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Editorial

Thoughts on Dentistry—2018

JA Platt, Editor

What is a general dentist, anyway? Today, my answer includes at least three parts and is biased by what I have experienced in the United States: 1) we had at least a tangible idea of how to answer this question over the past 50 years or so; 2) we have little idea of what the answer will be 10 to 20 years from now; and 3) the question is critical as dental education grapples with its purpose and function. As I think back over my clinical career as a general dentist, I ask myself, "Why am I in academic dentistry today?" After 16 years of full-time practice, I joined the world of full-time academic dentistry in 2000. Several factors contributed to my making the change. Among those, I did not like the way insurance companies were driving us further and further toward a procedure-driven mentality instead of a total-patient care mentality. I believe there is no way to win the battle against this pressure if we do not lay the groundwork with our students. I find this battle has not become easier, yet I still believe it to be key to what a general dentist will be in the years to come.

During my years in academics, I have often heard discussion about the evils of procedure-driven graduation requirements. Procedures become education currency, and this creates a numbers game that drives students away from a total-patient care mindset, just as it can for private practitioners. And on the other side of the argument is the understanding that without an adequate number of procedures, students do not gain the surgical skills needed to predictably provide the care needed for our patients. Twenty years ago, there was a significant push in dental education away from discipline-based departments. Many schools created mega-departments with the belief that this would enhance our ability to provide total-patient care and decrease emphasis on procedures. But what goes around, often comes around, and some of those mega-departments have been deconstructed back into discipline-based departments where numbers can still reign. What does that mean for the training of our students and for the direction of our profession?

If someone had told me 30 years ago that, before my career was over, dental students would no longer be able to replace missing canines as a matter of routine, I would have truly thought that they had lost their mind. But that is the state of our world. In one sense, this is a result of our success. Many procedures that were commonplace during the majority of my career are no longer so. Oral disease has not been eradicated, but the utilization of dental care has been changing.[1,2] During the last quarter of the 20th century, many successful dental practices were based on the endodontic and prosthodontic needs of all segments of the population. As overall dental health has improved for the upper and middle classes, we now find a greater percentage of restorative needs in the lower socioeconomic class and the geriatric population. This is challenging our system and, therefore, the reality of what a general dentist will actually be. We need to figure out how to respond.

In the practice world, one response has been the growth of larger group- and corporate-owned practices. Yes, there are other factors that contribute to this, but I would suggest that the need to increase volume to maintain desired cash-flow levels is one of the contributing factors. A large amount of the disease that needs to be treated exists in a portion of the population that does not have the financial resources to pay for that treatment. A basic understanding of economics suggests that this negative pressure on fees will lead toward a need for increased volume. This also puts added stress on dental education as we traditionally think about it. You see, some of these patients are those that used to frequent dental school clinics to take advantage of a reduced fee schedule. Now, many school fees are not so reduced, and if these patients pursue care, they may do so for similar or marginally higher fees without having to invest the large amount of time that the educational environment has traditionally demanded.

In academics, I believe we are largely kicking this challenge down the road. In some institutions, this may be tied to the reemergence of discipline-based

clinics, at least for early clinical experiences. I believe these clinics help calibrate early clinical experiences and solidify basic concepts. But even as this occurs, more procedures are being handed to residents, while fewer procedures are available for pre-doctoral students. I have heard it argued that residents in prosthodontics and the increased number of operative dentistry programs are finishing their training with levels of restorative experience somewhat comparable to what pre-doctoral students had 30 years ago. And our pre-doctoral students are graduating with the hope that they will find some way to pay their debts. What is it that we truly expect them to do?

Part of the current environment is the increased number of training programs in operative dentistry that we now see in the United States. For most of my career, there were four such programs. Today, the number is now in double digits. I contend that these and other advanced training and postgraduate year 1 programs will become more important for the next era of dentistry as traditional pre-doctoral programs are less and less able to provide the repetitions beyond competency essential to the training of our next generation of clinicians.

Isn't it time to embrace our reality? I see some progress with the growth of interprofessional education—working with teams of other health care providers in an educational environment. Oral health is an integral part of total health. We must embrace our role as oral health care providers; we contribute to the overall health of the people who come to see us. A dental career that thrives on the traditional restorative approaches of our past is becoming more and more difficult to achieve and, educationally, is being driven into the hands of a fewer number of well-trained practitioners.

Where does that leave the general dentist of the future? When leaving dental school, more advanced training may be a necessity for each new graduate. If so, that training should be available in the graduate's desired area of emphasis. In addition, new dentists must leave school understanding that they are overseeing total oral health care that is a *critical component of the overall health* of their patients. Knowing how to guide treatment for each patient should be at the core of a general dentist's activity. In this issue of *Operative Dentistry*, you will find an invited article from last February's Academy of Operative Dentistry Buonocore lecturer, Falk Schwendicke.[3] Its content should stimulate thought.

So, why did I jump to academic dentistry? Toward the top of the list of reasons was the desire to help students think less about procedure-driven care and more about total-patient care. What do I see today? Dental education is struggling to cope with what total-patient oral health care looks like in a world where traditional dental diseases do not have a great enough prevalence in the right populations to support all of the dentists being trained, at least not in our historical way of thinking. This in the midst of a remuneration system that demands surgical procedures be accomplished to pay off debt and earn a living. We are at a crossroads, and great leadership is needed to guide us through this period of change.

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Buonocore Lecture

Tailored Dentistry: From "One Size Fits All" to Precision Dental Medicine?

F Schwendicke

Clinical Relevance

Treatment needs and diagnostic/management options in dentistry have changed dramatically over the past five decades. A "one-size-fits-all" approach toward managing dental health/disease is increasingly insufficient. "Tailored dentistry" will be a cornerstone of daily clinical practice in the future.

SUMMARY

Over the past 30 years and fueled by both a rapidly evolving understanding of dental diseases and technological advances in diagnostics and therapy, dentistry has been changing dramatically. Managing dental caries and carious lesions had, for nearly a century, encompassed only a small number of basic concepts that were applied to virtually all patients and lesions, namely, invasive removal of any carious tissue regardless of its activity or depth and its replacement with restorative materials (amalgams or crowns for most of the past) or tooth removal and prosthetic replacement. Grounded in a deeper understanding of the disease "caries," its management—aiming to control the causes of the disease, to slow down or alleviate existing disease, and, only as a last resort, to remove its symptoms using a bur or forceps—has become more complex and diverse. In parallel and at nearly unprecedented speed, our patients are changing, as mirrored by ongoing debates as to the demographic and, with it, the social future of most high-income countries. This article describes how these changes will have a profound future impact on how we practice dental medicine in the future. It will deduce, from both demographic and epidemiologic trends, why there is the need to apply not one but rather the whole range of existing evidence-based concepts in an individualized (personalized) manner, hence increasing the effectiveness and efficiency of dental management strategies, and also describe how these strategies should be tailored according not only to our patients (their age and risk profiles) but also to the specific tooth (or site or lesion).

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TAILORING TO AGE-GROUPS

In many high-income countries, a dramatic demographic change is under way, characterized by population aging, shrinking, diversification, and geographic polarization (most rural areas get older and generally shrink, while urban areas grow and get younger or, at least, do not age). In parallel to this demographic change, the occurrence and distribution (prevalence, extent, and severity) of dental diseases has been changing drastically and is expected to change further in the future. For example, in the United States and Germany, tooth loss in adults and seniors has decreased staggeringly over the past 30 years, and edentulism is a rare phenomenon today in adults and will be rare in the future in seniors as well.[1-3] In parallel, periodontal disease is affecting a large part of the population, especially older people, who retain teeth now for long enough to develop periodontitis.[4] Consequently, with more teeth being retained and more of these teeth showing bone loss and hence exposed root surface, more (root and coronal) surfaces are at risk in older age.[5]

It is relevant to highlight that dental caries will thus no longer be a disease found mainly in children. In the past, the caries experience and thus the number of restored surfaces was already high in adolescence or early adulthood. In epidemiologic surveys, these surfaces could not experience caries once more (as such surveys measured mainly primary caries).[6] Consequently, new caries was mainly found in children and only seldom in adults or seniors (who did not even have many teeth at their high ages). Based on such measurements, one erroneously assumed that caries was a disease of children. This, however, is a misinterpretation. When accounting for the number of surfaces that had not been restored and were not missing (ie, were at risk), the increment of dental caries of an individual (measured as a percentage of new carious surfaces per all available surfaces at risk) seems to be rather constant.[7,8] With children in many countries of the world no longer exhibiting any carious lesions, it can be expected that new lesions will increasingly be found in adults or seniors.[9] This phenomenon is called morbidity compression and has been documented for a range of other chronic diseases as well.[10] Hence, the focus of managing dental diseases will shift—dental caries and associated preventive and restorative management will no longer be found or needed mainly in children or adolescents but rather in older age-groups.

This is especially true for root caries lesions due to the described reasons. This is problematic, as most concepts both for preventing and for restoratively managing carious lesions have been established for coronal, not root, lesions.[11] If additionally assuming that the population who will experience root caries lesions will be mainly the old and very old—often impaired in some way and suffering from poor oral hygiene due to limited dexterity, hyposalivation due to polypharmacy, and limited mobility (which significantly impacts the possibility of utilizing conventional dental care)[12]—it is obvious that the dentistry of the future will need to develop effective and efficient concepts to prevent and noninvasively manage root caries lesions. That is more true, as restorations for root lesions show high risk of failure and are in many cases hard to provide (considering access to the lesion, use of matrices, or moisture control).[13,14] In older, frail individuals, managing the dental biofilm has another relevant role: dental or denture biofilms significantly contribute to the risk of developing pneumonia.[15] Regularly maintaining full or partial dentures and providing professional oral hygiene has been found to significantly lower such risks[16] and could eventually even be cost effective due to averted hospitalization or decreased costs for managing dental complications.[17]

In summary, dental concepts of the future will need to be tailored to age-groups. Many concepts—at least in cariology—were developed for a younger population. This population is, to a large degree, no longer in great need of dental care. Instead, the need is shifted to older age-groups, which are additionally growing on an absolute level given the demographic changes occurring in many countries worldwide. Organizers of care, public health and clinical dental researchers, and practicing dentists, as well as stakeholders beyond dentistry, are called to action.[4]

TAILORING TO RISKS

With the decrease in the prevalence and extent of many oral diseases in a large portion of the population (at least those who are younger but increasingly also those who are middle aged), a small but stubborn proportion of the population has not benefited from this improvement in oral health. This small group carries a large portion or, in children, even the majority of the burden of oral diseases.[18] For example, in 12-year-old children, around 80% in Germany do not have any caries lesions, while the remaining 20% carry the total burden of caries experience.[3] This so-called polarization and the underlying inequality in oral health

distribution continues into adolescence and adulthood and can even be traced in seniors.[12,19-21] Such an unequal distribution of diseases can be seen for periodontitis and oral cancer as well and is generally common for many chronic diseases (eg, cardiovascular diseases or diabetes).[22-24] It is deeply grounded in wider social disparity and enters the health arena via a number of pathways, the discussion of which is beyond the scope of this paper.

With this polarization comes the need to identify high-risk individuals and discriminate them from the remaining larger part of the population with no or low risk. This is relevant, as both diagnostic and management strategies need to be tailored according to this risk. For example, dental caries detection increasingly aims to identify and assess early caries lesions using sensitive tools, such as fluorescence-based diagnostics. All sensitive tools—and most of the regularly introduced tools that are highly sensitive for early lesions—come with relatively high risk of false-positive diagnoses given the trade-off needed between sensitivity and specificity. If applied to low-risk populations, where the prevalence of lesions is low and existing lesions progress only slowly, the risk of overdetection is high. The chances of finding any lesions are relatively low,[25,26] and even if lesions are found, many of them are inactive (ie, do not require active therapy).[27] It is thus relevant to tailor the diagnostic strategy, including which diagnostic tool to use but also in which interval to apply it, to an individual's risk. Sensitive tools should be applied mainly in high-risk individuals and, if needed, in shorter intervals. In low-risk individuals, specific tools should be applied but not necessarily in high frequency (the risk of "missing" an active lesion and regretting it soon is low) and any diagnoses double-checked using a second diagnostic method if feasible (eg, fluorescence-based findings should be confirmed radiographically).

Therapies should also be tailored according to risk. For example, in low-risk individuals, the regular in-office application of fluoride varnish has been found to convey very limited effectiveness at low cost-effectiveness; that is, money spent here could be spent better elsewhere.[28,29] High-effort prevention should be applied in an individualized manner, while public health measures, such as a tax on sugar-sweetened beverages, are obviously cost effective even if they are not tailored to risks, which would also not be feasible.[30,31] Moreover, treatments for existing carious lesions should be tailored according to the certainty of the diagnosis. A large number of studies, for example, confirm that non-cavitated caries lesions can be effectively arrested using caries sealing or, for proximal surfaces, resin infiltration.[32-34] If in doubt of the surface status, it seems advisable to apply these methods and monitor the lesions for a certain time period instead of preemptively applying invasive means, which are irreversible.[25,26] Especially in low-risk individuals, these lesions are slowly progressing, even if they show some surface cavitation and neither sealing nor infiltration is effective. In contrast, placing restorations as first-line therapy puts the tooth on a spiral of ever-escalating retreatments given the limited life span of restorations, especially under less-than-optimal (ie, routine) conditions,[35,36] as discussed below.

If both diagnostics and treatments should be tailored according to risks, it is relevant to know how such risk assessment can be effectively performed. A number of risk assessment tools for caries have been developed. All of them build on assessing either risk factors, which are causally associated with the pathogenesis of dental caries (eg, dietary behavior or the presence of dental biofilm), or risk indicators, which are not directly related to the development of caries but have some indirect relationship with its pathogenesis (eg, socioeconomic status or dental utilization patterns or, most often, caries experience).[37] However, the predictive value (ie, the accuracy to predict new caries lesions or experience) of these models has been evaluated only on a very limited basis. Most of these parameters or tools have not been validated in populations other than those they were developed in (they may work well in that one but not another population). This problem is aggravated by so-called overfitting, in which the tool has been thoroughly optimized to predict new caries in the single population it was developed in but cannot replicate the high accuracy when applied to other groups. Moreover, when evaluating the single parameters used in these tools, most have only very limited value: nearly all parameters are not supported by quantitatively or qualitatively sufficient evidence (as collecting in-depth longitudinal data is challenging), and some show no accuracy at all but are similar to throwing a coin.[38] Currently, the most robust parameter is the existing caries experience, as it is a good indicator of past behavior and circumstances (including genetic aspects) and can, to a certain degree and assuming that behavior does not radically change, predict future caries experience.[8] Notably, however, this indicator is able to indicate caries risk only

when caries experience has already occurred; this is a significant disadvantage.

Overall, we are currently able to predict dental caries only on a very limited basis. Dentists nevertheless need to rely on the assessment systems we have, accepting their limitations and integrating them into their daily routines as best as possible.

TAILORING TO LESIONS

As described at the beginning of this article, dentistry was traditionally dominated by only a few available options for managing dental diseases (mainly caries). These were invasive and driven by the idea that dental caries was an infectious disease, requiring removal (eradication) of all causative bacteria.[39] This understanding of dental caries no longer holds. Currently, caries is understood as a disease driven by the transformation of a physiologic into a pathogenic biofilm (dysbiosis),[40] coming with an increased ability for acid release and demineralization of dental hard tissues, induced by abundant intake of fermentable carbohydrates (sucrose, glucose, and so on). This ecological plaque hypothesis understands the bacteria selection process and the resulting imbalance in the biofilm composition and activity as the driving forces behind the disease dental caries—and not biofilms *per se*.[41,42] Based on this understanding comes a significant shift in how dental caries and carious lesions are managed. Less invasive options, both for preventing new and for treating existing carious lesions, have been established and evaluated over the past five decades. Consequently, dentists today have a large number of available treatment options:

- Noninvasive strategies aim, without any breach of the dental surface, to prevent the occurrence of lesions or to manage existing carious lesions. Biofilm control (oral hygiene, chemical control, or probiotics), mineralization control (fluorides or calcium delivery), and dietary control (sugar restriction or substitution) fall into this group. Noninvasive strategies can control the disease of dental caries on the patient level (as they control the risk factors causing caries or the mineral loss as a result of the disease) as well as its symptoms (carious lesions) on-site at the tooth level.
- Microinvasive strategies involve conditioning the dental hard tissue, usually via acids, to retain something either on top of the tooth (usually sealants) or within it (resin infiltration). During this step, some micrometers of dental hard tissue are lost (hence *micro*invasive). Microinvasive strategies install a diffusion barrier onto or within the dental hard tissue, impeding acid diffusion into and mineral loss from it. Microinvasive strategies prevent new or arrest existing carious lesions but do not necessarily causally control the disease (the process) of dental caries (sealants and infiltration act on-site at the tooth level, not on a patient level). Sealing can be applied preventively and therapeutically, while caries infiltration can be used only to arrest existing (mainly proximal) lesions (the resin penetrates only porous, capillary active enamel).
- Invasive (restorative) interventions. These have been the traditional management strategy for dental caries, as described. However, restorations have a limited life span, ranging from decades (for small restorations placed under ideal conditions) to only a few years (for extensive restorations placed under routine dental care).[43,44] Each placed restoration will thus, at some point and strictly statistically speaking, require renewal. This would be unproblematic if this removal were possible without removing any further tissue. This, however, is unlikely, as restorations fail often due to either secondary caries or fracture of tooth/restorative material.[45,46] In both cases, some tooth tissue is lost during the complication. Moreover, when replacing most modern tooth-colored restorations, dentists usually remove some tooth tissue as well given the difficulties in discriminating it from the restorative material (which is adhesively bonded to the tooth).[47] Thus, with each restoration failure and replacement, additional tooth tissue is removed; this has been termed the spiral of re-restorations.[35,48] With this spiral, the invasiveness and the treatment costs increase, and, eventually, tooth retention is not always possible.[25,36,49] This is why nonrestorative (ie, noninvasive or microinvasive) alternatives for managing carious lesions are recommended today.[50,51]

The question that should be raised now is, Why do we not always resort to noninvasive or microinvasive instead of invasive (restorative) management of carious lesions? There are two reasons that restorations remain needed. First, noninvasive management strategies are thought to be largely invalid when a surface cavitation has been established, as the biofilm is now sheltered and cannot be effectively controlled. There is some evidence that under certain conditions, even cavitated lesions can be managed nonrestoratively (by installing a thorough cleaning regimen including regular application of fluoride); this is called nonrestorative cavity control. However, the few studies available show very

limited effectiveness of this measure for arresting lesions, possibly as the required behavior change of the patient and/or his or her caretaker is hard to achieve.[52-55] Second, one could resort to sealing all lesions, even cavitated ones. Under certain conditions, sealing has been found to also arrest cavitated lesions that harbor billions of bacteria, as the seal deprives the bacteria of dietary carbohydrates.[56,57] Sealed bacteria are no longer cariogenic (the lesion does not progress but is arrested), and their metabolic activity is radically altered/reduced (these effects are likely to be strain specific).[58,59] While a number of questions remain (do the metabolic products of these sealed bacteria have detrimental effects on the pulp?), clinical studies so far have shown convincingly that sealing also large amounts of bacteria does not induce clinical symptoms of pulpitis or necrosis.[53,56] Thus, with regard to controlling the lesion (its activity and the harbored bacteria), restorations are not needed.[34,60]

However, carious dentin is softer than sound dentin, and a relatively weak material, such as low filled resin, cannot withstand any forces exerted by biting onto it when it is not supported by firm or hard dentin.[61-63] Also, bond strengths of resins to carious dentin are much lower than those to sound dentin.[64,65] Consequently, sealants placed over larger amounts of carious dentin, such as in cavitated lesions, have been found to break or lose retention with high frequency. This would, if undetected, compromise the sealing effect and hence allow lesion progression.[57,66-70] Thus, sealing cavitated lesions is currently not possible using plastic sealants (the Hall Technique seals cavitated lesions in the primary dentition using stainless-steel crowns, but this is beyond the scope of this article).[71] In summary, the main aim of why, even today, carious tissue removal is still needed for most cavitated carious lesions is to maximize the longevity of the restoration.[72]

When performing carious tissue removal in cavitated lesions, today there are again more options available for dentists to choose from than in the past. When deciding on these options, a number of principles, agreed on by the International Caries Consensus Conference, should be followed:[72,73]

1) "avoid discomfort/pain and dental anxiety . . .
2) preserve non-demineralized as well as remineralizable tissue . . .
3) achieve an adequate seal by placing the peripheral restoration onto sound dentin and/or enamel, thus controlling the lesion and inactivating remaining bacteria . . .
4) maintain pulpal health by preserving residual dentin (avoiding unnecessary pulpal irritation/insult) and preventing pulp exposure . . .
5) maximize longevity of the restoration by removing enough soft dentin to place a durable restoration of sufficient bulk and resilience."[72]

Especially the last two points should be born in mind when different kinds of lesions are to be treated. For shallow or moderately deep carious lesions, where the pulp is not in danger of pulp exposure or irritation, the last principle can be prioritized. Maximizing restoration longevity helps to slow down the discussed spiral of re-restorations and hence increases tooth longevity. In deep lesions, however, the vitality of the pulp should be prioritized (assuming that the pulp is still vital and does not show signs of irreversible damage or necrosis). Especially pulp exposure should be, under all circumstances, avoided, as currently available treatments for exposed pulps are either often unsuccessful (eg, direct pulp capping, which shows satisfactory success only under optimal conditions[74-80] that are not necessarily replicable in each setting, tooth, or patient[81-83]) or are highly invasive (eg, root canal treatment, which has been found to also come with a significant risk of retreatment or extraction in routine dental practice settings[84,85]). Exposing the pulp often puts the tooth on a pathway to endodontic treatment and, with it, high treatment efforts and cost and decreased tooth longevity.

Consequently, in shallow or moderately deep lesions, dentists should remove as much carious dentin from the cavity as possible. In the periphery, hard dentin should remain, while centrally, firm (remineralizable) dentin should be left. This allows maximum restoration longevity but retains remineralizable dentin. In deep lesions (ie, those radiographically extending into the inner third or quarter of dentin or clinically extending close to the pulp), carious tissue removal should be less invasive and avoid pulp exposure at all costs: leathery or soft dentin can be left (in small, circumscribed areas) if needed, while peripherally, hard dentin should be left (to hermetically seal the lesion, depriving the sealed bacteria from their nutrition but also to ensure restoration longevity). Of course, for teeth with deep lesions but irreversible damage or necrosis, maintaining pulp vitality is no longer an aim, and root canal treatment should be performed after removing all carious tissue.[72]

In summary, dentists should tailor not only their management strategies to patients (with different age or risk profile) but also the specific tooth, site, or lesion they manage; for example, lesion depth, surface, status and activity are factors that need to be considered to appropriately assign one of the many currently available treatment options. For cavitated lesions, a tailored carious tissue removal is recommended as well.

CONCLUSIONS

With an increasingly complex distribution of diseases between different age groups and risk groups but also with the ever-larger number of available diagnostic and therapeutic options in today's dentistry, there is a strong demand for individualized, "tailored" approaches for managing the health of our patents, hence overcoming the formerly (also justified) one-size-fits-all approach. Tailored decision making has the potential to increase effectiveness and efficiency and reduce the risk of adverse events. However, the full scale of benefits of this approach have yet to be demonstrated.

Currently, many parts of medicine (and, as a part of that, dentistry) focus on stratification of patients (eg, according to age and risk, as described above) or teeth, sites, or lesions (eg, according to lesion depth or surface status). Such stratification is useful, as it requires only a small number of stratification parameters that then can be handled ("computed") by humans (in this case, dentists) with ease. For example, stratifying according to age or risks can be performed in only a few seconds or minutes and allows for somewhat tailored decision making.

Eventually, however, this is likely to move further on to a truly individualized ("precision") medicine. This will build on large amounts of (purposively or routinely collected) data: with decreasing costs and higher availability, microbiomic, proteomic, metabolomic, and genomic data may be used more frequently, allowing a more comprehensive understanding of the biologic foundations of health and disease in an individual patient. Routine data (eg, insurance claim or health records data) may also be used in this direction. The large amount of data will be more readily assessed, relying on vast computer power. Consequently, dentists will no longer mainly manage diseases any longer but rather will identify and understand patients' risks in their full complexity early on, allowing one to effectively modify these risks for a better long-term health outcome. Dentists will become the patients' lifetime oral health pilots.

Conflict of Interest

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

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Clinical Technique/Case Report

Color Match Between Composite Resin and Tooth Remnant in Class IV Restorations: A Case Series

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Clinical Relevance

A minimum of 14 days was needed to achieve a color match between the composite resin and the tooth remnant. The mock-up should be kept for at least 14 days to evaluate the color match of the composite resin in Class IV restorations.

SUMMARY

Rehydration of the tooth remnant and complete polymerization of the composite resin are aspects that should be considered in shade selection of composite resin. This article presents a case series of Class IV restorations performed to evaluate the color match between the composite resin and the tooth remnant. Thirteen Class IV restorations were performed in maxillary central incisors and evaluated according to the period following the restorative procedure: 10 minutes (baseline), 48 hours before and after finishing and polishing, and seven, 14, and 28 days. The color match of the restorations was evaluated by the ΔE values of the tooth remnant (TR) and the composite resin (CR) in each evaluation period using a spectrophotometer. The translucency, luminosity, and saturation were analyzed qualitatively in digital photographs of the restorations. The CR ΔE was statistically similar to the TR ΔE at 14 and 28 days ($p>0.05$). The saturation and luminosity of most of the restorations remained unchanged, but there was a greater change in the translucency. The color match of the composite resin in Class IV restorations was observed after 14 days of clinical assessment in this case series.

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INTRODUCTION

The reproduction of the optical characteristics of natural teeth is a challenge in anterior composite restorations.[1,2] The shade selection is carried out subjectively with a visual shade guide and generally under inadequate light conditions, which can lead to errors in color perception. It should be noted that color match in anterior composite restorations is obtained by rehydration of the tooth remnant and

complete polymerization of the composite resin, aspects that should be considered during restorative treatment.

Tooth dehydration occurs in restorative procedures because of operative field isolation. The absence of saliva makes the tooth lighter due to a change in the light refractive index. However, after removing the field of isolation, the tooth rehydrates and gradually returns to its original optical condition due to saliva absorption.[3-6]

Composite resin changes color after light curing due to a cross-linked polymerization reaction.[7-10] It is known that the conversion of monomers into polymers is about 75% in the first 10 minutes after photoactivation[11] and can increase after 24 hours.[12,13] Thus, the composite color change occurs during curing and as a postpolymerization reaction due to monomer conversion[7,9,14,15] and can persist over time due to water sorption of the materials.[8,10,13,16-20]

One method that can be used to evaluate the color match of the composite resin restoration is to measure the color of both the tooth remnant and the composite restoration with a spectrophotometer and then compare their respective values. A spectrophotometer indicates values of L*, a*, and b* in the CIELab color system. The L* parameter corresponds to the luminosity, whereas a* and b* correspond to the hue. The a* axis represents the red-green axis saturation and b* the blue-yellow saturation. The color difference (ΔE) is obtained from the individual changes in each parameter and is expressed as a single value.

It should be pointed out that because of tooth hydration and composite color change, a mock-up should be performed as a trial to assist in shade selection and ensure the predictability of the esthetic result in restorative treatment.[21,22] Furthermore, no clinical data are available to determine how long the mock-up should be kept in the mouth to evaluate the color match of the composite resin in Class IV restorations. Therefore, this article presents a case series of Class IV restorations performed to evaluate the color match of composite resin with the tooth remnant in Class IV restorations using spectrophotometric analysis and digital photographs.

CASE SERIES

Nine patients (four males and five females) aged between 20 and 30 years were randomly chosen among by two operators in the operative dentistry postgraduate clinic at the Federal University of Santa Catarina. The Institutional Review Board of the Federal University of Santa Catarina, Brazil, approved the study (protocol 1.197.856). Adults in need of restoration of at least one maxillary central incisor were included in the study.

In some cases, a diagnostic wax-up was made before conducting the restoration to obtain the palatal silicon matrix (Express XT, 3M ESPE, St Paul, MN, USA) used to layer the restorative material. After prophylaxis with prophylactic paste and a nylon brush, the shade selection was performed with a proprietary dual shade guide system, and the mock-up was used to confirm the esthetic appearance of the restoration. The mock-up was fabricated with the selected shades for each layer and was placed without etching; only the adhesive was applied. After three days, the mock-up was removed, and the teeth were restored with a nanohybrid light-activated composite resin (IPS Empress Direct, Ivoclar Vivadent, Schaan, Liechtenstein). First, the tooth remnant was etched with 37% phosphoric acid for 15 seconds on the dentin and 30 seconds on the enamel, extending 1 mm beyond the preparation margins. After the dentin was rinsed, it was protected with a cotton pellet, and the enamel was air-dried. A Single Bond Universal adhesive system (3M ESPE) was applied with a disposable brush (Microbrush, Coltène/Whaledent, Inc, Cuyahoga Falls, OH, USA) according to the manufacturer's instructions. The cavity was photocured for 20 seconds with an LED unit (Translux Blue, Heraeus Kulzer, Hanau, Germany) with a light intensity of 876 mW/cm^2 and calibrated in the beginning of the study.

The restoration was performed using the layering technique with the composite shades selected previously and confirmed with the mock-up. No bevel preparation was performed. See Figure 1 for the layering technique insertion sequence.[22] The finishing and polishing procedure was performed 48 hours after completing the restoration. The procedure was initiated with abrasive strips (3M ESPE) on the proximal surfaces. The facial surfaces were finished with sequential polishing discs of decreasing grit size (Sof-Lex Pop On, 3M ESPE), to improve the surface smoothness and remove small areas of excess at the interface. A carbide bur (FG 7664F, KG Sorensen, São Paulo, Brazil) was used to remove excess resin from the preparation margin. The surface texture was reproduced with a flame-shaped and tapered carbide burs (KG Sorensen) when it was needed. A felt disc (Diamond Felt Disc, FGM, Joinville, Brazil) with diamond polishing paste (Diamond Excel, FGM) was used to perform the final polishing.

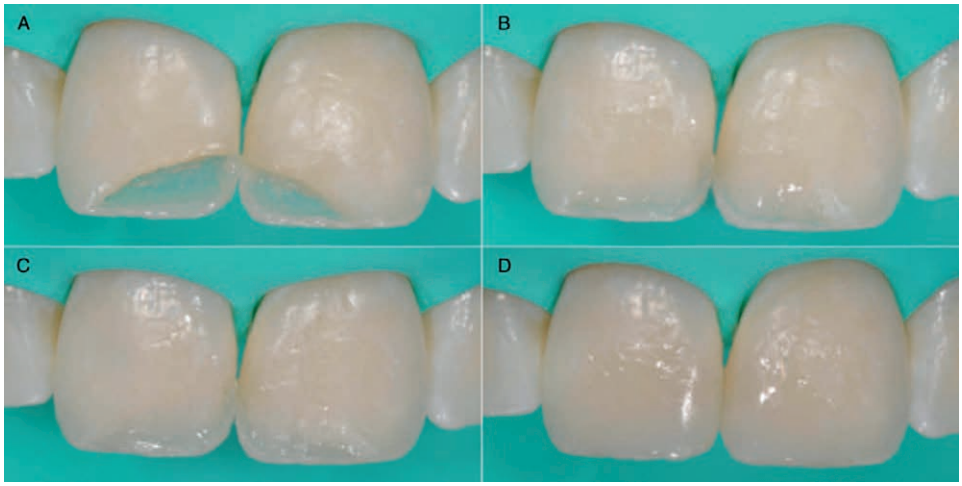

Figure 1. *Stratification of the resin composite restoration. (A): Reproduction of palatal enamel and opaque halo. (B): Reproduction of dentin. (C): Reproduction of the opalescent halo. (D): Reproduction of the facial enamel.*

A total of nine subjects with 13 Class IV restorations were enrolled. Two trained operative dentistry operators performed all the restorative procedures. The composite resin restoration and tooth remnant colors were measured with a spectrophotometer (Vita Easyshade, Vident, Brea, CA, USA) at different evaluation periods (n=13): 1) 10 minutes after completing the restoration (baseline), 2) 48 hours before finishing and polishing, 3) 48 hours after finishing and polishing, 4) seven days after the restorative procedure, 5) 14 days after the restorative procedure, and 6) 28 days after the restorative procedure. The remaining tooth area and the restoration were standardized for shade taking with two silicone matrixes (Express XT, 3M ESPE) fabricated for each patient: matrix 1 (a perforation in the tooth remnant region) and matrix 2 (a perforation in the central area of the restoration) (Figures 2 and 3). The perforation was compatible with the size of the device tip (6-mm diameter) and was made using a metallic device with well-formed borders. All assessments were made by an independent and calibrated evaluator. Evaluations were performed using the silicone matrixes positioned in the tooth, and spectrophotometric analysis was conducted without any colored background. Figures 4 through 9 represent clinical cases of restored teeth showing the before and after clinical images.

The color difference (ΔE) at each evaluation period was obtained from the L* a* b* values for each tooth and restoration according to the following formula:[23,24] $\Delta E = [(\Delta L)^2+(\Delta a)^2+(\Delta b)^2]^{1/2}$, where ΔL = L final − L initial, Δa = a final − a initial, and Δb = b final − b initial. Initial values were obtained at the baseline evaluation (10 minutes). The nonparametric Mann-Whitney test was used to compare the ΔE

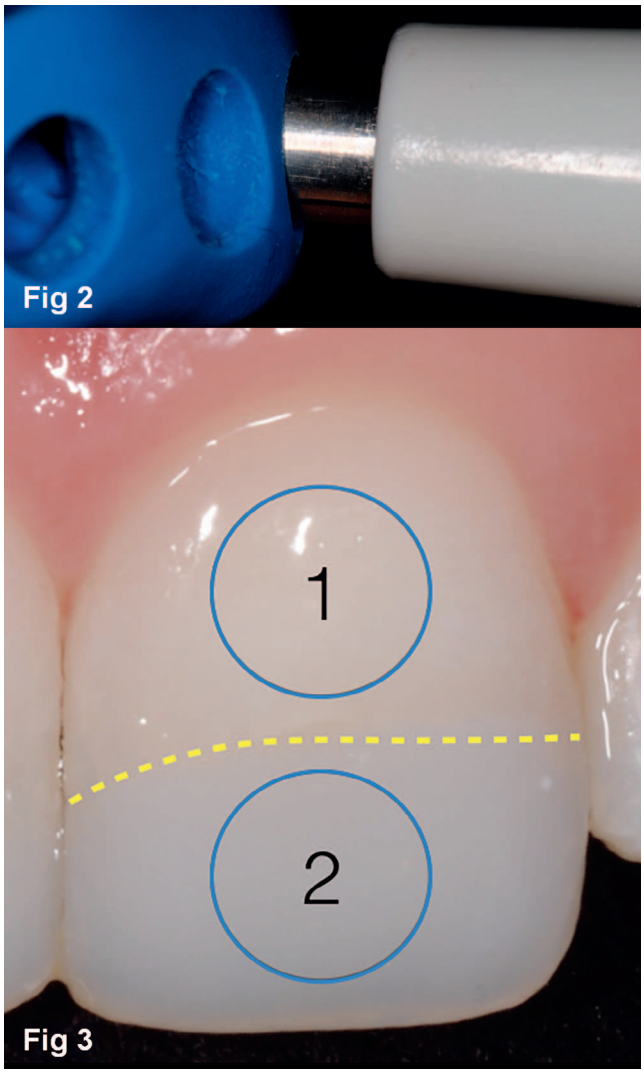

Figure 2. *Tip of the spectrophotometer placed in the labial perforation of the silicone matrix. Two matrixes were made for the same tooth. The first was used for individual evaluation of the tooth remnant and the second for the restoration.*

Figure 3. *Illustration of the measurement sites on the tooth remnant (1) and the restoration (2).*

Figure 4. *(A): Photo of the case 1 before treatment. (B): Photo of the case 1, 28 days after restorative procedures.*

Figure 5. *(A): Photo of the case 2 before treatment. (B): Photo of the case 2, 28 days after restorative procedures.*

Figure 6. *(A): Photo of the case 3 before treatment. (B): Photo of the case 3, 28 days after restorative procedures.*

Figure 7. *(A): Photo of the case 4 before treatment. (B): Photo of the case 4, 28 days after restorative procedures.*

Figure 8. *(A): Photo of the case 5 before treatment. (B): Photo of the case 5, 28 days after restorative procedures.*

Figure 9. *(A): Photo of the case 6 before treatment. (B): Photo of the case 6, 28 days after restorative procedures.*

values of the composite resin with those of the tooth remnant at each evaluation period. All the analyses were conducted at a significance level of 5% (α=0.05) using SPSS statistical software (version 20.0 for Mac, SPSS Inc, XXXX).

When the mean ΔE values of the composite resin were compared with those of the tooth remnant according to the evaluation period, a statistically significant difference was observed at the following evaluation periods: 48 hours (before polishing), 48 hours (after polishing), and seven days after the restorative procedure ($p<0.05$). At the 14-day and 28-day periods, the ΔE values of the composite were statistically similar to those of the tooth remnant ($p>0.05$) (Table 1).

Qualitative analysis of the restoration color was carried out with a digital photograph at each evaluation period. A digital camera (D90, Nikon, Tokyo, Japan) with a medical 120-mm lens (Nikon Sigma, Ronkonkoma, NY, USA) and configured at V125, ISO 150, and F32 was used to standardize the images. The images were assembled in a computer program (Keynote, Apple, Cupertino, CA, USA) and analyzed by an examiner according to translucency, saturation, and luminosity. Analysis of these criteria was best achieved by comparing the photographs in pairs according to the evaluation periods. In this procedure, an examiner compared the image of the analyzed period with the image of the previous period. Scores were established for each criterion: A (equal), B (decreased), and C (increased). These scores were respectively assigned to each restoration, after which the number of restorations for each score was obtained.

Table 1: *Mean and Standard Deviation Values of ΔE for Tooth Remnant and Composite Resin According to the Evaluation Period* [a]

Period of Evaluation	Tooth	Composite Resin	*p*-Value
48 h (before polishing)	4.94 ±1.36 A	3.69 ±0.99 B	0.011
48 h (after polishing)	4.50 ±1.53 A	3.15 ±0.90 B	0.015
7 d	4.36 ±1.26 A	3.09 ±0.73 B	0.005
14 d	4.62 ±1.36 A	3.98 ±1.46 A	0.209
28 d	3.84 ±1.12 A	3.07 ±1.13 A	0.058

[a] *Comparisons are valid only within rows. Means with identical letters on the same line for each period of evaluation are not statistically different (Mann-Whitney, p>0.05).*

The qualitative analysis indicated that saturation was equal during the 28-day period in most restorations. Furthermore, the luminosity remained unchanged in most restorations; however, in the cases with a significant amount of restoration, luminosity decreased during the 48 hours prior to finishing and polishing and during 14 days and also during 28 days, after which it increased. Moreover, 48 hours before finishing and polishing, translucency increased in most of the restorations and remained the same in the following periods. After seven and 14 days, the translucency remained unchanged in half of the restorations and increased in the rest, whereas at 28 days, most of the restorations presented the same translucency as the previous period (Table 2).

Table 2: *Number of Restorations (n) and Score Results for Saturation, Translucency, and Restoration Luminosity According to the Evaluation Period*[a]

Score / Evaluation Period	n	A	B	C	A	B	C	A	B	C
		Saturation			Translucency			Luminosity		
48 h (before polishing)	13	10	3	—	4	—	9	8	5	—
48 h (after polishing)	13	12	1	—	6	3	4	12	—	1
7 d	13	12	1	—	6	1	6	11	2	—
14 d	13	11	2	—	5	3	5	8	4	1
28 d	13	10	—	3	9	2	2	8	—	5

[a] *Score key: A (equal), B (decreased), C (increased).*

DISCUSSION

Composite resin color change is usually evaluated under laboratory conditions without considering the color match between the composite and the tooth remnant structure. The results of this study suggest that the color match of the composite resin in Class IV restorations occurred 14 days after the restorative procedure, considering that there was a statistical similarity of ΔE values between the composite resin and the tooth remnant at the evaluation periods of 14 and 28 days.

