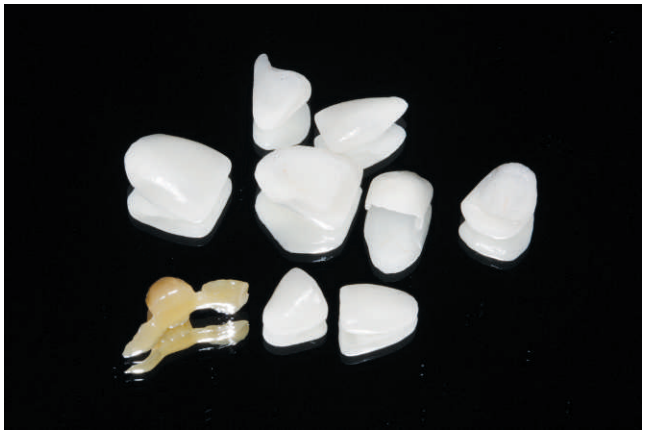


Figure 12. *Framework and porcelain veneers.*

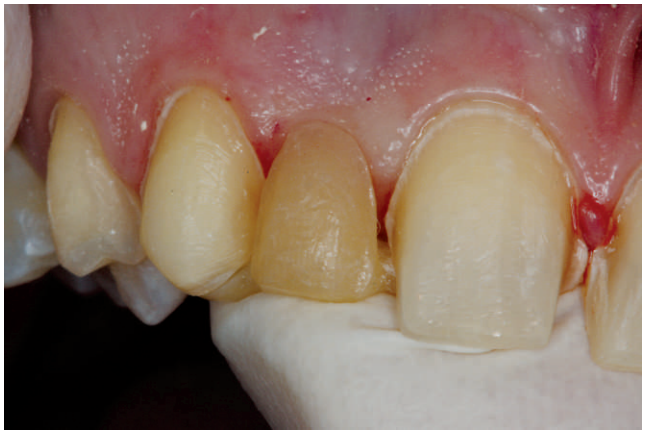


Figure 14. *Try-in of framework.*

shape, and contour that the patient had desired (Figures 15 through 18).

DISCUSSION

RBFPDs have been used as an alternative to implants, conventional metal ceramic bridges, and all ceramic bridges. Unfortunately, despite considerable modifications in design, materials, and preparation design, the survival rates are less than 76% after five years.[7] The literature reports that the failure is usually the result of debonding of the cast-metal framework from the luting cement. On some occasions the luting cement has debonded from the enamel. Detachment of the framework on only one abutment frequently resulted in extensive decay on that abutment. It is believed that the rigidity of the cast framework contributes to its detachment from the bonded surface. This is a consequence of the mobility of the abutment teeth under function, which results in tensile and compressive stresses at the boundary of the metal framework and the composite luting material. In the anterior esthetic zone the metal-free RBFPD has shown to be a viable alternative for single-tooth replacement. A possible explanation is the selection of a material that has a lower modulus of elasticity than do cast metals, thereby allowing for a reduction of the interface stresses. In addition, a framework that can bond more securely will reduce displacement. The mechanical properties of the fiber-reinforced composite (FRC) depend on the type, form, and quantity of the reinforcing fibers. The bonding ability will depend on the polymer matrix encasing the fibers.[8] Glass fiber possesses a high tensile strength, but the final strength of the FRC is dependent on other factors as well. Fiber-volume fraction, stress strain of the matrix, and the fiber-matrix interface all play a role. The most common types of fibers are electrical glass and silica glass (S-glass) fibers. The S-glass fibers have greater hardness and elastic modulus and present greater resistance to plastic deformation.[9] The fiber composites are produced with a pultrusion process that allows the fiber bundles to be pulled through an extruder at the same time as the

Figure 13. *Framework and porcelain veneers on model.*

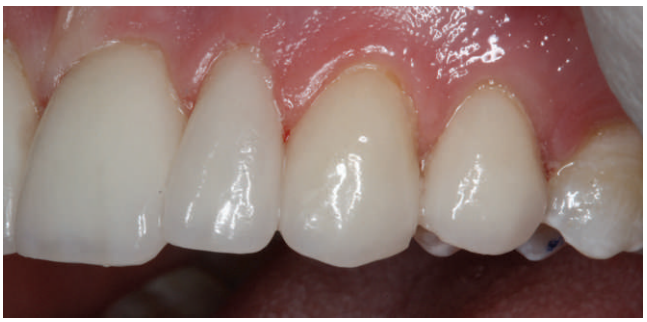


Figure 15. *Left lateral view immediately after insertion.*

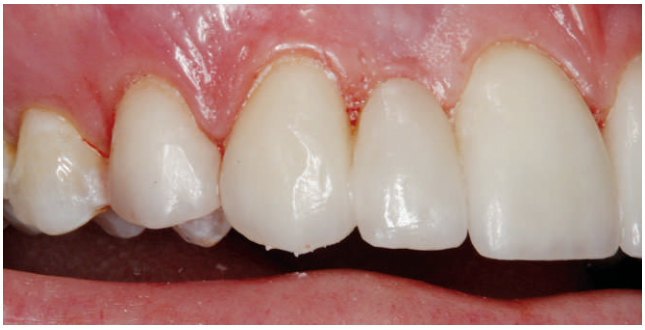


Figure 16. *Right lateral view immediately after insertion.*

polymer. This allows the polymer to penetrate the fiber bundle and creates the final cross-sectional shape. The framework used in this case, Sculpture/FibreKor, consists of the veneering composite Sculpture Plus Nano-Hybrid, a polycarbonate composite, and FibreKor material. FibreKor material is a unidirectional S-2 glass material. Unidirectional materials are termed anisotropic. They reinforce FRC materials to the utmost when stress is applied along the direction of the fibers.[9] The strength of these materials diminishes when a stress is applied in an oblique or perpendicular direction. The efficiency of any given level of fiber loading is called the Krechel factor. The Krechel factor is 1 for FiberKor. A material with a Krechel factor of 1 has the fibers oriented in one direction and gives the greatest amount of reinforcement for a given fiber loading. Woven fibers that reinforce FRC materials in two directions are described as orthotropic, with half the fibers in a longitudinal direction. The result is a reinforcing efficiency (Krechal factor) of 0.5.[9] The maximum bite force of normal incisors is 150–200 N.[10] The unidirectional fiber "FibreKor" can reinforce the framework when the forces are not

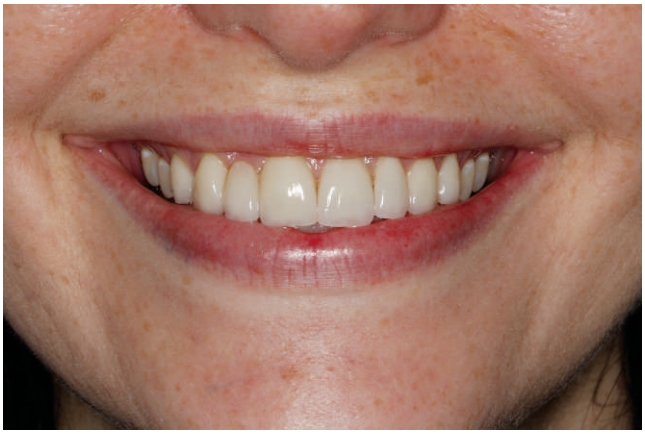


Figure 17. *Final labial smile.*

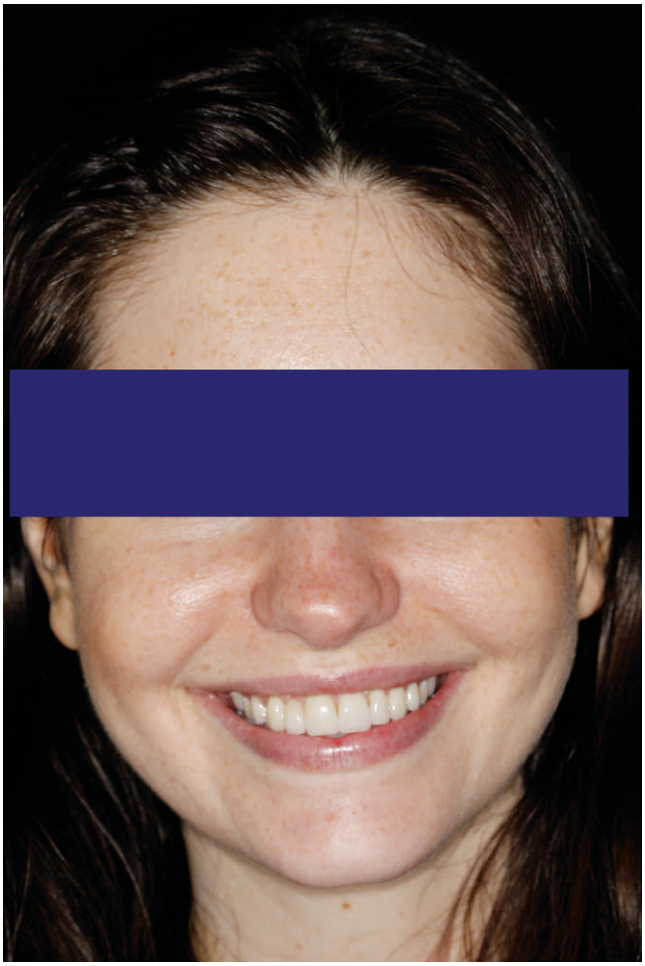


Figure 18. *Full facial smile.*

directed along the axis by placing the unidirectional fibers in multiple directions. When the direction of the force is not parallel to that of the fibers, the mechanical properties decrease and are more contingent on the resin matrix. In the pontic area a high percentage of unidirectional glass fibers are placed in a mesiodistal direction to maximize strength.[11]

The RBFPD allows an esthetic treatment option with minimal tooth modification.[12–14] The Encore Bridge requires a groove with a depth of 0.5 mm, a width of 1.0 mm, and a clearance of 1.0–1.5 mm incisally and cervically. While traditional metal ceramic complete-coverage fixed partial dentures will have the maximum strength and all-ceramic fixed partial denture will provide excellent esthetics, a more destructive tooth preparation will be required. The advent of better adhesives has led to the development of simplified, minimally invasive preparations. While the best bond strength is still to enamel, bond strengths to dentin are reasonably good.[15]

In vitro studies[16] demonstrated that FRC material displays flexural strength that is greater than or similar to that of metal alloys, but it has a lower flexural modulus. Composite resin is a brittle material and would be unable to support protracted occlusal forces in the pontic area of a bridge. Incorporation of fiber into the composite matrix results in increased strength and toughness.[17] The load-bearing capacity can be further increased by placing an additional piece of unidirectional FRC on the occlusal surface. However, the FRC must be placed in a buccolingual direction.[18] Patients with firm anterior occlusal contacts will require more preparation in order to obtain the additional 0.5 mm needed to assure adequate thickness of the wings.[2]

CONCLUSION

Esthetic assessment prior to initiating treatment is critical to achieving the best outcome. Proper guidance in protrusive and lateral excursions must be established to ensure longevity of the restorations. Selection of the best materials to achieve these parameters is of the utmost importance. A systematic approach leads to a more predictable result. The FRC fixed dental prostheses can present a viable substitute for a cast-metal resin-bonded bridge. It is minimally invasive and can often be repaired with direct composite resin if required.[19]

Acknowledgement

The authors wish to thank BonaDent Dental Laboratories (Seneca Falls, NY, USA) for the laboratory work.

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.

(Accepted 20 December 2011)

REFERENCES

1. Meyenberg KH, & Imoberdorf MJ (1997) The aesthetic challenge of a single tooth replacement: A comparison of treatment alternatives *Practical Periodontics Aesthetic Dentistry* **9(7)** 727-735.
2. Uribe F, Meiers JC, & Nanda R (2008) Fixed retention of congenitally missing lateral incisors using a chairside, prefabricated fiber-reinforced composite bridge *World Journal of Orthodontics* **9(4)** 349-354.
3. Dietschi D, & Schatz J-P (1997) Current restorative modalities for young patients with missing anterior teeth *Quintessence International* **28(4)** 231-240.
4. Miller MB (1993) Aesthetic anterior reconstruction using a combined periodontal/restorative approach *Practical Periodontics Aesthetic Dentistry* **5(8)** 33-40.
5. Leal FR, Cobb DS, Denehy GE, & Margeas R (2001) A conservative aesthetic solution for a single anterior edentulous space: Case report and one-year follow-up *Practical Procedures Aesthetic Dentistry* **13(8)** 635-641.
6. Hornbrook DS (1993) Anterior tooth replacement using a two-component resin-bonded bridge *Compendium of Continuing Education in Dentistry* **14(1)** 52-58.
7. Probster B, & Henrich GM (1997) 11-year follow-up study of resin-bonded fixed partial dentures *International Journal of Prosthodontics* **10(3)** 259-268.
8. Vallitu PK, & Sevelius C (2000) Resin-bonded, glass fiber-reinforced composite fixed partial dentures: A clinical study *Journal of Prosthetic Dentistry* **84(4)** 413-418.
9. Chong K-H, & Chai J (2003) Strength and mode of failure of unidirectional and bidirectional glass fiber-reinforced composite materials *International Journal of Prosthodontics* **16(2)** 161-166.
10. Fukuda T, Sugimoto Y, Yamashita S, Toyooka H, & Tanaka M (2011) Forces applied to the maxillary incisors during tracheal intubation and dental injury risks of intubation by beginners: A manikin study *Acta Anaesthesiol Taiwan* **49(1)** 12-15.
11. Freilich MA, Meirs JC, Duncan JP, & Goldberg AJ (2000) *Fiber-reinforced Composites in Clinical Dentistry* Quintessence Publishing, Carol Stream, Ill.
12. Feinman RA (1997) The aesthetic composite bridge *Practical Periodontics Aesthetic Dentistry* **9(1)** 85-89.
13. Fahl N Jr (1998) Restoration of the maxillary arch utilizing a composite resin buildup and fiber framework *Practical Periodontics Aesthetic Dentistry* **10(3)** 363-367.
14. Javaheri DS (2001) Replacement of an anterior tooth with a fiber reinforced resin bridge *Compendium of Continuing Education in Dentistry* **22(1)** 68-70.
15. Kimmes NS, Barkmeier WW, Erickson RL, & Latta MA (2010) Adhesive bond strengths to enamel and dentin using recommended and extended treatment times *Operative Dentistry* **35(1)** 112-119.
16. Freilich MA, Duncan JP, Alarcon EK, Eckrote KA, & Goldberg AJ (2002) The design and fabrication of fiber-reinforced implant prostheses *Journal of Prosthetic Dentistry* **88(4)** 449-454.
17. Shuman IE (2000) Replacement of a tooth with a fiber-reinforced direct bonded restoration *General Dentistry* **48(3)** 314-318.
18. Xie Q, Lassila LV, & Vallittu PK (2007) Comparison of load-bearing capacity of direct resin-bonded fiber-reinforced composite FPDs with four framework designs *Journal of Dentistry* **35(7)** 578-582.
19. Wolff D, Schach C, Kraus T, Ding P, Pritsch M, Mente J, Joerss D, & Staehle HJ (2011) Fiber-reinforced composite fixed dental prostheses: A retrospective clinical examination *Journal of Adhesive Dentistry* **13(2)** 187-194.

©Operative Dentistry, 2012, **37-6,** 584-590

Tooth Fragment Reattachment: The Natural Restoration

DP Lise • LC Cardoso Vieira • É Araújo
GC Lopes

Clinical Relevance

This technique is a conservative and predictable approach to restore fractured anterior teeth.

SUMMARY

The aim of this manuscript was to discuss some important considerations about tooth fragment reattachment and report the success of a clinical case in which a tooth fragment and direct composite resin were used to restore a fractured anterior tooth. Clinical and radiographic examination 12 months after trauma showed good esthetics and periodontal health.

*Diogo Pedrollo Lise, DDS student, MSD, Operative Dentistry, , Universidade Federal de Santa Catarina, Florianópolis, Brazil

Luiz Clovis Cardoso Vieira, DDS, MS, Operative Dentistry, Universidade Federal de Santa Catarina, Florianópolis, Brazil

Élito Araújo, DDS, MSD, Operative Dentistry, Universidade Federal de Santa Catarina, Florianópolis, Brazil

Guilherme Carpena Lopes, DDS, MSD, Operative Dentistry, Universidade Federal de Santa Catarina, Florianopolis, Brazil

*Corresponding author: Disciplina de Dentística, Sala 138, Universidade Federal de Santa Catarina, Campus Universitário Trindade, Florianópolis 88040–970, Brazil; e-mail: diogolise@hotmail.com

DOI: 10.2341/12-063-T

INTRODUCTION

One in four people suffer from some kind of oral trauma during childhood or youth, especially males.[1] The impact during trauma transfers a large amount of energy to a limited part of the tooth crown, leading to fracture of tooth structure. Motor vehicle collisions, sports, and home accidents are the most common causes of anterior tooth fractures. Because of their vulnerable position in the dental arch, upper central incisors are the most affected.[3] The risk of tooth fracture is further increased when an individual has severe overjet/overbite or anterior open bite.[4]

Dental fractures are classified by the World Health Organization as uncomplicated (cracks and/or enamel and dentin fractures) or complicated (with pulp exposure and/or periodontal involvement). Fortunately, uncomplicated fractures are more frequent and require less complex treatment.[2,5]

Dentists are often required to treat (usually as an emergency situation) the esthetic, functional, and emotional discomforts that tooth fractures can cause. Treatment strategies range from simple enamel polishing to prosthetic rehabilitation. The restorative choice is based on various factors, such as the extent of the fracture, a patient's age, dental eruption and root formation, occlusion, esthetic expectations, amount and quality of remaining

tooth, and pulpal and periodontal involvement.[6–8] If pulp becomes exposed, the priority of restorative care is to preserve vitality using a conservative approach (pulp capping, pulpotomy, or curettage), depending on the degree of bacterial contamination, root formation, pulp consistency, and bleeding.[2] When periodontal biologic width is compromised, surgical modification of the support tissues or orthodontic movement (extrusion) are necessary to enable restorative procedures and promote periodontal health.[9–11] However, a single anterior tooth osteotomy can result in poor esthetics (considering the bony architecture of the adjacent teeth), postoperative sensitivity (root exposure), and periodontal pockets.[12,13] Extrusion also has disadvantages, such as reduced cervical diameter (in relation to adjacent teeth), time required for stabilization (restraint), incisal abrasion to adjust the cervical-incisal tooth length, and consequent loss of optical characteristics.[11,14]

A perfect reproduction of the natural dental color, optical properties (such as translucency, opalescence, and fluorescence), shape, and surface texture is a challenge and requires great skill and dexterity when performing a direct composite restoration. Therefore, when a tooth fragment is viable and presents good adaptation to the remaining dental structure, it should be the first restorative option, with the restorative procedures performed immediately (just after fracture) or later (under more favorable conditions).[6,8] Although the first report of fragment reattachment dates from 1964, adhesive restorations were not possible until the late 1970s (without pin retention) using enamel/dentin etching associated with an adhesive system and composite resin.[16,17] Adhesive reattachment requires minimum healthy tooth reduction, has a predictable esthetic result, is usually faster than a complete composite restoration, and triggers a strong emotional effect because the patient feels relief by keeping one's own natural tooth.[8,18]

Modifications to both tooth and fragment prior to bonding have been proposed, with an estimated recovery of fracture resistance up to 97%.[19] Theoretically, these techniques (dentin groove, bevel, chamfer, or overcontour) remove fractured enamel prisms and retain prisms that are in a favorable position for effective etching.[17] Preparations can also be performed after bonding to improve esthetics by grinding the buccal fracture line and masking it with composite.[18,20,21] Indeed, restorations might result in fragment misfit or deficient composite esthetics over time because of composite abrasion or discoloration.[8,17,22] Simple reattachment recovers approximately 37–50% of the tooth fracture resistance.[23,24] Nevertheless, this procedure is feasible because retention relies on hybridization.[8,15,18,21,25,26] Similar bonding results are achieved with the use of adhesives alone or in combination with composites. However, there is a trend of improving fracture resistance when adhesives are associated with composite resin because they would fill possible interface gaps.[24]

Sometime after bonding, a fragment might exhibit a lighter shade (white) than the remaining. This occurs because of possible dentin dehydration and breakdown of collagen fibers.[27] To avoid discoloration, the patient should be instructed to store the tooth fragment in water immediately after trauma. Furthermore, hydrophilic adhesives require proper dentin hydration for optimal bonding.[28] If a tooth fragment is maintained in a dry state for more than one hour, it will achieve lower bond strength[29] and must be rehydrated for at least 30 minutes before bonding.[20] Complete rehydration and color match usually occurs after one week but could be delayed by several months or may never occur.[6,30,31]

Fragment debonding happens because of repeated trauma, nonphysiological use of the tooth, or horizontal pulling of the tooth.[22] The risk of debonding is higher for children since they have restricted control of incisive function and greater exposure to traumatic situations.[8] Some authors have demonstrated long-term reattachment prognoses that are better than composite restorations, reaching a 90% success rate after five years.[3,22] While the fracture line becomes undetectable in some cases, the patient should be informed about treatment limitations and future interventions.[32]

The following case report describes the management of anterior fractured teeth with periodontal involvement, where a fragment was used as the main restorative material.

CLINICAL CASE REPORT

A 13-year-old boy presented to the Federal University of Santa Catarina with fractured anterior teeth (#8, #9, and #10) as a result of a fall that occurred three months before (Figure 1). Radiographic evaluation of tooth #9 revealed an apical lesion and pulp necrosis. An enamel and dentin fracture, with subgingival extensions, was observed on tooth #8 (Figure 2), and the patient was in possession of the fragment. The provided fragment was stored in distilled water during the clinical examination.

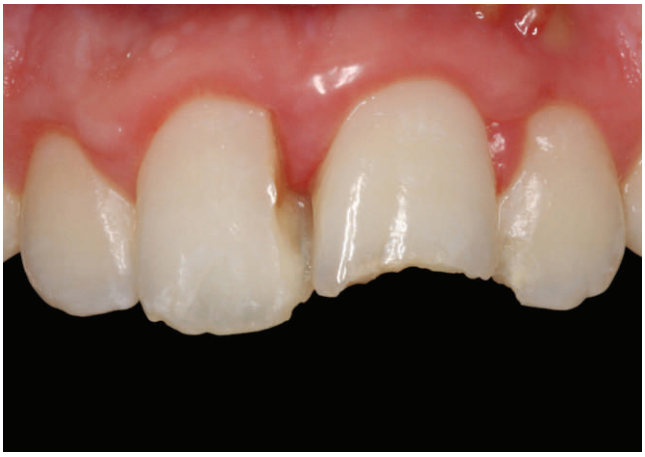


Figure 1. *Preoperative clinical image of fractured anterior teeth.*

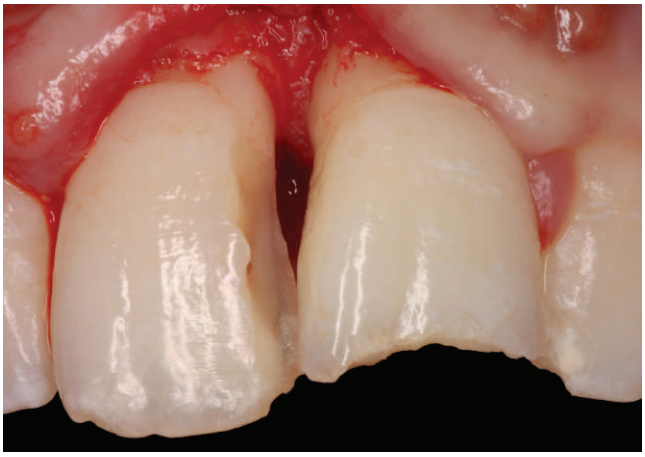


Figure 3. *Gingival flap enabled observation of fracture limit.*

An intrassulcular incision was made on teeth #8 and #9. A gingival flap was obtained to evaluate the relation between the fracture margin and alveolar bone, to provide adequate isolation of the operative field (essential for bonding procedures), and to verify the fit of the fragment. The fracture margin was located at the cementoenamel junction; therefore, an osteotomy was not necessary (Figure 3). After rubber dam isolation, excellent fragment adaptation was observed, and it was determined that the best treatment plan was to reattach the fragment (Figure 4). The fragment and tooth were etched with 35% phosphoric acid (Table 1) for 15 seconds and rinsed. The enamel was air dried while the dentin was maintained in a moist state using a cotton pellet. Two adhesive coats were then applied without light curing (Figure 5). A composite resin increment was placed over the entire fractured surface, and the fragment was positioned and properly adjusted (Figure 6). After the excess resin composite was removed, the restoration was light cured for 40 seconds each on the buccal and palatal surfaces using an LED unit with a 900 mW/cm^2 output (Figure 7). Polishing was performed using abrasive discs and polishing paste. The gingival flap was repositioned, and the papillae were sutured (Figure 8). After 10 days, the stitches were removed, and improved gingival health was observed (Figure 9). Tooth #9 was endodontically treated, and direct composite resin restorations were performed on the other fractured teeth. A clinical and radiographic examination 12 months after trauma showed the absence of a visible fracture line and good periodontal health (Figure 10).

Potential Problems

The development of effective adhesives and composite resins has made fragment reattachment the main

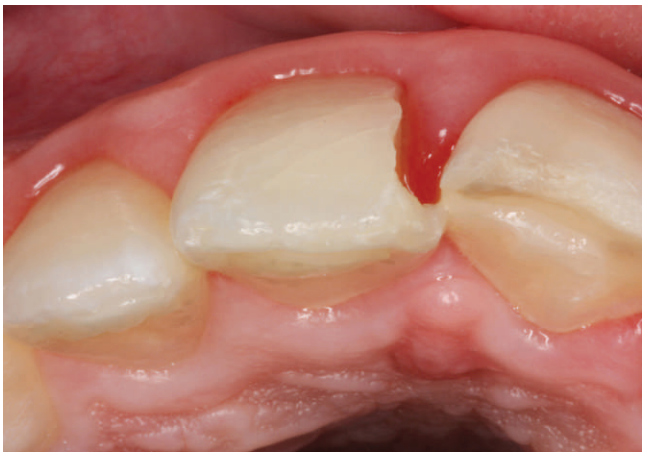


Figure 2. *Incisal view suggesting biological width involvement of tooth #8.*

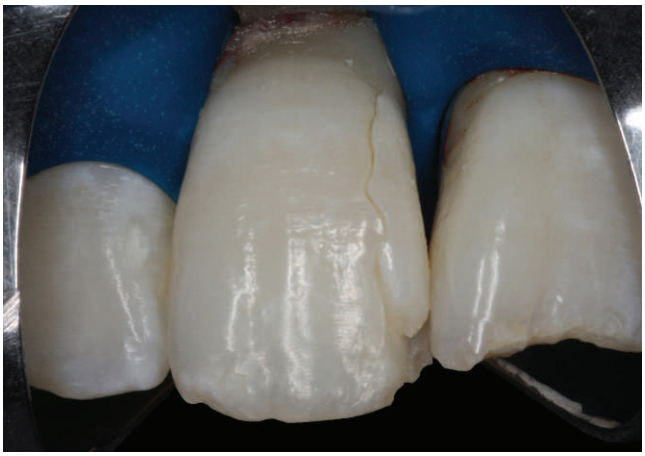


Figure 4. *Tooth fragment exhibited excellent adaptation, and it was used as the main restorative material.*

Table 1: *Materials Used*	
Etchant Gel (phosphoric acid gel 35%)	Coltène/Whaledent, Altstätten, Switzerland
Adper™ Single Bond 2 (etch&rinse two-step adhesive)	3M ESPE, St. Paul, MN, USA
KG Brush Fine (disposable applicator)	KG Sorensen, Cotia, SP, Brazil
Flash Lite 1401 (LED unit)	Discus Dental, Culver City, CA, USA
Composites 4 Seasons (composite resins)	Ivoclar Vivadent, Schaan, Liechtenstein
Sof-Lex (polishing discs)	3M ESPE, St. Paul, MN, USA
Diamond Excel (polishing paste)	FGM, Joinville, SC, Brazil
Diamond Flex (felt discs)	FGM, Joinville, SC, Brazil

choice for rehabilitating fractured anterior teeth, especially in young patients.[6] Because of its conservative nature, this technique reduces the need for a further restorative approach, as the fragment behaves similarly to the remaining tooth in regard to physiological wear and does not present signs of deterioration as early as composite resins (staining and surface texture loss).[3,15]

Clinicians who wish to master the technique and provide favorable esthetics and long-term results to their patients must have a working knowledge of the variations of the technique since some cases require a multidisciplinary approach, such as surgery or endodontics. In the presented case, it would not be prudent to utilize reattachment without surgical exploration to determine the extent of the fracture and to ensure a clean and dry operating field provided by rubber dam isolation. The success of the bonding protocol would be impaired by blood contamination from the gingival flap.[12] In order to facilitate fragment handling and prevent reattachment in an improper position, there are some strategies that can be utilized, such as fragment stabilization with low-fusion compound, gutta-percha, wax, or acrylic or silicone index, which maintains a reference of the adjacent teeth.[8,33] In the present case, the use of an index was not necessary because of the excellent fit between the fragment and remaining tooth structure.

According to the patient, the fragment was maintained intraorally and in position since the trauma occurred (three months). Even though the

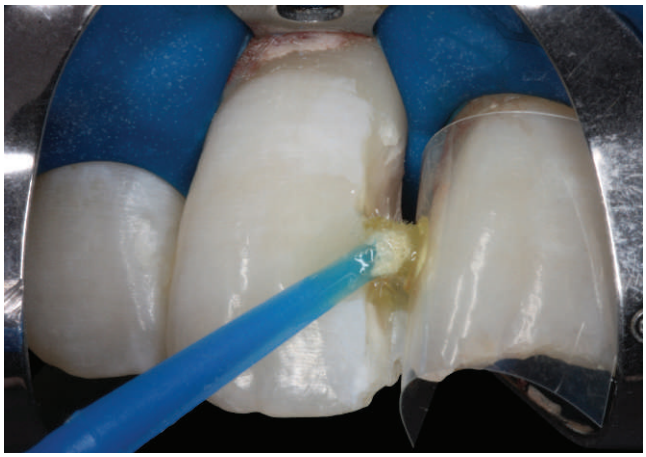


Figure 5. *Two adhesive layers were applied on etched substrates.*

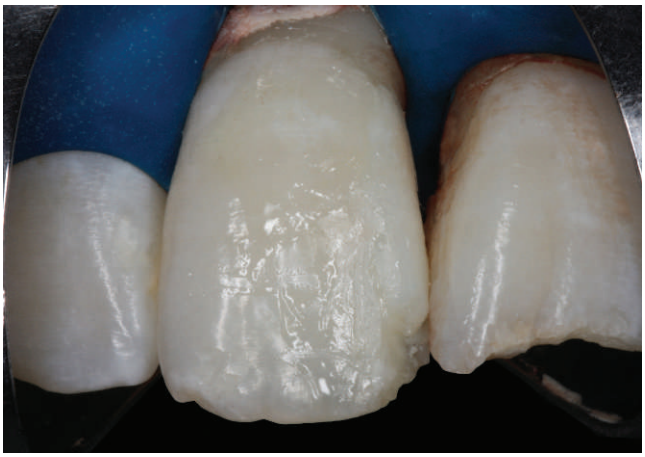


Figure 6. *Fragment adapted after remove of resin composite excess.*

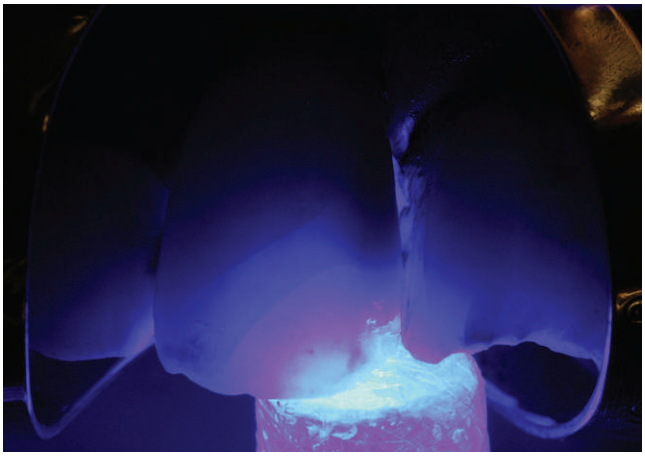


Figure 7. *Dental cracks on tooth #8 were observed during palatal light curing.*

fragment had been kept hydrated by saliva, the fragment was immersed in distilled water for 30 minutes prior to bonding to ensure adequate hydration and superior bond strength.[20,29] Mechanical preparation of the fragment was not performed before bonding, and its retention was based solely on hybridization.[8,15,21,25,26] Even with the possibility of using an adhesive system[24] only for fragment reattachment, composite resin was applied to fill possible gaps between the fragment and the remaining tooth structure and to improve the adaptation of the two pieces.

After three months, tenuous color mismatch was still observed between the fragment and tooth, but the patient was satisfied. However, even in cases where the fragment remains dehydrated for long periods of time and presents a contrasting color, reattachment has great value since complete rehydration may still occur.[31] One year after reattachment, the patient was pleased with the appearance of his anterior teeth, and no other esthetic treatment was necessary.

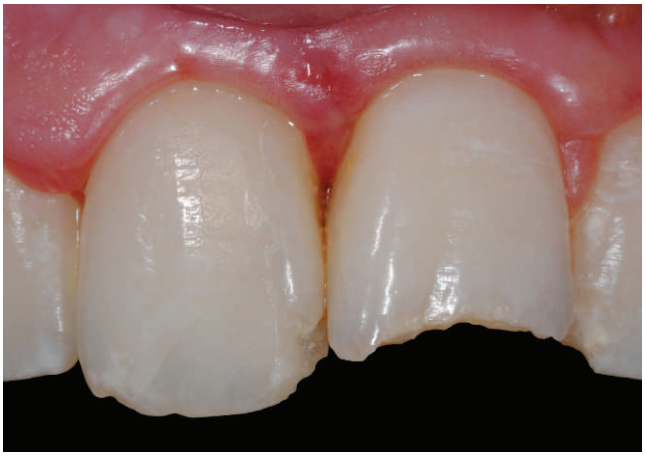


Figure 9. *After 10 days, improved gingival health was observed.*

In order to maintain oral health, reattachment cases must be followed for two years with clinical examination, vitality testing, periodontal probing, and radiographs.[8] It is very important that patients and their families understand the limitations of reattachment and take care to prevent fragment displacement. However, it is a dentist's responsibility to diagnose deleterious habits or traumatic activities that the patient may present and offer some protection, such as the use of a custom mouthguard.[4,34]

Advantages

- Conservative treatment
- Low cost and faster than direct restorative procedures
- Predictable esthetic result

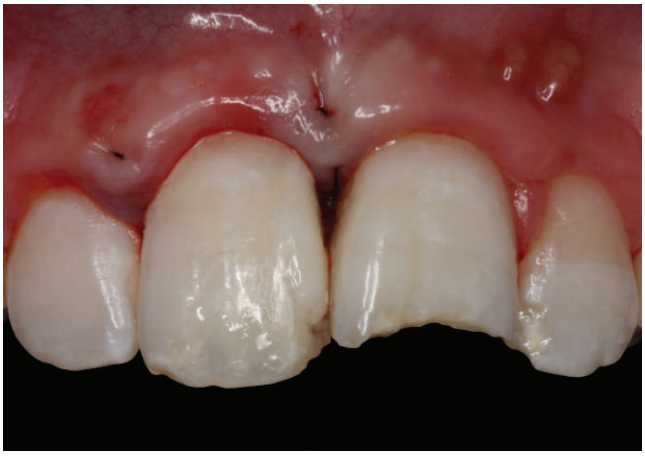


Figure 8. *Immediate view after gingival flap suture denotes tenuous color mismatch between the fragment and tooth #8.*

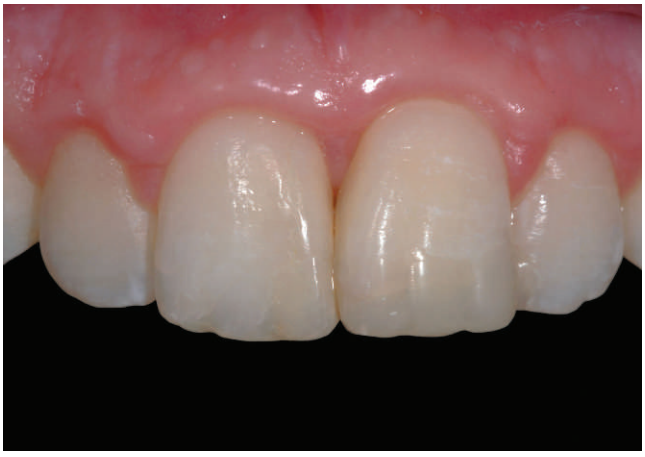


Figure 10. *Clinical view 12 months after trauma (composite direct restorations were realized on teeth #8, #9, and #10).*

Limitations

- Possible fragment debonding
- Incomplete fragment rehydration and color mismatch

CONCLUSIONS

- Considering the high incidence of dental fractures as a result of trauma, the working knowledge of the dentist regarding treatment possibilities is essential.
- Tooth fragment reattachment should be performed whenever possible because it is a simple, fast, and affordable procedure and presents a predictable esthetic result.

Conflict of Interest

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

(Accepted 30 April 2012)

REFERENCES

1. Andreasen JO (1970) Etiology and pathogenesis of traumatic dental injuries *Scandinavian Journal of Dental Research* **78(4)** 329-342.
2. Dietschi D, Jacoby T, Dietschi JM, & Schatz JP (2000) Treatment of traumatic injuries in the front teeth: Restorative aspects in crown fractures *Practical Periodontics and Aesthetic Dentistry* **12(8)** 751-758.
3. Zerman N, & Cavalleri G (1993) Traumatic injuries to permanent incisors *Endodontics and Dental Traumatology* **9(2)** 61-64.
4. Bastone EB, Freer TJ, & McNamara JR (2000) Epidemiology of dental trauma: A review of the literature *Australian Dental Journal* **45(1)** 2-9.
5. Reis A, Loguercio AD, Kraul A, & Matson E (2004) Reattachment of fractured teeth: A review of literature regarding techniques and materials *Operative Dentistry* **29(2)** 226-233.
6. Baratieri LN, Monteiro S Jr, & Andrada MAC (1990) Tooth fracture reattachment: Case reports *Operative Dentistry* **21(4)** 261-270.
7. Macedo GV, Diaz PI, Fernandes CAO, & Ritter AV (2008) Reattachment of anterior teeth fragments: A conservative approach *Journal of Esthetic and Restorative Dentistry* **20(1)** 5-20.
8. Baratieri LN, Monteiro S Jr, Andrada MAC, Vieira LCC, Cardoso AC, & Ritter AV (1995) *Estética: Restauraçoes Adesivas Diretas em Dentes Anteriores Fraturados* Quintessence, São Paulo.
9. Gargiulo AW, Wentz FM, & Orban B (1961) Dimensions and relations of the dento-gingival junction in humans *Journal of Periodontology* **32(3)** 261-266.
10. El-Askary F, Ghalab OH, Eldemerdash FH, Ahmed OIR, Fouad SA, & Nagy MM (2006) Reattachment of a several traumatized maxillary central incisor, one-year clinical evaluation: A case report *Journal of Adhesive Dentistry* **8(5)** 343-349.
11. Filho GRN, Machion L, Teixeira FB, Pimenta LAF, & Sallum EA (2002) Reattachment of an autogenous tooth fragment in a fracture with biologic width violation: A case report *Quintessence International* **33(3)** 181-184.
12. Baratieri LN, Monteiro S Jr, Cardoso AC, & Filho JCM (1993) Coronal fracture with invasion of the biologic width: A case report *Operative Dentistry* **24(2)** 85-91.
13. Stern N, & Becker A (1980) Forced eruption: Biological and clinical considerations *Journal of Oral Rehabilitation* **7** 395-403.
14. Saito CTMH, Guskuma MH, Gulinelli JL, Sonoda CK, Garcia-Júnior IR, Filho OM, & Panzarini SRP (2009) Management of a complicated crown-root fracture using adhesive fragment reattachment and orthodontic extrusion *Dental Traumatology* **25** 541-544.
15. Dean JA, Avery DR, & Swartz ML (1986) Attachment of anterior tooth fragments *Pediatric Dentistry* **8(2)** 139-143.
16. Chosak A, & Eidelman E (1964) Rehabilitation of a fractured incisor using the patient's natural crown: Case report *Journal of Dentistry for Children* **31(1)** 19-21.
17. Simonsen RJ (1979) Traumatic fracture restoration: An alternative use of the acid etch technique *Quintessence International* **10(2)** 15-22.
18. Brambilla GPM, & Cavallé E (2007) Fractured incisors: A judicious restorative approach—Part 1 *International Dental Journal* **57** 13-18.
19. Reis A, Francci C, Loguercio AD, Carrilho MR, & Rodrigues Filhos LE (2001) Re-attachment of anterior fractured teeth: Fracture strength using different techniques *Operative Dentistry* **26(3)** 287-294.
20. Capp CI, Roda MI, Tamaki R, Castanho GM, Camargo MA, & Cara AA (2009) Reattachment of rehydrated dental fragment using two techniques *Dental Traumatology* **25(1)** 95-99.
21. Rappelli G, Massaccesi C, & Putignano A (2002) Clinical procedures for the immediate reattachment of a tooth fragment *Dental Traumatology* **18(5)** 281-284.
22. Andreasen FM, Norén JG, Andreasen JO, Engelhardtsen S, & Lindth-Strömberg U (1995) Long-term survival of fragment bonding in the treatment of fractured crowns: A multicenter clinical study *Pediatric Dentistry* **26(10)** 669-681.
23. Farik B, Munksgaard EC, & Andreasen JO (2000) Impact strength of teeth restored by fragment-bonding *Endodontics and Dental Traumatology* **16(4)** 151-153.
24. Reis A, Kraul A, Francci C, de Assis TG, Crivelli DD, Oda M, & Loguercio AD (2002) Re-attachment of anterior fractured teeth: Fracture strength using different materials *Operative Dentistry* **27(6)** 621-627.
25. Worthington RB, Murchison DF, & Vandewalle KS (1999) Incisal edge reattachment: The effect of preparation

utilization and design *Quintessence International* **20(9)** 637-643.

26. Pagliarini A, Rubini R, Rea M, & Campese M (2000) Crown fractures: Effectiveness of current enamel-dentine adhesives in reattachment of fractured fragments *Quintessence International* **31(2)** 133-136.
27. Maia EAV, Baratieri LN, Andrada MAC, Monteiro S Jr, & Araújo EM (2003) Tooth fragmente reattachment: Fundamentals of the technique and two case reports *Quintessence International* **34(2)** 99-107.
28. Perdigão J, Van Meerbeek B, Lopes MM, & Ambrose WW (1999) The effect of a re-wetting agent on dentin bonding *Dental Materials* **15** 282-295.
29. Farik B, Musksgaard EC, Andreasen JO, & Kreiborg S (1999) Drying and rewetting anterior crown fragments prior to bonding *Endodontics and Dental Traumatology* **15(3)** 113-116.
30. Simonsen RJ (1982) Restoration of a fractured central incisor using original tooth fragment *Journal of the American Dental Association* **105(4)** 646-648.
31. Toshiriro K, & Rintaro T (2005) Rehydration of crown fragment 1 year after reattachment: A case report *Dental Traumatology* **21(5)** 297-300.
32. Chu FCS, Yim TM, & Wei SHY (2000) Clinical considerations for reattachment of tooth fragments *Restorative Dentistry* **31(6)** 385-391.
33. Álvares I, Sensi LG, Araújo EM Jr, & Araújo E (2007) Silicone index: An alternative approach for tooth fragment reattachment *Journal of Esthetic and Restorative Dentistry* **19(5)** 240-246.
34. Terry DA (2003) Adhesive reattachment of a tooth fragment: The biological restoration *Practical Periodontics and Aesthetic Dentistry* **15(5)** 403-409.

©Operative Dentistry, 2012, **37-6,** 591-601

Clinical Research

Randomized Clinical Trial of Two Resin-Modified Glass Ionomer Materials: 1-year Results

J Perdigão • M Dutra-Corrêa • SHC Saraceni
MT Ciaramicoli • VH Kiyan

Clinical Relevance

Although the quality of enamel margins may be a concern for the nanofilled resin-modified glass ionomer materials (RMGIC), surface roughness is still the major disadvantage for traditional RMGICs in non-stress-bearing areas.

SUMMARY

With institutional review board approval, 33 patients who needed restoration of noncarious cervical lesions (NCCL) were enrolled in this study. A total of 92 NCCL were selected and randomly assigned to three groups: (1) Ambar (FGM), a two-step etch-and-rinse adhesive (control), combined with the nanofilled composite resin Filtek Supreme Plus (FSP; 3M ESPE); (2) Fuji II LC (GC America), a traditional resin-modified glass ionomer (RMGIC) restorative material; (3) Ketac Nano (3M ESPE), a nanofilled RMGIC restorative material. Restorations were evaluated at six months and one year using modified United States Public Health Service parameters. At six months after initial placement, 84 restorations (a 91.3% recall rate) were evaluated. At one year, 78 restorations (a 84.8% recall rate) were available for evaluation. The six month and one year overall retention rates were 93.1% and 92.6%, respectively, for Ambar/FSP; 100% and 100%, respectively, for Fuji II LC; and 100% and 100%, respectively, for Ketac Nano with no statistical difference between any pair of groups at each recall. Sensitivity to air decreased for all three adhesive materials from the preoperative to the postoperative stage, but the difference was not statistically significant. For Ambar/FSP, there were no

*Jorge Perdigão, University of Minnesota, Minneapolis, MN, USA

Maristela Dutra-Corrêa, DDS, MS, PhD, Graduate Program in Dentistry, Paulista University–UNIP, São Paulo, Brazil

Cíntia H C Saraceni, DDS, MS, PhD, Graduate Program in Dentistry, Paulista University–UNIP, São Paulo, Brazil

Márcia T Ciaramicoli, DDS, MS, PhD, Department of Operative Dentistry, Paulista University–UNIP, São Paulo, Brazil

Vanessa H Kiyan, DDS, MS, Department of Operative Dentistry, Paulista University–UNIP, São Paulo, Brazil

*Corresponding author: 515 SE Delaware Street, 8-450 Moos Tower, Minneapolis, MN 55455, USA; e-mail: perdi001@umn.edu

DOI: 10.2341/11-415-C

statistical differences for any of the parameters from baseline to six months and from baseline to one year. For Fuji II LC, surface texture worsened significantly from baseline to six months and from baseline to one year. For Ketac Nano, enamel marginal staining increased significantly from baseline to one year and from six months to one year. Marginal adaptation was statistically worse at one year compared with baseline only for Ketac Nano. When parameters were compared for materials at each recall, Ketac Nano resulted in significantly worse color match than any of the other two materials at any evaluation period. At one year, Ketac Nano resulted in significantly worse marginal adaptation than the other two materials and worse marginal staining than Fuji II LC. Surface texture was statistically worse for Fuji II LC compared with the other two materials at all evaluation periods. The one-year retention rate was statistically similar for the three adhesive materials. Nevertheless, enamel marginal deficiencies and color mismatch were more prevalent for Ketac Nano. Surface texture of Fuji II LC restorations deteriorated quickly.

INTRODUCTION

Glass ionomer cements (GICs) have improved substantially since Wilson and Kent introduced these materials in the early 1970s.[1,2] GICs are self-adhesive materials that bond to tooth hard tissues through combined micromechanical and/or chemical bonding, in contrast to composite resins that only bond micromechanically. The ionic bond between the carboxyl groups of the polyalkenoic acid and hydroxyapatite in enamel and dentin is responsible for the chemical bonding ability of GICs.[3] Classical GICs set exclusively through an acid-base reaction between the polycarboxylate matrix and the fluoroaluminosilicate glass that results in the cross-linking of the polycarboxylate chains by metal ions from the glass.[3]

Resin-modified GICs (RMGICs) were developed to overcome some of the problems of early moisture sensitivity and low mechanical strength associated with classical GICs, yet maintain or improve their clinical advantages.[2–4] Whereas classical GICs set exclusively through an acid-base reaction, RMGICs undergo an additional free-radical polymerization.[3] RMGICs contain a monomer side chain grafted onto the polyalkenoic acid structure, such as 2-hydroxyethyl methacrylate (HEMA) or other monomer, which polymerizes through chemical and/or photo activation.[3,5] The chemical bonding of RMGICs to hydroxyapatite crystals in enamel and dentin has been demonstrated by x-ray photoelectron spectroscopy and Fourier-transformed infrared spectroscopy,[6,7] whereas the ability of these materials to mechanically interlock and form hybrid layers in dentin has been demonstrated by electron microscopy and confocal microscopy.[8–10]

Although RMGICs result in a more predictable adhesion to tooth structure than most resin-based adhesives,[11] their *in vitro* bond strengths are usually lower than those of resin-based adhesives.[12,13] This apparent paradox is a result of the low cohesive strength of the GIC material, which causes the material to fail intrinsically prior to debonding from the tooth surface.[14–16]

Several studies have reported the clinical effectiveness of GIC-based materials. Fuji II LC (GC America, Alsip, IL, USA) has resulted in excellent retention rates in noncarious cervical lesions (NCCL) up to five years.[17] This RMGIC has performed at the same level, or better, than two- and three-step etch-and rinse-adhesives in terms of retention rates.[13,17–19]

A new nanofilled RMGIC, Ketac Nano (3M ESPE, St Paul, MN, USA), has been recently introduced. Besides the typical GIC fluoroaluminosilicate glass, this material contains silane-treated silica nanofillers similar to those in Filtek Supreme Plus (3M ESPE, St Paul, MN, USA), and agglomerates or clusters of nano-sized zirconia/silica that appear as a single unit, which results in a highly packed filler composition (~69%).[20,21] According to the respective manufacturer, this new material has enhanced physical properties compared with those of Fuji II LC, a traditional restorative RMGIC.[21]

In light of the excellent clinical retention of the traditional RMGIC Fuji II LC in NCCL, it is relevant to compare its clinical performance with that of the new nanofilled RMGIC Ketac Nano, using an etch-and-rinse adhesive as the resin-based adhesive control. Therefore, the null hypothesis to test in this study is that the clinical retention of a new nanofilled RMGIC does not differ from that of a traditional RMGIC or that of a resin-based etch-and-rinse adhesive combined with a nanofilled composite resin.

METHODS AND MATERIALS

Before participating in the study, patients gave informed consent. Both the consent form and this

research protocol were reviewed and approved by the Paulista University (UNIP) Institutional Review Board. All 33 patients, with ages ranging from 30 to 79 years (average, 48.7 years), had been referred to the Operative Dentistry Clinic for the restoration of class V lesions. All patients received a dental exam by a member of the clinical faculty. The dental health status of patients was normal in all other respects. Patients with fewer than 20 teeth were not included in the study. All the other characteristics of dental status were considered normal, including the periodontal condition. Teeth included in the study had NCCL without undercuts. Teeth with carious lesions were excluded. Other exclusion criteria included

- History of existing chronic tooth sensitivity
- Bruxism and visible wear facets in the posterior dentition
- Known inability to return for recall appointments
- Fractured or visibly cracked candidate tooth
- Current desensitizing therapy, including desensitizing dentifrices or other over-the-counter products
- Chronic use of anti-inflammatory, analgesic, or psychotropic drugs
- Pregnancy or breast-feeding (potential conflicts with recall dates)
- Allergies to ingredients of resin-based restorative materials
- Orthodontic appliance treatment within the previous three months
- Abutment teeth for fixed or removable prostheses
- Teeth or supporting structures with any symptomatic pathology
- Existing periodontal disease or periodontal surgery within the previous three months

The teeth to be restored were vital (positive-response-to-cold sensitivity test), had a normal occlusal relationship with natural dentition, and had at least one adjacent tooth contact. Cavo-surface angles were not beveled and no retentive grooves were placed.

Materials, respective batch numbers, composition, and manufacturer's instructions for use are listed in Table 1. Approximately 92% of the lesions were classified in degree 1 or 2 in the University of North Carolina (UNC) sclerosis scale[22] (Table 2) and were equally distributed among the three groups. The distribution of restorations was 47.9% in the maxillary arch and 52.1% in the mandibular arch; 81.6% of restorations were placed in premolars or molars. Differences in lesion size and other characteristics were minimal.

A total of 92 NCCL were restored in this study. Each subject had two or three restorations placed, with each adhesive material applied to one tooth. The adhesive materials were randomly assigned with a separate randomization for each subject (adhesive material vs tooth): (1) Ambar (FGM, Joinville, Brazil), a two-step etch-and-rinse adhesive that was used as control. A nanofilled composite resin, Filtek Supreme Plus (FSP; 3M ESPE), was used with this etch-and-rinse adhesive; (2) Fuji II LC (GC America), a traditional RMGIC restorative material; (3) Ketac Nano (3M ESPE), a nanofilled RMGIC restorative material.

All operators had advanced clinical training in operative dentistry and were individually instructed by the study coordinator on how to apply each material. The insertion protocol for each restorative sequence was printed and posted in each dental unit so the operator was able to easily review the instructions before and while applying each material. Each operator inserted approximately the same number of restorations (± 2). Due to the specialized field of the operators, this study was not blind. All operative procedures were performed with cotton-roll isolation without local anesthesia.

Restorative materials were inserted in one increment because the NCCL were not deeper than 2 mm. The restorative material was polymerized for 40 seconds with a light-curing unit (Elipar Freelight 2, 3M ESPE). The intensity of the light exceeded 500 mW/cm^2. After polymerization, finishing was accomplished with aluminum oxide discs of decreasing abrasiveness (Sof-Lex XT, 3M ESPE).

Clinical Evaluation

In addition to the assessment of sensitivity immediately before insertion, postoperative sensitivity was assessed one week after the restorative procedure via telephone interview. Restorations were evaluated immediately after insertion, at six months, and at one year using the UNC-modified United States Public Health Service criteria[22] (*alfa*, *bravo*, *charlie*) for retention, color match, marginal staining, wear, marginal adaptation, surface texture, preoperative sensitivity (air syringe), and postoperative sensitivity (query) (Table 2). Two clinicians evaluated the restorations blindly at each recall but did not evaluate the restorations that they had inserted. In case there was no consensus, a third clinician evaluated the restoration. To help with the evalua-

Table 1: *Materials, Batch Numbers, Compositions, and Instructions for Use*

Material	Composition	Instructions for Use
Ambar, Lot 140410	Etchant: 37% silica-thickened phosphoric acid (H_3PO_4) gel (Condac 37) Adhesive: UDMA, HEMA, and other hydrophilic methacrylate monomers, acid methacrylated monomers, ethanol, silanated silica, photoinitiators, coinitiators, and stabilizers	Apply 37% H_3PO_4 to tooth surface for 15 s; rinse and dry (moist); apply 2 consecutive coats of adhesive and brush for 10 s each coat. Gently air thin for 10 s to evaporate solvent. Light cure for 10 s.
Fuji II LC, Lot 0912171	Cavity Conditioner: 20% polyacrylic acid, 3% aluminum chloride hydrate, distilled water, <0.1% food additive Blue No. 1 Liquid: 20%-22% polyacrylic acid, 30%-40% HEMA, 5%-7% 2,2,4,trimethyl hexamethylene dicarbonate, 4%-6% TEGDMA, 5%-15% proprietary ingredient Powder: Aluminosilicate glass	Apply Cavity Conditioner to enamel and dentin surfaces and leave undisturbed for 10 s; rinse with water for 10 s; gently air dry for 5 s, leaving a moist surface. Automatically mix capsules for 10 s; apply to enamel and dentin surfaces; light cure for 20 s.
Ketac Nano Light- curing Glass Ionomer Restorative Quick Mix Capsule, Lot N168565	Primer: Water (40%-50%); HEMA (35%-45%); Vitrebond copolymer (acrylic/itaconic acid copolymer) (10%–15%); photoinitiators Paste A + Paste B: water, Vitrebond copolymer, HEMA, PEGDMA, TEGDMA, Bis-GMA, fluoroaluminosilicate glass, silane-treated zirconia/silica, photoinitiators	Dispense the Ketac Nano primer into a well. Using a fiber tip, apply primer for 15 s to prepared semidry enamel and dentin surfaces. Replenish primer as needed to ensure surfaces are kept wet with primer for the recommended application time. Dry the primer using an air syringe for 10 s. Do not rinse. After drying, the primed surfaces will remain shiny in appearance. Light cure the primed surfaces for 10 s. The light-cured surfaces will appear shiny. Just prior to use, remove Quick Mix Capsule from foil package. When ready to dispense Ketac Nano restorative into preparation, lift orange mix tip until it is in a straight line with the capsule. Do not force beyond stop. Once the nozzle is swung open and activated do not reclose because this may cause a capsule failure. Place capsule into the applier gun such that the capsule holder engages the groove at the plunger end of the capsule. Press the capsule down into the holder as far as it will go. To dispense Ketac Nano, squeeze handle slowly to extrude a small amount of material approximately 2–3 mm in diameter outside the mouth to verify capsule function. Discard this material. Time to dispense paste from Quick Mix Capsule is 90 s. Exceeding the 90-s time may affect properties of Ketac Nano restorative or cause capsule failure. Dispense material directly into the preparation. Keep tip immersed in material to minimize air entrapment
Filtek Supreme Plus, Lots 8XA, 8GR, 8UU, 8EX, 8EK, 8JG, 8CL	Bis-GMA, UDMA, TEGDMA, Bis-EMA, silanated silica, silanated zirconia, photoinitiators	

Abbreviations: Bis-EMA, ethoxylated bisphenol-A dimethacrylate; Bis-GMA, bisphenol A diglycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; PEGDMA, polyethylene glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

tion, intraoral color photographs were collected at baseline and at the recall appointments. Clinical photographs consisted of digital images taken at 1.5× magnification using a Nikon D40X camera with a 200-mm Medical Nikkor lens (Nikon, Inc, Melville, NY, USA). Statistical analyses included the Mann Whitney nonparametric test to compare the performance of the three restorative materials at each recall, as well as the McNemar nonparametric test to compare the changes of each material from baseline

Table 2: *UNC-modified USPHS Direct Evaluation Criteria*	
Color match	*Alfa* = No mismatch in room light in 3–4 s.
	(Margins exempted from grading)
	(Interfacial staining should not affect grading)
	Bravo = Perceptible mismatch (but clinically acceptable)
	Charlie = Esthetically unacceptable (clinically unacceptable)
Marginal staining	*Alfa* = None
	Bravo = Superficial staining (removable, usually localized)
	Charlie = Deep staining (not removable, generalized)
Recurrent caries	*Alfa* = None
	Charlie = Present
Wear	*Alfa* = No perceptible wear (or only localized wear)
	Bravo = Generalized wear (but clinically acceptable)
	(<50% of margins are detectable)
	(Catches explorer going from material to tooth)
	Charlie = Wear beyond the DEJ (clinically unacceptable)
Marginal adaptation (ditching)	*Alfa* = Undetectable
	Bravo = Detectable (V-shaped defect in enamel only)
	(catches explorer going both ways)
	Charlie = Detectable (V-shaped defect to DEJ)
Surface texture	*Alfa* ewd = >Smooth (better than or equal to microfilled standard)
	Bravo = Rougher than microfilled standard
	Charlie = Pitted
Preoperative sensitivity	*Alfa* = None
	Charlie = Present
Postoperative sensitivity	*Alfa* = None
	Charlie = Present
Retention	*Alfa* = Retained
	Bravo = Partially retained
	Charlie = Missing
Fracture	*Alfa* = None
	Bravo = Small chip, but clinically acceptable
	Charlie = Failure due to bulk restoration fracture

Abbreviations: UNC, University of North Carolina; USPHS, United States Public Health Service

to six months and to one year (PASW Statistics 18.0, SPSS Inc, Chicago, IL, USA). The level of significance was set at $p < 0.05$.

RESULTS

At six months after initial placement, 84 restorations (a 91.3% recall rate) were evaluated. At one year, 78 restorations (a 84.8% recall rate) were available for evaluation. A summary of direct evaluations is shown in Table 3.

Two restorations were lost at six months for Ambar/FSP. All Fuji II LC and Ketac Nano restorations available for evaluation were retained. The six-month and one-year retention rates were 93.1% and

Table 3: *Summary of Direct Evaluations—Percentage of Restorations That Scored Alfa at Baseline (BL), Six Months, and One Year for Each Parameter*

	Ambar/Filtek Supreme Plus			Fuji II LC		
	BL	6 mo	1 y	BL	6 mo	1 y
Recall level	31/31=100%	29/31=93.6%	27/31=87.1%	31/31=100%	28/31=90.3%	26/31=83.9%
Retention	31/31=100%	27/29=93.1%	25/27=92.6%	31/31=100%	28/28=100%	26/26=100%
Color match	29/31=93.6%	25/29=86.2%	22/27=81.5%	31/31=100%	28/28=100%	26/26=100%
Marginal staining	31/31=100%	25/29=86.2%	22/27=81.5%	31/31=100%	28/28=100%	25/26=96.2%
Recurrent caries	31/31=100%	27/29=93.1%	25/27=92.6%	31/31=100%	28/28=100%	26/26=100%
Wear	31/31=100%	27/29=93.1%	25/27=92.6%	31/31=100%	28/28=100%	26/26=100%
Marginal adaptation	30/31=96.8%	23/29=79.3%	23/27=85.2%	30/31=96.8%	26/28=92.9%	26/26=100%
Pre-operative sensitivity	26/31=83.9%	———	———	28/31=90.3%	———	———
Post-operative sensitivity	30/31=96.8%	27/29=93.1%	24/27=88.9%	31/31=100%	28/28=100%	25/26=96.2%
Surface texture	30/31=96.8%	26/29=89.7%	25/27=92.6%	25/31=80.7%	16/28=57.1%	11/26=43.3%

92.6%, respectively, for Ambar/FSP; 100% and 100%, respectively, for Fuji II LC; and 100% and 100%, respectively, for Ketac Nano with no statistical difference between any pair of groups at each recall.

Sensitivity to air decreased for all three adhesive materials from the preoperative to the postoperative stage, but the difference did not reach statistical significance. For Ambar/FSP, there were no statistical differences for any of the parameters from baseline to six months and to one year. For Fuji II LC, surface texture worsened significantly from baseline to six months and baseline to one year ($p<0.016$ and $p<0.006$, respectively). For Ketac Nano, marginal staining (predominantly enamel margins) increased significantly from baseline to one year ($p<0.008$) and from six months to one year ($p<0.016$). Marginal adaptation was statistically worse at one year compared with baseline ($p<0.008$) only for Ketac Nano. When parameters were compared for pairs of adhesives at each recall (Figure 1, Table 4, Figure 2, and Table 5), Ketac Nano resulted in a significantly worse color match than the other two materials at any of the evaluation periods. At one year, Ketac Nano resulted in significantly worse enamel marginal adaptation than did the other two materials and worse marginal staining than did Fuji II LC. Surface texture was statistically worse for Fuji II LC compared with the other two materials at all evaluation periods.

The only *charlie* ratings were measured for the parameter retention (two lost restorations for Ambar at six months) and for preoperative and postoperative sensitivity.

DISCUSSION

We failed to reject the null hypothesis because the one-year clinical retention of the new nanofilled RMGIC was not statistically different from that of a traditional RMGIC or that of a resin-based etch-and-rinse adhesive combined with a nanofilled composite resin.

We used two RMGICs and one etch-and-rinse adhesive in the present clinical study. RMGICs do not require dentin/enamel phosphoric acid etching. Instead, an aqueous solution of polyacrylic acid is recommended to remove most of the smear layer and expose hydroxyapatite for chemical (ionic) bonding to dentin and enamel surfaces.[23,24] As a result of this

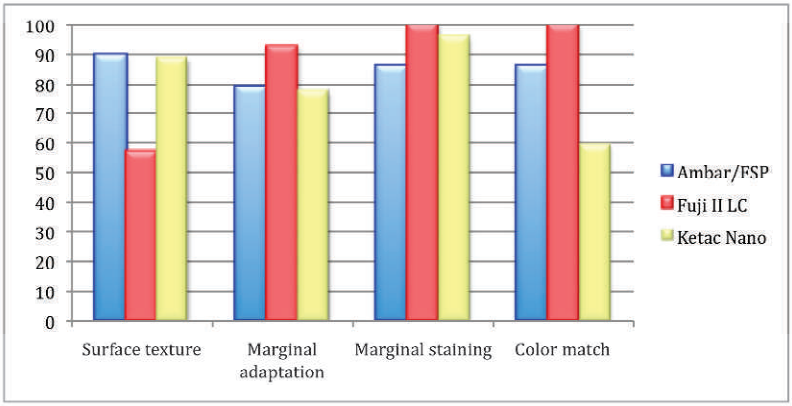
Table 3: Extended.

Ketac Nano		
BL	**6 mo**	**1 y**
30/30=100%	27/30=90.0%	25/30=83.3%
30/30=100%	27/27=100%	25/25=100%
19/30=63.3%	16/27=59.3%	15/25=60.0%
30/30=100%	26/27=96.3%	15/25=60.0%
30/30=100%	27/27=100%	25/25=100%
30/30=100%	26/27=96.3%	24/25=96.0%
29/30=96.7%	21/27=77.8%	17/25=68.0%
25/30=83.3%	———	———
30/30=100%	27/27=100%	24/25=96.0%
29/30=96.7%	24/27=88.9%	23/25=92.0%

mild surface demineralization (not removing all calcium from the demineralized area),[25] RMGICs that use a polyacrylic acid conditioner form a very thin hybrid layer.[20,23]

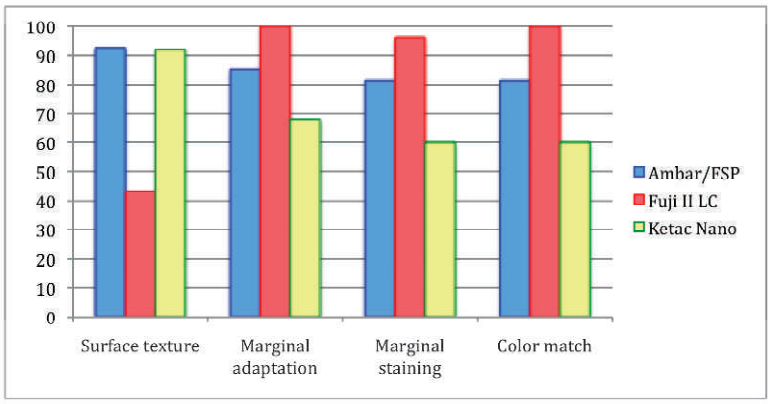
Microtensile dentin bond strengths increase when bur-prepared dentin is treated with the respective polyacrylic acid solution prior to the insertion of Fuji II LC.[26] On smear layer-free dentin, the bond strengths are very similar regardless of the use of the 20% polyacrylic acid solution prior to the insertion of this RMGIC, which attests that the smear layer must be treated to expose calcium bonding sites on the dentin surface. In another study, in which a smear layer was also created with a medium-grit bur, the dentin microtensile bond strengths for Ketac Nano were higher when the respective primer was used compared with nonprimed surfaces.[20] In a shear bond strength study the application of the respective primer improved the bond strengths for Ketac Nano, whereas bond strengths for Fuji II LC were not affected by the use of the respective cavity conditioner.[27]

Although Ketac Nano bonds to dentin *in vitro*, the bonding efficiency of Fuji II LC, as measured with the microtensile bond strength test, is still superior—14.4 megapascals (MPa) for Ketac Nano vs 31.4 MPa for Fuji II LC.[20] In the same study, Ketac Nano



	Surface texture	Marginal staining	Marginal adaptation	Color match
Fuji II LC vs. Ketac Nano	**0.0001**	NS	NS	**0.001**
Fuji II LC vs. Ambar/FSP	**0.022**	NS	NS	NS
Ketac Nano vs. Ambar/FSP	NS	NS	NS	**0.003**

Figure 1. *Percentage of restorations that scored alfa at six months.*



	Surface texture	Marginal staining	Marginal adaptation	Color match
Fuji II LC vs. Ketac Nano	**0.001**	**0.009**	**0.002**	**0.0001**
Fuji II LC vs. Ambar/FSP	**0.0001**	NS	NS	NS
Ketac Nano vs. Ambar/FSP	NS	NS	**0.036**	**0.025**

Figure 2. *Percentage of restorations that scored alfa at one year.*

was reported to interact with dentin and enamel very superficially, without ultrastructural evidence of demineralization and/or hybridization.[20] This phenomenon has been observed with another RMGIC used as base/liner (Vitrebond, 3M ESPE), which bonds to dentin without hybrid layer or gel phase formation and, therefore, only by chemical interaction.[25] According to Coutinho and coworkers, [20] the bonding mechanism of Ketac Nano relies primarily on the micromechanical infiltration into the substrate roughness, combined with the typical chemical bonding provided by the polyalkenoic acid copolymer. Because Ketac Nano contains a monomer and a photoinitiator in its primer (pH=3), it may form a resin coating on the dentin surface prior to the application of the restorative material. Consequently, Ketac Nano's primary bonding mechanism may be similar to that of mild self-etch resin adhesives, given that the increased enamel marginal staining and marginal adaptation resemble those of self-etch adhesives in clinical studies.[11] The secondary bonding mechanism may rely on the polyalkenoic acid copolymer chemical bonding to calcium in hydroxyapatite.

There has been some debate over the years as to whether all GIC-like materials are considered true GICs. Some of these GIC-like materials, such as compomers (polyacid-modified composite resins), have been marketed as belonging to the GIC family. However, compomers resemble composite resins in their physical properties.[28] Additionally, the acid-base reaction in compomers may be merely a surface phenomenon.[28] RMGICs, on the other hand, and in spite of containing a small amount of a polymerizable

Table 4: *Significant Differences of Restorations That Scored Alfa at Six Months*

	Surface Texture	Marginal Staining	Marginal Adaptation	Color Match
Fuji II LC vs. Ketac Nano	**0.0001**	NS	NS	**0.001**
Fuji II LC vs Ambar/FSP	**0.022**	NS	NS	NS
Ketac Nano vs Ambar/FSP	NS	NS	NS	**0.003**

Abbreviations: FSP, Filtek Supreme Plus; NS = not significantly different.

Table 5: *Significant Differences of Restorations That Scored Alfa at One Year*

	Surface Texture	Marginal Staining	Marginal Adaptation	Color Match
Fuji II LC vs Ketac Nano	**0.001**	**0.009**	**0.002**	**0.0001**
Fuji II LC vs Ambar/FSP	**0.0001**	NS	NS	NS
Ketac Nano vs Ambar/FSP	NS	NS	**0.036**	**0.025**

Abbreviations: FSP, Filtek Supreme Plus; NS = not significantly different.

monomer, will still undergo a true acid-base setting reaction. The quantity of the resin is limited to the extent that it will not interfere with the normal acid-base setting reaction, allowing for the ion exchange adhesion with tooth structure that is typical of GICs.[24,29] The increase in the relative resin and filler contents may result in a more attenuated acid-base reaction. Transmission electron microscopy studies in our laboratory (unpublished observations) have shown that the thickness of the silica gel that surrounds the aluminosilicate glass particles, as a result of the interaction of the polycarboxylic acid with the surface of the glass, is more pronounced in Fuji II LC than in Ketac Nano. A recent independent evaluation[30] also reported that Ketac Nano contains more resin than other RMGIC materials do and that its acid-base reaction rate is lower than that of competitive products. This may explain why the gel phase formation and consequent hybridization ability are more pronounced in Fuji II LC than in Ketac Nano.[20]

Although there were no statistical differences for any pair of materials for marginal staining and marginal adaptation at six months, Ketac Nano resulted in worse marginal adaptation than the two groups and worse marginal staining than Fuji II LC at one year. This means that the enamel bonding efficacy of Ketac Nano started to decrease after the six-month recall. Although RMGICs have the tendency for slightly more water sorption than conventional GICs,[31] Ketac Nano has been reported to compensate rapidly for polymerization shrinkage through hygroscopic expansion,[32] as measured by the amount of cusp deflection. When compared with FSP, the nanofilled RMGIC underwent a significant expansion after one week and continued to expand up to 24 months.[32] This characteristic may partially explain the issues with marginal adaptation around enamel margins. Although phosphoric acid etching might have improved the enamel marginal adaptation of Ketac Nano, the application of a RMGIC to acid-etched dentin precludes any ionic interaction with calcium, making hybridization the only viable bonding mechanism.[26] In fact, De Munck and coworkers[23] reported that dentin etching with phosphoric acid, prior to the application of Fuji Bond LC, enhances micromechanical interlocking at the expense of chemical bonding. Further studies with the nanofilled RMGIC should incorporate the enamel selective-etching technique to test the hypothesis that enamel etching improves the marginal adaptation of Ketac Nano.

Color match associated with RMGICs has been less than ideal in several clinical studies. One study[33] reported only 48% *alfa* ratings for one RMGIC after 18 months of clinical service, whereas another study found a poor shade match for two RMGICs at three years in a combination of noncarious and carious cervical lesions.[34] At five years, one clinical study reported a 86% *bravo* rating for one of the first restorative RMGICs.[35] In the present study Fuji II LC resulted in the best color match of the three restorative materials tested, although this difference was only significant when Fuji II LC was compared with Ketac Nano. However, given that surface texture showed signs of degradation starting at the six-month recall, we expect to see deterioration in color match for Fuji II LC in the upcoming 18- and 24-month recalls. Loss of anatomical form and wear have been associated with the deterioration of traditional RMGICs over two years.[33,34] Ketac Nano may have the advantage of more stable surface texture over a longer period of time and, therefore, may behave better than Fuji II LC in this regard. Only further clinical evaluations will test this hypothesis.

The decrease in the quality of surface texture for Fuji II LC has been reported in other clinical studies.[19,34,35] In a clinical trial of NCCL, 47.6% of Fuji II LC restorations were deemed "slightly rough or pitted" at three years, whereas 26.2% were evaluated as "rough, cannot be refinished."[19] In the same study, the cumulative failure rate of Fuji II LC at three years was 7%, whereas that of an acetone-

based two-step etch-and-rinse adhesive was 49%. In spite of the drastic decline in surface texture for Fuji II LC, the low failure rate attests to its bonding efficacy in NCCL.

Ketac Nano resulted in a poor color match starting at the baseline evaluation and remained stable thereafter. Although Ketac Nano's surface texture was comparable to that of the nanofilled composite resin used with Ambar, all operators experienced problems with color matching when using the nanofilled RMGIC. In contrast to other RMGICs that darken with time,[35,36] Ketac Nano restorations were perceived as lighter than the shade selected by the operator prior to starting the restorative procedure. This difficulty may have been a result of two factors. First, there was a reduced number of Ketac Nano shades made available by the respective manufacturer at the time that the restorations were inserted: A1, A2, A3, A3.5, and B2. Because enamel is thinner and dentin in NCCL is usually darker than dentin in the coronal part of the tooth, the availability of darker shades might have resulted in a better color match. Second, it has been reported that the lightness of Ketac Nano increases substantially with increased thickness of the material, as opposed to FSP, for which the respective lightness decreases with the thickness of the composite resin.[37]

Ambar (FGM) is a recently introduced two-step etch-and-rinse adhesive. The six-month clinical behavior in NCCL is comparable to that of the widely tested adhesive Adper Single Bond Plus (3M ESPE).[38] The clinical outcomes measured for Ambar/FSP in the present study were similar to those measured for either Adper Single Bond Plus or Adper Scotchbond Multi-Purpose (3M ESPE) in a recent clinical study using the same composite resin, following the same protocol,[39] which attests to the efficacy of Ambar in NCCL. Furthermore, the dentin-resin interfacial morphology and microtensile bond strengths of Ambar are comparable to those of Adper Single Bond Plus (3M ESPE), even after 20,000 thermal cycles.[40]

One year is a very short period to evaluate the long-term clinical behavior of dental adhesive materials. Nevertheless, this short-term evaluation may allow the ranking of materials regarding their initial bonding capability. All materials tested in this study resulted in retention rates above 90% at one year. Further studies are planned that involve medium- and long-term clinical evaluations of these three adhesive materials. Additionally, *in vitro* studies should test the hypothesis that selective enamel etching improves the enamel marginal integrity associated with Ketac Nano.

CONCLUSIONS

- The one-year retention rate was statistically similar for the three adhesive materials.
- Enamel marginal deficiencies and color mismatch were more prevalent for Ketac Nano.
- Surface texture of Fuji II LC restorations deteriorated quickly.

Conflict of Interest

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

(Accepted 10 January 2012)

REFERENCES

1. Wilson AD, & Kent BE (1971) The glass-ionomer cement, a new translucent dental filling material *Journal of Applied Chemistry and Biotechnology* **21(11)** 313.
2. Sidhu SK, & Watson TF (1995) Resin-modified glass ionomer materials. A status report for the American Journal of Dentistry *American Journal of Dentistry* **8(1)** 59-67.
3. Mitra SB (1991) Adhesion to dentin and physical properties of a light-cured glass-ionomer liner/base *Journal of Dental Research* **70(1)** 72-74.
4. Friedl KH, Powers JM, & Hiller KA (1995) Influence of different factors on bond strength of hybrid ionomers *Operative Dentistry* **20(2)** 74-80.
5. Nicholson JW, & Croll TP (1997) Glass-ionomer cements in restorative dentistry *Quintessence International* **28(11)** 705-714.
6. Mitra SB, Lee CY, Bui HT, Tantbirojn D, & Rusin RP (2009) Long-term adhesion and mechanism of bonding of a paste-liquid resin-modified glass-ionomer *Dental Materials* **25(4)** 459-466.
7. Fukuda R, Yoshida Y, Nakayama Y, Okazaki M, Inoue S, Sano H, Suzuki K, Shintani H, & Van Meerbeek B (2003) Bonding efficacy of polyalkenoic acids to hydroxyapatite, enamel, and dentin *Biomaterials* **24(11)** 1861-1867.
8. Yiu CK, Tay FR, King NM, Pashley DH, Sidhu SK, Neo JC, Toledano M, & Wong SL (2004) Interaction of glass-ionomer cements with moist dentin *Journal of Dental Research* **83(4)** 283-289.
9. Yip HK, Tay FR, Ngo HC, Smales RJ, & Pashley DH (2001) Bonding of contemporary glass ionomer cements to dentin *Dental Materials* **17(5)** 456-470.
10. Tay FR, Smales RJ, Ngo H, Wei SH, & Pashley DH (2001) Effect of different conditioning protocols on adhesion of a GIC to dentin *Journal of Adhesive Dentistry* **3(2)** 153-167.
11. Peumans M, Kanumilli P, De Munck J, Van Landuyt K, Lambrechts P, & Van Meerbeek B (2005) Clinical effectiveness of contemporary adhesives: A systematic review of current clinical trials *Dental Materials* **21(9)** 864-881.

12. Belli S, Unlu N, & Ozer F (2001) Bonding strength to two different surfaces of dentin under simulated pulpal pressure *Journal of Adhesive Dentistry* **3(2)** 145-152.
13. Van Meerbeek B, de Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, Van Landuyt K, Lambrechts P, & Vanherle G (2003) Buonocore Memorial Lecture. Adhesion to enamel and dentin: Current status and future challenges *Operative Dentistry* **28(3)** 215-235.
14. Inoue S, Van Meerbeek B, Abe Y, Yoshida Y, Lambrechts P, Vanherle G, & Sano H (2001) Effect of remaining dentin thickness and the use of conditioner on microtensile bond strength of a glass-ionomer adhesive *Dental Materials* **17(5)** 445-455.
15. Tay FR, Smales RJ, Ngo H, Wei SH, & Pashley DH (2001) Effect of different conditioning protocols on adhesion of a GIC to dentin *Journal of Adhesive Dentistry* **3(2)** 153-167.
16. Lin A, McIntyre NS, & Davidson RD (1992) Studies on the adhesion of glass-ionomer cements to dentin *Journal of Dental Research* **71(11)** 1836-1841.
17. Tyas MJ, & Burrow MF (2002) Clinical evaluation of a resin-modified glass ionomer adhesive system: Results at five years *Operative Dentistry* **27(5)** 438-441.
18. Peumans M, Van Meerbeek B, Lambrechts P, & Vanherle G (2003) Two-year clinical effectiveness of a resin-modified glass-ionomer adhesive *American Journal of Dentistry* **16(6)** 363-368.
19. van Dijken JW (2000) Clinical evaluation of three adhesive systems in class V non-carious lesions. *Dental Materials* **16(4)** 285-291.
20. Coutinho E, Cardoso MV, De Munck J, Neves AA, Van Landuyt KL, Poitevin A, Peumans M, Lambrechts P, & Van Meerbeek B (2009) Bonding effectiveness and interfacial characterization of a nanofilled resin-modified glass-ionomer *Dental Materials* **25(11)** 1347-1357.
21. Ketac Nano Light-Curing Glass Ionomer Technical Data Sheet; Retrieved online October 19, 2011 from: http://multimedia.3m.com/mws/mediawebserver?mwsId=66666UuZjcFSLXTtnxTclxMEEVuQEcuZgVs6EVs6E666666–&fn=ketac_nano_tds.pdf
22. Swift EJ, Perdigão J, Heymann HO, Wilder AD, Bayne SC, May KN, Sturdevant JR, & Roberson TM (2001) Clinical evaluation of a filled and unfilled dentin adhesive *Journal of Dentistry* **29(1)** 1-6.
23. De Munck J, Van Meerbeek B, Yoshida Y, Inoue S, Suzuki K, & Lambrechts P (2004) Four-year water degradation of a resin-modified glass-ionomer adhesive bonded to dentin *European Journal of Oral Sciences* **112(1)** 73-83.
24. Tyas MJ (2003) Milestones in adhesion: Glass-ionomer cements *Journal of Adhesive Dentistry* **5(4)** 259-266.
25. Coutinho E, Yoshida Y, Inoue S, Fukuda R, Snauwaert J, Nakayama Y, De Munck J, Lambrechts P, Suzuki K, & Van Meerbeek B (2007) Gel phase formation at resin-modified glass-ionomer/tooth interfaces *Journal of Dental Research* **86(7)** 656-661.
26. Cardoso MV, Delmé KIM, Mine A, Neves AA, Coutinho E, De Moor RJG, & Van Meerbeek B (2010) Towards a better understanding of the adhesion mechanism of resin-modified glass-ionomers by bonding to differently prepared dentin *Journal of Dentistry* **38(11)** 921-929.
27. Attar M, & Nathanson D (2011) Conditioner/primer effect on two resin modified glassionomers *Journal of Dental Research* **90(Special Issue A)** Abstract #1115 (http://www.dentalresearch.org).
28. Gladys S, Van Meerbeek B, Braem M, Lambrechts P, & Vanherle G (1997) Comparative physico-mechanical characterization of new hybrid restorative materials with conventional glass-ionomer and resin composite restorative materials *Journal of Dental Research* **76(4)** 883-894.
29. Mount GJ, Patel C, & Makinson OF (2002) Resin modified glass-ionomers: Strength, cure depth, and translucency *Australian Dental Journal* **47(4)** 339-343.
30. USAF Dental Evaluation & Consultation Service (2010) Ketac Nano Light-Curing Glass Ionomer Restorative (3M ESPE) (Project #07-025) (11/10). Retrieved online March 31, 2012 from: http://airforcemedicine.afms.mil/idc/groups/public/documents/afms/ctb_143573.pdf
31. Sidhu SK, Pilecki P, Sherriff M, & Watson TF (2004) Crack closure on rehydration of glass-ionomer materials *European Journal of Oral Sciences* **112(5)** 465-469.
32. Versluis A, Tantbirojn D, & DeLong R (2010) Does hygroscopic expansion compensate polymerization shrinkage? *Journal of Dental Research* **89(Special Issue A)** Abstract #455 (www.dentalresearch.org).
33. Neo J, Chew CL, Yap A, & Sidhu S (1996) Clinical evaluation of tooth-colored materials in cervical lesions *American Journal of Dentistry* **9(1)** 15-18.
34. Folwaczny M, Loher C, Mehl A, Kunzelmann KH, & Hickel R (2001) Class V lesions restored with four different tooth-colored materials—3-year results *Clinical Oral Investigations* **5(1)** 31-39.
35. Loguercio AD, Reis A, Barbosa AN, & Roulet JF (2003) Five-year double-blind randomized clinical evaluation of a resin-modified glass ionomer and a polyacid-modified resin in noncarious cervical lesions *Journal of Adhesive Dentistry* **5(4)** 323-332.
36. Maneenut C, & Tyas MJ (1995) Clinical evaluation of resin-modified glass-ionomer restorative cements in cervical 'abrasion' lesions: One-year results *Quintessence International* **26(10)** 739-743.
37. Hammady N, El-Kassas D, Fahmy O, & El Zogb A (2011) Influence of restorative material thickness on its color and translucency *Journal of Dental Research* **90(Special Issue A)** Abstract #642 (www.dentalresearch.org).
38. Loguercio AD, Ferri L, Ferreira TRC, & Reis A (2011) Clinical evaluation of a new simplified adhesive in cervical lesions *Journal of Dental Research* **90(Special Issue A)** Abstract #1149 (www.dentalresearch.org).
39. Perdigão J, Dutra-Corrêa M, Saraceni CHC, Ciaramicoli MT, Kiyan VH, & Queiroz CS (2012) Randomized clinical trial of four adhesion strategies: 18-month results *Operative Dentistry* **37(1)** 3-11.
40. Perdigão J, Gomes G, & Sezinando A (2011) Bonding ability of three ethanol-based adhesives after thermal fatigue *American Journal of Dentistry* **24(4)** 159-164.

©Operative Dentistry, 2012, **37-6,** 602-609

Laboratory Research

The Effect of Prophylaxis Method on Microtensile Bond Strength of Indirect Restorations to Dentin

CJ Soares • JC Pereira • SJB Souza
MS Menezes • SR Armstrong

Clinical Relevance

The use of cavity cleaners on dentin before indirect resin restoration cementation results in variation in microtensile bond strengths (μTBS). Prophylaxis with aluminum oxide air abrasion, pumice paste, or chlorhexidine produced significantly higher μTBS to dentin than were produced with hydrogen peroxide and a sodium bicarbonate jet.

*Carlos José Soares, DDS, MS, PhD, professor and chairman, Biomechanics Research Group, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlândia, Minas Gerais, Brazil

Janaína Carla Pereira, DDS, MS, Biomechanics Research Group, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlândia, Minas Gerais, Brazil

Silas Júnior Boaventura Sousa, DDS student, Biomechanics Research Group, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlândia, Minas Gerais, Brazil

Murilo Souza Menezes, DDS, MS, PhD, Professor, Biomechanics Research Group, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlândia, Minas Gerais, Brazil

Steven R Armstrong, DDS, PhD, professor and chairman, Department of Operative Dentistry, University of Iowa College of Dentistry, Iowa City, IA, USA

*Corresponding author: Universidade Federal de Uberlândia, Faculdade de Odontologia, Departamento de Dentística e Materiais Odontológicos, Avenida Pará, 1720, Bloco 2B, Sala 24, Campus Umuarama, Uberlândia, Minas Gerais, Brazil CEP, 38400–902; e-mail: carlosjsoares@umuarama.ufu.br

DOI: 10.2341/11-459-L

SUMMARY

The aim of this study was to evaluate the effect of different materials used for dentin prophylaxis on the microtensile bond strengths (μTBS) of adhesively cemented indirect composite restorations. Sixty bovine incisors had the buccal surface ground with wet #600-grit silicon carbide abrasive paper to obtain a flat exposed superficial dentin and were submitted to different prophylaxis protocols, as follows: 3% hydrogen peroxide (HydP); 0.12% chlorhexidine (Chlo); sodium bicarbonate jet (SodB); 50-μm aluminum oxide air abrasion (AirA); pumice paste (PumP), and control group–water spray (Cont). After prophylaxis protocols a resin composite block (3.0 mm × 5.0 mm × 5.0 mm) was adhesively cemented using dual resin cement (Rely X ARC). After 24 hours of water storage, specimens were serially sectioned perpendicular to the bonded interface into 1-mm-thick slices. Each specimen was trimmed with a diamond bur to an hourglass shape with a cross-sectional area of approximately 1.0 mm^2 at the bonded area. Specimens were

tested (μTBS) at 0.5 mm/min using a universal testing machine. Scanning electron microscopy was used to examine the effects of prophylaxis techniques on dentin. Bond strength data (MPa) were analyzed by one-way analysis of variance and failure mode by Fisher test (α=0.05). μTBS data, means (SD), were (different superscripted letters indicate statistically significant differences): AirA, 25.2 (7.2)[a]; PumP, 24.1 (7.8)[a]; Chlo, 21.5 (5.6)[a]; Cont, 20.6 (8.1)[a]; HydP, 15.5 (7.6)[b]; and SodB, 11.5 (4.4)[c]. The use of aluminum oxide air abrasion, pumice paste, and chlorhexidine before acid etching did not significantly affect μTBS to dentin; however, the use of hydrogen peroxide and sodium bicarbonate jet significantly reduced μTBS.

INTRODUCTION

Complete intertubular resin infiltration and hybrid layer formation are critical factors for successful dentin bonding.[1] Oral fluids such as saliva, blood, and crevicular fluid can cause chemical incompatibility with dental materials.[2] Adhesive wetting of the tooth substrate can be inhibited by the presence of blood,[3] saliva,[2] hard-tissue particles from operative procedures, and remnants of temporary cement.[4,5] Several agents are recommended for cleansing of the dentin substrate to optimize resin monomer penetration into the collagen network: these include chlorhexidine,[6,7] hydrogen peroxide,[8] pumice paste,[9,10] sodium bicarbonate jet,[9] and aluminum oxide air abrasion.[5,11]

Applying a mixture of flour of pumice and water results in partial removal of the smear layer and surface erosion as a result of the mechanical abrasion of the pumice particles.[12] Pumice paste has been shown to adequately clean oil-contaminated dentin for etch-and-rinse adhesives.[10] Aluminum oxide air abrasion used as a cavity cleanser produces a rough irregular surface with increased surface area that increases the wettability of adhesive systems to tooth structure to provide additional mechanical retention, similar to that associated with etched enamel.[11] Additionally, a mechanical cleansing protocol, utilized prior to definitive cementation, increases the bond strength of luting cement to dentin following eugenol-containing temporary cements.[4,5]

Different dentin-cleaning agents containing chlorhexidine digluconate isolated or associated with glass particles have been marketed with good performance.[7] The use of chlorhexidine as a cavity cleanser is based on its antimicrobial properties.[7] As a result of the cationic properties of the chlorhexidine, it easily binds to the tooth hydroxyapatite, the pellicle on the tooth surface, salivary proteins, bacteria, and phosphate groups.[6] Several studies have shown that the bond strength of some adhesives was compromised by the use of hydrogen peroxide.[13] The exact mechanism by which hydrogen peroxide affects dentin has yet to be fully understood. Hydrogen peroxide is capable of generating a hydroxyl radical, an oxygen-derived free radical that is known to accumulate in dentin.[14] Residual hydrogen peroxide solution in the collagen matrix and dentinal tubules may eventually break down into oxygen and water.[13] Liberation of oxygen could either interfere with resin infiltration into etched dentin or inhibit polymerization of resins that cure *via* a free-radical mechanism.[13]

The aim of this study was to analyze the influence of prophylaxis method (hydrogen peroxide, sodium bicarbonate jet, chlorhexidine, hydrogen peroxide, pumice paste, and air abrasion) on the microtensile bond strengths (μTBS) of indirect composite restorations fixed on bovine dentin. The null hypothesis was that the dental prophylaxis methods do not affect the μTBS of resin-based composite luted to bovine dentin.

MATERIALS AND METHODS

Sixty bovine teeth were extracted and stored in 0.2% thymol solution (Biopharma, Uberlândia, Brazil). The roots were cut off and the pulp was removed under water irrigation. Flat dentinal surfaces with standardized smear layers were created by grinding buccal surfaces with #600 silicon carbide papers (Norton, Campinas, Brazil). Specimens were randomly divided into six prophylaxis groups, as follows: 1) Cont, no cavity cleaner application, 15-second water-spray, and conventional etching (control group). 2) PumP, rubber cup application of pumice paste for 10 seconds, rinsed with water spray for 10 seconds, etching with 37% phosphoric acid (Adper Etching, 3M-ESPE, St Paul, MN, USA) for 15 seconds, and the surface rinsed with water spray for 15 seconds. 3) Chlo, 10-mL application of 0.12% chlorhexidine solution (Biopharma) for 10 seconds with rubber cup application, rinsed with water spray for 10 seconds, etching with 37% phosphoric acid (Adper Etching) for 15 seconds, and the surface rinsed with water spray for 15 seconds. 4) HydP, 10-mL application of 3% hydrogen peroxide solution (Biopharma) for 10 seconds with rubber cup application, rinsed with water spray for 10 seconds,

etching with 37% phosphoric acid (Adper Etching) for 15 seconds, and the surface rinsed with water spray for 15 seconds. 5) SodB, sodium bicarbonate jet at 60-pound pressure for 10 seconds from 5 mm perpendicular to the surface (Profi II, Dabi Atlante, São Paulo, Brazil); rinsed with water spray for 10 seconds, etching with 37% phosphoric acid (Adper Etching) for 15 seconds, and the surface rinsed with water spray for 15 seconds. 6) AirA, air abrasion with 50-μm aluminum oxide particles at four bars of pressure for 10 seconds from 5 mm perpendicular to the surface (Microjato Plus, Bio-Art, São Paulo, Brazil); rinsed with water spray for 10 seconds, etching with 37% phosphoric acid (Adper Etching) for 15 seconds, and the surface rinsed with water spray for 15 seconds.

Resin composite blocks (3.0 mm × 5.0 mm × 5.0 mm) were incrementally prepared in a silicon matrix with a microhybrid composite resin (Filtek Z250, 3M-ESPE). Each increment was light-cured with a quartz-tungsten-halogen curing lamp (XL 3000, 3M-ESPE) at 600 mW/cm^2 for 40 seconds, as measured by a radiometer (Radiometro, FANEM, São Paulo, SP, Brazil). The intaglio surface of the indirect resin restorations were air abraded with 50 μm aluminum oxide particles (Microjato Plus, Bio-Art) for 10 seconds with four bars of pressure at 10.0 mm of source-to-sample distance and silanized with a pre-hydrolyzed silane solution (Rely X Ceramic Primer, 3M-ESPE) for one minute.

A one-bottle adhesive system (Adper Single Bond 2, 3M-ESPE) was applied, air-dried, and light-cured for 20 seconds at 600 mW/cm^2. Restorations were cemented with a dual cured resin cement (RelyX ARC, 3M-ESPE), according to the manufacturer's instructions. A 500*g* load was applied for five minutes to standardize the luting cement thickness. Visible-light activation of superior and all lateral surfaces on the bonded blocks was performed for 40 seconds for each surface (XL3000, 3M-ESPE). Specimens were stored in distilled water at 37°C for 24 hours. Samples were positioned in a precision diamond cutting machine (1000 Isomet, Buehler Ltd, Lake Bluff, IL, USA) and serially sectioned perpendicular to the bonded interface to obtain four slices of approximately 1 mm in thickness per sample. Each slab was trimmed with a cylindrical diamond bur (#1090, Kg Sorensen, Barueri, SP, Brazil), resulting in an hourglass-shaped specimen with a cross-sectional bonded area of approximately 1.04 ± 0.05 mm^2. Each specimen was fixed to the microtensile testing device (Ciucchi device) with cyanoacrylate glue (Loctite Super Bonder, Henkel Loctite Corporation, Rocky Hill, CT, USA) and tested to failure in tension at 0.5 mm/min in a mechanical testing machine (EMIC DL-2000, São José dos Pinhais, PR, Brazil) to determine μTBS. After fracture, the specimen was removed from the testing apparatus and the cross-sectioned area measured at the site of fracture with a digital caliper (S235, Sylvac, Switzerland). A mean μTBS in MPa was reported for each tooth, with pretest failures ignored in the data analyses. All groups presented a normal and homogeneous distribution; therefore, one-way analysis of variance (ANOVA) with *post hoc* Tukey honestly significantly different (HSD) was used to determine whether there was a significant difference in the bond strengths among the prophylaxis methods.

All fractured specimens were observed under a stereomicroscope (Leika MZ12, Tokyo, Japan) and the failure mode was determined. The failure modes were classified as follows: apparently interfacial (within the adhesive joint), cohesive in dentin, cohesive in the resin cement or resin-based composite restoration, or mixed failures, in which the failures were recorded as the surfaces comprising the dominance of failure of each substrate. Differences in failure modes between groups were tested by Fisher exact test. A p-value of less than 0.05 was used as a criterion for statistical significance.

Additionally, to observe the effects of products used in dentin prophylaxis, specimens were wet-abraded to expose superficial dentin and submitted to the techniques used in this study. The specimens were allowed to dry in an oven at 37°C; next they were gold sputter-coated (MED 010, Balzers, Balzer, Liechtenstein) and observed under a scanning electron microscope (SEM; LEO 435 VP, LEO Electron Microscopy, Cambridge, UK). Representative areas of dentin-treated surfaces were photographed at 5000× magnification.

RESULTS

μTBS data are shown in Table 1. One-way ANOVA revealed a significant effect of prophylaxis methods ($p<0.001$). Tukey HSD test showed that the AirA, PumP, Chlo, and Cont groups presented significantly higher μTBS values than did the HydP and SodB groups, while HydP was significantly higher than SodB.

With regard to failure modes, the HydP (80.0%) and SodB (82.5%) were significantly more likely to produce interfacial failures than were the other three prophylaxis methods (67.5% for PumP, 62.5%

Table 1: *Microtensile Bond Strength Values (MPa) and Statistical Categories*

Prophylaxis Method	μTBS[a] Mean (SD)
AirA, Al_2O_3 air abrasion	25.2 (7.2) A
PumP, pumice paste	24.1 (7.8) A
Chlo, 0.12% chlorhexidine	21.5 (5.6) A
Cont, water-spray (control)	20.6 (8.1) A
HydP, 3% hydrogen peroxide	15.5 (7.6) B
SodB, sodium bicarbonate jet	11.5 (4.4) C

[a] *Different letters indicate significant differences by Tukey honestly significantly different test ($p<0.05$). μTBS, microtensile bond strengths; SD, standard deviation.*

for Chol, and 60% for AirA) or the control group (65%). Additionally, the SodB (15%) and HydP (12.5%) groups were more likely to produce pre-tested failure than were the other three prophylaxis methods (2.5% for PumP, Chol, and AirA) or the control group (5%). On the other hand, the AirA (15%), PumP (15%), Chol (12.5%), and Cont (10%) groups were more likely to have cohesive failure in the dentin mode than were the HydP (5%) and SodB (0%) groups. The same failure distribution was found with regard to mixed failures: AirA (15%), PumP (15%), Chol (12.5%), and Cont (10%) groups were more likely to have cohesive failure in the dentin mode than were the HydP (5%) and SodB (0%) groups (Table 2).

SEM revealed a rough irregular surface on the specimens treated with AirA (Figure 1A), crystalline deposits on the surface of specimens treated with SodB (Figure 1B), and the presence of surface grooves on PumP samples (Figure 1C). However, all of the specimens demonstrated the presence of a remnant smear layer prior to the etching acid (Figure 1A-E), which was removed after using phosphoric acid for all groups (Figure 1F).

DISCUSSION

The null hypothesis was rejected. This *in vitro* study showed that prophylaxis with 3% hydrogen peroxide solution and sodium bicarbonate jet negatively influenced the μTBS of the indirect resin restoration fixed to dentin with resinous cement, as compared to the control group. During bond procedures, isolation of the working field is crucial, as any contamination of a prepared tooth might have a detrimental effect on adhesion and retention of restorative materials.[5] In addition, any dentin prophylaxis method should be efficient while leaving no detrimental remnants of the cleaning agent.

Prophylaxis with 0.12% chlorhexidine solution applied prior to acid etching resulted in no significant effect on μTBS values of a luting procedure realized with dual-cure resinous cement and a one-bottle adhesive system (Table 1). Previous studies[6,15] have demonstrated that chlorhexidine application

Table 2: *Mode of Failure of Results Using Different Prophylaxis Methods*

Prophylaxis Method	Apparently Interfacial	Cohesive in Dentin	Cohesive in Resin Restoration or Resin Cement	Mixed	Premature Failure	Total
Cont	26	4	1	7	2	40
AirA	24	6	2	7	1	40
HydP	32	2	0	1	5	40
SodB	33	0	0	1	6	40
PumpP	27	6	1	5	1	40
Chlo	25	5	3	6	1	40

Abbreviations: Cont, water-spray (control); AirA, Al_2O_3 air abrasion; HydP, 3% hydrogen peroxide; SodB, sodium bicarbonate jet; PumP, pumice paste; Chlo, 0.12% chlorhexidine.

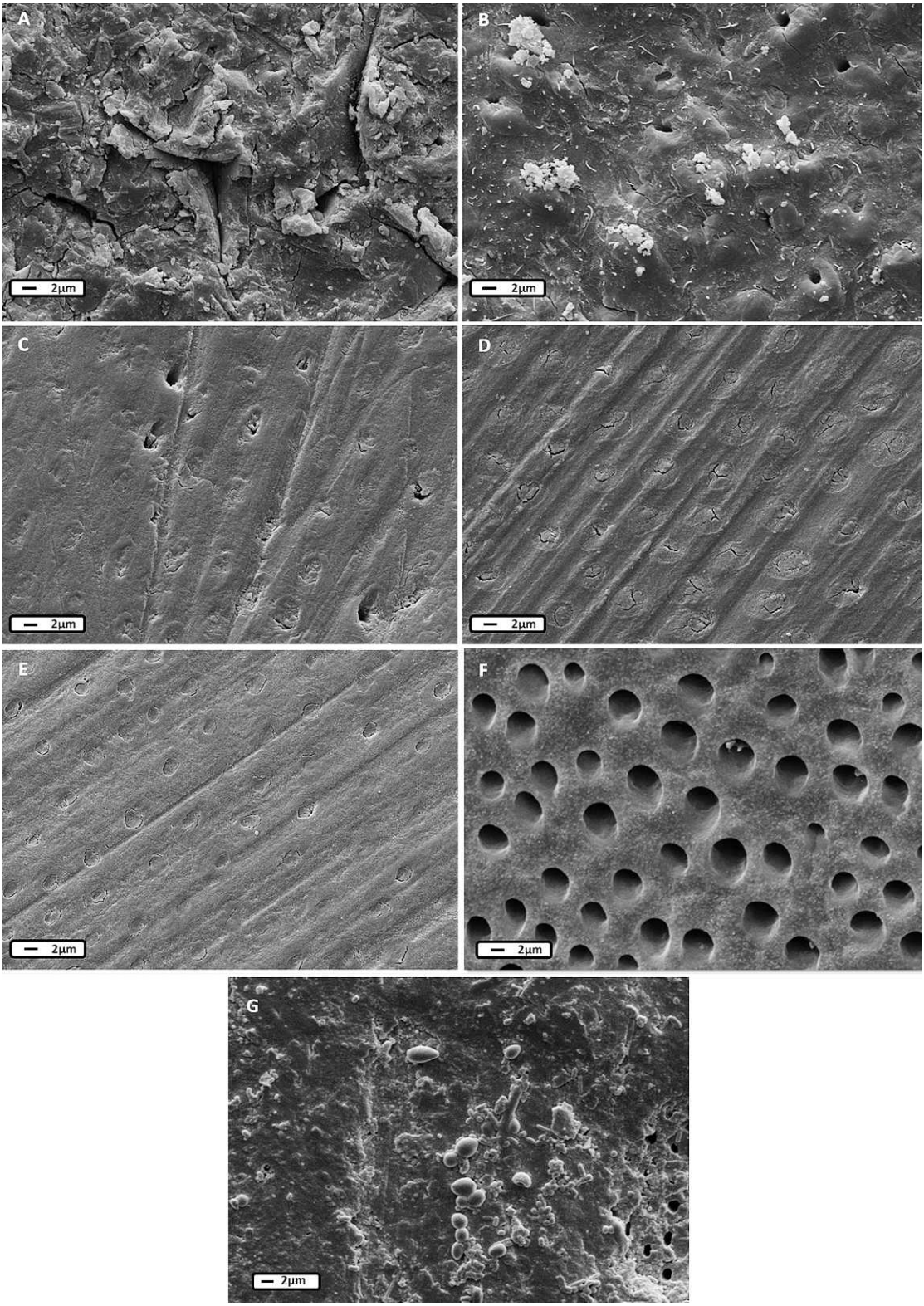


Figure 1. *A. Irregular dentin surface created by air abrasion with 50-µm aluminum oxide particles; B. deposition of crystals and granular residues over dentin surface treated with sodium bicarbonate jet; C. presence of surface grooves and partial removal of smear layer produced by pumice paste; D. dentin surface treated with hydrogen peroxide showing no apparent alteration; E. dentin surface treated with chlorhexidine showing no alteration on surface; F. representative image for all groups of the dentin surface without smear layer completely removed after phosphoric acid etching; G. dentin surface untreated produced by the lab technique. (original magnification 5000×)*

prior to acid etching had no adverse effects on immediate composite-adhesive bonds to dentin. Chlorhexidine applied after acid etching and before use of an adhesive system might be useful for the preservation of dentin bond strength.[16] Chlorhexidine remains the gold standard as an antiplaque and antigingivitis agent.[17] It has been shown that chlorhexidine has an affinity to bacteria, probably because of an interaction between the positively charged chlorhexidine molecule and negatively charged groups on the bacterial cell wall. This interaction increases the permeability of the bacterial cell wall and thus permits the agent to penetrate into the cytoplasm and cause death of the microorganism.[17] Since 0.12% chlorhexidine solution did not produce apparent morphological alteration (Figure 1E) and, additionally, did not produce a negative effect on bond strength to dentin,[6,15] and because it has an antibacterial effect, the use of this product may be a potentially good option for cleaning of cavity preparations before adhesive restorative procedures. It is important to emphasize that the 0.12% chlorhexidine solution used in this study differs from 0.12% mouthwash formulations, which would be contraindicated because they contain glycerin.

Previous studies[4,5] showed that aluminum oxide abrasion was effective in removing cement residues, resulting in better dentin wettability and, consequently, facilitating the infiltration of the adhesive system into the dentin after acid etching. However, another study[18] reported similar bond strengths when acid etching was associated with aluminum oxide abrasion or when it was performed in isolation. As the particles collide with a solid target, in this case the dentin surface, the kinetic energy of the particles is transferred, resulting in microscopic fractures of the target (Figure 1A).[5] As a result, contaminants may be removed from the surface, avoiding the negative effect on the μTBS of the indirect resin restoration.

Cleaning with pumice and water paste is widely considered to be necessary as the last step in cavity preparation or as the first step in the restorative process. Pumice paste does not result in residue deposition on dentin (Figure 1C). In addition to the elimination of residue, pumice paste constitutes an adequate cleaning technique that eliminates bacterial plaque and may produce a thinner smear layer, thus facilitating acid conditioning on dentin.

It is well known that hydrogen peroxide is a strong oxidant and is also weakly acidic, which could increase the wettability of the dentin surface.[19] Hydrogen peroxide affects the inorganic components of dentin through acidic demineralization and attacks the organic-rich intertubular dentin by collagen denaturation.[19] The reduction in bond strength is probably due to these properties of peroxide and their action on dental tissues. Hydrogen peroxide degradation results in the release of water and free radicals from oxygen; these products, however, show no apparent morphological alteration (Figure 1D). The reduction in bond strength of resin-based composite to dentin may also have been due to the presence of free radicals from oxygen that interfered with polymerization of the adhesive resin or adhesive resin cement.[20]

Residues of sodium bicarbonate precipitate on the dentin surface of specimens treated with sodium bicarbonate jet are clearly visible (Figure 1B). The residues of sodium bicarbonate and changes in superficial pH probably interfered with the action of the phosphoric acid, affecting adhesive system and dentin interaction.[21] Despite its efficiency in cleaning pits and fissures as a result of its high degree of penetration in these areas,[9] the sodium bicarbonate jet also has drawbacks, as its residue or pH surface alterations may be responsible for altering phosphoric acid action and for its poor performance on smooth surfaces. Failure analysis demonstrated that the failures for the sodium bicarbonate group were consistently within the interfacial region. The particle deposition resulted in a thin amorphous layer of carbonates and sodium over the dentin surface, which was difficult to remove, resulting in predominantly adhesive failures and six out of 40 pre-test failures.