This result is probably attributed to the tooth rehydration time[3-5] and the postpolymerization period of the composite.[8-10,17,19] Teeth with excessive dehydration recover their color after a period ranging from one month[6] up to 12 months due to water absorption in the mouth.[4,5] Thus, after completing Class IV restorations, the teeth gradually return to their original color over time. With respect to composite resin, laboratory studies show that different composite resins change color after photoactivation,[7-9] after water storage,[8,10,18,19] after thermocycling,[17] and after artificial aging.[13,15,16,20] This composite color change may be related to the color of the materials used and depends on the brand.[7,8,10,16,17,20]

The color match in Class IV restorations is also influenced by small variations in the thickness of the composite layers since the composite used to reproduce the enamel is more translucent than the dentin composite.[22,25] A thick translucent composite on the facial surface makes the restoration grayish because of differences in the index of light refraction of natural enamel and that of the restorative material.[26] In this study, the restorations were made with a minimum thickness of enamel composite, and the thickness of the artificial dentin provided adequate opacity without interfering with the restoration value.

The layering of the resin composite was conducted in this study based on the concept of natural stratification, which proposes the combination of optical properties from different resin layers,[22] and it allows achieving the esthetic result without needing a bevel.[27,28] Also, the finishing and polishing procedure was performed 48 hours after the restoration because as the degree of cure continues after the initial polymerization,[12] the delayed polishing time may improve the composite surface roughness and microhardness.[29,30]

Luminosity, saturation, and translucency are the color dimensions that most influence the appearance of natural teeth.[1,2,21] The results of the qualitative analysis showed that the saturation of most restorations remained unchanged over 28 days. Moreover, the luminosity of most restorations remained unchanged for 28 days. However, there was an increasing tendency for greater translucency in most restorations and a consequent reduction in luminosity, shown by some restorations over time, considering that greater translucency of composite dentin may reduce the luminosity of the restorations.[22,31]

The current study showed that the color difference (ΔE) of the composite among the evaluation periods ranged from 3.07 to 3.69 and that that of the tooth remnant ranged from 3.84 to 4.94 among the same periods (Table 1). Some of these color shifts go over the threshold value (ΔE=3.3) and may be noticeable.[32,33] This may attributed to the baseline color being measured 10 minutes after completion of the restoration, at which time the tooth may be dehydrated[3] and the composite not completely polymerized;[11] this could explain the expressive color differences between the natural tooth and the composite. Yet the resin composite colors used in this study were brighter colors: DA1, DB1, DBL-L, DBL-XL, EB1, EBL-L, and EBL-XL (IPS Empress Direct). Is it important to consider that the more luminous or less chromatic the

composite resin, the greater the tendency to change color.[14,16,18,20]

Despite the different color values obtained, the aim of this study was to evaluate the color match of composite resin with the tooth remnant at the evaluation periods and not the color change of the composite or of the tooth remnant over time. Future randomized double-blinded clinical studies with a greater number of restorations and evaluators are needed to confirm the results obtained.

CONCLUSION

A minimum period of 14 days was needed for the color match of composite resin in Class IV restorations. There was an increase in translucency at the 28-day time period; however, the restorations remained clinically satisfactory. Accordingly, it could be recommended that a mock-up be performed and kept for at least 14 days to check the shade selection.

Acknowledgement

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Ethics Committee of the Federal University of Santa Catarina. The approval code for this study is 1.197.856.

Conflict of Interest

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

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Use of a Modified Matrix Band Technique to Restore Subgingival Root Caries

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Clinical Relevance

A modified matrix technique used in combination with an RMGI restorative material is an effective approach for treating subgingival root caries.

SUMMARY

Given the increasing incidence of root caries in the elderly population, clinicians frequently must isolate and restore subgingival preparations. This article demonstrates a technique utilizing a modified Tofflemire matrix band that creates a preparation free of crevicular fluid and blood for restoration with resin-modified glass ionomer cement.

INTRODUCTION

Root caries have become one of the most significant patient management problems in the elderly population because these patients frequently exhibit xerostomia, often medication induced, which increases the incidence of root caries.[1]

Given the lack of enamel in cervical areas, resin-modified glass ionomer (RMGI) materials are an ideal choice for such restorations.[2-4] A recent systematic review found that RMGI restorations bond more effectively and durably in root surface restorations than do other adhesive materials,[5] while other studies demonstrate reasonable success in patients with xerostomia.[6,7] Unfortunately, RMGI restorative materials are difficult to sculpt when used for large restorations, which can result in extensive finishing being necessary. Further, cervical caries that extend subgingivally are particularly challenging to isolate because the tissue is usually inflamed and bleeds easily. This must be controlled to adequately place any adhesive restoration.[8]

Rubber dam isolation using a 212 clamp, placement of retraction cord, and minor periodontal surgery are effective adjuncts in achieving access in subgingival restorations,[9] but these can be complex and time consuming. Often, use of a customized matrix band offers a simpler approach to attaining isolation and minimizing finishing of RMGI restorations. Manufacturers produce a variety of prefabricated matrix shapes for this purpose, but these must be added to practice inventory. Modification of a matrix band already in inventory is

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simpler, and several authors have suggested the use of a modified Mylar strip for cervical RMGI restorations.[10,11] We favor a modified metal matrix band, which can be readily contoured.

The purpose of this clinical technique article is to present a simple modification of a metal matrix band that greatly facilitates isolation and minimizes the finishing needed for subgingival Class V restorations with RMGI materials.

CLINICAL CASE

Patient Description

A 60-year-old Caucasian male presented to the student clinic at the Dental College of Georgia, Augusta University, seeking comprehensive dental treatment. On review of his medical history, he was classified as ASA III (severe systematic controlled disease) due to diabetes, hypertension, and history of heart attack. The clinical exam revealed the following: all mandibular molars and teeth numbers 5 and 6 missing, generalized moderate-to-severe chronic periodontitis, generalized recession, gingival inflammation and bleeding, occlusion only on posterior teeth with an irregular occlusal plane. At the time of the exam, his caries risk was high because of multiple carious lesions, many missing and restored teeth, as well as fair oral hygiene and xerostomia due to medications (Figure 1).

Caries Management

The patient underwent scaling and root planning and was scheduled for extractions. It was recommended that he decrease the frequency with which he ingested carbohydrates and use a 5000 ppm NaF toothpaste and xylitol gum. Root caries were restored with RMGI restorations.

Treatment of Left Maxillary Lateral Incisor

The tooth shade was determined to be A3. Anesthesia was attained by infiltration with lidocaine HCl 2% with epinephrine 1:100,000 (Xylocaine, Septodont, Lancaster, PA, USA). Isolation was done with cotton rolls. Ultrapack retraction cord #00 (Ultradent Products, Inc, South Jordan, UT, USA) was placed in the facial gingival sulcus during cavity preparation because the lesion was at the level of the gingiva. Cavity preparation was initiated with a No. 2 carbide bur (SS White, Lakewood, NJ, USA) using a high-speed handpiece under constant water cooling, and caries removal was completed with a No. 6 latch-type carbide bur at slow speed (Figure 2).

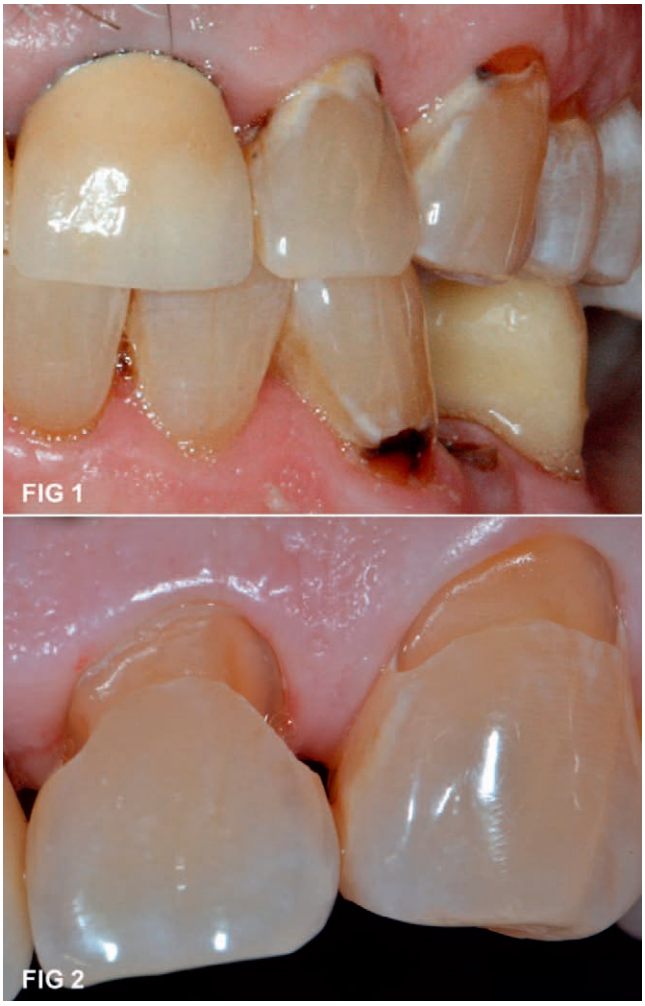
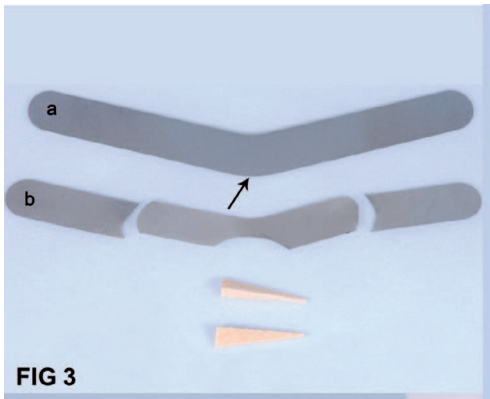


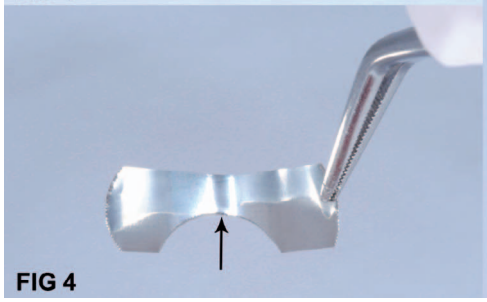
Figure 1. *Initial root caries.*

Figure 2. *Root caries prepared.*

Modified Matrix Technique

To form the custom matrix for this restoration, the center of the occlusal side of a universal Tofflemire No. 1 matrix band (JR Rand Corp, Deer Park, NY, USA) (Figure 3a, arrow) was trimmed with fixed-curve crown scissors to a width of 3 mm using a curvature opposite that of the band on that side. The two ends of the matrix band were then trimmed by 1.5 cm each (Figure 3b). The modified matrix was then molded using finger pressure to form the convex, arch-shaped facial contour of the tooth. The trimmed occlusal portion of the modified band became the gingival side of the custom matrix (Figures 4 and 5, arrow). Before placing the matrix, the retraction cord was removed. The modified matrix was then placed around the prepared tooth and into the gingival sulcus where necessary. Two small Wizard wooden wedges (Prestige Dental Products, Inc, Anaheim, CA, USA) were placed on





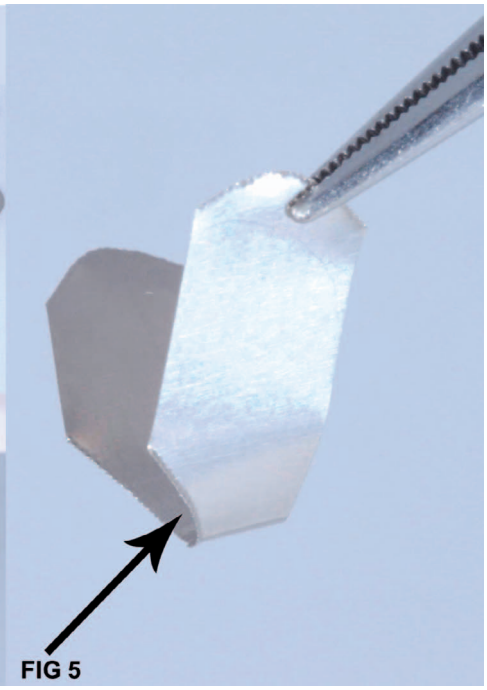


Figure 3. *Universal Tofflemire band. a. Arrow indicates the occlusal portion and area to be trimmed. b. Tofflemire band modified and wooden wedges.*

Figure 4. *Modified Tofflemire band. Arrow indicates concave area of the newly trimmed band, which will be placed toward the gingiva.*

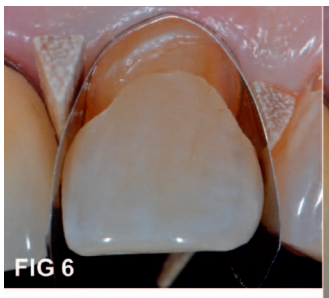
Figure 5. *Proximal view of modified Tofflemire band, which was curved using finger pressure. Arrow indicates gingival side.*

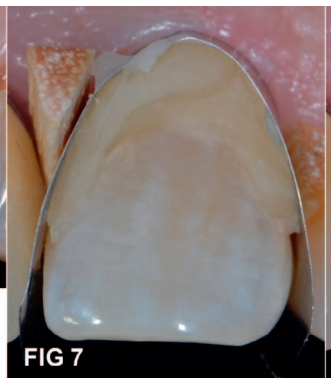
the mesial and distal sides to hold the matrix firmly in place and to seal the preparation against fluid contamination (Figure 6).

Restoration

The cavity surface was scrubbed with GC cavity conditioner (polyacrylic acid/aluminum chloride) with a microbrush for 10 seconds according to the manufacturer's instructions, then rinsed and lightly air dried. An A3 shade, resin-modified glass ionomer capsule (Fuji II LC, GC America, Alsip, IL, USA) was activated and mixed for 10 seconds at 4000 cpm, then the material was injected into the internal form of the cavity preparation from distal to mesial, allowing excess to cover the margins and producing an approximately 30% overcontour, and light cured for 20 seconds with an LED light (Valo, Ultradent; Figure 7). After light curing the RMGI but before finishing, the matrix was removed with a cotton forceps (Figure 8). A finishing diamond bur (FSD4, Komet USA, Rock Hill, SC, USA) was used to obtain final contours. Excess RMGI was removed from the gingival embrasures using thin 0.078″ finishing strips (Sof-Lex, 3M, St Paul, MN, USA). Medium and fine polishing discs (Sof-Lex XT, 3M) were used to obtain smooth surfaces and transitions (Figure 9).

Figure 10 shows the restorations after 2 years. The patient's treatment, which included removable partial dentures, was completed. Restoration of the left maxillary lateral incisor was intact, but some interproximal enamel decalcification was evident.





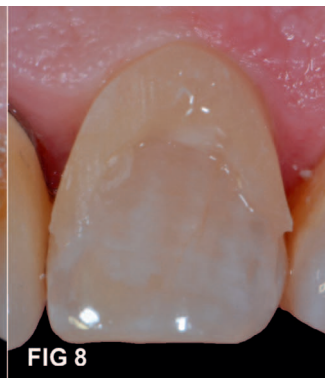
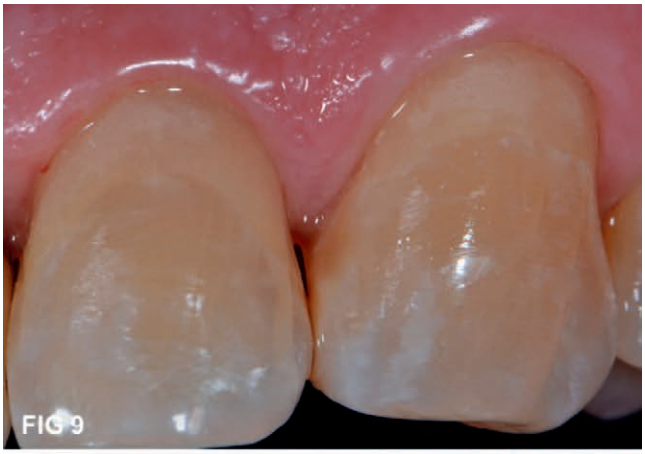


Figure 6. *Modified Tofflemire band and wooden wedges placed around preparation.*

Figure 7. *Resin modified glass ionomer in place.*

Figure 8. *Band and wedges removed.*



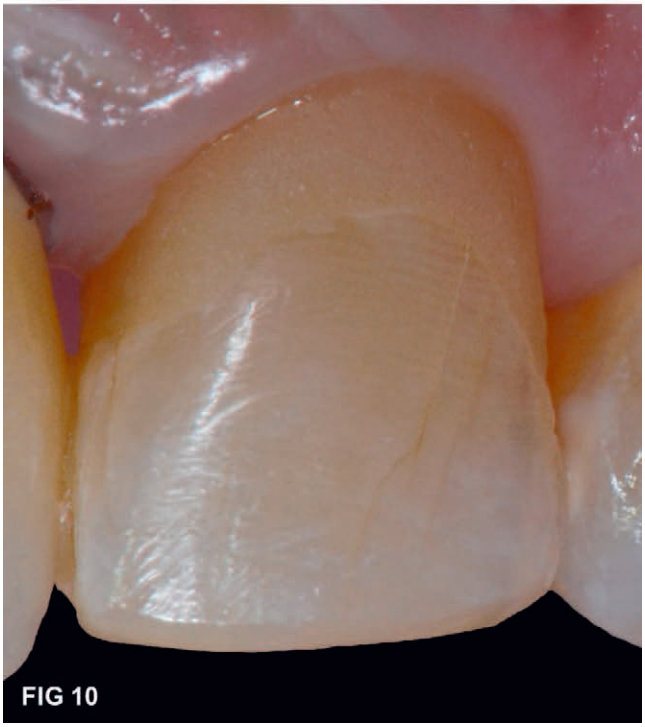


Figure 9. *Final restoration.*

Figure 10. *Restoration 2 years later.*

DISCUSSION

Restoring Class V subgingival preparations on buccal and lingual surfaces may be challenging if the lesion is extensive apically and interproximally, making it difficult to place an adequate restoration. The modified Tofflemire matrix band technique in combination with an RMGI restorative material was a good approach in this clinical case. An advantage of this technique is confinement of the restorative material in the matrix band, which produces a minimum of overhangs and proper interproximal contours; therefore, minimal finishing is required.

The advantage of using RMGI is that the materials can be applied in a bulk-fill technique without compromising marginal integrity. While these materials are not as smooth and translucent as enamel,[12] they can be overlaid with resin composite if desired.[13]

Treatment of high-risk patients such as this should include not only restorative measures but a well-designed caries-risk-management protocol and oral hygiene instructions.[14] After 2 years, the patient presented with interproximal enamel decalcification lesions (Figure 10), indicating that future caries management steps should be undertaken to increase effectiveness.

CONCLUSION

The modified matrix technique, combined with an RMGI restorative material, was an effective approach for treating the subgingival root caries of this patient.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Dental College of Georgia, Augusta University.

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, or company s presented in this article.

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Clinical Research

Preventive Use of a Resin-based Desensitizer Containing Glutaraldehyde on Tooth Sensitivity Caused by In-office Bleaching: A Randomized, Single-blind Clinical Trial

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Clinical Relevance

Application of Gluma Desensitizer prior to bleaching provides no significant decrease in sensitivity.

SUMMARY

Objective: To evaluate the risk and intensity of bleaching-induced tooth sensitivity (TS) after in-office bleaching following topical application of a resin-based glutaraldehyde desensitizer.

Methods: Thirty-three patients were randomly assigned to the experimental (Gluma Desensitizer Liquid, Heraeus Kulzer, Hanau, Germany) and placebo groups. The placebo or Gluma Desensitizer Liquid was applied for one minute prior to application of an in-office bleach-

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ing gel. Bleaching was performed with 35% hydrogen peroxide gel (three applications × 15 minutes each) over two sessions, one week apart. The color of the anterior teeth was evaluated before and 21 days after treatment using the VITA Classical shade guide, Bleachedguide 3D, and Easyshade spectrophotometer. TS during and after the bleaching was recorded according to the visual analog (VAS) and numerical rating (NRS) scales. All data were submitted to statistical analysis (α=0.05).

Results: There was no significant difference in absolute risk or intensity of TS between the two groups (risk and VAS, p=0.93 and 0.31, respectively; NRS, $p \geq 0.45$). At the end of the bleaching protocol, tooth whitening was observed in both groups, as evident from color change in shade guide units (ΔSGU, 4.1-7.1; both guides) and overall color change (ΔE, 7.4-9.3 units); however, there were no significant differences in whitening between the two groups ($p > 0.11$).

Conclusion: Gluma Desensitizer Liquid was not able to reduce the risk or intensity of TS. Bleaching efficacy was not affected by application of the desensitizer.

INTRODUCTION

Dental bleaching is a popular procedure for treatment of discolored teeth.[1-8] It is a very conservative, simple, and low-cost procedure.[8] Unfortunately, tooth sensitivity (TS) is a common side effect of bleaching, particularly with in-office bleaching protocols that employ relatively high concentrations of hydrogen peroxide.[4-6]

The incidence of TS after in-office bleaching has been reported to be relatively high.[1,3,8-11] In addition, while the intensity of TS after in-office bleaching has been reported as usually being moderate,[7,12,13] in some cases it is severe and irritating enough to cause patients to withdraw from treatment.[14] Although the mechanism of bleaching-induced TS is not well understood,[15] it seems to result from the passage of hydrogen peroxide through hard tissues to the pulp, where it induces a reversible inflammatory process[16,17] and might also directly stimulate nerves, leading to pain.[18]

Several approaches have been proposed to minimize this side effect caused by bleaching products. As reported in a recent systematic literature review, preemptive administration of oral drugs such as analgesics, anti-inflammatories, antioxidants, and corticosteroids[3,9,19,20] has been found to be ineffective in minimizing the risk or intensity of bleaching-induced TS.[21] Similarly, reports on the effect of topical application of potassium nitrate, fluorides, and remineralizing agents on in-office bleaching-induced TS are conflicting.[22-26]

Glutaraldehyde-based products exhibit satisfactory performance as desensitizing agents in treatment of dentin hypersensitivity as well as upon prior application in restorative procedures.[27-29] A recent study on the effect of prior application of a resin-based glutaraldehyde desensitizer gel (Gluma Desensitizer Power Gel, Heraeus Kulzer, Hanau, Germany) on in-office bleaching-induced TS[11] reported significant reduction of bleaching-induced TS during and after in-office whitening.[11]

In contrast, in a study on the effect of prior application of different desensitizing agents—including a fluoride varnish, sealant, and resin-based glutaraldehyde desensitizer (Gluma Desensitizer Liquid, Heraeus Kulzer)—on in-office bleaching-induced TS in patients with dentinal hypersensitivity, Ibrahim and Banna[30] reported no significant differences in performance between the resin-based glutaraldehyde desensitizer and resin-based adhesive without glutaraldehyde (sealant). Although different types of TS have been evaluated in both studies, these conflicting results[11,30] indicate the necessity of more clinical studies for evaluating whether the presence of glutaraldehyde in desensitizers helps minimize in-office bleaching-induced TS. In addition, it is not clear whether application of different glutaraldehyde-containing substances influences the efficacy of tooth whitening, because only one study to date has presented relevant data in terms of color change.[11] Therefore, this randomized clinical study aimed to evaluate the risk of bleaching-induced TS (primary outcome) after in-office bleaching following topical application of a resin-based glutaraldehyde desensitizer when compared with a placebo group. In addition, the intensity of bleaching-induced TS and efficacy of in-office bleaching were evaluated as secondary outcomes.

The null hypotheses of the present study were 1) the use of resin-based glutaraldehyde desensitizer liquid and placebo groups will yield similar risks to bleaching-induced TS, 2) both groups will have a similar intensity of bleaching-induced TS, and 3) both groups will have the same bleaching effectiveness.

METHODS AND MATERIALS

The ethics committee of the local university approved this clinical investigation (Protocol No.

1.422.841). The research protocol was registered in the Brazilian clinical trials registry (No. RBR-7YRR3S). The experimental design was planned in accordance with the Consolidated Standards of Reporting Trials statement.[31] Based on pre-established criteria, 33 volunteers from São Luís, Maranhão, Brazil, were selected for this study. Two weeks prior to bleaching, all volunteers received dental screening and dental prophylaxis with pumice and water in a rubber cup and signed informed consent forms.

Study Design

This was a randomized, single-blind, split-mouth clinical trial, with equal probability of a participant receiving either of two treatments. The study was conducted between March and June 2016 at the clinic of the School of Dentistry, Ceuma University, São Luis, Brazil.

Inclusion and Exclusion Criteria

Patients included in this clinical trial were at least 18 years old and had good general and oral health. Participants were recruited by means of local advertisement. A total of 33 participants were examined for fulfillment of the inclusion and exclusion criteria while seated in a dental chair (Figure 1). The inclusion criteria were as follows: caries-free maxillary and mandibular anterior teeth without restorations on the labial surfaces and central incisors of shade A2 or darker, as judged by comparison with a value-oriented shade guide (VITA Lumin, VITA Zahnfabrik, Bad Sackingen, Germany). The exclusion criteria were as follows: history of tooth whitening, presence of anterior restorations, pregnancy or lactation, and severe internal tooth discoloration (eg, tetracycline stains, fluorosis, or pulpless teeth), bruxism, or any other pathology (eg, recession or dentin exposure) that could cause sensitivity. These criteria were so stipulated because the participants would not be immediately eligible for cosmetic treatment such as bleaching if they had other restorative needs that required immediate attention. A week prior to bleaching, the participants were questioned about previous TS according to the criteria described in the TS Evaluation section. Patients with preexisting TS were excluded from the study.

Sample Size Calculation

The primary outcome of this study was absolute risk of TS. It was determined that 33 patients would be required to ensure an 80% probability of detecting a decrease in mean absolute risk of TS from 85% in the control group[12,23,25,26] to 55% in the experimental group (α=0.05). Also, determination of sample size for a second outcome, TS intensity, was performed. For TS intensity, it was determined that 22 patients would be required to exclude a mean difference of 2 in the visual analog scale (VAS) scores with 80% power and 5% alpha, considering that the standard deviation of VAS score is approximately 2. The limit of equivalence (difference of means) was considered based on the results of the VAS score in clinical trials that evaluated in-office bleaching.[3,9,20,26] The sample size was calculated on the website www.sealedenvelope.com. This study was powered to detect a significant effect.

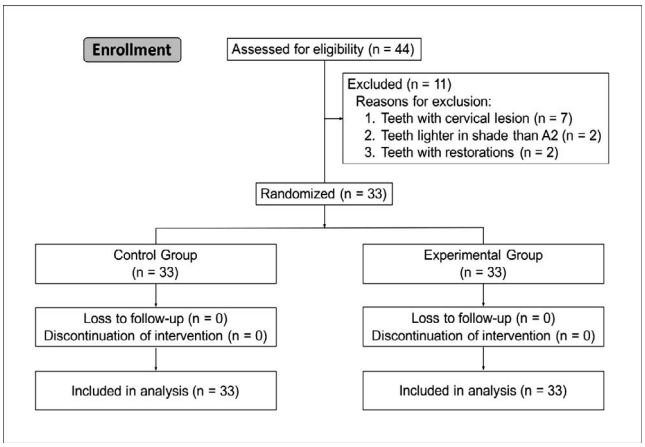

Figure 1. *Flow diagram of the clinical trial, including detailed information on excluded participants.*

Intervention

Randomization was performed using computer-generated tables prepared by a third party not involved in the study protocol. Details of group allocation were recorded on cards contained in sequentially numbered, opaque, sealed envelopes, prepared by a third party not involved in any phase of this clinical trial. To avoid disclosure of the randomization scheme, these envelopes were opened on the day of restorative intervention. Participants as well as the operator were blinded to group allocation and the study protocol.

The gingival tissue of teeth meant to be bleached was isolated using a light-polymerized resin dam (Top Dam, FGM Dental Products, Joinville, Brazil). For all patients, the left side received the treatment described first in the randomization list, while the right side received the treatment mentioned second. Either the placebo or experimental agent was applied to the buccal tooth surfaces and left

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

Product	Composition[a]	Application Regimen[a]
Gluma Desensitizer Liquid	2-hydroxyethyl methacrylate, glutaraldehyde, and purified water	1.Insert the lip retractor. 2.Apply the light-cured gingival barrier and perform light curing. 3.Actively apply the desensitizer on the labial surfaces of teeth with the aid of a micro-brush (10 seconds for each tooth). 4.Leave the product in contact with the labial surface for 60 seconds. 5.Dry the surface carefully by applying a stream of compressed air until the fluid film has disappeared and the surface is no longer shiny. 6.Rinse with water (10 seconds).
Whiteness HP Maxx 35%	35% hydrogen peroxide	1.Mix the two gel phases in a 3:1 ratio (drops) of peroxide to thickener. 2.With the aid of a nozzle, spread the gel on the buccal surfaces of teeth, forming a 0.5- to 1-mm-thick layer. 3.Leave the gel in for 15 min, and stir the product every 5 minutes to release bubbles. 4.Aspirate excess gel, wash the teeth, and repeat the application two more times (a total of three applications, amounting to 45 min of contact with teeth). 5.Aspirate excess gel, wash the teeth, and remove the gingival barrier.

[a] *According to the manufacturer's indications.*

undisturbed for 60 seconds. Teeth were then lightly air-dried until the fluid film disappeared and the surface was no longer shiny. During application of the placebo or experimental agent, the contralateral side was covered with gauze to avoid contact and mutual influence of the two treatments.

The experimental group was treated with Gluma Desensitizer Liquid (Heraeus Kulzer). Details regarding composition and mode of application are described in Table 1. The placebo had the same composition as the desensitizing agent, except for the absence of the active ingredients (ie, resin monomers and glutaraldehyde). The desensitizing and placebo agents were provided in bottles marked only with numbered codes that neither the clinicians nor patients could identify. Unfortunately, because of the peculiar smell of glutaraldehyde, the clinicians and patients were able to differentiate between the two treatments over the course of the study.

All participants received bleaching with 35% hydrogen peroxide gel (Whiteness HP 35, FGM Dental Products) according to the manufacturer's instructions (Table 1). Bleaching was performed over two sessions, a week apart. Participants were instructed to brush teeth regularly using toothpaste without desensitizing or bleaching agents.

Shade Evaluation

The 16 tabs of the VITA Classical shade guide were arranged from the highest (B1) to the lowest (C4) value. Although this scale is not linear in the truest sense, the shades were treated as representing a continuous and approximately linear ranking for the purpose of analysis. Change in tooth shade was calculated from the start of treatment to individual recall times according to the change in shade guide units (ΔSGU) from the darker toward the lighter end of the value-oriented list. Color was recorded at baseline, a week after the first and second bleaching sessions, and 21 days after the first bleaching session, using the VITA Classical shade guide, Bleachedguide 3D, and Easyshade Advance 4.0 spectrophotometer. Color evaluation was performed in a room under artificial lighting conditions, without interference from outside light. According to the American Dental Association guidelines, the mid-third of the labial surface of anterior teeth (central incisors) was considered as the area of interest for shade matching.[32]

A preliminary impression of the maxillary arch was acquired using high-putty Coltoflax silicone putty (Vigodent S/A Ind. Com., Rio de Janeiro, RJ, Brazil) to serve as a standard guide for the spectrophotometer probe. A window of 3-mm radius was created at the mid-third of the labial surface of the molded silicone guide using a metallic device with well-formed borders. A single calibrated operator evaluated tooth color in all participants using the VITA Easyshade spectrophotometer (Easyshade, Vident, Brea, CA, USA) before and 21 days after bleaching. The L*, a*, and b* measurements were recorded, with L* representing a value from 0 (black) to 100 (white) and a* and b* representing the shade, where a* was measured along the red-green axis and b* along the yellow-blue axis. Variation in color between the two assessment periods (ΔE) was determined using the following formula:

Table 2: *Number of Patients Who Experienced TS at Least Once During the Bleaching Regimen in Both Groups Along With Absolute Risk and Risk Ratio*

Treatment		No. of Participants With TS		Absolute Risk[a] (95% CI)	Risk Ratio (95% CI)
		Yes	No		
HP 35%	Gluma	24	9	68 (52-81)	1.20 (0.92-1.58)
	Placebo	29	4	82 (67-92)	

Abbreviations: CI, confidence interval; HP, hydrogen peroxide; TS, tooth sensitivity.
[a] *McNemar test (p=0.93).*

Table 3: *Intensity of Tooth Sensitivity According to the VAS[a] and NRS[b] in Both Study Groups*

Time Assessment	VAS[a]		NRS	
	Gluma	Placebo	Gluma	Placebo
Up to 1 h	1.66 ± 2.5	1.67 ± 2.3	1 (0/1)	1 (0/2)
1-24 h	2.48 ± 2.9	2.50 ± 2.9	1 (0/2)	1 (0/2)
24-48 h	0.54 ± 1.3	0.65 ± 1.6	0 (0/0)	0 (0/1)

Abbreviations: NRS, numerical rating scale; VAS, visual analog scale.
[a] *Mean ± standard deviation: two-way repeated-measures analysis of variance and Tukey test (p>0.31).*
[b] *Median (interquartile range): Kruskal-Wallis and Mann-Whitney tests (p≥0.45). There were no statistically significant differences in values between the two measurement scales.*

$$\mathrm{DE} = \left[(\mathrm{DL}*)^2 + (\mathrm{Da}*)^2 + (\mathrm{Db}*)^2\right]^{1/2}.$$

Evaluation of TS

Patients were asked to record any perception of TS immediately, 1 hour, 24 hours, and up to 48 hours after each session using a five-point verbal rating scale (0 = *none*, 1 = *mild*, 2 = *moderate*, 3 = *considerable*, and 4 = *severe TS*) and a 10-cm VAS with *no pain* and *worst pain* at opposite ends. Given that bleaching was performed over two sessions, the worst scores reported in the two sessions were considered for statistical analysis. The data were arranged into two categories: overall percentage of patients who reported TS at least once during treatment (absolute risk of TS) and TS intensity at each assessment point.

Statistical Analysis

Statistical analysis involved all randomly assigned participants and was performed according to the intention-to-treat protocol.[31] Absolute risk of TS was compared between the two groups by McNemar's test (α=0.05). Relative risk of TS and the confidence interval for effect size were also calculated.

Because the data exhibited normal distribution, bleaching-induced TS intensity measured by VAS scores was compared between the two groups by two-way repeated-measures analysis of variance and Tukey test. However, TS intensity measured by numerical rating scale (NRS) scores did not exhibit normal distribution and was therefore compared between the two groups at each assessment point by the Kruskal-Wallis and Mann-Whitney tests.

Color changes (ΔSGU according to both guides and ΔE between baseline and 21 months after bleaching) were compared between the two groups by the Student *t*-test. The significance level for all statistical tests was set at 0.05. All analyses were performed with SigmaPlot version 11.0 (Systat Software Inc, San Jose, CA, USA).

RESULTS

Participant Characteristics

A total of 44 participants were examined for verification of eligibility for the present study (Figure 1). There was no significant difference in baseline tooth color between the two treatment groups (placebo, 5.90 ± 2.6; experimental, 6.4 ± 2.7). The mean age of the participants was 23 ± 4 years (range, 18-40 years), and 55% of the patients were female.

Adherence to Protocol and Loss to Follow-up

All participants attended the recall visit 21 days after bleaching. Figure 1 presents the flow diagram for participant selection in different phases of the study.

Tooth Sensitivity

There was no significant difference in absolute risk of TS between the two groups (p=0.93; Table 2). With regard to intensity of TS, there was no significant difference between the two groups at any of the time points (VAS, p=0.31; NRS, $p\geq$0.45; Table 3).

Color Evaluation

The results of subjective and objective evaluation revealed significant whitening in both study groups. At the end of the bleaching protocol, both groups exhibited tooth whitening corresponding to 4.1 to 7.1 SGU (according to both guides), with ΔE ranging from 7.4 to 9.3 units (Table 4). The results of

Table 4: *Color Change in SGU (ΔSGU) and Overall Color Change (ΔE) From Baseline to 21 Days After Bleaching in the Two Treatment Groups*[a]

Color Evaluation Tool	Gluma	Placebo	*p* Value[b]
ΔSGU (Vita Classical)	5.0 ± 2.7	4.1 ± 2.5	0.12
ΔSGU (Vita Bleachedguide 3D)	7.1 ± 3.2	6.6 ± 3.6	0.55
ΔE	7.4 ± 3.6	9.3 ± 4.5	0.11

Abbreviation: SGU, shade guide units (Vita Classical and Vita Bleachedguide).
[a] *Values are presented as mean ± standard deviations.*
[b] *Student* t*-test paired.*

subjective (VITA Classical shade guide, $p=0.12$; VITA Bleachedguide 3D, $p=0.55$) and objective (spectrophotometry, $p=0.11$) evaluation supported the hypothesis of equivalence in treatment outcomes between the two groups after bleaching.

DISCUSSION

As mentioned in the introduction, topical application of several chemical agents has produced conflicting results in terms of its effect on TS due to in-office bleaching.[22-26] Therefore, it is important to evaluate alternative desensitizer agents that can reduce in-office bleaching-induced TS. Thus, the main purpose of this study was to evaluate if preventive application of a resin-based desensitizer liquid containing glutaraldehyde and hydrophilic monomers (Gluma Desensitizer Liquid) could reduce TS induced by bleaching.

Glutaraldehyde is a well-known biological fixative. There are several theories on the mechanism by which glutaraldehyde decreases dentin hypersensitivity.[33-36] The most accepted mechanism of action was elucidated by Schüpbach and others,[37] who suggested that, upon topical application of glutaraldehyde on the dentin surface, the reaction between glutaraldehyde and plasma proteins in dentin leads to precipitation of the latter. The results of a subsequent spectroscopic study confirmed that glutaraldehyde reacts with plasma proteins such as albumins and causes proteins to precipitate.[38] Another study demonstrated a reaction between glutaraldehyde–cross-linked albumin and the hydrophilic monomer (HEMA) present in the Gluma Desensitizer Liquid, resulting in the formation of a mixture of polyHEMA molecules.[38] These precipitates occlude open dentinal tubules beneath the surface, thus interfering with the hydrodynamics of dentinal fluid and preventing dentin sensitivity.[27-29]

However, it is necessary that glutaraldehyde and HEMA be able to penetrate through enamel and dentin along the same pathway as peroxide radicals.[39,40] Unfortunately, to the best of our knowledge, no study to date has evaluated transenamel penetration of glutaraldehyde or HEMA; this aspect deserves to be addressed in future studies. A tentative explanation is that the molecular sizes of glutaraldehyde (molar mass, 100 $gmol^{-1}$) and HEMA (molar mass, 130.14 $gmol^{-1}$) are lower compared with those of other high-molecular-size substances that have been proven to penetrate through enamel and dentin.[41,42] Also, because Gluma Desensitizer is used as a liquid, the solubility and diffusion coefficient of the molecule, as measured by Fick's second law, facilitate its penetration inside the pulp.[43-45] Therefore, it is reasonable to conclude that both substances can reach the dentin-pulp complex.

Unfortunately, the results of the present clinical trial demonstrated that, in comparison with teeth submitted for placebo treatment, prior application of Gluma Desensitizing Liquid for one minute reduced bleaching-induced TS by only 15%; however, this difference was not statistically significant. Also, no significant difference was observed in the intensity of TS, leading us to accept the first and second null hypotheses.

This result is contradictory to that of a previous study that employed a very similar product (Gluma Desensitizer Power Gel) for pretreatment before in-office bleaching. The compositions of Gluma Desensitizer Power Gel and Gluma Desensitizer Liquid are very similar, with the exception of the presence of pyrogenic silicic acid in the former.[46,47] Several studies have demonstrated that, upon application on the dentin surface, the gel reduces dentin permeability as effectively as the liquid desensitizer,[48,49] especially upon application at the same time. Similar results were observed in the present study as well as in the study by Mehta and others.[11] However, there are substantial differences in the in-office bleaching protocol between the two studies: although Mehta and others[11] performed in-office bleaching for only 15 minutes, in-office bleaching in the present study was performed with three 15-minute applications. Unfortunately, comparison of the results of TS observed in the present study with the results of the previous study[30] is not possible, mainly because Ibrahim and El Banna[30] evaluated subjects with existing dentin sensitivity and applied the desensitizers on the dentin surface.