The hydrogen peroxide solution and sodium bicarbonate jet resulted in more interfacial failure modes and pre-test failures than did the other prophylaxis methods. All other groups resulted in more mixed and cohesive failure than did HydP and SodB. This may be because 1) stress concentration at the interfacial region was not sufficient to create rupture of the bonded joint in more efficacious prophylaxis groups, and 2) hourglass specimen designs concentrate stress in the neck of the dentin or resin, therefore tending to produce cohesive or mixed failures.

There are limitations to the current study. The study results only pertain to the materials used in this study, since only one conventional adhesive system associated with only one dual-cure resin cement was tested. Additional studies are required to determine the effect of the prophylaxis methods using different types of self-adhesive resin cements and with previously contaminated dentin. Addition-

ally, the combination of pumice paste and chlorhexidine may be tested to evaluate for potential synergistic effects on dentin bond strength. The present study tested only 24-hour bond strength and did not include a durability challenge through water storage, thermocycling, cyclic loading, or their combination. Additionally, no temporary cement was used before the definitive indirect restoration cementation.

In a clinical situation, before the adhesive luting procedure the cavity preparation should be cleaned effectively; therefore, the use of mechanical prophylaxis methods that use aluminum oxide air abrasion, pumice paste, or chlorhexidine might be considered before the adhesive integration of the restoration, whereas the use of hydroxide peroxide solution and sodium bicarbonate jet should be avoided as prophylaxis methods before adhesive procedures.

CONCLUSIONS

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

1. The use of products as a cavity cleanser before etching dentin can influence μTBS;
2. The use of hydrogen peroxide and sodium bicarbonate jet significantly reduced μTBS values to dentin; and
3. Aluminum oxide air abrasion, chlorhexidine, and pumice paste before acid etching produced no negative effect on μTBS to dentin.

Acknowledgement

The authors are indebted to Dr EW Kitajima, Dr FAO Tanaka, and RB Salaroli (NAP/MEPAESALQ/USP, Brazil) for the SEM technical support. This study was supported by grants from FAPEMIG and CNPq.

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.

(Accepted 6 February 2012)

REFERENCES

1. Takahashi A, Sato Y, Uno S, Pereira PN, & Sano H (2002) Effects of mechanical properties of adhesive resins on bond strength to dentin *Dental Materials* **18(3)** 263-268.
2. Pinzon LM, Oguri M, O'Keefe K, Dusevish V, Spencer P, Powers JM, & Marshall GW (2010) Bond strength of adhesives to dentin contaminated with smoker's saliva *Odontology* **98(1)** 37-43.
3. Eiriksson SO, Pereira PN, Swift EJ, Heymann HO, & Siqurdsson A (2004) Effects of blood contamination on resin-resin bond strength *Dental Materials* **20(2)** 184-190.
4. Fonseca RB, Martins LR, Quagliatto PS, & Soares CJ (2005) Influence of provisional cements on ultimate bond strength of indirect composite restorations to dentin *Journal of Adhesive Dentistry* **7(3)** 225-230.
5. Chaiyabutr Y, & Kois JC (2008) The effects of tooth preparation cleansing protocols on the bond strength of self-adhesive resin luting cement to contaminated dentin *Operative Dentistry* **33(5)** 556-563.
6. Say EC, Koray F, Tarim B, Soyman M, & Gulmez T (2004) In vitro effect of cavity disinfectants on the bond strength of dentin bonding systems *Quintessence International* **35(1)** 56-60.
7. Saraç D, Bulucu B, Saraç YS, & Kulunk S (2008) The effect of dentin-cleaning agents on resin cement bond strength to dentin *Journal of the American Dental Association* **139(6)** 751-758.
8. Duke ES, Phillips RW, & Blumershine R (1985) Effects of various agents in cleaning cut dentine *Journal of Oral Rehabilitation* **12(4)** 295-302.
9. Sol E, Espasa E, Boj JR, & Canalda C (2000) Effect of different prophylaxis methods on sealant adhesion *Journal of Clinical Pediatric Dentistry* **24(3)** 211-214.
10. Matos AB, Oliveira DC, Vieira SN, Netto NG, & Powers JM (2008) Influence of oil contamination on in vitro bond strength of bonding agents to dental substrates *American Journal of Dentistry* **21(2)** 101-104.
11. Roeder LB, Berry EA III, You C, & Powers JM (1995) Bond strength of composite to air-abraded enamel and dentin *Operative Dentistry* **20(5)** 186-190.
12. Grasso CA, Caluori DM, Goldstein GR, & Hittelman E (2002) In vivo evaluation of three cleansing techniques for prepared abutment teeth *Journal of Prosthetic Dentistry* **88(4)** 437-441.
13. Lai SC, Mak YF, Cheung GS, Osorio R, Toledano M, Carvalho RM, Tay FR, & Pashley DH (2001) Reversal of compromised bonding to oxidized etched dentin *Journal of Dental Research* **80(10)** 1919-1924.
14. Timpawat S, Nipattamanon C, Kijsamanmith K, & Messer HH (2005) Effect of bleaching agents on bonding to pulp chamber dentine *International Endodontic Journal* **38(4)** 211-217.
15. Soares CJ, Pereira CA, Pereira JC, Santana FR, & do Prado CJ (2008) Effect of chlorhexidine application on microtensile bond strength to dentin *Operative Dentistry* **33(2)** 183-188.
16. Carrilho MR, Carvalho RM, de Goes MF, di Hipólito V, Geraldeli S, Tay FR, Pashley DH, & Tjäderhane L (2007) Chlorhexidine preserves dentin bond in vitro *Journal of Dental Research* **86(1)** 90-94.
17. Matthijs S, & Adriaens PA (2002) Chlorhexidine varnishes: A review *Journal of Clinical Periodontology* **29(1)** 1-8.
18. Chaves P, Giannini M, & Ambrosano GM (2002) Influence of smear layer pretreatments on bond strength to dentin *Journal of Adhesive Dentistry* **4(3)** 191-196.

19. Hu X, Ling J, & Gao Y (2010) Effects of irrigation solutions on dentin wettability and roughness *Journal of Endodontics* **36(6)** 1064-1067.

20. Bittencourt ME, Trentin MS, Linden MS, de Oliveira Lima Arsati YB, França FM, Flório FM, & Basting RT (2010) Influence of in situ post bleaching times on shear bond strength of resin-based composite restorations *Journal of the American Dental Association* **141(3)** 300-306.

21. Rosin C, Arana-Chavez VE, Netto NG, & Luz MA (2005) Effects of cleaning agents on bond strength to dentin *Brazilian Oral Research* **19(2)** 127-133.

©Operative Dentistry, 2012, **37-6,** 610-616

Effect of Silver Diamine Fluoride on Microtensile Bond Strength to Dentin

RL Quock • JA Barros • SW Yang
SA Patel

Clinical Relevance

Silver diamine fluoride (SDF), a caries arresting and preventing agent, does not adversely affect the bond strength of resin composite to non-carious dentin. As the evidence grows to support the role of SDF in caries management, its role in operative dentistry will also likely grow.

SUMMARY

The aim of this *in vitro* study was to investigate the effect of the cariostatic and preventive agent silver diamine fluoride (SDF) on the microtensile bond strength of resin composite to dentin. Forty-two caries-free, extracted molars were flattened occlusally and apically using a diamond saw, and the exposed occlusal dentin was polished with a series of silicon carbide papers, all under water irrigation. The teeth were then randomly divided into six groups of seven teeth each that were treated as follows: 1) Peak SE self-etch bonding agent; 2) 12% SDF + Peak SE; 3) 38% SDF + Peak SE; 4) Peak LC etch-and-rinse bonding agent; 5) 12% SDF + Peak LC; and 6) 38% SDF + Peak LC. Four-millimeter buildups of Amelogen Plus were incrementally placed on all teeth; after a 24-hour storage period in distilled water, the specimens were sectioned perpendicular to the adhesive interface to produce beams of cross-sectional surface area measuring approximately 1 mm^2. The beams were placed on a microtensile testing machine, which utilized a single-speed pump motor and force gauge at 20 kgf $\times$ 0.01 second to record maximum tensile force before failure occurred. Two-way analysis of variance and post hoc Tukey tests were performed to compare the effects of the SDF on microtensile bond strength, with statistical significance set at $\alpha = 0.05$. None of the experimental groups treated with different concentrations of SDF showed a significant difference in bond strength compared to the control groups, and there was no significant difference in bond strength between self-etch

*Ryan L Quock, DDS, University of Texas–Houston School of Dentistry, Restorative Dentistry & Biomaterials, Houston, TX, USA

Juliana A Barros, DDS, MS, University of Texas–Houston School of Dentistry, Restorative Dentistry & Biomaterials, Houston, TX, USA

Stephanie W Yang, BM, University of Texas–Houston School of Dentistry, Houston, TX, USA

Shalizeh A Patel, DDS, University of Texas–Houston School of Dentistry, Restorative Dentistry & Biomaterials, Houston, TX, USA

*Corresponding author: 6516 M. D. Anderson Blvd., Ste. 493, Houston, TX 77030, USA; e-mail: Ryan.Quock@uth.tmc.edu

DOI: 10.2341/11-344-L

and etch-and-rinse groups. However, the effect of SDF on self-etch bonded teeth compared to etch-and-rinse bonded teeth was statistically significant (p=0.0363), specifically at the 12% concentration. SDF does not adversely affect the bond strength of resin composite to non-carious dentin.

INTRODUCTION

Dental caries continues to be a prevalent disease, even in developed nations.[1] Traditionally the discipline and practice of operative dentistry have been intimately linked with our understanding of the caries disease process. Indeed, it has been noted that the primary reason directing operative preparation and filling of a tooth is to repair destruction from a carious lesion.[2] In recent years, advancement in our understanding of the caries process[3] and the development of adhesive restorative materials have created the ability to practice with a minimally invasive dentistry philosophy in mind.[4]

Silver diamine fluoride [$Ag(NH_3)_2F$] (SDF) is a solution that has been used to arrest dental caries in countries throughout the world since the early 1970s, primarily in pediatric populations.[5] The components of SDF solution are found in Table 1. Mainly marketed in East Asia and South America, SDF currently does not have Food and Drug Administration approval in the United States—only recently has SDF attracted the attention of the National Institutes of Health.[6] Although its mechanism of action is not well understood, it has been proposed that SDF's chemical components contribute the following benefits: silver-salts stimulate dentin sclerosis/calcification, silver nitrate acts to kill bacteria, and fluoride aids in remineralization and prevention.[7]

The technique for SDF use is fairly simple; a small cotton pellet or brush is used to paint approximately one drop of SDF onto the cavitated carious lesions in a quadrant for at least one minute.[5,7] As long as they are not into the pulp or symptomatic, frank cavitated lesions are preferable because access to salivary minerals enhances remineralization.[5] After the lesions have been exposed to SDF for the determined period of time, the SDF is rinsed off.[7] The main adverse effect that is commonly reported is that the caries lesion is stained dark by the SDF.[7]

Recently published studies[8–10] report that when applied to cavitated caries lesions with no removal of decayed tooth structure, SDF has demonstrated arrest of caries with no further progression after two to three years. It is notable that the lesions in these studies were not filled after treatment with SDF; cavitations were left open. Restorations are generally indicated for cavitated lesions in part to seal out bacteria-harboring plaque and to improve the cleansability of the tooth.[11] Although *in vitro* studies[12,13] indicate that SDF treatment is compatible with glass ionomer restorations, to the authors' knowledge there are no published English-language studies that address the effect of SDF on the bond strength of resin composite to dentin. This *in vitro* study aimed to test the following null hypotheses: 1) SDF has no effect on the bond strength of composite resin to dentin; and 2) the type of adhesive, etch-and-rinse vs self-etch, does not affect the adhesion of composite resin to dentin in teeth treated with SDF.

MATERIALS AND METHODS

Forty-two noncarious, extracted molars were collected according to human subjects' regulations at the University of Texas Health Science Center in Houston, TX (USA) and were stored in 0.9% sodium chloride/0.2% sodium azide solution. Occlusal enamel was ground flat using a model trimmer (Model 3C 1/2HP, Whip Mix, Louisville, KY, USA) under running water, then abraded and smoothed sequentially with 240-, 320-, and 600-grit silicon carbide (SiC) paper on a water-cooled lathe (Ecomet 6, Buehler, Evanston, IL, USA) to expose a flat dentin surface. Tooth roots were removed with a diamond bur and high-speed handpiece. Pulp chambers were cleaned with a large round bur and slow-speed handpiece as well as a spoon excavator; chambers were then filled with bonded resin composite to act as support.

The specimens were randomly divided into six groups of seven teeth each. Each group was further prepared as follows.

Group 1—Exposed flat dentin surface was treated with a self-etch bonding system (Peak SE, Ultradent, South Jordan, UT, USA), according to manufacturer's instructions.

Group 2—Exposed flat dentin surface was treated with 12% SDF solution (Ancarie 12% Cariostatico, Maquira Dental Products, Maringa, PR, Brazil) for three minutes, followed by a rinse for 30 seconds with distilled water.[9] Then the dentin surface was treated with the same self-etch bonding system used in group 1.

Group 3—Exposed flat dentin surface was treated with 38% SDF solution (Saforide, Toyo Seiyaku Kasei Ltd, Osaka, Japan) for three minutes, followed

Table 1: *Manufacturer and Composition of Silver Diamine Fluoride (SDF) Solutions[a] and Adhesives[b]*

Agent	Composition
Ancarie 12% Cariostatico[a] (Maquira Dental Products, Maringa, PR, Brazil)	Ammonium hydroxide, silver nitrate, hydrofluoric acid, water
Saforide[a] (Toyo Seiyaku Kasei Ltd, Osaka, Japan)	Ammonium hydroxide, silver nitrate, hydrofluoric acid, water
Peak SE™[b] Primer Bond (Ultradent, South Jordan, UT, USA)	 Bis-GMA, HEMA, ethanol, methacrylic acid, water Bis-GMA, HEMA, ethanol, methacrylic acid, silica filler
Peak LC™[b] Etch Primer Bond (Ultradent, South Jordan, UT, USA)	 35% Phosphoric acid Bis-GMA, HEMA, ethanol, methacrylic acid, water Bis-GMA, HEMA, ethanol, methacrylic acid, silica filler

Abbreviations: Bis-GMA, bisphenol A diglycidyl ether dimethacrylate; HEMA, 2-hydroxyethyl methacrylate.

by a rinse for 30 seconds with distilled water. Then the dentin surface was treated with the same self-etch bonding system used in group 1.

Group 4—Exposed flat dentin surface was treated with an etch-and-rinse bonding system (Peak LC, Ultradent), according to manufacturer's instructions.

Group 5—Exposed flat dentin surface was treated with 12% SDF solution (Ancarie 12% Cariostatico, Maquira Dental Products) for three minutes, followed by a rinse for 30 seconds with distilled water. Then the dentin surface was treated with the same etch-and-rinse bonding system used in group 4.

Group 6—Exposed flat dentin surface was treated with 38% SDF solution (Saforide, Toyo Seiyaku Kasei Ltd) for three minutes, followed by a rinse for 30 seconds with distilled water. Then the dentin surface was treated with the same etch-and-rinse bonding system used in group 4.

Following treatment with respective adhesives (according to manufacturer instructions), 4-mm-thick buildups of microhybrid composite (Amelogen Plus, Ultradent) were placed, with increments limited to 1 mm. Curing was accomplished with a halogen curing light (Optilux 501, Kerry, Danbury, CT, USA), which was verified to have a light output of 600 mW/cm^2 throughout the study, as indicated by the unit's radiometer.

After storage in distilled water for 24 hours at 37°C, the restored specimens were sectioned occluso-gingivally into serial slabs approximately 1.0 mm thick by a slow-speed water-cooled diamond saw (Isomet 11–1180, Buehler). Each slab was then sectioned into composite/tooth structure beams measuring approximately 1.0 × 1.0 mm in cross section by the same slow-speed water-cooled diamond saw. Ten to 15 beams were yielded from each restored specimen; seven beams from each specimen were chosen at random for testing, for a total of 49 beams for each test group. After storage in distilled water for 24 hours at 37°C, the bonded surface area of each beam was calculated using measurements taken by a digital caliper (Absolute Digimatic, Mitutoyo Corporation, Kawasaki, Kanagawa, Japan). The ends of the beams were affixed to the test block of a microtensile testing machine (BISCO, Schaumburg, IL, USA) with cyanoacrylate glue (Zapit, Dental Ventures of America, Corona, CA, USA). The microtensile testing machine utilized a single-speed pump motor and force gauge at 20 kgf × 0.01 second to record maximum tensile force before failure occurred; force gauge readings in kgf were converted to MPa using the following equation: $1\ kgf/_{mm}{}^2 = 9.80665$ MPa. The type of failure was observed and recorded as adhesive, cohesive in dentin, cohesive in resin, or mixed.

Although seven beams per tooth were tested for microtensile strength, multiple beams derived from the same tooth may not be considered independent

Table 2: *Resin/Dentin Microtensile Bond Strengths in MPa (Standard Deviation [SD])*[a]

Group	n, beams	Bond Strengths[b]	a	cd	cr	m
1: Control with Peak SE	49	32.96 (6.72) A,C	28.6	0.0	69.4	2.0
2: 12% SDF with Peak SE	49	23.99 (8.04) A,*	59.2	0.0	24.5	16.3
3: 38% SDF with Peak SE	49	32.73 (10.54) A,D	61.2	0.0	36.7	2.0
4: Control with Peak LC	49	30.81 (2.55) B,C	61.2	2.0	32.7	4.1
5: 12% SDF with Peak LC	49	39.00 (11.46) B,*	36.7	8.2	32.7	22.4
6: 38% SDF with Peak LC	49	34.41 (10.48) B,D	53.1	0.0	36.7	10.2

Abbreviation: SDF, silver diamine fluoride.
[a] Mean (SD) microtensile bond strength values (MPa) obtained on tested groups (n=7 teeth) and distribution of failure modes (%) (a = adhesive, cd = cohesive in dentin, cr = cohesive in composite resin, m = mixed).
[b] Mean bond strengths with same letters are not significantly different (α=0.05). Mean bond strengths indicated with an asterisk (*) are significantly different (α=0.05). Statistical analysis with two-way ANOVA and post hoc Tukey tests (α=0.05).

samples. Thus, the mean of the seven beams from each tooth was used as the value for that tooth, resulting in a sample size of seven per group. At a significance level of 5%, and assuming a hypothesized effect size no less than 1.25, a sample size of seven could provide a statistical power of 90%. Two-way analysis of variance (ANOVA) and post hoc Tukey tests were performed to compare the effects of the SDF on bond strengths, with statistical significance set at $\alpha = 0.05$. The statistical unit was teeth, not beams.

RESULTS

The study used a total of 42 teeth, with the bond strength for each tooth being determined by the average bond strength of seven beams from each tooth. The means and standard deviations for each tooth are shown in Table 2. None of the experimental groups treated with different concentrations of SDF showed a significant difference in bond strength compared to the control groups, and there was no significant difference in bond strength between self-etch and etch-and-rinse groups. However, the effect of SDF on self-etch bonding compared to etch-and-rinse bonding was statistically significant (p=0.0363). There was no significant difference between the self-etch and etch-and-rinse bond strengths for the control groups (no SDF) or the groups treated with 38% SDF solution, but bond strengths were significantly lower for self-etch vs etch-and-rinse when using a 12% SDF solution. In all groups, the most common failure types were adhesive and/or cohesive in resin. Cohesive in resin failures indicate that since the adhesive interface was still intact, actual bond strengths were likely higher than reported.

DISCUSSION

This study sought first to examine if pretreating noncarious dentin with SDF adversely affects the bond strength of composite resin to dentin. As noted earlier, the results from this study indicate no significant difference in bond strengths between control groups (no pretreatment with SDF) and experimental groups (pretreatment with 12% or 38% SDF). Based on the results of this study, the null hypothesis that SDF has no effect on the bond strength of composite resin to dentin is affirmed. The clinical implication of these findings is that if SDF is used to arrest and/or prevent dental caries in a tooth, bond strength to the noncarious dentin of that tooth will be unaffected.

Secondarily, this study sought to examine if self-etch or etch-and-rinse adhesives were preferable for dentin pretreated with SDF. The results from this study indicate that overall there was no significant difference in bond strength between self-etch or etch-and-rinse groups. However, pretreatment with 12% SDF resulted in significantly lower bond strengths for self-etch groups than for etch-and-rinse groups.

This finding is difficult to reconcile, since pretreatment with 38% SDF did not result in significantly different bond strengths for self-etch vs etch-

and-rinse groups. The authors offer the following twofold explanation. The significant difference in bond strength noted with pretreatment with 12% SDF might be related to one of the components of SDF solution—hydrofluoric acid.[14] It has been noted[15] that hydrofluoric acid generally has a detrimental effect on resin bond strength to dentin. Etching with phosphoric acid after exposure to hydrofluoric acid tends to result in higher bond strengths.[15] This phenomenon seems consistent with the finding in this study that etch-and-rinse adhesive outperforms self-etch adhesive when the dentin is pretreated with 12% SDF.

Although etch-and-rinse adhesive in this study also resulted in higher bond strength than did self-etch adhesive when the pretreatment was with 38% SDF, the difference was not statistically significant. This may be due to differences in manufacturing techniques. Saforide is produced and marketed in Japan, and it is only available in 38% solution. Ancarie Cariostatico is produced and marketed in South America, and it is available in 12% or 30% solution. Only 38% Saforide and 12% Ancarie Cariostatico were available to the authors for this study, so it was not possible to compare differing concentrations of SDF made by the same manufacturer. Based on the results of this study, with its limitations, the null hypothesis that the type of adhesive does not affect the adhesion of composite resin to dentin in teeth treated with SDF is rejected. In light of this, it may be preferable to utilize an etch-and-rinse adhesive following pretreatment of dentin with SDF.

The traditional approach to the operative management of cavitated carious lesions is to mechanically remove soft, bacteria-rich, infected dentin prior to filling the cavity with a suitable restorative material[11]; based on the current evidence, this is still a reasonable perspective because highly infected dentin is not remineralizable.[3] However, there is evidence to indicate that removal of soft infected dentin may not be necessary.[16] For example, a landmark 10-year clinical study[17] of ultraconservative restorations indicated that if a well-sealed adhesive resin restoration is placed over infected dentin, the result is an arrested lesion. Furthermore, it has been noted[18] that if a well-sealed resin restoration is placed over infected dentin in a cavitated lesion, bacterial count and activity are reduced over time. From a biological perspective, these findings present an intriguing challenge to the need to remove infected dentin prior to restoration. However, the discussion of need for or extent of caries excavation seems likely to continue, as it has been recently reported[19] that underlying soft infected dentin may compromise the fracture strength of a composite resin filling.

In contrast to bacteria-rich, infected dentin, affected dentin is relatively low in bacterial count and structurally maintains enough collagen to remineralize[3]—it does not need to be removed prior to restorative filling. Similarly, lesions that have arrested do not need to be excavated.[11] Dentin caries that is either affected or arrested tends to demonstrate lower bacterial activity than does infected dentin—What if the active caries process found in infected dentin could be arrested on contact by the dental practitioner? And would there still be rationale for excavation, especially considering the evidence that caries does not progress under sealed restorations?[16–18] Recent clinical studies[8–10] indicate that SDF might be able to address at least the first question, with regard to arresting dental caries.

Based upon the success that SDF has demonstrated *in vivo* in arresting and preventing caries,[8–10] as well as its apparent initial bonding compatibility *in vitro* with glass ionomers[12,13] and composite resins, SDF may potentially find an important niche in the operative dentist's arsenal. The existing clinical studies[8–10] already seem to indicate the usefulness of SDF in a public health setting. With regard to the biological management of caries, SDF seems to offer the ability to chemically control or eliminate the spread of the disease in a frank cavity. This property may reduce the need to use a handpiece or hand instrument to mechanically remove highly infected dentin—the potential to prepare and place a filling without the "drill" would be appealing to both the conservative-minded dentist and the anxious-minded patient.[20] Indeed, evidence indicating that well-sealed margins are the key to halting the progress of caries[16–18] seems to lend support to the use of SDF for this purpose, which itself adds a preventive effect *via* its fluoride content.[7] At least one case report[21] in the literature details placing a glass ionomer restoration over a SDF-treated lesion; in this case, caries is removed from the dentino-enamel junction but left pulpally.

Despite the prospects that SDF offers for cariology and minimally invasive dentistry, questions still remain—the authors can identify at least the following questions that would benefit from further investigation:

- The actual mechanism of action of SDF on a carious lesion is still not well understood[6,7]—What

type of interactions occur at a molecular level that lead to the observed clinical results? One *in vitro* study[22] indicates a strong antibacterial effect of SDF, as it inhibited *Streptococcus mutans* biofilm formation on demineralized dentin.

- What is the nature of the carious dentin after treatment with SDF? Although clinical studies[8–10] have reported that SDF arrests caries, is the tooth structure more akin to affected or sclerotic dentin in physical properties (ie, hardness), or is it simply softened dentin minus active infection? Chu and Lo[23] reported that superficial dentin of primary teeth may experience an increase in microhardness, but deeper dentin appears to be less affected. This may have implications for the longevity of whatever restoration is placed over the lesion.[19]
- Does SDF exert a cytotoxic effect on pulpal cells? SDF is not known to produce pulpal damage[24]; Gotjamanos[25] reported a favorable response in primary teeth treated with SDF, including the formation of reparative dentin. The thickness of dentin required to protect the pulp chamber in SDF-treated teeth is still unknown.
- The final question is an esthetic question. It known that SDF stains the caries lesion with dark coloration[7,24]; furthermore, our study found that noncarious dentin treated with SDF will also stain after the resin adhesive is light-cured—this phenomenon occurred in both self-etch and etch-and-rinse teeth. The dark staining, whether in carious or noncarious dentin, may create an esthetic challenge when the restorative material is composite. An Australian group[12,21,22] has been investigating the use of potassium iodide to mask the staining with a white precipitate; the long-term effect of this treatment, as well as its interaction with various restorative materials, has yet to been determined.

CONCLUSION

To conclude, SDF seems to present exciting possibilities for the minimally invasive treatment and prevention of dental caries. Although this *in vitro* study indicates the bonding compatibility of SDF-treated noncarious dentin with a resin composite filling, further investigation will be beneficial in determining the role of SDF in operative dentistry.

Acknowledgements

The authors would like to acknowledge Drs Jarvis Chan and Tilly Peters for their technical assistance in preparing this article.

Conflict of Interest

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

(Accepted 23 January 2012)

REFERENCES

1. US Department of Health and Human Services (2000) Oral Health in America: A Report of the Surgeon General—Executive Summary *Rockville, MD: US Department of Health and Human Services, National Institute of Dental and Craniofacial Research, National Institutes of Health*.
2. Roberson TM, Heymann HO, & Swift EJ (2002) *Sturdevant's Art and Science of Operative Dentistry,* 4th ed. Mosby, St Louis, Mo.
3. Gonzalez-Cabezas C (2010) The chemistry of caries: Remineralization and demineralization events with direct clinical relevance *Dental Clinics of North America* **54(3)** 469-478.
4. Murdoch-Kinch CA, & McLean ME (2003) Minimally invasive dentistry *Journal of the American Dental Association* **134(1)** 87-95.
5. Walker D, & Yee R (2011) Arrest of caries technique (ACT): Appropriate technology for the clinician and for disadvantaged communities in Nepal; Retrieved online June 20, 2011 from: http://www.dentaid.org/data/dentaid/downloads/Arrest_of_Caries_Technique.pdf
6. Milgrom P, Zero DT, & Tanzer JM (2009) An examination of advances in science and technology of prevention of tooth decay in young children since the Surgeon General's Report on Oral Health *Academic Pediatrics* **9(6)** 404-409.
7. Rosenblatt A, Stamford TCM, & Niederman R (2009) Silver diamine fluoride: A caries "silver-fluoride bullet." *Journal of Dental Research* **88(2)** 116-125.
8. Yee R, Holmgren C, Mulder J, Lama D, Walker D, & van Palenstein Helderman W (2009) Efficacy of silver diamine fluoride for arresting caries treatment *Journal of Dental Research* **88(7)** 644-647.
9. Llodra JC, Rodriguez A, Ferrer B, Menardia D, Ramos T, & Morato M (2005) Efficacy of silver diamine fluoride for caries reduction in primary teeth and first permanent molars of schoolchildren: A 36-month clinical trial *Journal of Dental Research* **84(8)** 721-724.
10. Chu CH, Lo ECM, & Lin HC (2002) Effectiveness of silver diamine fluoride and sodium fluoride varnish in arresting dentin caries in Chinese pre-school children *Journal of Dental Research* **81(1)** 767-770.
11. Kidd EAM (1998) The operative management of caries *Dental Update* **25(3)** 104-110.
12. Knight GM, McIntyre JM, & Mulyani (2006) The effect of silver fluoride and potassium iodide on the bond strength of auto cure glass ionomer cement to dentine *Australian Dental Journal* **51(1)** 42-5.
13. Yamaga M, Koide T, & Hieda T (1993) Adhesiveness of glass ionomer cement containing tannin-fluoride prepa-

ration (HY agent) to dentin—An evaluation of adding various ratios of HY agent and combination with application diamine silver fluoride *Dental Materials Journal* **12(1)** 36-44.

14. Ditterich RG, Vasconcellos Romanelli MCMO, Rastelli MC, Czlusniak GD, & Wambier DS (2006) Diamine silver fluoride: A literature review *Publicacao Universidade Estadual Ponta Grossa Ciencia Biologica da Saude* **12(2)** 45-52.
15. Loomans BA, Mine A, Roeters FJ, Opdam NJ, De Munck J, Huysmans MG, & Van Meerbeek B (2010) Hydrofluoric acid on dentin should be avoided *Dental Materials* **26(7)** 643-649.
16. Ricketts D, Kidd E, Innes NPT, & Clarkson JE (2006) Complete or ultraconservative removal of decayed tissue in unfilled teeth *Cochrane Database of Systematic Reviews* **19(3)** CD003808.
17. Mertz-Fairhurst EJ, Curtis JW, Ergle JW, Rueggeberg FA, & Adair FM (1998) Ultraconservative and cariostatic sealed restorations: Results at year 10 *Journal of the American Dental Association* **129(1)** 55-66.
18. Oong EM, Griffin SO, Kohn WG, Gooch BF, & Caufield PW (2008) The effect of dental sealants on bacteria in caries lesions: A review of the evidence *Journal of the American Dental Association* **139(3)** 271-278.
19. Hevinga MA, Opdam NJ, Frencken JE, Truin GJ, & Huysmans MCDNJM (2010) Does incomplete caries removal reduce strength of restored teeth *Journal of Dental Research* **89(11)** 1270-1275.
20. Quock RL, Patel SA, Falcao FA, & Barros JA (2011) Is a drill-less dental filling possible? *Medical Hypotheses* **77(3)** 315-317.
21. Knight GM, McIntyre JM, Craig GG, & Mulyani (2010) Leave decay in my cavity? You must be kidding! *Dentistry Today* **29(2)** 130-133.
22. Knight GM, McIntyre JM, Craig GG, Mulyani, Zilm PS, & Gully NJ (2009) Inability to form a biofilm of *Streptococcus mutans* on silver fluoride and potassium iodide-treated demineralized dentin *Quintessence International* **40(2)** 155-161.
23. Chu CH, & Lo ECM (2008) Microhardness of dentine in primary teeth after topical fluoride applications *Journal of Dentistry* **36(6)** 387-391.
24. Chu CH, & Lo EC (2008) Promoting caries arrest in children with silver diamine fluoride: A review *Oral Health and Preventive Dentistry* **6(4)** 315-321.
25. Gotjamanos T (1996) Pulp response in primary teeth with deep residual caries treated with silver fluoride and glass ionomer cement ('atraumatic' technique) *Australian Dental Journal* **41(5)** 328-334.

©Operative Dentistry, 2012, **37-6,** 617-624

Influence of Surface Roughness on Mechanical Properties of Two Computer-aided Design/ Computer-aided Manufacturing (CAD/CAM) Ceramic Materials

S Flury • A Peutzfeldt • A Lussi

Clinical Relevance

Dental practitioners who use chairside computer-aided design/computer-aided manufacturing (CAD/CAM) technology should carefully finish and polish their CAD/CAM ceramic restorations as the present study showed that a decrease in surface roughness improved mechanical properties, that is, led to an increase in surface hardness, elastic modulus, and flexural strength for both CAD/CAM ceramic materials.

*Simon Flury, Dr. Med. Dent., research assistant, University of Bern, Department of Preventive, Restorative and Pediatric Dentistry, Bern, Switzerland

Anne Peutzfeldt, DDS, PhD, senior researcher, University of Bern, Department of Preventive, Restorative and Pediatric Dentistry, Bern, Switzerland

Adrian Lussi, Dr. Med. Dent., Dipl. Chem. Ing., professor University of Bern, Head Department of Preventive, Restorative and Pediatric Dentistry, Bern, Switzerland

*Corresponding author: University of Bern, Department of Preventive, Restorative and Pediatric Dentistry, Freiburgstrasse 7, Bern, 3011, Switzerland; e-mail: simon.flury@zmk.unibe.ch

DOI: 10.2341/11-391-L

SUMMARY

The aim of this study was to evaluate the influence of surface roughness on surface hardness (Vickers; VHN), elastic modulus (EM), and flexural strength (FLS) of two computer-aided design/computer-aided manufacturing (CAD/CAM) ceramic materials. One hundred sixty-two samples of VITABLOCS Mark II (VMII) and 162 samples of IPS Empress CAD (IPS) were ground according to six standardized protocols producing decreasing surface roughnesses (n=27/group): grinding with 1) silicon carbide (SiC) paper #80, 2) SiC paper #120, 3) SiC paper #220, 4) SiC paper #320, 5)

SiC paper #500, and 6) SiC paper #1000. Surface roughness (Ra/Rz) was measured with a surface roughness meter, VHN and EM with a hardness indentation device, and FLS with a three-point bending test. To test for a correlation between surface roughness (Ra/Rz) and VHN, EM, or FLS, Spearman rank correlation coefficients were calculated. The decrease in surface roughness led to an increase in VHN from (VMII/IPS; medians) 263.7/256.5 VHN to 646.8/601.5 VHN, an increase in EM from 45.4/41.0 GPa to 66.8/58.4 GPa, and an increase in FLS from 49.5/44.3 MPa to 73.0/97.2 MPa. For both ceramic materials, Spearman rank correlation coefficients showed a strong negative correlation between surface roughness (Ra/Rz) and VHN or EM and a moderate negative correlation between Ra/Rz and FLS. In conclusion, a decrease in surface roughness generally improved the mechanical properties of the CAD/CAM ceramic materials tested. However, FLS was less influenced by surface roughness than expected.

INTRODUCTION

Direct ceramic restorations produced with chairside computer-aided design/computer-aided manufacturing (CAD/CAM) systems are milled by burs coated with diamond abrasive particles of 50- to 60-μm grit size, for example (for Sirona CEREC system; diamond abrasive particles of D64 [≈ mesh 260] grit size; Sirona, Bensheim, Germany). As a result, these ceramic restorations initially show a high surface roughness. The high surface roughness needs to be reduced—normally obtained through finishing and polishing—since surface roughness greatly influences esthetical, biological, and mechanical properties of ceramic restorations. Restoration surfaces of high roughness tend to increase discoloration,[1] may facilitate plaque accumulation,[2,3] and lead to abrasion and increased wear of antagonists.[4,5] Finally, high surface roughness has generally been described to negatively influence porcelain strength.[6–9] Literature is sparse as to the effect of surface roughness on mechanical properties of CAD/CAM ceramic materials. Therefore, the present study aimed to investigate the influence of different surface roughnesses (Ra and Rz) on surface hardness (Vickers; VHN), elastic modulus (EM), and flexural strength (FLS) of one feldspathic and one leucite-reinforced CAD/CAM ceramic material.

The working hypothesis to be tested was that surface roughness influenced mechanical properties with a strong negative correlation between surface roughness and VHN, EM, and FLS for a given ceramic material.

METHODS AND MATERIALS

Sample Preparation

A total of 324 ceramic samples were produced. One hundred sixty-two samples were made of a feldspathic CAD/CAM ceramic material (VITABLOCS Mark II for CEREC; size I8, Vita Zahnfabrik, Bad Säckingen, Germany), and 162 were made of a leucite-reinforced ceramic material (IPS Empress CAD for CEREC; size I8, Ivoclar Vivadent AG, Schaan, Liechtenstein). Specifications of the two ceramic materials are listed in Table 1. To obtain the samples, 81 blocks of each ceramic material were cut in half, and the metal stubs were removed (Isomet Low Speed Saw, Isomet, Lake Bluff, IL). Each cut surface was then ground according to one of six standardized grinding protocols. The standardized grinding was performed with a grinding machine (Tegra Pol 15/Tegra Pol 1, Struers, Ballerup, Denmark) and grinding papers of six different grit sizes (silicon carbide [SiC] papers, diameter 200 mm, Struers; Table 2) to obtain six groups of decreasing surface roughness (n=27/group). All grinding protocols were carried out under water cooling at a speed of 200 rpm for 15 seconds and with a pressure of 15 N. Three samples could be ground simultaneously using mountings made of a self-curing acrylic resin (Paladur, Heraeus Kulzer, Hanau, Germany) to fix the samples in the machine. The SiC paper was changed after each group of three samples had been ground. The samples were then ultrasonically cleaned (TUC-150, Telsonic AG, Bronschhofen, Switzerland) for 1 minute in 100% ethanol and air dried.

Measurement of Surface Roughness

The ground surfaces of the ceramic samples were profilometrically analyzed with a surface roughness meter (Perthometer S2, Mahr GmbH, Göttingen, Germany): the average surface roughness (Ra; μm) and the arithmetic mean height of the surface profile (Rz; μm) were measured. Three measurements per sample were determined over a transverse length of Lt=5.600 mm, with a cutoff value of 0.8 mm and a stylus speed of 0.5 mm/second. The sample was turned 45° for each new measurement. From the three Ra and Rz values per sample, a mean Ra and a mean Rz value were calculated. During the experimental period, the surface roughness meter was monitored with a calibration device (Mahr GmbH) on each day prior to measuring.

Table 1: *Ceramic Materials Used (Manufacturer Information)*

Material	Brand Name (Manufacturer)	Lot No.	Shade	Average Particle Size, μm	Composition	% by Weight
Feldspathic ceramic material	VITABLOCS Mark II for CEREC (Vita, Bad Säckingen, Germany)	0000100038	2M3C	4	SiO_2	56–64
					Al_2O_3	20–23
					Na_2O	6–9
					K_2O	6–8
Leucite-reinforced ceramic material	IPS Empress CAD for CEREC (Ivoclar Vivadent AG, Schaan, Liechtenstein)	N42417	HT A3	1–5	SiO_2	60–65
					Al_2O_3	16–20
					Na_2O	3.5–6.5
					K_2O	10–14

Measurement of VHN and EM

Six VHN and six EM (GPa) measurements per ceramic sample were made simultaneously on the ground surfaces with a hardness indentation device at a force of 10 g for 15 seconds (Fischerscope HM2000, Helmut Fischer GmbH, Sindelfingen, Germany). The six measurements were made in two parallel lines of three measurements each. Each of the two lines was located at a distance of 1 mm from two opposing edges. Programming of the hardness indentation device and reproducible placement of the sample ensured that the indentations were made within exactly the same position on all samples. From the six VHN and EM values per sample, a mean VHN and a mean EM value were calculated.

Measurement of FLS

The two parallel lines along which the indentations had been made on each ceramic sample were marked with a felt pen. Then, the sample was mounted in a diamond blade low-speed saw (Isomet Low Speed Saw, Isomet) to cut one plate of approximately 2-mm thickness from each ceramic sample (range: 1.8-2.2 mm; mean value [standard deviation], 2.03 mm [0.8]). The actual thickness of each ceramic plate was measured with a digital caliper (Mitutoyo IP 65, Kawasaki, Japan) for later calculation of the FLS (MPa). The plates were then ultrasonically cleaned for 1 minute in deionized water and air dried. The plates were placed in a Zwick Z010 universal testing machine (Zwick GmbH & Co, Ulm, Germany) fitted with a custom-made, three-point bending jig (M. E. Mueller Institute, Bern, Switzerland). The ground surface was orientated toward the bearers of the three-point bending jig, ensuring that the felt pen–marked lines were parallel to the bearers. Thus, the plates were loaded from the nonground surface at a

Table 2: *The Six Groups of Decreasing Surface Roughness for Both Ceramic Materials (n=27/Group and Material) According to Standardized Grinding with Silicon Carbide Papers of Six Different Grit Sizes*

Group	Grit # (DIN)	US-Standard (ANSI)	Abrasive Particle Size, μm (Manufacturer Information)
1	80	80	~200
2	120	100–120	~125
3	220	220	~68
4	320	~280	~46
5	500	~360	~30
6	1000	500	~18

Table 3: *Surface Roughness of the Six Groups (Ra/Rz; Minima [Min], Median, and Maxima [Max]) and Decrease in Ra/Rz Compared With the Antecedent Group (Except Group 1) for Both Ceramic Materials*

	Group 1			Group 2				Group 3				Group 4			
	Min	**Median**	Max	Min	**Median**	Max	Decrease	Min	**Median**	Max	Decrease	Min	**Median**	Max	Decrease
Feldspathic ceramic material															
Ra, μm	1.17	**1.68**	2.15	0.82	**1.11**	1.52	−0.57	0.41	**0.56**	0.76	−0.55	0.30	**0.40**	0.58	−0.16
Rz, μm	7.63	**9.95**	12.77	5.46	**7.47**	10.00	−2.48	3.07	**4.00**	5.06	−3.47	2.23	**2.95**	4.26	−1.05
Leucite-reinforced ceramic material															
Ra, μm	1.17	**1.57**	2.29	0.73	**1.11**	1.40	−0.46	0.37	**0.53**	0.73	−0.58	0.25	**0.38**	0.52	−0.15
Rz, μm	7.63	**10.18**	14.30	5.15	**7.31**	8.88	−2.87	2.74	**3.80**	5.43	−3.51	1.72	**2.67**	3.89	−1.13

cross-head speed of 1.0 mm/minute. The breaking load (F_{max}; N) was recorded (testXpert software, V9.0, Zwick GmbH & Co), and the FLS of each plate was calculated in analogy to ISO 6872:[10] FLS = $3F_{max}l / 2bd^2$, where l (mm) was the center-to-center distance between bearers (6.4 mm), b (mm) was the width of the plate (8.3 mm for VITABLOCS Mark II; 8.0 mm for IPS Empress CAD), and d (mm) was the thickness of the plate measured as described above.

Statistical Analysis

To test for a correlation between surface roughness (Ra/Rz) and VHN, EM, or FLS, Spearman rank correlation coefficients were calculated. Calculations were performed with R version 2.13.0 (The R Foundation for Statistical Computing, Vienna, Austria; www.R-project.org).

RESULTS

Surface roughness (Ra/Rz; minima, median, and maxima) as well as the decrease in Ra/Rz compared with the antecedent group (except group 1) is shown in Table 3 for both ceramic materials. The influence of surface roughness (Ra/Rz) on VHN, EM, and FLS of all samples are shown in Figure 1 for the feldspathic ceramic material and in Figure 2 for the leucite-reinforced ceramic material. In each figure, dots of the same color represent the ceramic samples of one group, with each dot indicating the surface roughness (Ra and Rz) and the corresponding VHN, EM, or FLS of one of the 27 ceramic samples in each of the six groups.

Spearman rank correlation coefficients over all six groups for a given material showed a strong negative correlation between surface roughness (Ra and Rz) and VHN or EM but only a moderate negative correlation between surface roughness (Ra and Rz) and FLS (Table 4).

DISCUSSION

In the present study, six groups of decreasing surface roughness were produced on two CAD/CAM ceramic materials by grinding of the ceramic samples with SiC papers. Group 1 intended to mimic the surface roughness of a dental ceramic restoration after the milling process by diamond burs during CAM. Groups 2 to 6 were produced according to the range of surface roughnesses (Ra/Rz) obtained in a previous study with different polishing methods, in which the same ceramic materials and the same profilometric measurement conditions were used.[11] Thus, group 2 showed Ra and Rz values very similar to those produced by a bur coated with diamond particles of 40-μm grit size, whereas group 6 showed Ra and Rz values very similar to those produced with superior polishing methods.

First, the present study showed a strong correlation between surface roughness and VHN, with decreasing surface roughness leading to increasing VHN. Clinically, an increase in VHN implies an increase in resistance to abrasion and thus to a decrease in wear on the surface of a ceramic restoration. An explanation for the correlation shown between surface roughness and VHN is that a high surface roughness implicates unevenness

Table 3: Extended.

Group 5				Group 6			
Min	**Median**	Max	Decrease	Min	**Median**	Max	Decrease
Feldspathic ceramic material							
0.19	**0.27**	0.36	−0.13	0.11	**0.15**	0.20	−0.12
1.46	**2.07**	2.99	−0.88	0.84	**1.14**	1.46	−0.93
Leucite-reinforced ceramic material							
0.17	**0.25**	0.34	−0.13	0.07	**0.15**	0.19	−0.10
1.42	**1.80**	2.48	−0.87	0.57	**1.02**	1.50	−0.78

with pronounced grooves (eg, scratches and undulations) on the surface of a material. Consequently, the tip of the hardness indentor gets into contact with the elevated parts of the rough surface first. However, the tip does not (or only toward the end of the measurement) reach the entire surface of a material including the surface at the bottom of the grooves. Hence, grooves did not account for the ceramic VHN measurement and thus led to lower VHN. In contrast, SiC paper with abrasive particles of small grit size led to a more planar surface with less pronounced grooves. A more planar surface offers continuous resistance to the entire tip of the indentor and thus leads to higher VHN. Although determination of VHN has been described as suitable for measuring the surface hardness of brittle materials such as ceramic materials,[12] VHN measurements on rough surfaces might not correspond to the actual hardness of the ceramic material but might rather characterize the topography of the surface, which can be regarded as a limitation of the method used in the present study. Moreover, it is unclear to what extent any effects of grinding influenced the VHN (eg, effects of compression/compaction[13,14] or thermal changes when grinding is performed with SiC papers comprising abrasive particles of small grit size).

Second, the present study showed a strong correlation between surface roughness and EM, with decreasing surface roughness leading to increasing EM. Clinically, an increase in EM implies a (relative) increase in stiffness or rigidity of a ceramic restoration and thus higher resistance to deformation. The correlation shown between surface roughness and EM may not be surprising considering that EM was simultaneously determined with the same hardness indentation device as was VHN. It remains to be investigated if the EM of a ceramic material determined by means of an indentor (ie, micromechanically determined on the surface) is indeed accurate and if it correlates with EM (macromechanically) determined by bending/tensile tests. Although not quantitatively confirmed, the slope of the stress-strain curves obtained during the measurements of FLS looked similar regardless of surface roughness, which suggests that the EM for a given ceramic material depends on material-dependent, intrinsic properties rather than on surface roughness.

Third, the present study generally showed that decreasing surface roughness led to increasing FLS, which is also supported by previous studies.[8,9,15] Clinically, an increase in FLS implies a higher resistance to chipping and fracture of a ceramic restoration and thus higher longevity. As reflected by the correlation shown in the present study, however, a decrease in surface roughness only moderately increased FLS. De Jager and coworkers concluded that although surface roughness primarily determined strength of a ceramic material, a decrease in strength may also occur when areas and concentrations of stress are present inside the ceramic material.[7] A limited influence of surface roughness on strength of ceramic materials was described in a study of Albakry and coworkers, who observed a poor correlation between surface roughness and FLS of two ceramic materials and concluded that not only surface roughness but also porosity, microstructural stresses, or surface and bulk defects may influence the FLS of ceramic materials.[14]

CONCLUSIONS

- Reducing the surface roughness generally improved the mechanical properties of the CAD/CAM ceramic materials tested. Therefore, direct ceramic restorations produced with chairside CAD/CAM systems need to be finished and polished carefully.
- The working hypothesis was partly accepted. Whereas the correlation between surface roughness and VHN or EM was indeed strong, the correlation between surface roughness and FLS was only moderate.
- The latter correlation indicates that FLS of the CAD/CAM ceramic materials tested might not have been solely influenced by surface roughness.

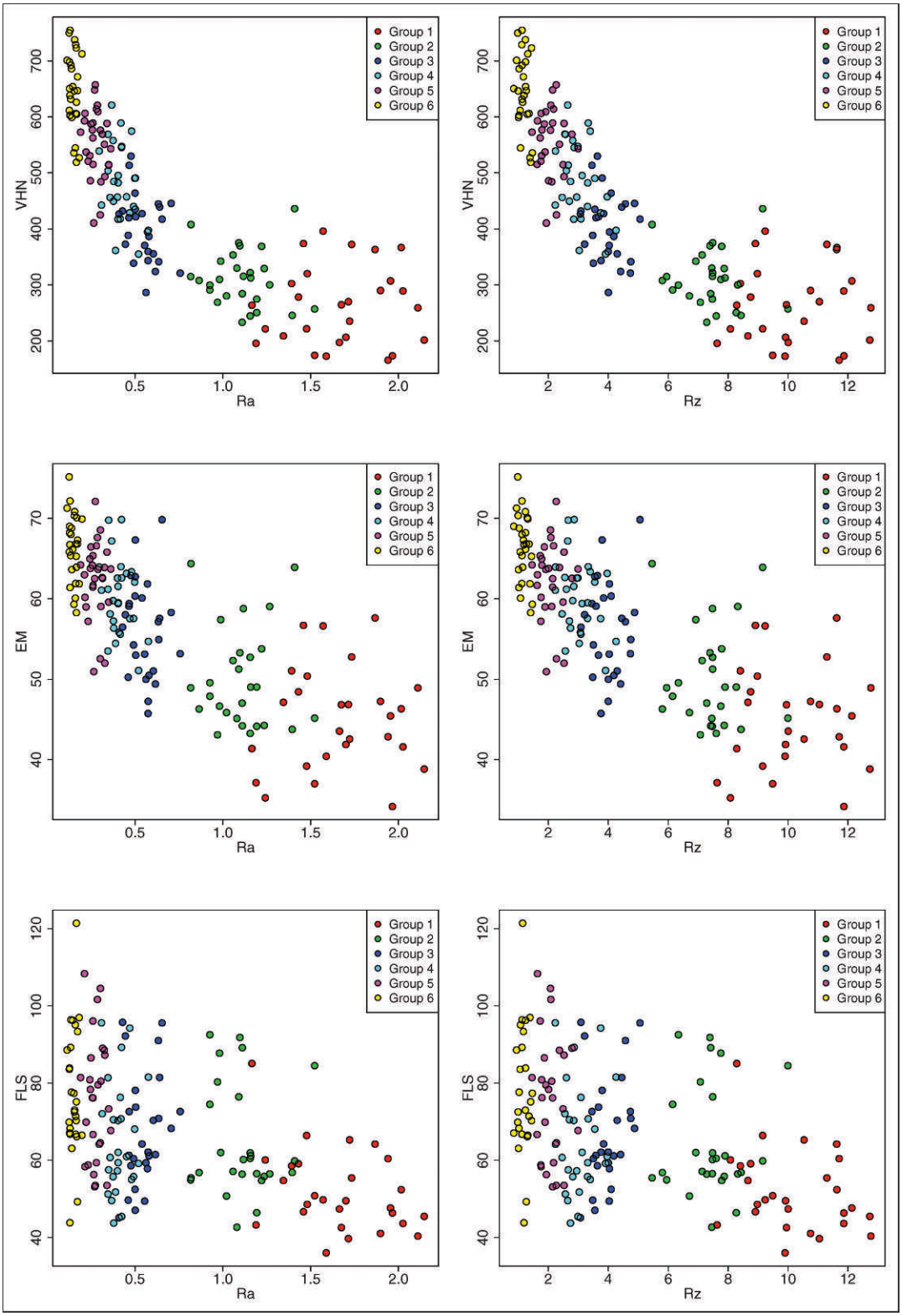


Figure 1. *Feldspathic ceramic material: surface roughness (Ra/Rz; μm) and surface hardness (Vickers; VHN), elastic modulus (EM; GPa), and flexural strength (FLS; MPa) of all samples in the six groups.*

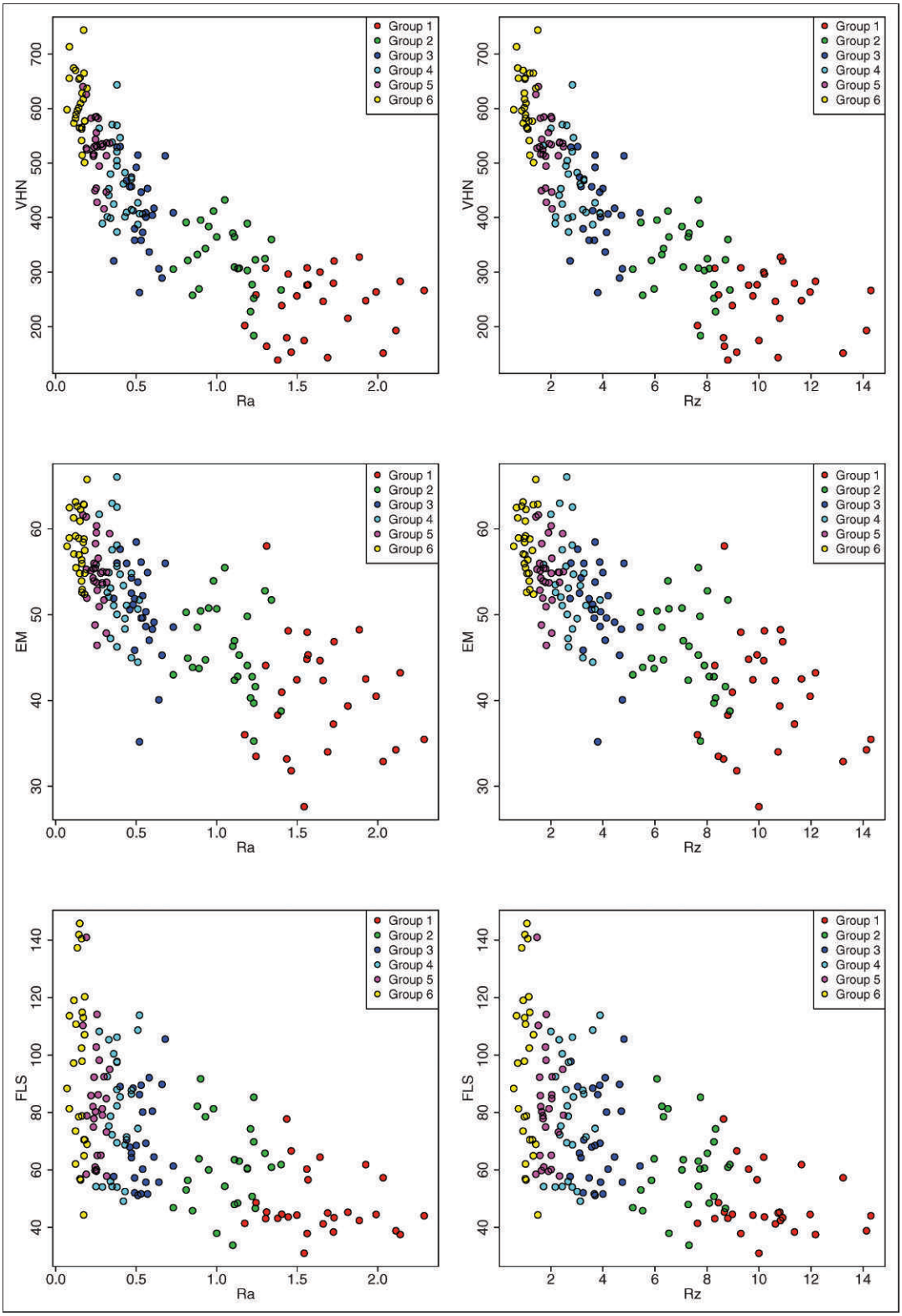


Figure 2. *Leucite-reinforced ceramic material: surface roughness (Ra/Rz; μm) and surface hardness (Vickers; VHN), elastic modulus (EM; GPa), and flexural strength (FLS; MPa) of all samples in the six groups.*

Table 4: *Correlation Between Surface Roughness (Ra/Rz) and Surface Hardness (Vickers; VHN), Elastic Modulus (EM), and Flexural Strength (FLS) for Both Ceramic Materials (Spearman Rank Correlation Coefficients Over All Six Groups for Both Ceramic Materials)*

Correlation	Spearman Rank Correlation Coefficient
Feldspathic ceramic material	
Ra: VHN	−0.9033
Rz: VHN	−0.9000
Ra: EM	−0.7943
Rz: EM	−0.7910
Ra: FLS	−0.4827
Rz: FLS	−0.4804
Leucite-reinforced ceramic material	
Ra: VHN	−0.9025
Rz: VHN	−0.9001
Ra: EM	−0.7856
Rz: EM	−0.7816
Ra: FLS	−0.6054
Rz: FLS	−0.6111

Acknowledgements

The authors are indebted to Vita Zahnfabrik and Ivoclar Vivadent AG for the ceramic blocks. Furthermore, the authors would like to thank S. Hayoz and Prof. Dr. J. Hüsler, Institute of Mathematical Statistics and Actuarial Science, University of Bern, for statistical advice.

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.

(Accepted 27 December 2011)

REFERENCES

1. Yilmaz C, Korkmaz T, Demirköprülü H, Ergün G, & Ozkan Y (2008) Color stability of glazed and polished dental porcelains *Journal of Prosthodontics* **17(1)** 20-24.
2. Bollen CM, Lambrechts P, & Quirynen M (1997) Comparison of surface roughness of oral hard materials to the threshold surface roughness for bacterial plaque retention: a review of the literature *Dental Materials* **13(4)** 258-269.
3. Kawai K, Urano M, & Ebisu S (2000) Effect of surface roughness of porcelain on adhesion of bacteria and their synthesizing glucans *Journal of Prosthetic Dentistry* **83(6)** 664-667.
4. Al-Wahadni AM, & Martin DM (1999) An *in vitro* investigation into the wear effects of glazed, unglazed and refinished dental porcelain on an opposing material *Journal of Oral Rehabilitation* **26(6)** 538-546.
5. Heintze SD, Cavalleri A, Forjanic M, Zellweger G, & Rousson V (2008) Wear of ceramic and antagonist—a systematic evaluation of influencing factors *in vitro* *Dental Materials* **24(4)** 433-449.
6. Griggs JA, Thompson JY, & Anusavice KJ (1996) Effects of flaw size and auto-glaze treatment on porcelain strength *Journal of Dental Research* **75(6)** 1414-1417.
7. De Jager N, Feilzer AJ, & Davidson CL (2000) The influence of surface roughness on porcelain strength *Dental Materials* **16(6)** 381-388.
8. Fischer H, Schäfer M, & Marx R (2003) Effect of surface roughness on flexural strength of veneer ceramics *Journal of Dental Research* **82(12)** 972-975.
9. Nakamura Y, Hojo S, & Sato H (2010) The effect of surface roughness on the Weibull distribution of porcelain strength *Dental Materials Journal* **29(1)** 30-34.
10. ISO-Standards (2008) ISO 6872 Dentistry—Ceramic materials *Geneva: International Organization for Standardization* **3rd edition**.
11. Flury S, Lussi A, & Zimmerli B (2010) Performance of different polishing techniques for direct CAD/CAM ceramic restorations *Operative Dentistry* **35(4)** 470-481.
12. Hamouda IM, El-Waseffy NA, Hasan AM, & El-Falal AA (2010) Evaluation of an experimental dental porcelain *Journal of the Mechanical Behaviour of Biomedical Materials* **3(8)** 610-618.
13. Giordano R, Cima M, & Pober R (1995) Effect of surface finish on the flexural strength of feldspathic and aluminous dental ceramics *International Journal of Prosthodontics* **8(4)** 311-319.
14. Albakry M, Guazzato M, & Swain MV (2004) Effect of sandblasting, grinding, polishing and glazing on the flexural strength of two pressable all-ceramic dental materials *Journal of Dentistry* **32(2)** 91-99.
15. Lohbauer U, Müller FA, & Petschelt A (2008) Influence of surface roughness on mechanical strength of resin composite versus glass ceramic materials *Dental Materials* **24(2)** 250-256.

©Operative Dentistry, 2012, **37-6,** 625-633

Evaluation of Chemical Treatment on Zirconia Surface with Two Primer Agents and an Alkaline Solution on Bond Strength

FC Lorenzoni • VP Leme • LA Santos
PCG de Oliveira • LM Martins • G Bonfante

Clinical Relevance

Achieving a reliable bond to zirconia is still a challenge. The chemical conditioning methods, which do not require the micromechanical topographical changes, enhanced the shear bond strength to zirconia.

*Fabio Cesar Lorenzoni, PhD candidate student, Department of Prosthodontics, Bauru School of Dentistry, São Paulo University, Bauru, Brazil

Viviane Paes Leme, DDS, Bauru, Brazil

Lívia Aguiar Santos, DDS, MS, Department of Prosthodontics, Bauru School of Dentistry, University of São Paulo, Bauru, Brazil

Pedro Cesar Garcia de Oliveira, DDS, MS, PhD, Department of Prosthodontics, Bauru School of Dentistry, University of São Paulo, Bauru, Brazil

Leandro de Moura Martins, DDS, MS, PhD , Federal University of Amazonas, School of Dentistry, Amazonas, Brazil

Gerson Bonfante, DDS, MS, PhD, Department of Prosthodontics, Bauru School of Dentistry, University of São Paulo, Bauru, Brazil

*Corresponding author: al. Octávio Pinheiro Brisola, 9-75, Bauru, São Paulo 17012-180, Brazil; e-mail: fcesarlorenzoni@yahoo.com.br

DOI: 10.2341/11-216-L

SUMMARY

Objectives: **This study evaluated the effect of an alkaline solution and two 10-methacryloyloxydecyl dihydrogen phosphate (MDP)-based primer agents on bond strength to zirconia (yttria-stabilized tetragonal zirconium polycrystal [Y-TZP]) through the shear bond strength (SBS) test.**

Materials and Methods: **Sixty square-shaped Y-TZP samples were embedded in an acrylic resin mold, polished, and randomly assigned to one of six groups (n=10) according to treatment surface: group CR, no treatment (control); group NaOH, 0.5 M NaOH; group AP, Alloy Primer; group ZP, Z-Primer Plus; group NaOH-AP, 0.5 M NaOH + Alloy Primer; and group NaOH-ZP, 0.5 M NaOH + Z-Primer Plus. The resin cement (Rely X U100) was applied inside a matrix directly onto the Y-TZP surface, and it was light-cured for 40**

seconds. The samples were stored in distilled water at 37°C for 24 hours prior to the test, which was performed in a universal machine at a crosshead-speed of 0.5 mm/min. The data were analyzed by one-way analysis of variance and Tukey tests ($p<0.05$). Light stereomicroscopy and scanning electron microscopy were used to assess the surface topography and failure mode.

Results: **The SBS was significantly affected by the chemical treatment ($p<0.0001$). The AP group displayed the best results, and the use of NaOH did not improve SBS results relative to either AP or ZP. The samples treated with Alloy Primer displayed mainly mixed failures, whereas those conditioned with Z-Primer Plus or with 0.5 M NaOH presented a balanced distribution of adhesive and mixed failure modes.**

Conclusions: **The use of a NaOH solution may have modified the reactivity of the Y-TZP surface, whereas the employment of a MDP/6-4-vinylbenzyl-*n*-propyl amino-1,3,5-triazine-2,4-dithione–based primer enhanced the Y-TZP bond strength.**

INTRODUCTION

Fully ceramic systems are attractive materials because of their excellent properties, including high biocompatibility and esthetic potential.[1] The first use of zirconia as a biomaterial involved its use in artificial limbs,[2] whereas its introduction to clinical dentistry has been more recent. Three percent yttria-stabilized tetragonal zirconium polycrystal (Y-TZP) presents a flexural strength and fracture toughness of 1100 MPa[3] and 6.27 MPa $m^{1/2}$,[4] respectively, which are higher than those of alumina (514 MPa[5] and 4.78 MPa $m^{1/2}$ [4]) or lithium disilicate (407 MPa[6] and 3.0 MPa $m^{1/2}$ [7]) ceramics. Nowadays, Y-TPZ is a safe treatment option for single or multiple tooth replacements, including those located in the posterior region.[8–10]

Whereas the main failure mode of all-ceramic systems involves bulk fracture (except for Y-TZP–based restorations),[11] the porcelain veneer fracture represents the most prevalent mechanical complication observed in Y-TZP–based restorations, showing rates of 15%[12] and 20%.[13] Several *in vitro*[14–18] and *in vivo*[12,13,19,20] studies have addressed the reasons behind the chipping of porcelain veneers. On the other hand, a recent three-year follow up[21] determined that 7% of all Y-TZP crowns placed in the molar region lost retention. Although the loss of retention has never been a topic of interest with Y-TZP restorations (because it has never been reported), efforts have been undertaken to establish ways to achieve a reliable bond between Y-TZP crowns and luting agents.[22–34] Comparatively, data from a literature review[11] showed a 2.8% loss of retention rate in all-ceramic systems (except for zirconia) after five years, while for a metal-ceramic system the loss of retention rate decreases to 0.7% after 10 years.[35] Thus, the effective bond strength between the substrate and ceramic plays an important role in enhancing the longevity of restorative treatments[25,29,36–40] and in preventing microleakage.[32]

Although alternatives such as sandblasting with Al_2O_3 particles,[37] tribochemical silica coating,[25] selective infiltration etching,[23,41] and experimental hot etching solution[41] have enhanced the zirconia bond strength, the concept employed for all of these methods relies on the micromechanical interlocking between the zirconia and the cement. Whereas sandblasting has been associated with microcracks, which may decrease the mechanical properties of Y-TZP,[42,43] neither selective infiltration etching or experimental hot etching solution has not been examined with regard to this defect to date. Furthermore, these procedures are invasive methods and often require special equipment. Moreover, there is no consensus about the most appropriate or reliable method with which to bond Y-TZP restorations.

Taking into account the above-mentioned concerns associated with sandblasting,[43] the chemical interaction between zirconia and the luting agent must be evaluated. The chemical adhesion potential of zirconia is low as a result of its inertness (ie, it presents a nonpolar surface),[44] which hampers its union with cements.[27] However, it has been discussed[45] that an increased availability of hydroxyl groups was found at the implant surface of a zirconia/alumina nanocomposite after 15 M sodium hydroxide solution (NaOH) treatment. In addition, durable bond strength may be achieved by employing acid monomers, such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP)-based primers.[32]

Considering that strong chemical bonds with zirconia have been proven to be difficult as a result of zirconia's nonreactive surface,[34] the present study sought to assess *in vitro* the effect of hydroxylation of the zirconia surface on shear bond strength (SBS) with either the combination or exclusion of two commercially available primers. The following null hypothesis was tested: no differences in SBS will be

observed among the groups treated with the different primers with or without the use of NaOH.

MATERIALS AND METHODS

Four pre-sintered blocks of Y-TZP (IPS ZirCAD, InLab Blocks C15, Ivoclar Vivadent, Schaan/Liechtenstein, Germany) were sintered in a furnace (Sintramat, Ivoclar Vivadent) at 1500°C for eight hours. Subsequently, each block was cut on a machine saw (Isomet™ 1000, Buehler, Lake Bluff, IL, USA) at a speed of 500 rpm under constant irrigation with water, producing 60 square-shaped zirconia specimens with dimensions of $1 \times 10 \times 10$ mm. Next, each square-shaped specimen was embedded in acrylic resin mold (ie, so that one side of the Y-TZP square was not clouded by the resin acrylic) to be attached into the shear test jig (Figure 1). All of the materials used in this study are described in Table 1.

The Y-TZP surface was polished with 320-, 400-, and 600-grit carbide silicon papers[28] under abundant water irrigation in a polishing machine (Struers Polisher Metallographic DP-10, São Paulo, SP, Brazil). Subsequently, the Y-TZP surfaces were cleaned with 96° GL alcohol in an ultrasonic bath (Unique USC700, Indaiatuba, SP, Brazil) for three minutes.[46,47] The specimens were randomly divided into six experimental groups (n=10) according to the surface treatment employed, as follows: group CR (control): No treatment was provided to the zirconia surface; group NaOH: A 0.5 M NaOH was applied in excess to the zirconia surface for 60 seconds and then it was washed out with distilled water for 20 seconds and dried with a gentle oil-free air stream for 10 seconds; group AP: The Alloy Primer was applied on the zirconia surfaces in excess with a microbrush and it was allowed to react with the surface for 60 seconds. Afterward, a gentle oil-free air stream was directed for 10 seconds to dry the surface; group ZP: Z-Primer Plus was applied on the zirconia surfaces, also in excess, with a microbrush and it was allowed to react for 60 seconds. Subsequently, a gentle oil-free air stream was applied for 10 seconds to dry the surface; group NaOH-AP: As described for the NaOH group, the 0.5 M NaOH solution was applied on the zirconia surface; this was followed by the application of the Alloy Primer (as described for the AP group); and group NaOH-ZP: As described for the NaOH group, the 0.5 M NaOH solution was applied on the zirconia surface; this was followed by the application of the Z-Primer Plus (as described for the ZP group).

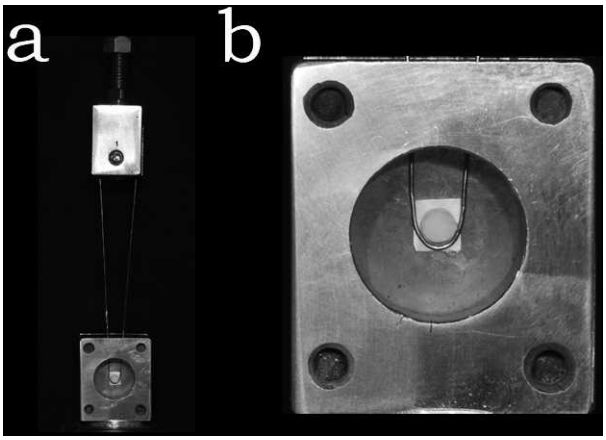


Figure 1. *Shear bond strength testing configuration.*

Afterwards, a polytetrafluoroethylene matrix of 3 mm internal diameter and 3 mm internal height was placed on the center of the Y-TZP square[28] and filled with the luting agent (Rely X U100, 3M ESPE, St Paul, MN, USA). Two minutes after the start of mixing, the cement was light-activated with a halogen light-curing unit (750 mW cm^{-2}; Optilight Plus, Gnatus, Ribeirão Preto, SP, Brazil) for 40 seconds. The preparation of the luting agent and the curing time followed the manufacturer's guidelines. Prior to the tests, all specimens were stored in deionized water in a chamber (Estufa de Secagem 410/1ND, Nova Ética, São Paulo, SP, Brazil) at 37°C for 24 hours. The SBS tests were performed in a universal testing machine (Emic-2000, São José dos Pinhais, PR, Brazil) with a 50-KgF load cell at a constant crosshead speed of 0.5 mm/min. The force was concentrated on the ceramic/cement interface. The shear bond strength (σ) values (MPa) were determined from the following equation:

$$\sigma = \frac{P}{A},$$

in which P is the maximum load (Newtons) required to produce the fracture, and A is the adhesive cross-sectional area (where $A = \pi r^2$). The r denotes the diameter of the bonded area divided by 2, which was measured with a digital caliper (Mitutoyo, Tokyo, Japan).

Additionally, the fractured surfaces were inspected with a light stereomicroscope (Zeiss Stereomicroscope Leica MZE, Mannheim, Germany) at 20× magnification, aided by an external light source (Leica CL5 150D). Using the grading method[26] employed to assess failure modes in zirconia/cement interfaces, the failures were classified into the following categories: 1) cohesive failures in cement; 2) adhesive failures between the ceramic and

Table 1: *Materials, Chemical Compositions, and Manufacturers of the Investigated Materials*

Material	Composition and Batch No.	Manufacturer
e.max ZirCAD	Zirconium dioxide partially stabilized by yttrium in the tetragonal phase (Batch L 15418)	Ivoclar Vivadent, Schaan, Liechtenstein, Germany
Alloy Primer	Acetone, 10-ethacryloyloxydecyl dihydrogen phosphate (MDP), 6-(4-vinylbenzyl-*n*-propyl) amino-1,3,5-triazine-2,4-dithione (VBATDT) (C6415)	Kuraray Medical, Tokyo, Japan
Z-Primer Plus	Biphenyl dimethacrylate, hydroxyethyl methacrylate, ethanol (0900011498)	Bisco, Schaumburg, IL, USA
Rely X U100	Base: fiberglass, acrylic acid ester, dimethacrylate trietilenoglico, silica treated with silane and sodium persulfate	3M ESPE, St Paul, MN, USA
	Catalyst: fiberglass, substitute dimethacrylate, silica treated with silane, *p*-toluenesulfonate sodium and calcium hydroxide (349178)	
Sodium Hydroxide (NaOH)	NaOH 0.5 M, pH 13	Fórmulas Bauru, SP, Brazil

cement; and 3) mixed failures (a combination of adhesive and cohesive failures). Representative specimens from each group were examined under scanning electron microscope (SEM) (Model 3500S, Hitachi Ltd, Osaka, Japan). The data obtained from the SBS test were evaluated using one-way analysis of variance to analyze the chemical surface treatments of Y-TZP. A *post hoc* Tukey test was applied to identify pairwise differences among the tested groups. Both tests employed a pre-set significance level of 5%.

RESULTS

The SBS was significantly affected by the chemical treatment ($p<0.0001$). The highest bond strength values were observed in the NaOH-AP (12.71 MPa) and AP (11.94 MPa) groups and were statistically higher than in other groups. The bond strength value for the NaOH group (6.74 MPa) was comparable to values for the ZP (6.89 MPa) and NaOH-ZP (5.85 MPa) groups, although it was lower than those of the AP and NaOH-AP groups. The use of 0.5 M NaOH did not improve the SBS outcomes relative to either AP or ZP Primers. The CR group (3.69 MPa) showed the lowest bond strength. The average values of SBS, along with standard deviations and minimum and maximum values, are shown in Table 2.

The failure analysis showed that the AP and NaOH-AP groups mainly exhibit mixed failure modes, whereas the NaOH, NaOH-ZP, and ZP groups displayed a balanced distribution between adhesive and mixed failure modes. The results from inspections of the Y-TZP surfaces are presented in Table 3. SEM analysis of one representative failure mode from each group is illustrated in Figure 2. Neither group showed a cohesive failure type. No samples showed spontaneous debonding prior to testing.

Table 2: *Bond Strength, Standard Deviation (SD), and Minimum and Maximum Values Are Described in MPa*

Group	Bond Strength (±SD)[a]	Minimum Value	Maximum Value
CR	3.69 (1.45) A	1.22	6.01
NaOH	6.74 (1.91) B	3.72	9.21
AP	11.94 (3.30) C	8.46	20.07
ZP	6.89 (1.99) B	4.58	10.42
NaOH-AP	12.71 (1.86) C	9.50	15.02
NaOH-ZP	5.85 (2.12) AB	3.05	9.07

Abbreviations: AP, Alloy Primer; CR = control; NaOH, sodium hydroxide; ZP, Z-Primer Plus.
[a] *Identical letters show that the values were not statistically different (Tukey test, $p>0.05$).*

Table 3: *Failure Mode Classifications After Shear Bond Strength (SBS)*

Failure Mode	CR, %	NaOH, %	AP, %	ZP, %	NaOH-AP, %	NaOH-ZP, %
Adhesive	100	40	0	50	20	60
Cohesive	0	0	0	0	0	0
Mixed	0	60	100	50	80	40

Abbreviations: AP, Alloy Primer; CR = control; NaOH, sodium hydroxide; ZP, Z-Primer Plus.

DISCUSSION

Achieving a reliable and adequate bond strength to Y-TZP has proven to be difficult,[48] in part because of Y-TZP's microstructure and chemical composition, which prevent the action of the hydrofluoric acid etch (ie, it does not make any changes with regard to the surface morphology of zirconia).[49,50] Unlike glasses and porcelains,[48] it also fails to respond to silanization procedures.[51] This study evaluated the effect of application of 0.5 M NaOH solution either with or without the association of two primer agents (Alloy Primer and Z-Primer Plus) on bond strength to zirconia. The results revealed that all of the provided chemical treatments increased the SBS when compared with the CR group, which supports the rejection of the null hypothesis.

Although the surface treatment with 0.5 M NaOH did not increase statistically the SBS results when combined with either AP or ZP Primers, it improved the bond strength when compared with the CR group. Two main reasons might explain such a finding. First, it can be speculated that there was activation of the zirconia surface due to the increased availability of hydroxyl groups (OH) after the application of the 0.5 M NaOH. This factor may have favored the acid-base reaction between the metal oxides present on the zirconia surface with both cement and primer agents, which are admittedly acidic. Second, the surface energy may have been raised (mainly the polar component), which could increase the wettability of the zirconia surface, and, as a result, it may have favored the bond reacting between the metal oxides on the zirconia surface and the functional monomer present in the

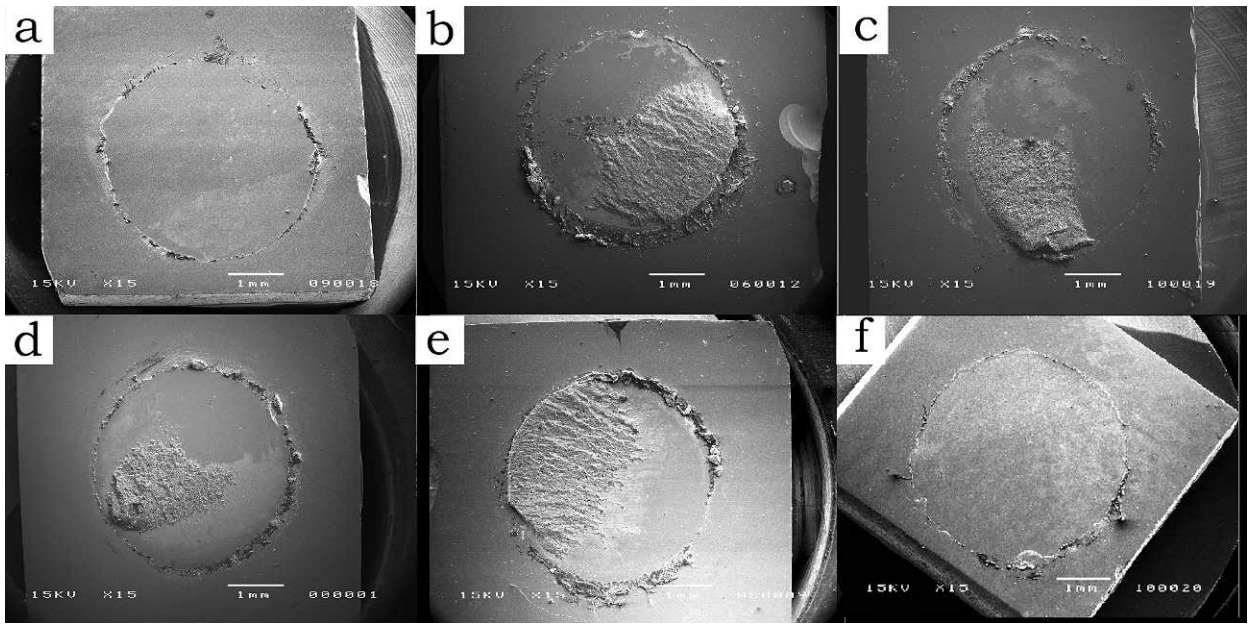


Figure 2. *SEM images showing representative failure modes of each group after SBS testing. (a, f) Images from CR and NaOH-AP groups, respectively, depicting the adhesive failure mode. Images (b), (c), (d), and (e) show the mixed failure mode for AP, ZP, NaOH, and NaOH-AP groups, respectively.*

composition of both primer agents and the resin cement evaluated in this study. When the 0.5 M NaOH solution was associated with Alloy Primer, the best SBS result was recorded. However, this outcome may be more significantly related to the composition of the primer than to the benefits provided by the NaOH solution, since the AP and NaOH-AP results were statistically similar. In addition, this strong base did not affect the performance of the primer itself because there were no statistical differences between the AP and NaOH-AP groups or between the ZP and NaOH-ZP groups. However, the result of the NaOH group was lower when compared with a value limit 10–13 MPa suggested as the minimum for acceptable clinical bonding {Thurmond, 1994 #23}.

Y-TZP surface conditioning with primers has been proposed to increase the bond strengths between cements and zirconia[52]; however, the composition of these primers can result in different bond strength values[6] because they contain different monomers (eg, MDP, 4-methacryloxy- ethyl trimellitate anhydride, thiophosphate methacryloyloxyalkyl derivates, and zirconate coupler).[33] The Alloy Primer was specifically developed to enhance the bond strength in metal surfaces (fabricant information). Therefore, as a result of its chemical composition, the Alloy Primer can result in an improved performance when used in metal alloys. This primer presents two active monomers, MDP and 6-4-vinylbenzyl-*n*-propyl amino-1,3,5-triazine-2,4-dithione (VBATDT), that set up bonds to precious and nonprecious metal oxides, respectively. MDP has been considered an important monomer in bonding to zirconia[31]; thus, it was chosen for evaluation. In this study, the best SBS results were found in the AP and NaOH-AP groups. Considering the composition of the Alloy Primer, it can be speculated that the interaction between MDP and VBATDT (ie, because MDP aids in VBATDT's reaction with the precious metal oxides, which improves the bond strength) produced more reliable bond strength to the zirconia oxide layer. Therefore, this interaction was not investigated in the present study. The NaOH-AP and AP groups were associated with mixed failure modes, which may indicate that adhesions between the cement and ceramics were effective. This finding may be related to the double carbon bonds located at the end of both MDP and VBATDT molecules, which may have bonded to monomers present in the cement composition. High bond strength between Alloy Primer and Rely X U100 have been reported, even after aging.[32]

On the other hand, the results from the NaOH-ZP and ZP groups were greater than those of the CR group, although they were lower when compared with the AP and NaOH-AP groups. This may be related to Z-Primer Plus composition; it contains organophosphate and carboxylic acid monomers.[33] Although both primers present the MDP molecule, the differences found between the two may be a sign that monomers other than MDP can affect the bond strength.[32] Moreover, the Z-Primer Plus produced better SBS values when associated with methacrylate-based monomer (Bis-GMA)–based composites because the presence of acid monomers may weaken the links between Z-Primer Plus and the methacrylate group of the self-etching resin luting cements.[33] The adhesive failure was the main mode associated with the ZP and NaOH-ZP groups (pointing to weaknesses in the zirconia-resin cement bond interface); however, such outcomes do not agree with the findings of a previous report[33] that observed mixed failures as the main failure mode. However, those authors[33] sandblasted all zirconia surfaces with 50 μm aluminum oxide (Al_2O_3) particles. A cohesive failure in zirconia has never been reported with this type of test,[28] and, as expected, it was not found in the present study.

Several *in vitro* studies have attempted to increase zirconia bond strength. Although sandblasting seems to be a mandatory condition to achieve a reliable bond strength to zirconia,[52,53] it may induce defects in the zirconia surface, which may serve as crack initiation sites,[54] decreasing the long-term survival of all-ceramic crowns.[42,43] Many other attempts, such as those involving selective infiltration etching,[22] application of fused glass micro pearls,[48] and the tribochemical silica coating process,[24,55] also have displayed positive results on Y-TZP bond strength, but all of them depend on mechanical damages in the Y-TZP surface. Nevertheless, efforts should be directed to achieving a chemical protocol that does not require any special equipment or produce mechanical damage in the Y-TZP surface. As such, the use of MDP-based primers and alkaline solution seem to contribute to this approach.

This study presented outcomes limited to initial testing after 24 hours in a wet condition. However, the effect of hydroxylation on surface zirconia and its interaction with primer agents should further be evaluated through the use of a thermocycling method, which is often employed to simulated clinical conditions.[52,56,57] However, Y-TZP surface pretreatments proposed in the literature still warrant further clinical prospective controlled stud-

ies to evaluate their real effect on Y-TZP restoration in the long term.

CONCLUSIONS

Within the limitations of this study it was possible to conclude that the use of a 0.5 M NaOH solution modified the reactivity of the zirconia surface, whereas the application of a MDP/VBATDT-based primer enhanced the bond strengths between flat zirconia surfaces and resin luting.

Acknowledgements

This study was supported, in part, by grants from CAPES-Brazil (process No. 4941-10-1). The authors also acknowledge the Bauru Integrated Center for Research (CIP-I).

Conflict of Interest Declaration

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

(Accepted 13 December 2011)

REFERENCES

1. Haselton DR, Diaz-Arnold AM, & Hillis SL (2000) Clinical assessment of high-strength all-ceramic crowns *Journal of Prosthetic Dentistry* **83(4)** 396-401.
2. Cales B (2000) Zirconia as a sliding material: Histologic, laboratory, and clinical data *Clinical Orthopaedics and Related Research* **379** 94-112.
3. Pittayachawan P, McDonald A, Petrie A, & Knowles JC (2007) The biaxial flexural strength and fatigue property of Lava Y-TZP dental ceramic *Dental Materials* **23(8)** 1018-1029.
4. Yilmaz H, Aydin C, & Gul BE (2007) Flexural strength and fracture toughness of dental core ceramics *Journal of Prosthetic Dentistry* **98(2)** 120-128.
5. Chen YM, Smales RJ, Yip KH, & Sung WJ (2008) Translucency and biaxial flexural strength of four ceramic core materials *Dental Materials* **24(11)** 1506-1511.
6. Aboushelib MN, Mirmohamadi H, Matinlinna JP, Kukk E, Ounsi HF, & Salameh Z (2009) Innovations in bonding to zirconia-based materials Part II: Focusing on chemical interactions *Dental Materials* **25(8)** 989-993.
7. Guazzato M, Albakry M, Ringer SP, & Swain MV (2004) Strength, fracture toughness and microstructure of a selection of all-ceramic materials Part II. Zirconia-based dental ceramics *Dental Materials* **20(5)** 449-456.
8. Conrad HJ, Seong WJ, & Pesun IJ (2007) Current ceramic materials and systems with clinical recommendations: A systematic review *Journal of Prosthetic Dentistry* **98(5)** 389-404.
9. Della Bona A, & Kelly JR (2008) The clinical success of all-ceramic restorations *Journal of the American Dental Association* **139(Supplement)** 8S-13S.
10. Sailer I, Gottnerb J, Kanelb S, & Hammerle CH (2009) Randomized controlled clinical trial of zirconia-ceramic and metal-ceramic posterior fixed dental prostheses: A 3-year follow-up *International Journal of Prosthodontics* **22(6)** 553-560.
11. Pjetursson BE, Sailer I, Zwahlen M, & Hammerle CH (2007) A systematic review of the survival and complication rates of all-ceramic and metal-ceramic reconstructions after an observation period of at least 3 years. Part I: Single crowns *Clinical Oral Implants Research* **18(Supplement 3)** 73-85.
12. Raigrodski AJ, Chiche GJ, Potiket N, Hochstedler JL, Mohamed SE, Billiot S, & Mercante DE (2006) The efficacy of posterior three-unit zirconium-oxide-based ceramic fixed partial dental prostheses: A prospective clinical pilot study *Journal of Prosthetic Dentistry* **96(4)** 237-244.
13. Sailer I, Feher A, Filser F, Gauckler LJ, Luthy H, & Hammerle CH (2007) Five-year clinical results of zirconia frameworks for posterior fixed partial dentures *International Journal of Prosthodontics* **20(4)** 383-388.
14. Bonfante EA, Coelho PG, Navarro JM Jr, Pegoraro LF, Bonfante G, Thompson VP, & Silva NR (2010) Reliability and failure modes of implant-supported Y-TZP and MCR three-unit bridges *Clinical Implant Dentisty and Related Research* **12(3)** 235-243.
15. Coelho PG, Bonfante EA, Silva NR, Rekow ED, & Thompson VP (2009) Laboratory simulation of Y-TZP all-ceramic crown clinical failures *Journal of Dental Research* **88(4)** 382-386.
16. Coelho PG, Silva NR, Bonfante EA, Guess PC, Rekow ED, & Thompson VP (2009) Fatigue testing of two porcelain-zirconia all-ceramic crown systems *Dental Materials* **25(9)** 1122-1127.
17. Kim B, Zhang Y, Pines M, & Thompson VP (2007) Fracture of porcelain-veneered structures in fatigue *Journal of Dental Research* **86(2)** 142-146.
18. Kim JH, Kim JW, Myoung SW, Pines M, & Zhang Y (2008) Damage maps for layered ceramics under simulated mastication *Journal of Dental Research* **87(7)** 671-675.
19. Molin MK, & Karlsson SL (2008) Five-year clinical prospective evaluation of zirconia-based Denzir 3-unit FPDs *International Journal of Prosthodontics* **21(3)** 223-227.
20. Tinschert J, Schulze KA, Natt G, Latzke P, Heussen N, & Spiekermann H (2008) Clinical behavior of zirconia-based fixed partial dentures made of DC-Zirkon: 3-Year results *International Journal of Prosthodontics* **21(3)** 217-222.
21. Ortorp A, Kihl ML, & Carlsson GE (2009) A 3-year retrospective and clinical follow-up study of zirconia single crowns performed in a private practice *Journal of Dentistry* **37(9)** 731-736.
22. Aboushelib MN, Kleverlaan CJ, & Feilzer AJ (2007) Selective infiltration-etching technique for a strong and durable bond of resin cements to zirconia-based materials *Journal of Prosthetic Dentistry* **98(5)** 379-388.

23. Aboushelib MN, Matinlinna JP, Salameh Z, & Ounsi H (2008) Innovations in bonding to zirconia-based materials: Part I *Dental Materials* **24(9)** 1268-1272.
24. Atsu SS, Kilicarslan MA, Kucukesmen HC, & Aka PS (2006) Effect of zirconium-oxide ceramic surface treatments on the bond strength to adhesive resin *Journal of Prosthetic Dentistry* **95(6)** 430-436.
25. de Oyague RC, Monticelli F, Toledano M, Osorio E, Ferrari M, & Osorio R (2009) Influence of surface treatments and resin cement selection on bonding to densely-sintered zirconium-oxide ceramic *Dental Materials* **25(2)** 172-179.
26. Kobayashi K, Blatz MB, Saito A, Koizumi H, & Matsumura H (2009) Influence of priming agents on the short-term bond strength of an indirect composite veneering material to zirconium dioxide ceramic *Quintessence International* **40(7)** 545-551.
27. Lohbauer U, Zipperle M, Rischka K, Petschelt A, & Muller FA (2008) Hydroxylation of dental zirconia surfaces: Characterization and bonding potential *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **87(2)** 461-467.
28. Nothdurft FP, Motter PJ, & Pospiech PR (2009) Effect of surface treatment on the initial bond strength of different luting cements to zirconium oxide ceramic *Clinical Oral Investigation* **13(2)** 229-235.
29. Oyague RC, Monticelli F, Toledano M, Osorio E, Ferrari M, & Osorio R (2009) Effect of water aging on microtensile bond strength of dual-cured resin cements to pre-treated sintered zirconium-oxide ceramics *Dental Materials* **25(3)** 392-399.
30. Phark JH, Duarte S Jr, Blatz M, & Sadan A (2009) An in vitro evaluation of the long-term resin bond to a new densely sintered high-purity zirconium-oxide ceramic surface *Journal of Prosthetic Dentistry* **101(1)** 29-38.
31. Wolfart M, Lehmann F, Wolfart S, & Kern M (2007) Durability of the resin bond strength to zirconia ceramic after using different surface conditioning methods *Dental Materials* **23(1)** 45-50.
32. de Souza GM, Silva NR, Paulillo LA, De Goes MF, Rekow ED, & Thompson VP (2010) Bond strength to high-crystalline content zirconia after different surface treatments *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **93(2)** 318-323.
33. Magne P, Paranhos MP, & Burnett LH Jr (2010) New zirconia primer improves bond strength of resin-based cements *Dental Materials* **26(4)** 345-352.
34. Piascik JR, Swift EJ, Thompson JY, Grego S, & Stoner BR (2009) Surface modification for enhanced silanation of zirconia ceramics *Dental Materials* **25(9)** 1116-1121.
35. Walton TR (1999) A 10-year longitudinal study of fixed prosthodontics: Clinical characteristics and outcome of single-unit metal-ceramic crowns *International Journal Prosthodontic* **12(6)** 519-526.
36. Amaral R, Ozcan M, Bottino MA, & Valandro LF (2006) Microtensile bond strength of a resin cement to glass infiltrated zirconia-reinforced ceramic: The effect of surface conditioning *Dental Materials* **22(3)** 283-290.
37. Blatz MB, Chiche G, Holst S, & Sadan A (2007) Influence of surface treatment and simulated aging on bond strengths of luting agents to zirconia *Quintessence International* **38(9)** 745-753.
38. Burke FJ, Fleming GJ, Nathanson D, & Marquis PM (2002) Are adhesive technologies needed to support ceramics? An assessment of the current evidence *Journal of Adhesive Dentsitry* **4(1)** 7-22.
39. Della Bona A, Anusavice KJ, & Hood JA (2002) Effect of ceramic surface treatment on tensile bond strength to a resin cement *International Journal of Prosthodontic* **15(3)** 248-253.
40. Senyilmaz DP, Palin WM, Shortall AC, & Burke FJ (2007) The effect of surface preparation and luting agent on bond strength to a zirconium-based ceramic *Operative Dentistry* **32(6)** 623-630.
41. Casucci A, Osorio E, Osorio R, Monticelli F, Toledano M, Mazzitelli C, & Ferrari M (2009) Influence of different surface treatments on surface zirconia frameworks *Journal of Dentistry* **37(11)** 891-897.
42. Zhang Y, Lawn BR, Malament KA, Van Thompson P, & Rekow ED (2006) Damage accumulation and fatigue life of particle-abraded ceramics *International Journal of Prosthodontics* **19(5)** 442-448.
43. Zhang Y, Lawn BR, Rekow ED, & Thompson VP (2004) Effect of sandblasting on the long-term performance of dental ceramics *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **71(2)** 381-386.
44. Hao L, Lawrence J, & Chian KS (2005) Osteoblast cell adhesion on a laser modified zirconia based bioceramic *Journal of Materials Science: Materials in Medicine* **16(8)** 719-726.
45. Uchida M, Kim H-M, Kokubo T, Nawa M, Asano T, Tanaka K, & Nakamura T (2002) Apatite-forming ability of a zirconia/alumina nano-composite induced by chemical treatment *Journal of Biomedical Materials Research* **60(2)** 277-282.
46. Blatz MB, Sadan A, Arch GH Jr, & Lang BR (2003) In vitro evaluation of long-term bonding of Procera AllCeram alumina restorations with a modified resin luting agent *Journal of Prosthetic Dentistry* **89(4)** 381-387.
47. Blatz MB, Sadan A, Martin J, & Lang B (2004) In vitro evaluation of shear bond strengths of resin to densely-sintered high-purity zirconium-oxide ceramic after long-term storage and thermal cycling *Journal of Prosthetic Dentistry* **91(4)** 356-362.
48. Kitayama S, Nikaido T, Maruoka R, Zhu L, Ikeda M, Watanabe A, Foxton RM, Miura H, & Tagami J (2009) Effect of an internal coating technique on tensile bond strengths of resin cements to zirconia ceramics *Dental Materials Journal* **28(4)** 446-453.
49. Derand P, & Derand T (2000) Bond strength of luting cements to zirconium oxide ceramics *Internaltional Journal of Prosthodontics* **13(2)** 131-135.
50. Ozcan M, & Vallittu PK (2003) Effect of surface conditioning methods on the bond strength of luting cement to ceramics *Dental Materials* **19(8)** 725-731.

51. Blatz MB, Sadan A, & Kern M (2003) Resin-ceramic bonding: A review of the literature *Journal of Prosthetic Dentistry* **89(3)** 268-274.

52. Kern M, Barloi A, & Yang B (2009) Surface conditioning influences zirconia ceramic bonding *Journal of Dental Research* **88(9)** 817-822.

53. Lehmann F, & Kern M (2009) Durability of resin bonding to zirconia ceramic using different primers *Journal of Adhesive Dentistry* **11(6)** 479-483.

54. Thompson JY, Stoner BR, Piascik JR, & Smith R (2011) Adhesion/cementation to zirconia and other non-silicate ceramics: Where are we now? *Dental Materials* **27(1)** 71-82.

55. Bottino MA, Valandro LF, Scotti R, & Buso L (2005) Effect of surface treatments on the resin bond to zirconium-based ceramic *International Journal of Prosthodontics* **18(1)** 60-65.

56. Attia A, Lehmann F, & Kern M (2011) Influence of surface conditioning and cleaning methods on resin bonding to zirconia ceramic *Dental Materials* **27(3)** 207-213.

57. Kern M, & Wegner SM (1998) Bonding to zirconia ceramic: Adhesion methods and their durability *Dental Materials* **14(1)** 64-71.

©Operative Dentistry, 2012, **37-6,** 634-640

Ceramic Primer Heat-Treatment Effect on Resin Cement/Y-TZP Bond Strength

LH Silva • AKF Costa • JRC Queiroz
MA Bottino • LF Valandro

Clinical Relevance Statement

The adhesion of yttrium-stabilized tetragonal zirconia polycrystal ceramics to resin cements is unstable. Even if heat treatment of the metal/zirconia primer improves early bond strength, it is not effective for bond improvement under aging and therefore should not be recommended.

SUMMARY

The purpose of this study was to evaluate the effect of different heat-treatment strategies for a ceramic primer on the shear bond strength of a 10-methacryloyloxydecyl-dihydrogen-phosphate (MDP)-based resin cement to a yttrium-stabilized tetragonal zirconia polycrystal (Y-TZP) ceramic. Specimens measuring 4.5 × 3.5 × 4.5 mm^3 were produced from Y-TZP presintered cubes and embedded in polymethyl methacrylate (PMMA). Following finishing, the specimens were cleaned using an ultrasound device and distilled water and randomly divided into 10 experimental groups (n=14) according to the heat treatment of the ceramic primer and aging condition. The strategies used for the experimental groups were: GC (control), without primer; G20, primer application at ambient temperature (20°C); G45, primer application + heat treatment at 45°C; G79, primer application + heat treatment at 79°C; and G100, primer application + heat treatment at 100°C. The specimens from the aging groups were submitted to thermal cycling (6000 cycles, 5°C/55°C, 30 seconds per bath) after 24 hours. A cylinder of MDP-based resin cement (2.4 mm in diameter) was con-

Lucas Hian da Silva, DDS, MS, PhD student, Biomaterials and Oral Biology, University of São Paulo (USP), São Paulo, Brazil

Anna Karina Figueiredo Costa, DDS, MS, PhD student, Dental Materials and Prosthodontics, São Paulo State University(UNESP), São José dos Campos, Brazil

Jose Renato Cavalcanti de Queiroz, DDS, MS, PhD, postdoc student, Physics, Aeronautical Institute of Technology (ITA), São José dos Campos, Brazil

Marco Antonio Bottino, DDS, PhD, professor, Dental Materials and Prosthodontics, São Paulo State University (UNESP), São José dos Campos, Brazil

*Luiz Felipe Valandro, DDS, PhD, associate professor, Restorative Dentistry (Prosthodontics), Federal University of Santa Maria, Santa Maria, Brazil

*Corresponding author: R Floriano Peixoto 1184, Santa Maria, RS 97015-372, Brazil; e-mail: lfvalandro@hotmail.com

DOI: 10.2341/11-374-L

structed on the ceramic surface of the specimens of each experimental group and stored for 24 hours at 37°C. The specimens were submitted to a shear bond strength test (n=14). Thermal gravimetric analysis was performed on the ceramic primer. The data obtained were statistically analyzed by two-way analysis of variance and the Tukey test (α=0.05). The experimental group G79 without aging (7.23 ± 2.87 MPa) presented a significantly higher mean than the other experimental groups without aging (GC: 2.81 ± 1.5 MPa; G20: 3.38 ± 2.21 MPa; G100: 3.96 ± 1.57 MPa), showing no difference from G45 only (G45: 6 ± 3.63 MPa). All specimens of the aging groups debonded during thermocycling and were considered to present zero bond strength for the statistical analyses. In conclusion, heat treatment of the metal/zirconia primer improved bond strength under the initial condition but did not promote stable bonding under the aging condition.

INTRODUCTION

Reliable adhesion of resin cements to yttrium-stabilized tetragonal zirconia polycrystal (Y-TZP)-based ceramics would improve marginal adaptation, prevent microleakage, and increase retention in situations where it does not exist.[1] However, adhesion between Y-TZP ceramics and resin cements is still a challenge because this ceramic substrate is highly crystalline and dense and consequently is characterized as a nonetchable material.[1,2]

Studies have shown that modifications on the Y-TZP ceramic surface using airborne particle-abrasion approaches, such as the tribochemical silica coating method followed by silanization[1,3] or sandblasted with aluminum oxide particles followed by application of ceramic primers with a chemical functional group increase the bond strength with 10-methacryloyloxydecyl-dihydrogen-phosphate (MDP)-based resin cement. The metal/zirconia primer contains an adhesive phosphate monomer that consists of a bifunctional molecule with 1) a polymerizable organic chain that reacts with the restorative material, 2) an acidic group that reacts with oxides present on the Y-TZP surface, and 3) a spacer group that affects the hydrophilicity, flexibility, and wettability of this molecule.[4,5]

Studies suggest that heat treatment of silanes can improve both their resistance to hydrolysis and the bond strength of glass ceramics to resin cements.[6–8] This treatment provides energy to the system, promoting the evaporation of the solvent and by-products formed in the chemical reactions of this layer. Furthermore, heat could enhance the polymerization of the molecule's organic portion.[9] Due to the similar molecular structure (bifunctional molecules), these mechanisms could also occur in the ceramic primer; consequently, heat could optimize the resin bond strength to Y-TZP ceramics.

Thus, the purpose of this study was to evaluate the effect of different heat-treatment strategies for a ceramic primer on the shear bond strength (SBS) of a MDP-based resin cement to a Y-TZP ceramic. The hypothesis was that the heat treatment of the ceramic primer would improve the resin bond strength.

MATERIALS AND METHODS

Y-TZP Specimen Production

The brand name, manufacturer, and chemical composition of the materials used in this study are listed in Table 1.

Sintered, block-shaped Y-TZP ceramics (Vita Zanhfabrik, Bad Säckingen, Germany) measuring $4.5 \times 3.5 \times 4.5$ mm^3 were embedded in polymethyl methacrylate (PMMA) (Classico, São Paulo, Brazil), maintaining one side of the block free for resin-cement application. They were wet ground finished with 200- to 1500-grit sandpapers (Politriz PSK-2V, ERIOS Equipamentos Técnicos e Científicos Ltda, São Paulo, Brazil) and then ultrasonically cleaned in distilled water for 10 minutes (Vitasonic II, Vita Zanhfabrik).

Subsequently, the blocks were randomly divided into 10 groups according to the surface conditioning method applied and the aging condition (n=14). The experimental groups and treatment strategies are schematically presented in Table 2.

Surface Conditioning Methods

All the testing groups, with the exception of GCd and GCa (control groups, no surface treatment), received an application of metal/zirconia primer (Ivoclar Vivadent Ltda, São Paulo, Brazil) using a microbrush rubbed upon the zirconia surface for 20 seconds. The primer was left for 180 seconds, in accordance with the manufacturer's recommendations, at room temperature and 50% relative humidity, to allow the chemical reactions of the primer and Y-TZP surface to occur. Then, the excess primer was removed by air spray free from oil contamination.

A heat treatment in a ceramic oven (EDG 1800, EDG Equipamentos, São Paulo, Brazil) was per-

Table 1: *Materials Used in the Study*

Brand	Type and Material	Composition	Manufacturer
InCeram 2000 YZ	Yttria stabilized tetragonal zirconia	Zircon oxides, yttrium oxides	Vita Zanhfabrik, Germany
Metal/zirconia primer	Ceramic primer	Tertiary butyl alcohol, methyl isobutyl ketone, phosphonic acid acrylate, benzoylperoxide	Ivoclar Vivadent, Schaan, Leichtenstein
Panavia F (paste A)	Resin cement	10-methacryloyloxydecyl dihydrogen phosphate, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated silica filler, silanated colloidal silica, dl-camphorquinone, catalysts, initiators	Kuraray Medical Inc, Kurashiki, Okayama, Japan
Panavia F (paste B)	Resin cement	Hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler catalysts, accelerators, pigments	Kuraray Medical Inc, Kurashiki, Okayama, Japan

formed for 60 seconds in the G45, G79, and G100 testing groups, in accordance with the temperature previously described for each group (Table 2).

Bonding Procedure

A dual-cure resin cement (Panavia F 2.0, Kuraray Medical Inc, Osaka, Japan) was mixed and applied in a standardized cylindrical plastic mold (2.4 mm diameter, height ~2 mm) and placed upon the zirconia surface of the specimens. The cement was mixed in accordance with the manufacturer's instructions, inserted into the plastic mold using a plastic hand instrument, and light activated (Dentsply SmartLite PS, Dentsply International, York, PA, USA) from above for 40 seconds. Removal of the plastic mold was performed gently by cutting the side with a no. 15 scalpel blade. The specimens from the heat-treated groups (Table 2) were stored for 24 hours at room temperature to guarantee the polymerization of the resin cement and then submitted to the SBS test. The specimens from the aging groups (Table 2) were submitted to the thermal cycling (6,000 cycles, 5°C/55°C, 30 seconds per bath) after 24 hours.

SBS Test and Failure Analysis

The specimens were mounted in the jig of a universal testing machine (EMIC, São José dos Pinhais, Brazil), and a knife-edge shearing rod running at a crosshead speed of 1.0 mm/min was used to load the adhesive interface until failure occurred. The maximum force to produce failure was recorded (N) using a corresponding software. Then the bond strength (MPa) was calculated (force in N / adhered area in mm^2).

Following the shear bond test, the fractured specimens were analyzed under an optical microscope (Mitutoyo FM, Mitutoyo, São Paulo, Brazil) at

Table 2: *Testing Groups* (n=*14*)

Groups	Heat-treatment Strategies of the Metal/Zirconia Primer	Condition
GCd	None (Control)	Dry[a]
G20d	Room temperature 20°C	
G45d	Thermal treatment at 45°C	
G79d	Thermal treatment at 79°C	
G100d	Thermal treatment at 100°C	
GCa	None (control)	Aging[b]
G20a	Room temperature 20°C	
G45a	Thermal treatment at 45°C	
G79a	Thermal treatment at 79°C	
G100a	Thermal treatment at 100°C	

[a] *The shear bond test was performed 24 hours after cement application.*
[b] *The shear test was performed after the aging regime.*

Table 3: *Results for the SBS by the Tukey Test*

Condition	Groups (n=14)	Shear Bond Strength (MPa)	Homogeneous Grouping[a]
Dry	GCd	2.81 ± 1.5	C
	G20d	3.38 ± 2.21	C
	G45d	6.00 ± 3.63	AB
	G79d	7.23 ± 2.87	A
	G100d	3.96 ± 1.57	BC
Aging	GCa	0.0	D
	G20a	0.0	D
	G45a	0.0	D
	G79a	0.0	D
	G100a	0.0	D

[a] *Different letters in same column show statistical differences with a significance level of 5%.*

20× magnification and by scanning electron microscopy (JEOL JSM-7401F, JEOL, Tokyo, Japan) using the secondary electron (SE) mode (70×) to inspect the failure type. The failure types were classified according to the following criteria: (A) along the ceramic/cement adhesive interphase; (B) cohesive in the ceramic; (C) cohesive in the resin cement; and (D) mixed (A+C).

Bond strength data were analyzed by two-way analysis of variance (ANOVA) and the Tukey test (α=0.05).