Previous studies have demonstrated that the higher the number of applications or the longer the application of in-office bleaching agents on enamel, the greater the extent of hydrogen peroxide pene-

tration to the pulp chamber[39,40] and the more intense the adverse effects on pulp cells,[16,17] with the consequence being increased TS. This was confirmed in a recent randomized clinical trial, where the authors compared the effects of different durations of in-office bleaching on bleaching-induced TS. The results revealed that the proportion of patients with TS upon a single 15-minute application of in-office bleaching gel (60%) was lower compared with that upon two to three 15-minute applications of the gel in the same session (80%-95%).[50] This leads us to conclude that at shorter durations of application of in-office bleaching gel—as that in the study by Mehta and others[11]—application of Gluma Desensitizer for only one minute is enough to reduce bleaching-induced TS. However, these application times are inadequate for bringing about a decrease in TS when relatively high concentrations of hydrogen peroxide are applied, as observed in the 3 × 15-minute application protocol.

Several studies have demonstrated that application of glutaraldehyde only slightly increases the mechanical properties of dentin at shorter application times (eg, one minute).[51,52] However, upon application for longer durations, the cross-linking effect of glutaraldehyde increases significantly.[53,54] Since application of Gluma Desensitizer results in closure of dentinal tubules because of cross-linking, thus hindering the easy passage of peroxide radicals into the pulp chamber,[37] increasing the application time of the desensitizer will further impair the passage of hydrogen peroxide, thus minimizing bleaching-induced TS. However, this hypothesis has yet to be proven, and further studies are required to evaluate the effect of longer application times of Gluma Desensitizer Liquid or Gel on reducing bleaching-induced TS.

The results of evaluation of color change in the present study indicated that prior application of the Gluma desensitizing agent or placebo had no effect on tooth whitening, leading us to also accept the third null hypothesis. This is a common finding when at-home or in-office bleaching are evaluated,[55-60] and it can be explained by the fact that, usually, the desensitizer agents used did not contain any colorants in their composition.

Both study groups exhibited significant degrees of tooth whitening, ranging from 4.1 to 7.1 units in terms of ΔSGU and 7.4 to 9.3 units in terms of ΔE. These results correspond with those of previous randomized clinical trials that employed relatively high concentrations of hydrogen peroxide, applied three times for 15 minutes each.[7,20,50]

CONCLUSION

Prior application of Gluma Desensitizer Liquid for one minute did not significantly reduce the risk or intensity of in-office bleaching-induced TS, although a slight reduction in risk of TS was observed. Prior application of Gluma Desensitizer Liquid did not jeopardize the whitening effect of in-house bleaching.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of Ceuma University. The approval code for this study is Protocol No. 1.422.841.

Conflict of Interest

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

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Prospective Clinical Study of Zirconia Full-coverage Restorations on Teeth Prepared With Biologically Oriented Preparation Technique on Gingival Health: Results After Two-year Follow-up

R Agustín-Panadero • B Serra-Pastor • A Fons-Font • MF Solá-Ruíz

Clinical Relevance

Tooth preparation with the biologically oriented preparation technique prior to restoration by zirconia fixed prostheses is a safe treatment option that provides excellent clinical outcomes, with greater gingival thickness and gingival margin stability.

SUMMARY

Objectives: To evaluate the clinical behavior of one-piece complete-coverage crowns and fixed partial dentures (FPDs) on teeth with vertical preparation without finish line biologically oriented preparation technique (BOPT).

Methods and Materials: This prospective study included 52 patients requiring treatment with restorations in the esthetic region: 74 crowns and 27 FPDs. The sample included a total of 149 teeth that were prepared vertically without finish line. The sample was divided into two groups: one-piece crowns and FPDs, all with zirconia cores, feldspathic ceramic veneer, and a 0.5-mm prosthetic finish line of zirconia. All procedures were carried out at the University of Valencia from 2013 to 2014. The following parameters were evaluated over a two-year follow-up: oral hygiene, periodontal state, gingival thickening, gingival margin stability, the presence of complications, and restoration survival rate. Patient satisfaction

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with treatment was assessed by means of a visual analogue scale (VAS).

Results: Two years after treatment, 80.5% of treated teeth remained free of gingival inflammation and bleeding. Mean gingival thickening was 0.41 ± 0.28 mm for one-piece crowns and 0.38 ± 0.36 mm for FPDs. Gingival margin stability was 100%, but 2% of the sample presented biological complications. The VAS patient satisfaction scores were eight out of a maximum score of 10.

Conclusions: Two years after treatment, vertical preparation without finish line produces gingival thickening, margin stability, and optimal esthetics. Neither crowns nor FPDs presented any mechanical complications.

INTRODUCTION

Maintaining gingival tissue stability is one of the main challenges when restoring teeth with fixed prostheses in the esthetic region.[1,2] One of the most frequent complications when teeth are restored with tooth-supported fixed prostheses is gingival recession that occurs over time.[3] When the restorations are in the anterior region, this can compromise esthetics and lead to biological and functional problems.[4-7]

Gingival margin recession around tooth-supported fixed prostheses is largely associated with iatrogenic effects produced during dental preparation or caused by inadequate prosthetic fit, which can cause chronic inflammation leading to gingival margin recession around the restoration.[7,8]

Tooth preparation prior to placing fixed prostheses can be classified as three types (Figure 1): horizontal finish line such as rounded shoulder margin, knife-edge finishing line,[9,10] or without finish line, the latter described by Loi as biologically oriented preparation technique (BOPT).[5]

BOPT is a protocol in which the crown's anatomical emergence profile corresponding to the cemento-enamel junction (CEJ) is eliminated to create a new junction with the prosthesis at the moment it is placed.[5,11,12] The protocol for fabricating the interim prosthesis is of key importance as this determines the new emergence that will support the gingival margin and guide healing, reinsertion, and thickening of the gingival tissue; this will be reproduced when the definitive prosthesis is placed. The clinical experience of the authors who have published in the literature on the BOPT technique is that they appreciate an increase in the gingival thickness and better soft tissue stability in the restorations,[5,11,12] but there is no scientific evidence with prospective clinical studies in the literature.

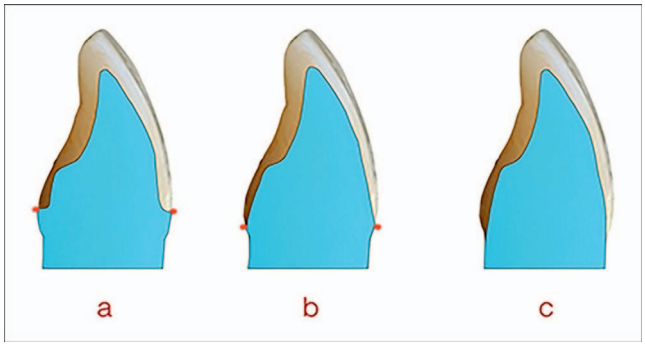

Figure 1. *(a): Rounded shoulder margin with horizontal stop for the final restoration marked with red points. (b): Knife-edge finish line with horizontal stop for the final restoration marked with red points. (c) BOPT preparation technique without horizontal stop.*

The aim of this study was to evaluate the clinical behavior of full-coverage restorations made with zirconia cores, feldspathic ceramic veneer, and a 0.5-mm prosthetic finish line of zirconia on teeth prepared without finish line over a two-year follow-up, registering probing depth, inflammation, gingival thickness and margin stability, any resulting complications, and the restoration survival rate.

METHODS AND MATERIALS

Fifty-two patients were selected who were attending the Prosthetics Clinic at the Department of Dental Medicine, Faculty of Medicine and Dentistry, University of Valencia, Spain. The sample consisted of 22 men and 30 women between 18 and 65 years of age. All were treated between January 2013 and January 2014.

Inclusion criteria included patients older than 18 years, nonsmokers, in good or well-managed periodontal health, and with former treatment in the anterior sector (one-piece crowns or fixed partial dentures [FPDs]) requiring replacement because of differences between the gingival margin and the restorative margin that created an esthetic problem, discoloring, secondary caries, or some other complication (Figure 2).

The study protocol was approved by the University of Valencia Clinical Trial Committee (No. H1448361523684). Patients gave their informed consent in writing to take part.

The sample included a total of 149 teeth (incisors, canines, and premolars), divided into two groups according to the type of prosthetic rehabilitation to

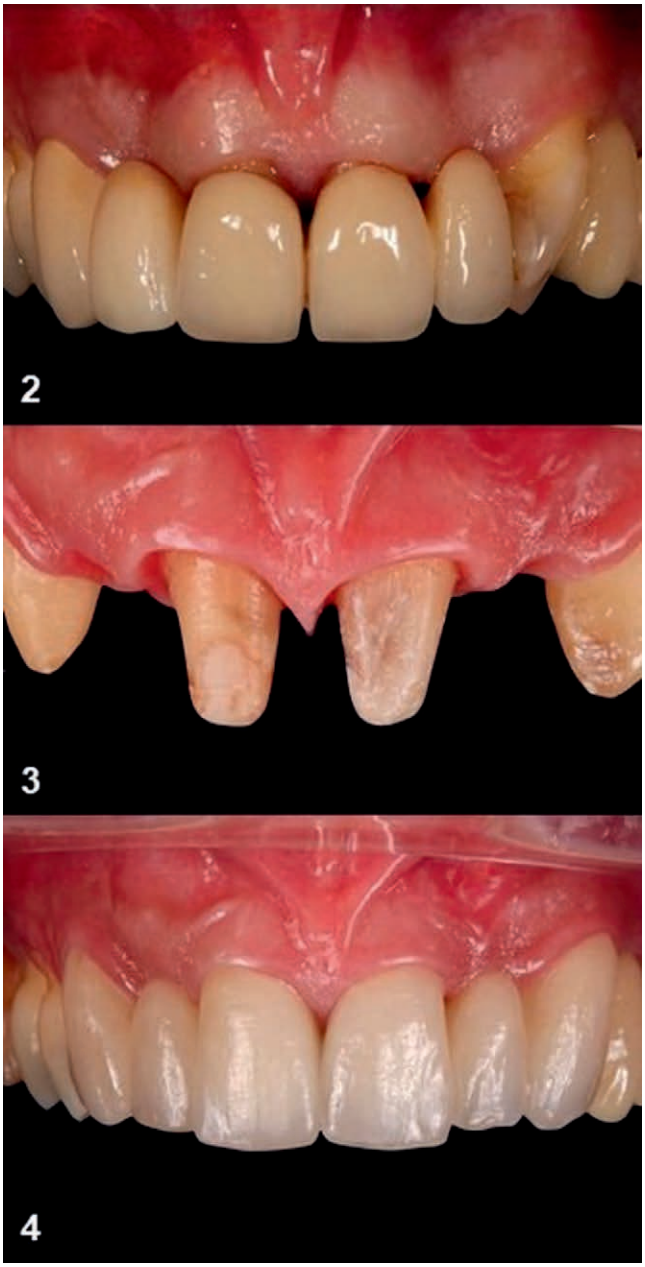

Figure 2. *Labial view of earlier treatment in anterior region with fixed prosthesis presenting recession of the gingival margin and recession.*

Figure 3. *Labial view of dental and soft-tissue preparation after eight weeks of maturation with the provisional prosthesis.*

Figure 4. *Definitive restorations with zirconia core after two years (labial view).*

be performed: one-piece crowns or FPDs (three-unit FPDs). To perform comparable analyses of FPDs and crowns, each tooth was analyzed individually (74 teeth supporting crowns and 75 teeth supporting FPDs).

In all cases, tooth preparation, the provisionalization phase, and laboratory procedures were carried out by a single clinician, following the simplified BOPT protocol described by Agustín and others.[12] Dental preparation eliminated the preexisting finish line (situated supragingival) using a turbine hand piece and 100-/200-μm cone diamond bur of 1.2-mm diameter (862.534.012, BOPT drills; Sweden & Martina, Due Carrare, Italy). The bur was inserted into the gingival sulcus at an angle of 10-15° to the tooth's axis[11]; in this way, the tooth and the gingival tissue underwent rotary curettage, producing bleeding in the gingival sulcus. Afterward, the provisional prosthesis was fabricated with self-polymerizing acrylic resin (Sintodent, Sintodent s.r.l, Rome, Italy) to create a new cementoenamel-prosthetic junction, situated in the gingival sulcus at a depth of 0.5 to 1 mm, with consideration of the biological width.[5,11,12]

Interim restorations were not removed until the soft tissues had completely matured—a period of 8 to 12 weeks (Figure 3). At this point, impressions were taken to fabricate the definitive prosthesis using the two-step impression technique, placing double gingival retraction cord to prevent gingival collapse.

Lastly, the definitive restorations were fitted, with zirconia core (Lava Frame Zirconia, 3M ESPE, Germany) and feldspathic ceramic veneer (Lava Ceram, 3M ESPE) fabricated using the stratification technique, covering up to 0.5 mm before the end of the restoration and a 0.5-mm prosthetic finish line of zirconia (Figure 4). All prostheses were cemented with temporary cement (Temp Bond Clear, Kerr Dental, Orange, CA, USA) during the first two months. After this time, we checked that everything was correct and the restorations were cemented with glass ionomer cement (Ketac Cem Radiopaque, 3M ESPE). It is advisable to use definitive radiopaque cements to check radiologically the correct removal.

Clinical Patient Follow-up

A follow-up protocol was established, with the first checkup shortly after treatment (one week after definitive prosthesis cementation with glass ionomer cement), and at three months, six months, one year, and two years later.

The following parameters were registered at each follow-up visit: frequency of tooth brushing, probing depth (PD), gingival inflammation and bleeding, the presence of any complications, and marginal stability. Marginal stability was assessed using a millimeter-calibrated periodontal probe (PCPUNC156, Hu-Friedy, Des Plaines, IL, USA) to measure the distance (in millimeters) from the cementoenamel-prosthetic junction to the gingival margin.

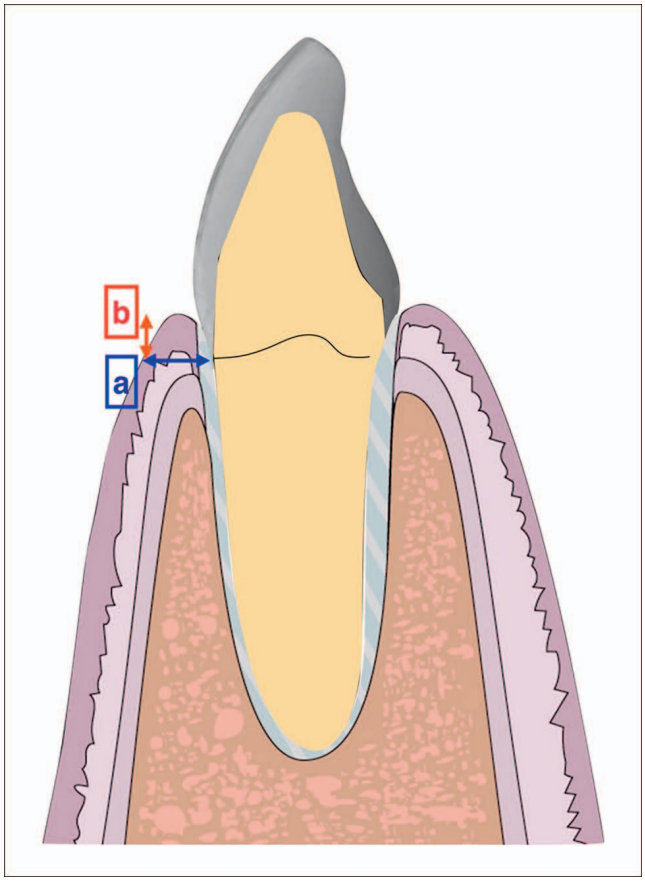

Figure 5. *(a): Measuring gingival thickness. (b): Vertical distance in relation to gingival margin for measuring vestibular gingival thickness.*

Gingival thickness was measured around each tooth at the first checkup (one week after treatment) and at the last checkup (two years after treatment). The measurements were taken under local anesthesia introducing a millimeter-calibrated periodontal probe (PCPUNC156, Hu-Friedy) horizontally 2 mm below the vestibular gingival margin. To standardize this measurement, a transparent guide was fabricated following an Essix splint-type design. Measurements were taken buccally at a 2-mm distance from the gingival margin (Figure 5) indicated in a little hole in the transparent guide and reproduced at the same exact point two years after treatment completion (final measurement). The exact gingival thickness in millimeters was estimated by introducing an endodontic rubber stopper in the periodontal probe and checking the measurements with an endodontic rule.

Finally, the degree of patient satisfaction was assessed at the last visit (two years after treatment) using a visual analogue scale (VAS).[13]

Statistical Analysis

Statistical analysis was performed using IBM SPSS Statistics 21.0 software. Parametric tests were applied with significance set at $p<0.05$. Fisher exact test was applied with a 95% confidence level. Student *t*-test was used to compare independent samples with a power of 0.85.

RESULTS

Analyzing the data obtained during the two-year follow-up, 76.8% of patients presented very good oral hygiene maintenance, brushing two or three times a day, while the other 23.2% of patients brushed only once a day.

At the start of treatment, PD values were 3 mm or less in all samples; during the follow-up, only 4.1% of teeth restored with crowns and 5.6% of teeth supporting FPDs showed some variation. A total of 120 treated teeth (80.5%) remained without gingival inflammation or bleeding, while 29 (19.5%) did show inflammation and/or bleeding. The presence of adequate periodontal parameters (PD of 3 mm or less, absence of gingival inflammation and bleeding) was statistically significantly related to good oral hygiene maintenance.

For teeth supporting one-piece crowns, initial mean gingival thickness was 1.26 mm (SD ±0.48 mm), increasing to 1.67 ± 0.58 mm at the end of the two-year follow-up (Table 1). This represents a mean increase of 0.41 mm (SD±0.28 mm) with statistical significance ($p<0.001$, *t*).

For teeth supporting FPDs, initial mean gingival thickness was 1.14 ± 0.42 mm, increasing to 1.52 ± 0.43 mm (Table 1). This represents a mean increase of 0.38 ± 0.36 mm, also with statistical significance ($p<0.001$, *t*; Figure 6)

Gingival margin stability was 100% for all one-piece crowns and FPDs ($p=0.999$; Table 2); no mucogingival alterations were observed around any of the restorations.

The total number of complications registered represented 2% of the treated teeth. Two cases of pulpitis were found (1.3%), and there was a single case of root fracture of a tooth that had undergone endodontic treatment before the start of the trial, which necessitated extraction of the tooth (0.7%). No mechanical complications—cracks or fractures—were observed in any of the restorations. The total survival index of the restorations supported by teeth prepared with BOPT was 100%.

Table 1: *Changes in Gingival Thickness (in Millimeters) During the Clinical Follow-up Period*

	Gingival Thickness		
	Total	Tooth Supporting Crown	Tooth Supporting FPD
Initial thickness			
n	149	74	75
Mean	1.20	1.26	1.14
Standard deviation	0.45	0.48	0.42
Minimum	0.50	0.50	0.50
Maximum	2.50	2.50	2.00
Median	1.00	1.00	1.00
Final thickness			
n	149	74	75
Mean	1.59	1.67	1.52
Standard deviation	0.51	0.58	0.43
Minimum	1.00	1.00	1.00
Maximum	3.00	3.00	2.50
Median	1.50	1.50	1.50

Lastly, the degree of patient satisfaction assessed by VAS showed a mean value of 8.3 ± 1.2 with statistical significance ($p<0.001$).

DISCUSSION

Establishing a good relationship between dental restorations and the periodontum is crucial to the long-term clinical success of treatment and its esthetic harmony.[1,2] Gingival health and stability around fixed prostheses protects against recession of the gingival margin, which can expose the tooth-restoration finish line and so compromise esthetics.[3-6] Gingival recession is associated with several factors, including gingival biotype (quality and quantity of keratinized gingival tissue), iatrogenesis during the dental preparation phase, chronic inflammation, and inadequate prosthetic marginal fit.[7,8]

Several studies have indicated that subgingival restorations with a conventional finish line are associated with periodontal inflammation and possible gingival recession.[12-14] The present study obtained good gingival health outcomes in terms of PD, inflammation, and bleeding; the few cases that presented increased probing depth and signs of inflammation were associated with the patient's poor oral hygiene regime.

The clinical experience of BOPT reported in the literature[5,11,12] has found that the technique produces increases in gingival thickness and generates better soft-tissue stability in the medium and long term in comparison with other preparations with chamfered finish lines. A four-year prospective study by Peláez and others[15] studied the periodontal behavior of 20 FPDs made of zirconia core and feldspatic veneer, prepared with subgingival chamfer finish line. Of the teeth, 89.47% suffered gingival margin migration, whereby the finish line becomes juxta- or supragingival, and only 10.53% of the teeth maintained the initial subgingival margin.[15] These data show that there is a problem of gingival margin stability in teeth prepared with subgingival horizontal finish lines; however, in the present trial, 100% of teeth prepared with BOPT maintained their initial margin position and produced gingival thickening (mean thickening of 0.41 mm for crowns and 0.38 mm for FPDs) during the two-year follow-up.

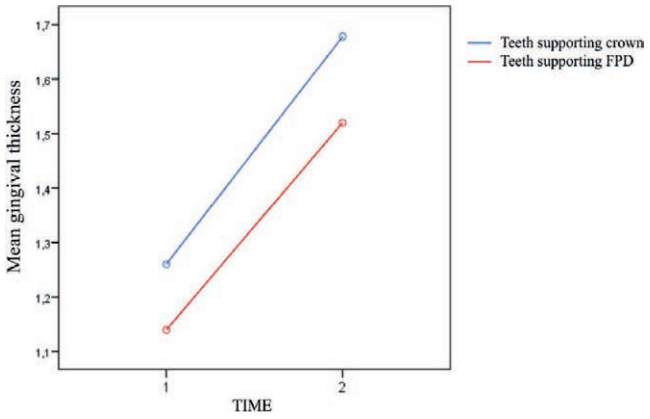

Figure 6. *Gingival thickening during follow-up period.*

In the BOPT technique, the four-week waiting period in the provisional phase is an initial disadvantage that is then compensated by an optimal gingival stability, a correct adaptation of the tissue to the new ovoid morphologies, and a thickening of the gingival tissue according to the results obtained in this study, but it is necessary to take into account that, because there are no long-term clinical studies on this technique, it has not been possible to compare our results with literature following the same procedure.

Regarding the restoration material and its survival, some studies have shown that contemporary ceramic materials such as zirconia offer sufficient resistance to fracture to allow this type of vertical preparation of the tooth stump without a horizontal finish line in the anterior region.[12] Reich and others[16] obtained higher strength with zirconium oxide crowns with a 0.5-mm knife-edge finish line in comparison with crowns with a chamfer finish line. These results concur with the present study in which the restoration survival rate was 100%.

Table 2: *Total Gingival Margin Stability According to Group (Crowns and Fixed Partial Dentures)*

Gingival Margin Stability	Total		Crowns		FPDs	
	N	%	*n*	%	*n*	%
Total	149	100.0%	74	100.0%	75	100.0%
0	149	100.0%	74	100.0%	75	100.0%

CONCLUSIONS

Teeth prepared with BOPT and restored with zirconia crowns or FPDs presented a 100% survival rate. According to the present results, the technique generates gingival thickening (a mean thickening of 0.41 mm for crowns and 0.38 mm for FPDs), as well as gingival margin stability in 100% of samples. The technique provides high periodontal tissue and gingival margin stability, provided the patient maintains adequate oral hygiene. More longitudinal prospective clinical studies are needed to confirm the present findings in the longer term.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Ethics Committee for Human Research of the Commission for Ethics in Experimental Research of the University of Valencia. The approval code for this study is 30-11-2015.

Conflict of Interest

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

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Effect of Surface Sealant Reapplication on Clinical Performance of HEMA-containing and HEMA-free Self-etch Adhesives: Two-year Results

N Tekçe • M Demirci • S Tuncer • SA Göktürk

Clinical Relevance

The application of a surface sealant on Class I restorations is promising for decreasing marginal discoloration and particularly for improving marginal adaptation.

SUMMARY

Purpose: To evaluate the clinical performance of one-step self-etch adhesives over two years with and without the application of a surface sealant.

Methods and Materials: In total, 160 restorations in 40 patients were performed for occlusal caries. Each patient received four Class I restorations, which included a 2-hydroxyethyl methacrylate (HEMA)–containing (Clearfil S3 Bond) and HEMA-free (G-aenial Bond) one-step self-etch adhesive system with and without surface sealant. Half of the restored teeth received Fortify Plus (Bisco) surface sealant material, and the other half were polished with Sof-Lex discs only. Two experienced calibrated examiners clinically evaluated the restorations at baseline and at one- and two-year recalls according to the modified US Public Health Service criteria. The filled surface sealant material was reapplied at each evaluation period.

Results: After two years, none of the restorations had failed. There were no significant differences between the two dentin adhesives with or without a surface sealant application among the evaluation periods. Each dentin adhesive with and without surface sealant showed significant changes from the clinically ideal (Alfa) to clinically acceptable (Bravo) with regard to marginal discoloration, marginal adaptation, and surface texture. Sealed restorations exhibited lower ideal restoration rates with regard to color matching and surface texture and higher ideal restoration rates

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with respect to marginal adaptation compared with unsealed restorations. In addition, the surface sealant application reduced the marginal discoloration of the HEMA-free one-step self-etch adhesive.

Conclusions: The two-year success rates of HEMA-containing and HEMA-free self-etch adhesives with and without surface sealing application were excellent. Although the surface sealant application was not effective with regard to changes in color matching and surface texture, it improved the marginal adaptation of the dentin adhesive and the marginal discoloration of a HEMA-free adhesive.

INTRODUCTION

Shrinkage stresses compete with resin-dentin bonds during resin composite polymerization in such a way that bond failure can be caused, depending upon the configuration and depth of the cavity and the restorative technique used.[1] The configuration factor (C) affects dentin adhesion.[2] The restoration shape is defined by C-factor, which is the proportion of the bonded to the unbonded surface in a restoration.[3] This proportion is greatest in box-like cavities in which there are five bound walls and a single free surface.[4] For clinical circumstances, the proportion of bonded to nonbonded (free) surfaces can reach a maximum C = 5. The increased shrinkage stress rate that develops with an increasing C-value leads to a decrease in the stress-relieving flow capacity of the restorations.[3] Composites are bonded to more than two dentin walls in three-dimensional Class V cavity models in bovine teeth. Flow is significantly restricted in this situation, and contraction stress values can exceed bond strength, which leads to separation.[5] For this reason, in Class I cavities with a high configuration factor, a certain amount of stress is caused when the resin composite is bonded.[2]

Marginal adaptation becomes an important clinical sign of adhesive degradation in composite restorations.[6,7] Sealing marginal gaps through rebonding requires an unfilled resin bonding agent to cover the margins of finished restorations to eliminate the adverse effect of polymerization shrinkage and to ensure better quality and more durable marginal adaptation.[8-11] Unfilled resin seals marginal gaps and decreases microleakage by penetrating into interfacial microgaps.[10,12]

One-step self-etch or so-called "all-in-one" adhesives are accepted as user-friendly materials because the number of steps required in the bonding protocol is reduced. With this system, etching, priming, and adhesive application stages are combined, and, therefore, technique sensitivity diminishes.[13,14] A hydrophilic monomer, 2-hydroxyethyl methacrylate (HEMA), is especially incorporated in adhesive formulations.[15] Current all-in-one adhesives commonly contain HEMA, a well-known co-monomer, that functions as a wetting agent and diffusion promoter of resin into the exposed collagen and prevents phase separation between hydrophilic and hydrophobic monomers.[16,17] However, HEMA makes it difficult to remove water from the adhesive by decreasing the vapor pressure, and this residual water may interfere with the polymerization of adhesive monomers, thereby affecting the quality of the hybrid layer.[18] This negative effect was overcome by introducing HEMA-free self-etch adhesives, which isolate water.[17] HEMA-free self-etch adhesives are a mixture of hydrophilic and hydrophobic contents, solvent, and water. These adhesives are prone to phase separation, which partially accounts for their lower bonding effectiveness. However, strongly air-drying the phase-separated adhesive might be an appropriate clinical technique for removing substantial interfacial water for HEMA-free adhesives, which, when applied accurately, are expected to result in a less hydrophilic (no HEMA) and thus more hydrolysis-resistant adhesive interface in the long term.[16,19,20] The omission of HEMA from adhesive formulations is considered an advantage for removing most of the water that would otherwise weaken the bond.[20] Very strong air-drying appeared sufficient to remove the water droplets.[20]

Acceptable clinical results were reported with sealed restorations that had been maintained after three and 10 years.[6,21] Sealing restorations improved marginal adaptation and staining.[6] A three-year clinical study[21] showed that marginal sealing of defective resin-based composite and amalgam Class I and Class II restorations were conservative and simple procedures that increased the longevity of restorations. Therefore, it is important to understand the clinical success of surface sealing on composite restorations with HEMA-containing or HEMA-free all-in-one self-etch adhesives.

Our aim was to evaluate the clinical performance of HEMA-containing and HEMA-free all-in-one self-etch adhesives with and without a surface sealing process in Class I cavities. The first null hypothesis was that there would be no significant differences between the clinical performance of HEMA-containing and HEMA-free all-in-one self-etch adhesives in

Table 1: *The Brand Names, Chemical Compositions, and Manufacturers' Instructions for Application*

Material (Manufacturer)	Type	Composition	Manufacturers' Instructions
G-aenial Bond (GC Corp, Tokyo, Japan)	HEMA-free one-step self-etch adhesive	4-MET, UDMA, phosphate monomer, DMA component, fumed silica filler, acetone, water, photoinitiator	Shake adhesive bottle. Apply adhesive. Leave for 10 s. Dry thoroughly for 5 s with oil-free air under maximum air pressure. Light-cure for 10 s.
Clearfil S3 Bond (Kuraray Medical Inc, Tokyo, Japan)	One-step self-etch adhesive with HEMA	10-MDP, HEMA, Bis-GMA, water, ethanol, silanated colloidal silica, camphorquinone, photoinitiator	Apply adhesive. Leave for 20 s. Dry by high-pressure blowing for more than 5 s. Light-cure for 10 s.
Fortify Plus Filled Surface Sealant (Bisco, Schaumburg, IL, USA)	Microfilled surface sealant material	Bis-EMA, UDMA, 17.3 vol % 0.4 μm amorphous silica filler	Etch the surface of the restoration and approximately 1-2 mm beyond the tooth/restoration margin for 15 s. Apply a thin layer to previously etched surfaces using a scrubbing motion. Air-thin by blowing a gentle stream of air over this layer to assure an even distribution. Light-cure sealant for 10 s.
Clearfil Majesty Posterior (Kuraray Medical Inc, Tokyo, Japan)	Superfilled nanohybrid composite	Organic content: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate	Place the chosen shade product into the cavity in 1.5-mm increments. Light-cure the resin for 20 s.
		Inorganic content: Glass ceramics, surface-treated alumina microfiller (1.5 μm), silica filler (20 nm)	
		Filler (wt/vol %): 92/83	

Abbreviations: Bis-EMA, ethoxylated bisphenol A dimethacrylate; Bis-GMA, bisphenol glycidyl methacrylate; DMA, dimethacrylate; 4-MET, 4-methacryloxyethyl trimellitic acid; HEMA, 2-hydroxyethyl methacrylate; PENTA, dipentaerythritol penta-acrylate phosphate; TEGDMA, triethylene glycol dimethacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; UDMA, urethane dimethacrylate.

Class I cavities. The second null hypothesis was that annual surface sealant reapplication would not significantly affect the clinical performance of HEMA-containing and HEMA-free all-in-one self-etch adhesives in Class I cavities.

METHODS AND MATERIALS

Study Design

The study was approved by the Ethics Committee of Kocaeli University, Faculty of Dentistry (KOU KAEK 2014/239). Table 1 shows the materials used in the study. The restorations were performed between July and December 2014 in the Department of Restorative Dentistry, Faculty of Dentistry at Kocaeli University. In total, 40 patients (15 males and 25 females) aged 18-55 years (mean age: 23.3 years) were included in the study (Figure 1). The inclusion criteria were patients who needed four direct Class I composite restorations, those with good oral hygiene and with no active pulpal or periodontal diseases, whose permanent first or second molars/premolars required restorations because of the presence of occlusal carious lesions and were in occlusion with antagonist teeth.[22-24] Patients were excluded according to the following criteria: patients with uncontrolled parafunction, those presenting with poor oral hygiene and those disinterested in or refusing of oral hygiene instructions, those with molars and premolars with carious lesions on a surface other than the occlusal surface or with pulp exposure during carious tissue excavation, those having sensitivity to percussion or spontaneous pain from the related tooth, and patients with periodontal or gingival disease.[22-24] Each patient received four restorations for primary caries on occlusal surfaces. All teeth had opposing and adjacent tooth contacts. The distribution of Class I restorations with sealing and without sealing according to dentin adhesives, composite material type, and teeth numbers were as shown in Table 2.

Treatment Protocol

Each patient received four Class I restorations, which included a HEMA-containing (Clearfil S3 Bond, Kuraray Medical Inc, Tokyo, Japan) or HEMA-free (G-aenial Bond, GC Corp, Tokyo, Japan) one-step self-etch adhesive system, a HEMA-containing adhesive

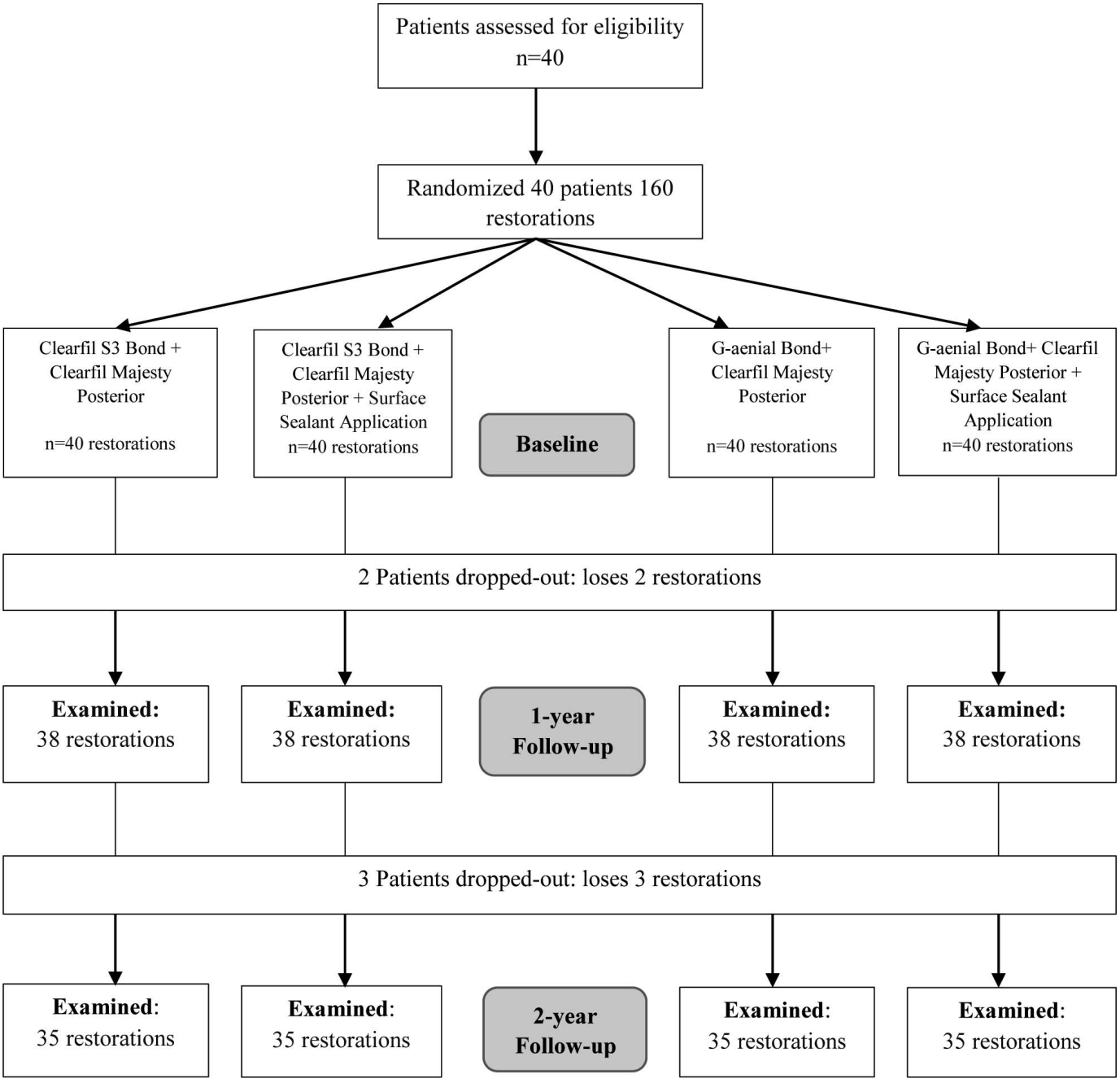

Figure 1. *Flow diagram for history of restorations.*

(Clearfil S3 Bond) with a surface sealant, or a HEMA-free adhesive (G-aenial Bond) with surface sealant. All restorations were performed with the same super-filled nanohybrid composite (Clearfil Majesty Posterior, Kuraray Medical Inc). Randomization was performed by selecting the HEMA-containing dentin adhesive and tooth number by flipping a coin, followed by the selection of restoration type, also determined by the flip of a coin.

Restorative Procedure

First, the teeth were cleaned using pumice water and a rubber cup to remove the surface stains and any residual dental plaque. The lesions were diagnosed macroscopically with a probe; they involved fissures that had reached the dentin, but in which lateral spread was limited and localized to the dentin. Cavity preparation only involved removal of enamel and dentin carious tissues. The average

Table 2: *Distribution of Class I Restorations with Sealing and Without Sealing According to Dentin Adhesives, Composite Material Types, and Teeth Number*

Materials	n	Tooth No.															
		14	15	16	17	24	25	26	27	34	35	36	37	44	45	46	47
Clearfil S3 Bond + Clearfil Majesty Posterior	40	—	—	5	6	—	1	4	4	—	—	6	3	1	—	6	4
Clearfil S3 Bond + Clearfil Majesty Posterior + surface sealant application	40	—	—	5	4	1	—	—	7	—	1	2	13	—	—	—	7
G-aenial Bond + Clearfil Majesty Posterior	40	—	—	4	7	1	—	3	3	—	—	3	7	—	—	8	4
G-aenial Bond + Clearfil Majesty Posterior + surface sealant application	40	—	—	6	4	—	—	5	3	—	1	4	—	—	—	8	9
		—	—	20	21	2	1	12	17	—	2	15	23	1	—	22	24

facio-lingual width of the cavities was approximately one-third of the intercuspal width. The cavity margins were not left in occlusal contact. Isolation of cavities was provided with cotton rolls and saliva ejectors.[25] After isolating the cavities, the same experienced practitioner (NT), who was familiar with the materials used in the present study, performed the tooth preparation and applied the materials per the manufacturers' instructions (Table 1). Polymerization was performed using an Elipar S10 (3M ESPE, St Paul, MN, USA) at no less than 1200 mW/cm^2. The composite shade was selected using the corresponding composite guide or custom composite samples. If the restorations had a depth greater than 2 mm, the composite was applied incrementally. First, a super-filled nanohybrid composite (Clearfil Majesty Posterior) was applied in layers no greater than 2 mm with an oblique incremental placement technique. Then the composite increment was light-cured for 20 seconds, in accordance with the manufacturer's instructions. Occlusion and articulation were checked after the restorations were completed. Then the removal of excess material and finishing were performed using microfine finishing diamonds (8368.204.023 Komet, Gebr Brasseler, Lemgo, Germany). Finally, the restorations were polished using Sof-Lex abrasive disks (3M ESPE). For restorations that required surface sealant, the surface sealant (Fortify Plus, Bisco, Schaumburg, IL, USA) was applied in line with the manufacturer's instructions (Table 1). This step was repeated at the end of one year to enhance the clinical effectiveness of marginal sealing. The patients were informed about the evaluation periods and their cooperation was requested.

Evaluation

Two experienced calibrated examiners from the Department of Restorative Dentistry at Kocaeli University evaluated the restorations using a dental explorer and mirror, according to the modified US Public Health Service (USPHS) criteria (Table 3).[25-27] The examiners were not involved in the operation or insertion of the restorations and were fully blind to the experimental protocol. For training purposes, the examiners were given a set of photographs as a reference to illustrate each score for each criterion. Then they clinically evaluated 20 Class I restorations with two days' separation between examinations. These restorations were not included in the present study. The evaluation phase of the study was performed only when at least 85% intraexaminer and interexaminer agreement was achieved in the calibration phase.[28] At baseline and one- and two-year recalls, color match, wear and loss of anatomic form, marginal discoloration, caries, marginal adaptation, and surface texture were evaluated and scored as Alfa (A) = ideal clinical findings, Bravo (B) = clinically acceptable, Charlie (C) = clinically unacceptable and requiring restoration replacement, and Delta (D) = fractured restoration, mobile, or missing and requiring immediate replacement. Also, in restorations with surface sealant, after clinical evaluation of surface sealant application at the end of one year, surface sealant was again reapplied. This surface sealant reapplication was evaluated at the end of two years (another one year). Thus, surface sealant reapplication was evaluated annually. Conflicts in scoring were resolved through consensus.

Statistical Analysis

Statistical analysis was performed using SPSSWIN 20.0 (SPSS, Chicago, IL, USA). The data obtained were statistically analyzed using the Friedman test to examine changes that occurred throughout the two-year evaluation period (Table 4). Comparisons of data between the two dentin adhesives with or without a surface sealing were performed using the Mann-Whitney *U*-test, Kruskal-Wallis one-way

Table 3: *Direct Clinical Evaluation Criteria (Modified USPHS Criteria)*

Rating	Aspect	Method
Color match		
Alfa (A)	There is no mismatch in color, shade, and/or translucency between the restoration and the adjacent tooth structure.	Visual inspection
Bravo (B)	There is a mismatch in color, shade, and/or translucency between the restoration and the adjacent tooth structure, but the mismatch is within the normal range of tooth color, shade, and/or translucency.	Visual inspection
Charlie (C)	The mismatch is between restoration and adjacent tooth structure outside the normal range of tooth color, shade, and/or translucency.	Visual inspection
Cavosurface marginal discoloration		
Alfa (A)	There is no discoloration anywhere on the margin between the restoration and the tooth structure.	Visual inspection
Bravo (B)	There is discoloration anywhere on the margin between the restoration and the tooth structure, but the discoloration has not penetrated along the margin of the restorative material in an enamel direction and can be polished away.	Visual inspection
Charlie (C)	The discoloration has penetrated along the margin of the restorative material in an enamel direction.	Visual inspection
Wear/anatomic form		
Alfa (A)	The restoration is not undercontoured: that is, the restorative material is not discontinuous with existing anatomic form.	Visual inspection and explorer
Bravo (B)	The restoration is undercontoured: that is, the restorative material is discontinuous with existing anatomic form, but sufficient restorative material is not missing so as to expose the enamel or base.	Visual inspection and explorer
Charlie (C)	Sufficient restorative material is missing so as to expose the enamel or base.	Visual inspection
Caries		
Alfa (A)	There is no evidence of caries contiguous with the margin of the restoration.	Visual inspection
Bravo (B)	There is evidence of caries contiguous with the margin of the restoration.	Visual inspection
Marginal adaptation		
Alfa (A)	There is no visible evidence of a crevice along the margin into which the explorer will penetrate.	Visual inspection and explorer
Bravo (B)	There is visible evidence of a crevice along the margin into which the explorer will penetrate. The enamel or base is not exposed.	Visual inspection and explorer
Charlie (C)	There is visible evidence of a crevice along the margin into which the explorer will penetrate. The enamel or base is exposed.	Visual inspection and explorer
Delta (D)	The restoration is fractured or missing in part or *in toto*.	Visual inspection and explorer
Surface texture		
Alfa (A)	Surface of restoration is smooth.	Explorer
Bravo (B)	Surface of restoration is slightly rough or pitted, can be refinished.	Explorer
Charlie (C)	Surface deeply pitted, irregular grooves (not related to anatomy), cannot be refinished.	Explorer
Delta (D)	Surface is fractured or flaking.	Explorer

analysis of variance, and the Dunn post hoc test. When a statistically significant difference was identified for any assessed criterion, the Dunn post hoc test was used for multiple comparisons between each recall time interval (Tables 5-7). Kaplan-Meier survival analysis was used to determine the probability of the clinical survivability of the two dentin adhesives with or without the surface sealing for a given time period (Table 4). *P*-values of <0.05 were considered statistically significant. Interexaminer and intraexaminer agreement was tested using Cohen kappa coefficient.

RESULTS

After one year, two patients with eight restorations left the study. At the end of two years, three patients with 12 restorations did not return (Figure 1). After one and two years, the cumulative recall rates for patients were 95% and 87.5%, respectively.

The Cohen kappa coefficient (0.87) revealed strong agreement between the examiners, with no statistical difference between them ($p>0.05$). The Kaplan-Meier survival analyses are given in Table 4. After one and two years, no restorations failed, giving a 100% success rate for each evaluation period.

Table 4: *Results of Clinical Evaluation of a HEMA-containing and a HEMA-free One-step (All-in-one) Self-etch Adhesive with and Without Surface Sealing Process in Class I Restorations Using Modified USPHS Criteria. Observations Are Shown in Percent (Cumulative Number of Restorations)*

Time	Groups	Recall Rate	Retention		Color Match			Marginal Discoloration		
			A	C	A	B	C	A	B	C
Baseline	Clearfil S3 Bond	100 (40)	100 (40)	—	100 (40)	—	—	100 (40)	—	—
	G-aenial Bond	100 (40)	100 (40)	—	100 (40)	—	—	100 (40)	—	—
	Clearfil S3 Bond + surface sealant application	100 (40)	100 (40)	—	100 (40)	—	—	100 (40)	—	—
	G-aenial Bond + surface sealant application	100 (40)	100 (40)	—	100 (40)	—	—	100 (40)	—	—
1 year	Clearfil S3 Bond	95.0 (38)	100 (38)	—	94.7 (36)	5.3 (2)	—	86.8 (33)	13.2 (5)	—
	G-aenial Bond	95.0 (38)	100 (38)	—	94.7 (36)	5.3 (2)	—	86.8 (33)	13.2 (5)	—
	Clearfil S3 Bond + surface sealant application	95.0 (38)	100 (38)	—	97.4 (37)	2.6 (1)	—	84.2 (32)	15.8 (6)	—
	G-aenial Bond + surface sealant application	95.0 (38)	100 (38)	—	89.5 (34)	10.5 (4)	—	89.5(34)	10.5 (4)	—
2 Year	Clearfil S3 Bond	87.5 (35)	100 (35)	—	94.3 (33)	5.7 (2)	—	82.9 (29)	17.1 (6)	—
	G-aenial Bond	87.5 (35)	100 (35)	—	91.4 (32)	8.6 (3)	—	77.1 (27)	22.9 (8)	—
	Clearfil S3 Bond + surface sealant application	87.5 (35)	100 (35)	—	91.4 (32)	8.6 (3)	—	82.9 (29)	17.1 (6)	—
	G-aenial Bond + surface sealant application	87.5 (35)	100 (35)	—	85.7 (30)	14.3 (5)	—	85.7 (30)	14.3 (5)	—

Abbreviations: A, Alfa; B, Bravo; C, Charlie; D, Delta; HEMA, 2-hydroxyethyl methacrylate.

Statistical analyses revealed no significant differences ($p>0.05$) between Clearfil S3 Bond and G-aenial Bond dentin adhesives with or without surface sealants within each evaluation period with regard to the defined parameters.

Only the G-aenial Bond with the surface sealant showed statistically significant differences ($p=0.015$) between baseline and one-year rates and between baseline and two-year rates with respect to color matching (Tables 5 and 7). After two years, 94.3% of Clearfil S3 Bond, 91.4% of G-aenial Bond, 91.4% of Clearfil S3 Bond with sealant and 85.7% of G-aenial Bond with sealant restorations were scored as clinically ideal (Alfa) with respect to color match. For Clearfil S3 Bond and G-aenial Bond dentin adhesive with or without a surface sealant, statistically significant differences ($p<0.05$) were determined between baseline and one-year rates and baseline and two-year rates with respect to marginal discoloration and surface texture (Tables 5 and 7). After two years, 17.1% of Clearfil S3 Bond and Clearfil S3 Bond restorations with sealant showed marginal discoloration, and 22.9% of G-aenial Bond and 14.3% of G-aenial Bond restorations with sealant showed marginal discoloration. This marginal discoloration was a result of the adhesive system. However, this discoloration was superficial, located on a nonspecific part of the enamel, did not penetrate toward the pulp along the margin of the restorative material, and could be polished away. Regarding surface texture, 82.9% of Clearfil S3 Bond restorations, 77.1% of G-aenial Bond restorations, 80% of Clearfil S3 Bond restorations with sealant, and 74.3% of G-aenial Bond restorations with sealant were clinically ideal after two years. Regarding the marginal adaptation rate, there were statistically significant differences ($p=0.014$) between baseline and two-year rates of Clearfil S3 Bond without the surface sealant (Table 7). Statistically significant differences were determined between baseline and two-year rates ($p=0.008$) and between the one-year and two-year rates ($p=0.046$) of G-aenial Bond without the

Table 5: p-*values (statistical difference) Between Baseline and One Year*

Groups	Retention	Color Match	Marginal Discoloration	Wear/Anatomic Form	Caries	Marginal Adaptation	Surface Texture
Clearfil S3 Bond	1.0 (NS)	0.135 (NS)	0.025 (S)	0.368 (NS)	1.0 (NS)	0.083 (NS)	0.025 (S)
G-aenial Bond	1.0 (NS)	0.097 (NS)	0.025 (S)	0.097 (NS)	1.0 (NS)	0.083 (NS)	0.046 (S)
Clearfil S3 Bond + surface sealant application	1.0 (NS)	0.097 (NS)	0.014 (S)	1.0 (NS)	1.0 (NS)	0.046 (S)	0.046 (S)
G-aenial Bond + surface sealant application	1.0 (NS)	0.046 (S)	0.046 (S)	0.317 (NS)	1.0 (NS)	0.025 (S)	0.008 (S)

Abbreviations: NS, not significant; S, significant ($p<0.05$*).*

Table 4: Extended.

Time	Wear/Anatomic Form			Caries		Marginal Adaptation				Surface Texture			
	A	B	C	A	B	A	B	C	D	A	B	C	D
Baseline	100 (40)	—	—	100 (40)	—	100 (40)	—	—	—	100 (40)	—	—	—
	100 (40)	—	—	100 (40)	—	100 (40)	—	—	—	100 (40)	—	—	—
	100 (40)	—	—	100 (40)	—	100 (40)	—	—	—	100 (40)	—	—	—
	100 (40)	—	—	100 (40)	—	100 (40)	—	—	—	100 (40)	—	—	—
1 year	97.4 (37)	2.6 (1)	—	100 (38)	—	92.1 (35)	7.9 (3)	—	—	86.8 (33)	13.2 (5)	—	—
	97.4 (37)	2.6 (1)	—	100 (38)	—	92.1 (35)	7.9 (3)	—	—	89.5 (34)	10.5 (4)	—	—
	100 (38)	—	—	100 (38)	—	89.5 (34)	10.5 (4)	—	—	89.5 (34)	10.5 (4)	—	—
	97.4 (37)	2.6 (1)	—	100 (38)	—	86.8 (33)	13.2 (5)	—	—	81.6 (31)	18.4 (7)	—	—
2 Year	97.1 (34)	2.9 (1)	—	100 (35)	—	82.9 (29)	17.1 (6)	—	—	82.9 (29)	17.1 (6)	—	—
	91.4 (32)	8.6 (3)	—	100 (35)	—	80 (28)	20 (7)	—	—	77.1 (27)	22.9 (8)	—	—
	100 (35)	—	—	100 (35)	—	85.7 (30)	14.3 (5)	—	—	80.0 (28)	20.0 (7)	—	—
	88.6 (31)	11.4 (4)	—	100 (35)	—	82.9 (29)	17.1 (6)	—	—	74.3 (26)	25.7 (9)	—	—

surface sealant (Tables 6 and 7). Also, for the Clearfil S3 Bond and G-aenial Bond with the surface sealant, there were statistically significant differences between baseline and one-year rates (p=0.046 and p=0.025, respectively) (Table 5) and baseline and two-year rates (p=0.025 and p=0.014, respectively) (Table 7). In addition, 82.9% of Clearfil S3 Bond restorations, 80% of G-aenial Bond restorations, 100% of Clearfil S3 Bond restorations with sealant, and 82.9% of G-aenial Bond restorations with sealant were clinically ideal (Alfa) with respect to marginal adaptation after two years. Only the G-aenial Bond with the surface sealant showed statistically significant differences (p=0.046) between the baseline and two-year rates with regard to wear or loss of anatomic form (Table 7). After two years, 97.1% of Clearfil S3 Bond, 91.4% of G-aenial Bond, 100% of Clearfil S3 Bond restorations with sealant, and 88.6% of G-aenial Bond restorations with sealant were clinically ideal with regard to wear and anatomic form. After two years, none of the restorations demonstrated caries.

DISCUSSION

After two years, there were no significant differences between the clinical performance of HEMA-containing and HEMA-free one-step self-etch adhesives with regard to color match, marginal discoloration, wear and loss of anatomic form, caries, marginal adaptation, and surface texture. Therefore, the first null hypothesis must be accepted. With the exception of caries, there was a decline in restoration performance from clinically ideal to clinically acceptable with respect to all criteria evaluated in the study. In our study, the two-year survival rates for Clearfil S3 Bond and G-aenial Bond restorations were 100%. In agreement with our results, other studies[29,30] reported 100% success rates after two and three years for Class I cavities. In accordance with our findings, it was reported[31] that the restorations performed well overall and were successful at the two-year recall for Class I/II restorations, regardless of which bonding agent was used. Moreover, 100% success rates were obtained for a nanohybrid composite material (Grandio) with a self-etch adhesive (Futur-

Table 6: p-*values (statistical difference) Between One and Two Years*

Groups	Retention	Color Match	Marginal Discoloration	Wear/Anatomic Form	Caries	Marginal Adaptation	Surface Texture
Clearfil S3 Bond	1.0 (NS)	0.135 (NS)	0.317 (NS)	0.368 (NS)	1.0 (NS)	0.083 (NS)	0.317 (NS)
G-aenial Bond	1.0 (NS)	0.097 (NS)	0.083 (NS)	0.097 (NS)	1.0 (NS)	0.046 (S)	0.046 (S)
Clearfil S3 Bond + surface sealant application	1.0 (NS)	0.097 (NS)	1.0 (NS)	1.0 (NS)	1.0 (NS)	0.317 (NS)	0.083 (NS)
G-aenial Bond + surface sealant application	1.0 (NS)	0.317 (NS)	0.317 (NS)	0.083 (NS)	1.0 (NS)	0.317 (NS)	0.157 (NS)

Abbreviations: NS, not significant; S, significant ($p<0.05$*).*

Table 7: p-*values (statistical difference) Between Baseline and Two Years*

Groups	Retention	Color Match	Marginal Discoloration	Wear/Anatomic Form	Caries	Marginal Adaptation	Surface Texture
Clearfil S3 Bond	1.0 (NS)	0.135 (NS)	0.014 (S)	0.368 (NS)	1.0 (NS)	0.014 (S)	0.014 (S)
G-aenial Bond	1.0 (NS)	0.097 (NS)	0.005 (S)	0.097 (NS)	1.0 (NS)	0.008 (S)	0.005 (S)
Clearfil S3 Bond + surface sealant application	1.0 (NS)	0.097 (NS)	0.014 (S)	1.0 (NS)	1.0 (NS)	0.025 (S)	0.008 (S)
G-aenial Bond + surface sealant application	1.0 (NS)	0.025 (S)	0.025 (S)	0.046 (S)	1.0 (NS)	0.014 (S)	0.003 (S)

Abbreviations: NS, not significant; S, significant ($p<0.05$).

abond NR) after two years.[32] However, the adhesive system used in that study was different from the adhesive used in our study. Another study[33] that clinically evaluated self-etch adhesives in posterior restorations reported 96% and 95.2% retention rates for Adper Prompt L-Pop and iBond, respectively, and a 100% retention rate for Clearfil S3 Bond and One-Step Plus in Class I/II restorations after two years; compared with our study, slightly lower or the same percentage success rates were obtained in these studies. Unlike the present study, Class I and II restorations in these studies were evaluated together. In addition, with the exception of Clearfil S3 Bond, different adhesives and composite materials were used in those studies. Therefore, cavity location and size and composite material and adhesive variability may account for the different failure rates between our study and those in the literature. However, a meta-analysis on prospective studies[34] concerning survival of direct resin restorations in posterior teeth showed a 1.46% mean annual failure rate in short-term studies that included Class I/II restorations. When both short- and long-term studies were considered, recall rate, ratio of Class I fillings to Class II fillings, observation period, and study size (number of restorations and patients) each significantly influenced the overall failure rate.

The data obtained after two years shows the performance of this surface sealant reapplication after another one year because the surface sealant was reapplied again in the first-year recall. Thus, data for restoration with surface sealant related to all criteria evaluated in the study were obtained annually. The annual reapplication of the surface sealing did not significantly affect the clinical performance of a HEMA-containing and a HEMA-free one-step self-etch adhesive in regard to the evaluation criteria used at the end of two years. Thus, the second null hypothesis must be accepted. With the exception of caries, there was a decline in the performance of the restorations from clinically ideal (Alfa) to clinically acceptable (Bravo) with respect to the evaluation criteria. The two-year survival rates for Clearfil S3 Bond and G-aenial Bond restorations with sealant were 100%. None of the restorations failed, resulting in a 100% success rate. In agreement with our findings, a study[6] that evaluated sealed composite after 10 years in Class I and II cavities reported no failures in Class I restorations. However, the survival rate of Class II restorations was 85%.

After two years, color change was observed for Clearfil S3 Bond and G-aenial Bond restorations without sealant from clinically ideal (Alfa) to clinically acceptable (Bravo) (Table 4), and these changes were not statistically significant ($p>0.05$). It was reported[29,30] that ideal restorations with regard to color match were 100% and 96% in Class I restorations that did not include sealant application protocols after two and three years, respectively. However, 86.5% of restorations that included nanofill and nanohybrid composite materials exhibited ideal color match after 30 months in Class I restorations.[23] These results partially agree with our findings, in which similar or lower ideal restoration rates were observed. In our study, we used the same brand of composite material for all adhesives to exclude potential intervening variables.[33] After two years, color changes were observed for Clearfil S3 Bond and G-aenial Bond restorations with sealant from clinically ideal to clinically acceptable (Table 4); these changes were not statistically significant ($p>0.05$), with the exception of G-aenial Bond restorations with sealant (Tables 5-7). The color change was only statistically significant for G-aenial Bond restorations with sealant between baseline and one year ($p=0.046$) (Table 5) and between baseline and two years ($p=0.025$) (Table 7). In addition, Clearfil S3 Bond and G-aenial Bond restorations with sealant showed a greater color change than was seen without sealant restorations of Clearfil S3 Bond and G-aenial Bond between one and two years. On the other hand, it was reported[35] that 75% of sealed and only 33% of unsealed restorations were rated Alfa with regard to color

match in Class I and II restorations at a five-year recall. This difference may be associated with the difference in evaluation times between that study and our study. In contrast to our findings, it was shown[36] that the surface sealant did not alter the color stability of the tested materials after artificial aging using ultraviolet radiation and staining solutions. We used a different surface sealant material including amorphous silica filler; therefore, this may have affected the color change. In addition, *in vivo* conditions may cause different results compared to *in vitro* conditions. Fortify Plus contains ethoxylated bisphenol A dimethacrylate resin (Bis-EMA). The Bis-EMA component, which is present in many restorative resin composites, was considered to contribute increased staining of composite resin coated with Fortify Plus surface sealant.[37]

After two years, Clearfil S3 Bond and G-aenial Bond restorations without surface sealant exhibited significant marginal discoloration and the deterioration of marginal adaptation. However, these changes were clinically acceptable. In partial agreement with the present study, a clinically acceptable marginal discoloration rate, which was 14.3%-22.9% in our study, was reported[23,29,30] as 0%-27% in Class I restorations after three and two years and after 30 months. In addition, with regard to marginal adaptation, the ideal restoration rates in these studies were found to be 56%-92%. The differences in results between trials are accounted for by differences in the adhesives used, chemical and physical properties of the materials, compositions of brands, and duration of the clinical studies.[23] However, in another study[33] that used the same adhesive (Clearfil S3 Bond), ideal restorations rates were, respectively, 54.5% and 50% with regard to marginal discoloration and marginal adaptation in Class I/II restorations after two years. The comparatively lower ideal restoration rates may have been caused by cavity size differences because they included both Class II and Class I cavities.

In our study, although there were no statistical differences, Clearfil S3 Bond restorations without sealant showed lower marginal discoloration and deterioration of marginal adaptation than did G-aenial Bond restorations without sealant. Clearfil S3 Bond had 10-MDP (10-methacryloyloxydecyl dihydrogen phosphate) in its chemical structure. The "Adhesion-decalcification" idea reports that this specific functional monomer can interact ionically with hydroxyapatite, forming self-assembled "nano-layers." Combined with nano-layering, stable MDP-calcium salt deposition will contribute to clinical longevity of the hybrid layer and thus the bond to dentin.[38] However, HEMA-free one-step adhesives are prone to phase separation; therefore, they are complex blends of solvents, water, and hydrophilic and hydrophobic ingredients. This may explain their lower bonding effectiveness[16] and may account for differences between Clearfil S3 Bond and G-aenial Bond restorations.

After two years, Clearfil S3 Bond and G-aenial Bond restorations with surface sealant exhibited significant marginal discoloration and the deterioration of marginal adaptation. However, these changes were clinically acceptable. In addition, in the present study, G-aenial Bond restorations with surface sealant exhibited lower marginal discoloration than did G-aenial Bond restorations without surface sealant. Moreover, Clearfil S3 Bond and G-aenial Bond restorations with surface sealant both showed higher clinically ideal (Alfa) restoration rates than did restorations without sealant with regard to marginal adaptation. It may be said that the surface sealant improved the quality of the marginal seal, especially for G-aenial Bond restorations with surface sealant. In agreement with this finding, Femiano and others[39] observed that the use of a hydrophobic bonding agent for resealing direct restorations showed the small deteriorations of marginal seal, such as overhang resin or brown line at finish lines in enamel in the short term. On the other hand, after 24 months, restorations without the additional marginal seal showed a greater prevalence of gaps that retained the probe or included probe penetration of more than 1 mm. On this basis, the authors[39] concluded that the quality of marginal seal could be improved by applying an enamel adhesive on the margins of finished direct resin restorations, thereby increasing their longevity. Dickinson and Leinfelder[35] found that over five years, sealed restorations showed a higher rate of ideal restorations than did unsealed restorations with respect to marginal discoloration and marginal integrity. They reported that surface-penetrating sealant had the potential to penetrate and fill microstructural defects, including defects both on the occlusal surface of the restoration and at the restoration-preparation interface. Thus, their surface-penetrating sealant was effective at enhancing marginal integrity.[35] In addition, it was reported[6] that sealing the defective margins of restorations improved marginal staining and marginal adaptation parameters, although the findings of this study were similar to those associated with the group without sealing by the 10th year.

With respect to wear and anatomic form, 97.1% of Clearfil S3 Bond and 91.4% of G-aenial Bond restorations without sealants were ideal (Alfa). In partial agreement with our findings, the ideal restoration rates were reportedly 85.7%-93% after two years, 88%-100% after three years, and 94.6% after 30 months.[23,29-31,33] The difference in rates between our study and others may have been caused by the use of different composite materials. In addition, only G-aenial Bond restorations with surface sealant exhibited significantly lower rates of ideal restoration with regard to wear and anatomic form after two years. However, there was no statistically significant difference between Clearfil S3 Bond and G-aenial Bond restorations with and without sealant. On the other hand, the G-aenial Bond restorations with the surface sealant exhibited statistically significant differences ($p=0.046$) between the baseline and two-year rates (Table 7). Thus, the surface sealant was effective at reducing the wear rate of Clearfil S3 Bond restorations, but ineffective at reducing the wear rate of G-aenial Bond restorations. In partial support of our finding, Dickinson and Leinfelder[35] found that after two years, the values for the loss of material was 33.8 μm for unsealed restorations and 26.2 μm for sealed samples, and they stated that the unfilled surface sealant was effective at reducing the wear rates of the composite resin.

No restorations exhibited caries that were contiguous with their margin. In accordance with our findings, no caries were found in Class I restorations after either two or three years.[29,30] However, after 30 months, the caries rates were respectively reported[23] as 0% and 2.7% for nanohybrid and nanofill composite restorations in Class I restorations.

Regarding surface texture, 82.9% of Clearfil S3 Bond and 77.1% of G-aenial Bond restorations were ideal. In partial agreement with our finding, the ideal restoration rates were reported as 59.9%-100% in Class I restorations after two years, three years, and 30 months.[23,29,30] In addition, in Class I/II restorations, the achieved clinically ideal (Alfa) restorations rates were 71.4%-100% after two years.[33,40] The differences in results between our study and those of others may be explained by the differences in the compositions of brands and the physical and chemical properties of the materials used.[23] However, although there were no significant differences in the present study, surface-sealed Clearfil S3 Bond and G-aenial Bond restorations exhibited lower ideal restoration rates than did non–surface-sealed restorations with respect to surface texture. In agreement with our findings, a previous study[35] found no significant differences between sealed and unsealed restorations over five years; however, unsealed restorations had a higher percentage of Alfa ratings until the fifth-year evaluation. Furthermore, surface-penetrating sealants did not improve the roughness of the nanofiller composite resin, which supports our findings.[41] Nevertheless, the surface roughness values of G-aenial Posterior and Filtek Ultimate Universal Restorative increased significantly after the application of surface sealant, and sealant application had no significant effect on the microhardness of Clearfil Majesty Posterior, which was used in the present study.[42] Our study found that surface roughness values increased following the application of surface sealant, compared with unsealed teeth after two years, which may be explained by the relatively high filler content (17.3% vol) and particle size of Fortify Plus.[42] This is supported by the finding that sealant performance worsened compared with that of controls after six months of tooth brushing when filler was added, as in Fortify Plus. The wear in the organic matrix of this sealant potentially allowed the filler to protrude or become lost, which caused a rougher surface.[41]

CONCLUSIONS

None of the restorations failed after two years, and there were no significant differences between the clinical performance of HEMA-containing and HEMA-free all-in-one self-etch adhesives with and without surface sealing in Class I restorations over the same time period. Regarding marginal discoloration, marginal adaptation, and surface texture, each dentin adhesive, no matter whether with or without seal, showed significant changes from clinically ideal to clinically acceptable. Sealed restorations exhibited greater changes in color matching and surface texture than did unsealed restorations. However, the sealing process improved the marginal adaptation of restorations compared with unsealed restorations, and the sealing process reduced the marginal discolorations of the HEMA-free self-etch adhesive restorations.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Kocaeli University, Basiskele, Kocaeli, Turkey. The approval code for this study is KOU KAEK 2014/239.

Conflict of Interest

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

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Effect of Magnification on the Precision of Tooth Preparation in Dentistry

M Eichenberger • N Biner • M Amato • A Lussi • P Perrin

Clinical Relevance

Magnification devices can improve the precision of tooth preparation by dentists.

SUMMARY

Objectives: To evaluate the impact of magnification aids on the precision of tooth preparation under simulated clinical conditions.

Methods and Materials: Two plastic blocks marked with a geometric shape were fixed in a dental phantom head: a circle as the distal surface of tooth 16 (UNS 3) and a y-shaped figure as the occlusal surface of tooth 36 (UNS 19). Sixteen dentists (mean age: 39 years; range: 26-67 years) prepared the geometric shapes from the inside to the boundary line with a cylindrical bur and water-cooling. The boundary line had to be touched but not erased. Chair-side assistance was provided to simulate the clinical situation. Tooth 16 was prepared under indirect vision via a dental mirror. Tooth 36 was prepared under direct vision A) without magnification aids, B) with Galilean loupes, 2.5× and light-emitting diode light, and C) with a microscope, 6.4× and coaxial light. The preparation procedure was performed three times in different sequences of the magnification devices and with a break of at least 1 week between each procedure. The correctly prepared contour and the incorrectly prepared areas were evaluated in relation to the whole circumference of the geometric shapes.

Results: For both values the precision was significantly higher when a microscope was used, followed by preparation using loupes; precision was lowest without magnification aids ($p<0.0001$). This was true for both indirect and direct vision ($p<0.05$).

Conclusions: Magnification devices improved the precision of tooth preparation under simulated clinical conditions.

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INTRODUCTION

The use of magnification aids is widespread in professions requiring manual dexterity and preci-

sion. In dentistry, loupes and operating microscopes have become part of the normal equipment of many dentists. They improve near visual acuity and help to compensate for visual deficiencies.[1-3] Recent studies with miniaturized visual tests on the basis of microfilms have shown a high variability in the near visual acuity of dentists. They found that acuity declined with increasing age of dentists older than 40 years.[1,3-6] The influence of magnification aids on visual performance was evaluated in the same studies. Galilean and Keplerian loupes improved near visual acuity and could compensate for presbyopia in persons older than 40 years. The results of Keplerian loupes were superior to those of Galilean loupes due to their higher magnification. The performance of the operating microscope was outstanding and highly superior compared with loupes.[1,3-5] These basic studies did not evaluate the influence of visual acuity on the quality of dental diagnostics and therapy, however. The subjective conviction that magnification devices improve the precision of manual work is not supported by the weak scientific evidence in this field. Most studies of magnification aids and dental treatment are of low scientific rigor, such as expert opinions,[7-10] case reports,[11-13] and case series.[14,15] The few scientific studies that included a control group or followed a standardized study design reported ambiguous results, and some authors found that magnification devices per se did not lead to better diagnostics or better treatment results.[16-22]

The aim of the present study was to evaluate the impact of optical magnification on the precision of tooth preparation under simulated clinical conditions. The null hypothesis was that magnification has no influence on the precision of tooth preparation.

METHODS AND MATERIALS

Test Subjects

Sixteen dentists participated in the study (mean age: 39 years; median age: 31 years; range: 26-67 years). The dentists were employees of the dental school (n=10) and private dental practitioners (n=6). Inclusion criteria were 1) experience with dental loupes and operating microscopes and 2) near visual acuity in the range of a reference group of dentists as determined in an earlier study.[5] The inclusion threshold for experience was the daily use of both loupes and a microscope, ascertained by questioning the participating dentists. Near visual acuity was assessed by a visual test.

Visual Test

Each participating dentist underwent a near vision test as described by Eichenberger and others.[5] The test was performed without magnification aids but with participants wearing their prescription glasses, if needed. The distance was 300 mm, or the focal distance of the correction glasses.

Geometric Shapes

Plastic teeth (OK T 14 and UK T 14, KaVo Dental AG, Biberach, Germany) of a dental phantom head were prepared for the insertion of standardized geometric shapes from a plastic block (A-PTM 99-001, Frasaco, Tettnang, Germany). A geometric circle was fixed as the distal surface of tooth 16 (universal numbering system: tooth 3) and a y-shaped figure as the distal surface of tooth 36 (universal numbering system: tooth 19) in order to simulate a typical indirect and direct preparation (Figure 1A,B). The plastic blocks were reversibly fixed with superglue (Pattex flüssig 3g, Henkel, Düsseldorf, Germany), which allowed reuse of the teeth for standardization purposes. The phantom head with the teeth described earlier was positioned on the dental chair habitually used by patients of the respective dentists to simulate a typical patient setting.

Preparation Procedure

The cavities were prepared using a handpiece (5:1, KaVo Dental), a cylindrical diamond bur (120-μm grit, 1-mm diameter, ISO 806 314 156 524 010 4.0, Intensiv SA, Montagnola, Switzerland), water-cooling, and compressed air. Chair-side assistance was provided by one of the authors (M.E.). The preparation proceeded from inside to the boundary line, with a predetermined limit of preparation depth between 1.5 and 2.5 mm. This depth was indicated by the colored layers in the plastic block. The black line of the geometric shape had to be touched without erasing it. The preparation time was limited to 5 minutes. Tooth 16 was prepared under indirect vision via a dental mirror (TOPvision FS Rhodium, Hahnenkratt GmbH, Königsbach-Stein, Germany). Tooth 36 was prepared under direct vision, using the dental mirror to check the preparation. Each dentist prepared the shape of tooth 16, followed by the shape of tooth 36, under the following conditions:

A. Naked eye, that is, no magnification devices except prescription glasses and customary operating light

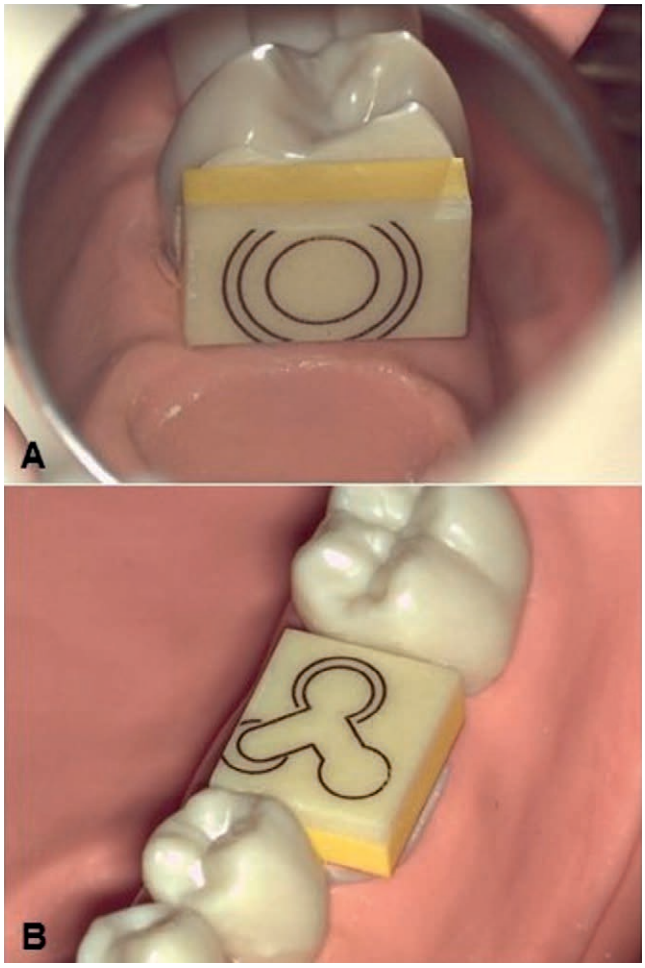

Figure 1. *(A) The circle was fixed as the distal surface of tooth 16 to simulate the clinical situation of indirect preparation (viewed from the 12-o'clock position). (B) The y-shaped figure was fixed as the occlusal surface of tooth 36 to simulate the clinical situation of direct preparation.*

B. Customary Galilean loupes with coaxial light-emitting diode light source; 2.5× magnification factor
C. Operating microscope with integrated light source (Leica, Heerbrugg, Switzerland); 6.4× magnification factor

The test was performed three times in different sequences (A-B-C; B-C-A; C-A-B) with a break of at least 1 week between the tests.

Evaluation of the Prepared Geometrical Shapes

The unprepared surface of the geometrical shapes was colored (Schwan-Stabilo Marker, Heroldsberg, Germany). Photographs of the geometric shapes were taken at 10× magnification using a light microscope (Leica M 420) equipped with a video camera (Leica DFC 495) and linked to a computer. The ideal geometric shapes were superimposed to the photographs of each preparation using the program LAS V4.6.1 (Leica). These superimpositions allowed the user to evaluate the correctly prepared contour (mm) and the sum of overprepared and underprepared areas (mm^2). These values were set in relation to the whole circumference and resulted in two qualitative values for the preparation.

Statistical Analyses

For statistical analysis, the software program R version 3.3.0 (http://www.r-project.org/) was used. The significance level was set at $\alpha=0.05$. The medians of the three preparation sequences were used for the statistical analysis. Descriptive statistics included minimum, maximum, mean, median, and standard deviations. The numeric outcomes were analyzed for differences between the three experimental conditions (eye, loupe, microscope). Because of the small sample size this was done using a nonparametric analysis of variance for longitudinal data according to Brunner and others.[23] The *p*-values were adjusted to take into account the multiple comparisons using the Bonferroni-Holm correction. Post hoc tests were performed without *p*-value adjustment if global tests showed significant main effects or interactions with other variables. Additional questions (ie, on indirect vs direct vision) were answered by performing post hoc Wilcoxon signed-rank tests without *p*-value adjustment.

RESULTS

The near visual test resulted in a mean visual acuity of 1.18, a median of 1.20, and a range of 0.86 to 1.57. These values are within the range of the reference group studied by Eichenberger and others.[5] All test subjects could therefore be included in the study.

The summarized data of both test teeth showed highly significant differences between the three experimental conditions (eye, loupe, microscope) for the percentage of correctly prepared circumference and for the size of the incorrectly prepared area in relation to the circumference ($p<0.0001$, Figures 2 and 3).

A separate analysis of the two teeth allowed a comparison to be made between direct (tooth 36) and indirect vision (tooth 16). The percentage of correctly prepared circumferences is presented in Figure 4 for the three optical conditions and the two teeth separately. For both teeth the percentage of correctly prepared circumferences was significantly higher when a microscope was used, followed by Galilean

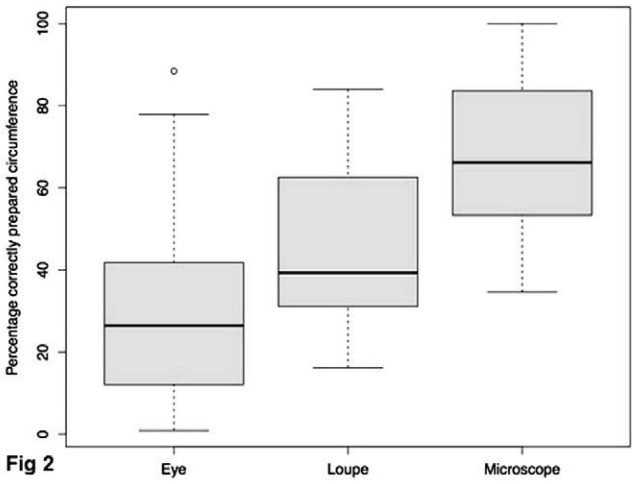

Fig 2

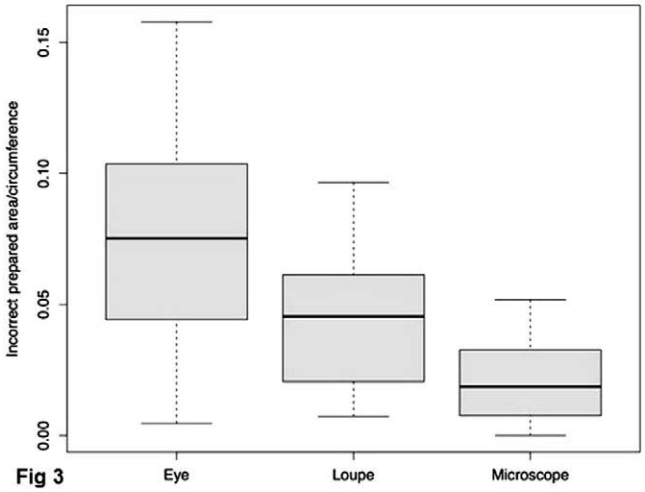

Fig 3

Figure 2. *The summarized data of both teeth showed that the percentage of correctly prepared circumferences was significantly higher when a microscope was used (mean: 67.8%; standard deviation [SD]: 17.6%), followed by loupes (mean: 44.9%; SD: 18.2%) and no magnification aids (mean: 31.2%; SD: 22.7%) (*p*<0.0001).*

Figure 3. *Summarized data of both teeth showed that the incorrectly prepared areas in relation to the circumference were significantly lower when a microscope was used (mean: 0.021 mm^2/mm; standard deviation [SD]: 0.015 mm^2/mm), followed by loupes (mean: 0.045 mm^2/mm; SD: 0.025 mm^2/mm) and no magnification aids (mean: 0.076 mm^2/mm; SD: 0.042 mm^2/mm) (p<0.0001).*

loupes, and was lowest with the naked eye (or wearing prescription glasses) ($p<0.05$). A significantly better performance under direct vision than indirect vision was found for preparations made with the naked eye ($p=0.0076$) and using the Galilean loupe ($p=0.044$). When dentists used the microscope, the difference between direct and indirect vision was not significant ($p>0.05$).