Thermal Analyses

A metal/zirconia primer solution was positioned in a platinum sample holder, its mass considered to be 15 mg. Thermal gravimetric and differential scanning calorimetry analyses (TG and DSC, respectively) were performed, which initiated at room temperature and increased to 200°C with a heat rate of 10°C/min at 50 mL/min under nitrogen flow.

Thermal analysis is used as a complementary technique in the identification of weight variation with heat treatment (TG) and to measure the enthalpy of the system and the heat capacity of reactions (DSC) when the temperature is increased.

Table 4: *Percentage of Fracture Types for the Experimental Groups (Dry Condition)*

Groups	Type of Failure			
	A	B	C	D
GC	100	0	0	0
G20	100	0	0	0
G45	71.44	0	0	28.57
G79	71.44	0	14.28	14.28
G100	78.57	0	0	21.42

RESULTS

All the specimens in the aged groups debonded during thermocycling and were considered to present zero bond strength for the statistical analyses. All the failures of these prematurely debonded specimens presented a type (A) adhesive failure of the Y-TZP ceramic and cement resin.

Two-way ANOVA revealed that the SBS values were significantly affected by surface treatment (p=0.000) and the interaction with aging (p=0.000). The results of the Tukey multiple comparison test revealed that G79d (7.23 ± 2.87 MPa) presented higher values compared with negative (GCd: 2.81 ± 1.5 MPa) and positive (G20d: 3.38 ± 2.21 MPa) control groups and G100d (3.96 ± 1.57 MPa). G45d (6 ± 3.63 MPa) presented statistically similar results compared with G79d (Table 3).

The fracture analysis of the specimens revealed different fracture patterns: adhesive failure along the interfacial region between the luting agent and the Y-TZP ceramic surface, mixed failure (cohesive fracture of the luting agent combined with adhesive failure), and cohesive in the luting agent (Table 4). The typical mixed fracture pattern is presented in Figure 1.

The TG and DSC analyses (Figure 2) showed mass loss accompanied by an endothermic reaction with a peak at 57°C and a reverse exothermic reaction at 79°C that peaked at 142°C, followed by a slightly downfall, then remained constant from 162°C.

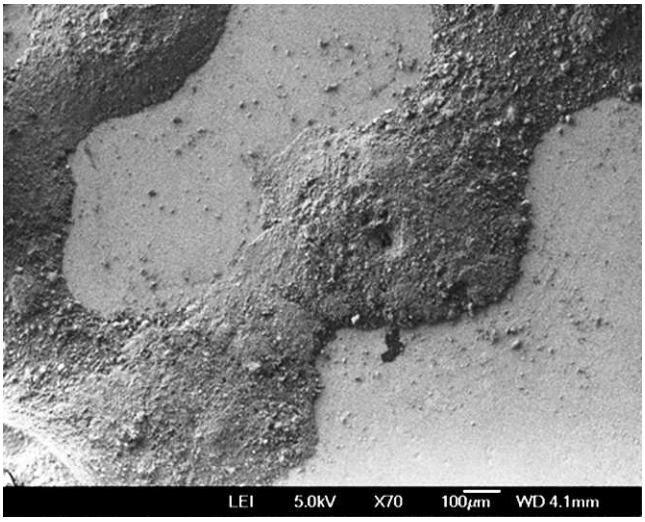


Figure 1. *Representative SEM micrograph of a mixed fracture pattern specimen.*

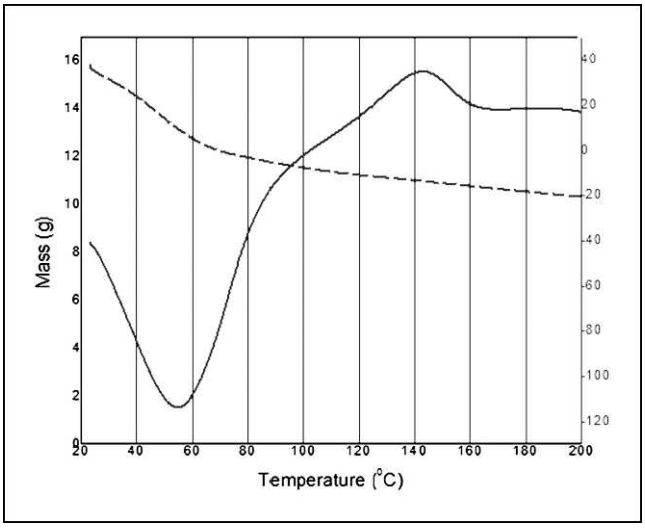


Figure 2. *Thermal behavior of the metal/zirconia primer due to temperature increase: TG (dashed line) and DSC (continuous line).*

DISCUSSION

It is known that the cohesive resistance of Y-TZP ceramics surpasses the adhesive resistance between them and resin cements, decreasing the chances of ceramic failure during the test. The choice of the SBS test was made considering this fact and to simplify comparison of the results with other studies reported in the literature that used the same methodology.[1,5,10,11,12]

Many studies regarding adhesion to Y-TZP ceramic have been conducted due to the fact that the bond between this material and resin cement is not stable.[1] Its nonetchable characteristic prevents the formation of an adhesive interface capable of resisting hydrolysis, leading to degradation and bonding failure.[13]

The heat treatment for bonding agents indicated for etchable ceramics improves the bond strength resistance with resin cements.[1,7] This positive effect could have been caused by the increase in siloxane links in the bonding agent and the solvent evaporation present in the mixture.[7,8] Solvent molecules stored in the adhesive interface could influence the degradation process, accelerating hydrolysis.[14]

The current study applied heat treatment to the metal/zirconia primer used for dentistry and indicated the positive influence of this treatment on the SBS in the dry condition. The primer was subjected to this process up to 200°C, as demonstrated in the TG analysis (Figure 2). It displayed continuous mass loss due to the increase in temperature, suggesting a continuous chemical reaction. The objective of the DSC analysis in this study was to detect the initial temperature of the thermal processes responsible for mass loss and its qualitative characterization, endothermic or exothermic.[15] The modifications that occurred in the material are linked to the chemical processes and transformations caused by the absorption and release of heat, which are clearly present in Figure 2. In the first phase, the primer absorbs heat and results in the loss of volatile substances (solvent and water produced during the primer reaction with the ceramic surface) up to 60°C. Beyond this temperature, mass loss decreased and an inversion of the curve began at 80°C, indicating the onset of exothermic reactions in the primer, with a peak at 140°C caused by the polymerization and posterior chemical degradation of the material, starting at 162°C. Polymerization began at 80°C, as verified by the DSC, negatively influencing the bond strength results for the G100d group.

One important fact is that the chemical composition of the primer used in this study is 70% alcohol (solvent) and its ebullition point is close to 80°C. Within this temperature range, alcohol molecules and by-products resulting from the hydrolysis of phosphonic acid stored in the polymeric network are probably eliminated, according to the results obtained for bond strength, which were greater for the G79d group.

Other studies suggest that silane copolymerization could retard the diffusion of water molecules, preventing the impact of hydrolysis on the adhesive interface.[16] Nevertheless, even with its positive influence on the results, the heat treatment promoted values that were too low to consider it an efficient

alternative for resin bonding improvement for non-etchable Y-TZP ceramics. Although this consideration is related only to the metal/zirconia primer, the aging groups confirm that heat treatment alone was insufficient to promote stable bonding between resin cements and Y-TZP ceramics when only chemical adhesion is required. A consideration must be made regarding the heat treatment time, which was standardized to the time recommended by the manufacturer when the primer is applied under room temperature. However, extending the heat-treatment time and also changing the temperature of ceramic surface could improve the bond strength.

In agreement with the current results, previous research showed that the use of primer solution before resin cement application improved initial bond strength to zirconia ceramics[17]; however, no chemical adhesion was observed between Panavia (Kuraray Medical) and a flat Y-TZP ceramic when this interface was submitted to aging.[18,19]

Several techniques to improve this bond resistance have been reported in the literature, and the use of advanced technologies and chemical modification of the ceramic surface have demonstrated some success at improving the bond strength. The use of film deposition,[20,21,22] selective infiltration etching as a surface pretreatment, nanostructural alumina coating of the Y-TZP surface, or the addition of glass ceramics to the internal region of the zirconia-based ceramics in order to improve bond resistance and permit the use of conventional silane treatment have all been reported.[23,24,25]

Other studies have been trying to find alternative adhesion approaches that are functional in the working environment. Further studies regarding bonding agents have also been conducted.[17,23] The efficiency of this primer is associated with the increased roughness of the surface, consequently promoting better micromechanical retention, improving surface free energy, and increasing the contact area between the ceramic and the bonding agent.[19] That there are so many studies attempting to improve the bond resistance of this adhesive interface is due to the fact that no consensus exists regarding the best treatment to be applied at the Y-TZP adhesive surface. A surface treatment that permits an adhesive cementation of restorations made of Y-TZP frameworks would maximize their clinical application and contribute to preventing marginal infiltration and discoloration.[1,5,10,17,18,21,26]

Heat treatment of the ceramic primer associated with air abrasion using alumina particles may produce relevant results, though sandblasting methods seem to promote crack formation, compromising the material's performance in fatigue tests.[5,27] Clinical studies are necessary to validate this surface treatment as a valid alternative.

Therefore, new studies are required to further investigate the importance of copolymerization on bond resistance following interfacial aging, using other ceramic primers and heating ceramic primers associated with different surface treatments. Heat treatment of ceramic primers should be studied separately to find an optimal temperature capable of promoting the optimal results for each bonding agent.

CONCLUSION

Within the limitations of this study, it can be concluded that heat treatment of the metal/zirconia primer improved the early bond strength but did not promote stable bonding under the aging condition. The aging condition is fundamental for assessing the real capacity for bond improvement of any pretreatment of the Y-TZP surface.

Conflict of Interest

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

(Accepted 5 December 2011)

REFERENCES

1. Ozcan M, Nijhuis H, & Valandro LF (2008) Effect of various surface conditioning methods on the adhesion of dual-cure resin cement with MDP functional monomer to zirconia after thermal aging *Dental Materials Journal* **27(1)** 99-104.
2. Blatz MB, Sadan A, & Kern M (2003) Resin-ceramic bonding: A review of the literature *Journal of Prosthetic Dentistry* **89(3)** 268-274.
3. Amaral R, Ozcan M, Valandro LF, Balducci I, & Bottino MA (2008) Effect of conditioning methods on the microtensile bond strength of phosphate monomer-based cement on zirconia ceramic in dry and aged conditions *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **85(1)** 1-9.
4. Moszner N, Zeuner F, Pfeiffer S, Schurte I, Rheinberger V, & Drache M (2001) Monomers for adhesive polymers, 3a synthesis, radical polymerization and adhesive properties of hydrolytically stable phosphonic acid monomers *Macromolecular Materials and Engineering* **286(4)** 225-231.
5. Nothdurft FP, Motter PJ, & Pospiech PR (2009) Effect of surface treatment on the initial bond strength of different

luting cements to zirconium oxide ceramic *Clinical Oral Investigations* **13(2)** 229-235.

6. Hooshmand T, van Noort R, & Keshvad A (2002) Bond durability of the resin-bonded and silane treated ceramic surface *Dental Materials* **18(2)** 179-188.
7. Hooshmand T, van Noort R, & Keshvad A (2004) Storage effect of a pre-activated silane on the resin to ceramic bond *Dental Materials* **20(7)** 635-642.
8. Queiroz JRC, Benetti P, Özcan M, Oliveira LFC, Della Bona A, Takahashi FE, & Bottino MA (2011) Surface characterization of feldspathic ceramic using ATR FT-IR and ellipsometry after various silanization protocols *Dental Materials* **28(2)** 189-196.
9. Ishida H, & Koening JL (1980) Effect of hydrolysis and drying on the siloxane bonds of a silane coupling agent deposited on E-glass fibers *Journal of Polymer Science Part B: Polymer Physics* **18** 233-237.
10. Phark JH, Duarte S Jr, Blatz M, & Sadan A (2009) An *in vitro* evaluation of the long-term resin bond to a new densely sintered high-purity zirconium-oxide ceramic surface *Journal of Prosthetic Dentistry* **101(1)** 29-38.
11. Kumbuloglua O, Lassila LVJ, User A, & Vallittu PK (2006) Bonding of resin composite luting cements to zirconium oxide by two air-particle abrasion methods *Operative Dentistry* **31(2)** 248-255.
12. Blatz MB, Sadan A, Martin J, & Lang B (2004) *In vitro* evaluation of shear bond strengths of resin to densely-sintered high-purity zirconium-oxide ceramic after long-term storage and thermal cycling *Journal of Prosthetic Dentistry* **91(4)** 356-362.
13. Valandro LF, Ozcan M, Amaral R, Leite FP, & Bottino MA (2007) Microtensile bond strength of a resin cement to silica-coated and silanized In-Ceram zirconia before and after aging *International Journal of Prosthodontics* **20(1)** 70-72.
14. El-Nahhal IM, & El-Ashgar NM (2007) A review on polysiloxane-immobilized ligand systems: Synthesis, characterization and applications *Journal of Organometallic Chemistry* **692(14)** 2861-2886.
15. Becker CR, Currano LJ, Churaman WA, & Stoldt CR (2010) Thermal analysis of the exothermic reaction between galvanic porous silicon and sodium perchlorate *ACS Applied Materials & Interfaces* **2(11)** 2998-3003.
16. Abel M-L, Allington RD, Digby RP, Porritt N, Shaw SJ, & Watts JF (2006) Understanding the relationship between silane application conditions, bond durability and locus of failure *International Journal of Adhesion & Adhesives* **26(1–2)** 2-15.
17. Kitayama S, Nikaido T, Takahashi R, Zhu L, Ikeda M, Foxton RM, Sadr A, & Tagami J (2010) Effect of primer treatment on bonding of resin cements to zirconia ceramic *Dental Materials* **26(5)** 426-432.
18. Özcan M, Kerkdijk S, & Valandro LF (2008) Comparison of resin cement adhesion to Y-TZP ceramic following manufacturers' instructions of the cements only *Clinical Oral Investigations* **12(3)** 279-282.
19. Wolfart M, Lehmann F, Wolfart S, & Kern M (2007) Durability of the resin bond strength to zirconia ceramic after using different surface conditioning methods *Dental Materials* **23(1)** 45-50.
20. Derand T, Molin M, & Kvam K (2005) Bond strength of composite luting cement to zirconia ceramic surfaces *Dental Materials* **21(12)** 1158-1162.
21. Piascik JR, Swift EJ, Thompson JY, Grego S, & Stoner BR (2009) Surface modification for enhanced silanation of zirconia ceramics *Dental Materials* **25(9)** 1116-1121.
22. Queiroz JRC, Duarte DA, Souza ROA, Fissmer SF, Massi M, & Bottino MA (2011) Deposition of SiO_x thin films by reactive magnetron sputtering: Influence of plasma parameters on the adhesion properties between Y-TZP and resin cement for application in dental prosthesis *Materials Research* **14(2)** 212-216.
23. Aboushelib MN, Mirmohamadi H, Matinlinna JP, Kukk E, Ounsi HF, & Salameh Z (2009) Innovations in bonding to zirconia-based materials. Part II: Focusing on chemical interactions *Dental Materials* **25(8)** 989–993.
24. Jevnikar P, Krnel K, Kocjan A, Funduk N, & Kosmac T (2010) The effect of nano-structured alumina coating on resin-bond strength to zirconia ceramics *Dental Materials* **26(7)** 688-696.
25. Kitayama SN, T, Maruka R, Zhu L, Ikeda M, Watanabe A, Foxton R, Miura H, & Tagami J (2009) Effect of an internal coating technique on tensile bond strengths of resin cements to zirconia ceramics *Dental Materials Journal* **28(4)** 446-453.
26. Ohlmann B, Rammelsberg P, Schmitter M, Schwarz S, & Gabbert O (2008) All-ceramic inlay-retained fixed partial dentures: Preliminary results from a clinical study *Journal of Dentistry* **36(9)** 692-696.
27. Zhang Y, Lawn BR, Malament KA, Van Thompson P, & Rekow ED (2006) Damage accumulation and fatigue life of particle-abraded ceramics *International Journal of Prosthodontics* **19(5)** 442-448.

©Operative Dentistry, 2012, **37-6,** 641-649

In Vitro Marginal Fit of Three All-Ceramic Crown Systems Before and After Cementation

GA Borges • JS Faria • P Agarwal
AM Spohr • L Correr-Sobrinho • BAS Miranzi

Clinical Relevance

The results of this study suggest that the evaluated resin-modified glass ionomer cement and resin cement will increase the marginal discrepancy after cementation of the evaluated ceramics.

SUMMARY

Statement of the Problem: **Full-coverage all-ceramic restorations are widely used. The impact of various classifications of luting agent on marginal discrepancies is not well understood.**

Purpose: The purpose of this study was to evaluate the cervical fit of all ceramic crowns (IPS e.maxPress, Cergogold, and In Ceram) on bovine teeth with two luting agents before and after cementation.

Materials and Methods: Ninety bovine incisors were embedded in resin. The coronal portions of the teeth were prepared to receive full-coverage crowns. Thirty crowns of 7.0 ± 0.5 mm height, 8.0 mm cervical diameter, and 4.2 mm incisal diameter were fabricated for each ceramic system. The crowns were seated on the teeth, and the marginal discrepancy was measured using a measuring microscope. Then, 15 crowns of each ceramic system were luted on the teeth with resin cement (Variolink II) or resin-modified glass ionomer cement (Rely X luting), and the marginal discrepancy was measured. The results were submitted to analysis of variance, t test and Tukey's test ($p<0.05$).

Results: The three ceramic systems showed cervical fits after cementation statistically

*Gilberto Antonio Borges, PhD, University of Uberaba, Dental Materials and Restorative Dentistry, Minas Gerais, Brazil

Jordana S. Faria, DDS, master student, Uberaba University, Dental Materials, Dental Materials and Restorative Dentistry, Uberaba University, Minas Gerais, Brazil

Parul Agarwal, BDS, DDS, MSD, assistant professor of prosthodontics, Department of Restorative Dentistry, Indiana University, School of Dentistry, Indianapolis, IN, USA

Ana Maria Spohr, PhD, professor, Pontifical Catholic University of Rio Grande do Sul, Dental Materials, Porto Alegre, Brazil

Lourenço Correr-Sobrinho, PhD, UNICAMP, Dental Materials Division, São Paulo, Brazil

Benito Andre Silveira Miranzi, PhD, University of Uberaba, Minas Gerais, Brazil

*Corresponding author: University of Uberaba, Dental Materials and Restorative Dentistry, Nene Sabino Avenue, 1801, Uberaba, Minas Gerais 38055500, Brazil; e-mail: gilberto.borges@uniube.br

DOI: 10-2341/11-012-L

inferior to cervical fits before cementation for the two cements. The IPS e.maxPress showed values for cervical fit statistically superior to Cergogold before cementation. No statistically significant difference was found between IPS e.maxPress and In Ceram and In Ceram and Cergogold. After cementation, no statistically significant difference was found for the three ceramics systems when luted with resin or resin-modified glass ionomer luting agents.

Conclusions: Within the limitations of this study, it can be concluded that both cements studied increase the marginal discrepancy between the crown and the preparation for the three ceramic systems evaluated.

INTRODUCTION

Dental ceramics have increasingly become the best choice for achieving natural looking restorations and are appropriate materials to mimic destroyed or missing dental structures.[1,2] These materials have desirable characteristics such as chemical stability, biocompatibility, high compressive strength, and a coefficient of thermal expansion similar to that of tooth structure.[3–5] Moreover, recent progress in material technology and processing of ceramic restorations has expanded their indication for use due to more reliable results.[6] Because of the fact that ceramics are indirect restorations and have to be cemented, there will be always be a space between the restoration and preparation. If this space is large, more luting material is exposed to the oral environment. Bacterial plaque can accumulate in this area and can result in gingival inflammation, caries, and pulp lesions, resulting in restoration failure.[7,8] In addition, a large gap can create stress concentrations, which could reduce the final strength of the restoration.[9]

Different types of cement have been used, and there have been controversial opinions about which kind would be more appropriate.[6,7] When considering all-ceramic restorations, it has been reported that fracture strength can be improved by using resin cement. It has been argued that this kind of cement should be the preference when cementing all-ceramic restorations.[10,11] The development of resin-modified glass ionomer cement was intended to improve some physical properties that were present in the conventional glass ionomer cements with better esthetics, working time, and adhesion.[12,13] Furthermore, it has been shown that resin-modified glass ionomer cement does not decrease the final strength of all ceramic crowns.[14]

Marginal discrepancies have been evaluated extensively; however, relatively small sample sizes and low numbers of measurements per specimen have limited statistical analysis.[15] As few as four measurements per specimen have been reported, even though Groten et al[16] stated that at least 50 measurements are required to achieve relevant information. A variety of methodologies have reported marginal discrepancies between 19 and 160 μm.[14–24] It has also been shown that discrepancies between the master die and inner surface of a crown will increase the gap. Steel dies or resin dies have been employed to measure the marginal accuracy of indirect restorations.[25–27] Although the use of these materials results in more accurate standardization of the abutment preparation, they do not supply the specific character of dental hard tissues.[28–32] In the present study, bovine teeth were used because they are histologically and morphologically similar to human dentin and are easier to obtain.[33]

The difference in marginal gap created using resin cement and resin-modified glass ionomer cement with different ceramic systems has not been thoroughly reported. The aim of this study was to evaluate the cervical fit of all-ceramic crowns (IPS e.maxPress, Cergogold, and In Ceram) on bovine teeth with two cements before and after cementation. The null hypotheses were 1) the type of cement does not affect the marginal adaptation of the three different ceramic systems and 2) there is no difference before and after cementation with respect to the marginal adaptation of the three different ceramic systems.

METHODS AND MATERIALS

All materials studied are listed in Table 1.

Ninety bovine mandibular incisors were collected and stored in a 10% formalin solution.[16] Calculus deposits and soft tissue were removed from the selected teeth with a scaler and cleaned with a bristle brush and nonfluoridated flour of pumice (Zircate Prophy Paste, Dentsply, Milford, DE). Mechanical retention was made in the root of the tooth before embedding with autopolymerizing acrylic resin (Jet, Clássico-Produtos Odontológicos, São Paulo, Brazil) in polyvinyl chloride tubes (Marron, Tigre, Joinvile, Brazil). The teeth were placed upright with the long axes parallel to the height of the tube, and the cemento-enamel junctions (CEJ) were placed 3 mm above the resin. The assembled specimens were attached to a lathe (Nardini-ND 250 BE, São Paulo, Brazil) with a grinding device and preparation accomplished under water spray. The

Table 1: *Materials Used in the Study*[a]

Material	Manufacturer	Type	Composition
IPS e.maxPress	Ivoclar Vivadent, Schaan, Liechtenstein	Lithium disilicate ceramic	SiO_2, Al_2O_3, La_2O_3, MgO, ZnO, K2O, Li_2O, P_2O_5
e.maxCeram	Ivoclar Vivadent	Feldspatic porcelain	SiO_2, K_2O, ZnO, ZrO_2, Li_2O, CaO, Na_2O, Al_2O_3
Cergogold	Degussa Dental, Hanau, Germany	Leucite ceramic	SiO_2, Al_2O_2, K_2O, Na_2O, CaO
Duceragold	Degussa Dental	Feldspatic porcelain	SiO_2, Al_2O_3, K_2O, Na_2O, CaO, BaO, SnO_2, Li_2O, F,Sb_2O_3, CeO2, B_2O_3, TiO_2
InCeram Alumina	Vita Zanfabrik, Seefeld, Germany	Infiltrated Alumina ceramic	Al_2O_3, La_2O_3, SiO_2, CaO, other oxides
VM7	Vita Zanfabrik	Feldspatic porcelain	SiO_2, Al_2O_3, B_2O_3, Na_2O, K_2O, CaO, TiO_2
Variolink II	Ivoclar Vivadent	Dual-polymerizing resin luting agent	HEMA, dimethacrylate, phosphoric acid acrylate, highly-dispersed silicon dioxide, initiators and stabilizers in an alcohol solution; the brush is coated with initiators
Rely X luting	3M ESPE, Seefeld, Germany	Resin-modified glass ionomer cement	A: fluoroaluminosilicate, potassium persulfate, ascorbic acid, opacifying agent B: 30%-40% copolymer of acrylic and itaconic acids, 25%-35% 2-hydroxyethyl-methacrylate, 25%-35% water

[a] *Manufacturer information.*

final dimensions of the preparations were 7.0 ± 0.5 mm in height, 8.0 mm for the cervical diameter, and 4.2 mm for the incisal diameter, which resulted in a peripheral surface convergence of 8°. This degree of convergence was used since it has been shown that retention increases considerably as the taper decreases.[24] A 0.8-mm-deep shoulder finish line with a rounded internal line angle was prepared using a diamond instrument (No. 5850-018; Brasseler USA, Savannah, GA). All sharp angles were rounded, and all cervical margins were located 1.0 ± 0.2 mm above the CEJ (Figure 1). All teeth were measured after the preparation using a precision electronic micrometer (Electronic Micrometer; LS Starrett, Athol, MA) with an accuracy of 0.002 mm.[16]

The 90 prepared teeth were divided into three groups (n=30) as follows: In Ceram Alumina, IPS e.maxPress, and Cergogold. An impression was made for each prepared tooth with a polyvinyl siloxane impression material (Express, 3M ESPE, St Paul, MN) using a custom-made impression tray fabricated with acrylic resin. Then, type IV gypsum (Fuji Rock, GC America, Aslip, IL) was poured to produce dies. The teeth were stored in distilled water at 37°C until the cementation process.

The dies were coated with one layer of die spacer (Spacelaquer Ducera Lay, Degussa Huls, Hanau, Germany) to approximately 1 mm above the finish line. For IPS e.maxPress and Cergogold, the dies were isolated with lubricating oil (Die Lube, Dentaurum J.P. Winkelstroeter KG, Pforzheim, Germany) and 0.7-mm-thick wax patterns were prepared over the master dies using a wax-dipping unit (Hotty, Renfert, Hilzingen, Germany). Following the preparation of the wax patterns, each pattern was sprued and invested in an investing ring. A two-stage burnout sequence was used: temperature increased 5°C/minute to 250°C and held for 30

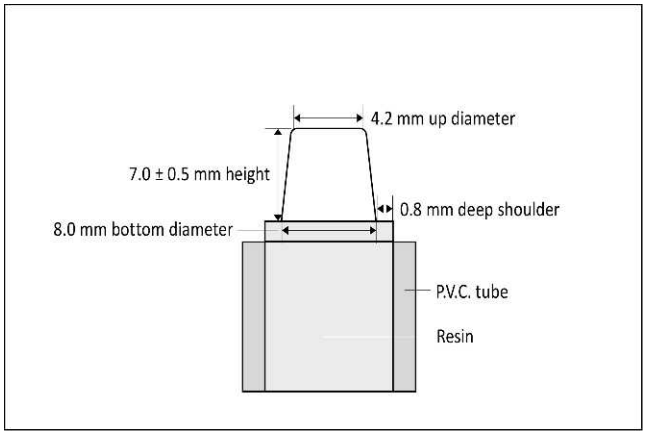


Figure 1. *Schematic diagram of full-coverage preparation.*

minutes before increasing the temperature at 5°C/minute to 850°C and holding for 1 hour. After the preheating stage, the investment cylinders were immediately transferred to the pressing furnace (EP500, Ivoclar AG, Schaan, Liechtenstein). The pressing temperatures for Empress 2 core and Cergogold core ceramics were 920°C and 850°C, respectively. Following the pressing procedure, the investment cylinders were removed from the pressing furnace and cooled for 2 hours in a ventilated room. The cooled specimens were divested by grit blasting with 80-μm glass beads (Williams glass beads, Ivoclar North America, Amherst, NY). Before etching, the sprues were cut away, and excess sprue segments were removed by grinding from the specimen surfaces using water as a coolant. The core specimens were placed in one plastic bottle containing 20 mL of 1% hydrofluoric acid solution (Invex Liquid, Ivoclar AG), and these bottles were placed in an ultrasonic bath. After etching, the specimens were cleaned under running tap water for 10 seconds and then dried thoroughly. These procedures were performed by a certified dental technician.

For the In Ceram alumina, three layers of die spacer (Vita Zanfabrik, Seefeld, Germany) were applied on the stone die surface to approximately 1 mm above the finish line. Impressions were made using a polyvinyl siloxane impression material (Express, 3M ESPE) with a plastic ring. These impressions were poured with In Ceram special plaster using a liquid-to-powder ratio of 0.23 mL/g to make refractory models. In Ceram powder slip was prepared according to the manufacturer's instructions and was applied to the models. A sculpturing device was used to ensure a uniform core thickness.[19] The stabilizer was applied, and the coping was fired on the plaster dies and infiltrated with glass. Excess glass was removed with a diamond bur. These procedures were conducted in an authorized laboratory by a certified technician. The final dimension of IPS e.maxPress and Cergogold copings were 0.7 mm and 0.5 mm for In Ceram.

The veneer porcelains (e.max Ceram , Ivoclar for IPS e.maxPress core; VM7, Vita Zahnfabrik for In Ceram core and Duceragold, Degussa Dental for Cergogold core) were applied to the core materials, which had been placed in a split brass mold to make a complete crown shape with a stratification porcelain thickness measuring 0.1 mm for IPS e.maxPress and Cergogold and 0.3 mm for In Ceram specimens in the cervical region and increasing in thickness in accordance with the taper angle. Following veneer porcelain sintering, the final dimensions of the crowns were 0.8 mm in the cervical region, 1.0 mm in the mid-facial region, and 1.5 mm in the incisal region.[16]

Measurements of Marginal Adaptation Before Cementation

Tooth surfaces were cleaned with a bristle brush and nonfluoridated flour of pumice (Zircate Prophy, Dentsply). Each crown was placed onto its preparation under a constant controlled pressure of 9 kgf for 1 minute using a pneumatic pressure machine (developed in the Dental Materials Laboratory of the University of Campinas School of Dentistry, Piracicaba, Brazil). A metallic device was designed to maintain the assembled tooth/restoration at a reproducible position and allow measurement on a measuring optical microscope (Nikon measurescope UM2, Nikon Co, Tokyo, Japan) at original magnification 50×. The accuracy of the microscope was ±0.5 μm. The marginal discrepancy was evaluated by measuring the gap between the edge of the crown and prepared tooth margin. Sixteen diametrically placed marks were created on the root tooth surface 2 mm below the prepared margin using a round diamond bur (#1011, KGSorensen, São Paulo, Brazil). The distance of the gap was measured at each demarcated area with four different measurements. These measurements were taken from the prepared margin to the edge of the crown (Figures 2 and 3).

Application of Cement

Variolink II (Ivoclar-Vivadent, Schaan, Liechtenstein). Tooth surfaces were cleaned with a bristle brush and nonfluoridated flour of pumice (Zircate Prophy, Dentsply). The dentin was treated for 15

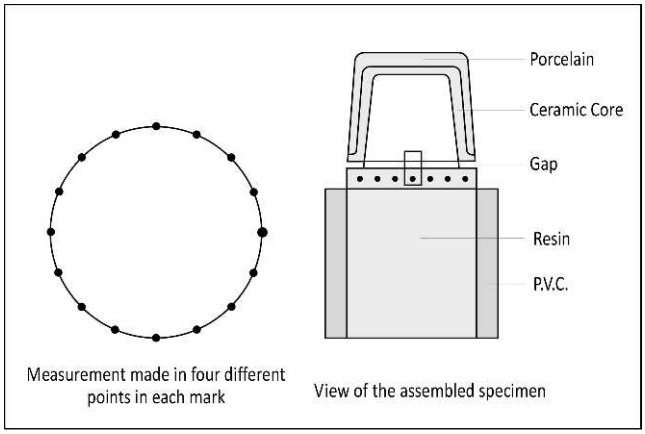


Figure 2. *Points of measurement and view of the assembled specimen.*

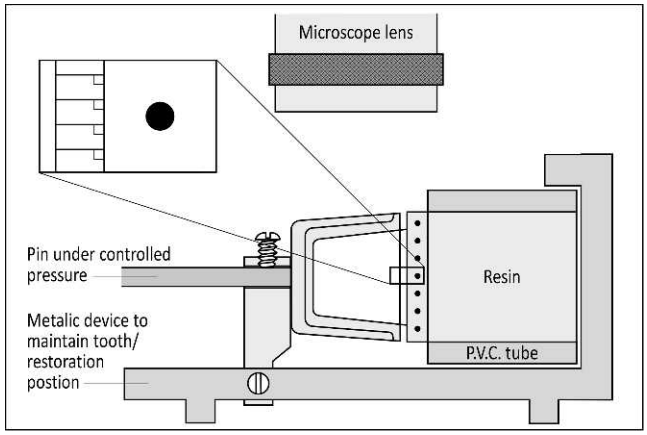


Figure 3. *Assembled specimen under the microscope.*

seconds with 35% phosphoric acid and rinsed for 10 seconds under running tap water. Excess water was removed with a cotton pellet, leaving a moist surface. Two consecutive coats of adhesive were then applied using a saturated microbrush tip. The ceramic surface was etched with 10% hydrofluoric acid (Ácido hidrofluorídrico, Dentsply Brazil, Petropolis, RJ, Brazil) for 1 minute (Cergogold and In Ceram) or for 20 seconds (IPS e.maxPress), followed by rinsing for 1 minute. Samples were then ultrasonically cleaned with distilled water for 10 minutes and dried with oil-free air. The silane agent Monobond S (Ivoclar-Vivadent) was applied, and the surface was dried after 1 minute using compressed air. The resin cement was mixed and applied to the internal ceramic crown surface. A load of 454 gf was applied while the excess cement was removed. The cement was light-cured (XL3000, 3MEspe) for 40 seconds on each side (labial, lingual, medial, and distal) of the crown, resulting in 160 seconds of light polymerization for each crown with 500 mW/cm^2 light intensity. Ten minutes after the start of the mix, specimens were immersed in distilled water at 37°C and stored until testing.

Rely X luting (3M ESPE). The procedures were the same as for the aforementioned resin cement, but the dentin was simply cleaned and did not receive any adhesive application. The specimens were stored in distilled water for 24 hours before making the measurements.

Measurements of Marginal Adaptation After Cementation

The marginal discrepancy was measured as described for marginal adaptation measurment before cementation (Figures 2 and 3). However, the pressure was not applied because the crowns were already cemented. ANOVA one-way, post hoc Tukey and t test were applied (Table 2). The marginal adaptations were determined, and since two different cements were used and the readings were before and after cementation, the data were statistically analyzed using two-way analysis of variance (ANOVA), with the independent variables being the cement and the time of evaluation (before and after cementation) (Tables 3, 4 and 5). Means and standard deviations were calculated for each material and each condition. Individual comparisons between the materials and conditions were made using a Tukey Honestly Significant Difference test to determine significant differences. All statistical testing was performed with α=0.05.

RESULTS

The one-way ANOVA (Table 2) revealed significant differences in the marginal discrepancy values ($p<0.001$). Tables 3, 4, and 5 show the mean marginal discrepancies and standard deviations. Table 3 displays the values of the three ceramic systems before and after cementation with resin-modified glass ionomer cement. It can be seen that before cementation, IPS e.maxPress resulted in a discrepancy significantly higher than Cergogold, and both did not differ from In Ceram ($p<0.05$). The same situation was observed after cementation ($p<0.05$). When comparing the discrepancies within each ceramic before and after cementation, the results after cementation were statistically significantly higher than before cementation ($p<0.05$).

Table 2: *Statistical Analysis*[a]

Causes of Variation	Applied Test	Mean	SD	*F*	*p*
Ceramic before	ANOVA one-way			13.0445	0.0000631
In Ceram		81.43 B	17.27		
e.maxPress		95.65 A	19.54		
Cergogold		71.51 B	18.31		
Ceramic after	ANOVA one-way			9.4975	0.0003803
In Ceram		122.92 A	17.27		
e.maxPress		137.97 A	40.69		
Cergogold		101.95 B	26.43		
Before and after	*t*-test paired				<0.0001
Before		82.87	20.74		
After		121.0	35.09		
Cement	*t*-test not paired				0.8405
Glass ionomer		121.7	33.66		
Resin cement		120.2	36.84		

[a] *Means followed by the same letter indicate no statistical difference.*

Table 4 shows the mean values of discrepancies of the three ceramic systems before and after cementation with resin cement. It can be observed that before cementation, IPS e.maxPress resulted in a discrepancy significantly higher then Cergogold, and both IPS e.maxPress and Cergogold did not differ from In Ceram ($p<0.05$). The same situation was observed after cementation ($p<0.05$). When comparing the discrepancies within each ceramic before and after cementation, the results after cementation showed a statistically significant increase ($p<0.05$). Table 5 compares the mean values of discrepancies between the two cements for the three ceramic systems. It shows that there was no statistically significant difference between the cements for the ceramics evaluated.

DISCUSSION

The null hypotheses that the type of cement does not affect the marginal adaptation of the three different ceramic systems and that the time of evaluation (before and after cementation) does not affect the marginal adaptation of the three different ceramic systems were rejected by the results. The marginal discrepancy of the three ceramic systems evaluated was affected by the cement and by the time of evaluation (Tables 3 and 4). These results seem to correlate well with *in vitro* studies assessing the discrepancies of ceramic restorations.[19,20] Many studies evaluating the marginal discrepancy of all-ceramic crowns have been published;[15,17,19–21] however, no study was found comparing resin-modified glass ionomer cement and resin cement before and after cementation for different ceramic restoration systems. It is well known that

Table 3: *Marginal Discrepancies of the Three Ceramic Systems Before and After Cementation With Resin-Modified Glass Ionomer Cement (Means and Standard Deviations [SD] in μm[a]*

Ceramic Type	Before Cementation			After Cementation		
	No. of Spec	Mean	SD	No. of Spec	Mean	SD
IPS e.maxPress	15	83.13[a] A	25.04	15	137.82[a] B	44.44
In Ceram	15	77.04[ab] A	18.32	15	122.67[ab] B	18.88
Cergogold	15	66.20[b] A	20.19	15	104.64[b] B	25.28

[a] *Means followed by the same superscript letters within each column and capital letters within the row indicate no statistical difference at the 95% confidence level (p>0.05).*

resin cement is the most popular luting agent when using all-ceramic restorations[34]; however, resin-modified glass ionomer cement has been used to cement all-ceramic restorations, and some authors argue that this cement has advantages.[13,14] Even though controversy exists regarding a clinically acceptable marginal adaptation, the study by McLean and von Fraunhofer[18] proposed that a restoration would be successful if marginal gaps of less than 120 μm could be obtained. Before cementation, the marginal discrepancy of the all-ceramic systems evaluated in the current study was within this clinically acceptable standard, and it is in agreement with other studies[19–21] that found marginal discrepancies deemed clinically acceptable. Nevertheless, the current results do not agree with Grey et al[26] that found discrepancies larger than 120 μm for conventional In Ceram crowns. After cementation, Cergogold and In Ceram showed acceptable discrepancy values for both cements evaluated, but IPS e.maxPress resulted in a discrepancy larger than recommended (Tables 3, 4, and 5). Some studies have evaluated the marginal discrepancies without taking the cementation process into consideration.[35–37] Evaluating discrepancies without luting them is not reflective of clinical reality because the cement and the cementation process play a relevant role in the final discrepancy achieved. In the current study, although a space was created to allow the cement to flow into the space between the tooth and internal ceramic surface, the results for the all-ceramic and cement combinations increased the discrepancy after cementation. The convergence angle could influence the final marginal discrepancy acquired since an angle with a higher divergence would permit easier displacement of the cement. In the current study, the 8° taper angle might not have allowed the same flow of the cements as a clinical preparation would, because clinical angles range between 12° and 20°, and increased taper could affect the crown retention.[23,24] However, the current results are in agreement with previous studies that found an increase in the marginal discrepancy after cementation using approximately the same taper-

Table 4: *Marginal Discrepancies of the Three Ceramic Systems Before and After Cementation With Resin Cement (Means and Standard Deviations [SD] in μm)[a]*

Ceramic Type	Before Cementation			After Cementation		
	No. of Spec	Mean	SD	No. of Spec	Mean	SD
IPS e.maxPress	15	101.50[b] A	21.20	15	138.10[b] B	38.13
In Ceram	15	85.83[a] A	15.22	15	123.20[a] B	34.55
Cergogold	15	76.82[a] A	15.06	15	99.26[a] B	28.16

[a] *Means followed by the same superscript lowercase letters within each column and uppercase letters within the row indicate no statistical difference at the 95% confidence level (p>0.05).*

Table 5: *Marginal Discrepancies of the Three Ceramic Systems After Cementation With Resin Cement and Resin-Modified Glass Ionomer Cement (Means and Standard Deviations [SD] in μm)*[a]

Ceramic Type	RMGI			Resin Cement		
	No. of Spec	Mean	SD	No. of Spec	Mean	SD
IPS e.maxPress	15	123.20 A	38.13	15	137.80 A	44.44
In Ceram	15	138.10 A	34.55	15	122.70 A	18.88
Cergogold	15	99.26 A	28.16	15	104.60 A	25.28

[a] *Means followed by the same capital letters within each row indicate no statistical difference at the 95% confidence level (p>0.05).*

ing.[19,20] Further study should be pursued to delineate the optimum convergence angle for adhesively retained all-ceramic restorations. Despite the fact that ceramic restorations have been used extensively, the cementation procedure is one of the most critical parts of the overall process. In the present study, the conditions were well controlled. Even though clinically, the practitioner cares about the final fit of the restoration, it is not possible to control all of the steps as in an experimental study. Since an increase in marginal discrepancy was seen in a well-controlled environment, it would seem important for the clinician to pay close attention to environmental factors that might interfere with the cement line thickness.

CONCLUSION

Within the limitations of this study, it can be concluded that the cementation increases the marginal gap between the crown and the preparation for the three ceramic systems evaluated regardless of the type of cement evaluated.

Acknowledgements

This investigation was supported by the University of Uberaba, Brazil. The authors thank Jeffrey A. Platt for aid in manuscript preparation.

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.

(Accepted 13 January 2012)

REFERENCES

1. Heydecke G, Thomason JM, Lund JP, & Feine JS (2005) The impact of conventional and implant supported prostheses on social and sexual activities in edentulous adults: results from a randomized trial 2 months after treatment *Journal of Dentistry* **33(8)** 649-657.
2. Raptis NV, Michalakis KX, & Hirayama H (2006) Optical behavior of current ceramic systems *International Journal of Periodontics and Restorative Dentistry* **26(1)** 31-41.
3. Mitchell CA, & Orr JF (2005) Engineering properties and performance of dental crowns *Proceedings of the Institution of Mechanical Engineers* **219(4)** 245-255.
4. Chu S, & Ahmad I (2005) A historical perspective of synthetic ceramic and traditional feldspathic porcelain *Practical Procedures and Aesthetic Dentistry* **17(9)** 593-598.
5. Xible AA, de Jesus Tavarez RR, de Araujo Cdos R, & Bonachela WC (2006) Effect of silica coating and silanization on flexural and composite-resin bond strengths of zirconia posts: an in vitro study *Journal of Prosthetic Dentistry* **95(3)** 224-229.
6. Fradeani M, D'Amelio M, Redemagni M, & Corrado M (2005) Five-year follow-up with Procera all-ceramic crowns *Quintessence International* **36(2)** 105-113.
7. Santos GC Jr, Santos MJ, & Rizkalla AS (2009) Adhesive cementation of etchable ceramic esthetic restorations *Journal of Canadian Dental Association* **75(5)** 379-384.
8. Orstavik D, & Orstavik J (1976) In vitro attachment of *Streptococcus sanguis* to dental crown and bridge cements *Journal of Oral Rehabilitation* **3(2)** 139-144.
9. Tuntiprawon M, & Wilson PR (1995) The effect of cement thickness on the fracture strength of all-ceramic crowns *Australian Dentistry Journal* **40(1)** 17-21.
10. Kelly JR, Giordano R, Pober R, & Cima MJ (1990) Fracture surface analysis of dental ceramics: clinically failed restorations *International Journal of Prosthodontics* **3(5)** 430-440.
11. Groten M, & Probster L (1997) The influence of different cementation modes on the fracture resistance of feldspathic ceramic crowns *International Journal of Prosthodontics* **10(2)** 169-177.
12. Kim Y, Hirano S, & Hirasawa T Physical properties of resin-modified glass-ionomers *Dental Materials Journal* **17(1)** 68-76.

13. Chan WD, Yang L, Wan W, & Rizkalla AS (2006) Fluoride release from dental cements and composites: a mechanistic study *Dental Materials* **22(4)** 366-373.
14. Leevailoj C, Platt JA, Cochran MA, & Moore BK (1998) In vitro study of fracture incidence and compressive fracture load of all-ceramic crowns cemented with resin-modified glass ionomer and other luting agents *Journal of Prosthetic Dentistry* **80(6)** 699-707.
15. Yeo IS, Yang JH, & Lee JB (2003) *In vitro* marginal fit of three all-ceramic crown systems *Journal of Prosthetic Dentistry* **90(5)** 459-464.
16. Groten M, Axmann D, Probster L, & Weber H (2000) Determination of the minimum number of marginal gap measurements required for practical in-vitro testing *Journal of Prosthetic Dentistry* **83(1)** 40-49.
17. Kern M, Schaller HG, & Strub JR (1993) Marginal fit of restorations before and after cementation *in vivo International Journal of Prosthodontics* **6(6)** 585-591.
18. McLean JW, & von Fraunhofer JA (1971) The estimation of cement film thickness by an in vivo technique *Brazilian Dentistry Journal* **131(3)** 107-111.
19. Beschnidt SM, & Strub JR (1999) Evaluation of the marginal accuracy of different all-ceramic crown systems after simulation in the artificial mouth *Journal of Oral Rehabilitation* **26(7)** 582-593.
20. Groten M, Girthofer S, & Probster L (1997) Marginal fit consistency of copy-milled all-ceramic crowns during fabrication by light and scanning electron microscopic analysis in vitro *Journal of Oral Rehabilitation* **24(12)** 871-881.
21. Balkaya MC, Cinar A, & Pamuk S (2005) Influence of firing cycles on the margin distortion of 3 all-ceramic crown systems *Journal of Prosthetic Dentistry* **93(4)** 346-355.
22. Leempoel PJB, Lemmens PL, Snoek PA, & van't Hof MA (1987) The convergence angle of tooth preparations for complete crowns *Journal of Prosthetic Dentistry* **58(4)** 414-416.
23. Nordlander J, Weir D, Stoffer W, & Ochi S (1988) The taper of clinical preparations for fixed prosthodontics *Journal of Prosthetic Dentistry* **60(2)** 148-151.
24. Jorgensen KD (1955) The relationship between retention and convergence angle in cemented veneer crowns *Acta Odontologica Scandinavica* **13(1)** 35-40.
25. Bottino MA, & Valandro LF (2007) The influence of cervical finish line, internal relief, and cement type on the cervical adaptation of metal crowns *Quintessence International* **38(7)** 425-432.
26. Grey NJ, Piddock V, & Wilson MA (1993) *In vitro* comparison of conventional crowns and a new all-ceramic system *Journal of Dentistry* **21(1)** 47-51.
27. Lee KB, Park CW, Kim KH, & Kwon TY (2008) Marginal and internal fit of all-ceramic crowns fabricated with two different CAD/CAM systems *Dental Materials Journal* **27(3)** 422-426.
28. Limkangwalmongkol P, Kee E, Chiche GJ, & Blatz MB (2009) Comparison of marginal fit between all-porcelain margin versus alumina-supported margin on Procera Alumina crowns *Journal of Prosthodontics* **18(2)** 162-166.
29. Comlekoglu M, Dundar M, Ozcan M, Gungor M, Gokce B, & Artunc C (2009) Influence of cervical finish line type on the marginal adaptation of zirconia ceramic crowns *Operative Dentistry* **34(5)** 586-592.
30. Krifka S, Anthofer T, Fritzsch M, Hiller KA, Schmalz G, & Federlin M (2009) Ceramic inlays and partial ceramic crowns: influence of remaining cusp wall thickness on the marginal integrity and enamel crack formation in vitro *Operative Dentistry* **34(1)** 32-42.
31. Federlin M, Krifka S, Herpich M, Hiller KA, & Schmalz G (2007) Partial ceramic crowns: influence of ceramic thickness, preparation design and luting material on fracture resistance and marginal integrity *in vitro Operative Dentistry* **32(3)** 251-260.
32. Federlin M, Schmidt S, Hiller KA, Thonemann B, & Schmalz G (2004) Partial ceramic crowns: influence of preparation design and luting material on internal adaptation *Operative Dentistry* **29(5)** 560-570.
33. Nakamichi I, Iwaku M, & Fusayama T (1983) Bovine teeth as possible substitutes in the adhesion test *Journal of Dentistry Research* **62(10)** 1076-1081.
34. Hill EE (2007) Dental cements for definitive luting: a review and practical clinical considerations *Dental Clinic of North America* **51(3)** 643-658.
35. Holden JE, Goldstein GR, Hittelman EL, & Clark EA (2009) Comparison of the marginal fit of pressable ceramic to metal ceramic restorations *Journal of Prosthodontics* **18(8)** 645-648.
36. Al-Rabab'ah MA, Macfarlane TV, & McCord JF (2008) Vertical marginal and internal adaptation of all-ceramic copings made by CAD/CAM technology *European Journal of Prosthodontics Restorative Dentistry* **16(3)** 109-115.
37. Kokubo Y, Tsumita M, Kano T, Sakurai S, & Fukushima S (2011) Clinical marginal and internal gaps of zirconia all-ceramic crowns *Journal of Prosthodontic Research* **55(1)**:40-43.

©Operative Dentistry, 2012, **37-6,** 650-659

The Effects of Scaling and Root Planing on the Marginal Gap and Microleakage of Indirect Composite Crowns Prepared With Different Finish Lines: An *In Vitro* Study

D Angerame • R Sorrentino • D Cettolin
F Zarone

Clinical Relevance Statement

Due to the surface alterations caused by mechanical periodontal maintenance, the 90° shoulder and the chamfer preparation proved to be a viable option for the fabrication of composite crowns, whereas the beveled 90° shoulder and the feather edge should not be recommended. The amorphous debris due to scaling and root planing could increase bacteria accumulation over time; consequently, hygienic maintenance should be stressed in the presence of composite restorations in order to reduce the need for root mechanical instrumentation.

Daniele Angerame, MD, DDS, associate professor, Dept. Medical, Surgical and Health Sciences, University of Trieste, Trieste, Italy

*Roberto Sorrentino, DDS, MSc, PhD, Department of Fixed Prosthodontics, University Federico II, Naples, Italy.

Davis Cettolin, DH, School of Dental Hygiene, University of Trieste, Trieste, Italy

Fernando Zarone, MD, DDS, professor, Department of Fixed Prosthodontics, University Federico II, Naples, Italy

*Corresponding author: Via S Pansini 5, Naples, 80131, Italy; e-mail: errestino@libero.it

DOI: 10.2341/11-347-L

SUMMARY

The present *in vitro* study aimed to assess the effects of root surface mechanical instrumentation on the marginal integrity and adaptation of resin composite crowns. The following null hypotheses were tested: no differences exist between finish line and 1) marginal gap or 2) marginal microleakage before and after manual mechanical periodontal maintenance.

A total of 56 intact human mandibular molars were randomly distributed into four groups and subjected to standardized tooth prepara-

tions for indirect composite crowns with different marginal finish lines (90° shoulder, beveled 90° shoulder, feather edge, chamfer). One-half of the specimens was used as a control and remained untreated, and the remaining half was subjected to root surface procedures simulating five years of semestral mechanical supportive periodontal treatment. The marginal gap and microleakage were evaluated and statistically analyzed.

The specimens used as controls showed lower mean marginal gaps than those subjected to the simulated periodontal treatment, whereas the latter showed lower microleakage than the control crowns. Statistically significant differences were recorded for both the experimental variables.

The root surface procedures resulted in altered surfaces of the composite crowns. The marginal gap increased after the treatment, whereas the marginal microleakage was reduced. The 90° shoulder and the chamfer preparation could be considered a viable option to fabricate composite crowns, but the beveled 90° shoulder and the feather edge should not be recommended.

INTRODUCTION

Metal-ceramic prostheses still represent the gold standard in single crowns due to their optimal mechanical resistance, acceptable esthetics, and well-documented clinical longevity.[1–3] All-ceramic restorations were introduced in clinical practice to improve esthetic performances, eliminating the underlying grayish metal framework.[1–4] Polycrystalline ceramics, just like alumina and zirconia, combine tooth-like esthetic appearance with optimal mechanical properties[1–4] and require computer-aided design/computer-aided manufacturing (CAD-CAM) fabrication.[1–3] Conversely, resin composite full-coverage indirect restorations may reduce manufacturing costs up to 60%.[1] As a consequence, the interest toward composite crowns as a less expensive, promising interim alternative to metal and all-ceramic restorations has increased in the last years.[1,4,5–12]

Composite crowns can be considered as long-term temporary restorations, due to inferior esthetics and wear resistance compared with traditional ceramic prostheses.[1,12,14] Nevertheless, some authors have proposed them as permanent restorations thanks to the introduction of efficient dentinal adhesives, the development of resin composites with higher fracture toughness and wear resistance, and the possibility of CAD-CAM processing.[14–18] Indirect composite restorations are easy to use and less time-consuming compared with traditional procedures.[8,12] Moreover, they require less invasive preparations with minimal depth reductions, preserving more sound tooth structure: According to some authors, this could be paramount to increasing the survival probability of a restoration, particularly after endodontic treatment.[1,4,7,19,20] As a consequence of the absence of metal frameworks, prosthetic finish lines can be placed at the gingival margin, reducing gingival irritation.[21]

Quite scarce data are available regarding the clinical performances of composite crowns.[14] In an *in vitro* study,[22,23] adhesively luted posterior composite crowns showed failure loads higher than 1000 N. Acceptable fracture resistance[4,24–28] and promising results for clinical use after fatigue tests[29,30] were reported in laboratory investigations. Preliminary clinical studies[1,4] reported variable results depending on composite systems, occlusal thickness and type of cement. Probability survival rates of 96% and 88.5% were indicated after 3 and 5 years, respectively.[1,9] Success rates lower than those of all-ceramic restorations were reported after 3 years of function.[12] Estimated overall survival rates of 53% ± 14% at restoration level and 79% ± 11% at tooth level were reported in a controlled clinical trial.[8] As to complications, increased plaque accumulation was noticed, restricting the indication for permanent restorations.[1,5] With the introduction of modern composite materials, it is possible to reduce the above mentioned disadvantages;[4,18,31] nonetheless, further *in vivo* studies will be necessary to evaluate the stability and longevity of composite crowns in the oral environment.[1]

Hand and ultrasonic scaling and root planing of root surfaces are the most commonly used mechanical procedures for supportive periodontal care.[32–36] Such instrumentation may cause minor structural alterations of both root surface and restoration margins; the consequent roughening may result in unacceptable restorative margins associated with increased plaque accumulation and high risk of secondary caries.[21,33,35,36] Consequently, the side effects of mechanical periodontal care procedures should be limited so as not to interfere with marginal adaptation of restorations.[21,33,37] Plastic dental scalers were proposed to avoid scratching restorative materials so as not to affect their surface texture; nevertheless, some authors suggested that they

could quickly become ineffective due to the greater hardness of restorative materials.[33] Sharp metal curettes are still the standard instruments for periodontal maintenance therapy.[38]

High-quality margins and smooth surfaces enhance biocompatibility of restorative materials because plaque retention is minimized and gingival inflammation prevented.[21,33,39] Several studies investigated the textural changes of restorations caused by mechanical periodontal maintenance.[21,33,37–41] Discordant results were reported for composite resin restorations: some authors reported significant altered composite surfaces after hand and ultrasonic scaling,[40] whereas other investigations demonstrated no damaging effects after scaling and jet-polishing procedures.[41] Such controversial results were probably due to the different characteristics of the periodontal treatment (eg, time, pressure, angulation, type of instruments) and techniques of investigation (eg, qualitative, quantitative, type of measurement).[21,32,37–41]

The present *in vitro* study aimed to assess the effects of root surface mechanical instrumentation on the marginal integrity and adaptation of resin composite crowns.