The incorrectly prepared areas in relation to the circumference (mm^2/mm) are presented in Figure 5 for tooth 16 and tooth 36. For both teeth the difference between the naked eye, Galilean loupes, and the microscope was significant ($p<0.05$). A significant difference between direct and indirect vision was noted for the naked eye ($p=0.0052$) but not for the Galilean loupe ($p=0.093$) or the microscope ($p=0.597$).

DISCUSSION

The literature on the effect of using magnification devices on the precision of dental procedures is controversial. To the best of our knowledge no standardized protocol has so far been used to test the impact of loupes or an operating microscope on the precision of tooth preparations. The aim of the present study was to evaluate the effect of magnification on tooth preparation under simulated clinical conditions using a standardized protocol.

To avoid any bias due to limitations of dentists' near vision, a standardized visual test at dental working distance was performed on the study participants.[5] Most of the previous studies about the impact of magnification devices on clinical skills have not tested the dentists' near visual performance, although weak natural near visual acuity might affect the dentists' clinical performance.[16,19,24-28]

To prevent bias resulting from fatigue or training effects, the dentists performed three preparation cycles in rotating order of the visual conditions with a break of at least 1 week between each procedure. The median results of the three cycles were used for statistical analysis to exclude outliers by accidental preparation defaults.

The circle on the distal surface of tooth 16 and the y-shaped figure on the occlusal surface of tooth 36 were chosen to represent common cavities in these locations. The choice of these two locations also allowed comparison of direct vs indirect vision corresponding to the clinical situation. The finding that direct vision allowed a significantly higher precision than indirect vision for preparations made with the naked eye but not for those made using the microscope is of clinical interest and should be further investigated.

The precision of tooth preparation was measured by two values: 1) the percentage of correctly prepared circumference quantified the general precision, and 2) the dimensions of the incorrectly prepared areas were quantified in relation to the circumference, thus giving a weight of the respective imperfections. Both values showed that a highly significantly better performance was obtained using the microscope, followed by Galilean loupes and,

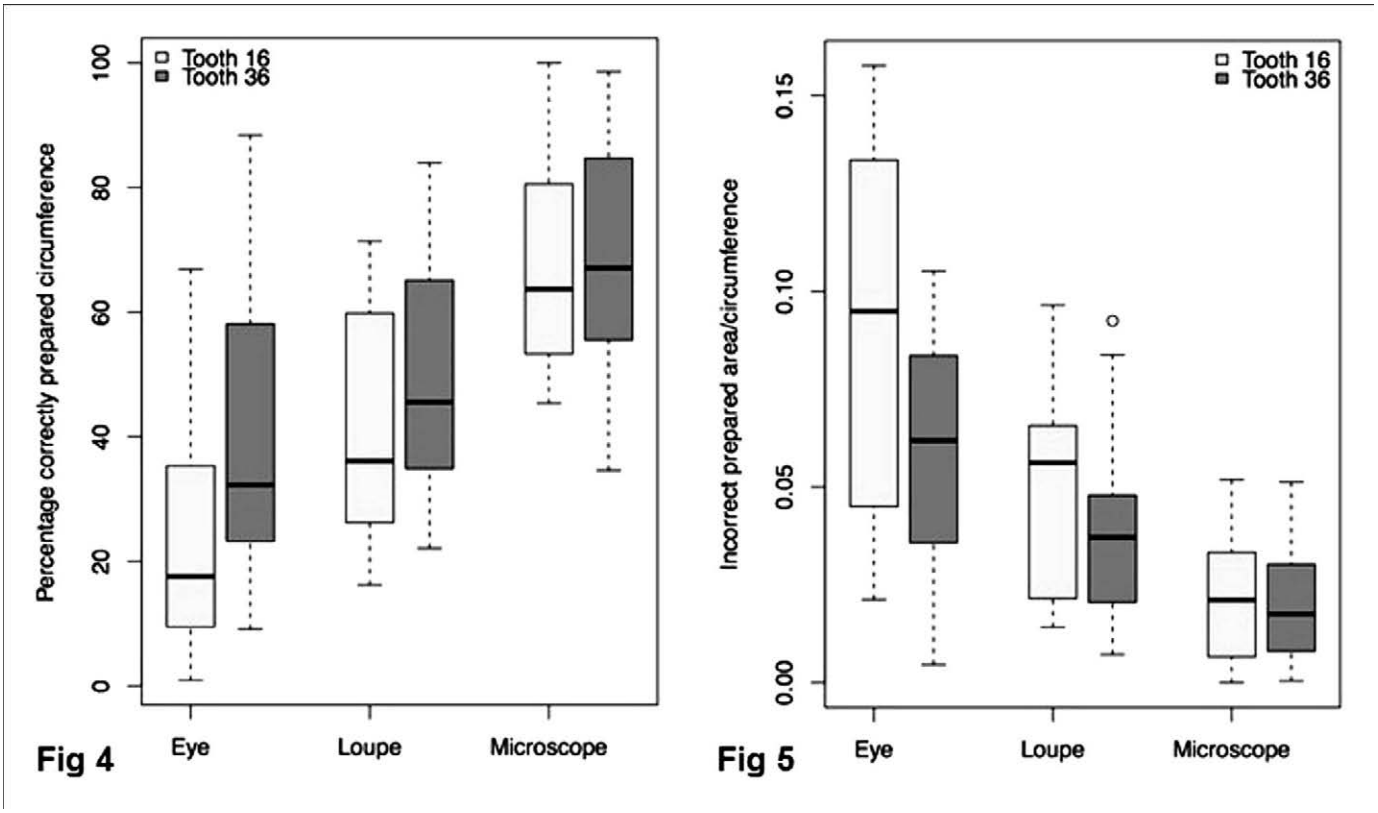

Figure 4. *Percentage of correctly prepared circumferences for direct vision (tooth 36) vs indirect vision (tooth 16). Performance under direct vision was significantly better for preparations made with the naked eye and the Galilean loupe but not the microscope.*

Figure 5. *Incorrectly prepared areas for direct vision (tooth 36) vs indirect vision (tooth 16). Performance under direct vision was significantly better for preparations made with the naked eye but not for those made using loupes or the microscope.*

lastly, the naked eye (with prescription glasses if needed). The results indicate a direct influence of magnification devices on the precision of dental work. This supports commonly expressed expert opinions[29-31] but is in contrast to the results of some experimental studies,[16,17,20] where magnification aids per se did not lead to better clinical outcomes. The inclusion criterion of daily use by the study subjects of all magnification aids tested is essential to avoid bias resulting from lack of expertise. This strict inclusion criterion has not been described in earlier studies and might be a possible explanation for the different outcomes. This criterion, on the other hand, drastically limits the number of potential test subjects and caused the restriction on Galilean loupes in this study. Since earlier studies showed a superior visual performance of Keplerian loupes, it would be interesting to evaluate their impact in a future study. The effects of age and near visual acuity were not further investigated due to the limited number of participants.

CONCLUSION

Magnification devices improved the precision of tooth preparations in a simulated clinical setting. Highly significant differences were noted between preparations made using the optically sophisticated operating microscope, Galilean loupes with coaxial illumination and the naked eye (plus prescription glasses if needed). This was true for direct and indirect vision. The protocol evaluated in this study allowed for an objective assessment of different impacts, for example, magnification aids and direct vs indirect vision, on the precision of tooth preparation.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Kantonale Ethikkommission Bern. There was no approval number or code and documentation was provided.

Conflict of Interest

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

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Literature Review

Influence of Surface Treatment on Composite Adhesion in Noncarious Cervical Lesions: Systematic Review and Meta-analysis

AC Rocha • WLO Da Rosa • AR Cocco • AF Da Silva • E Piva • R-G Lund

Clinical Relevance

Although the meta-analysis demonstrated a positive effect of dentin surface treatment, further clinical trials are needed to determine the best treatment option for improving the retention of resin composite in NCCLs.

SUMMARY

The purpose of this study was to analyze the influence of dentin surface treatments on the retention rate of resin composite restorations in non-carious cervical lesions (NCCLs). Seven randomized clinical trials were included in this review. Data regarding retention rate, type of surface treatment, and the main characteristics of studies were analyzed. Two reviewers performed a literature search up to December 2016 in eight databases: PubMed (Medline), Lilacs, Ibecs, Web of Science, BBO, Scopus, Scielo and The Cochrane Library. Only clinical trials evaluating dentin surface treatments in resin composite restoration in NCCLs were included. Noncontrolled clinical trials, reviews, editorial letters, case reports, case series and studies published in a language other than English, Portuguese, or Spanish were not included. The included studies evaluated different surface treatments, such as using an adhesive system with a frictional technique, drying the dentin, and removing sclerotic dentin by using a bur and applying EDTA before primer use. The analysis considering the mechanical removal of dentin surface with a bur and the application of an adhesive system in a frictional mode showed

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these treatments improved retention rates of the resin composite restorations in NCCLs ($p<0.05$). There is evidence in the literature suggesting that the mechanical removal of dentin surface with a bur and the application of an adhesive system in a frictional mode could improve the retention rates of resin composite restorations in NCCLs. However, the studies showed high heterogeneity, and additional clinical trials are needed to determine the best dentin treatment option in NCCLs.

INTRODUCTION

The prevalence of noncarious cervical lesions (NCCLs) varies from 5% to 85% and has been increasing in recent years due to an aging population, especially in premolars.[1-5] Among the main dental materials used for the treatment of NCCLs are resin composites. They are used to protect the affected teeth against the loss of a healthy tooth structure, to improve esthetics, and to treat dental hypersensitivity, as well as when the affected tooth is used as lateral support for a removable partial denture.[6,7]

Treatment of NCCLs remains a major challenge for dentists, and studies have reported that the retention loss of NCCLs can vary from 0% to 50%.[8] Restoration loss especially occurs because of the difficulty of dental material adhesion;[9] a high degree of sclerosis can exist, and a high amount of minerals can impair the adequate establishment of a hybrid layer.[6,8] Besides, many studies have reported the loss of retention and marginal discoloration after cervical restorations with resin composites.[8,10] To minimize these problems, tooth surface treatment has been suggested to improve resin composite adhesion in NCCLs.[11-13]

The surface treatment techniques that were proposed include surface irrigation with EDTA, adhesive application with a frictional technique, and drying the dentin before adhesive application.[11,13-15] Some studies have already evaluated the effects of surface treatment on NCCL treatment, but a question still remains about whether clinicians should consider using these approaches to improve the adhesion of resin composites. Therefore, the aim of this study was to analyze the influence of dentin surface treatments on the retention rate of resin composite restorations in NCCLs. Our hypothesis evaluated whether surface treatments could improve resin composite retention.

METHODS AND MATERIALS

The protocol of this review was registered in the PROSPERO international database for systematic reviews (CRD42014010018). This systematic review is reported according to the Preferred Reporting Items for Systematic Reviews and Meta-Analysis (PRISMA Statement).[16] To formulate the question from evidence-based practice, the following PICO (Population, Intervention, Comparison and Outcomes) was established: the population was patients who present with NCCLs; the intervention was dentin surface treatment (any nonconventional procedure performed prior to resin composite restoration, such as surface irrigation with EDTA, adhesive application with a frictional technique, drying the dentin before adhesive application); the comparison was teeth without dentin surface treatment; and the outcome was retention rate (complete or partial loss of the restoration). The research question was as follows: Does dentin surface treatment improve the retention rate of resin composite restorations in NCCLs?

Search Strategies

The literature search was carried out by two independent reviewers of studies published from 1940 to December 2016. Eight databases were screened, including PubMed (Medline), Lilacs, Ibecs, Web of Science, BBO, Scopus, Scielo, and The Cochrane Library, using the search strategy developed for PubMed (Medline) and adapted for other databases (Table 1). The references cited in the included papers were also checked to identify other potentially relevant articles. After the identification of articles in the databases, the articles were imported into Endnote X7 software (Thompson Reuters, Philadelphia, PA, USA) to remove duplicates.

Study Selection

Two authors independently assessed the titles and abstracts of all the documents. The studies were analyzed according to the selection criteria described in Table 2. Full copies of all the potentially relevant studies were identified. Those appearing to meet the inclusion criteria or for which there were insufficient data in the title and abstract to make a clear decision were selected for full analysis. The full-text papers were assessed independently and in duplicate by two authors. Any disagreement regarding the eligibility of the included studies was resolved through discussion and consensus or by a third reviewer. Only

Table 1: *Search Strategy Used in PubMed (MedLine)*

Search	Search Terms
#3	Search #1 AND #2
#2	"Non-carious cervical lesions" OR "Non-carious cervical lesion" OR "non-carious, cervical lesion" OR "non-carious cervical lesions" OR "Tooth Wear"[Mesh] OR "tooth wear" OR "Tooth Wears" OR "Wear, Tooth" OR "Wears, Tooth" OR "Dental Wear" OR "Dental Wears" OR "Wear, Dental" OR "Wears, Dental" OR "Tooth Loss"[Mesh] OR "tooth loss" OR "Loss, Tooth" OR "Tooth Cervix"[Mesh] OR "Cervix, Tooth" OR "Cementoenamel Junction" OR "Cementoenamel Junctions" OR "Junction, Cementoenamel" OR "Junctions, Cementoenamel" OR "Cervix Dentin" OR "CEJ" OR "Tooth Abrasion"[Mesh] OR "Abrasion, Tooth" OR "Abrasion, Dental" OR "Dental Abrasion" OR "Tooth Erosion"[Mesh] OR "Erosion, Tooth" OR "Erosions, Tooth" OR "Tooth Erosions" OR "class V restorations" OR "composite class V"
#1	"Clinical Trial" [Publication Type] or clinical trial or ""Study Characteristics" [Publication Type] or study characteristics or randomized controlled trial[pt] OR controlled clinical trial[pt] OR randomized controlled trials[mh] OR random allocation[mh] OR double-blind method[mh] OR single-blind method[mh] OR clinical trial[pt] OR clinical trials[mh] OR ("clinical trial"[tw]) OR ((singl*[tw] OR doubl*[tw] OR trebl*[tw] OR tripl*[tw]) AND (mask*[tw] OR blind*[tw])) OR ("latin square"[tw]) OR random*[tw] OR research design[mh:noexp] OR follow-up studies[mh] OR prospective studies[mh] OR cross-over studies[mh] OR control*[tw] OR prospectiv*[tw] OR volunteer*[tw])

papers that fulfilled all eligibility criteria were included.

Data Extraction

The data were extracted using a standardized form. If there was some information missing, the authors of the included papers were contacted via e-mail to retrieve any missing data. The following data were tabulated: study design, publication year, country, number of patients, gender, age, evaluation criteria, follow-up (months), and number of teeth evaluated (Table 3). The characteristics of the included studies, such as selection criteria, surface treatment, control group, and restoration characteristics (brand, company and country), were also analyzed (Table 4).

Assessment of Risk of Bias

The methodologic quality was assessed by the two reviewers. Studies were evaluated and classified according to Cochrane guidelines[17] for the following items: selection bias (sequence generation, allocation concealment), performance and detection bias (blinding of operators or participants and personnel), bias due to incomplete data, reporting bias (selective reporting, unclear withdrawals, and missing outcomes), and other bias (including industry sponsorship bias).

Statistical Analysis

The analyses were performed using Review Manager Software version 5.2 (The Nordic Cochrane Centre, The Cochrane Collaboration, Copenhagen, Denmark). The global analysis was carried out using a random-effects model, and pooled-effect estimates were obtained by comparing the risk difference of each dentin surface treatment group with the conventional protocol (control); $p<0.05$ was considered statistically significant. Multiple groups from the same study were analyzed according to Cochrane guidelines for combining groups.[18] Subgroup analyses were also performed considering the mechanical removal of dentin surface with a bur, the application of an adhesive system in a frictional mode, or in dry dentin. Statistical heterogeneity of the treatment effects among studies were assessed using the Cochran's Q test and the inconsistency I^2 test, in

Table 2: *Inclusion and Exclusion Criteria*

PICO	Inclusion Criteria	Exclusion Criteria
Population	Studies of participants with: ■ Noncarious cervical lesions	
Intervention	Studies with subjects who have undergone to the following procedures: ■ Pretreatment of dentin surface before resin composite restoration in noncarious cervical lesions	Studies with subjects who have undergone to the following procedures: ■ Used restorative dental materials different than resin composite (such as glass-ionomer cements) in noncarious cervical lesions
Outcomes	Studies investigating: ■ Retention rate	
Study design	■ Prospective or retrospective clinical trials	■ Noncontrolled clinical trials, reviews, editorial letters, case reports, case series ■ Studies published in a language other than English, Portuguese, or Spanish

Table 3: *Description of Demographic Data, Study Design, and Main Objectives of Included Studies*

Study (Authors)	Year	Country	Study Design	Number of Patients	Sex (Number)		Age (years)	Evaluation Criteria	Follow-Up (Months)	Number of teeth
					Women	Men				
Van Dijken[13]	2010	Sweden	RCT	72	42	30	42-84	USPHS	6, 12, 18, 24, 36, 48, 60, 72, 84, and 96 months	112
Loguercio and others[26]	2011	Brazil	RCT	40	63	57	20->49	USPHS	6, 12, and 24 months	120
Dalkiliç and Omurlu[14]	2012	Turkey	RCT	29	13	16	30-70	Modified USPHS	3, 12, and 24 months	158
Luque-Martinez and others[11]	2015	Chile	RCT	48	22	26	40-48	FDI	6, 12, and 18 months	77
Zander-Grande and others[12]	2014	Brazil	RCT	31	19	12	20->49	FDI	6, 12, and 24 months	124
Zander-Grande and others[35]	2011	Brazil	RCT	40	29	11	20->49	USPHS	6, 12, and 24 months	160
Perdigão and others[15]	2014	United States	RCT	39	15	24	20->49	FDI; USPHS	6 and 18 months	196

FDI, World Dental Federation; RCT, randomized clinical trial; USPHS, United States Public Health Service.

which values greater than 50% were considered indicative of substantial heterogeneity.[17]

RESULTS

Search Strategy

A total of 4040 potentially relevant records were identified from all the databases, of which 1046 were duplicates. No additional studies were identified as relevant after a search of the reference lists. Figure 1 is a flowchart that summarizes the article selection process according to the PRISMA Statement.[12] After the title and abstract examination, 2971 studies were excluded because they did not meet the eligibility criteria. Of the 23 studies retained for detailed review, 16 studies were not included because 11 evaluated the restoration material without surface treatment[19-29] and five papers were *in vitro* studies.[30-34] A total of seven studies fulfilled all of the selection criteria and were included in the qualitative analysis.

Descriptive Analysis

The studies were published between 2010 and 2015. All studies were randomized clinical trials. The sample size ranged from 29 to 72 subjects. A total of 947 teeth were evaluated in this review for all the included clinical trials. The ages of the patients ranged from 20 to 84 years old. All clinical studies had a minimum of 18 months of follow-up. Five studies evaluated the restorations according to United States Public Health Service (USPHS) criteria, and three used World Dental Federation (FDI) criteria.

Two studies evaluated the application of the adhesive system with the frictional technique,[12,15] and two others analyzed the effects of dry and moist dentin.[12,19] Additionally, one study investigated the removal of sclerotic dentin through the use of a bur,[14] and in another, only the roughness of the tooth surface with a bur was investigated.[36] Moreover, only one study applied EDTA before primer use in NCCLs.[11]

Risk of Bias of Included Studies

Concerning the quality assessment (Figure 2), these studies presented a low risk of bias for most of the biases that were analyzed. Only two studies[13,14] did not report blinding the participants and personnel, as well as blinding the outcome assessments.

Meta-Analysis

A meta-analysis was performed for the six randomized clinical trials. Considerable heterogeneity was observed in this analysis (I^2=55%). The analysis considering the mechanical removal of dentin surface with a bur (Figure 3A) and the application of an adhesive system in a frictional mode (Figure 3B) showed these treatments improved retention rates of the resin composite restorations in NCCLs ($p<0.05$). However, when considering only the application of an adhesive system in dry dentin compared with a control, no statistically significant differences were observed (Figure 3C).

DISCUSSION

The hypothesis that was evaluated was accepted once our meta-analysis demonstrated that dentin surface treatments could improve resin composite retention in NCCLs. All studies were randomized, and in general, they presented a low risk of bias because the evidence obtained from them had high quality. Among the surface treatments suggested to

Table 4: *Main Characteristics of the Included Studies*

Study	Selection Criteria	Surface Treatment	Control Group
Van Dijken[13]	Healthy subjects; treatment of NCCL	Surface roughened by a diamond bur followed by application of an etch-and-rinse or self-etch adhesive	Surface not roughened before adhesive application
Loguercio and others[26]	Healthy subjects; no dentin hypersensitivity; treatment of NCCL	Slight rubbing action: adhesive lightly spread on the surface for approximately 10 seconds (pressure was equivalent to approximately 4.0±1.0*g*). An air stream was applied for 10 seconds at a distance of 20 cm. Vigorous rubbing action: adhesive rigorously agitated on the surface for approximately 10 seconds (pressure equivalent to approximately 34.5±6.9*g*). An air stream was applied for 10 seconds at a distance of 20 cm	No rubbing action: adhesive only spread over the entire surface for approximately 3 seconds and left undisturbed for 7 seconds. An air stream was applied for 10 seconds at a distance of 20 cm.
Dalkiliç and Omurlu[14]	Healthy subjects; treatment of NCCL	Outer surface of the sclerotic dentin was removed by roughening with a diamond bur, then etch-and-rinse or self-etch adhesive was applied.	Adhesive application without prior removal of sclerotic dentin
Luque-Martinez and others[11]	Healthy subjects; treatment of NCCL	Surfaces were treated with 17% EDTA for 2 minutes, copiously rinsed with water for 30 seconds, and slightly dried with an air stream for five to 10 seconds while keeping the dentin surface slightly moist before the adhesive was applied.	Adhesive application without surface treatment with EDTA
Zander-Grande and others[12]	Healthy subjects; treatment of NCCL	Adhesive was rigorously agitated on the entire dentin surface for approximately 15-20 seconds. A microbrush was used to scrub the dentin surface under manual pressure (equivalent of approximately 34.5±6.9*g*). An airstream was applied for 10 seconds at a distance of 20 cm. The air-dry pressure used was 40 psi.	Adhesive only spread over the entire surface for approximately three to five seconds and was left undisturbed for 15 to 20 seconds. An airstream was applied for 10 seconds at a distance of 20 cm.
Zander-Grande and others[35]	Healthy subjects; not included teeth with dentin sclerosis; treatment of NCCL	Adhesive application with dry dentin	Adhesive application with moist dentin
Perdigão and others[15]	Healthy subjects; treatment of NCCL	Adhesive application with dry dentin	Adhesive application with moist dentin

Table 4: *Main Characteristics of the Included Studies (ext.)*

Study	Restoration	Dropouts	Failures
Van Dijken[13]	*Adhesive System:* Clearfil SE Bond (Kuraray Co. Ltd., Osaka, Japan) in self-etch mode PQ 1 (Ultradent, South Jordan, Utah, United States) in etch-and-rinse mode *Composite Resin:* Tetric Ceram (Ivoclar/Vivadent, Schaan, Liechtenstein) Point 4 (Kerr Corp., Orange, United States)	Seven restorations	Retention
Loguercio and others[26]	*Adhesive System:* Prime & Bond NT (Dentsply DeTrey, Konstanz, Germany) in etch-and-rinse mode *Composite Resin*: Esthet-X (Dentsply DeTrey, Konstanz, Germany)	None	Retention
Dalkiliç and Omurlu[14]	*Adhesive System:* Single Bond (3M ESPE, St. Paul, Minnesota, United States) in etch-and-rinse mode Clearfil SE Bond (Kuraray Medical, Tokyo, Japan) in self-etch mode XENO III (Dentsply/DeTrey, Konstanz, Germany) in self-etch mode *Composite Resin:* Filtek Supreme (3M ESPE, St. Paul, Minnesota, United States)	Six patients (94 restorations)	Retention
Luque-Martinez and others[11]	*Adhesive System:* Adper Easy One (3M ESPE, St. Paul, Minnesota, United States) in self-etch mode *Composite Resin:* Filtek Z350XT (3M ESPE, St. Paul, Minnesota, United States)	Six patients	Retention
Zander-Grande and others[12]	*Adhesive System:* Adper Prompt L-Pop (3M ESPE, St. Paul, Minnesota, United States) in self-etch mode XENO III (Dentsply Caulk, Milford, DE, United States) in self-etch mode *Composite Resin:* Filtek Z250 (3M ESPE, St. Paul, Minnesota, United States) Esthet X (Dentsply Caulk, Milford, DE, United States)	None	Retention
Zander-Grande and others[35]	*Adhesive System:* One-Step Universal Dental Adhesive System (Bisco, Schaumburg, Illinois, United States) in etch-and-rinse mode Adper Single Bond Plus Adhesive (3M ESPE, St. Paul, Minnesota, United States) in etch-and-rinse mode *Composite Resin:* Filtek Z250 (3M ESPE, St. Paul, Minnesota, United States)	None	Retention
Perdigão and others[15]	*Adhesive System:* Scotchbond Universal Adhesive (3M ESPE, St. Paul, Minnesota, United States) in etch-and-rinse, self-etch and selective etching mode *Composite Resin:* Filtek Supreme Ultra (3M ESPE, St. Paul, Minnesota, United States)	1 patient	Retention, marginal staining and postoperative sensitivity

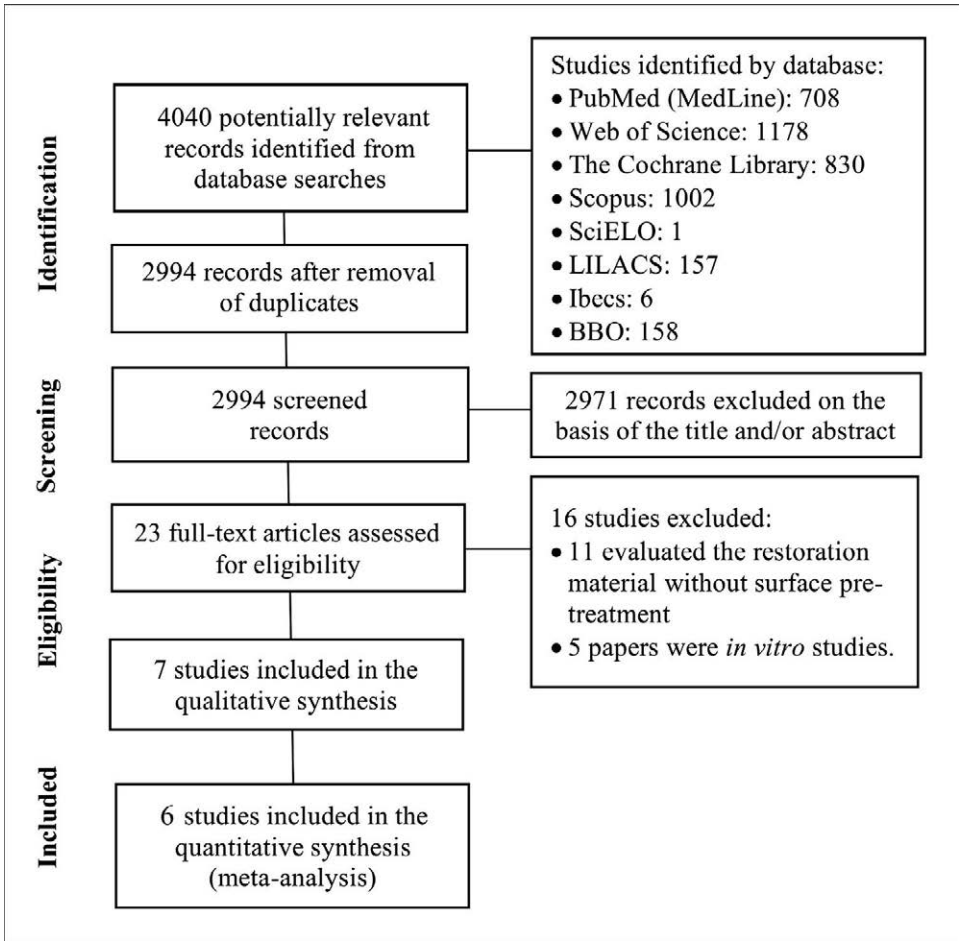

Figure 1. *Search flow (as described in the PRISMA statement).*

improve resin composite retention, two studies evaluated the application of an adhesive system in a frictional mode.[12,26] Both studies reported that vigorous application of the adhesive system improved the retention of restorations in NCCLs. One of the studies compared two different frictional techniques, including a slight rubbing action and the vigorous application of adhesives, and the researchers concluded that the vigorous application could be a clinical approach to improve the retention of resin composite in NCCLs.[26] It was reported that this approach improved the bond strength of self-etch adhesives to enamel[37,38] and to dentin.[38–41] In dentin, the active adhesive system application may have improved smear layer dissolution, micromechanical interlocking and chemical interactions.[42]

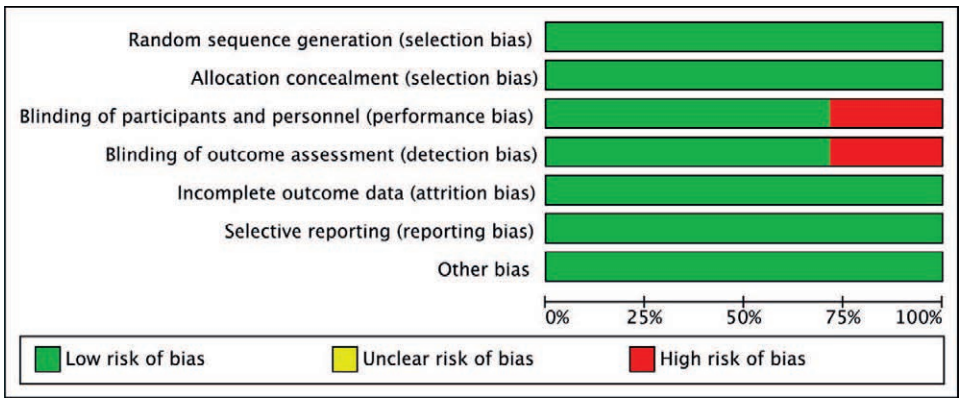

Figure 2. *Risk of bias graph: authors' judgments about each risk of bias item presented as percentages across all included studies.*

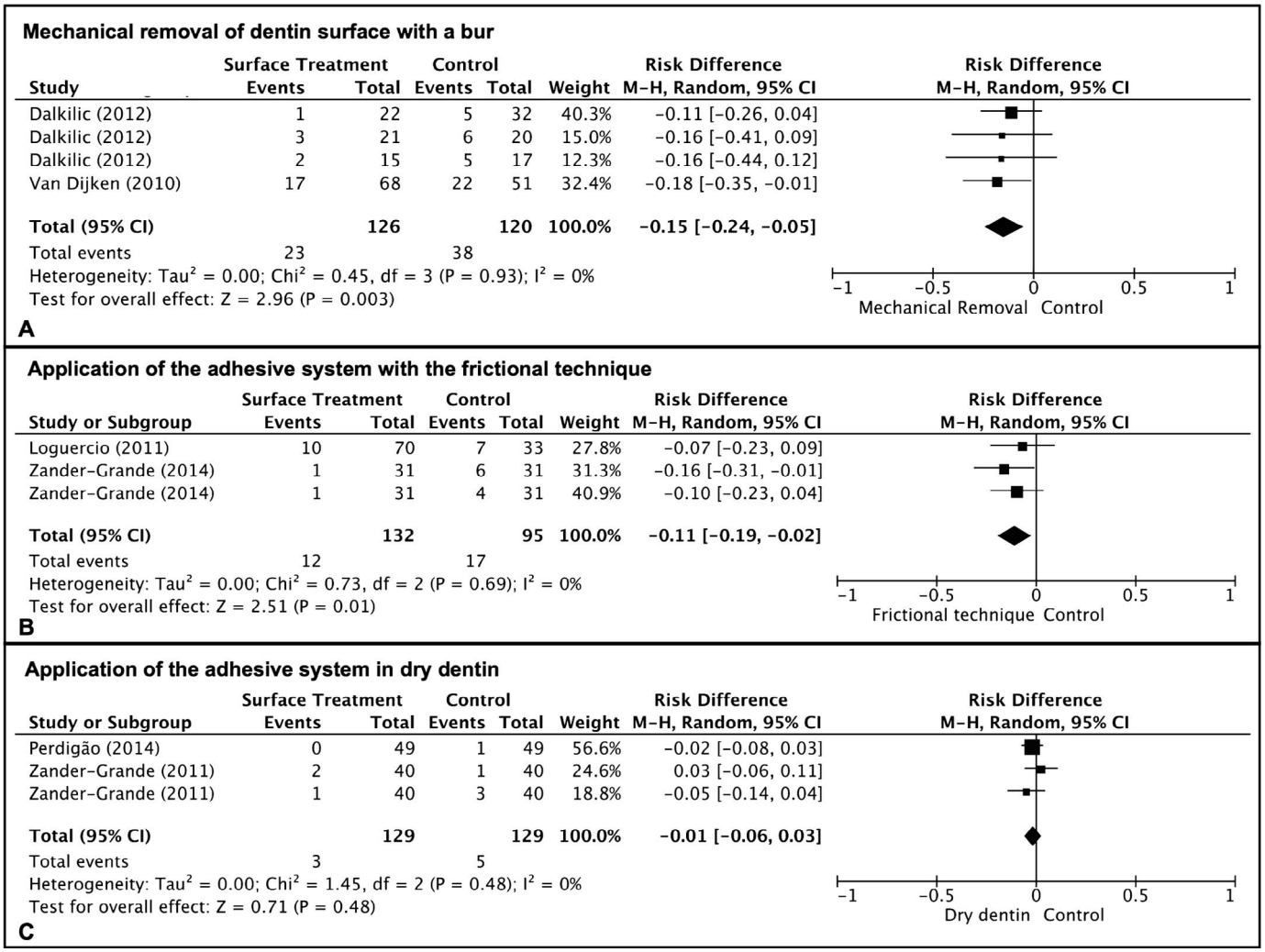

Figure 3. *Meta-analysis considering (A) the mechanical removal of dentin surface with a bur, (B) the application of an adhesive system in a frictional mode or (C) in dry dentin.*

Although clinical trials evaluating this approach are limited,[14] the available literature suggests that adhesive application in a frictional mode could improve the retention rates of resin composite.

One of the factors that could compromise adhesion in NCCLs is the presence of dentin sclerosis, which consists of a hypermineralized dentin that has difficult resin composite adhesion.[6,43] It was reported that 54%, 22%, and 2% of NCCL presented mild, moderate, and heavy dentin sclerosis, respectively.[2] Alternative strategies for adhesion to sclerotic dentin have been recommended by previous researchers.[6] It was observed that lesions were slightly roughened before adhesive system application showed lower loss rates of restoration compared with unroughened adhesives.[13,14] This difference probably occurred because these lesions had a high degree of sclerosis. In the present review, one study investigated the removal of the surface layer of sclerotic dentin through the use of a bur[14] and showed no improvement in the retention and marginal staining after 24 months. On the other hand, one clinical trial showed that roughening the tooth surface with a bur prior to an etch-and-rinse or self-etch adhesive application improved retention rates after 8 years for adhesives used in both sclerotic and nonsclerotic dentin.[13]

Furthermore, many failures of restoration are due to the technique sensitivity of the adhesive systems, and some studies have questioned the technique of using moist dentin. It is necessary to preserve demineralized dentin to allow adequate resin monomer infiltration, and this sensitivity technique is a major challenge for students or inexperienced dentists.[44–46] Due to these disadvantages, other dentin surface treatments that were

evaluated included the application of an adhesive system over dry and moist dentin.[15,35] It was suggested that the maintenance of demineralized dentin in a dry state could minimize the negative effects of water on hybrid layer formation.[15] Additionally, universal adhesives were evaluated in two studies, and they were applied in self-etch, etch-and-rinse, and selective enamel etching modes, which allowed the clinician to decide on a specific adhesive protocol that was best suited for the cavity that was being prepared.[46] Studies that evaluated the application of these adhesives in etch-and-rinse or self-etch modes,[15,35] both in moist and dry dentin, demonstrated that the behavior of the adhesive was not dependent on the bonding strategy that was used after 18[15] and 24 months.[35] Further studies with longer follow-up periods are needed to evaluate if dry dentin can increase the retention rate of resin composite in NCCLs.

Another method that was evaluated was the application of EDTA, which could dissolve the smear layer and permit a chemical bond between the primer/bond and the dentinal collagen and calcium.[47,48] As a consequence, better interaction of the adhesive with the sclerotic dentin might occur, which would improve the etching pattern and the interaction between self-etch adhesives and sclerotic dentin.[11,49,50] EDTA is widely recognized as an effective inhibitor of endogenous metalloproteinases (MMPs),[51] and the application of 17% EDTA for two minutes could significantly reduce the activity of dentin MMPs.[52] EDTA also chelates zinc and calcium ions that are essential for MMP activity.[53,54] *In vitro* studies have reported promising results after preliminary conditioning with EDTA, which produced a shallow demineralization of the dentin.[49,50] In this review, one study evaluated the application of 17% EDTA for two minutes prior to adhesive application and showed a significant increase in the retention rates of composite restorations in NCCLs after 18 months.[11] It is reported that EDTA could improve adhesion of the self-etch adhesives to sclerotic dentin[49,50,55] by producing selective dissolution of hydroxyapatite and "kidnapping" the metallic ions presented in dentin,[56,57] enhancing the interaction of the self-etch adhesive with the tissue.[49,50,55]

The studies also varied in terms of the evaluation methods used for clinical assessment of the restorations of NCCLs. Most studies used the USPHS and FDI criteria.[58] The USPHS mainly evaluated marginal adaptation staining, retention, fracture, marginal discoloration, postoperative sensitivity and recurrence of caries,[13,26,35] whereas FDI evaluated esthetic properties (staining margin), functional properties (fractures and retention, marginal adaptation), and biological properties (postoperative sensitivity, secondary caries).[11,12] We used the outcome "retention rate" that both criteria analyzed, which allowed the comparisons. A recent study[15] that compared both methods concluded that the FDI criteria were more sensitive to small variations in the clinical outcomes than were the USPHS criteria when evaluating restorations of NCCLs.

One limitation of our review is that the included studies presented substantial heterogeneity, which was probably due to the different surface treatments that were evaluated, follow-up periods, evaluation criteria, materials that were tested and outcomes that were assessed. This high heterogeneity and the small amount of studies regarding surface treatment in NCCLs made other comparisons difficult. Furthermore, an important factor that influences retention of NCCL restoration is the kind of adhesive system (etch-and-rinse or self-etch) used. However, due to the small number of included studies, the results of this study could not be controlled for this confounder. Further studies comparing different dentin surface treatments need to be performed to determine the best treatment option that can improve the longevity of resin composite in NCCLs.

In summary, the influence of dentin surface treatment in the retention rates of resin composite restorations in NCCLs was dependent on the strategy that was used. The application of an adhesive system in a frictional mode and the mechanical removal of dentin surfaces with a bur improved retention rates of resin composite restorations in NCCLs. Although the meta-analysis demonstrated a positive effect of treatment, only a few studies were already available in the literature, and further clinical trials are needed to determine the best treatment option. Additionally, there was not enough evidence to support this conclusion over longer-term follow-up, as the majority of included studies evaluated the outcomes for up to 24 months.

CONCLUSIONS

There is evidence in the literature suggesting that the mechanical removal of dentin surfaces with a bur and the application of an adhesive system in a frictional mode may improve the retention rates of resin composite restorations in NCCLs. However, the studies showed high heterogeneity, and addi-

tional clinical trials are needed to compare different strategies in longer-term follow-ups and determine the best treatment option for resin composite in NCCLs.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the PROSPERO International Database for systematic reviews. The approval code for this study is CRD42014010018.

Conflict of Interest

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

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Laboratory Research

Effect of Mold Type and Diameter on the Depth of Cure of Three Resin-Based Composites

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Clinical Relevance

Wide multisurface composite restorations may achieve a greater depth of cure than a narrower restoration, especially when a less opaque matrix is used. Clinicians should not attempt to light cure a 4 mm increment of conventional composite, even with an increased exposure time.

SUMMARY

Objective: To evaluate the effects of different mold materials, their diameters, and light-curing units on the mechanical properties of three resin-based composites (RBC).

Methods and Materials: A conventional nano-filled resin composite (Filtek Supreme Ultra, 3M Oral Care, St Paul, MN, USA) and two bulk-fill composites materials, Tetric Evoceram Bulk fill (Ivoclar Vivadent, Schaan, Liechtenstein) and Aura Bulk Fill (SDI, Bayswater, VIC, Australia), were tested. A total of 240 specimens were fabricated using metal or white semitransparent Delrin molds that were 4 or 10 mm in diameter. The RBCs were light cured for 40 seconds on the high-power setting of either a monowave (DeepCure-S, 3M Oral Care) or polywave (Bluephase G2, Ivoclar Vivadent) light-emitting diode (LED) curing unit. The depth of cure was determined using a scraping test, according to the 2009 ISO 4049 test method. Data were analyzed using multivariate analysis of variance followed by Tukey multiple comparison test ($p<0.05$).

Results: In general, when used for 40 seconds, both LED curing lights achieved the same depth of cure ($p=0.157$). However, the mold material and its diameter had a significant effect on the depth of cure of all three RBCs ($p<0.0001$).

Conclusion: Curing with either the polywave or monowave LED curing light resulted in the same depth of cure in the composites. The greatest depth of cure was always achieved using the 10-mm-diameter Delrin mold. Of the three RBCs tested, both Tetric Bulk Fill and Aura achieved a 4-mm depth of cure when tested in the 10-mm-diameter metal mold. Tetric Bulk Fill was the most transparent and

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had the greatest depth of cure, and the conventional composite had the least depth of cure. Very little violet (<420 nm) light penetrated through 6 mm of any of the RBCs.

INTRODUCTION

Photopolymerizable resin-based composites have become the material of choice for direct restorations, and as a consequence of the recommendations of the 2013 Minamata agreement, they are replacing dental amalgam.[1] Using a resin-based composite (RBC) allows for more conservative tooth preparation designs, improved reparability, and an esthetic tooth-colored restoration.[2]

Resin-based composites are highly cross-linked polymeric materials that contain pigments and filler particles that determine their final properties.[3,4] While the resin matrix is considered to be the skeleton of the RBC, the inorganic fillers improve wear resistance, reduce polymerization shrinkage, reduce the coefficient of thermal expansion, and reduce water absorption.[3]

Camphorquinone (CQ) is the most commonly used photoinitiator, with ethyl-4-(N, N-dimethyl-amino) benzoate often used as an amine co-initiator. CQ is activated by a broad range of light but is most sensitive to blue light at approximately 468 nm, and also to light below 320 nm. Thus, CQ allows photopolymerization of the resin after it has been irradiated with blue light from a dental curing unit that delivers light in the 400- to 500-nm wavelength range.[3]

To overcome some of the yellow color–related side effects of using CQ as a photoinitiator, some light-shaded or translucent RBCs also include alternative photoinitiators such as 2,4,6-trimethylbenzoyldiphenylphosphine oxide (TPO), 1-phenyl-1,2-propanedione (PPD), or the germanium based initiator, Ivocerin.[5] Improved color stability and a higher degree of conversion has been reported for RBCs that contain TPO.[6,7] The PPD initiator is sometimes used in combination with CQ to reduce its yellowing effects, enhance the photoinitiation reaction, and possibly reduce the polymerization stress that is produced by the cured RBC.[8,9] The dibenzoylgermanium compound, Ivocerin, has a higher photoinitiation activity and also absorbs visible light over a wider range of wavelengths (from 370 to 460 nm).[10,11]

The maximum degree of conversion (DC) for light activated dimethacrylate polymers is never 100%, but instead, it ranges between 43% and 75% depending on the brand of RBC and measurement method.[12] Ideally, the RBC should achieve a high degree of monomer conversion so that the manufacturer's desired physical properties and the most biocompatible RBC are achieved in the tooth.[13,14] The depth of cure (DOC) of the RBC can be affected by many factors, including exposure time, radiant exposure, the type of the light-curing unit used, thickness of RBC, its volume, and the type of photoinitiator used within the RBC.[15,16]

Although considered to be the gold standard, incremental placement of 2-mm-thicknesses of RBC is time-consuming. Incremental placement also increases the chance of void incorporation and contamination between each layer of RBC.[17,18] To reduce placement time, reduce the possibility of contamination between increments, minimize polymerization shrinkage, and allow improved polymerization at greater depths, bulk-fill RBCs have been introduced.[18] These bulk-fill materials should provide adequate curing of 4- to 5-mm-thick increments of RBC.[11,19] This is due to the use of improved photoinitiator systems, improved matching of the refractive indices between the resin and the filler, and overall increased translucency of the matrix that allows greater penetration of light down into the deeper areas of the RBC.[20]

Different types of light-curing units (LCU) are available, such as the conventional quartz tungsten halogen (QTH), plasma arc, and light-emitting diode (LED) curing light units.[21] QTH units emit a broad spectrum of light from 400-500 nm that is compatible with the most commonly used photoinitiator, CQ, and all newer generations of photoinitiators.[22] However, QTH units have several limitations: the high operating temperature of the QTH bulb, a short life span for the QTH bulb of only about 50 working hours, and a relatively large device size.[23] In the mid-1990s, the first generation of LED curing lights was introduced.[24] These LED units emitted blue light in a narrow range of wavelengths that were compatible with the activation range of CQ.[25,26] The LED emitter should last for thousands of hours, and they can be battery powered (cordless).[25] Consequently, the use of QTH units has fallen, and battery-operated LED units now dominate the market. The first generation of LED units delivered only a low power output, and their curing efficacy was questionable until the next generation of higher-power LED units was developed.[27] These LED LCUs deliver equivalent or higher powers than the QTH units, and some of them emit light in two or more different wavelength ranges (they are sometimes

called third-generation broad-spectrum or polywave LED LCUs). These units produce both violet (shorter) wavelength and blue (longer) wavelengths of light from two or more different LED emitters located within the unit. The violet light is used to activate photoinitiators that are most sensitive to light that is shorter than 420 nm in wavelength,[7,28] whereas the blue light activates the CQ photoinitiator.[28] Therefore, these polywave LED LCUs can activate all of the currently used photoinitiators.[7,29] Santini and others[30] reported higher DC values in 2-mm-thick specimens of RBCs that contained the TPO photoinitiator when cured with a polywave LED LCU compared with when a monowave LED LCU was used. On the other hand, when Menees and others,[31] in 2015, evaluated the DOC of Tetric Evoceram Bulk Fill and Filtek Bulk Fill Posterior RBCs cured with either a monowave (Elipar S10 delivering 13.5 J/cm^2) or a polywave (Bluephase G2 delivering 10.9 J/cm^2) LCU, they found that Tetric Evoceram Bulk Fill showed a deeper DOC than Filtek Bulk Fill Posterior in a 4 mm wide metal mold, but not in a tooth mold. They found no statistically significant effect of the different LED LCU types on the DOC, despite the different photoinitiators used within the RBCs. This result was surprising because according to the manufacturer, the polywave LCU should be expected to better activate the Ivocerin photoinitiator used in Tetric Evoceram Bulk Fill and provide an improved DOC.[31] A similar result was reported in 2016 by Issa and others,[32] who tested the nanohardness and elastic modulus of Tetric Evoceram Bulk Fill RBC cured with polywave LED LCUs in 6-mm-diameter metal molds. Their study design allowed them to determine which wavelengths were delivered to different regions of the test specimens, and they found no differences in the tested properties of Tetric Bulk Fill to a depth of 4 mm when mostly lower-wavelength violet light was delivered compared with when mostly longer-wavelength blue light was delivered. However, it was reported that delivering mostly lower-wavelength violet light adversely affected the DOC of Filtek Bulk Fill flowable (FBFF,) whereas delivering mostly longer-wavelength blue light improved the properties of FBFF.[32]

Clearly, study methodology has an impact on the DOC of RBCs. A previous study in 1993 by Harrington and Wilson[33] tested the DOC of RBCs using white polytetrafluoroethylene, black Nylotron, and stainless-steel mold materials of 4-mm diameter. A greater DOC was found with white molds.[33] Similarly semitransparent white Delrin molds may better mimic the optical properties of tooth than the completely opaque stainless-steel mold that is used in the 2009 ISO 4049 standard.[34] However, there are many different types and opacities of Delrin, and an opaque metal mold has some advantages. Rueggeberg and others,[34] in 2016, evaluated RBCs DOC using different mold materials and diameters. The RBCs were packed in one increment into split Delrin or metal stainless-steel molds that had either 4-, 6-, or 10-mm diameter holes. They reported that RBCs cured within white Delrin molds showed greater DOC than RBCs that were photocured in metal molds. Of note, increasing the mold diameter resulted in greater DOC values. They concluded that the mold material and diameter both have a significant impact on the DOC, but they used only one brand of bulk-fill RBC.[34]

The International Standards Organization (ISO) provides guidelines for laboratory studies to test the properties of dental polymer–based restorative materials.[35] According to ISO 4049:2009, the material should be cured within a stainless-steel mold that should be 4 mm in diameter and at least 2 mm longer than twice the claimed DOC.[20] Immediately after curing the RBC from the top, the soft uncured RBC at the bottom is scraped away using a plastic instrument. The maximum length of hard RBC is measured and divided by two to determine the DOC.[36] Contemporary bulk-filling RBCs are intended to be used in cavities that are much greater than this 4-mm diameter, and thus, the 4-mm-diameter mold specified in ISO 4049 may not be applicable when testing bulk-fill RBCs, but this requires confirmation.

This *in vitro* study investigated the effect of two mold materials (metal vs white semitransparent Delrin), the diameters of the tested specimens, and types of LCU on the DOC of three RBCs. The null hypotheses of this *in vitro* study were the following:

1) There will be no difference in the DOC of the three tested composite materials when made in the metal mold specified in the ISO 4049 test compared with a similar mold made of Delrin.
2) There will be no difference in the DOC of the three tested composite materials when made in the 4-mm mold specified in the ISO 4049 test compared with the larger 10-mm-diameter mold.
3) There will be no difference in the DOC of the three tested composite materials when either a high-power polywave LED or high-power monowave LED LCU is used.

Table 1: *Resin-Based Composite Resins and Light-Curing Units Used in the Study*

Material (Shade)	Type	Manufacturer	Lot No.
Filtek Supreme Ultra (2AB)	Conventional	3M ESPE, St Paul, MN, USA	N751605
Tetric EvoCeram Bulk Fill (IVA)	Bulk fill	Ivoclar Vivadent, Schaan, Liechtenstein	U55063
Aura Bulk Fill (BKF)	Bulk fill	SDI, Australia	151614
Bluephase G2	LED Polywave	Ivoclar Vivadent, Schaan, Liechtenstein	222788
Elipar DeepCure-S	LED Monowave	3M ESPE, St Paul, MN, USA	932125

4) There will be no difference in the transmission of the lower wavelengths (violet) of light through all three RBCs.

METHODS AND MATERIALS

Three RBCs were evaluated: two bulk-fill RBCs (Tetric Evoceram Bulk Fill shade IVA [Ivoclar Vivadent, Schaan, Liechtenstein] and Aura Bulk Fill universal shade [SDI, Bayswater, VIC, Australia]) and one conventional nano-composite RBC that is intended to be used in at most a 2 mm increment (Filtek Supreme Ultra shade A2B, 3M Oral Care, St Paul, MN, USA; Table 1). Metal (M) and semitransparent white Delrin (D) split molds of 15-mm depth and a 4- or 10-mm internal diameter opening were used to prepare the composite specimens (Figure 1).[35] The molds were placed on a polyester strip over a glass slide, and then uncured RBC was packed in one increment into the mold. A polyester strip was used to cover the uncured RBC in the mold. The RBCs were then light cured using either the polywave LED (Bluephase G2, Ivoclar Vivadent) or the monowave LED (Elipar DeepCure-S, 3M Oral Care) for 40 seconds, with the curing tip perpendicular to the mold surface and centered directly over the opening (Table 1). To standardize exposure times and to deliver sufficient radiant exposure, the same 40-second exposure time recommended by the manufacturer of Aura was used for both lights and all RBCs.

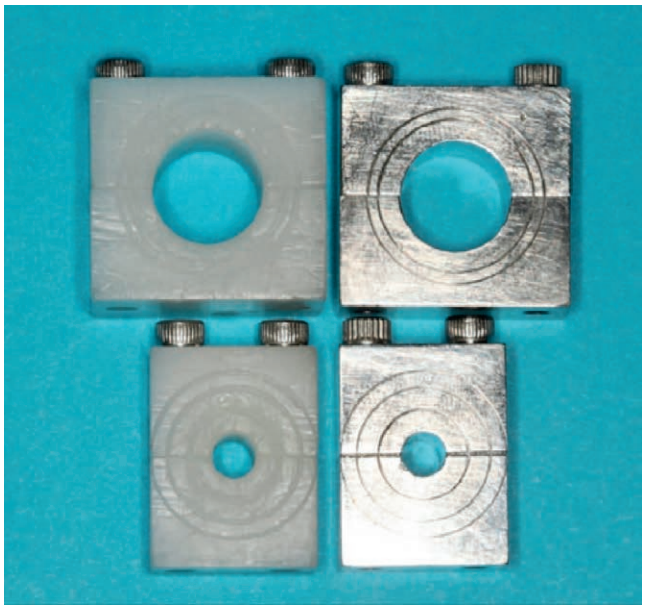

Figure 1. *Split molds (Delrin and metal) with a 4- and a 10-mm internal diameter.*

The irradiance, radiant exposure, and spectral emission from the two LCUs were measured using a 6-inch integrating sphere (Labsphere, North Sutton, NH, USA) connected to a fiber-optic spectrometer (USB 4000, Ocean Optics, Dunedin, FL, USA). This fiber-optic system was calibrated before the experiment using the internal reference lamp contained within the sphere. The output from each LCU was measured through both a 10-mm-diameter aperture and a 4-mm-diameter aperture placed at the entrance to the integrating sphere. The 10-mm-diameter aperture matched the diameter of the end of the light guides, and the 4-mm-diameter aperture matched the diameter of the 4-mm molds. Thus, in this case, the sphere measured the same spectral radiant power that would be received by the 4-mm specimens and not the total output emitted from the LCU. Spectrasuite v2.0.162 software (Ocean Optics) was used for data collection and analysis. These data were considered as the control values for the light reaching the RBCs.

Immediately after light exposure, the RBC specimens were removed. The uncured RBC was manually scraped away using a plastic spatula, the maximum length of the remaining hard, cured resin measured to the nearest 0.01 mm using a digital caliper (Mitutoyo, Canada Inc, Mississauga, ON, Canada) and the values divided by two according to the ISO 4049 test method.[20] A total of 240 specimens were prepared (two mold materials × two diameters × three RBCs × two LEDS × 10 repeats). A random sequence of RBC material, mold size and type, and LED unit was used to make 10 specimens for each condition.

Light Transmission Through the RBCs

The amount of light transmitted through a standard 6-mm-long specimen of each cured RBC was measured. This length was chosen because it was the length of hard specimens that could be consistently obtained. The spectral radiant power emitted from the bottom of the 6-mm thick specimens of RBC in the respective molds was measured using a 6-inch integrating sphere (Labsphere) connected to a fiber-optic spectrometer (USB 4000, Ocean Optics). This fiber-optic system was calibrated before the experiment using the internal reference lamp contained within the sphere. Spectrasuite v2.0.162 software (Ocean Optics) was used for data collection and analysis.

Statistical Analysis

Data were analyzed using SPSS Pc + version 21.0 (IBM Inc, Chicago, IL, USA) statistical software. Descriptive statistics (mean and standard deviation) were used to describe the quantitative outcome variable (RBC DOC). The data were tested graphically and by homogeneity of variances, to determine whether the data were normally distributed. Multivariate analysis of variance (ANOVA) was used to compare the mean values of DOC, in relation to three RBCs, two mold materials, two diameters, and two LED LCUs. Multivariate ANOVA followed by post hoc Tukey multiple comparison tests were used to assess the significance (α=0.05) of each study variable in relation to the DOC values.

RESULTS

Although not detectable by the human eye or by a dental radiometer, the Elipar DeepCure-S delivered different emission spectra from the Bluephase G2. The Elipar DeepCure-S delivered a higher power compared with the Bluephase G2 through the 4-mm-diameter aperture, 256 mW compared with 166 mW for the G2. When measuring power through a 10-mm-diameter aperture, both LEDs delivered similar power values, with the polywave LED curing light delivering 820 mW and the monowave LED curing light delivering 807 mW. There was a statistically significant difference for three variables tested (RBC, mold type, and LCU; Table 2).

Effect of RBC Type on the DOC

There was significant difference in the mean values of DOC in relation to the three RBCs. Tetric Evoceram Bulk-Fill composite showed the greatest mean DOC value compared with the other two RBCs, namely, Aura and Filtek Supreme Ultra (Table 2). The conventional material Filtek Supreme Ultra showed the least DOC ($p<0.0001$).

Effect of Mold Material and Diameter on DOC

There was a statistically significant difference in mean depth cure values between the two mold materials and the two mold diameters (Table 2). The white Delrin mold material always produced a significantly greater DOC compared with the metal mold ($p<0.0001$). Also, significantly higher DOC was always found in the molds that had a 10-mm internal diameter compared with the molds with the 4-mm internal diameter ($p<0.0001$).

Effect of LED Type on DOC

There was no statistically significant difference in the mean DOC values when either the Bluephase G2 or DeepCure-S (p=0.157) were used for 40 seconds (Table 2).

Light Transmission Through the RBCs

When using the Deep Cure-S, at the bottom of the 6-mm-long cured specimen of each brand of RBC, approximately 0.4% (Supreme Ultra), 1.9% (Aura), and 2.6% (Ivoclar Bulk Fill) of the original amount of light was transmitted when using the 10-mm-diameter metal mold, and approximately 0.2% (Supreme Ultra), 0.9% (Aura), and 1.5% (Ivoclar Bulk Fill) when using the 4-mm-diameter metal mold (Figure 2). As for the 10-mm-diameter Delrin mold, approximately 0.5% (Supreme Ultra), 2.2% (Aura), and 2.9% (Ivoclar Bulk Fill) of the original amount of light was transmitted, compared with approximately 3.3% (Supreme Ultra), 5.2% (Aura), and 6.2% (Ivoclar Bulk Fill) when using the 4-mm-diameter Delrin mold (Figure 2).

When using the Bluephase G2, at the bottom of this 6-mm-long cured specimen of each brand of RBC, approximately 0.4% (Supreme Ultra), 1.9% (Aura), and 3.1% (Ivoclar Bulk Fill) of the original amount of light was transmitted when using the 10-mm-diameter metal mold and approximately 0.2% (Supreme Ultra), 1.0% (Aura), and 1.5% (Ivoclar Bulk Fill) when using the 4-mm-diameter metal mold (Figure 3). As for the 10-mm-diameter Delrin mold, approximately 0.6% (Supreme Ultra), 2.4% (Aura), and 3.5% (Ivoclar Bulk Fill) of the original amount of light was transmitted, compared with approximately 4.6% (Supreme Ultra), 7.0% (Aura), and 8.3% (Ivoclar Bulk Fill) when using the 4-mm-diameter Delrin mold (Figure 3). For all three RBCs,

Table 2: *Depth of Cure (Means and SDs) of Different Resin-Based Composites When Cured With the Bluephase G2 or the DeepCure-S Curing Lights in the 4-mm and 10-mm Delrin and Metal molds* [a]

Resin-Based Composites	Mean Depth of Cure ± SD, mm				Multivariant ANOVA Test	
	Group			Mean ± SD		
	Curing Light	Mold Type	Diameter, mm			
Aura Bulk Fill	Bluephase G2	Delrin	4	5.04 ± 0.03$_{e}$	Resin-based composite	<0.0001
			10	6.16 ± 0.19**$_{j}$		
		Metal	4	3.98 ± 0.09*$_{c}$		
			10	5.03 ± 0.02$_{e}$		
	DeepCure	Delrin	4	5.06 ± 0.04$_{e}$		
			10	5.81 ± 0.09$_{h}$		
		Metal	4	4.1 ± 0.08$_{c}$	Curing light	0.157
			10	4.98 ± 0.03$_{e}$		
Filtek Supreme Ultra A2B	Bluephase G2	Delrin	4	5.02 ± 0.02$_{e}$		
			10	5.27 ± 0.09**$_{g}$		
		Metal	4	3.42 ± 0.07*$_{a}$		
			10	4.09 ± 0.04$_{c}$		
	DeepCure	Delrin	4	5.04 ± 0.02$_{e}$	Mold material type	<0.0001
			10	4.80 ± 0.08$_{f}$		
		Metal	4	3.61 ± 0.08$_{b}$		
			10	3.99 ± 0.06$_{c}$		
Tetric Bulk Fill IVA	Bluephase G2	Delrin	4	5.04 ± 0.02$_{e}$		
			10	6.73 ± 0.12**$_{i}$		
		Metal	4	4.36 ± 0.17*$_{d}$	Mold diameter	<0.0001
			10	5.09 ± 0.02$_{e}$		
	DeepCure	Delrin	4	5.08 ± 0.05$_{e}$		
			10	6.68 ± 0.19$_{i}$		
		Metal	4	4.78 ± 0.16$_{f}$		
			10	5.11 ± 0.03$_{e}$		

[a] *Maximum length divided by 2 as per ISO 1049. n=10 for each group; significant difference at p<0.05. According to post hoc Tukey multiple comparison test, means with the same subscript letter are not statistically different and means with different subscript letters are statistically different.*
** Lowest mean values for each RBC.*
*** Highest mean values for each RBC.*

the lower wavelengths of light (below 420 nm) were almost completely filtered out after passing through 6 mm of cured resin (Figure 4).

DISCUSSION

This *in vitro* study evaluated the effect of two different mold materials and mold diameters on the DOC of two bulk-fill RBCs (Tetric Bulk Fill and Aura Bulk Fill) and one conventional nanofilled-resin composite that is recommended to be used in at most a 2 mm increment (Filtek Supreme Ultra). The 4-mm-diameter metal molds were the molds specified in the ISO 4049 standard. There was a significant difference when using the semitransparent white Delrin compared with the metal molds. The Delrin material always resulted in a greater DOC for all tested RBCs ($p<0.0001$). Thus, the first null hypothesis was rejected. The second null hypothesis that different diameters of molds will have no significant effect on the DOC on the three RBC materials was also rejected, because the wider mold diameter (10-mm internal diameter) always produced a greater DOC when compared with the 4-mm internal diameter molds ($p<0.0001$). With regard to the third hypothesis, there were no differences (p=0.157) between curing either with the monowave or polywave LED when they were used for 40 seconds, and therefore, this hypothesis was accepted. The lower wavelengths of light (below 420 nm) were almost completely filtered out after passing through 6 mm of all three RBCs, and the fourth hypothesis was accepted (Figure 4).

With regard to the effect of different mold types on curing RBCs, our study found that the DOC results

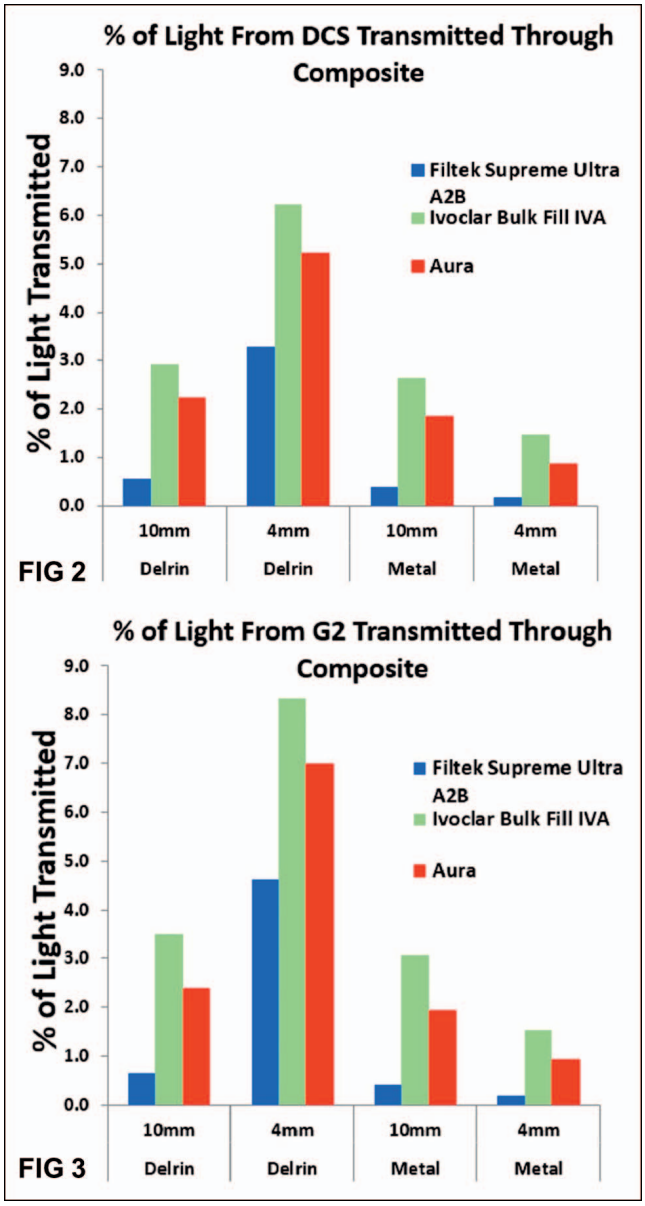

Figure 2. *DeepCure-S: Percentage of light delivered to the surface emitted from the bottom of 6-mm-thick cured specimens of each RBC in the 4- and 10-mm-diameter Delrin and metal molds.*

Figure 3. *Bluephase G2: Percentage of light delivered to the surface emitted from the bottom of 6-mm-thick cured specimens of each RBC in the 4- and 10-mm-diameter Delrin and metal molds.*

for both a conventional and a bulk-fill RBC made in the Delrin mold were always greater than those made in the metal mold. This was in agreement with other studies and suggests that the 4-mm metal mold specified in the 2009 ISO 4049 specifications will underestimate the DOC that will occur in a large tooth restoration.[34,37] This reduction in the DOC when smaller-diameter molds are used has been previously reported and is believed to occur because

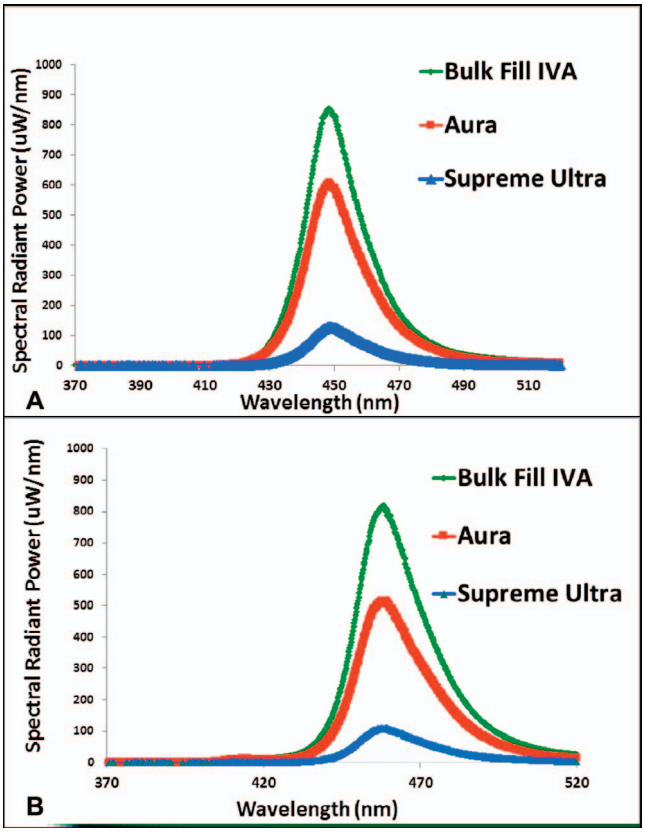

Figure 4. *Spectral radiant power (uW/nm) from the DeepCure-S (a) and the Bluephase G2 (b) emitted from the bottom of the 6-mm-thick specimens of RBC (note the absence of the lower wavelengths of light below 420 nm from the G2).*

the metal mold walls prevent any light transmission from the mold into the RBC, thus resulting in lower DOC.[34,37] In addition, the top surface area of the 4-mm-diameter mold was 12.56 mm^2, whereas the top area of the 10-mm-diameter cylinder was 78.50 mm^2. Thus, the 10-mm-diameter mold would allow 6.25 times as much light to enter into the mold, and this likely also improved the DOC of the RBCs. When using a bulk-fill RBC, Rueggeberg and others[34] also reported greater DOC for RBCs cured with Delrin molds when compared with metal stainless-steel molds.[34] In a different study, Erickson and Barkmeier[37] also reported lower DOC of a conventional RBC (Z100, 3M Oral Care) cured within opaque mold materials. A possible clarification of this finding is that the DOC is influenced by the light absorption and/or reflection properties of different mold materials. The white Delrin mold allows light to be transmitted down the sides of the mold in addition to light passing through the RBC, and thus, more light is received than from just the top alone. Since more light energy is delivered to the RBC specimens, and a greater DOC occurs.[34,37]

The Bluephase G2 delivered a slightly greater radiant power compared with the Deep Cure-S (820 mW compared with 807 mW), but the increased power and the different wavelengths of light from the polywave LED curing light did not produce a statistically significant difference ($p=0.157$) in the DOC of the three RBCs. A possible explanation is the large amount of energy delivered to the RBCs, because both LCUs were used for 40 seconds. The radiant power that arrived at the top of the 10-mm-diameter specimens was 820 mW for the G2 light and 807 mW for the Deep Cure-S light. In contrast, the radiant power that arrived at the top of the 4-mm-diameter specimens was less (166 mW) for the Bluephase G2 light compared with 256 mW for the DeepCure-S light. Although the Bluephase G2 was a more powerful light, the lower radiant power delivered to the top of the 4-mm-diameter specimens by the Bluephase G2 was likely due to the relative inhomogeneity in the light output from this LCU compared to the DeepCure-S. Whereas there is only one emitter in the DeepCure-S, the Bluephase G2 uses four LED emitters to deliver light in two different wavelength ranges. The location of these four emitters and the reflector produces an inhomogeneous light output, thus explaining why the 4-mm-diameter specimens received less light.[38] This difference in the radiant power delivered to the 4-mm-diameter specimens from the Bluephase G2 compared with the DeepCure-S probably accounts for the 20.55% increase in the DOC for the Bluephase G2 samples made in the 10-mm-diameter (regardless of mold and RBC types) when compared with the 4-mm samples. In contrast, the DeepCure-S 10-mm samples increased by only 13.36%.

Figures 2 to 4 support previous reports that the bulk-fill materials are more transparent compared with a conventional RBC.[39] Also, as expected, more light was transmitted when the white Delrin molds were used compared with the metal molds. Figures 2 to 4 also highlight the differences between the RBCs and show that very little violet light (<420 nm) was transmitted through 6 mm of all three RBCs. The increased absorbance of the violet light below 420 nm from the polywave LCU partly occurs because of the Rayleigh scattering of light, where the filler particles within the RBC tend to scatter more light at the shorter wavelengths.[40] Thus, the power and benefit of the violet light emitted from the polywave LCU will be lost as the thickness of the RBC increases.[31] Of note, the monowave LED curing light used in this study (DeepCure-S) emitted light from 430 to 480 nm, with a peak at 455 nm. Since approximately 50% of the light absorption for Ivocerin still occurs at 440 nm, this monowave LED delivers a functional emission spectrum that overlaps the Ivocerin absorbance range and allows this particular LCU to activate both the Ivocerin and CQ photoinitiators. Consequently, the need to use a polywave curing light to light cure this bulk-filling RBC should be questioned. However, it should be acknowledged that not all monowave LED LCUs will deliver a functional amount of light between 430 to 460 nm.[41]

This *in vitro* study found the greatest light transmission and DOC in Tetric Evoceram Bulk Fill followed by Aura Bulk Fill, and Filtek Supreme Ultra had the least. Of the three RBCs tested, both bulk-fill RBCs achieved a 4-mm DOC when tested in both the 4- and the 10-mm-diameter metal mold as per ISO 4049. In contrast, when the DOC was tested in the 4-mm-diameter metal mold, the conventional material, Filtek Supreme, did not achieve a 4-mm DOC, but it did in the 10-mm-diameter mold. This is to be expected because bulk-fill RBCs use more efficient photoinitiator systems, and by matching the refractive indices of their fillers and matrix, they increase the amount of transmitted light.

This *in vitro* study provides important information for clinicians when curing a deep restoration with a single increment of RBC. The conventional RBC, Filtek Supreme Ultra, was light cured for four times the minimum recommended exposure time (40 seconds instead of 10 seconds), and yet it still had the shallowest DOC. This shows that if the clinician wishes to place a 4-mm increment, the clinician must use a bulk-fill RBC, and they should not just try curing a conventional RBC for a longer time. Second, the DOC will be less in small cavities with narrow openings compared with larger, wider cavities (eg, a mesial occlusal distal restoration in a molar tooth).

The 10-mm internal diameter metal molds used in this study resemble the worst-case clinical condition when restoring a multisurface restoration with a metallic matrix band.[42] The mesial-distal dimensions of MOD cavity preparation in mandibular molar teeth are on average 11 mm in the mesiodistal dimension, 10.5 mm in the buccolingual dimension at the crown, and 9 mm at the cervix.[43] Although both bulk-fill RBCs achieved a 4-mm DOC when tested in the 4- and 10-mm-diameter metal mold as per ISO 4049, the clinician should recognize that the DOC will be less adjacent to the metal matrix band.

The results also show that the 4-mm-diameter metal mold specified in the 2009 ISO 4049 standard will underestimate the DOC, and this 4-mm-diameter mold may not be ideal when testing bulk-fill RBCs that are intended to fill larger cavities. Instead, a mold diameter that is similar to the light tip diameter may be preferable. Since the mold diameter and material significantly affect the DOC of both conventional and bulk-fill RBCs, the reader should pay careful attention to the mold diameter and opacity used in any study.

CONCLUSIONS

Within the limitations of this study, the following was concluded:

1) Of the three RBCs tested, only the bulk-fill products achieved a 4-mm DOC when tested in the 4-mm-diameter metal mold as per ISO 4049.
2) When used for 40 seconds, both the polywave LED curing light and the monowave LED curing light produced the same DOC for the same conditions, however the conventional composite still did not achieve a 4-mm DOC when tested in the 4-mm-diameter metal mold.
3) Increasing the mold diameter from 4 to 10 mm meant that the RBCs received a greater radiant power and resulted in greater DOC values.
4) Greater DOC is achieved in white semitransparent Delrin molds compared with metal molds.
5) All three RBCs achieved a 4-mm DOC when tested in the 10-mm-diameter metal mold.
6) The use of bulk-fill RBCs increased the DOC up to 28% compared with a conventional RBC.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of King Saud University.

Conflict of Interest

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

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Influence of Different CAM Strategies on the Fit of Partial Crown Restorations: A Digital Three-dimensional Evaluation

M Zimmermann • A Valcanaia • G Neiva • A Mehl • D Fasbinder

Clinical Relevance

Ideal fit of CAD/CAM fabricated indirect restorations is important for high clinical long-term success. Insufficient CAM milling strategies may lead to adaption discrepancies of the restoration resulting in poor occlusal fit and microleakage.

ABSTRACT

Objective: CAM fabrication is an important step within the CAD/CAM process. The internal fit of restorations is influenced by the accuracy of the subtractive CAM procedure. Little is known about how CAM strategies might influence the fit of CAD/CAM fabricated restorations. The aim of this study was to three-dimensionally evaluate the fit of CAD/CAM fabricated zirconia-reinforced lithium silicate ceramic partial crowns fabricated with three different CAM strategies. The null hypothesis was that different CAM strategies did not influence the fitting accuracy of CAD/CAM fabricated zirconia-reinforced lithium silicate ceramic partial crowns.

Methods and Materials: Preparation for a partial crown was performed on a maxillary right first molar on a typodont. A chairside CAD/CAM system with the intraoral scanning device CEREC Omnicam (Dentsply Sirona, York, PA, USA) and the 3+1 axis milling unit CEREC MCXL was used. There were three groups with different CAM strategies: step bur 12 (12), step bur 12S (12S), and two step-mode (12TWO). The zirconia-reinforced lithium silicate ceramic Celtra Duo (Dentsply Sirona) was used as the CAD/CAM material. A new

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3D method for evaluating the fit was applied, consisting of the quadrant scan with the intraoral scanning device CEREC Omnicam. The scan of the PVS material adherent to the preparation and the preparation scan were matched, and the difference analysis was performed with special software OraCheck (Cyfex AG, Zurich, Switzerland). Three areas were selected for analysis: margin (MA), axial (AX), and occlusal (OC). Statistical analysis was performed using 80% percentile, one-way ANOVA, and the *post hoc* Scheffé test with α=0.05.

Results: Statistically significant differences were found both within and between the test groups. The aspect axial fit results varied from 90.5 ± 20.1 μm for the two-step milling mode (12TWO_AX) to 122.8 ± 12.2 μm for the milling with step bur 12S (12S_AX). The worst result in all groups was found for the aspect occlusal fit with the highest value for group 12S of 222.8 ± 35.6 μm. Group two-step milling mode (12TWO) performed statistically significantly better from groups 12 and 12S for the occlusal fit ($p<0.05$). Deviation patterns were visually analyzed with a color-coded scheme for each restoration.

Conclusions: CAM strategy influenced the internal adaptation of zirconia-reinforced lithium silicate partial crowns fabricated with a chairside CAD/CAM system. Sensible selection of specific areas of internal adaptation and fit is an important factor for evaluating the CAM accuracy of CAD/CAM systems.

INTRODUCTION

Computer aided design/computer aided machining (CAD/CAM) technology has become a common fabrication technique for dental restorations.[1] The CAD/CAM workflow is composed of three essential steps.[2] The first step is to record the intraoral geometry of the dentition in a computer program in the form of a digital file. The second step involves a software program for computer modeling of the desired shape of the proposed restoration. The third step involves machining the designed restoration from a millable restorative CAD/CAM material. CAD/CAM technology is available for both laboratory and in-office applications.

The CAM process for current chairside CAD/CAM systems is subtractive milling. The CAM milling unit may utilize either carbide or diamond instruments for shaping the designed restoration from a prefabricated block of restorative CAD/CAM material. The main instruments used for milling acrylic and zirconia material are carbide, whereas diamond instruments are the chief type used for grinding resin-based and glass ceramic materials.

The accuracy of the CAM procedure is an obvious key factor in the final fit and adaptation of the restoration. There are several primary items that can influence the accuracy of the CAM milling process. Both milling instrument geometries, such as diameter, length, and type of instrument, and the CAD/CAM software parameter setting will influence the relief space created during the milling process between the imaged tooth preparation and the internal surface of the restoration.[3-6] The different machinability of CAD/CAM materials may be an additional factor influencing the internal adaption of CAD/CAM fabricated restorations. Brittle ceramic materials may behave differently from resin-based composite materials when milled with a CAD/CAM system.[7,8] In the literature, the overall selection of all individual CAM manufacturing parameters is often summarized with the term "CAM strategy."[2,6]

The most commonly used in-office CAD/CAM milling machines are 4-axis milling units. Today, the most popular chairside CAD/CAM system is the CEREC system. The MCXL milling unit (Dentsply Sirona, York, PA, USA) is a 3+1 axis milling machine that contains two or four motors with the option to use different instruments for milling. For fabricating glass-ceramic restorations, three different CAM strategies are currently available for the CEREC MCXL milling unit. When grinding glass-ceramics, the MCXL milling unit can be equipped with three different-sized diamond instruments (step bur 12S, step bur 12, and cylinder pointed bur 12S).