Two different null hypotheses were tested:

1. There is no difference between the finish line of indirect composite crowns and their marginal gap before and after manual mechanical periodontal maintenance.
2. There is no difference between the finish line of indirect composite crowns and their marginal microleakage before and after manual mechanical periodontal maintenance.

METHODS AND MATERIALS

A total of 56 intact human mandibular molars with no obvious pathology or restorations and extracted for periodontal reasons were selected. Dental plaque, calculus, and periodontal debris were removed by means of ultrasonic scaling. The teeth were disinfected by immersion in 5% sodium hypochlorite for five minutes and stored in 0.9% NaCl physiological solution at 37°C.

The teeth were randomly distributed into four groups, each containing 14 specimens. Silicone impressions of the teeth were made before tooth preparation in order to be used as templates to check the removal of dental tissues. Each group was subjected to standardized tooth preparations for indirect composite crowns with different marginal finish lines (Figure 1), as follows:

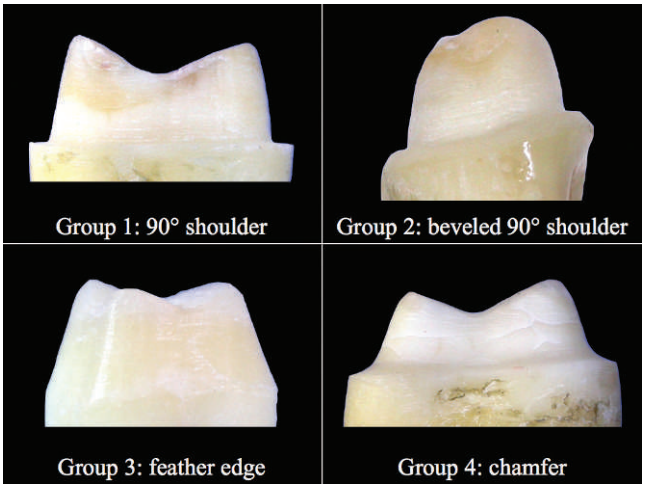


Figure 1. *Standardized tooth preparations and finish lines for indirect composite crowns: G1: 90° shoulder, G2: beveled 90° shoulder, G3: feather edge, G4: chamfer.*

- Group 1 (G1): 90° shoulder
- Group 2 (G2): beveled 90° shoulder
- Group 3 (G3): feather edge
- Group 4 (G4): chamfer

The specimens were prepared by means of calibrated, diamond rotary cutting instruments (GS.341.ISO.013, GSD.18.ISO.015, 4035.ISO.014, GSD.4.ISO.015, Intensiv Dental Production, Grancia, Switzerland) mounted on a parallel milling machine under constant water irrigation. The preparations were standardized with 1.5-mm occlusal reduction, 1.0-mm axial reduction, and 0.5-mm margin preparation; preparation depth was checked using the aforementioned silicone templates. The marginal finish lines were placed 0.5 mm coronal to the cementoenamel junction. The finishing of the preparation margins was performed using Arkansas stones mounted on a low-speed handpiece under constant water irrigation. Tooth preparations were performed with 4× magnification loupes by the same experienced prosthodontist.

Precision impressions of the preparations were taken by means of calibrated custom trays and polyether impression materials (Permadyne Penta L, 3M ESPE, Seefeld, Germany). Master casts were obtained using type IV dental stone (Fuji Rock, GC Corporation, Tokyo, Japan). Each cast was coated with a surface conditioner to seal porosities (Kleen Lube, Kerr Corporation, Orange, CA, USA), and a thin layer of a die spacer was applied to simulate the luting agent (Quick Set Spacer, Kerr Corporation).

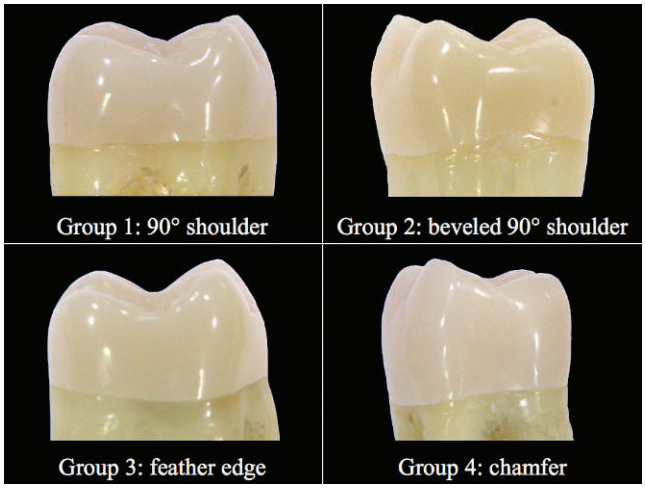


Figure 2. *Indirect composite crowns after cementation: G1: 90° shoulder, G2: beveled 90° shoulder, G3: feather edge, G4: chamfer.*

The buildup of crowns was performed by the same experienced dental technician with a microfilled hybrid resin composite (Gradia Direct, GC Corporation) using the incremental technique. Each increment was light cured for 10 seconds with a laboratory light-curing unit (Steplight, GC Corporation) for a whole polymerization time of five minutes per crown. The passive fit of the restorations was tried on the master casts and verified using a silicone disclosing agent (Fit Checker, GC Corporation). The composite crowns were polished with a hard, fine-grained polishing paste (Universal polishing paste, beige, Renfert GmbH, Hilzingen, Germany).

The adhesive cementation technique was used to lute the restorations onto the teeth. A gluing wax was used to protect the outer surfaces and margins of the crowns; then, the inner surface of each restoration was sandblasted with 50-100 μ of aluminum oxide particles. Afterward, the crowns were subjected to vaporization to clean any aluminum oxide remnant. The teeth were etched with 37% orthophosphoric acid for 15 seconds, rinsed with water for 15 seconds, and dried with cotton pellets. A thin layer of bonding (RelyX ARC Scotchbond 1 XT, 3M ESPE) was applied onto both the inner surfaces of the crowns and the preparations and light cured with 600 mW/cm^2 (Demetron Optilux 501, Kerr Corporation) for 10 seconds on each surface, as recommended by the manufacturer. The halogen light was tested for output before each cure (for consistency) in order to avoid halogen aging that could interfere with polymerization and cure. A dual-cure resin cement (RelyX ARC, 3M ESPE) was applied inside each restoration, and the crowns were carefully seated onto the preparations. Cement excess was removed with cotton pellets; then, the samples were carefully rinsed and dried to prevent fiber contamination. Each surface was light cured for 40 seconds, and the dual-cure cement was allowed to self-cure for two more minutes. Once the crowns were luted (Figure 2), the fit of restorations was visually checked with 4× magnification loupes.

Precision impressions and master casts of the cemented restorations were made as previously described. Moreover, detailed digital pictures of each crown surface (ie, buccal, palatal, mesial, and distal) were taken (Nikon D100 reflex, Nikon Corporation, Tokyo, Japan).

The specimens were stored in physiological saline solution at 37°C for 24 hours. Then, the specimens were kept in artificial saliva and underwent thermal aging by 50,000 cycles from 5°C to 55°C for 30 seconds each (Willytec/SD Mechatronik, Feldkirchen-Westerham, Germany).

A total of 36 indirect composite restorations (ie, nine per group) were subjected to root surface procedures simulating five years of semestral mechanical supportive periodontal treatment.[39,43] Each crown surface was scaled and root planed with Gracey curettes 7/8 (Immunity, Hu-Friedy, Chicago, IL, USA) by the same experienced dental hygienist. Forty continuous overlapping strokes were used across the restoration margins with a movement of about 2 mm (ie, from 1 mm apical to 1 mm coronal to the margin) at 15 mm/sec with a force of 5 N. Twenty strokes were made with the tip held parallel, whereas another 20 strokes were made with the tip held diagonal to the longitudinal axis of the teeth; the direction of the strokes was standardized using a goniometer linked to the holder of the specimens. A digital dynamometer was linked to the curettes in order to standardize the force of the strokes; the holder of the dynamometer was linked to a metal ring that could slide around a horizontal metal bar in order to follow the curettes during the instrumentation (Figure 3).

Precision impressions, master casts, and detailed digital pictures of the scaled and root planed restorations were made as previously described.

The marginal gap (MG) of the composite crowns was evaluated on 16 teeth, four per group; in each group, one-half of the specimens were randomly used as controls and not subjected to any treatment (A), whereas the remaining half was subjected to the simulated periodontal treatment (B). A CAD digital software (AutoCAD, Autodesk Inc, San Rafael, CA, USA) was used for the analysis. Ten consecutive

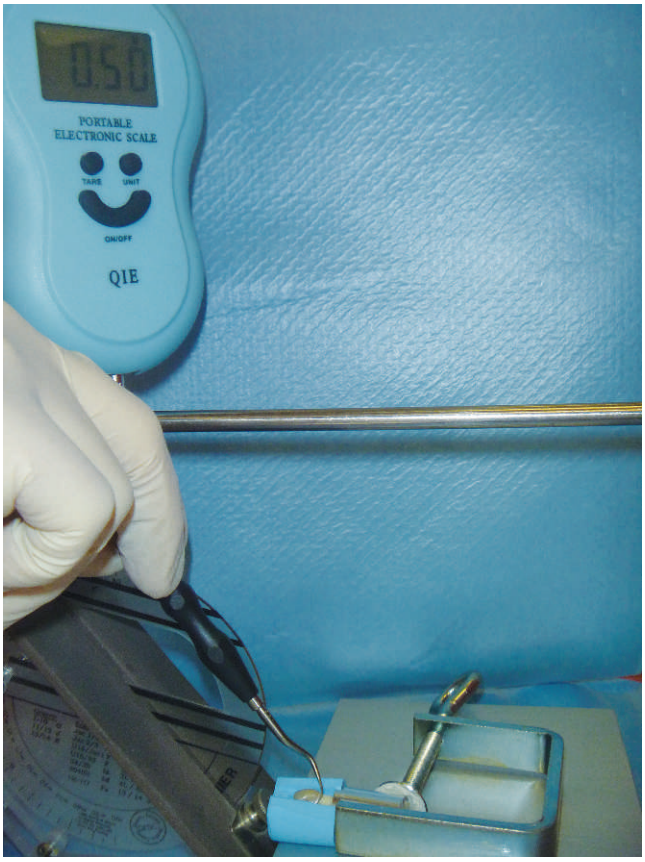


Figure 3. *Dynamometric appliance used to standardized the force and the direction of the strokes.*

points were univocally identified and measured on each surface of the specimens (Figure 4); consequently, each specimen was characterized by 40 consecutive points. In each group, 160 measurements were assessed, for a whole of 640 measured

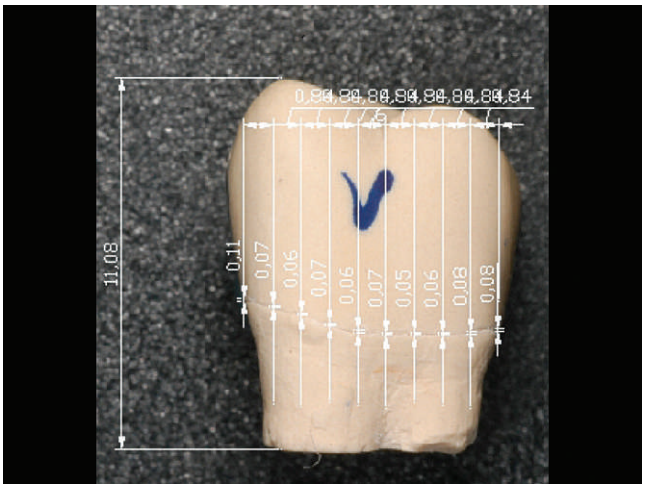


Figure 4. *Point-to-point measurements of the marginal gap by means of CAD digital software.*

Figure 5. *Specimens covering and margin exposure for microleakage test.*

points. Point-to-point measurements were performed before and after the simulated periodontal treatment.

The marginal microleakage (MM) of the composite crowns was evaluated on 40 teeth, 10 per group; in each group, one-half of the specimens were randomly used as controls and not subjected to any treatment (A), whereas the remaining half was subjected to the simulated periodontal treatment (B).

Wax and three layers of an isolating varnish were placed 1 mm coronal to the margin of the crowns; the root surfaces were fully covered with the same materials (Figure 5). Afterward, the specimens were immersed in a methylene blue supersaturated solution at room temperature for 10 minutes. The specimens were rinsed with water to remove the remnants of methylene blue, and the isolating materials were removed with a spatula.

A separating disk (Inline Wheel Slim, BM Dentale, Torino, Italy) was used to cut eight slices per specimen, for a whole of 80 sections per group. Detailed digital pictures of the slices were taken as

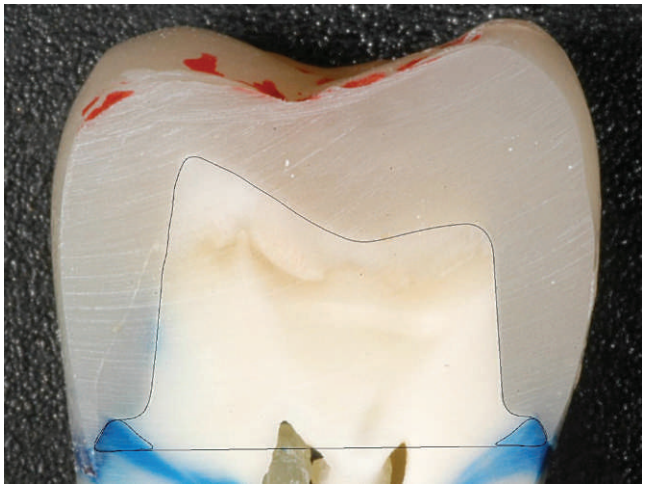


Figure 6. *Microleakage analysis by means of dedicated digital imaging and measurement software.*

previously described. Each section was analyzed for microleakage using dedicated digital imaging and measurement software (Image Pro Plus, Media Cybernetics, Bethesda, MD, USA) (Figure 6). The following parameters were recorded on each section:

- length of the adhesive interface
- linear marginal microleakage
- percentage of linear marginal microleakage
- margin-to-margin preparation area
- preparation area microleakage
- percentage of preparation area microleakage

Both the marginal gap and microleakage measurements were statistically analyzed by means of dedicated software (SPSS 11.0, SPSS Inc, Chicago, IL, USA); the analysis of variance (ANOVA), Scheffé test, and Student *t*-test were performed. In particular, the Scheffé test is a parametric, multi-comparison method applied to the set of estimates of all possible contrasts among the factor level means, so as to involve contrasts of more than two means at a time. A p-value < 0.05 was used in the rejection of the null hypotheses.

RESULTS

Marginal Gap

The mean values and standard deviations in millimeters of the marginal gap measurements are summarized in Table 1. The specimens used as controls (A) showed lower mean marginal gaps than those subjected to the simulated periodontal treatment (B); therefore, scaling and root planing procedures always increased the marginal gap of the composite crowns irrespective of the finish line design. The lowest mean value was recorded for the specimens prepared with the 90° shoulder, whereas the highest was recorded for the beveled 90° shoulder preparation.

Table 1: *Mean Values and Standard Deviations of the Marginal Gap Measurements (mm)*

Group	Not Treated (A)	Treated (B)
G1	0.091 ± 0.065	0.258 ± 0.114
G2	0.133 ± 0.050	0.463 ± 1.875
G3	0.134 ± 0.051	0.409 ± 0.134
G4	0.101 ± 0.029	0.247 ± 0.064

The ANOVA revealed statistically significant differences in the control crowns (A, $p<0.0001$) but not in the periodontally treated specimens (B, $p>0.05$).

The Scheffé test performed on subgroups A showed statistically significant differences between G1-MG-A vs G2-MG-A ($p<0.0001$), G1-MG-A vs G3-MG-A ($p<0.0001$), and G2-MG-A vs G4-MG-A ($p<0.0001$).

The Student *t*-test for independent samples was used to compare the marginal gap of the specimens prepared with the same prosthetic geometry but subjected or not to the simulated periodontal treatment: Statistically significant differences were shown in all groups (G1-MG-A vs B: $p<0.0001$, G2-MG-A vs B: $p<0.05$, G3-MG-A vs B: $p<0.0001$, G4-MG-A vs B: $p<0.0001$).

Microleakage

The mean values and standard deviations in percentage of the linear and area microleakage measurements are summarized in Table 2. The treated specimens (subgroups B) showed mean linear and area microleakage values lower than those noticed in the control crowns (subgroups A).

As to the linear measurements of the controls (A), the specimens prepared with the chamfer (G4) showed the lowest microleakage, whereas the teeth prepared with the beveled 90° shoulder (G2) were found to have the highest rate of infiltration. Differently, among the treated specimens (B), the crowns prepared with the 90° shoulder (G1) showed no linear microleakage, whereas the highest values

Table 2: *Mean Values and Standard Deviations in Percentage of the Linear and Area Microleakage Measurements*

Group	Not Treated (A)		Treated (B)	
	Linear	Area	Linear	Area
G1	1.551 ± 1.735	0.145 ± 0.342	0	0
G2	18.066 ± 16.655	13.947 ± 18.610	5.429 ± 2.025	2.060 ± 3.851
G3	1.933 ± 5.667	0.365 ± 1.156	0.359 ± 0.824	0.040 ± 0.099
G4	0.362 ± 0.570	0	0.235 ± 0.887	0

were recorded again for the beveled 90° shoulder finish line (G2).

As regards the area microleakage measurements of the controls (A), the crowns prepared with the chamfer (G4) showed no infiltration, whereas the highest percentage of microleakage was recorded for the beveled 90° shoulder finish line (G2). Among the treated specimens (B), no microleakage was noticed for both the crowns prepared with 90° shoulder (G1) and the chamfer (G4). Once more, the beveled 90° shoulder finish line (G2) showed the highest percentage of area infiltration.

The ANOVA revealed statistically significant differences for both linear and area microleakage among both the control crowns (A, $p<0.0001$) and the periodontally treated specimens (B, $p<0.0001$).

As to both the controls (A) and the treated crowns (B), the Scheffé test showed statistically significant differences for both the analyzed variables between G2-MM-A vs G1-MM-A ($p<0.0001$), G2-MM-A vs G3-MM-A ($p<0.0001$), G2-MM-A vs G4-MM-A ($p<0.0001$), G2-MM-B vs G1-MM-B ($p<0.0001$), G2-MM-B vs G3-MM-B ($p<0.0001$), and G2-MM-B vs G4-MM-B ($p<0.0001$).

The Student *t*-test for independent samples, used to compare both the marginal linear and area microleakage of the specimens prepared with the same prosthetic geometry but subjected or not to the simulated periodontal treatment, showed statistically significant differences in G1-MM-A vs B for both linear ($p<0.0001$) and area microleakage ($p<0.01$) and in G2-MM-A vs B for both linear ($p<0.0001$) and area microleakage ($p<0.005$).

According to these results, both the tested null hypotheses were rejected.

DISCUSSION

Although presenting significant differences, all the tested specimens showed a clinically acceptable marginal adaptation, irrespective of the finish line design.

According to the results of the present *in vitro* study and of other similar investigations,[38,41] the marginal gaps always increased after the simulated periodontal treatment. Conversely, scaling and root planing procedures reduced both the linear and area microleakage.

As regards the control specimens not treated with the simulated periodontal treatment (subgroups MG-A), the crowns prepared with 90° shoulder (G1) and chamfer (G4) showed mean marginal gaps $\leq$100 μ, within the range of clinical acceptability,[2,3] whereas the restorations prepared with beveled 90° shoulder (G2) and feather edge (G3) showed higher mean marginal gaps of about 130 μ. Similar to the control specimens, after simulated periodontal treatment (subgroups MG-B), the crowns prepared with 90° shoulder (G1) and chamfer (G4) showed mean marginal gaps of about 250 μ, lower than those recorded for the restorations prepared with beveled 90° shoulder (G2) and feather edge (G3) that reached mean marginal gaps of about 400–450 μ.

The marginal gap values recorded in the present study in the absence of periodontal instrumentation (subgroups MG-A) were comparable to those obtained in other similar investigations.[20,38,41,43] After the simulated periodontal treatment (subgroups MG-B), the resulting marginal gap measurements were higher than those recorded for the controls, reaching doubled or tripled values. Such increases were proportional to the gap evidenced in the absence of the periodontal treatment.

As regards the feather-edge preparation, significant marginal gap values were recorded, but they were not proportional to the microleakage. It could be speculated that this was probably due to the combination of two phenomena: the tapering of the preparation, which favors an even distribution of the luting agent, and the orthogonal or slightly oblique cut of the dentinal tubules, which could enhance the creation of the adhesive hybrid layer. Nonetheless, in the presence of the feather-edge preparation, the marginal gap could worsen over time.

According to the results of the present investigation, composite crowns prepared with beveled 90° shoulder and feather edge could fracture during function and periodic periodontal treatments more easily than restorations prepared with other finish lines due to their thin margins. As a consequence, beveled shoulders and feather edges should not be considered a first choice for the fabrication of composite crowns.

As to the marginal microleakage, a direct proportionality between linear and area infiltration was noticed in both control (A) and treated (B) crowns. The 90° shoulder (G1), the feather-edge (G3), and the chamfer (G4) finish lines showed lower marginal microleakage than the beveled 90° shoulder (G2); as regards the specimens not subjected to any instrumentation, the latter showed massive marginal microleakage and the rate of microleakage seemed scarcely related to the dimensions of the marginal gap. This was probably due to the fact that, in the presence of the same marginal gap, the beveled preparation exposed a greater number of dentinal tubules than other finish geometries. Another possible explanation for such a massive microleakage with the beveled 90° shoulder is the higher cement thickness due to a more difficult flow of the luting agent during cementation;[38,41] subsequently, the crown margin was probably impacted by a more critical polymerization shrinkage. The latter hypothesis is just a speculation and should be verified experimentally.

According to the results of the present *in vitro* study, no correlation between the marginal gap and microleakage was noticed. The root surface instrumentation significantly reduced the marginal microleakage; these data seem in contrast to the recorded increased marginal gap after the periodontal treatment. This was probably due to the debris compacted at the level of the margin by the curettes moving in an apical-coronal direction, increasing the resistance to microleakage; such a speculation should be verified by further experimental investigations.

The amorphous debris could increase bacteria accumulation over time; as a consequence, both professional and domiciliary hygiene maintenance should be stressed in the presence of composite restorations in order to reduce the need of scaling and root planing procedures. As regards the domiciliary oral hygiene procedures, the use of spongy dental flosses and interproximal brushes should be recommended to the patients. Furthermore, if scaling and root planing are needed, dentists should polish the margins of composite crowns by means of rotary bristles and interproximal finishing strips.

Moreover, the reduction of microleakage in the treated specimens could also be explained considering that the instrumentation removed areas of infiltrated margins; nevertheless, the fractures that occurred at the level of some margins, particularly in the specimens prepared with the chamfer finish line (G4), did not influence significantly the reduction of microleakage after the periodontal treatment.

CONCLUSION

Within the limitations of the present *in vitro* study, the following conclusions can be drawn:

- The manual mechanical periodontal instrumentation altered the surface of composite crowns and the marginal gap increased proportionally after such treatment.
- The 90° shoulder and the chamfer finish lines revealed lower marginal gaps both before and after periodontal treatment and showed a stable marginal integrity.
- The beveled 90° shoulder and the feather edge presented with a higher risk of damage due to scaling with curettes.
- The 90° shoulder, the feather-edge, and the chamfer preparations showed lower marginal microleakage both before and after periodontal treatment.
- The mechanical root surface instrumentation reduced the marginal microleakage.

According to the results of the present *in vitro* study, the 90° shoulder and the chamfer preparation proved to be a viable option for the fabrication of composite crowns, whereas the beveled 90° shoulder and the feather edge should not be recommended.

Conflict of Interest

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

(Accepted 21 November 2011)

REFERENCES

1. Lehmann F, Spiegl K, Eickemeyer G, & Rammelsberg P (2009) Adhesively luted, metal-free composite crowns after five years *Journal of Adhesive Dentistry* **11(6)** 493-498.
2. Zarone F, Sorrentino R, Vaccaro F, Russo S, & De Simone G (2005) Retrospective clinical evaluation of 86 Procera AllCeram anterior single crowns on natural and implant-supported abutments *Clinical Implant Dentistry and Related Research* **7(Supplement 1)** S95-S103.
3. Sorrentino R, Galasso L, Tetè S, De Simone G, & Zarone F (2012) Clinical evaluation of 209 all-ceramic single crowns cemented on natural and implant-supported abutments with different luting agents: a 6-year retrospective study *Clinical Implant Dentistry and Related Research* **14(2)** 184-197.
4. Ohlmann B, Gruber R, Eickemeyer G, & Rammelsberg P (2008) Optimizing preparation design for metal-free composite resin crowns *Journal of Prosthetic Dentistry* **100(3)** 211-219.
5. Behr M, Rosentritt M, & Handel G (2003) Fiber-reinforced composite crowns and FPDs: A clinical report *International Journal of Prosthodontics* **16(3)** 239-243.
6. Emilson CG, & Lindquist B (2004) The clinical performance of composite resin crowns after 5–8 years *Journal of Dental Research* **83(Special Issue A)** 223.
7. Fokkinga WA, Kreulen CM, Bronkhorst EM, & Creugers NH (2007) Up to 17-year controlled clinical study on post-and-cores and covering crowns *Journal of Dentistry* **35(10)** 778-786.
8. Fokkinga WA, Kreulen CM, Bronkhorst EM, & Creugers NH (2008) Composite resin core-crown reconstructions: An up to 17-year follow-up of a controlled clinical trial *International Journal of Prosthodontics* **21(2)** 109-115.
9. Rammelsberg P, Spiegl K, Eickemeyer G, & Schmitter M (2005) Clinical performance of metal-free polymer crowns after 3 years in service *Journal of Dentistry* **33(6)** 517-523.
10. Roeters JJ (2001) Extended indications for directly bonded composite restorations: A clinician's view. *Journal of Adhesive Dentistry* **3(1)** 81-87.
11. Torbjorner A, & Fransson B (2004) A literature review on the prosthetic treatment of structurally compromised teeth *International Journal of Prosthodontics* **17(3)** 369-376.
12. Vanoorbeek S, Vandamme K, Lijnen I, & Naert I (2010) Computer-aided designed/computer-assisted manufactured composite resin versus ceramic single-tooth restorations: A 3-year clinical study *International Journal of Prosthodontics* **23(3)** 223-230.
13. Nakamura T, Imanishi A, Kashima H, Ohyama T, & Ishigaki S (2001) Stress analysis of metal-free polymer crowns using the three-dimensional finite element method *International Journal of Prosthodontics* **14(5)** 401-405.
14. Fokkinga WA, Le Bell AM, Kreulen CM, Lassila LV, Vallittu PK, & Creugers NH (2005) *Ex vivo* fracture resistance of direct resin composite complete crowns with and without posts on maxillary premolars *International Endodontic Journal* **38(4)** 230-237.
15. Freiberg RS, & Ferracane JL (1998) Evaluation of cure, properties and wear resistance of Artglass dental composite *American Journal of Dentistry* **11(5)** 214-218.
16. Kern M, Strub JR, & Lü XY (1999) Wear of composite resin veneering materials in a dual-axis chewing simulator *Journal of Oral Rehabilitation* **26(5)** 372-378.
17. Ohlmann B, Trame JP, Dreyhaupt J, Gabbert O, Koob A, & Rammelsberg P (2008) Wear of posterior metal-free polymer crowns after 2 years *Journal of Oral Rehabilitation* **35(10)** 782-788.
18. Suzuki S, Nagai E, Taira Y, & Minesaki Y (2002) *In vitro* wear of indirect composite restoratives *Journal of Prosthetic Dentistry* **88(4)** 431-436.
19. Nagasiri R, & Chitmongkolsuk S (2005) Long-term survival of endodontically treated molars without crown coverage: a retrospective cohort study *Journal of Prosthetic Dentistry* **93(2)** 164-170.
20. Cho L, Song H, Koak J, & Heo S (2002) Marginal accuracy and fracture strength of ceromer/fiber-reinforced composite crowns: Effect of variations in preparation design *Journal of Prosthetic Dentistry* **88(4)** 388-395.
21. Vermilyea SG, Prasanna MK, & Agar JR (1994) Effect of ultrasonic cleaning and air polishing on porcelain labial margin restorations *Journal of Prosthetic Dentistry* **71(5)** 447-452.
22. Lehmann F, Eickemeyer G, & Rammelsberg P (2004) Fracture resistance of metal-free composite crowns—Effects of fiber reinforcement, thermal cycling, and cementation technique *Journal of Prosthetic Dentistry* **92(3)** 258-264.
23. Rammelsberg P, Eickemeyer G, Erdelt K, & Pospiech P (2000) Fracture resistance of posterior metal-free polymer crowns *Journal of Prosthetic Dentistry* **84(3)** 303-308.
24. Krejci I, Mueller E, & Lutz F (1994) Effects of thermocycling and occlusal force on adhesive composite crowns *Journal of Dental Research* **73(6)** 1228-31232.
25. Behr M, Rosentritt M, Latzel D, & Handel G (2003) Fracture resistance of fiber-reinforced vs non-fiber-reinforced composite molar crowns *Clinical Oral Investigations* **7(3)** 135-159.
26. Behr M, Rosentritt M, Sikora MI, Karl P, & Handel G (2003) Marginal adaptation and fracture resistance of adhesively luted glass fibre-composite reinforced molar crowns with different inner crown surfaces *Journal of Dentistry* **31(7)** 503-508.
27. Fokkinga WA, Kreulen CM, Le Bell-Rönnlöf AM, Lassila LV, Vallittu PK, & Creugers NH (2006) Fracture behavior of structurally compromised non-vital maxillary premolars restored using experimental fiber reinforced composite crowns *American Journal of Dentistry* **19(6)** 326-332.
28. Suzuki C, Miura H, Okada D, & Komada W (2008) Investigation of stress distribution in roots restored with different crown materials and luting agents *Dental Materials Journal* **27(2)** 229-236.

29. Schenke F, Hiller KA, Schmalz G, & Federlin M (2008) Marginal integrity of partial ceramic crowns within dentin with different luting techniques and materials *Operative Dentistry* **33(5)** 516-525.

30. Attia A, Abdelaziz KM, Freitag S, & Kern M (2006) Fracture load of composite resin and feldspathic all-ceramic CAD/CAM crowns *Journal of Prosthetic Dentistry* **95(2)** 117-123.

31. Ku CW, Park SW, & Yang HS (2002) Comparison of the fracture strengths of metal-ceramic crowns and three ceromer crowns *Journal of Prosthetic Dentistry* **88(2)** 170-175.

32. Research, Science, and Therapy Committee of the American Academy of Periodontology (2001) Treatment of plaque-induced gingivitis, chronic periodontitis, and other clinical conditions *Journal of Periodontology* **72(12)** 1790-1800.

33. Lee SY, Lai YL, & Morgano SM (1995) Effects of ultrasonic scaling and periodontal curettage on surface roughness of porcelain *Journal of Prosthetic Dentistry* **73(3)** 227-232.

34. Cobb CM (1996) Non-surgical pocket therapy: Mechanical *Annals of Periodontology* **1(1)** 443-490.

35. Eberhard J, Ehlers H, Falk W, Açil Y, Albers HK, & Jepsen S (2003) Efficacy of subgingival calculus removal with Er:YAG laser compared to mechanical debridement: An *in situ* study *Journal of Clinical Periodontology* **30(6)** 511-518.

36. Chan YK, Needleman IG, & Clifford LR (2000) Comparison of four methods of assessing root surface debridement *Journal of Periodontology* **71(3)** 385-393.

37. Carey HM, & Daly CG (2001) Subgingival debridement of root surfaces with a micro-brush: Macroscopic and ultrastructural assessment *Journal of Clinical Periodontology* **28(9)** 820-827.

38. Ruhling A, Wulf J, Schwahn C, & Kocher T (2004) Surface wear on cervical restorations and adjacent enamel and root cementum caused by simulated long-term maintenance therapy *Journal of Clinical Periodontology* **31(4)** 293-298.

39. Ritz L, Hefti AF, & Rateitschak KH (1991) An *in vitro* investigation on the loss of root substance in scaling with various instruments *Journal of Clinical Periodontology* **18(9)** 643-647.

40. Bjornson EJ, Collins DE, & Engler WO (1990) Surface alteration of composite resins after curette, ultrasonic, and sonic instrumentation: An *in vitro* study *Quintessence International* **21(5)** 381-389.

41. Gorfil C, Nordenberg D, Liberman R, & Ben-Amar A (1989) The effect of ultrasonic cleaning and air polishing on the marginal integrity of radicular amalgam and composite resin restorations. An *in vitro* study *Journal of Clinical Periodontology* **16(3)** 137-139.

©Operative Dentistry, 2012, **37-6,** 660-664

Quantification of Peroxide Ion Passage in Dentin, Enamel, and Cementum After Internal Bleaching With Hydrogen Peroxide

RM Palo • I Bonetti-Filho • MC Valera
CHR Camargo • SEA Camargo • C Moura-Netto
C Pameijer

Clinical Relevance

It is not possible to extrapolate the results directly to the clinical setting; however, hydrogen peroxide when placed in the pulp chamber passes through dental hard tissues and reaches the external surface and periodontal tissues.

Renato Miotto Palo, DDS, MsC, PhD, professor of endodontics, UNESP–Univ Estadual Paulista, São José dos Campos, SP, Brazil

Idomeo Bonetti-Filho, DDS, MsC, PhD, associate professor of endodontics, UNESP–Univ Estadual Paulista, Araraquara, SP, Brazil

Marcia Carneiro Valera, DDS, MsC, PhD, associate professor of endodontics, UNESP–Univ Estadual Paulista, São José dos Campos, SP, Brazil

Carlos Henrique Ribeiro Camargo, DDS, MsC, PhD, associate professor of endodontics, UNESP–Univ Estadual Paulista, São José dos Campos, SP, Brazil

*Samira Esteves Afonso Camargo, DDS, MsC, PhD, professor of biochemistry, UNESP–Univ Estadual Paulista, São José dos Campos, SP, Brazil

Cacio Moura-Netto, DDS, MsC, PhD, professor of endodontics, Universidade Paulista–UNIP, São Paulo, SP, Brazil

Cornelis Pameijer, DDS, DSc, PhD, professor emeritus, University of Connecticut, Farmington, CT, USA

SUMMARY

The aim of this study was to evaluate the amount of peroxide passage from the pulp chamber to the external enamel surface during the internal bleaching technique. Fifty bovine teeth were sectioned transversally 5 mm below the cemento-enamel junction (CEJ), and the remaining part of the root was sealed with a 2-mm layer of glass ionomer cement. The external surface of the samples was coated with nail varnish, with the exception of standardized circular areas (6-mm diameter) located on the enamel, exposed dentin, or cementum surface of the tooth. The teeth were divided into three

*Corresponding author: Av. Fco José Longo, 777, Jd São Dimas, São José dos Campos /SP, Brazil, 12245-000; e-mail: samira@fosjc.unesp.br

DOI: 10.2341/11-334-L

experimental groups according to exposed areas close to the CEJ and into two control groups (n=10/group), as follows: GE, enamel exposure area; GC, cementum exposed area; GD, dentin exposed area; Negative control, no presence of internal bleaching agent and uncoated surface; and Positive control, pulp chamber filled with bleaching agent and external surface totally coated with nail varnish. The pulp chamber was filled with 35% hydrogen peroxide (Opalescence Endo, Ultradent). Each sample was placed inside of individual flasks with 1000 μL of acetate buffer solution, 2 M (pH 4.5). After seven days, the buffer solution was transferred to a glass tube, in which 100 μL of leuco-crystal violet and 50 μL of horseradish peroxidase were added, producing a blue solution. The optical density of the blue solution was determined by spectrophotometer and converted into microgram equivalents of hydrogen peroxide. Data were submitted to Kruskal-Wallis and Dunn-Bonferroni tests (α=0.05). All experimental groups presented passage of peroxide to the external surface that was statistically different from that observed in the control groups. It was verified that the passage of peroxide was higher in GD than in GE ($p<0.01$). The GC group presented a significantly lower peroxide passage than did GD and GE ($p<0.01$). It can be concluded that the hydrogen peroxide placed into the pulp chamber passed through the dental hard tissues, reaching the external surface and the periodontal tissue. The cementum surface was less permeable than were the dentin and enamel surfaces.

INTRODUCTION

The use of tooth whitening techniques has become a common treatment modality aimed at achieving esthetically desirable appearance. This procedure preserves tooth structure and avoids invasive restorative interventions to correct color anomalies.[1]

In order for intrinsic stains to be removed by the action of bleaching agents, the oxidizing ions must penetrate enamel and dentin,[2–5] leading to an oxidation reaction. Hydrogen peroxide (HP) is a strong oxidizing agent that can act on dentin, modifying its mechanical and chemical properties. The bleaching mechanism of HP has not yet been fully established, and some controversy exists.[6,7]

The HP dissociates into free radicals with unpaired electrons and is being reduced by releasing these electrons, while as a consequence the substances that accept the electrons are being bleached by oxidation. This results in a molecular rupture and in a change in the energy absorption at a molecular level within the pigmented dental organic matrix, forming simpler and smaller molecules that reflect light in a different way, creating a successful bleaching effect.[1,8,9]

The permeability of teeth increases after penetration of oxidative ions,[3,4,7] allowing the passage of these ions to the external root surface and consequently to the periodontal ligament. Another potential consequence of the bleaching action can be the development of cervical root resorption. This can occur as a result of the fact that HP and other oxygen radicals penetrate the periodontal membrane, causing tissue and cellular destruction.[4,10] Nevertheless, use of 30% HP and sodium perborate (SP) as bleaching agents[3,11,12] has been extensively practiced by those in the dental profession.

In order to quantify the peroxide ions that are released by a bleaching agent from the pulp chamber through exposed dentin, dentin that is covered by enamel or by cementum during internal bleaching procedures, this study tested the following null hypothesis: (H0) the bleaching agent will not pass through the dental hard tissues.

MATERIALS AND METHODS

This project was developed in accordance with the Research Ethics Code (approved under No. 002451/2008-PH/CEP). Fifty freshly extracted bovine lateral incisors from early calves of approximately the same age were cleaned, immersed in physiological saline, and kept in a freezer (−18°C) until use. Access openings were prepared on the lingual surface with a high-speed hand piece and diamond bur under copious water-cooling. Pulp tissue was extirpated and the pulp chamber irrigated with saline.

The roots were sectioned perpendicular to the long axis, 5 mm below the cemento-enamel junction (CEJ), using a carbide disc in a low-speed hand piece. A glass ionomer cement (Vidrion R, SS White, Rio de Janeiro, Brazil) plug was placed 2 mm apical from the CEJ, serving as a barrier to the root canal. The root below the glass ionomer barrier was etched with 35% phosphoric acid gel, rinsed, and sealed with a light-curing bonding agent (Single Bond Adhesive, 3M ESPE, Minneapolis, MN, USA) and a light-curing resin composite (TPH, Dentsply/Caulk Div., Milford, DE, USA).

Passage of HP was evaluated by application to dimensionally standardized areas on the external

surface of the tooth. The external surface of the samples was coated with nail varnish, with the exception of standardized circular areas (6-mm diameter) located on the enamel, dentin-exposed, or cementum surface of the tooth. The circular areas of exposed dentin with 6-mm width and 2-mm depth were created using cylindrical diamond burs (No. 2094, KG Sorensen Ind. Ltda, Barueri, SP, Brazil).

In order to make the specimens waterproof, two layers of transparent nail varnish were applied. After removal of the labels dimensionally standardized areas were available for measurements.

The teeth were divided into three experimental groups according to exposed areas close to the CEJ and into two control groups (n=10/group), as follows: GE, enamel exposure area; GC, cementum exposed area; GD, dentin exposed area; Negative control, no presence of internal bleaching agent and uncoated surface; and Positive control, pulp chamber filled with bleaching agent and external surface totally coated with nail varnish.

The pulp chamber was filled with 35% HP (Opalescence Endo, Ultradent Products Inc, South Jordan, UT, USA), followed by a resin composite filling (TPH, Dentsply/Caulk Div.). After light-curing the remainder of the access opening was sealed after etching with 37% phosphoric acid (Alpha Etch gel, DFL, Rio de Janeiro, Brazil) for 15 seconds, rinsing, lightly air-drying, and application of a bonding agent (Single Bond Adhesive, 3M ESPE). After light-curing for 20 seconds (780 mW/mm^2), a resin composite (TPH, Dentsply/Caulk Div.) was placed and light-cured for 20 seconds.

The specimens were placed in a reservoir containing 1000 μL of acetate buffer solution, 2M (pH=4.5), making sure that the exposed areas were submerged. The acetate buffer was necessary to stabilize the HP that might pass from the pulp chamber to the external surface. Specimens were then stored in an incubator at 37°C at 100% relative humidity. After seven days, the acetate buffer solution was removed from the reservoirs using micropipettes and transferred to a glass tube. The reservoirs were rinsed twice with deionized water. One hundred microliters of 0.5 mg/mL leuco-crystal violet (Sigma Chemical Co, Sigma-Aldrich, São Paulo, Brazil) and 50 μL of 1 mg/mL enzyme horseradish peroxidase (Sigma Chemical Co, Sigma-Aldrich) were also added to each tube, and the solution was diluted to 3 mL with distilled water. The optical density of the resulting blue color in the tubes was measured with a spectrophotometer (UV Spectrophotometer, UV-1203, Shimadzu, Kyoto, Japan) at a wavelength of 596 nm.

Table 1: *Amount of Peroxide (μg/mL) That Penetrated From the Pulp Chamber to the External Root Surface During Internal Bleaching (SD = Standard Deviation)*

Groups	Mean, μg/mL (±SD)
Group enamel	0.957 (1.366±0.766)
Group cementum	0.886 (1.630±0.582)
Group dentin	0.965 (2.234±0.758)
Negative control	0.033 (0.099±0.000)
Positive control	0.024 (0.043±0.003)

A standard curve, in which eight points of HP were associated at intervals of 0.25 μg/mL until 2.0 μg/mL, according to their corresponding absorbance, was used to convert the optical density values obtained from the samples into microgram equivalents of HP. The results were statistically analyzed using Kruskal-Wallis and Dunn-Bonferroni tests ($p<0.05$).

RESULTS

The amount of peroxide (μg/mL; mean ± SD) that penetrated from the pulp chamber through the tooth structure is shown in Table 1. All experimental groups presented passage of peroxide bleaching agent to the root surface and were statistically different from the negative and positive control groups ($p<0.05$). It was verified that the ion penetration was higher in GD (0.965 μg/mL; 2.234 ± 0.758), followed by GE (0.957 μg/mL; 1.366 ± 0.766). GC demonstrated passage of peroxide that was lower, at 0.886 μg/mL (1.630 ± 0.582), than that of GD and GE ($p<0.05$).

The data were submitted to the Dunn-Bonferroni statistical test and it was verified that control and experimental groups showed statistically significant differences ($p<0.05$). The Kruskal-Wallis test showed no statistically significant difference among the experimental groups ($p>0.05$).

DISCUSSION

External and internal bleaching agents are able to penetrate into dentin.[12–14] These bleaching agents reduce the calcium and phosphate ions of the dental

hard tissues, promoting a higher permeability in these tissues. Carrasco and others[13] showed that different bleaching agents can increase the permeability of the coronal dentin of endodontically treated teeth.

The passage of HP occurs mainly as a result of its low molecular weight and ability to denature proteins, which increases the ion movement through the enamel, dentin, and cementum. Dental hard tissues present an organic content; the HP penetrates through these structures, promoting increased porosity and loss of substances of the protein matrix as a result of free radical oxidation.

The present study verified that the penetration of the peroxide from the pulp chamber to the external root surface occurred in all experimental groups. This was demonstrated by measuring ions from the bleaching agents passing through dentin, enamel, and cementum. These results are not in accordance with those of Kehoe,[15] who reported that cementum presented a barrier for ion passage.

The amount of bleaching agent that passages through the tooth structure is influenced by enamel, dentin, and cementum thickness. It is known that the higher the enamel, dentin, or cementum thickness, the less will be the passage of bleaching agents. In our study we observed a lower passage of HP in the cementum that can be correlated with the different thicknesses of the dental hard tissues.

Fuss and others[16] evaluated the diffusion of a mixture of SP plus HP and calcium hydroxide. They determined that the whitening materials, in contrast to calcium hydroxide, offered an easier and faster diffusion through dentin. This faster diffusion is probably due to the small size molecules of the bleaching agents.[5,16,17] Additionally, the cervical region is more permeable compared to other areas of the root,[18] even when the bleaching agent is sealed within the pulp chamber. Pressure builds up inside the pulp chamber, because of the oxygen liberation caused by HP and SP decomposition. This pressure forces the bleaching agents into the dentinal tubules.[7,19] HP passage as well as other oxygen radicals are not desirable and must be minimized. When in contact with tissues, these ions may cause cellular and tissue destruction.[10]

In the present study, it is of interest to note that oxidative ions were able to pass through exposed dentin, dentin covered by enamel, and dentin covered by cementum. However, it is known that this junction may exhibit flaws that expose the dentin to the periodontal tissue in this area. According to Neuvald and Consolaro,[20] this is the case for almost 10% of teeth. Under these conditions, ions coming from the bleaching agents can reach these tissues easily and in high quantities, thus initiating an inflammatory reaction.

A significant aspect that must be taken into consideration is that dentin autoimmunity presents specific proteins, which often are not accessible to the human immunologic recognition cells. Despite its organization, the direct exposure of noncollagen proteins to the bone maintains the dentin proteins incorporated in a mineralized matrix and acts as a sequestrated antigen.[12]

Nevertheless, with exposure, in the case of an inflammatory process at the cervical region, the cells are not recognized as their own by the human immune system. A specific response will start, represented by cellular intent on eliminating antigens, in which macrophages will act as the main executors. However, if the CEJ presents an irregular anatomical form, the initiator mechanism of resorptive processes requires the presence of local factors, such as cytokine liberation that activate clastic cells Other factors can cause external cervical resorptions, such as bleaching agents, trauma, and induced dental movement.[21]

In the present study, we used bovine teeth as they presented similar size and age, promoting a better standardization of the specimens. The diameter of dentin tubules in bovine teeth is smaller and the intertubular dentin area is larger than in human teeth.[22] On the other hand, the human tooth and its structural and morphologic characteristics can influence the passage of bleaching agents. A previous study[22] showed that human teeth are more permeable to bleaching agents than bovine teeth.

Based on the results of this study, the null hypothesis (H0) was rejected. Thus, this study demonstrated that the oxidative power of the HP was able to pass through all hard dental tissues. It could be concluded that when placed into the pulp chamber, the HP passes through dental hard tissues, reaching the external surface and periodontal tissues.

Conflict of Interest

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

(Accepted 10 January 2012)

REFERENCES

1. Plotino G, Buono L, Grande NM, Pameijer CH, & Somma F (2008) Nonvital tooth bleaching: A review of the literature and clinical procedures *Journal of Endodontics* **34(4)** 394-407.
2. Wainwright WW, & Lemoine FA (1950) Rapid diffuse penetration of intact enamel and dentin by carbon-14-labeled urea *Journal of the American Dental Association* **41(2)** 135-145.
3. Simon RH, Scoggin CH, & Patterson D (1981) Hydrogen peroxide causes the fatal injury to human fibroblast exposed to oxygen radicals *Journal of Biological Chemistry* **256(14)** 7181-7186.
4. Haywood VB, Leech T, Heymann HO, Crumpler D, & Bruggers K (1990) Nightguard vital bleaching: Effects on enamel surface texture and diffusion *Quintessence International* **21(10)** 801-804.
5. Gökay O, Ziraman F, Cali Asal A, & Saka OM (2008) Radicular peroxide penetration from carbamide peroxide gels during intracoronal bleaching *International Endodontic Journal* **41(7)** 556-560.
6. Rodrigues LM, Vansan LP, Pécora JD, & Marchesan MA (2009) Permeability of different groups of maxillary teeth after 38% hydrogen peroxide internal bleaching *Brazilian Dental Journal* **20(4)** 303-306.
7. Lima AF, Fonseca FM, Cavalcanti AN, Aguiar FH, & Marchi GM (2010) Effect of the diffusion of bleaching agents through enamel on dentin bonding at different depths *American Journal of Dentistry* **23(2)** 113-115.
8. McEvoy SA (1989) Chemical agents for removing intrinsic stains from vital teeth. II. Current techniques and their clinical application *Quintessence International* **20(6)** 379-384.
9. Bucheli JC, García GA, Méndez SR, Osorio NR, Lombana N, & Muñoz HR (2008) The effect of tooth bleaching on substance p expression in human dental pulp *Journal of Endodontics* **34(12)** 1462-1465.
10. Halliwel B, & Gutteridge JM (1984) Oxygen toxicity, oxygen radicals, transition metals and disease *Biochemistry Journal* **219(1)** 1-14.
11. Camps J, Pommel L, Aubut V, & About I (2010) Influence of acid etching on hydrogen peroxide diffusion through human dentin *American Journal of Dentistry* **23(3)** 168-170.
12. Palo RM, Valera MC, Camargo SE, Camargo CH, Cardoso PE, Mancini MN, & Pameijer CH (2010) Peroxide penetration from the pulp chamber to the external root surface after internal bleaching *American Journal of Dentistry* **23(3)** 171-174.
13. Carrasco LD, Fröner IC, Corona SA, & Pécora JD (2003) Effect of internal bleaching agents on dentinal permeability of non-vital teeth: Quantitative assessment *Dental Traumatology* **19(2)** 85-89.
14. Hannig C, Weinhold HC, Becker K, & Attin T (2010) Diffusion of peroxides through dentin in vitro with and without prior use of a desensitizing varnish *Clinical Oral Investigation* **15(6)** 863-868.
15. Kehoe JC (1987) pH reversal following in vitro bleaching of pulpless teeth *Journal of Endodontics* **13(1)** 6-9.
16. Fuss Z, Szajkis S, & Tagger M (1989) Tubular permeability to calcium hydroxide and to bleaching agents *Journal of Endodontics* **15(8)** 362-364.
17. Ramp WK, Arnold RR, Russell JE, & Yancey JM (1987) Hydrogen peroxide inhibits glucose metabolism and collagen synthesis in bone *Journal of Periodontology* **58(5)** 340-344.
18. Chen JH, Xu JW, & Shing CX (1993) Decomposition rate of hydrogen peroxide bleaching agents under various chemical and physical conditions *Journal of Prosthetic Dentistry* **69(1)** 46-48.
19. Marshall FJ, Massler M, & Dute HL (1960) Effects of endodontic treatment on permeability of root dentin *Oral Surgery Oral Medicine Oral Pathology Oral Radiology, and Endodontics* **13(1)** 208-223.
20. Neuvald LR, & Consolaro A (2000) Cementoenamel junction: Microscopic analysis and external cervical resorption *Journal of Endodontics* **26(9)** 503-508.
21. Rotstein I, Dankner E, Goldman A, Heling I, Stabholz A, & Zalkind M (1996) Histochemical analysis of dental hard tissues following bleaching *Journal of Endodontics* **22(1)** 23-25.
22. Camargo SEA, Valera MC, Camargo CHR, Mancini MNG, & Menezes MM (2007) Penetration of 38% hydrogen peroxide into the pulp chamber in bovine and human teeth submitted to office bleach technique *Journal of Endodontics* **33(9)** 1074-1077.

©Operative Dentistry, 2012, **37-6,** 665-671

Effects of Food-simulating Liquids on Surface Properties of Giomer Restoratives

TJM Kooi • QZ Tan • AUJ Yap
W Guo • KJ Tay • MS Soh

Clinical Relevance

Giomer restoratives, like other direct and indirect composites, are softened by food-simulating liquids, especially citric acid and ethanol. They are also roughened by citric acid.