Although restorative material manufacturers work closely with software engineers to determine optimum milling paths for a specific material, milling paths and milling instruments are often preset in the CAD/CAM software. Especially for chairside CAD/CAM systems parameters, the different CAM milling strategies are often predefined and are not adjustable.

Zirconia-reinforced lithium silicate-ceramic (ZLS) is a new CAD/CAM glass-ceramic material that contains 10% by weight of 500-800 nm zirconia dispersed within a glass matrix.[9] It is available as a completely crystallized block. ZLS ceramics can be used for high-strength partial coverage crowns, and the postmilling processing that might affect restora-

tion fit is not required. ZLS CAD/CAM restorations do not need oven firing as do lithium disilicate ceramic CAD/CAM restorations such as e.max CAD (Ivoclar Vivadent, Schaan, Liechtenstein). ZLS ceramics can be hand polished prior to adhesive insertion and are thus suitable for chairside esthetic CAD/CAM restorations.

No controlled studies are available that investigate the possible change in dimensional fit and adaptation for ZLS restorations milled with different CAM strategies. The aim of this study was to three-dimensionally evaluate the marginal fit and internal adaptation of chairside CAD/CAM fabricated ZLS partial crowns using different CAM strategies. The null hypothesis was that different CAM strategies do not influence the marginal fit and internal adaption of chairside CAD/CAM fabricated ZLS partial crowns.

METHODS AND MATERIALS

This study represents an *in vitro* study. Preparation of a master partial crown was performed on a typodont on the maxillary right first molar. Tooth preparation was done according to recommended guidelines for all-ceramic partial crowns.[10] The preparation guidelines were 1.5 mm anatomical reduction of the palatal cusp with a butt-joint facial margin and an occlusal plateau with a mesiodistal standard inlay preparation. All internal angles were rounded and the deviation angle of the axial walls varied between 4° to 6°.

A chairside CAD/CAM system (CEREC, Dentsply Sirona) was used to fabricate the partial crowns. The powder-free intraoral scanning system CEREC Omnicam (Dentsply Sirona) was used to make a quadrant scan of the preparation. The manufacturer's recommendations of the scanning technique were respected.[11] The CAD design was performed with CAD software (CEREC SW v.4.0) using the biogeneric individual design mode. The parameter settings for the restoration were set to the manufacturer's recommendations with a spacer of 80 μm, margin thickness of 0 μm, minimum radial thickness of 400 μm, and minimum occlusal thickness of 1500 μm. Zirconia-reinforced lithium silicate-ceramic (ZLS) Celtra Duo (Dentsply Sirona) was selected as the CAD/CAM material.

A 3+1 axis milling unit CEREC MCXL (Dentsply Sirona) was used for CAM fabrication. There were three different groups corresponding to the three different CAM strategies available for grinding glass-ceramics with the MCX milling unit (group 12, group 12S, and group 12TWO). The MCXL milling unit was equipped with different milling instruments for each group: Groups 12 and 12TWO used two microfine diamonds, step bur 12, and cylinder pointed bur 12S; group 12S, two microfine diamonds, step bur 12S, and cylinder pointed bur 12S. The diameter sizes of the instruments were as follows: step bur 12, tip diameter 0.95 mm; step bur 12S, tip diameter 1.35 mm; and cylinder pointed bur 12S, tip diameter 1.75 mm. All diamond instruments had 65-micron grit grain size. Step bur instruments were used for subtractive milling of the inner surface of the restoration and cylinder pointed instruments for the outer surface. Geometry for the milling instruments used in this study is shown exemplarily in Figure 1.

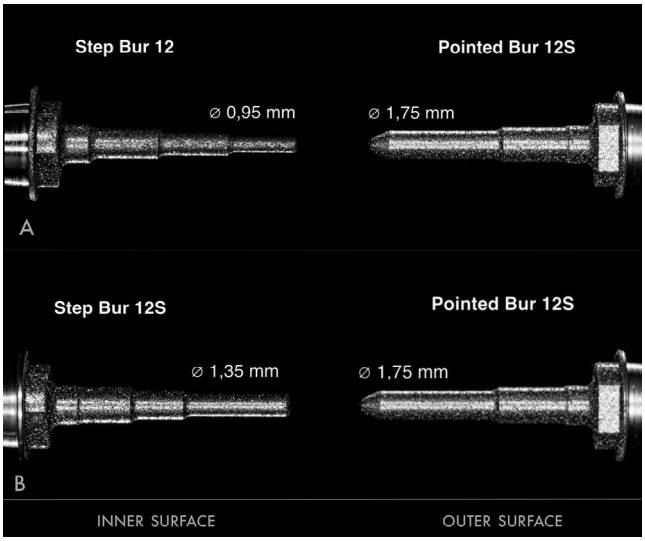

Figure 1. *Diamond instruments used for fabricating zirconia-reinforced partial crown CAD/CAM restorations with the CEREC MCXL milling unit. Different instrument setups (A, B) and milling paths were used for the respective groups; group 12: normal milling with instruments A; group 12S: normal milling with instruments B; and group 12TWO: two-step milling with instruments A.*

Milling modes were different for the respective groups. Group 12: normal milling mode (Figure 1: instruments A); group 12S: normal milling mode (Figure 1: instruments B); group 12TWO: two-step milling mode (Figure 1: instruments A). With the normal milling mode, the partial crown restoration was already ground in its final form. With the two-step milling mode, the restoration was first ground with the 200 μm restoration material left before the rest of the material was removed with the same instruments in a second circulation of the instruments. The milling time was almost double for group 12TWO than for groups 12 and 12S. In total, there were three different CAM strategies using different

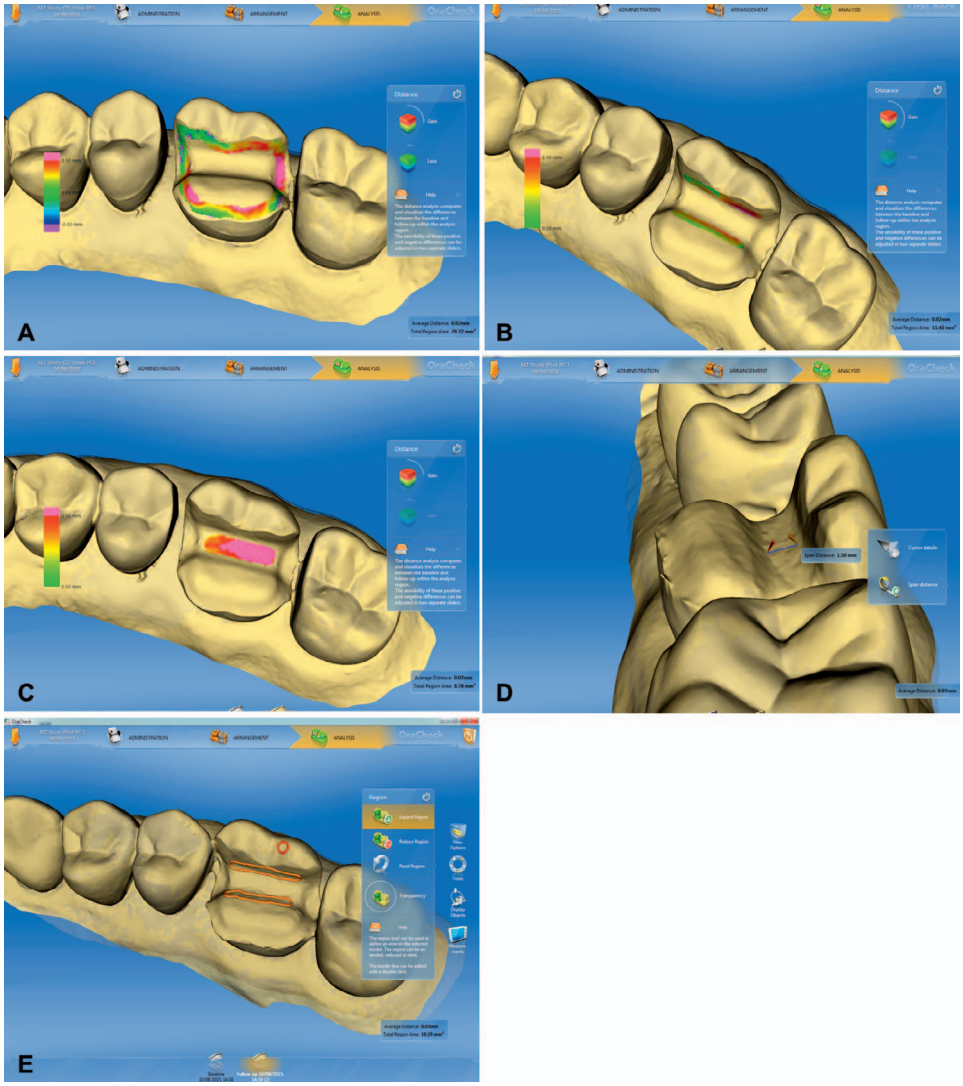

Figure 2. *Three-dimensional evaluation of the margin fit and internal adaptation of two-step milling mode fabricated partial crown. Three areas were selected: (A) margin; (B) axial; and (C) occlusal. Difference analysis with software OraCheck. Deviation pattern color coded with (+100 μm; red). Digital measuring tools such as "span distance" implemented within OraCheck software were used to ensure identical selection of the respective areas (D, E).*

milling instruments and milling paths that were investigated in this study.

For each test group, 10 partial crown restorations were fabricated (n = 10). Milling instruments and water were changed after each 10 restorations. No internal adjustments nor any postprocessing protocols were made on the restorations after grinding.

In this study, a special three-dimensional technique with a proprietary software program (OraCheck, Cyfex AG, Zurich, Switzerland) was done to evaluate the marginal fit and internal adaptation. First, the preparation was scanned using an intraoral scanner (CEREC Omnicam, Dentsply Sirona). Second, a polyvinylsiloxane impression recording the marginal fit and internal adaptation of each restoration was performed. The inner surface of each restoration was wiped with a lubricant (Vaseline) and a thin layer of light body polyvinylsiloxane impression material (Aquasil Ultra LV, Dentsply Sirona) was applied to the inner surface of the partial crown. The restoration was seated on the master preparation with moderate finger pressure for 15 seconds with approximately 25.0 N. Excess PVS material was carefully removed from the margins. After a setting time of 2 minutes, the partial crown was carefully removed from the preparation with the polyvinylsiloxane impression material left on its surface. Then, a second quadrant scan with the intraoral scanning device CEREC Omnicam (Dentsply Sirona) of the preparation was performed with the PVS material covering the preparation. The second scan was thus a replica of the adhesive cement space representing the marginal fit and internal adaptation.

Dimensional differences between the two recorded quadrant scans were analyzed with a proprietary

Table 1: *Results of Margin Fit and Internal Adaptation of ZLS Partial Crowns Fabricated With Three Different CAM Strategies (12, 12TWO, 12S)* [a]

Group	Area	n	Mean	SD	Min	Max	95% Confidence Interval	
							Lower	Upper
(12) step bur 12, normal milling	MA	10	120.4	11.9	103.8	141.6	111.9	128.9
	AX	10	96.9	12.0	80.2	117.6	88.4	105.5
	OC	10	215.8	14.4	201.8	250.0	205.5	226.0
(12TWO) step bur 12, two-step milling	MA	10	110.3	22.2	71.1	148.8	94.4	126.1
	AX	10	90.5	20.1	63.9	130.6	76.1	104.8
	OC	10	155.0	40.1	108.1	244.8	126.4	183.7
(12S) step bur 12S, normal milling	MA	10	144.6	14.4	121.4	164.2	134.3	154.9
	AX	10	122.8	12.2	111.1	142.9	114.1	131.5
	OC	10	222.8	35.6	176.7	297.0	197.3	248.3

[a] *Three areas were selected for 3D analysis: margin (MA), axial (AX), and occlusal (OC). Difference values were calculated as 80% percentile (μm).*

three-dimensional software program (OraCheck, Cyfex AG) for each test's partial crown. The principle of OraCheck software has recently been described in the literature.[12] First, the two scans were imported into the OraCheck software and superimposed using the software's best-fit algorithm. Second, subtractive analysis was performed by an automatic calculation of the distances between previously selected areas of interest. A point-to-surface distance approach was used in the study. An approximately 20,000 points-per-surface matching process was selected. There were three different regions of interest to evaluate the marginal fit and internal adaption. The margin area included the circumferential area of the preparation within 0.5 mm of the preparation margin line. The axial adaptation included a 0.5 mm diameter region for both the entire inner buccal and oral walls of the preparation. The occlusal surface (OC) adaptation included a 1.5 mm diameter occlusal plateau within the mesial and distal inlay slot preparation. The respective areas selected are shown in Figure 2 A-C. Selection of the respective areas, such as "span distance," was ensured with digital measuring instruments, implemented within the OraCheck software. The method of selecting the respective areas for the axial and occlusal aspect of the partial crowns is shown exemplarily in Figure 2 D-E.

The differences between the two superimposed digital files were measured by mathematically calculating the 80% percentile value. Values were exported as a CSV file and imported into statistical analysis software (SPSS v24.0, IBM Statistics, Armonk, NY, USA). The Kolmogorov-Smirnoff test was used for normal distribution of the data. The Levène test was used for homogeneity of variances. Descriptive statistics, including the mean, median, standard deviation, and 95% confidence interval, were calculated for each group. Statistical analysis was performed with one-way ANOVA and *post hoc* Scheffé test (α=0.05).

RESULTS

The results showed a normal distribution with equality of variances. Results for the fitting accuracy of a partial crown restoration with different CAM strategies are shown in Table 1. A box plot with median values is shown in Figure 3.

One-way ANOVA and *post hoc* Scheffé tests revealed statistically significant differences for the values both within and between the test groups ($p<0.05$). Homogenous subsets for one-way ANOVA with *post hoc* Scheffé test for all test groups are shown in Table 2.

For the aspect axial fit, results varied from 90.5 ± 20.1 μm (group 12TWO_AX) to 122.8 ± 12.2 μm (group 12S_AX). For the aspect margin fit, results varied from 110.3 ± 22.2 μm (group 12TWO_MA) to 144.6 ± 14.4 μm (group 12S_AX). The worst fit was found for the aspect occlusal fit in all groups, with the highest value for group 12S with 222.8 ± 35.6 μm (group 12S_OC). For the aspect occlusal fit, two-step milling with step bur 12 (group 12TWO_OC) performed statistically significantly better than did groups 12 (group 12_OC; $p<0.01$) and 12S (group 12S_OC; $p<0.01$). For both aspects axial fit and marginal fit, no statistically significant differences were found within all three test groups ($p>0.05$).

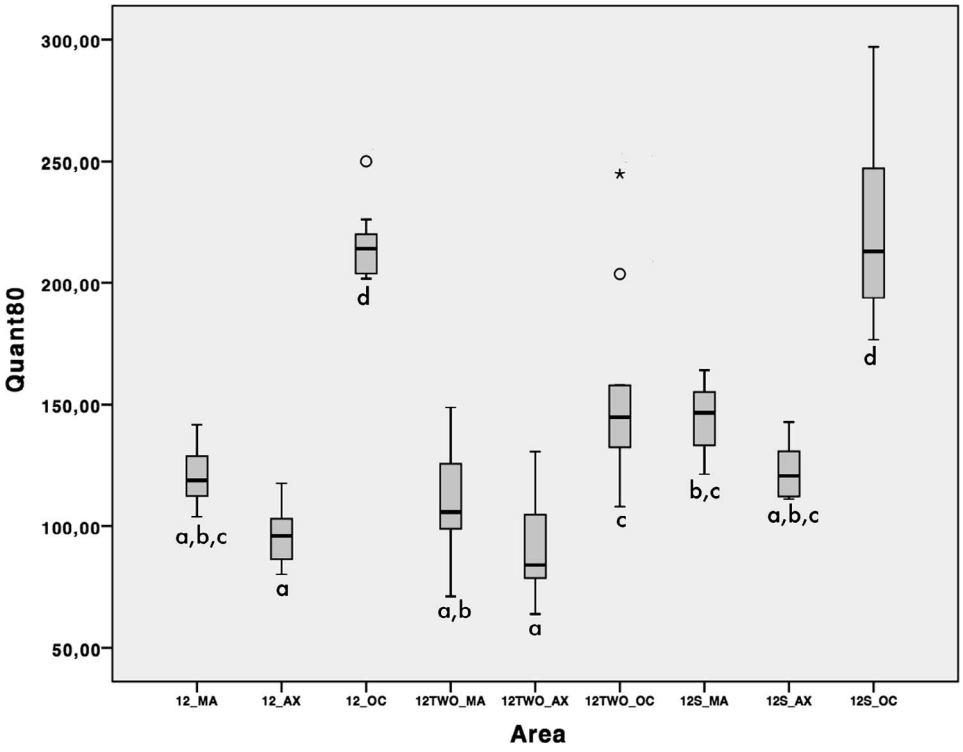

Figure 3. *Box plot for evaluating margin fit and internal adaptation of partial crowns fabricated with three different CAM strategies (step bur 12; 12), (two-step mode; 12TWO), and (step bur 12S; 12S). Three areas were selected for 3D analysis: margin (MA), axial (AX), and occlusal (OC). Mean precision for test groups is represented by the bar, circles represent outliers. Difference values were calculated as 80% percentile (μm). No statistically significant difference for box plots with same superscript letters.*

DISCUSSION

The aim of this study was to three-dimensionally evaluate the fit of CAD/CAM-fabricated ZLS partial crowns fabricated with three different CAM strategies. Based on the results found in this study, the null hypothesis is rejected. Statistically significant differences were found both within and between the test groups ($p<0.05$). For all aspects of internal fit investigated in this study, the two-step milling mode (group 12TWO) performed superiorly to normal milling with step bur 12 (group 12) and normal milling with step bur 12S (group 12S). Statistical significance could be found only for aspect OC between the three groups ($p<0.05$). In terms of area, the milling accuracy of occlusal areas performed worst for all milling strategies. In terms of milling strategies, the milling accuracy with instrument step burs 12S (group 12S) performed worst for all areas. There are several results that need to be discussed.

The CEREC MCXL used in this study is a 3+1 axis milling machine. The CAD/CAM block can be rotated and moved vertically while two instruments work simultaneously within the other three dimensions.

Table 2: *Homogenous Subsets for Test Groups. No Statistically Significant Difference for Values Within One Subset Group.* [a]

Material_Area	n	Subsets for Alpha = 0.05			
		1	2	3	4
12TWO_AX	10	90.5			
12_AX	10	96.9			
12TWO_MA	10	110.3	110.3		
12_MA	10	120.4	120.4	120.4	
12S_AX	10	122.8	122.8	122.8	
12S_MA	10		144.6	144.6	
12TWO_OC	10			155.0	
12_OC	10				215.8
12S_OC	10				222.8
Sig		.268	.195	.185	1.000

[a] *Statistical analysis with one-way ANOVA and* post hoc *Scheffé test. Significance level (α=0.05) (μm).*

The step bur instrument mounted on the left motor of the MCXL milling unit is used for subtractive milling of the inner surface, whereas the cylinder-pointed instrument mounted on the right motor is used for subtractive milling of the outer surface. Because of the limited degree of freedom of the CEREC MCXL milling unit, only specific sides of the instruments are used for the subtractive milling process. Steep areas of the inner surface such as axial walls and outer marginal contours are milled with the outer edge of the step bur instrument. Flat areas of the inner surface are milled with the tip of the instrument. Thus, because of dimensional discrepancies between instrument size and inner geometry of the restoration, milling inaccuracies might occur predominantly for occlusal surfaces of the CAD/CAM restoration. This affirmation is consistent with the results found in this study. All milling strategies showed the worst internal fit for the occlusal area, while the two-step milling mode (group 12TWO) performed statistically significantly better. Marginal areas and axial walls showed fewer milling discrepancies and a better internal fit. These findings are a direct result of the technical specifications of the CEREC MCXL milling unit.

The tip diameter of the step burs used in this study varied from 0.95 mm for the step bur 12 to 1.35 mm for step bur 12S. To ensure milling accuracy of flat surfaces such as the occlusal plateau of partial crowns, the tip diameter of the instrument is extremely important. When oversized instruments such as the step bur 12S are used, so-called over-milling occurs, resulting in a poor internal fit of the restoration. The results found in this study are in high agreement with this statement. The highest discrepancies for the occlusal area were found for group 12S. Group 12TWO_OC performed statistically significantly better than did groups 12_OC and 12S_OC. Results of this study are also in high agreement with published literature about the fit of different types of CEREC restorations where occlusal areas of crowns also showed the poorest fit of internal adaptation.[13,14]

Results of this study demonstrate that, even if identical milling instruments are used, the milling pathway might be highly decisive for the internal fit of restorations. For groups 12TWO and 12, the MCXL milling unit was equipped with the identical milling instrument step bur 12, but the machine used a different milling pathway for the instruments. The milling pathway is generally characterized by the x-, y-, and z-position of the instrument and its feeding rate as well as its revolutions per minute.[2] The higher the feeding rate, the higher the revolutions, the more abrasive the milling instrument, and the more material that is removed during subtractive CAD/CAM milling. Little can be found in the literature about the influence of different milling strategies on the internal fit of restorations. Most studies focus on the influence of the different parameter settings on the internal fit of restorations.[6,15]

CAD/CAM milling strategies must take into account the respective CAD/CAM material. In this study, ZLS Celtra Duo (Dentsply Sirona) was selected as the material. ZLS ceramic is a new CAD/CAM glass-ceramic material containing 10% by weight 500-800 nm zirconia dispersed within a glass matrix.[9] The material indication of ZLS ceramic includes full coverage restorations such as partial crowns. The crystallite size of ZLS is smaller than that of lithium disilicate ceramics such as e.max CAD (Ivoclar Vivadent), thus making ZLS ceramic highly favorable for subtractive CAD/CAM milling procedures.[9] Other studies have reported that various margin stabilities of CAD/CAM materials are a direct result of their material composition.[16] Compared with ZLS ceramics, particle-filled composite materials might possess a superior margin stability and, thus, better machinability, resulting in a superior internal fit of CAD/CAM restorations. It would also be interesting to investigate the influence of CAM strategies on different CAD/CAM materials.

Interestingly, no recommendation is given by the manufacturer of the 3+1 axis CAM machine that has been used in this study for the ideal CAM strategy for ZLS. However, our findings suggest that ideal CAM strategies are possible with respect to the respective CAM machining process and its parameters such as type of CAD/CAM material and restoration design. Ideally, by combining all individual parameters possible for the CAM machining process, the final CAM machining outcome and thus the marginal and internal fit of the restoration could be significantly improved.

Grinding glass ceramic materials is generally accomplished with diamond instruments. Little is reported in the literature about the influence of tool wear on the milling accuracy of CAD/CAM restorations.[17] In this study, diamond instruments were renewed after each 10 restorations to minimize the effect of tool wear. It might be interesting to further investigate the aspect of tool wear as a function of the respective CAM strategy used. The idea of the two-step milling mode is to reduce the pressure of the milling instruments on the restoration by

initially leaving about 200 μm rest material on the object to be milled. On the one hand, this approach might result in fewer material break-outs and thus a better internal fit of restorations, but on the other hand, this might also prolong the durability of the milling instruments. Further investigation seems to be necessary to elucidate this aspect in more detail.

The method of evaluating the fit of a restoration needs discussion. In the literature, mostly 2D methods in the form of a replica technique are used.[18] However, 2D methods seem to be less favorable than 3D as only point-to-point measurements can be carried out. The 2D method does not allow an entire circumferential analysis and can thus be seen only as an approximation because preselected points and distances instead of real geometries are used. In this study, approximately 20,000 points were used for the 3D analysis of each specimen. This is in strong contrast to the usual three to five linear measurements usually performed for 2D analysis. 3D methods may thus provide a better interpretation of restoration fit. Only a few 3D methods have been described in the literature, and all those are highly technique sensitive and not easily applicable.[19,20] The 3D method described in this study using intraoral scanning devices represents a far more clinically applicable approach. However, it does not represent a full digital approach and can be designated a 3D hybrid method as it describes the digitalization of an analog silicone layer and its further digital assessment.

The *in vivo* precision of the CEREC Omnicam intraoral scanner used in this study has been described in the recent literature for quadrant and full-arch scans with 37.4 ± 8.1 μm and 48.6 ± 11.6 μm, respectively (mean ± SD).[21,22] In this study, quadrant scans of a typodont were performed with local analysis of a single tooth area. Digital models are always a result of a matching process of single images with specific overlapping areas. Insufficient scientific data are available that analyze intraoral scanners' local accuracy for small regions such as a single tooth. It is important to mention that both complex geometric information, lack of surface texture details, *in vivo* conditions, and incorrect scanning strategy might significantly worsen the local scanning accuracy of intraoral scanners. Our own data (not yet published) reveal that the ideal *in vitro* accuracy of the CEREC Omnicam can be up to 10-15 μm for single teeth.

Many studies investigating the fit of restorations focus only on the overall internal fit.[5] This study represents a unique approach, investigating specific areas of internal fit three-dimensionally dependent on different CAM strategies using intraoral scanning. Many factors influencing the final fit of restorations have been described in the literature.[6,23,24] Several consequences have been reported for poor internal fit of restorations such as secondary caries, periodontal inflammation, retention loss, pulpal inflammation, and reduced fracture toughness.[25-27] This study thus demonstrates the clinical importance of the proper choice of CAM strategy as one important variable for the fitting accuracy of CAD/CAM fabricated restorations within the digital workflow. Because of the rapid development of CAD/CAM technology, improvements in the field of CAM fabrication are highly likely to occur in the future.

CONCLUSION

CAM strategy influenced the internal adaptation of ZLS partial crowns fabricated with a chairside CAD/CAM system. Sensible selection of specific areas of internal adaptation and fit is an important factor in evaluating CAM accuracy of CAD/CAM systems.

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 presented in this article.

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Effect of Different Computer-aided Design/Computer-aided Manufacturing (CAD/CAM) Materials and Thicknesses on the Fracture Resistance of Occlusal Veneers

JP Andrade • D Stona • HR Bittencourt • GA Borges • LH Burnett Júnior • AM Spohr

Clinical Relevance

Occlusal veneers composed of IPS e.max CAD, Vita Enamic, or Lava Ultimate with an 0.6-mm thickness are promising materials for the restoration of eroded posterior teeth.

SUMMARY

The aim was to evaluate, *in vitro*, the influence of different computer-aided design/computer-aided manufacturing (CAD/CAM) materials (IPS e.max CAD, Vita Enamic, and Lava Ultimate) and thicknesses (0.6 mm and 1.5 mm) on the fracture resistance of occlusal veneers. Sixty human third molars were prepared to simulate advanced erosion of the occlusal surface, and the teeth were randomly divided into six experimental groups (n=10) according to the material and thickness used to build the veneers. Ten sound teeth formed the control group. The veneers were adhesively luted and submitted to mechanical cyclic loading (1 million cycles at 200-N load). The fracture resistance test was performed in a universal testing machine. The failures were classified as "reparable" and "irreparable." According to two-way analysis of variance and the Tukey test, the interaction (material × thickness) was significant ($p=0.013$). The highest fracture resistance was obtained for IPS e.max CAD at a 1.5-mm thickness (4995 N) and was significantly higher compared to the other experimental groups ($p<0.05$). The lowest fracture resistance was obtained for Vita Enamic at 0.6 mm (2973 N), although this resistance

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was not significantly different from those for IPS e.max CAD at 0.6 mm (3067 N), Lava Ultimate at 0.6 mm (3384 N), Vita Enamic at 1.5 mm (3540 N), and Lava Ultimate at 1.5 mm (3584 N) ($p>0.05$). The experimental groups did not differ significantly from the sound teeth (3991 N) ($p>0.05$). The failures were predominantly repairable. The occlusal veneers of IPS e.max CAD, Vita Enamic, and Lava Ultimate, with thicknesses of 0.6 mm and 1.5 mm, obtained fracture resistances similar to those associated with sound teeth.

INTRODUCTION

The progressive reduction of dental enamel is a biological consequence of advancing age. However, the premature loss of this tissue may be due to the action of acidic foods and beverages, gastroesophageal reflux disease, bulimia nervosa, medications, and the reduction of salivary flow itself. These lesions are called dental erosion and have noncarious characteristics.[1,2]

The treatment of dental erosion should be approached by addressing the etiological factor of the disease, avoiding the progression of mineral loss. This mineral loss is slow, gradual, and often painless.[3] Dental erosion is usually not observed by patients and parents, and it is many times only diagnosed at an advanced stage when a substantial loss of dental tissue has occurred.[4] The recommended treatments to restore molars and premolars with severe erosion are indirect composite resin or ceramic restorations, such as inlays, onlays, or full crowns. However, some of these approaches can be very invasive.[5] Therefore, less invasive procedures should be the first treatment choice because of the advantages they offer, such as dental tissue preservation, maintenance of pulp vitality, and low rates of sensitivity.[6,7]

Computer-aided design/computer-aided manufacturing (CAD/CAM) technology became popular during the last decade for the preparation of restorations. This technology allows professionals to easily and quickly make restorations in a single session. Different materials are supplied in the form of blocks that are milled to obtain the restorations.[8]

Among CAD/CAM materials, reinforced ceramics, such as lithium disilicate (IPS e.max CAD), have recently expanded their indications to include thinner restorations. Ultrathin lithium disilicate occlusal veneers on posterior teeth have been shown[9] to be a conservative alternative to traditional inlays, onlays, and full crowns, with promising results.

Recently, a new material called a hybrid ceramic was launched on the market (Vita Enamic). This material consists of a hybrid structure of two interpenetrating networks of ceramic and polymer. Vita Enamic has been developed to allow faster milling of the ceramic block as well as ultrathin restorations (0.2-0.5 mm) with good mechanical behavior after luting.[10]

Composite resins represent another alternative for obtaining thin restorations in the occlusal region (Lava Ultimate), with superior fatigue resistance in comparison with the lithium disilicate–reinforced ceramic.[9] Lava Ultimate is a nano-filled resin composite, and this material has higher flexural strength and fracture toughness than do resin composites polymerized by a dental curing light.[11]

There is little information about the fracture resistance of Vita Enamic occlusal veneers compared with other restorative materials.[12] In view of the importance of dental tissue preservation, it is relevant to evaluate the fracture resistance of ultrathin occlusal veneers made with different restorative materials. Therefore, the aim of this study was to evaluate, *in vitro*, the influence of CAD/CAM restorative materials (IPS e.max CAD, Vita Enamic, and Lava Ultimate) and their thickness (0.6 mm and 1.5 mm) on the fracture resistance of teeth restored with occlusal veneers. The study was carried out under the following hypotheses: 1) the different thicknesses of the occlusal veneers do not influence the fracture resistance of the restored teeth; and 2) the different restorative materials do not influence the fracture resistance of the restored teeth.

METHODS AND MATERIALS

Tooth Selection

Human third molars, extracted for therapeutic reasons, were obtained after approval from the ethics committee (55675416.7.0000.5336). The teeth were examined under 10× magnification to verify the absence of cracks, caries, restorations, or fractures. The teeth were cleaned of gross debris, disinfected in 0.5% chloramine T for 24 hours, and then stored in distilled water at 4°C. The water was changed every week, and the teeth were used within six months. The buccal-palatal and mesiodistal dimensions of each tooth were measured with a digital caliper rule (500-197-20 Mitutoyo, Kawasaki, Japan). A variation of 0.5 mm was allowed for each measurement to standardize the dimensions of the teeth. In total, 70 teeth were selected, which were randomly distributed among seven different groups (n=10), as follows:

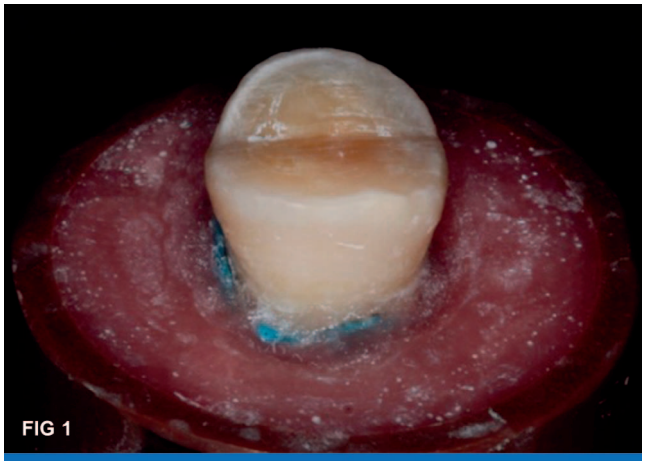

Figure 1. *Tooth preparation.*

Figure 2. *(a) Model of occlusal veneers with a thickness of 0.6 mm at the central groove and (b) model of occlusal veneers with a thickness of 1.5 mm at the central groove.*

1) sound teeth (control); 2) IPS e.max CAD 0.6 mm; 3) IPS e.max CAD 1.5 mm; 4) Vita Enamic 0.6 mm; 5) Vita Enamic 1.5 mm; 6) Lava Ultimate 0.6 mm; and 7) Lava Ultimate 1.5 mm.

Periodontal Ligament Simulation

A layer of adhesive (Universal Tray Adhesive, Zhermack, Rovigo, Italy) was applied with a brush on the root portion of each tooth. After the adhesive was dried, a layer of regular-viscosity vinyl polysiloxane was applied (Express Standard, 3M ESPE, St Paul, MN, USA) to artificially represent the periodontal ligament.

Tooth Preparation

Each tooth was mounted in a plastic cylinder filled with self-cured acrylic resin up to 2 mm below the cemento-enamel junction (CEJ) and then stored in distilled water at 4°C. The tooth preparation was performed according to the methods in the study by Magne and others.[13] A standardized preparation was performed on all teeth to simulate advanced erosion of the occlusal surface. First, the occlusal dentin was exposed by the selective removal of the occlusal enamel by applying a 4138 diamond bur (KG Sorensen, Cotia, SP, Brazil) at high speed with a water coolant. The buccal and lingual margins were maintained at approximately 5 mm from the CEJ and 2.3 to 2.6 mm above the central groove, maintaining the inclination of the cusps. The finished dental preparation was obtained with a 4138F diamond bur (KG Sorensen) (Figure 1). The diamond bur was replaced every five preparations.

Manufacturing the Occlusal Veneers

The occlusal veneers were made by CAD/CAM using Cerec software (version 4.0.2, Sirona Dental Systems GmbH, Bensheim, Germany). The tooth preparation was sprayed with reflective titanium (VITA Zahnfabrik, Bad Säckingen, Germany) to create the opaque surface needed for scanning with an optical three-dimensional intraoral camera, creating a three-dimensional virtual impression. The shape of the occlusal veneers was designed with an individual biogeneric copy from a right second lower molar. The thickness of the occlusal veneers was defined in the software according to each experimental group (Figure 2). The virtual die spacer used was 50 μm without removal of retention.

Sixty occlusal veneers were fabricated in the milling unit: 20 in lithium disilicate glass-ceramic (IPS e.max CAD), 20 in hybrid ceramic (Vita Enamic) and 20 in nanoceramic resin (Lava Ultimate) (Table 1). Ten teeth remained sound (control). Of the 20 occlusal veneers with each material, 10 had a thickness of 0.6 mm and 10 had a thickness of 1.5 mm, the latter being the usual thickness recommended by the manufacturers of these different trademark products.

The occlusal veneers milled in IPS e.max CAD were crystallized in a ceramic furnace (Programat P300, Ivoclar Vivadent, Schaan, Liechtenstein) for 30 minutes at a final temperature of 850°C under vacuum. After removal of the sprue and polishing with rubber tips (Diagloss, Edenta, Au, Switzerland), the veneers were glazed at 770°C. Vita Enamic veneers were mechanically polished with the Vita Enamic polishing set (VITA). Lava Ultimate veneers were mechanically polished with silicone tip Optimize (TDV, Pomerode, SC, Brazil) and diamond paste Enamelize (Cosmedent, Chicago, IL, USA) with a felt disk.

Luting Procedure

The luting procedures are described in Table 2. The resin cement was applied to the inner surface of the occlusal veneer. Immediately, the veneer was placed on the preparation and a load of 1 kg was applied by means of a metallic tool. The excess resin cement

Table 1: *Materials for Computer-aided Design/Computer-aided Manufacturing (CAD/CAM), Composition, and Manufacturers*[a]

Material	Composition	Manufacturer
IPS e.max CAD Lithium disilicate ceramic	SiO_2, Li_2O, K_2O, MgO, Al_2O_3, P_2O_5, and other oxides	Ivoclar Vivadent, Schaan, Liechtenstein
Vita Enamic Hybrid ceramic	Ceramic: silicon dioxide 58%-63%, aluminum oxide 20%-23%, sodium oxide 9%-11%, potassium oxide 4%-6%, boron trioxide 0.5%-2%, zirconia and calcium oxide; Polymer part (25%): UDMA and TEGDMA	VITA Zahnfabrik, Bad Säckingen, Germany
Lava Ultimate nanoceramic resin	Silica nanomers (20 nm), zirconia nanomers (4-11 nm), nanocluster particles derived from the nanomers (0.6-10 nm), silane coupling agent, resin matrix (Bis-GMA, Bis-EMA, UDMA, and TEGDMA)	3M ESPE, St Paul, MN, USA

Abbreviations: Bis-EMA, ethoxylated bisphenol A dimethacrylate; Bis-GMA, bisphenol A diglycidyl dimethacrylate; HEMA, hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate
[a] *The chemical composition information was obtained from the manufacturer's material safety data sheets.*

was removed, followed by light-curing with an LED curing unit (Bluephase N, Ivoclar Vivadent) for 20 seconds in each face with a light intensity of 1000 mW/cm^2. The light intensity of the curing unit was monitored every five specimens with the aid of an LED radiometer (SDI, Bayswater, Victoria, Australia). The specimens were stored in distilled water at 37°C for 24 hours. After this period, the restorations received cyclic mechanical loading.

Cyclic Mechanical Loading

The specimens were submitted to cyclic loading using the ER-11000 (Erios, São Paulo, SP, Brazil) cycling machine. The load profile was shaped as a sine wave and was always in contact with the occlusal surface of the veneers at 200 N using 1,000,000 cycles at 1 Hz in distilled water at 37°C. At the end of the cyclic loading, the presence or absence of luting failures, fractures, chips, or cracks on the veneers was observed with the aid of a 10× loupe (Olympus, Tokyo, Japan). The following classifications were used: a) success (unchanged); b) failure (fractures, chips, or cracks); or c) survival (a failure that did not interfere with the esthetics or use of the restoration).[14]

Fracture Resistance Testing

The fracture resistance testing was performed in a universal testing machine DL-2000 (EMIC, São José

Table 2: *Application Steps of the Luting Procedures*

Treatment/Material	IPS e.max CAD Vita Enamic	Lava Ultimate
Tooth surface treatment	• Apply the 37% phosphoric acid (N Etch; Ivoclar Vivadent, Schaan, Liechtenstein) on enamel for 30 s and on dentin for 15 s • Rinse the surface for 30 s and dry the surface with cotton buds • Apply the ExciTE F DSC adhesive system (Ivoclar Vivadent, Schaan, Liechtenstein) with a microbrush and dry gently for 5 s	• Apply the 35% phosphoric acid (3M, St Paul, MN, USA) only on enamel for 30 s • Rinse the surface for 30 s and dry the surface with cotton buds • Apply the Single Bond Universal adhesive system (3M, St Paul, MN, USA) with a microbrush for 20 s and dry gently for 5 s.
Treatment of the inner surface of the veneers	• Apply the 5% hydrofluoric acid (Condac Porcelana, FGM, Joinville, SC, Brazil) for 20 s on IPS e.max CAD and for 60 s on Vita Enamic • Rinse the surface for 30 s and air-dry • Apply a layer of universal primer (Monobond-N, Ivoclar Vivadent, Schaan, Liechtenstein) and dry gently for 5 s	• Sandblasting with 50 μm aluminum oxide for 5 s. • Rinse the surface for 30 s and air-dry • Apply a layer of the Single Bond Universal adhesive system and dry gently for 5 s
Resin cement	• Mix equal lengths of the base and catalyst pastes of Variolink N (Ivoclar Vivadent, Schaan, Liechtenstein) for 15 s and put on the inner surface of the occlusal veneer	• Mix equal lengths of the base and catalyst pastes of RelyX Ultimate resin cement (3M, St Paul, MN, USA) for 15 s and put on the inner surface of the occlusal veneer

Table 3: *Fracture Resistance Means (N) of the Experimental Groups*[a]

Groups	n	Fracture Resistance Means, N	Standard Deviations
Vita Enamic 0.6 mm	10	2973 A	635
IPS e.max CAD 0.6 mm	10	3067 A	933
Lava Ultimate 0.6 mm	9	3384 A	922
Vita Enamic 1.5 mm	10	3540 A	986
Lava Ultimate 1.5 mm	10	3584 A	954
IPS e.max CAD 1.5 mm	8	4995 B	855

[a] *Means followed by different letters represent significant differences according to Tukey test (α=0.05).*

dos Pinhais, PR, Brazil) using a cell load of 10 kN and a crosshead speed of 1 mm/min. A metal sphere with a diameter of 6 mm was attached to the load cell, which was connected to the mobile arm of the testing machine. The metal sphere was positioned to achieve tripodization of contacts along the cuspal inclines over the central fossa. The compression load was applied parallel to the long axis of the restored tooth until it fractured. The maximum force was recorded in Newtons (N).