SUMMARY

This study examined the effects of food-simulating liquid (FSL) on the hardness and roughness of giomer restoratives based on pre-reacted glass ionomer (PRG) technology. The materials investigated included a regular (Beautifil II [BT]) and a recently introduced injectable (Beautifil Flow Plus F00 [BF]) hybrid PRG composite. A direct hybrid composite (Filtek Z250 [ZT]) and an indirect hybrid composite (Ceramage [CM]) were used for comparison. The materials were placed into customized square molds (5 mm × 5 mm × 2.5 mm), covered with Mylar strips, and cured according to manufacturers' instructions. The materials were then conditioned in air (control), distilled water, 50% ethanol solution, and 0.02 N citric acid at 37°C for seven days. Specimens (n=6) were then subjected to hardness testing (Knoop) and surface profilometry. Data were analyzed using one-way analysis of variance and *post hoc* Scheffe test ($p<0.05$). Mean Knoop hardness values for the control group (air) ranged from 53.4 ± 3.4 (BF) to 89.5 ± 5.2 (ZT), while mean surface roughness values values ranged from 0.014 ± 0.002 (ZT) to 0.032 ± 0.001 (BT). All materials were significantly softened by FSL. The degree of soften-

*Adrian UJ Yap, PhD, MSc, BDS, Grad Dip Psychotherapy, Raffles Hospital; Faculty of Dentistry, National University of Singapore, Singapore; School of Science and Technology, SIM University, Singapore

Terence Jun Ming Kooi, NA, Faculty of Dentistry, National University of Singapore, Singapore

Qing Zhen Tan, NA, Faculty of Dentistry, National University of Singapore, Singapore

Weidi Guo, NA, Faculty of Dentistry, National University of Singapore, Singapore

Koong Jiunn Tay, NA, Faculty of Dentistry, National University of Singapore, Singapore

Mui Siang Soh, PhD, MSc, BSc, Institute of Materials Research and Engineering, Singapore

*Corresponding author: Lower Kent Ridge Road, Singapore, Singapore 119083, Singapore; e-mail: aujyaprd@gmail.com

DOI: 10.2341/11-419-L

ing by the different FSLs was material dependent. The hardness of giomers was most affected by citric acid and ethanol. The smoothest surface was generally observed with the control group. Giomer restoratives were significantly roughened by citric acid.

INTRODUCTION

Resin-based composite materials are widely used in restorative dentistry. Clinically, composite restorations are exposed either intermittently or continuously to chemical agents found in saliva, food, and beverages.[1] Results of previous *in vitro* studies[2–4] have shown that food substances can significantly affect the hardness and roughness of composites. Giomers or pre-reacted glass ionomer (PRG) composites are the latest type of glass ionomer–composite hybrid materials, in which glass ionomer fillers (consisting of fluorosilicate particles pre-reacted with polyacrylic acid) are incorporated into a resin matrix. Coupling agents bond the fillers to the matrix and catalysts are added to initiate polymerization of the material. Giomers, like other dental composites, also require bonding agents to adhere to tooth structure. They are light-activated, easy to handle, and release fluoride.

The clinical performance of giomers has been evaluated in several studies.[5,6] In an eight-year trial involving Class I and II restorations, Gordan and others[5] reported no restoration failures. Significant changes were observed only for marginal adaptation at occlusal surfaces and marginal staining at proximal surfaces. In addition, no significant difference in clinical performance was observed[6] between giomer and microfilled composite restorations in Class V cavities after three years. *In vitro* studies[7,8] comparing giomers to resin-modified glass ionomer cements showed that giomers had significantly higher flexural strength. Giomers were also found to be harder than minifilled composite resins and ormocers[9] and had better polishability than did conventional glass ionomers.[10] The high-fluoride release and recharge properties of giomers minimize recurrent caries and demineralization.[11] Based on several studies,[12–16] giomers have a reported caries inhibiting effect of 14%-35% compared to non–fluoride-releasing restorative materials.

Information regarding the influence of food-simulating liquids (FSLs) on the surface hardness and roughness of giomer restoratives employing PRG technology is still not widely available in the literature. By virtue of their pre-reacted glass ionomer fillers, these materials may behave differently when compared to composites based on zirconia and other fillers.

This study investigated the effects of FSL on the surface properties of two types of giomer restoratives. It was hypothesized that the effects of FSL on giomers will differ from those of conventional composites in view of their novel PRG technology.

MATERIALS AND METHODS

The materials investigated included two giomers (Beautifil II [BT] and Beautifil Flow Plus F00 [BF], Shofu, Kyoto, Japan), a direct hybrid composite (Filtek Supreme Z250 [ZT], 3M-ESPE, St Paul, MN, USA), and an indirect hybrid composite (Ceramage [CM], Shofu). The technical profiles of the various restoratives evaluated are shown in Table 1.

The materials were placed into customized square molds (5 mm × 5 mm × 2.5 mm) and covered with Mylar strips. A glass slide was placed and pressure was applied to remove any excess material. The specimens were then light-cured according to manufacturers' instructions (Table 2). Twenty-four specimens of each material were fabricated and randomly assigned into control (air) and treatment (distilled water, 50% ethanol solution, and 0.02 N citric acid) groups in clusters of six. The specimens were conditioned in individual vials containing the different FSL at 37°C for seven days.

After conditioning, each specimen was air-dried and subjected to a 3.0-mm line scan across the center of the specimen using a surface profilometer (Surftest, Mitutoyo Corp, Tokyo, Japan) with a probe diameter of 5 μm. Surface roughness value (Ra), which is the arithmetic average of the absolute values based on the vertical deviations of the roughness profile from the mean line calculated by the computer, was recorded. Hardness testing was then carried out using a digital microhardness tester (FM Series Microhardness Tester, Future Tech, Tokyo, Japan) to attain the Knoop hardness value (KHN). A load of 500 gf with a dwell time of 15 seconds was applied to the central top surface of each specimen *via* an indentator. Statistical analysis was done using one-way analysis of variance and *post hoc* Scheffe test at a significance level of 0.05. A Pearson correlation test was also conducted to determine the relationship between the surface hardness and roughness of the individual composites.

RESULTS

The means for KHN and Ra of the four composite restoratives are shown in Tables 3 and 4. Mean KHN

Table 1: *Chemical Composition of the Different Composite Restoratives*

Material, Lot No.	Composition	wt%	Filler Size, μm	Shade
Beautifil II, 051003	Bis-GMA (bisphenol A diglycidyl ether dimethacrylate)	7.5	0.01–4.0 0.8 (mean)	A2
	TEGDMA (triethyleneglycol dimethacrylate)	<5		
	Aluminofluoro-borosilicate glass, Al_2O_3	83.3		
	DL-camphorquinone			
Beautifil Flow Plus F00, 091013	Bis-GMA	15–25	0.01–4.0 0.8 (mean)	A2
	TEGDMA	12–14		
	Aluminofluoro-borosilicate glass, Al_2O_3	67.3		
	DL-camphorquinone			
Filtek Z250, N183958	Bis-GMA	1–10	0.01–3.5 0.6 (mean)	A2
	TEGMA	<5		
	Bis-EMA (bisphenol A polyethylene glycol diether dimethacrylate)	1–10		
	UDMA (diurethane dimethacrylate)	1–10		
	Zirconia/silica	82		
Ceramage, 071060	UDMA	Proprietary	Proprietary	A2B
	Zirconia/silica (amorphous)	73	Proprietary	

for the control group (air) ranged from 53.4 ± 3.4 (BF) to 89.5 ± 5.2 (ZT), while mean Ra values ranged from 0.014 ± 0.002 (ZT) to 0.032 ± 0.001 (BT). Results of statistical analyses are reflected in Tables 5 through 7.

The degree of softening by the different FSLs was material dependent. The greatest hardness was observed for the control group. Conditioning in ethanol generally resulted in the greatest softening. The giomers were also affected by citric acid. For the control group, ZT was significantly harder than all of the materials evaluated. No significant difference in KHN between BT and CM was noted, and BF was significantly softer than all of the other materials. When conditioned in distilled water, similar results were observed. After conditioning in citric acid, ZT and CM had comparable KHN. Both materials were significantly harder than BT and BF. KHN for CM and ZT were, again, not significantly different after conditioning in ethanol. The hardness values of CM were significantly greater than those of BT and BF, and ZT was significantly harder than that of BF.

Table 2: *Manufacturers' Curing Instructions*

Materials	Mode	Curing Unit	Curing Time, s
Beautifil II	Light cure	Elipar S10 (3M-ESPE)	20
Beautifil Flow Plus F00	Light cure	Elipar S10 (3M-ESPE)	20
Filtek Z250	Light cure	Elipar S10 (3M-ESPE)	20
Ceramage	Oven cure	Solidlite (Shofu)	240

Table 3: *Mean Knoop Hardness (KHN) for the Composites in the Different Food-simulating Liquids (FSLs)*[a]

Materials	Air	Distilled Water	Citric Acid	Ethanol
Beautifil II	77.6 (2.4)	63.8 (2.0)	41.4 (3.0)	42.0 (1.4)
Beautifil Flow Plus F00	53.4 (3.4)	40.5 (2.3)	37.4 (4.3)	32.6 (4.3)
Filtek Z250	89.5 (5.2)	71.7 (4.3)	74.9 (1.8)	50.7 (7.6)
Ceramage	75.4 (8.3)	59.3 (3.4)	69.9 (1.6)	56.3 (9.6)

[a] *Standard deviations (SDs) in parentheses.*

Table 4: *Mean Surface Roughness (Ra) for the Composites in the Different Food-simulating Liquids (FSLs)*[a]

Materials	Air	Distilled Water	Citric Acid	Ethanol
Beautifil II	0.032 (0.001)	0.025 (0.002)	0.083 (0.002)	0.033 (0.004)
Beautifil Flow Plus F00	0.020 (0.006)	0.040 (0.004)	0.057 (0.003)	0.041 (0.005)
Filtek Z250	0.014 (0.002)	0.017 (0.002)	0.017 (0.003)	0.022 (0.002)
Ceramage	0.025 (0.012)	0.027 (0.003)	0.029 (0.003)	0.031 (0.005)

[a] *Standard deviations (SDs) in parentheses.*

Regardless of conditioning medium, ZT had the smoothest surface among the materials evaluated. After conditioning in air and citric acid, BT was significantly rougher than ZT. BT also had higher Ra values than BF and CM after exposure to citric acid. After conditioning in distilled water and ethanol, BF was significantly rougher than the other materials. Significant negative correlations between hardness and roughness were observed for all composites except for CM. The correlation for ZT was strong.

Table 5: *Comparison of Mean Knoop Hardness (KHN) of the Composites in Different Food-simulating Liquids (FSLs)*

FSL	Materials
Air	ZT > BT, CM > BF
Distilled water	ZT > BT, CM > BF
Citric acid	ZT, CM > BT, BF
Ethanol	CM > BT, BF ZT > BF

Abbreviations: BT, Beautifil II; BF, Beautifil Flow Plus F00; ZT, Filtek Z250; CM, Ceramage.

DISCUSSION

Hardness is defined as the resistance of a material to permanent indentation.[17] Studies[18,19] have linked low hardness values to inferior surface wear resistance. Worn and roughened surfaces may be plaque retentive, allowing bacterial flora to flourish, leading to increased caries risk and periodontal inflammation.[20] The liquids used to condition the materials in this study are among those recommended in guidelines from the US Food and Drug Administration to be used as food simulators.[21,22] Aqueous ethanol-water solution simulates alcoholic liquids and is also the medium of choice for accelerated ageing of composite restorations, as its solubility parameter is comparable to that of bisphenol A diglycidyl ether dimethacrylate (bis-GMA).[2,23] The latter is one of the most commonly utilized resin monomers in composite resins. Citric acid (0.02 N) stimulates acid in foodstuffs such as vegetables, fruits, candy, and syrup, as well as certain beverages. Distilled water simulates the wet oral environment, while air serves

Table 6: *Comparison of Mean Surface Roughness (Ra) of the Composites in Different Food-simulating Liquids (FSLs)*

FSL	Materials
Air	BT > ZT
Distilled water	BF > CM, BT > ZT
Citric acid	BT > BF > CM > ZT
Ethanol	BF > BT, CM > ZT

Abbreviations: BT, Beautifil II; BF, Beautifil Flow Plus F00; ZT, Filtek Z250; CM, Ceramage.

Table 7: *Comparison of Mean Knoop Hardness (KHN) of the Composites in Different Food-simulating Liquids (FSLs)*

Materials	FSLs
Beautifil II	Air > distilled water > ethanol, citric acid
Beautifil Flow Plus F00	Air > distilled water > ethanol, air > citric acid
Filtek Z250	Air > citric acid, distilled water > ethanol
Ceramage	Air, citric acid > ethanol, air > distilled water

as the control medium. As the most significant changes in hardness have reportedly[3] occurred during the first week of exposure to FSL, a seven-day conditioning period was selected for this investigation.

Oxygen inhibits the surface polymerization of composite resins. The depth of inhibition in atmospheric air ranges between 25 μm and 105 μm and varies between composites.[24] The materials were adapted against a Mylar strip to minimize the oxygen inhibition layer[24] and were cured according to manufacturers' instructions. This method produced a consistent smooth surface across all specimens.[25] The latter ensures accurate hardness readings and prevents discrepancies associated with finishing/polishing procedures. The unpolished surface is, however, matrix-rich and may result in a greater degree of softening. It is therefore less characteristic of the bulk material.[26,27] As the materials were not subject to any mechanical forces, any observed changes in hardness and surface roughness can be attributed to exposure to the FSL.

BF is marketed as a flowable hybrid composite to be used as a restorative, base, and liner. "Flowability" is mainly achieved by its lower filler loading of approximately 67.3% weight. The filler loading values of the other materials investigated were higher and ranged from 73% to 83.3% weight. A general trend is observed between increased filler loading and improved hardness, compressive strength, and stiffness.[28,29] BF had the lowest hardness values in the control medium and was significantly softer than ZT and CM for all test mediums. The lower filler fraction in BF could have played a significant role in its lower hardness as compared to the other composites. The higher hardness values of ZT and CM can also be attributed to the use of zirconia-silicate fillers over alumino-fluoro-borosilicate glass fillers in the giomers. In addition, CM was oven-cured (Solidlite, Shofu) under heat and high-voltage (600-W) light.[30,31]

The giomers investigated were found to be significantly degraded by citric acid. This may be attributed to the greater susceptibility of fluorosilicate glass fillers to degradation by weak acids.[32] The giomer materials and ZT contain bis-GMA as part of their resin matrix. Ethanol has a solubility index similar to that of bis-GMA,[2,23] which enhances its disintegration. In addition, triethyleneglycol dimethacrylate (TEGDMA) has been shown[33] to have the greatest amount of liquid sorption in 50% ethanol-water solution when compared to bis-GMA, bisphenol A polyethylene glycol diether dimethacrylate, and diurethane dimethacrylate.

Liquid uptake will leach unreacted components from the resin matrix, causing a reduction in mechanical properties. Diffusion of solvent into the resin network interferes with bonding, separates the chains, and interrupts the arrangement of the polymer chains in the compound, causing significant reduction of the material's physical properties.[33] BF, which contained the highest percentage weight of bis-GMA and TEGDMA, was observed to be most affected by ethanol as compared to the other three materials. Distilled water exerts a similar degradative effect through liquid sorption and dissolution of the resin matrix.[33]

Roughness parameters are dependent on several factors, such as filler particle size, percentage of surface area occupied by filler particles, hardness, degree of polymer conversion to resin matrix, and filler-matrix interaction.[34] For all FSLs, the Ra

values of the giomer composites were significantly higher than that of ZT. The greater surface roughness corresponded to the larger average particle sizes (0.8 μm) of BT and BF, as compared to the smaller average particle sizes (0.6 μm) found in ZT. The fillers exposed on the surface of giomers after surface degradation by the FSL are consequently coarser, leading to a rougher surface profile. All roughness values of the materials were, however, below the threshold surface roughness for bacterial retention (Ra=0.2 μm).[20] The values may have little bearing in a clinical setting, as an initial unpolished surface is uncommon. The negative correlation between roughness and hardness was significant for all materials except CM. It was strong for ZT (r=−0.746). The surface roughness of giomers was therefore weakly associated with their surface hardness. A softer surface corresponded to a rougher giomer surface.

CONCLUSIONS

Within the limitations of this *in vitro* study,

1. Giomers, like other direct and indirect composites, were degraded by FSLs.
2. Hardness of giomers was significantly affected by citric acid and ethanol.
3. Roughness of giomers was significantly affected by citric acid.
4. With the exception of the indirect composite, significant and negative correlations were observed between hardness and roughness.

Conflict of Interest

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

(Accepted 20 December 2011)

REFERENCES

1. Yap AU, Tan SH, Wee SS, Lee CW, Lim EL, & Zeng KY (2001) Chemical degradation of composite restoratives *Journal of Oral Rehabilitation* **28(11)** 1015-1021.
2. McKinney JE, & Wu W (1985) Chemical softening and wear of dental composites *Journal of Dental Research* **64(11)** 1326-1331.
3. Kao EC (1989) Influence of food-simulating solvents on resin composites and glass-ionomer restorative cement *Dental Materials* **5(3)** 201-208.
4. Yap AU, Low JS, & Ong LF (2000) Effect of food-simulating liquids on surface characteristics of composite and polyacid-modified composite restoratives *Operative Dentistry* **25(3)** 170-176.
5. Gordan VV, Mondragon E, Watson RE, Garvan C, & Mjör IA (2007) A clinical evaluation of a self-etching primer and a giomer restorative material: Results at eight years *Journal of the American Dental Association* **138(5)** 621-627.
6. Matis BA, Cochran MJ, Carlson TJ, Guba C, & Eckert GJ (2004) A three-year clinical evaluation of two dentin bonding agents *Journal of the American Dental Association* **135(4)** 451-457.
7. Sulaiman E, Yeo YM, & Chong YT (2007) The flexural strengths of five commercially available tooth-coloured restorative materials *Annals of Dentistry, University of Malaya* **14(1)** 39-45.
8. Yamazaki T, Schricker SR, Brantley WA, Culbertson BM, & Johnston W (2006) Viscoelastic behavior and fracture toughness of six glass-ionomer cements *Journal Prosthetic Dentistry* **96(4)** 266-272.
9. Yap AU, Wang X, Wu X, & Chung SM (2004) Comparative hardness and modulus of tooth-colored restoratives: A depth-sensing microindentation study *Biomaterials* **25(11)** 2179-2185.
10. Yap AUJ, & Mok BYY (2002) Surface finish of a new hybrid aesthetic restorative material *Operative Dentistry* **27(2)** 161-166.
11. Naoum S, Ellakwa A, Martin F, & Swain M (2011) Fluoride release, recharge and mechanical property stability of various fluoride-containing resin composites *Operative Dentistry* **36(4)** 422-432.
12. Dionysopoulos P, Kotsanos N, Koliniotou-Koubia E, & Tolidis K (2003) Inhibition of demineralization in vitro around fluoride releasing materials *Journal of Oral Rehabilitation* **30(12)** 1216-1222.
13. Gonzalez EH, Yap AU, & Hsu SC (2004) Demineralization inhibition of direct tooth-colored restorative materials *Operative Dentistry* **29(5)** 578-585.
14. Dionysopoulos P, Kotsanos N, Papadogiannis Y, & Konstantinidis A (1998) Artificial caries formation around fluoride-releasing restorations in roots *Journal of Oral Rehabilitation* **25(11)** 814-820.
15. Francci C, Deaton TG, Arnold RR, Swift EJ Jr, Perdigão J, & Bawden JW (1999) Fluoride release from restorative materials and its effects on dentin demineralization *Journal of Dental Research* **78(10)** 1647-1654.
16. Donly KJ, & Grandgenett C (1998) Dentin demineralization inhibition at restoration margins of Vitremer, Dyract and Compoglass *American Journal of Dentistry* **11(5)** 245-248.
17. Anusavice KJ (2007) Mechanical properties of dental materials. In: Anusavice KJ (ed) *Phillips' Science of Dental Materials,* 11th edition WB Saunders Company, Philadelphia, Pa 96-97.
18. Say EC, Civelek A, Nobecourt A, Ersoy M, & Guleryuz C (2003) Wear and microhardness of different resin composite materials *Operative Dentistry* **28(5)** 628-634.
19. Kawai K, Iwami Y, & Ebisu S (1998) Effect of resin monomer composition on toothbrush wear resistance *Journal of Oral Rehabilitation* **25(4)** 264-268.
20. Bollen CM, Lambrechts P, & Quirynen M (1997) Comparison of surface roughness of oral hard materials

to the threshold surface roughness for bacterial plaque retention: A review of the literature *Dental Materials* **13(4)** 258-269.

21. Food and Drug Administration, Bureau of Foods (1976) *Guidelines for Chemistry and Technology* Department of Health, Education and Welfare, Washington, DC 11.
22. Food and Drug Administration, Division of Food Chemistry and Technology (1988) *Recommendations for Chemistry Data for Indirect Food Additives Petitions* Department of Health, Education and Welfare, Washington, DC 9.
23. Wu W, & McKinney JE (1982) Influences of chemicals on wear of dental composites *Journal of Dental Research* **61(10)** 1180-1183.
24. Finger W, & Jorgensen DK (1976) Inhibition of polymerization by oxygen in composite filling materials and enamel sealers *Schweizerische Monatsschrift Fur Zahnheilkunde* **86(8)** 812-824.
25. Stanford WB, Fan PL, Wozniak WT, & Stanford JW (1985) Effect of finishing on color and gloss of composites with different fillers *Journal of American Dental Association* **110(2)** 211-213.
26. Hachiya Y, Iwaku M, Hosoda H, & Fusayama T (1984) Relation of finish to discoloration of composite resins *Journal of Prosthetic Dentistry* **52(6)** 811-814.
27. Shintani H, Satou J, Satou N, Hayashihara H, & Inoue T (1984) Effects of various finishing methods on staining and accumulation of Streptococcus mutans HS-6 on composite resins *Dental Materials* **1(6)** 225-227.
28. St Germain H, Swartz ML, Phillips RW, Moore BK, & Roberts TA (1985) Properties of microfilled composite resins as influenced by filler content *Journal of Dental Research* **64(2)** 155-160.
29. Li Y, Swartz ML, Phillips RW, Moore BK, & Roberts TA (1985) Effect of filler content and size on properties of composites *Journal of Dental Research* **64(12)** 1396-1401.
30. Mandikos MN, McGivney GP, Davis E, Bush PJ, & Carter JM (2001) A comparison of the wear resistance and hardness of indirect composite resins *Journal of Prosthetic Dentistry* **85(4)** 386-395.
31. Gregory WA, Berry S, Duke E, & Dennison JB (1992) Physical properties and repair bond strength of direct and indirect composite resins *Journal of Prosthetic Dentistry* **68(3)** 406-411.
32. Roulet JF, & Wälti C (1984) Influence of oral fluid on composite resins and glass-ionomer cement *Journal of Prosthetic Dentistry* **52(2)** 182-189.
33. Sideridou ID, & Karabela MM (2011) Sorption of water, ethanol or ethanol/water solutions by light-cured dental dimethacrylate resins *Dental Materials* **27(10)** 1003-1010.
34. Marghalani HY (2010) Effect of filler particles on surface roughness of experimental composite series *Journal of Applied Oral Science* **18(1)** 59-67.

Departments

Faculty Positions

CLINICAL ASSISTANT/ASSOCIATE PROFESSOR
University of Illinois at Chicago
Restorative Dentistry—Advanced Operative/ General Dentistry

The Department of Restorative Dentistry at the University of Illinois at Chicago under the leadership of Dr. Stephen Campbell is seeking applications for two full-time faculty positions (non-tenure track) beginning July 1, 2013 at the Clinical Assistant/ Associate Professor levels. Future tenure track options exist for qualified/successful candidates. Responsibilities include preclinical and clinical instruction in all aspects of the restorative sciences. Qualifications include a DDS/DMD degree, and advanced training in operative dentistry or general dentistry (board eligibility/certification desirable where appropriate, but not required). Prior teaching experience is desirable. Candidates must be eligible for licensure in Illinois. Candidates with training and/or experience in research are preferred.

For fullest consideration, apply online at https://jobs.uic.edu/job-search/job-details?jobID=21259 and submit a cover letter, CV and names of three (3) references by November 09, 2012. Salary and academic rank commensurate with experience and qualifications.

For any questions regarding this position, please contact Ms. Anna Panova, UIC College of Dentistry (M/C 555), 801 S. Paulina Street, Chicago, IL 60612 or e-mail annap22@uic.edu.

UIC is AA/EOE.

GEORGIA HEALTHSCIENCES UNIVERSITY
College of Dental Medicine Instructor/ Assistant/Associate Professor
Oral Rehabilitation—Prosthodontics/ Restorative Dentistry
Req #'s 5950 and 5792

Two full-time, tenure/non-tenure track faculty positions. Teaching responsibilities include participation in clinical and preclinical courses in prosthodontics, operative dentistry, and/or restorative dentistry at the pre-doctoral and post-doctoral level. Preference will be given to candidates with significant clinical experience, post-graduate training in prosthodontics/operative dentistry/general dentistry or prior experience in dental education. Participation in faculty practice and research is expected. Applicants must be a graduate of an ADA accredited dental school or 2 year postgraduate training program and be licensed in a state or eligible for licensure by the Georgia Board of Dentistry. Salary and academic rank are commensurate with qualifications. AA/ EEO/Equal Access/ADA Employer. Interested candidates should apply for this position on-line at www.georgiahealth.edu/faculty jobs/, the requisition number provided above, and the department name. For further information, please email mmyers@georgiahealth.edu

Application Deadline: Until Filled

University of Maryland—Operative Dentistry

The Department of Endodontics, Prosthodontics and Operative Dentistry, University of Maryland is seeking candidates for two full time faculty positions at the rank of Assistant, Associate or Full Professor in Operative Dentistry. Responsibilities include didactic and clinical teaching in Cariology, Operative Dentistry, Dental Anatomy and Occlusion, and Esthetic Dentistry. Demonstrated record of successful research, involvement in leadership in dentistry at the local or national levels, and other scholarly activities are highly desirable. Requirements for the position include a DDS/ DMD degree, or foreign equivalent, and eligibility for a Maryland dental license. Preference will be given to candidates with Masters or Ph.D. degrees, expertise in cariology and caries risk assessment, funded research, demonstrated innovative approaches to curricular development and/or continuing education and productivity in other scholarly activities. An opportunity for intramural practice is available and candidates are encouraged to participate. Appointment track, salary and academic rank will be commensurate with qualifications and experience. The positions will be available until filled. Qualified applicants should send a letter of interest, curriculum vitae and names and addresses of three references to Dr. Ashraf Fouad, Chair, Department of Endodontics, Prosthodontics and Operative Dentistry, School of Dentistry, University of Maryland, 650 West

Baltimore Street, Room Rm. 4221, Baltimore, MD 21201-1586, or electronic versions of the above to afouad@umaryland.edu

The University of Maryland is an AA/EEO/ADA employer and has a strong commitment to the principle of diversity in all areas.

Online Only

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

One-Year Clinical Evaluation of Composite Restorations in Posterior Teeth: Effect of Adhesive Systems

RH Sundfeld • RS Scatolin • FG Oliveira • LS Machado • RS Alexandre • MLMM Sundefeld

Clinical Relevance:

Class I composite restorations, placed using either a total etch or a self-etch bonding system, showed equally satisfactory clinical performance after one year.

DOI: 10.2341/10-375-C

The Bond of Different Post Materials to a Resin Composite Cement and a Resin Composite Core Material

D Stewardson • A Shortall • P Marquis

Clinical Relevance:

When selecting endodontic post materials, clinicians should be aware that fiber-reinforced composite (FRC) posts do not achieve a chemical bond to resin composites. The surfaces of different FRC posts need to be roughened by grit blasting with aluminum oxide particles to optimize micromechanical bonding.

DOI: 10.2341/11-409-L

Staff Letter

Dear Members of our Parent Academies,

An agreement was reached early this year to make some changes to the subscriptions of the Academy Members. The new agreement specifies that Academy Members will receive their copy of the journal online only starting with issue 1 of volume 38 (2013). For those members interested in receiving a paper copy, it is available for an additional $40.00 paid via your dues to your academy. For those of you who belong to more than one academy, you can send a request to our office to have your other dues pay for the paper upgrade. Upon confirmation of dues receipt from the second academy, we will begin sending the paper copies you requested.

If you run into any technical issues, please contact our support desk at helpdesk@allenpress.com, or for questions on this new policy, please contact your Academy's secretary.

Happy reading,

Operative Dentistry Office Staff

©Operative Dentistry, 2012, **38-1,** E1-E8

One-Year Clinical Evaluation of Composite Restorations in Posterior Teeth: Effect of Adhesive Systems

RH Sundfeld • RS Scatolin • FG Oliveira
LS Machado • RS Alexandre • MLMM Sundefeld

Clinical Relevance

Class I composite restorations, placed using either a total etch or a self-etch bonding system, showed equally satisfactory clinical performance after one year.

Renato Herman Sundfeld, PhD, MS, DDS, Araçatuba Dental School, Restaorative Dentistry, Rua José Bonifácio, 1193, Araçatuba, 16015-050, Brazil

Renata Siqueira Scatolin, DDS, MS candidate, Dental School of Ribeirão Preto—USP, Restorative Dentistry, Rua José Bonifácio 1193, Araçatuba, São Paulo, Brazil

Fernanda Garcia Oliveira, DDS, MS, Araçatuba Dental School—UNESP, Department of Restorative Dentistry, Rua José Bonifácio, 1193, Araçatuba, SP, Brazil

Lucas Silveira Machado, DDS, MS, Araçatuba Dental School—UNESP, Department of Restorative Dentistry, Rua José Bonifácio, 1193, Araçatuba, Brazil

Rodrigo Sversut Alexandre, DDS, MS, PhD, collaborator research, Araçatuba Dental School, Restorative Dentistry, Rua José Bonifácio, 1193, Araçatuba, Brazil

Maria Lucia Marçal Mazza Sundefeld, DDS, MS, PhD, Araçatuba Dental School—UNESP, Department of Biostatistics, Rua José Bonifácio, 1193, Araçatuba, SP, Brazil

DOI: 10.2341/10-375-C

SUMMARY

This clinical study assessed the performance of posterior composite resins applied with the Adper™ Single Bond Plus (SB) and Adper™ Scotchbond SE (SE) adhesive systems and Filtek™ Supreme Plus composite resin, using modified US Public Health Service criteria. A total of 97 restorations were placed in posterior teeth by two calibrated operators. Application of the materials followed manufacturers' instructions. The restorations were evaluated by two examiners at baseline and after one year. Statistical analyses were conducted using the proportion test at a significance level of 5% ($p<0.05$). All the restorations evaluated (ie, 100%) received an alpha rating for the criteria of marginal discoloration and marginal integrity at baseline. At one year, for marginal discoloration, 64.6% of SB and 61.2% of SE

received an alpha rating. For marginal integrity, 72.9% of SB and 77.6% of SE received an alpha rating. The other restorations received bravo ratings for both criteria. None of the teeth that received the restorative systems presented caries lesions around the restorations. A total of eight teeth presented postoperative sensitivity one week after baseline, five with SB and three with SE; the symptom had disappeared one year later. One year later, composite resin restorations using either adhesive system showed satisfactory clinical performance.

INTRODUCTION

The introduction of resin-based adhesive materials was revolutionary for the restoration of anterior teeth, and their use was extended to posterior teeth; however, initial clinical evaluations of resin-based restorations in posterior teeth showed shortcomings in this restorative procedure compared with restorations performed with dental amalgam, particularly in regard to marginal discoloration, wear resistance, and incidence of secondary caries,[1,2] factors possibly related to the restorative technique employed and the properties of the adhesive systems in relation to dental tissues, especially dentin tissue.

Since then, researchers and manufacturers have developed and analyzed adhesive systems and techniques for good marginal sealing in dental restorations. In view of this concern, various types of adhesive systems have been developed, including the total etching adhesive system[3] and self-etching adhesive systems.[3,4]

Total etching adhesive systems using phosphoric acid etching have shown excellent clinical performance in terms of durability and bond strength to enamel,[5] but these results are not commonly observed with self-etching adhesive systems.[6] However, when a self-etching adhesive is applied to dentin tissue, the smear layer is not washed out.[7] Thus, pulpal pressure is not modified, explaining the low sensitivity found in a longitudinal clinical study by Gordan and Mjor[8] in 2002 in which resin-based restorative material and self-etching primer in posterior restorations did not result in significant short- or long-term postoperative sensitivity.[9,10]

Although several laboratory studies of adhesive systems have been described in the literature, clinical studies are necessary to determine whether problems identified in the laboratory are clinically significant.

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

Material	Composition
Adper™ Single Bond Plus	Bis-GMA; HEMA; copolymer of polyalkenoic acid, ethanol, water, and photoinitiator
Adper™ Scotchbond SE Plus	Bottle A (primer): water, HEMA, surfactant, and dye pink.
	Bottle B (bond): UDMA, TEGDMA, TMPTMA, HEMA, MHP zircon nanofiller, and canphoroquinona
Filtek Supreme Plus	Bis-GMA, BIS-EMA, UDMA, TEGDMA, and inorganic filler

The aim of this clinical study was to evaluate posterior nanofilled composite resin restorations using a two-step total etching or two-step self-etching adhesive system at baseline (one week later) and one year later. The response variables were marginal discoloration, marginal adaptation, caries lesions, and postoperative sensitivity.

MATERIALS AND METHODS

Experimental Design

A total of 97 class I restorations were placed in 15 patients (10 women and 5 men) of the Dental School of Araçatuba—UNESP with good oral hygiene and ages ranging between 13 and 21 years. Each patient had at least one restoration done with one of the adhesive systems employed in this study.

The selected teeth were restored because they presented primary or secondary caries lesions or restorations with amalgam or composite resin that needed replacing either because of recurrent caries lesions or fractures or even for esthetic reasons; all teeth were in occlusion. The clinical procedures were performed by two calibrated operators. The study was approved by the Ethics Committee of the Institution (process no. 2008-01502). The procedures were explained to the subjects, who gave their written consent to participate.

A nanofilled composite resin, Filtek Supreme Plus (FS;3M ESPE Dental Products, St Paul, MN, USA), in association with a two-step total etching adhesive system, Adper Single Bond Plus (SB), and a two-step self-etching adhesive system, Adper Scotchbond SE Plus (SE; 3M/ESPE; Table 1), were used in this

study. The factors were adhesive systems on two levels (two-step total etching and two-step self-etching), and the response variables were marginal discoloration, marginal adaptation, caries lesions, and postoperative sensitivity. Randomization was used to perform restorations, and there was always present in each patient at least one restoration of each adhesive used.

Operative Procedures

All restoration procedures were performed after anesthesia and dental prophylaxis with pumice and water. The cavity was opened or the existing restoration removed using a cylindrical diamond bur number 1092 (KGSorensen, Industria e Comercio Ltda, São Paulo, Brazil) mounted in a high-speed water-cooled hand piece. In the cave-surface enamel, beveling was not conducted. When carious lesions were found, they were removed with hand instruments and low-speed spherical drills in sizes compatible with the sizes of the lesions. A rubber dam was used. Only deep cavities received a thin layer of resin-modified glass ionomer Fuji II LC (GC Corp, Tokyo, Japan) as a liner, following the manufacturer's instructions.

All cavities were restored with a nanofilled resin composite, Filtek Supreme Plus (3M ESPE). For 48 cavities, a total etching adhesive system (SB) was used, and for 49 cavities, a two-step self-etching adhesive system (SE) was used. The cavities were restored in a randomized way. Table 1 shows the distribution of restorations among selected patients in the maxillary and mandibular arches. The materials were applied according to the manufacturers' instructions as follows.

Adper Single Bond Plus—The entire cavity was etched with 35% Scotch Etchant (3M ESPE) for 15 seconds in dentin tissue and 60 seconds in the enamel, rinsed with water spray, and air-dried. After drying, the enamel surface was completely dry, and the dentin had a moistened appearance . The adhesive system was applied with a microbrush and light cured for 20 seconds with a halogen light source, an Ultralux (Dabi Atlante, Ribeirão Preto, Brazil) with a power of 450 mW/cm^2.

Adper Scotchbond SE Plus—The cavity preparation was rinsed with water spray and air-dried. The self-etch adhesive system (SE) was composed of liquids designated A and B. Liquid A was initially applied to the cavity with a microbrush, immediately followed by liquid B, which was applied to the cavity for 20 seconds under moderate pressure. The adhesive was air-dried for 10 seconds. After that, a second layer of liquid B was applied, and light polymerization was performed for 10 seconds using an Ultralux (Dabi Atlante) halogen light appliance with a power of 450 mW/cm^2.

Filtek Supreme Plus—Following an incremental technique, the composite materials were applied using the oblique layering technique, with each layer not exceeding 2 mm. Each increment was light polymerized separately for 40 seconds using an Ultralux light-curing unit with a power of 450 mW/cm^2 Ultralux (Dabi Atlante).

The occlusal adjustment was performed with carbon paper (Accufilm: MDF for Parkell). The restorations were finished with diamond point number 1190 (KGSorensen, Indústria e Comércio Ltda), followed by the application of Enhance finishing points (Dentsply, Indústria e Comércio Ltda, Rio de Janeiro, Brazil).

Clinical and Statistical Analysis

Clinical analysis of the restorations was performed at baseline (one week after the procedure) and after one year in a duly illuminated operative field. Two duly calibrated independent clinicians not involved in the original placement evaluated the restorations after their placement at baseline and after one year, using an exploratory probe number 5 and a buccal mirror. In the case of disagreement over assessments, the examiners had to reach a consensus, considering the factors of marginal adaptation, marginal discoloration, the presence of marginal caries lesions, and postoperative sensitivity, using modified USPHS criteria[11] (Table 2).

The results were analyzed using the kappa test to evaluate the degree of reproducibility between the two examiners. Statistical analysis was conducted using the proportion test at a significance level of 5% ($p<0.05$).

RESULTS

The kappa test reported 80% concordance between the two examiners. All the restorations evaluated received an alpha rating for the criteria of marginal discoloration and marginal integrity at baseline. The results of clinical evaluations one year after the baseline exam are shown in Figures 1 and 2 for marginal discoloration and marginal integrity, respectively. After one year, for marginal discoloration, 64.6% of SB received an alpha rating, compared with 61.2% for SE adhesive systems. For marginal integrity, 72.9% of SB and 77.6% of SE adhesive

Table 2: *Modified US Public Health Service[14] Criteria*

Category	Code	Criteria
Marginal discoloration	ALPHA/A	Absence of marginal color alteration of restoration
	BRAVO/B	Alteration of marginal color of restoration, in a small extension
	CHARLIE/C	Alteration of marginal color of restoration, in a large extension
	DELTA/D	Alteration of marginal color of restoration, in the full extension
Caries lesions[a]	ALPHA/A	Absence of caries lesion at the restoration margin
	BRAVO/B	Presence of caries lesion at the restoration margin
Postoperative sensitivity one week after restoration (baseline)	ALPHA/A	Absence of sensitivity
	BRAVO/B	Presence of sensitivity
Postoperative sensitivity one year after restoration	ALPHA/A	Absence of sensitivity
	BRAVO/B	Presence of sensitivity
Marginal adaptation	ALPHA/A	Absence of gap at restoration margin; the explorer does not catch at the tooth/restoration interface
	BRAVO/B	Presence of gap at the restoration margin, with retention of the explorer at the tooth/restoration interface, but without dentinal exposure
	CHARLIE/C	Presence of gap at the restoration margin, with retention of the explorer at the tooth/restoration interface, but with dentinal exposure
	DELTA/D	Presence of gap at the restoration margin, with retention of the explorer at the tooth/restoration interface, with dentinal exposure and the restoration fractured and/or showing mobility

[a] *A region of the restoration margin was considered carious if the explorer caught or resisted removal, after moderate pressure, and if one of the following factors was observed: presence of softened dental tissue or marginal white stain lesion with evidence of demineralization.*

systems received an alpha rating. The remaining restorations were cataloged with a bravo rating for both criteria.

The proportion test revealed no statistically significant differences between SB and SE adhesive systems for marginal discoloration (p=0.732) or marginal integrity (p=0.597) after one year.

Postoperative sensitivity was observed in eight restored teeth (five SB and three SE) one week later. None of the restorations were cataloged with charlie or delta rating criteria or lost during the evaluation period of one year, and no sensitivity was found in the one-year follow-up. No verified secondary caries were found around the restorations one year later.

DISCUSSION

Adhesive systems are constantly being improved; however, their development has been so rapid that long-term clinical data on specific products are rarely available because of the regular introduction of "improved" versions,[9] justifying the need for laboratory research and longitudinal clinical studies.

In this study, we evaluated the clinical performance of posterior composite resin restorations made with a two-step total etching adhesive system

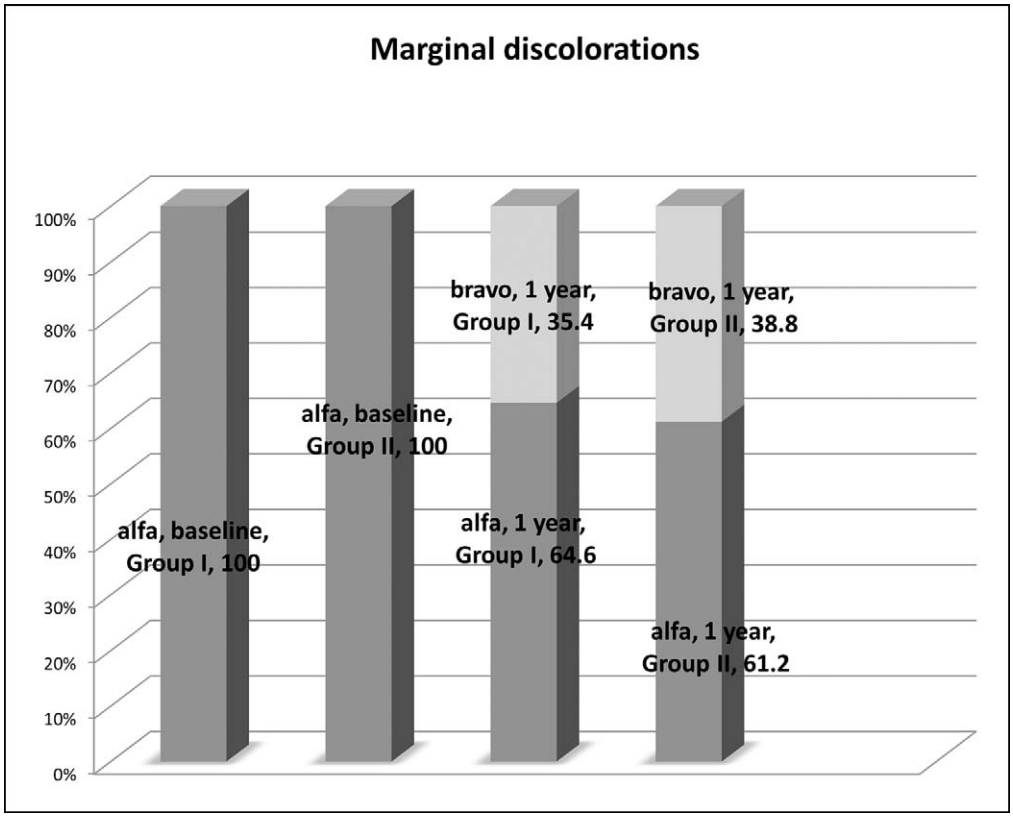


Figure 1. *Scores for marginal discoloration of the clinical evaluation of posterior composite restorations carried out with Filtek™ Supreme Plus and the SB and SE adhesive systems*

(SB) or a two-step self-etching adhesive system (SE) over a period of 12 months. It was observed that no restorations were lost, and there were no significant differences between restorations applied with either adhesive system in the variables of secondary caries incidence, postoperative sensitivity, marginal discoloration, or marginal adaptation. Similar clinical observations have been made in other clinical studies.[9–13]

It has been scientifically established that etching on the enamel surface followed by the application of an adhesive system is able to penetrate the dental surface,[6,14] but this has not been observed with self-etching adhesive systems.[6,14] However, our results showed similar marginal discoloration and integrity among the restorations applied with SE or SB, probably as a result of adequate incremental technique, avoiding simultaneous bonding of the composite to the opposite walls, reducing cavity configuration factors,[15] low polymerization contraction by nanofilled composite resin, and adequate light on the polymerization unit.[16–18] It is worthwhile to consider that, after polymerization, the direct class I resin composite restorations present a three-dimensional cavity configuration that favors the retention of restorative material, unlike those of direct class IV and V resin composite restoration cavities such that their retention is maintained almost exclusively by the action of the adhesive system used.[19–22]

Furthermore, we must consider that adequate solvent evaporation, the application according to the manufacturer's recommendations, and the use of a rubber dam during the restorative procedure improved the performance of the adhesive.[23–25]

After one year, the majority of restorations applied with either adhesive system, SB or SE, and the nanofilled composite resin presented with an alpha rating, with 64.6% and 61.2% marginal discoloration and 72.9% and 77.6% marginal integrity for SB and SE adhesive systems, respectively. The other restorations were given bravo ratings. When marginal discoloration was observed in some restorations, it was slight.

Whereas during a rigorous adhesive restorative procedure in posterior teeth a nonpurposeful excess of composite resin material can be left beyond the

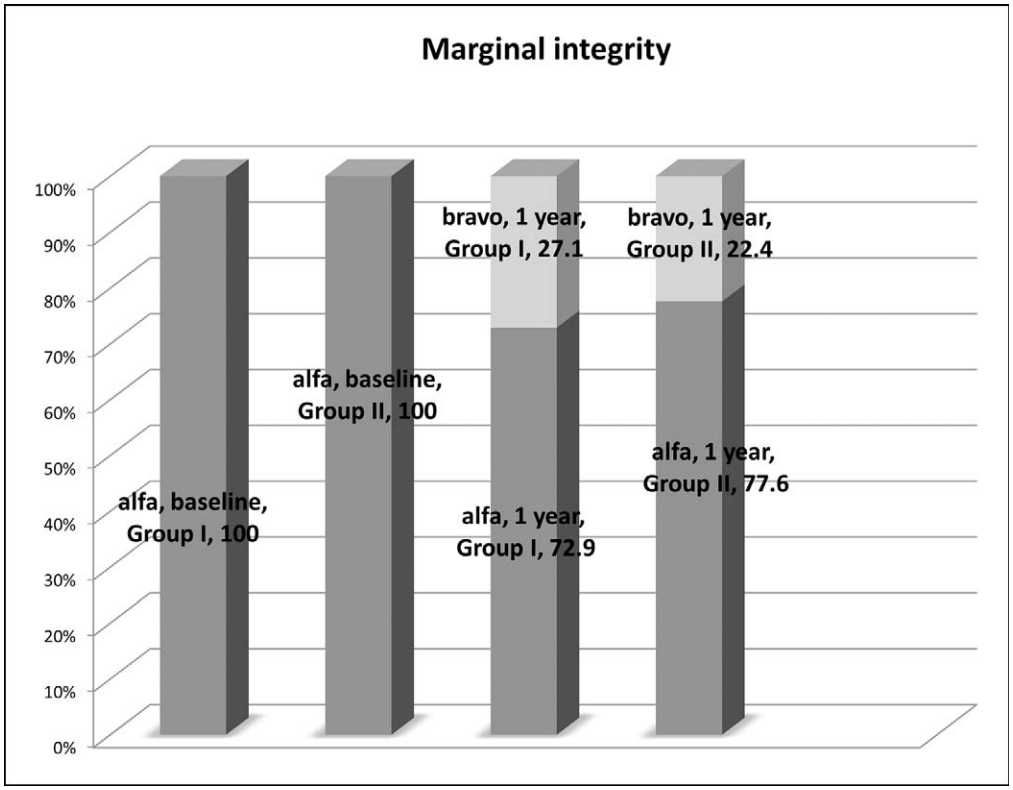


Figure 2. *Scores for marginal integrity of the clinical evaluation of posterior composite restorations carried out with Filtek™ Supreme Plus and the SB and SE adhesive systems*

cavity margin, considered "overfilling,"[26] this undesired effect, even when slight, can contribute to small marginal fractures, leading to bravo scores for marginal integrity. It is worth noting, however, that cataloged restorations with bravo scores are still considered clinically acceptable because of the rigorous criteria used, precluding the need for replacement of these restorations.[27]

Postoperative sensitivity must be considered an important factor in the success of restorations; in this study, 10.4% of SB restorations and 6.1% of SE restorations showed slight postoperative sensitivity one week after the restorations (baseline exam), but no sensitivity was found in the one-year follow-up. Possibly, this postoperative sensitivity verified only during the baseline exam is related to the dimensions of the cavity preparations, the enamel marginal sealing, and the occlusal adjustments accomplished after treatment.[25] However, the sensitivity was not present after one year.

Marginal imperfections and secondary caries lesions in posterior composite restorations are some of the factors that predict their need for replacement.[28] However, this study found no restorations with secondary caries lesions one year later for either adhesive system employed. Observations consistent with those of Bekes[27] in 2007 and Akimoto[29] in 2007 reinforced the finding that marginal imperfections, such as those found in this study and receiving a bravo rating, do not necessarily lead to secondary caries lesions.[11]

It is important to emphasize that this study evaluated only direct class I resin composite restorations, where all the margins were in enamel, without dentinal exposure at the tooth/restoration interface; dentin substrate is more challenging for any composite bonding system, so our results are not applicable to restorations with borders in the dentin or root. Assessment in future years will be necessary to gain useful information regarding material performance.

Early clinical results are promising, but long-term assessment is required for more precise conclusions. Thus, we must consider that the success of bonded restorations is determined mainly by the excellence of the technique employed, with appropriate application of the adhesive system and adherence to the steps for insertion, polymerization, finishing, and polishing of these restorations.[30] Likewise, we cannot overlook the fact that the collaboration of

the patients will certainly significantly influence the longevity of their restorations.[11]

CONCLUSION

This clinical evaluation of composite resin restorations in posterior teeth performed with a nanofilled composite resin and a one-bottle total etching adhesive system or self-etching adhesive system presented good clinical performance after one year.

ACKNOWLEDGEMENTS

This study was supported by the São Paulo State Research Foundation (FAPESP) grant # 2008/55738–6.

CONFLICT OF INTEREST

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

(Accepted 14 November 2011)

REFERENCES

1. Osborne JW, Gale EN, & Ferguson GW (1973) One-year and two-year clinical evaluation of composite resin x amalgam. *Journal of Prosthetic Dentistry* **30(5)** 795-800.
2. Phillips RW, Avery DR, Mehra R, Swartz ML, & McCune RJ (1973) Observations on a composite resin for class II restorations: Three-year report *Journal of Prosthetic Dentistry* **30(6)** 891-897.
3. Tay FR, & Pashley DH (2003) Have dentin adhesives become too hydrophilic? *Journal of the Canadian Dental Association* **69(11)** 726-731.
4. Perdigao J (2007) New developments in dental adhesion *Dental Clinics of North America* **51(2)** 333-357, viii.
5. Ramires-Romito AC, Reis A, Loguercio AD, Hipólito VD, de Goes MF, Singer JM, Grande RH. (2007) Microtensile bond strength of sealant and adhesive systems applied to occlusal primary enamel. *American Journal of Dentistry* **20(2)** 114-120.
6. Sundfeld RH, de Oliveira CH, da Silva AM, Briso AL, & Sundfeld ML (2005) Resin tag length of one-step and self-etching adhesives bonded to unground enamel *Bulletin of Tokyo Dental College* **46(3)** 43-49.
7. Tay FR, & Pashley DH (2001) Aggressiveness of contemporary self-etching systems. I: Depth of penetration beyond dentin smear layers *Dental Materials* **17(4)** 296-308.
8. Gordan VV, & Mjor IA (2002) Short- and long-term clinical evaluation of post-operative sensitivity of a new resin-based restorative material and self-etching primer *Operative Dentistry* **27(6)** 543-548.
9. Celik C, Arhun N, & Yamanel K (2010) Clinical evaluation of resin-based composites in posterior restorations: 12-month results. *European Journal of Dentistry* **4(1)** 57-65.
10. Swift EJ Jr, Ritter AV, Heymann HO, Sturdevant JR, & Wilder AD Jr (2008) 36-month clinical evaluation of two adhesives and microhybrid resin composites in class I restorations. *American Journal of Dentistry* **21(3)** 148-152.
11. Rezwani-Kaminski T, Kamann W, & Gaengler P (2002) Secondary caries susceptibility of teeth with long-term performing composite restorations *Journal of Oral Rehabilitation* **29(12)** 1131-1138.
12. Cvar JF, & Ryge G (2005) Reprint of criteria for the clinical evaluation of dental restorative materials. 1971 *Clinical Oral Investigations* **9(4)** 215-232.
13. Gaengler P, Hoyer I, & Montag R (2001) Clinical evaluation of posterior composite restorations: The 10-year report *Journal of Adhesive Dentistry* **3(2)** 185-194.
14. De Alexandre RS, Sundfeld RH, Giannini M, & Lovadino JR (2008) The influence of temperature of three adhesive systems on bonding to ground enamel *Operative Dentistry* **33(3)** 259-268.
15. Carvalho RM, Pereira JC, Yoshiyama M, & Pashley DH (1996) A review of polymerization contraction: The influence of stress development versus stress relief *Operative Dentistry* **21(1)** 17-24.
16. Correr AB, Sinhoreti MA, Correr Sobrinho L, Tango RN, Schneider LF, & Consani S (2005) Effect of the increase of energy density on Knoop hardness of dental composites light-cured by conventional QTH, LED and xenon plasma arc *Brazilian Dental Journal* **16(3)** 218-224.
17. Ferracane JL (2006) Hygroscopic and hydrolytic effects in dental polymer networks *Dental Materials* **22(3)** 211-222.
18. Ferracane JL, Ferracane LL, & Musanje L (2003) Effect of light activation method on flexural properties of dental composites *American Journal of Dentistry* **16(5)** 318-322.
19. Peumans M, Kanumilli P, De Munck J, Van Landuyt K, Lambrechts P, & Van Meerbeek B (2005) Clinical effectiveness of contemporary adhesives: A systematic review of current clinical trials *Dental Materials* **21(9)** 864-881.
20. Van Meerbeek B, Braem M, Lambrechts P, & Vanherle G (1993) Two-year clinical evaluation of two dentine-adhesive systems in cervical lesions *Journal of Dentistry* **21(4)** 195-202.
21. Van Meerbeek B, Kanumilli P, De Munck J, Van Landuyt K, Lambrechts P, & Peumans M (2005) A randomized controlled study evaluating the effectiveness of a two-step self-etch adhesive with and without selective phosphoric-acid etching of enamel *Dental Materials* **21(4)** 375-383.
22. Van Meerbeek B, Perdigao J, Lambrechts P, & Vanherle G (1998) The clinical performance of adhesives *Journal of Dentistry* **26(1)** 1-20.
23. Tay FR, Pashley DH, Garcia-Godoy F, & Yiu CK (2004) Single-step, self-etch adhesives behave as permeable membranes after polymerization. Part II. Silver tracer penetration evidence *American Journal of Dentistry* **17(5)** 315-22.
24. Tay FR, Pashley DH, Suh B, Carvalho R, & Miller M (2004) Single-step, self-etch adhesives behave as permeable membranes after polymerization. Part I. Bond

strength and morphologic evidence *American Journal of Dentistry* **17(4)** 271-278.

25. Tay FR, Pashley DH, Yiu C, Cheong C, Hashimoto M, Itou K, Yoshiyama M, King NM. (2004) Nanoleakage types and potential implications: Evidence from unfilled and filled adhesives with the same resin composition *American Journal of Dentistry* **17(3)** 182-190.
26. Briso AL, Mestrener SR, Delicio G, Sundfeld RH, Bedran-Russo AK, de Alexandre RS, Ambrosano GM. (2007) Clinical assessment of postoperative sensitivity in posterior composite restorations *Operative Dentistry* **32(5)** 421-426.
27. Bekes K, Boeckler L, Gernhardt CR, & Schaller HG (2007) Clinical performance of a self-etching and a total-etch adhesive system—2 year results. *Journal of Oral Rehabilitation* **34(11)** 855-861.
28. Gaengler P, Hoyer I, Montag R, & Gaebler P (2004) Micromorphological evaluation of posterior composite restorations—A 10-year report. *Journal of Oral Rehabilitation* **31(10)** 991-1000.
29. Akimoto N, Takamizu M, & Momoi Y (2007) 10-year clinical evaluation of a self-etching adhesive system. *Operative Dentistry* **32(1)** 3-10.
30. Leinfelder KF, Radz GM, & Nash RW (1998) A report on a new condensable composite resin. *Compendium of Continuing Education in Dentistry* **19(3)** 230-232.

©Operative Dentistry, 2012, **38-1,** E1-E12

The Bond of Different Post Materials to a Resin Composite Cement and a Resin Composite Core Material

D Stewardson • A Shortall • P Marquis

Clinical Relevance

When selecting endodontic post materials, clinicians should be aware that fiber-reinforced composite (FRC) posts do not achieve a chemical bond to resin composites. The surfaces of different FRC posts need to be roughened by grit blasting with aluminum oxide particles to optimize micromechanical bonding.

SUMMARY

Purpose: **To investigate the bond of endodontic post materials, with and without grit blasting, to a resin composite cement and a core material using push-out bond strength tests.**

Materials and Methods: **Fiber-reinforced composite (FRC) posts containing carbon (C) or glass (A) fiber and a steel (S) post were cemented into cylinders of polymerized restorative composite without surface treatment (as controls) and after grit blasting for 8, 16, and 32 seconds. Additional steel post samples were sputter-coated with gold before cementation to prevent chemical interaction with the cement. Cylindrical composite cores were bonded to other samples. After sectioning into discs, bond strengths were determined using push-out testing. Profilometry and electron microscopy were used to assess the effect of grit blasting on surface topography.**

Results: **Mean (standard deviation) bond strength values (MPa) for untreated posts to resin cement were 8.41 (2.80) for C, 9.61(1.88) for A, and 19.90 (3.61) for S. Prolonged grit blasting increased bond strength for FRC posts**

*Dominic Stewardson, PhD, BDS, University of Birmingham, College of Medical and Dental Sciences, School of Dentistry, Birmingham, United Kingdom

Adrian Shortall, DDS, BDS, FDS RCPS, FFD RCSI, University of Birmingham, College of Medical and Dental Sciences, School of Dentistry, Birmingham, United Kingdom

Peter Marquis, PhD, BSc, FIM, University of Birmingham, College of Medical and Dental Sciences, School of Dentistry, Birmingham, United Kingdom

*Corresponding author: University of Birmingham, College of Medical and Dental Sciences
School of Dentistry, St Chads Queensway, Birmingham, B4 6NN, United Kingdom;
d.a.stewardson@bham.ac.uk

DOI: 10.2341/11-409-L

but produced only a minimal increase for S. After 32 seconds, mean values were 20.65 (4.91) for C, 20.41 (2.93) for A, and 22.97 (2.87) for S. Gold-coated steel samples produced the lowest bond strength value, 7.84 (1.40). Mean bond strengths for untreated posts bonded to composite cores were 6.19 (0.95) for C, 13.22 (1.61) for A, and 8.82 (1.18) for S, and after 32 seconds of grit blasting the values were 17.30 (2.02) for C, 26.47 (3.09) for A, and 20.61 (2.67) for S. FRC materials recorded higher roughness values before and after grit blasting than S. With prolonged grit blasting, roughness increased for A and C, but not for S.

Conclusions: **There was no evidence of significant bonding to untreated FRC posts, but significant bonding occurred between untreated steel posts and the resin cement. Increases in the roughness of FRC samples were material dependent and roughening significantly increased bond strength values ($p<0.05$). Surface roughening of the tested FRC posts is required for effective bonding.**

INTRODUCTION

The most frequent mode of failure reported for post-retained crowns is loss of retention of the post[1,2]; therefore, much research into post-retained crowns has focused on the factors that increase post retention.[3] In addition, with prefabricated posts, effective bonding of the post to the root may result in reduced stress in the root.[4,5] Prefabricated posts made from fiber-reinforced composite (FRC) materials are now available in addition to traditional metal posts. Manufacturers have suggested that FRC posts will bond to composite resins, both as luting cements and as core materials, and this is given as one of the principal reasons for choosing FRC posts in preference to metal posts. However, because prefabricated FRC posts are thoroughly cured in an industrial process, there may be little potential for chemical bonding to resin composite cements or cores. Therefore, the attachment of resins to FRC posts is likely to be through mechanical interlocking alone. The surfaces of metal prefabricated posts have large-scale features, such as serrations and indentations, which are incorporated to increase the retention of cements to posts.[6] FRC posts, with a few exceptions, do not have macroscopic retention features as these weaken the composite structure. Microroughening of metal posts using airborne particle abrasion (grit blasting) increases their retention[3,7] and has therefore also been used to enhance the retention of FRC posts to resin composite cements and cores.[8,9] The degree of roughness is likely to affect the retention produced and needs to be quantified if the effects on post retention are to be related to different surface treatment methods. To the authors' knowledge, in only one study to date has the roughness produced on the surface of posts by grit blasting been measured.[10] The degree of roughening will depend on many factors, including type of abrasive material and particle size, air pressure, distance between the grit-blasting nozzle and the target surface, nozzle aperture, angle of incidence of the particle jet and duration of abrasion. Comparing those studies where details of the grit-blasting method are given, there is little consistency in the pressures, durations, and distances selected, and there are no descriptions of how the abrasion around the cylindrical surface was managed.[8,9,11,12] As FRC materials contain different fibers, the roughening effects are likely to be material dependent. Grit blasting may damage the surface fibers, introduce flaws, and reduce the flexural strength of the post material. If the grit blasting is prolonged, a significant amount of material may be removed, thereby decreasing the post diameter and resulting in increased flexibility of the post.

The resin cements used to lute posts may be described as conventional composite cements based on bis-GMA (bisphenol glycidyl methacrylate) or as adhesive cements that contain additional functional monomers such as 4-META (4-methacryloyloxyethyl trimellitate anhydride) or MDP (10-methacryloyloxydecyl dihydrogen phosphate). These agents bond to metal oxides and are included to increase the affinity to calcified tissues and to metallic restorations.[13] Such cements would be expected to develop a chemical bond with the oxide layer formed on the surface of non-precious metal posts. The type of fiber used in many glass fiber composite posts is E-glass (electrical), which contains aluminum and calcium oxides.[14] Chemical bonding could occur between these oxides and the MDP. It is important for clinicians to understand how to achieve effective bonding of posts so as to improve retention and reduce the risks of root fracture. Therefore, it is necessary to investigate the bond of resin composites to currently available prefabricated post materials and examine the effect of surface roughening of post materials on their adherence. Many investigations compare the retention of different posts luted with different cements in natural roots and rank the different combinations according to the load required to dislodge the post.[15,16] Failure may have occurred

between the post and the cement, within the cement, or between the cement and the root. If the aim is to compare just the bond of a cement to a post, then a test setup is required in which the bond of the cement to the root is reliable and is the strongest interface. Alternatively, to test the bond of cement to root, then the cement/post bond must be the strongest link. Push-out tests in natural roots reveal only which combination of materials resists removal; they do not test specific interfaces. The objectives of this study were

1. to investigate the bond of different prefabricated endodontic post materials by comparing the push-out bond strength of three selected endodontic post materials bonded to a resin luting cement and to a resin composite core material;
2. to quantify the effect of grit blasting on the post materials; and
3. to assess the effect of grit blasting for periods of 8, 16, and 32 seconds on the bond strength of the post materials.

METHODS AND MATERIALS

Samples of a carbon fiber (Composipost, RTD, Grenoble, France) and a glass fiber (Aesthetiplus, RTD) endodontic post material and stainless steel (Arenastock, Letchworth, UK) were obtained as rods 100-mm long and 2 mm in diameter. These were then cut using a diamond disc into lengths appropriate for the tests as described in the sections that follow.

Surface Treatment of Posts

The surface of samples was roughened by grit blasting with 50 μm aluminum oxide powder and a Danville microetcher (Danville Engineering, San Ramon, CA, USA) connected to the main compressed air supply of a dental unit at a pressure of 2.3 to 2.4 bars. Each of the post materials was cut into 50-mm lengths, and nylon cubes with central holes 2 mm in diameter were pushed onto either end. In the lid of a transparent plastic box, a 50-mm long slot was cut, and samples were secured with soft wax inside the box below and parallel to the slot. The slot width was approximately the same as the width of the nozzle of the grit-blasting unit. This allowed the nozzle to be moved along the length of the post at a constant separation of 10 mm. After initial trials, it was possible to repeatedly maintain the time taken to pass the nozzle from one end of the slot to the other by hand, to approximately 1 second. After one aspect had been treated, the sample was rotated by 90°, and grit blasting was repeated until each aspect had been abraded for 2, 4, or 8 seconds (giving a total of 8, 16, or 32 seconds per sample)

For the stainless steel material, additional samples of untreated stainless steel were coated with a 150-nm film of gold using a sputter-coating machine, Emitech K550x (Emitech Ltd, Ashford, UK) to prevent any chemical bond from occurring between the metal oxides and the MDP in the Panavia 21. This would allow the researchers to separate the contribution of chemical bonding to the adhesion of the steel posts from the effect of surface roughening.

Evaluation of Surface Roughness

Qualitative assessment of the post surfaces before and after the different abrasion times was made with a scanning electron microscope (SEM, JEOL 5300, Jeol Ltd, Tokyo, Japan). Roughness was quantitatively measured using a profilometer (FormTalysurf Series 2, Taylor Hobson Ltd, Leicester, UK). Samples of the posts were secured while the ruby stylus was drawn around the circumference for a distance of 0.8 mm, and the average roughness, R_A, was measured at four sites along the sample on one surface and four sites measured after the sample was rotated through 180°. Eight measurements were made on each of four samples of each post before (as controls) and after the three grit-blasting periods chosen, and the mean roughness was compared.

Push-Out Bond-Strength Testing

To evaluate the bond between the three different post materials and the luting cement, resin composite cylinders approximately 50 mm in length were produced from Tetric composite shade A2 (Ivoclar Vivadent, Schaan, Liechtenstein). The material was expressed into thin-walled (0.8 mm) glass tubes with an internal diameter of 6.7 ± 0.05 mm and then polymerized in a light-curing oven (Visio-Lux, 3M/ESPE, St Paul, MN, USA) for 14 minutes. The irradiance of this unit was calculated as 67.6 ± 3.1 mW/cm^2.[17] After one week's storage at room temperature (23°C) and humidity (45% to 50%), these were then cut into 10-mm lengths using a water-cooled slow-speed saw (Isomet, Buehler, Lake Bluff, IL, USA). A central hole was cut through each cylinder using a 2.1-mm steel twist drill in a bench press. The walls of this post space were roughened to create a frictional key for the cement lute with a coarse-grade silicon carbide grinding paste (Anglo Abrasives Ltd, Manchester, UK) applied for 30 seconds using the smooth shank of a 1.6-mm twist drill from which the cutting flutes had been

removed. The channel was cleaned with an alcohol-containing gel (Purell, GOJO industries, Milton Keynes, UK), followed by copious amounts of tap water. After drying, the channel walls were coated with Panavia 21 (Kuraray, Kurashiki, Japan), a resin cement containing MDP as an adhesion promoter. A spiral root filler was used to ensure even coating of the walls.[18,19] Fifteen-millimeter lengths of the stainless steel, Composipost, and Aesthetiplus post materials, untreated as controls or grit-blasted for 8, 16, or 32 seconds, were luted into four cylinders and allowed to cure for 48 hours. Gold-coated steel post samples were also cemented into an additional four composite cylinders. To hold the cylinders for sectioning, 10-mm nylon cubes were fabricated and a 2-mm hole was prepared through the center. The portion of post material protruding from the composite cylinder was pushed into the hole in the cube, which could then be gripped in the sample holder of a water-cooled slow-speed saw and each composite cylinder cut into five discs approximately 1.4-mm thick. Twenty disc samples of each material were produced.

Samples were also created to derive the push-out bond strength between the post materials and a composite resin core. Four 15-mm lengths of the posts were secured into nylon cubes, coated with a bonding agent (Excite, Ivoclar Vivadent, Schaan, Liechtenstein), air thinned, and polymerized in the light-curing oven for one minute. Next, four 10-mm-long sections were cut from transparent cylindrical plastic pipettes (LP Italiana SPA, Milan, Italy) with an internal diameter of 7 mm and lightly lubricated with petroleum jelly and placed around the posts resting on the nylon cubes. This mold was carefully filled with Tetric composite shade A2 and polymerized and stored as described before for one week. Each sample was then cut into five slices as described earlier (20 for each post material). To compare the push-out bond strength of grit-blasted posts to core composite with that of untreated (control) posts, each post material was abraded for a single period of 32 seconds, and cores were built up and cured as described previously and subsequently sectioned to produce 20 sample discs. It was decided to grit-blast the samples bonded to core composite for only a single time of 32 seconds after observing the limited effect that shorter durations of grit blasting had had on push-out bond strength to the luting cement.

A jig was constructed to carry out the push-out tests; it consisted of a steel plate attached to the base of an Instron universal testing machine (model 5544, Instron UK, High Wycombe, UK). In the center of the plate was a 3.25-mm-diameter hole over which the sample disc was placed. A 3-mm-long, 1.5-mm-diameter steel punch was attached to the upper part of the machine and aligned with the center of the hole in the base plate. The punch was lowered at a crosshead speed of 0.5 mm per minute and the load required to dislodge the post from the composite disc was recorded.

The push-out bond strength was calculated using the following equation:[20]

$$\text{Bond strength} = F_{\max} \div \pi DH$$

where $F_{\max}$ was the load required to dislodge the post (Newtons), D was the mean diameter, and H the mean thickness of the disc (millimeters); the value of π was taken as 3.142.

After failure, each sample was examined under the SEM to identify where failure had occurred, and images were obtained of representative examples.

Statistical Analysis

Statistical analysis of all data was carried out using the statistical package SPSS (version 16, SPSS Inc, Chicago, IL, USA). The data were first examined using Shapiro-Wilk and Levene's tests, which confirmed a normal distribution and homogeneity of variance. The mean loads were then compared for statistically significant differences using independent samples t-tests or one or two-way analysis of variance (ANOVA), as appropriate, with post hoc Scheffé tests (α=0.05).

RESULTS

Surface Roughness

Comparison of the surfaces as seen under the SEM showed that the untreated steel presented a relatively smooth surface with a few shallow indentations and linear grooves (Figure 1). After grit blasting for 8 seconds there was a widespread but apparently superficial increase in roughness. There was a more obvious increase in roughness after 16 seconds but little additional increase in roughness was obvious after 32 seconds of grit blasting.

The surface of the Composipost samples showed little change after 8 seconds of grit blasting compared with untreated (plain) samples (Figure 2). Surface changes become more apparent after 16 seconds but it was difficult to differentiate between the roughness observed after 16 seconds or 32 seconds. The increase in irregularity appears to be

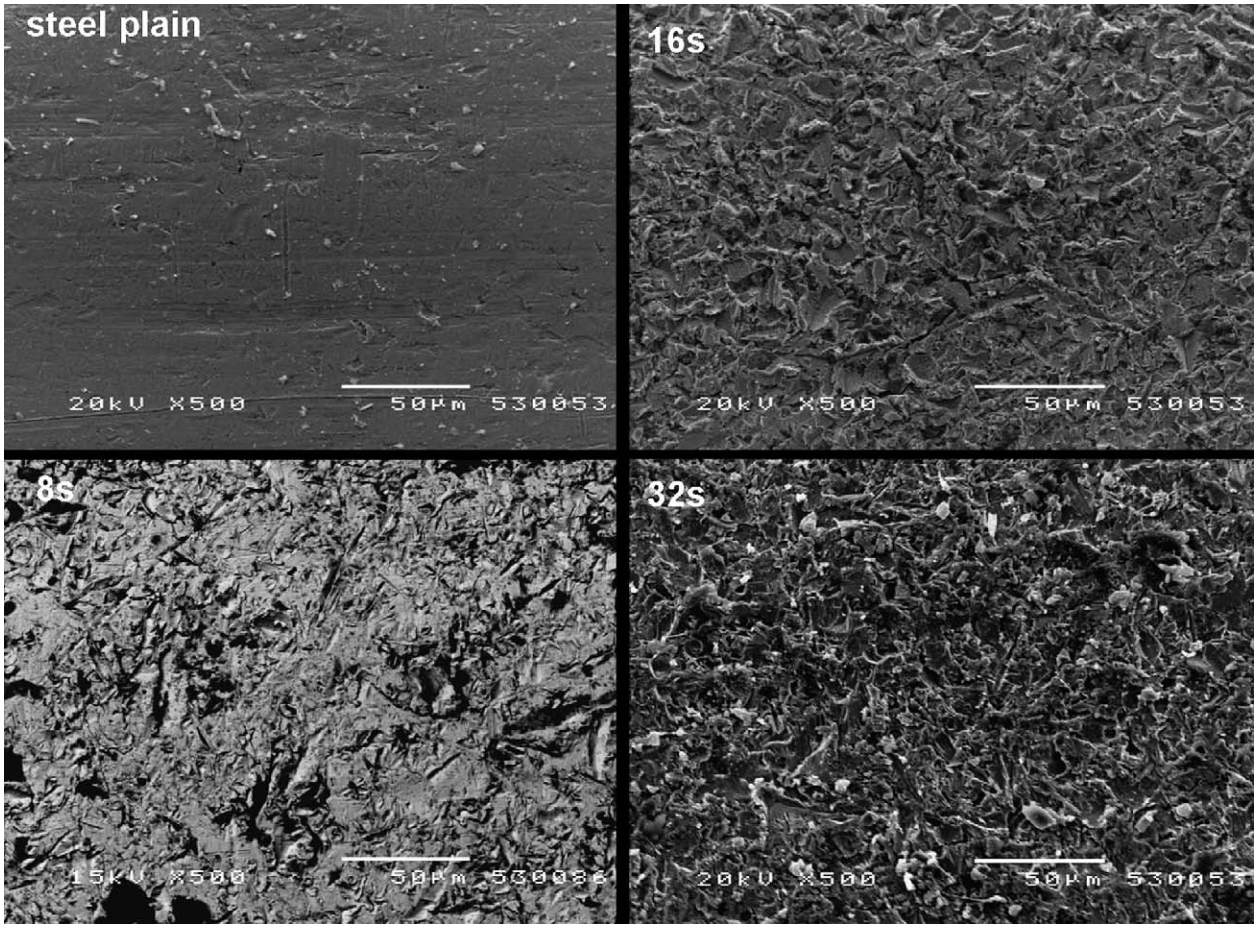


Figure 1. *Representative SEM images of stainless steel, untreated and after grit blasting for 8, 16 , and 32 seconds.*

due in part to the creation of surface voids by the loss of sections of fibers from the surface.

The samples of the untreated Aesthetiplus material had a more irregular surface than the Composipost, which is composed of smaller-diameter fibers. After 8 seconds of grit blasting there was a noticeable change in the surface appearance. After 16 seconds the roughness became more apparent but little difference could be seen between the surfaces after 16 seconds or 32 seconds. As with Composipost, loss of some superficial fibers produced furrows on the surface but these were of a greater diameter in the Aesthetiplus (Figure 3).

Profilometry

The mean roughness determined by the profilometer for each material and grit-blasting time is shown in Figure 4. It may be seen that Aesthetiplus had the highest mean R_A before and after each period of grit blasting, and grit blasting produced a proportionally smaller increase in roughness on the stainless steel than occurred for either FRC material. Analysis with one-way ANOVA and post hoc Scheffé tests ($p=0.05$) showed that for both FRC materials there was a significant increase in mean R_A after 8 seconds and again after 16 seconds of grit blasting ($p<0.005$) but there was no significant difference in the roughness between 16 seconds and 32 seconds ($p=0.858$ Composipost; $p=0.875$ Aesthetiplus). The stainless steel showed a significant difference in mean R_A after grit blasting for 8 seconds ($p<0.001$) but there was no significant difference in roughness after 16 seconds ($p=0.969$) or after 32 seconds ($p=0.083$).

Between materials, the surface of the untreated Aesthetiplus was significantly rougher than the other two untreated materials, which were not significantly different from each other. After 8 seconds and after 16 seconds of grit blasting, the mean R_A for each material was significantly different from the others ($p<0.01$). After 32 seconds, stainless steel was significantly less rough than the

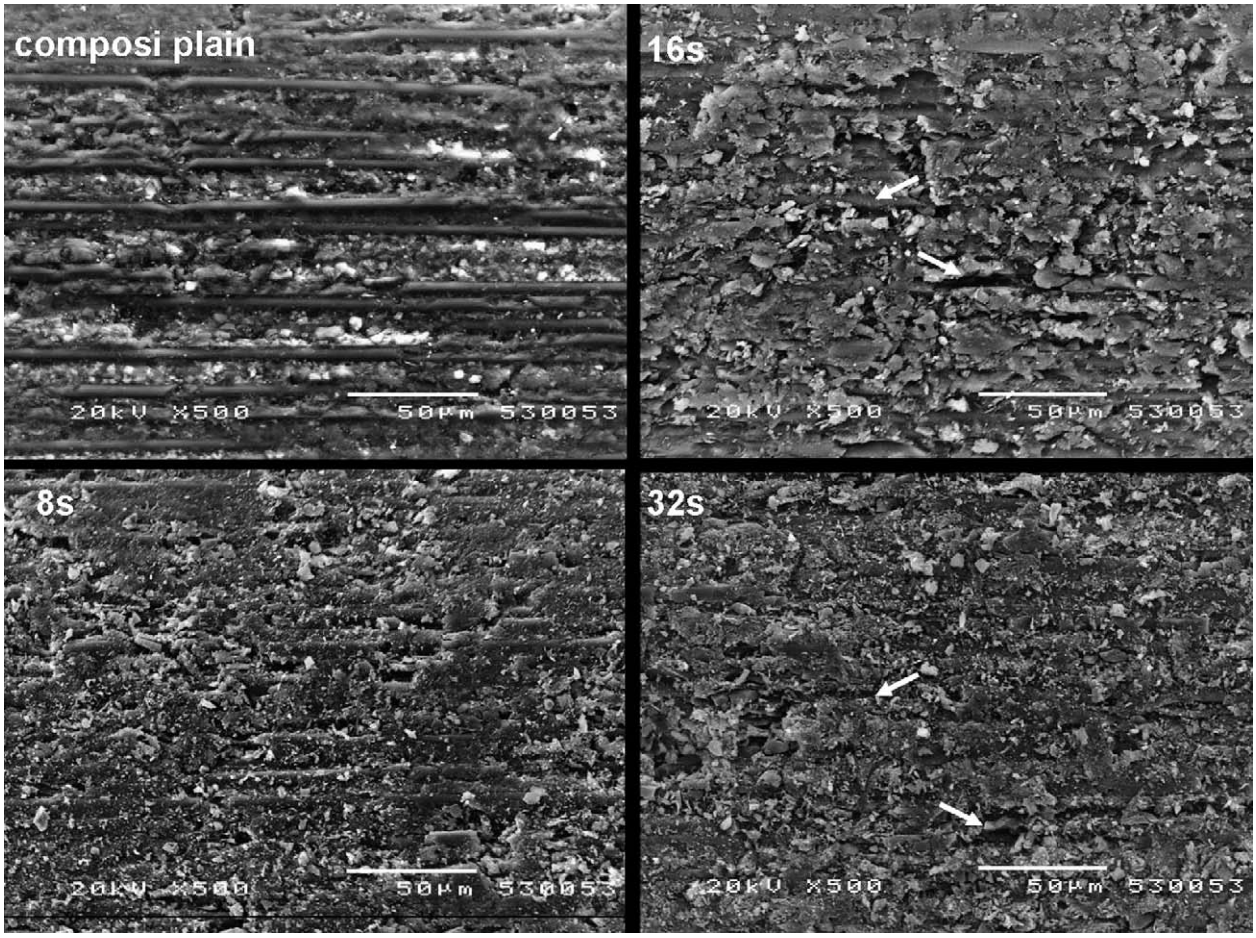


Figure 2. *SEM images of the surface of samples of Composipost before and after grit blasting for 8, 16, and 32 seconds. Arrows indicate areas where short sections of fibers may have been plucked from the surface of the sample.*

FRC materials ($p<0.001$), which showed no significant difference between them ($p=0.06$).

Push-Out Bond Strengths to Luting Cement

The mean values for push-out bond strengths of the post materials luted into the composite cylinders are recorded in Table 1. After the FRC posts received grit blasting, the bond strengths increased with increasing abrasion time. The plain steel posts recorded much higher strength values than the plain FRC post, and grit blasting of the steel posts was associated with only a slight further increase in mean push-out bond strength. Increasing durations of grit blasting were associated with increases in push-out bond strengths for the FRC posts. The gold-coated steel samples recorded the lowest mean bond strength values.

Statistical analysis using two-way ANOVA ($p=0.05$) showed significant main effects for both material ($p=0.007$) and post surface treatment ($p=0.003$) and a significant interaction between material and surface treatment ($p<0.001$). One-way ANOVA and post hoc Scheffé tests were then performed. The push-out bond strengths of the untreated FRC posts showed no statistically significant difference ($p=0.41$) but both were significantly lower than that of untreated steel ($p<0.001$). Comparing the effects of the surface treatment used, after 8 seconds of grit blasting, the shear strength of Composipost had increased but was significantly lower than that of Aesthetiplus ($p=0.032$), which was lower than that of steel ($p<0.001$). After 16 seconds of grit blasting, the bond strength of Composipost was lower but not significantly lower than that of Aesthetiplus ($p=0.055$). The latter mean bond strength value was significantly lower than that for the steel posts ($p<0.001$). After 32 seconds there was no significant difference among the mean bond strengths of the materials ($p>0.05$).

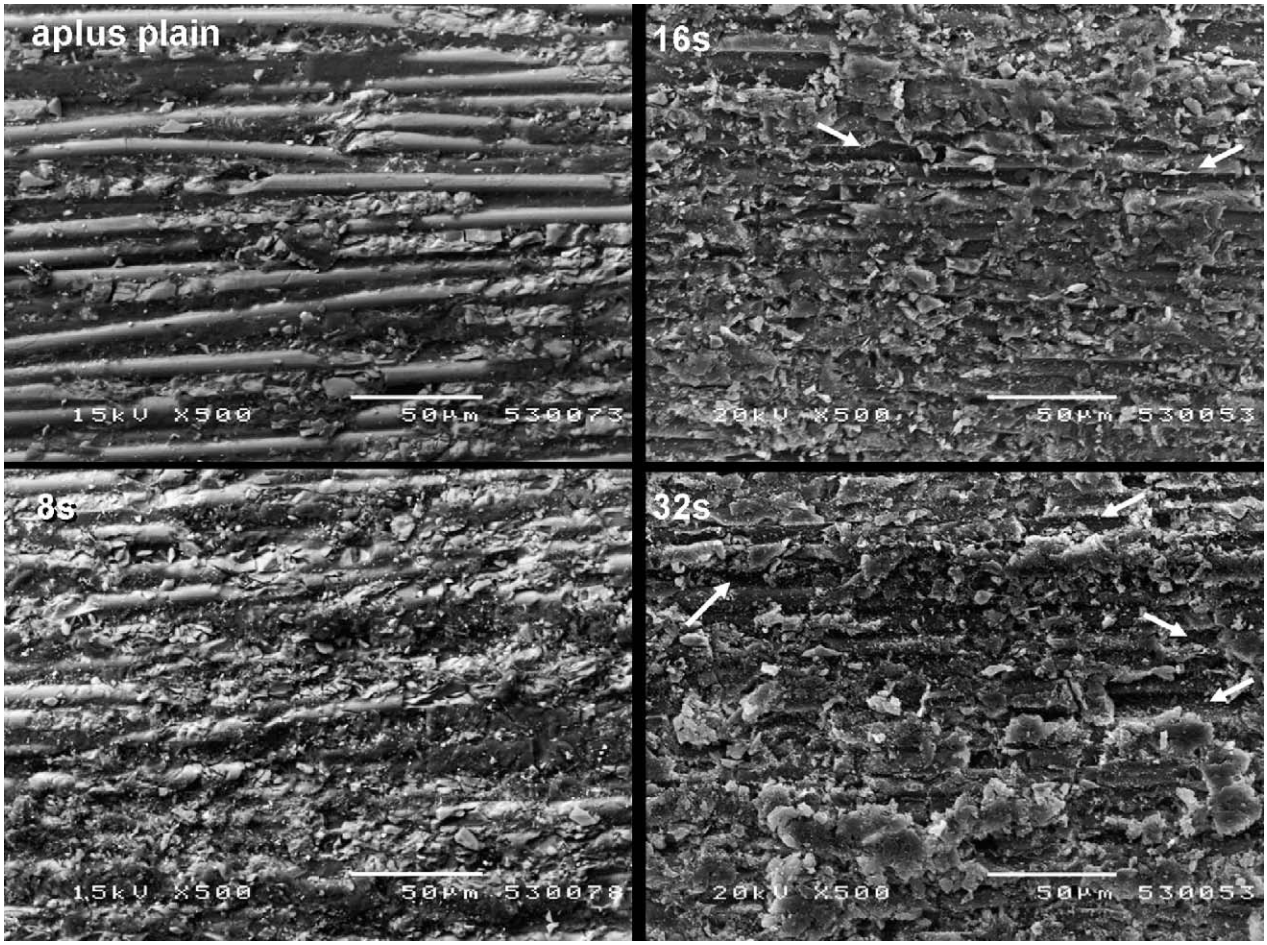


Figure 3. *Images of the surface of samples of Aesthetiplus before and after grit blasting for 8, 16, and 32 seconds. Arrows indicate furrows remaining where fibers may have been lost.*

The increase in bond strength showed different trends among the materials. For stainless steel, push-out bond strength was not significantly increased after 8 seconds of grit blasting ($p=0.893$). The difference in bond strength between 8 seconds and 16 seconds just reached significance ($p=0.039$) but there was no significant difference between the bond strength after 16 seconds and after 32 seconds ($p=0.999$). The push-out bond strength of Composipost after 8 seconds of grit blasting was not significantly different from that of the untreated material ($p=0.205$). There was a significant increase between 8 seconds and 16 seconds ($p=0.003$) and again between 16 seconds and 32 seconds ($p=0.02$). For Aesthetiplus, there was a significant increase in bond strength after 8 seconds of grit blasting ($p=0.001$) and between 8 seconds and 16 seconds of grit blasting ($p<0.001$) but no significant difference in the bond strength between 16 seconds and 32 seconds ($p=0.829$).

Examination of the samples after push-out testing showed that almost no cement was present on either dislodged untreated FRC post, suggesting that failure had occurred at the interface of the FRC post and the lute (Figure 5A,B) On the stainless steel samples the post surfaces were entirely covered with cement, and failure appeared to have occurred between the cement and the composite cylinder surface (Figure 5C). A mixture of appearances was observed on the failed push-out samples of grit-blasted Aesthetiplus. Among the samples treated for 8 seconds, surface fibers were visible, and there were only small areas of cement attached, suggesting that failure had occurred between the cement and the post. On the samples treated for 32 seconds, a thick layer of cement could be seen over most of the surface with little remaining attached to the composite cylinder (Figure 5D). Failure appeared to be predominantly the result of separation of the cement from the composite. Samples abraded for 16 seconds showed a mix of the two appearances. With increas-

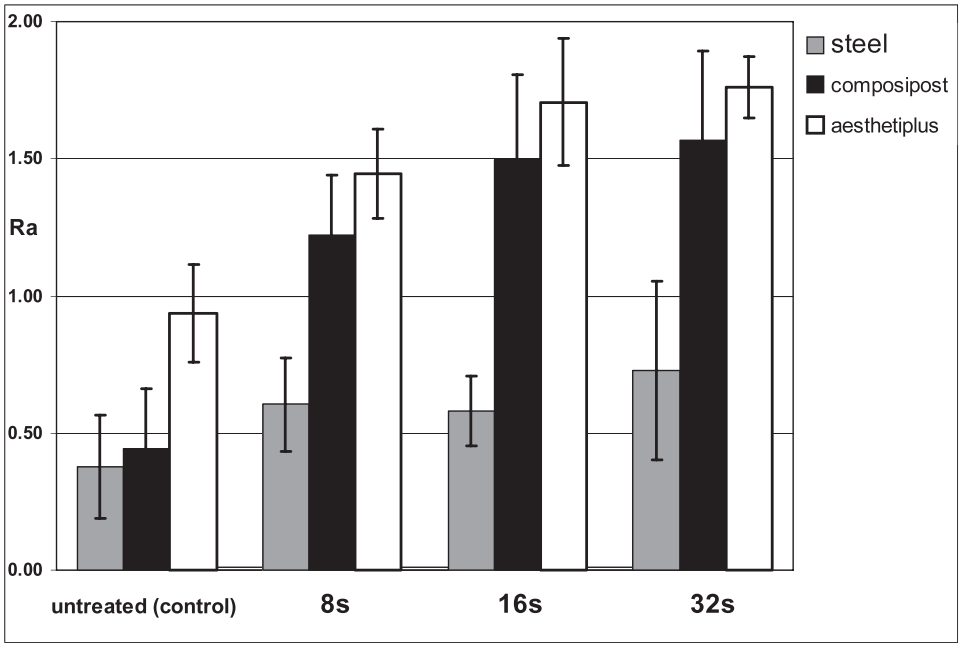


Figure 4. *Graph comparing the average roughness values (R_A) for the post materials before (control) and after grit blasting for 8, 16, and 32 seconds. Error bars indicate standard deviation.*

ing grit blasting there was some increase in the amount of cement fragments adherent to the surfaces of the Composipost samples after failure. However, even after 32 seconds of grit blasting there were several areas where fibers were clearly visible on the post surface, indicating little attachment of cement at these points (Figure 5E). Most of the cement layer was also identifiable attached to the surrounding composite, indicating that the mode of failure was mainly adhesive between the cement and the Composipost surface with some cohesive failure within the cement. When the steel posts had been gold-coated and tested, there was no cement visible on the post surface after failure (Figure 5F).

Push-Out Bond Strengths to Core Composite

The mean push-out bond strength values for the samples are shown in Table 2. Statistical analysis using independent samples *t*-tests showed that there was a significant difference between the bond strengths of the untreated and the grit-blasted samples of each material ($p<0.001$).

One-way ANOVA and post hoc Scheffé tests indicated that the bond of the core composite to the untreated Composipost was significantly weaker than to stainless steel ($p<0.001$) and that the bond with the steel was significantly weaker than that of the plain Aesthetiplus ($p<0.01$). After grit blasting for 32 seconds, the same significant differences between push-out bond strength values were also identified ($p<0.001$).

DISCUSSION

In this study, a push-out test was used to evaluate post retention. A variety of other methods have been employed to test the retention of posts; pull-out tests[21] produce a nonuniform distribution of stresses and are difficult to carry out on FRC posts because the post will be damaged by the gripping jaws of the loading machine. Microtensile testing has been adapted to evaluate the bond between posts cemented into tooth roots and between posts and core materials. However, the samples produced in this situation do not have planar surfaces arranged perpendicular to the separating force.[22,23] The interface is curved, and the stress field across the surface will be different from that in a standard microtensile test.

Push-out tests have also been used to calculate bond strength values[20] and can allow the bond at different depths within a post space to be compared. The preparation of samples is less technique sensitive than for microtensile testing[24]; there is less variability in the data[25] and a more uniform distribution of stress occurs.[26] In this study, samples were stored for one week to ensure that polymerization of the self-cure cement was complete. No immersion or artificial aging of samples was carried

Table 1: *Comparison of Push-Out Bond Strengths to Panavia 21 for Each of the Post Materials Before and After Grit Blasting*

Material	Treatment	Mean Push-Out Bond Strength (MPa)	SD
Steel	Untreated	19.90[de]	3.61
Steel	Grit blasting for 8 s	20.43[de]	3.09
Steel	Grit blasting for 16 s	23.21[e]	2.37
Steel	Grit blasting for 32 s	22.97[e]	2.87
Steel	Gold coated	7.84[a]	1.40
Composipost	Untreated	8.41[a]	2.80
Composipost	Grit blasting for 8 s	11.18[ab]	3.13
Composipost	Grit blasting for 16 s	16.29[cd]	4.67
Composipost	Grit blasting for 32 s	20.65[de]	4.91
Aesthetiplus	Untreated	9.61[ab]	1.88
Aesthetiplus	Grit blasting for 8 s	13.93[bc]	3.07
Aesthetiplus	Grit blasting for 16 s	19.51[de]	4.36
Aesthetiplus	Grit blasting for 32 s	20.41[de]	2.93

out as the aim of this study was to assess the potential for post materials to bond to a resin composite core and cement. Therefore, no attempt was made to simulate a clinical environment. Had the results revealed strong bonding for the FRC posts, it would then have been appropriate to challenge the samples with, for example, a thermocycling regimen before using the results to predict clinical performance.

The results of the push-out bond strength tests and the examination of the failed samples show that the Panavia resin cement developed a greater bond to the stainless steel material than to either of the FRC materials. This is likely because of a chemical reaction occurring between the cement and the steel as, when the steel was coated with gold, the bond strength value decreased below that recorded for the FRC materials. The gold coating will have prevented the MDP component of the cement from reacting with the base metal surface oxides. The post also had a relatively smooth surface and so very little mechanical locking could have occurred to increase bonding. The higher elastic modulus of the steel may also have been a contributing factor because it has been demonstrated that bond strength values can be affected by the difference in modulus of the two bonded materials.[27] However, the push-out bond strength values of the posts to either the core composite or the composite cement showed no direct relation with the modulus of the post because the untreated Composipost, which has an intermediate modulus,[28] recorded lower bond strength than either the steel or untreated Aesthetiplus materials.

The untreated FRC posts recorded low bond strengths, and although this was not statistically significant, the bond strengths correlated with the surface roughness determined for the untreated FRC posts and the gold-coated steel. From this and the observation that failure occurred between the cement and the post, it may be assumed that, in contrast to steel, no significant chemical bonding occurred between the Panavia cement and the untreated FRC materials. The push-out bond strengths also increased in conjunction with the increase in surface roughness produced by the grit blasting. The surface roughness of the steel was not greatly increased by this treatment and little increase in bond strength was noted. However, in view of the already high bond strengths noted for untreated steel and the proportion of fractures in the composite cylinder to which they were attached, it is unlikely that higher values could be achieved in this test setup without exceeding the ultimate strength of the composite. The increase in push-out bond strength produced by grit blasting is consistent with similar increases in retention reported in pull-out tests[8] and push-out tests[29] of grit-blasted glass fiber posts.

The method of grit blasting described did not ensure absolute consistency of treatment between samples but a greater degree of reproducibility was possible than that achieved with the methods described among other similar studies.[7–9] The glass fiber posts increased their roughness in less time than did the carbon fiber posts, which suggests that they are less resistant to the effects of grit blasting than the carbon fiber material, but with longer periods of grit blasting, the roughness produced was similar between the two materials. Although push-out bond strengths were also similar between the FRC posts after prolonged grit blasting, failure among the carbon fiber post samples appeared to

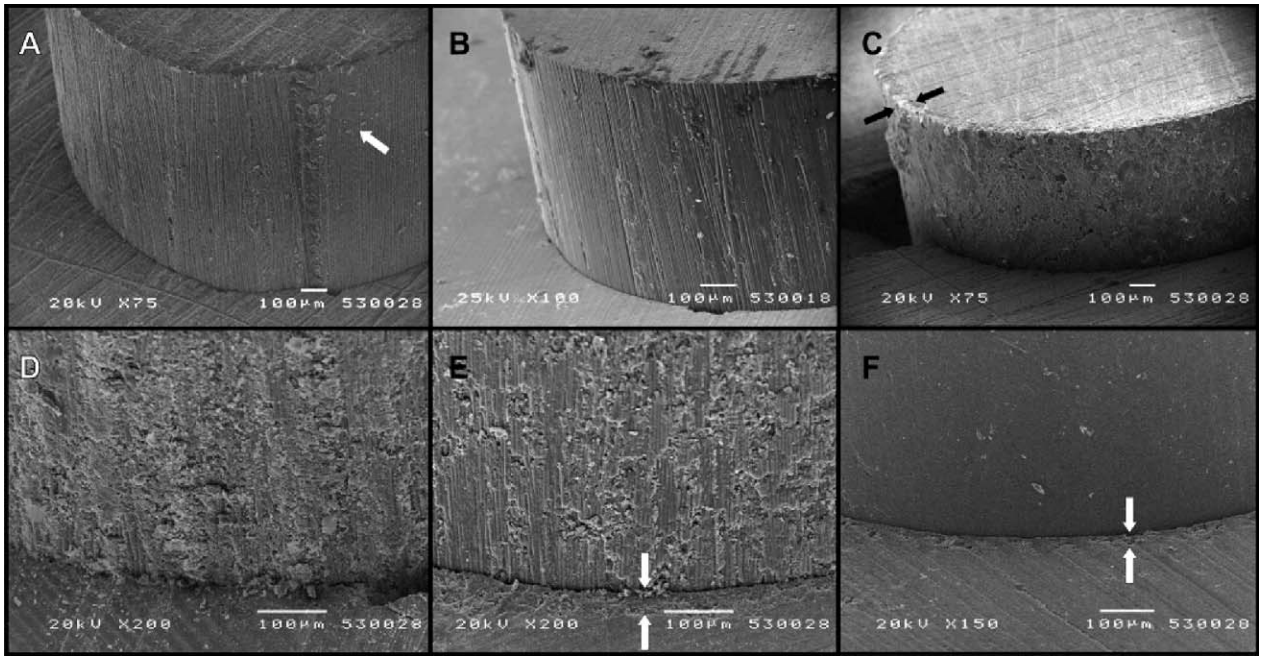


Figure 5. *SEM images of failed push-out test samples showing the following (A): Small areas of luting cement attached (arrowed) to untreated Aesthetiplus sample. (B): No significant attachment of luting cement to the post surface of untreated Composipost. (C): Untreated stainless steel push-out sample with fracture of surrounding composite cylinder. All the luting cement appears to be attached to the surface of the post (arrows). (D): Higher magnification image of grit-blasted Aesthetiplus with substantial attachment of cement to the post surface and no layer of cement attached to the surrounding composite. (E): Surface of grit-blasted Composipost sample with surface fibers visible and a luting cement layer attached to the composite disc (arrowed). (F): Gold-coated stainless steel sample showing the cement lute layer attached to the inner wall of the composite disc (arrowed) and no cement visible on the post surface.*

have occurred mainly between the post and the cement, whereas more of the glass fiber samples had failed between the cement and the composite. This could be because carbon fibers are less readily wetted than are glass fibers.[30]

Table 2. *Comparison of Push-Out Bond Strengths to the Core Composite of Each of the Post Materials Before and After Grit Blasting*

Material	Treatment	Mean Push-Out Bond Strength (MPa)	SD
Steel	Untreated	8.82	1.18
Steel	Grit blasting for 32 s	20.61	2.67
Composipost	Untreated	6.19	0.95
Composipost	Grit blasting for 32 s	17.30	2.02
Aesthetiplus	Untreated	13.22	1.61
Aesthetiplus	Grit blasting for 32 s	26.47	3.09

In the push-out tests of the posts bonded to core composite, bond strengths were not clearly related to the surface roughness of the posts. The untreated and grit-blasted glass fiber Aesthetiplus posts had rougher surfaces than the other materials and recorded higher bond strength to the core composite. However, the carbon fiber Composipost recorded the lowest bond strengths with and without grit blasting, despite having a rougher surface than the stainless steel posts. Surface roughness alone does not explain these results. Wetting differences among the materials or possible chemical interactions with components of the bonding agent may also have a part to play and requires further investigation.

The effect of grit blasting showed some differences between the materials. With the steel, there was little quantifiable increase in roughness even after 32 seconds of treatment. Both FRC materials showed a much greater increase in roughness with grit blasting, which was more pronounced with the glass fiber post. However, the effect on surface roughness appeared to be reaching its limit after 32 seconds. With both FRC posts, loss of surface material was becoming noticeable after 32 seconds, more so on the

glass fiber samples. There would therefore seem to be an optimum duration for grit blasting at which significant roughness can be produced without significant destruction occurring. Other investigations have reported that grit-blasting procedures caused no significant effect on flexural properties of FRC posts[12,31] but in these studies the grit-blasting methods used appeared to be less aggressive than the 32-second protocol selected here. The use of more intensive applications or longer periods of grit blasting are unlikely to produce a significant further increase in surface roughness but could result in a progressive decrease in the diameter of FRC posts, an increase in their flexibility, and a reduction in the maximum load-bearing capacity of the post.

CONCLUSIONS

The results of this study show that Panavia cement bonded effectively to smooth steel post materials but that no significant bonding occurred to FRC post materials. To enhance the retention of an FRC post when using Panavia cement, it is therefore necessary to roughen its surface. Grit blasting increases the micromechanical retention but the degree of roughening produced and the time needed to achieve sufficient surface roughening varies between different FRC materials.

Conflict of Interest

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

(Accepted 26 April 2012)

REFERENCES

1. Mentink AG, Meeuwissen R, Kayser AF, & Mulder J (1993) Survival rate and failure characteristics of the all metal post and core restoration *Journal of Oral Rehabilitation* **20(5)** 455-461.
2. Bergman B, Lundquist P, Sjogren U, & Sundquist G (1989) Restorative and endodontic results after treatment with cast posts and cores *Journal of Prosthetic Dentistry* **61(1)** 10-15.
3. Stockton LW (1999) Factors affecting retention of post systems: a literature review *Journal of Prosthetic Dentistry* **81(4)** 380-385.
4. Asmussen E, Peutzfeldt A, & Sahafi A (2005) Finite element analysis of stresses in endodontically treated, dowel-restored teeth *Journal of Prosthetic Dentistry* **94(4)** 321-329.
5. Mendoza DB, Eakle WS, Kahl EA, & Ho R (1997) Root reinforcement with a resin-bonded preformed post *Journal of Prosthetic Dentistry* **78(1)** 10-14.
6. Standlee JP, & Caputo AA (1993) Effect of surface design on retention of dowels cemented with a resin *Journal of Prosthetic Dentistry* **70(5)** 403-405.
7. Nergiz I, Schmage P, Platzer U, & McMullan-Vogel CG (1997) Effect of different surface textures on retentive strength of tapered posts *Journal of Prosthetic Dentistry* **78(5)** 451-457.
8. Balbosh A, & Kern M (2006) Effect of surface treatment on retention of glass-fiber endodontic posts *Journal of Prosthetic Dentistry* **95(3)** 218-223.
9. Radovic I, Monticelli F, Goracci C, Cury AH, Coniglio I, Vulicevic ZR, Garcia-Godoy F, & Ferrari M (2007) The effect of sandblasting on adhesion of a dual-cured resin composite to methacrylic fiber posts: microtensile bond strength and SEM evaluation *Journal of Dentistry* **35(6)** 496-502.
10. Nergiz I, Schmage P, Ozcan M & Platzer U (2002) Effect of length and diameter of tapered posts on the retention *Journal of Oral Rehabilitation* **29(1)** 28-34.
11. Soares CJ, Santana FR, Pereira JC, Araujo TS, & Menezes MS (2008) Influence of airborne-particle abrasion on mechanical properties and bond strength of carbon/epoxy and glass/bis-gma fiber-reinforced resin posts *Journal of Prosthetic Dentistry* **99(6)** 444-454.
12. Cheleux N, Sharrock P, & Degrange M (2007) Surface treatments on quartz fiber post: influence on adhesion and flexural properties *American Journal of Dentistry* **20(6)** 375-379.
13. Miller BH, Nakajima H, Powers JM, & Nunn ME (1998) Bond strength between cements and metals used for endodontic posts *Dental Materials* **14(5)** 312-320.
14. Wallenberger FT, Watson JC, & Li H (2001) Glass fibers In: Miracle DB, & Donaldson SL (eds) *ASM Handbook Volume 21*; Composites ASM International Materials Park, Ohio 27-34.
15. Perdigao J, Geraldeli S, & Lee IK (2004) Push-out bond strengths of tooth-colored posts bonded with different adhesive systems *American Journal of Dentistry* **17(6)** 422-426.
16. Kececi AD, Ureyen Kaya B, & Adanir N (2008) Micro push-out bond strengths of four fiber-reinforced composite post systems and 2 luting materials *Oral Surgery Oral Medicine Oral Pathology Oral Radiology Endodontology* **105(1)** 121-128.
17. Palin WM, Fleming GJ, & Marquis PM (2005) The reliability of standardized flexure strength testing procedures for a light-activated resin-based composite *Dental Materials* **21(10)** 911-919.
18. Turner CH (1981) Cement distribution during post cementation *Journal of Dentistry* **9(3)** 231-239.
19. Reel DC, Hinton T, Riggs G, & Mitchell RJ (1989) Effect of cementation method on the retention of anatomic cast post and cores *Journal of Prosthetic Dentistry* **62(2)** 162-165.
20. Dhert WJ, Verheyen CC, Braak LH, de Wijn JR, Klein CP, de Groot K, & Rozing PM (1992) A finite element analysis of the push-out test: influence of test conditions *Journal of Biomedical Materials Research* **26(1)** 119-130.

21. Gallo JR III, Miller T, Xu X, & Burgess JO (2002) In vitro evaluation of the retention of composite fiber and stainless steel posts *Journal of Prosthodontics* **11(1)** 25-29.

22. Mallmann A, Jacques LB, Valandro LF, Mathias P, & Muench A (2005) Microtensile bond strength of light- and self-cured adhesive systems to intraradicular dentin using a translucent fiber post *Operative Dentistry* **30(4)** 500-506.

23. Aksornmuang J, Foxton RM, Nakajima M, & Tagami J (2004) Microtensile bond strength of a dual-cure resin core material to glass and quartz fibre posts *Journal of Dentistry* **32(6)** 443-450.

24. Bouillaguet S, Troesch S, Wataha JC, Krejci I, Meyer JM, & Pashley DH (2003) Microtensile bond strength between adhesive cements and root canal dentin *Dental Materials* **19(3)** 199-205.

25. Goracci C, Tavares AU, Fabianelli A, Monticelli F, Raffaelli O, Cardoso PC, Tay F, & Ferrari M (2004) The adhesion between fiber posts and root canal walls: comparison between microtensile and push-out bond strength measurements *European Journal of Oral Sciences* **112(4)** 353-361.

26. Soares CJ, Santana FR, Castro CG, Santos-Filho PC, Soares PV, Qian F, & Armstrong SR (2008) Finite element analysis and bond strength of a glass post to intraradicular dentin: comparison between microtensile and push-out tests *Dental Materials* **24(10)**:1405-1411.

27. Della Bona A, & van Noort R (1995) Shear vs. tensile bond strength of resin composite bonded to ceramic *Journal of Dental Research* **74(9)** 1591-1596.

28. Stewardson DA, Shortall AC, Marquis PM, & Lumley PJ (2010) The flexural properties of endodontic post materials *Dental Materials* **26(8)** 730-736.

29. Bitter K, Priehn K, Martus P, & Kielbassa AM (2006) In vitro evaluation of push-out bond strengths of various luting agents to tooth-colored posts *Journal of Prosthetic Dentistry* **95(4)** 302-310.

30. Walsh PJ (2001) Carbon fibers In: Miracle DB, & Donaldson SL (eds) *ASM Handbook Volume 21*; Composites ASM International Materials Park, Ohio. 35-40.

31. D'Arcangelo C, D'Amario M, Vadini M, De Angelis F, & Caputi S (2007) Influence of surface treatments on the flexural properties of fiber posts *Journal of Endodontics* **33(7)** 864-867.

OPERATIVE DENTISTRY
CORPORATE SPONSORS

These Dental Manufacturers have joined *Operative Dentistry* in our commitment to publish quality dental literature in a timely manner. We thank them for their support.

DENTSPLY CAULK

ivoclar vivadent

ULTRADENT

GC America Inc.

SDI
SOUTHERN DENTAL INDUSTRIES

3M ESPE

BISCO
Specialist In Adhesive and Composite Technology
Bringing Science to the Art of Dentistry™

kuraray

NOVEMBER/DECEMBER 2012 • VOLUME 37 • NUMBER 6 • 561-676

EDITORIAL

BUONOCORE LECTURE

CLINICAL TECH/ CASE REPORT

CLINICAL RESEARCH

LABORATORY RESEARCH

DEPARTMENTS

ONLINE ONLY

OPERATIVE DENTISTRY *volume 37 • number 6 • pages 561-676* *november/december 2012*