Failures Analysis

After the fracture resistance testing, the specimens were visually assessed to determine the type of failure. The classification followed the criteria described by Beltrão and others,[15] as follows: 1) repairable and 2) irreparable. A fracture was considered irreparable when the fracture line divided the tooth into two parts at the floor level of the pulp chamber. The fractures were considered repairable when the fracture line involved only the restoration or all or part of the cusps.

Statistical Analysis

The fracture resistance values were submitted to the Kolmogorov-Smirnov normality test. As there was normality, the results were analyzed by two-way analysis of variance (ANOVA) (veneer thickness × material), followed by Tukey test. One-way ANOVA followed by Tukey test was applied to compare the fracture resistance of the sound teeth (control) with that of the experimental groups. The significance level was 5%. The software used was SPSS 10.0 (SPSS Inc, Chicago, IL, USA).

RESULTS

After cyclic loading, no cracks, chips, or fractures were observed in any sample.

According to two-way ANOVA, the material factor (p=0.031), the thickness factor (p=0.0004), and the interaction between the material and thickness (p=0.013) were significant.

A significantly higher fracture resistance was obtained for IPS e.max CAD 1.5 mm (4995 N) than for the other experimental groups ($p<0.027$). Lower fracture resistances, not significantly different from each other, were obtained for Vita Enamic 0.6 mm (2973 N), IPS e.max CAD 0.6 mm (3067 N), Lava Ultimate 0.6 mm (3384 N), Vita Enamic 1.5 mm (3540 N), and Lava Ultimate 1.5 mm (3584 N) ($p>0.550$) (Table 3).

There was a fracture of the acrylic resin base of three specimens, and the fracture resistance could not be obtained from these samples. These fractures occurred in one Lava Ultimate 0.6-mm specimen, two IPS e.max CAD 1.5-mm specimens, and one sound tooth.

According to one-way ANOVA, the fracture resistance of the sound teeth (3991 N) did not differ significantly from that of the experimental groups ($p>0.199$). Figure 3 compiles the fracture resistance means of the sound teeth (control) and experimental groups.

The failures were predominantly repairable for Lava Ultimate 0.6 mm, IPS e.max CAD 0.6 mm, IPS e.max CAD 1.5 mm, Vita Enamic 0.6 mm, and Vita Enamic 1.5 mm. The fractures were predominantly irreparable in sound teeth and Lava Ultimate 1.5 mm (Table 4).

Figure 4 shows examples of a reparable fracture (a) and an irreparable fracture (b).

DISCUSSION

There was no significant difference in the fracture resistances of 0.6-mm and 1.5-mm-thick veneers made of Lava Ultimate and Vita Enamic. For IPS e.max CAD, the fracture resistance was significantly higher at a thickness of 1.5 mm compared to a thickness of 0.6 mm. Therefore, the first hypothesis was rejected.

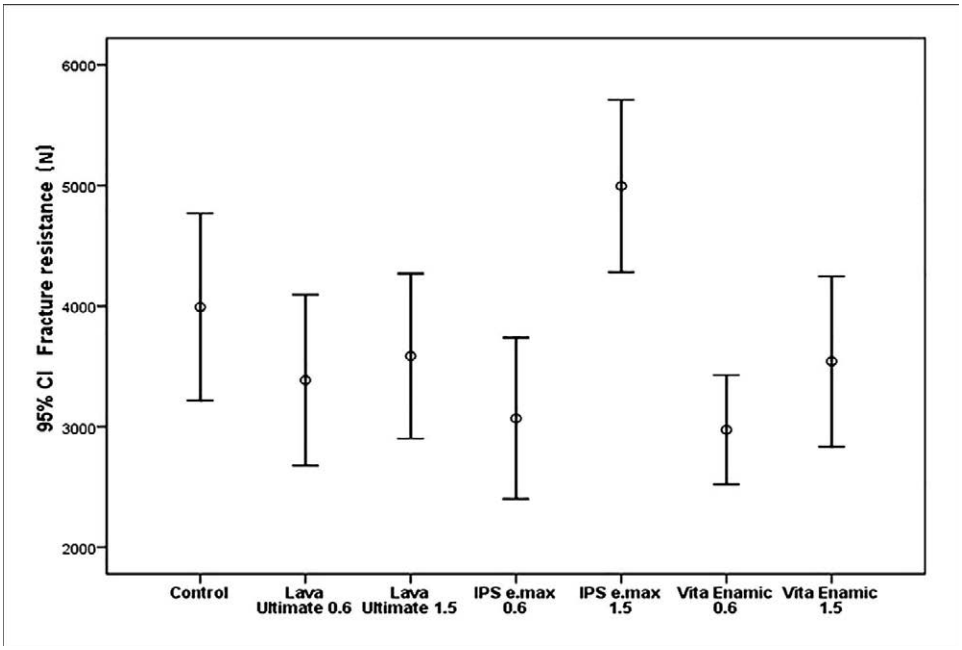

Figure 3. *Mean fracture resistances (N) of the sound teeth and experimental groups.*

Manufacturers of Lava Ultimate, IPS e.max CAD, and Vita Enamic indicate that restorations with a minimum thickness of 1.5 mm on the occlusal surface of posterior teeth will support masticatory loads. However, the results of the present study corroborate other *in vitro* studies[9,12,13,16,17] showing that it is possible to treat severe erosive lesions on posterior teeth with minimal wear using CAD/CAM ceramic and composite resin materials.

All of the restored teeth, regardless of the thickness of the occlusal veneers, obtained values above the maximum masticatory forces in humans. The maximum posterior masticatory force in an individual with no history of parafunction is approximately 424 N for women and 630 N for men.[18] In individuals with parafunction, the masticatory force in molars can vary from 780 N to 1120 N.[19] Additionally, there was no significant difference between the fracture resistance obtained for any of the experimental groups and the sound teeth (control). This finding is important because it shows that even when using ultrathin (0.6-mm) occlusal veneer restorations, the restored teeth showed similar fracture resistance to sound teeth. The possibility of making ultrathin occlusal veneers allows for a more conservative preparation with minimal wear to the tooth structure. It is believed that these positive results are due in part to the adhesive luting technique that allows intimate contact between the dental substrate, luting agent, and restorative material, so that the occlusal forces are applied and dissipated through the tooth, periodontal ligament, and alveolar bone.[20,21] Additionally, indirect restorations luted by the adhesive luting technique provided greater fracture resistance than do conventional luting techniques, such as zinc phosphate cement.[22] Therefore, the use of adhesive restorations has been recommended for reinforcing the remaining dental structure,[23,24] even if this recovery of resistance is only partial.[25,26]

In the present study, the luting protocol was not standardized for all groups, because the manufacturers of Lava Ultimate and IPS e.max CAD recommend the use of their own resin cement—RelyX Ultimate and Variolink N, respectively. The manufacturer of Vita Enamic recommends the use of VITA ADIVA full-adhesive (VITA Zahnfabrik), which is not sold in Brazil. The Brazilian representative of VITA, the Wilcos Company (Petrópolis, Rio de Janeiro, Brazil), recommends the use of resin cement in combination with an adhesive. For

Table 4: *Fracture Analyses of the Different Groups*

Group/Fracture Analysis	Repairable Fracture	Irreparable Fracture	Total
Sound teeth	3	6	9
IPS e.max CAD 0.6 mm	8	2	10
IPS e.max CAD 1.5 mm	6	2	8
Vita Enamic 0.6 mm	9	1	10
Vita Enamic 1.5 mm	6	4	10
Lava Ultimate 0.6 mm	6	3	9
Lava Ultimate 1.5 mm	4	6	10

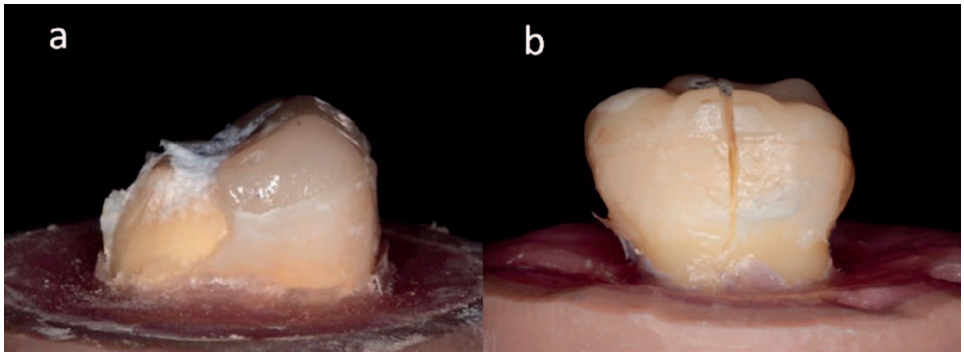

Figure 4. *(a) Reparable fracture—the fracture line involved the restoration and the cusp, and (b) irreparable fracture—the fracture line divided the tooth into two parts at the floor level of the pulp chamber.*

convenience, Variolink N was used for the luting of the occlusal veneers made with Vita Enamic.

For the adhesive luting of Lava Ultimate, the Single Bond Universal adhesive system was applied using the selective enamel etching technique. In this way, the self-etching mode on dentin was selected because better results are obtained from this mode than from the etch-and-rinse mode.[27] The ExciTE F DSC adhesive system was used for luting IPS e.max CAD and Vita Enamic, and the total-etch technique was applied because the manufacturer of this adhesive recommends this technique. To treat the inner surface of the restorations, the Lava Ultimate veneers were sandblasted with aluminum oxide particles, creating superficial irregularities that allow micromechanical retention with the applied adhesive.[28] The IPS e.max CAD and Vita Enamic veneers were etched with 5% hydrofluoric acid for 20 seconds and 60 seconds, respectively, as recommended by the manufacturers. The Monobond N primer, which contains silane, was applied to both materials. Associating hydrofluoric acid with silane is the most effective surface treatment with which to potentiate the bond between the lithium disilicate ceramic surface and the adhesive material.[29] The silane enhances the chemical bond between the silicon-containing materials and the resinous material used for luting.[30] Both IPS e.max CAD and Vita Enamic contain silicon in their composition. Silane was not applied to the Lava Ultimate veneers because it is not recommended by the manufacturer. However, such a procedure could be performed since the Lava Ultimate has nanoceramic silica particles. In addition, silane improves the wettability of the material surface, allowing greater contact between the adhesive and the restorative material.[31,32] The manufacturer probably does not recommend silane application because the Single Bond Universal adhesive system contains silane. The exact percentage of the silane present in this adhesive is not known, but studies[33,34] have shown that it is not enough to effectively optimize the ceramic-resinous bonding, as compared with a separate application of silane.

Of the three CAD/CAM restorative materials used to make the occlusal veneers, the highest fracture resistance was obtained with the IPS e.max CAD with a 1.5-mm thickness, which was significantly superior to the Lava Ultimate and Vita Enamic veneers with a 1.5-mm thickness. Therefore, the second hypothesis was rejected.

Lava Ultimate is a nanoceramic resin material with a high rate of polymerization that has a modulus of elasticity of 13 GPa and a flexural strength of 200 MPa (manufacturer's information; 3M ESPE).[35] Vita Enamic is referred to as a hybrid ceramic, having a modulus of elasticity of 30 GPa and a flexural strength of 150-160 MPa (manufacturer's information; VITA Zahnfabrik).[36] IPS e.max CAD is a lithium disilicate ceramic with a modulus of elasticity of 95 GPa and a flexural resistance of 360 MPa (manufacturer's information; Ivoclar Vivadent).[37] In another study[9] that used a fatigue resistance methodology different from the methodology used herein, ultrathin occlusal veneers with a 0.6-mm thickness made from composite resins (Paradigm MZ100 and XR) had a higher fatigue resistance when compared to ceramic materials (IPS Empress CAD and IPS e.max CAD). According to the authors,[9] the similarity of the modulus of elasticity between composite resin (16 GPa) and dentin (20.3 GPa)[38] may play a key role in the greater fatigue resistance of composite resins. However, with the fracture resistance methodology employed in the present study, it seems that the modulus of elasticity, as well as the flexural strength, had limited influence on the fracture resistance for Lava Ultimate and Vita Enamic with thicknesses of 0.6 and 1.5 mm and for the IPS e.max CAD with a 0.6-mm thickness. For these groups, despite differences in the modulus of elasticity and flexural strength between these materials, there was no significant

difference in the fracture resistance. In addition, the stronger but brittle IPS e.max CAD ceramic was more affected by reducing the thickness to 0.6 mm, and it seems that the higher flexural strength (360 MPa) of this material may have contributed to its reaching the highest fracture resistance with a 1.5-mm thickness.

In the present study, most of the fractures were repairable, except for Lava Ultimate with a thickness of 1.5 mm. As a result of its viscoelastic properties, Lava Ultimate has the capacity to accumulate the applied force on its surface and dissipate it rapidly along the tooth structure when the limit of proportionality is exceeded. This may promote catastrophic or irreparable dental fractures[39] and may account for the occurrence of a greater number of irreparable fractures in the 1.5-mm-thick Lava Ultimate veneers. However, in studies[9,13] using the fatigue resistance methodology, catastrophic failures did not occur, and cracks were limited to the restorative material when the occlusal veneers with 0.6-mm and 1.2-mm thicknesses were made with composite resins (Paradigm MZ100 and XR) and ceramics (Empress CAD and IPS e.max CAD). This methodology of fatigue resistance provides more useful results for examining the resistance of a restoration, since most the failures that occur in the oral cavity are caused by fatigue and not by a constant axial load.[9,32] The present study used the mechanical test of fracture resistance in which a constant axial load is applied to the specimens until fracture. This is a limitation of the study, since failure by constant axial load does not represent clinical reality. Despite this, this test is an important parameter for comparing the fracture resistance between different materials and restorative techniques.

Cyclic mechanical loading is an *in vitro* aging methodology that aims to submit the specimens to a cyclic load to reproduce the masticatory loads that are applied to the restorations. In the present study, teeth restored with occlusal veneers were submitted to 1,000,000 cycles with a 200-N load. In this way, approximately four years of normal functionality was simulated, since every 250,000 cycles are equivalent to one year of average mastication.[40-42] For all of the experimental groups, mechanical cyclic loading did not cause luting failure, fractures, or cracks on the veneer surface.

The present study evaluated 0.6-mm-thick veneers, which are considered ultrathin restorations. However, a study by Egbert and others[12] tested the fracture resistance of occlusal veneers with a 0.3-mm thickness using Lava Ultimate, Paradigm MZ 100, and Vita Enamic and found promising and favorable fracture resistances. Therefore, it seems that the use of thicknesses smaller than 0.6 mm could be feasible for the restoration of eroded teeth.

The transfer of laboratory results to the clinic should be done with caution, since *in vitro* studies cannot reproduce the real conditions inside the oral cavity. However, the results presented here suggest that occlusal veneers of Lava Ultimate, IPS e.max CAD, and Vita Enamic with a 0.6-mm thickness are promising candidates for the restoration of eroded teeth.

CONCLUSIONS

- The occlusal veneers of IPS e.max CAD, Vita Enamic, and Lava Ultimate, with thicknesses of 0.6 mm and 1.5 mm, obtained fracture resistances similar to those of sound teeth.
- Ultrathin occlusal veneers (0.6 mm) appear to be a promising restorative procedure in eroded posterior teeth.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Ethics Committee from Pontifical Catholic University of Rio Grande do Sul. The approval code for this study is 55675416.7.0000.5336.

Conflict of Interest

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

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Does Shortened Application Time Affect Long-Term Bond Strength of Universal Adhesives to Dentin?

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Clinical Relevance

Although a shorter application time may be clinically appealing, higher bond strength was achieved when adhesives were applied according to the manufacturer's instructions. Therefore, clinicians should perform the bonding procedure strictly according to the manufacturer's directions.

SUMMARY

Objective: This study evaluated the effect of shortened application time on long-term bond strength with universal adhesives.

Methods and Materials: Three universal adhesives were used: Clearfil Universal Bond (CU, Kuraray Noritake Dental Inc, Tokyo, Japan), Scotchbond Universal Adhesive (SB, 3M ESPE, St Paul, MN, USA) or G-Premio Bond (GP, GC Corp, Tokyo, Japan). Sixty molars were cut to expose midcoronal dentin and prepared with a regular diamond bur. Each adhesive was applied either according to the manufacturer's instruction or with shortened time. Specimens were stored in distilled water at 37°C for 24 hours and then cut into resin-dentin sticks. Microtensile bond strength (μTBS) was tested after either 24 hours or 1 year of water storage. Data were analyzed by the three-way ANOVA and Duncan tests (α=0.05). Fracture modes were analyzed under a scanning electron microscope (SEM). One dentin stick per group was selected after fracture mode analysis and further observed using transmission electron microscopy (TEM). Six additional dentin discs were prepared and conditioned with each adhesive under the different application time to observe the adhesive-smear layer interaction by SEM.

Results: Shortened application time affected the μTBS ($p<0.001$) while storage time did not affect bond strength ($p=0.187$). A significant effect of shortened application time on μTBS was observed in the CU at 1 year and in the GP at both storage times.

Conclusions: One-year storage time had no effect on the μTBS of universal adhesives to

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bur-cut dentin. The performance of universal adhesives can be compromised when applied using a shortened application time.

INTRODUCTION

Current dental adhesives are being developed to be more user-friendly. Recently, a simplified application technique taking less time appears to be driving the attention for new product development. Universal adhesives are appealing to many clinicians because they can be applied by either self-etching or etch-and-rinse.[1,2] Previous studies have shown that bonding performance of universal adhesives was similar regardless of bonding technique both *in vitro*[2,3] and *in vivo*.[4,5] In the case of self-etching technique, they are similar to typical all-in-one self-etching adhesives. Recently, even the one-step self-etching adhesives appear to be too slow. G-Premio Bond, the newly developed product from GC Corp, claims that high bond strength can be achieved even when applied with reduced application time (optional manufacturer's recommendation from a Japanese brochure). However, a previous report has demonstrated some drawbacks of reduced application time on 24-hour resin-dentin bond strength.[6]

In general, the dentin surfaces in *in vitro* studies are prepared mainly by SiC paper and a diamond bur, although the diamond bur is more clinically relevant. In addition, due to different characteristics of the smear layer,[7,8] more impact on self-etching adhesives was observed when bonded to bur-cut dentin.[6,9] Furthermore, from a previous study, various-size pores were observed on the adhesive surface of the fractured dentin beams prepared by the diamond bur. Thus, a crucial effect on long-term adhesion was expected.[6] Regarding the longevity of restorations, long-term water storage has been used to evaluate the durability of the resin-dentin bond in the oral cavity.[10-12] This bond degradation was observed after long-term storage due to the combined effect of resin dissolution[10] and collagen degradation.[13] Therefore, the aims of this study were to evaluate the effects of application time and long-term storage time on resin-dentin microtensile bond strength (μTBS) of three universal adhesives to bur-cut dentin. The null hypotheses were that (1) the adhesive application time has no effect on the strength of the resin-dentin bonds of universal adhesives and (2) the resin-dentin bond strength of universal adhesives is not affected by storage time.

METHODS AND MATERIALS

Tooth Selection and Preparation

Sixty-six extracted noncarious human third molars were used. They were stored in an aqueous solution of 0.5% chloramine-T at 4°C and used within 6 months after extraction. The teeth were collected under a protocol reviewed and approved by the university ethics committee. The teeth were cut with a gypsum model trimmer under water cooling to expose the midcoronal dentin. A light microscope was used to confirm that no enamel remained on the dentin surface. A uniform dentin surface was prepared with the tapered regular grit diamond bur (diamond point FG, #103R, Shofu, Kyoto, Japan) in a high-speed handpiece with copious water spray and five light-pressure strokes per tooth.

Adhesive and Bonding Procedure

The teeth were randomly divided into three experimental groups according to the adhesives used: CU (Clearfil Universal Bond, Kuraray Noritake Dental Inc, Tokyo, Japan), SB (Scotchbond Universal Adhesive, 3M ESPE, St Paul, MN, USA), and GP (G-Premio Bond, GC Corp, Tokyo, Japan). Material compositions and details are provided in Table 1. The teeth assigned for each adhesive were further randomly divided into two subgroups (n=10). Each subgroup was bonded with the adhesive applied either according to the manufacturer's instructions or under a shortened application time (optional manufacturer's instructions for GP).[6] Two 2-mm layers of resin composite (Clearfil AP-X, Kuraray Noritake Dental Inc, Tokyo, Japan) were built up. Each layer was light-cured for 20 seconds using an Optilux 401 (Demetron/Kerr, Orange, CA, USA) at $\geq$ 550 mW/cm^2.

Microtensile Bond Strength Test and Fracture Mode Analysis

After storage of the bonded teeth in water at 37°C for 24 hours, each tooth was sectioned into beams (cross-sectional area approximately 1 mm^2) using an Isomet diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA). Then three beams per tooth were randomly selected from the central portion of the crown. The mean bond strength obtained from these 3 beams was used for statistical calculation, resulting in 5 values per group. In total, fifteen beams from five teeth were tested immediately after sectioning (24-hour test), while the remaining 15 beams selected in the same manner were kept in distilled water at 37°C for 1 year (1-year test).

Table 1: *Adhesive System (Batch Number), Composition, and Application Procedures*

Adhesive (Batch Number)	pH*	Composition	Manufacturers' Instructions	Shortened Application Time
Clearfil Universal Bond (000002)	2.3	10-MDP, Bis-GMA, HEMA, ethanol, hydrophilic aliphatic dimethacrylate, colloidal silica, dl-camphorquinone, silane coupling agent, water	1. Apply the adhesive to the dentin surface with applicator brush and rub it for 10 s. 2. Dry the dentin surface sufficiently by gently blowing air for more than 5 s until the adhesive does not move. 3. Light cure for 10 s.	1. Drop the adhesive directly from the bottle on the surface. 2. Dry immediately by gently blowing air for 5 s. 3. Light cure for 10 s.
G-Premio Bond** (1411061G, 1510131)	1.5	10-MDP, 4 methacryloxyethyltrimellitate anhydrate, dimethacrylate monomer, distilled water, acetone, photo initiators, fine silica powder	1. Apply using a microbrush. 2. Leave undisturbed for 10 s after application. 3. Dry thoroughly for 5 s with oil-free air under maximum air pressure. 4. Light cure for 10 s.	1. Drop the adhesive directly from the bottle on the surface. 2. Dry immediately for 5 s under maximum air pressure. 3. Light cure for 10 s.
Scotchbond Universal (572054, 609623)	2.7	10-MDP, HEMA, silane, dimethacrylate resins, Vitrebond copolymer, filler, ethanol, water, initiators	1. Apply adhesive on the surface and rub it for 20 s. 2. Gently air-dry the adhesive for approximately 5 s for the solvent to evaporate. 3. Light cure for 10 s.	1. Drop the adhesive directly from the bottle on the surface. 2. Dry immediately by gently blowing air for 5 s. 3. Light cure for 10 s.

Abbreviations: 10-MDP, 10-methacryloxydecyl dihydrogen phosphate; Bis-GMA, bisphenol A diglycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate.
** The pH for SB and CU was obtained from Ref 1. For GP, it was supplied by the manufacturer.*
*** The shortened application time is an optional application mode suggested by the manufacturer.*

The beams were fixed onto a Ciucchi's jig with cyanoacrylate glue (Model repair 2 Blue, Dentsply-Sankin, Otahara, Japan) and subjected to a tensile force at a crosshead speed of 1 mm/min in a desktop testing apparatus (EZ test, Shimadzu, Kyoto, Japan). μTBS was expressed in MPa, and data were analyzed by three-way ANOVA and Duncan tests (α=0.05). The fractured specimens were carefully removed from the jig and mounted on an aluminum stub, then coated with Pt-Pd for 150 seconds. The fracture modes were determined using SEM (S-4000, Hitachi, Tokyo, Japan) at an accelerating voltage of 10 kV with low magnification (80×). Fracture mode categories were classified and the specific features of fractured surfaces were further observed at high magnification (10,000×).[6]

TEM Observation of Resin-Dentin Interface

After fracture mode analysis, representative dentin beams from each subgroup having μTBS values close to the mean were further prepared for TEM observation.[14] Specimens were fixed overnight in 2.5% glutaraldehyde containing 0.1M sodium cacodylate buffer at pH 7.4, and then rinsed with the same buffer. Subsequently, the specimens were dehydrated in ascending grades of ethanol series and embedded in epoxy resin (Epon 812, Polysciences, Inc, Warrington, PA, USA). Sections of 75-nm to 90-nm thickness through the resin-dentin interface were obtained using a diamond knife (Diatome, Bienne, Switzerland) in an ultramicrotome (Ultracut, UCT, Leica, Vienna, Austria). The sections were observed with a transmission electron microscope (H-800, Hitachi, Tokyo, Japan) operating at 75 kV.

Observation of Dentin Surface Treated With Adhesives

To observe dentin surface characteristics after adhesives application, approximately 2-mm dentin discs of six additional teeth were prepared by using a diamond saw. The midcoronal dentin surfaces were prepared by a regular diamond bur as previously described. Each disc was assigned to one of six groups according to the adhesives (CU, SB, and GP) and application times (manufacturer's instructions vs shortened application time) involved. Immediately after applying with the specific adhesive and application time without light curing, the discs were immersed in 100% acetone for 1 minute to remove the applied adhesive,[15] dehydrated with ethanol, and dried with hexamethyldisilazane.[16] The surfaces were then examined under SEM (3,000×) to observe the morphological changes on dentin resulting from the various adhesive application protocols.

RESULTS

μTBS and Fracture Modes

There were no pretest failures in this study. There were significant effects of adhesive (F=47.740, $p<0.001$) and application time (F=31.199,

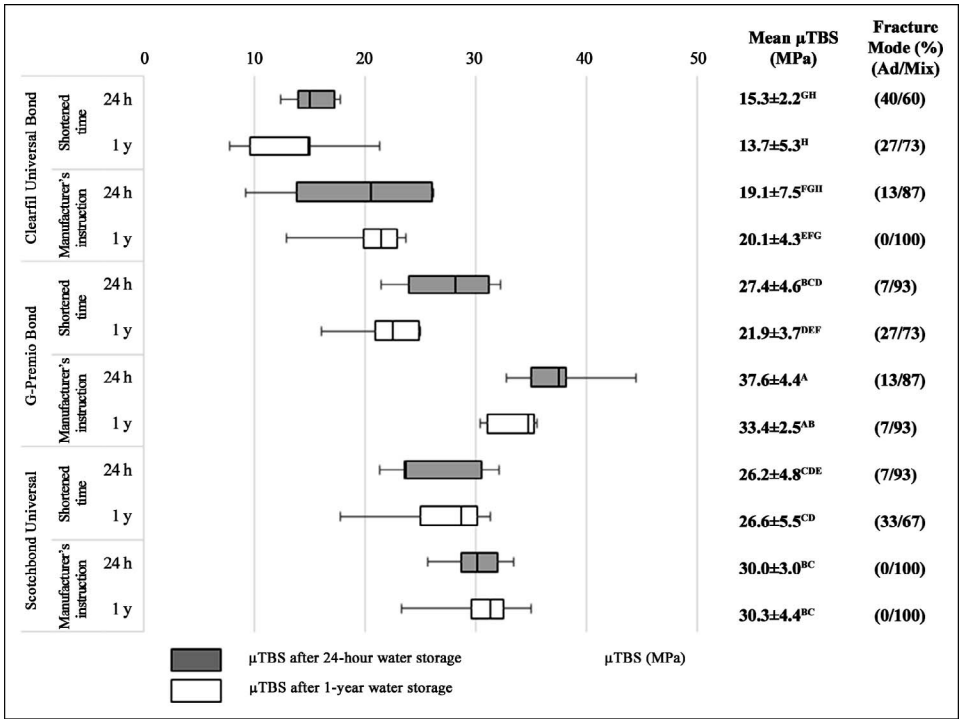

Figure 1. *Box-whisker plots of the μTBS to dentin and percentage of fracture mode. Different letters indicate statistically significant differences (p<0.05). S = shortened application time; MI = manufacturer's instructions; Ad = adhesive failure; Mix = mixed failure.*

$p<0.001$), but none of storage time ($F=1.789$, $p=0.187$). Only interaction between adhesive and application time was significant ($F=3.387$, $p=0.042$).

In general, higher μTBSs were observed when all the adhesives were applied according to the manufacturer's instructions for both testing periods of 24 hours and 1 year (Figure 1). At 24 hours, significantly higher bond strengths were observed for GP when applied with longer application time, and the significance was sustained after 1-year storage. Application time had no significant effect on either CU or SB at 24 hours. After 1-year storage, significant effects of application time were observed for both GP and CU, but not for SB. Storage time alone did not seem to significantly affect bond strength. When each combination of adhesive and application protocol was analyzed for the effect of storage time, no significant differences were observed. Similar bond strengths (range 23 to 36 MPa) were exhibited by GP and SB within each test time and application protocol. Conversely, CU always resulted in significantly lower bond strengths for each of the tested subgroups.

The most frequent fracture mode was classified as mixed failure wherein the failure was observed within the adhesive layer with some part of cohesive failure in dentin (Figure 1). Representative SEM images of the adhesive layer from the fractured surfaces at high magnification (10,000×) are shown in Figure 2. Various size porosities were detected in all tested groups. They were uniformly distributed on the entire surface of the adhesive layer except those in SB. A pore-free area was observed when applied with SB according to the manufacturer's instructions (Figure 2f and 2l). Greater size of porosities was detected in GP followed by CU and SB, respectively. They were predominately round, having a diameter of submicron size. Only a few of those in GP were above 1-2 μm. The porosities were smaller when applied to the adhesives having the longer application time. No difference was detected between the fractured beams at 24 hours and at 1-year storage.

TEM Observation of Resin-Dentin Interface

Hybrid layers ranging from 200 nm to 1000 nm thick were observed for all adhesives at the different testing conditions (Figure 3,4). One interesting finding was the striking difference observed in the adhesive layer when 24 hours was compared with 1-year images for all groups. The dense presence of fillers that can be observed in all images at 24 hours seemed to disappear in the specimens after 1 year of storage (comparing 24 hours with 1-year images for

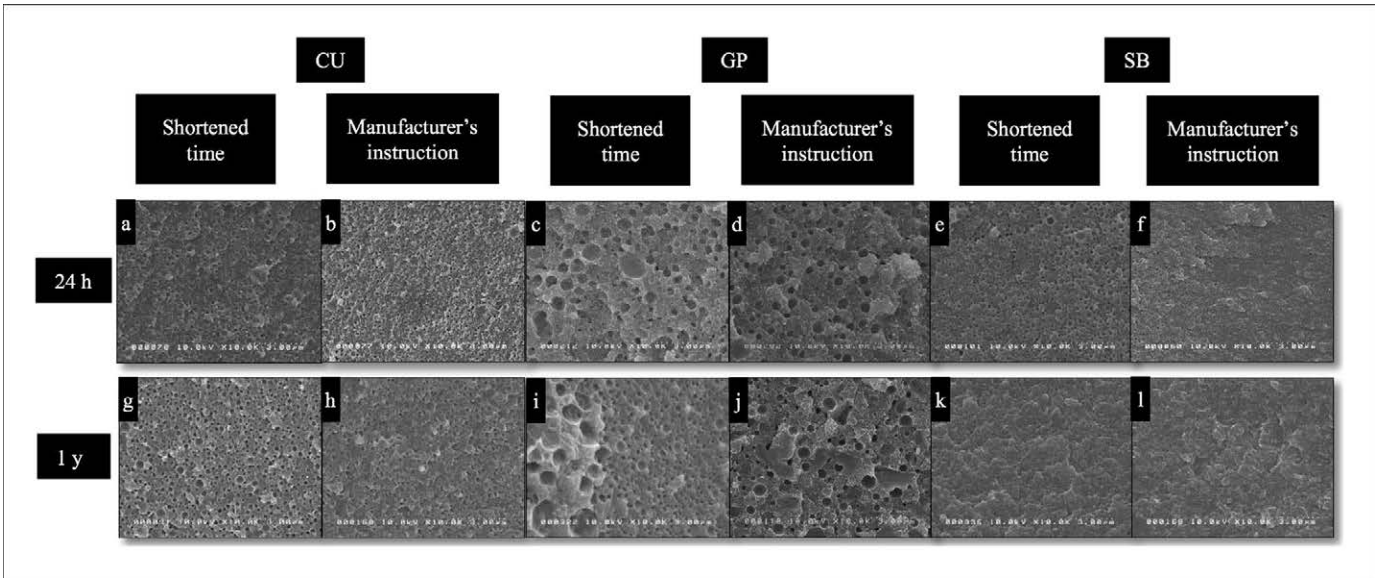

Figure 2. *Representative SEM images of adhesive layer from fractured beams at high magnification (10,000×).*

each group, Figures 3 and 4). With the exception of GP, in which the presence of some remaining fillers could be detected after 1 year, storage in water for 1 year rendered the adhesive layer devoid of fillers, and voids could be observed where the fillers were before. Some voids entrapped in the adhesive layer were detected in the GP (Figure 3e)

Observation of Dentin Surface Treated With Adhesives

Remnants of the smear layer were observed in all groups regardless of application time (Figure 5). An intact smear layer was observed when CU and SB were applied with a shortened application time (Figure 5a,c), whereas partial loss of the smear layer was detected when GP was used (Figure 5b). Conversely, application of the adhesives according to the manufacturer's directions clearly resulted in further dissolution of the smear layer and exposure of the underlying dentin, including some collagen fibrils (Figure 5d-f).

DISCUSSION

In this study, higher bond strength was demonstrated in all universal adhesives using the manufacturer's recommended time compared with those with shortened application time. However, statistically significant differences ($p<0.05$) were noticed for GP (at both storage times) and CU (at long-term storage). Therefore, the first null hypothesis that application mode would not affect bond strength of the adhesives was rejected. After 1 year storage, the μTBS of all adhesives tested were not statistically significantly different from those having 24-hour storage. Therefore, the study failed to reject the second null hypothesis that resin-dentin bond strength of universal adhesives would not be affected by storage time ($p=0.187$).

All adhesives showed higher μTBS when applied using the manufacturer's recommended time. This is in line with previous studies.[6,17] However, a significant effect of application time was observed in three out of six pairs (Figure 1). This might be explained by the different material compositions, adhesive-smear layer interaction, and method of application. According to the adhesives' composition, GP is acetone based whereas CU and SB are ethanol-water based (Table 1). Due to the high vapor pressure of acetone and the recommended burst of air, longer application time enhanced solvent evaporation in the GP, resulting in better integrity of the adhesive layer (compare Figure 2c,i vs 2d,j). This could be the reason why the significant effect of application time on μTBS was observed in the GP.

Regarding ethanol-based adhesives, when water is added as a solvent to ethanol-solvated monomer, water and ethanol can form a hydrogen bond both with each other and with the monomers.[18] In addition, the vapor pressure of ethanol is lower than that of acetone.[19] Therefore, it is likely that the shortened application time was insufficient for the solvent to evaporate from the ethanol-water–based adhesive. Nevertheless, the significant effect of reduced application time on the bond strength was

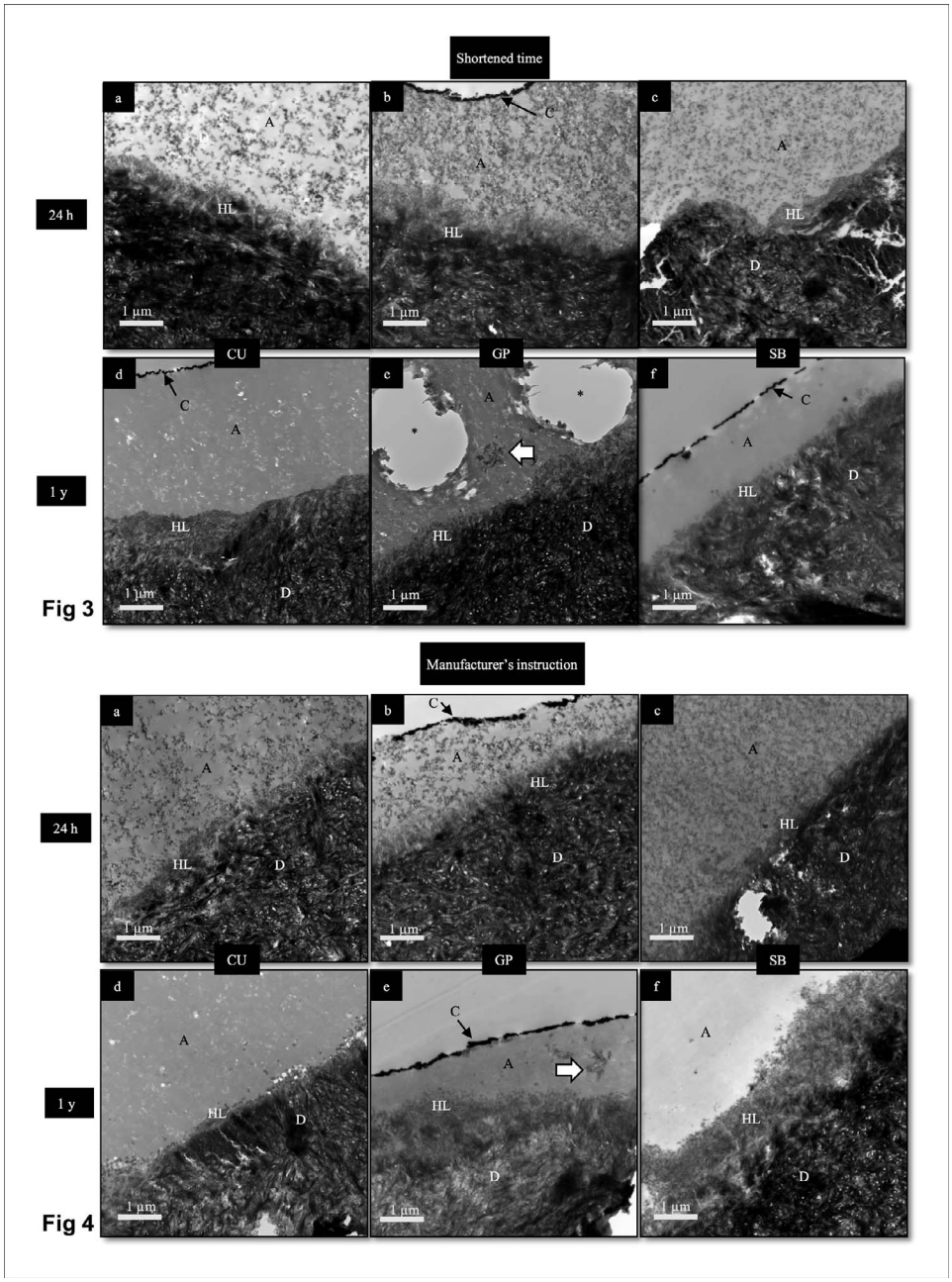

Figure 3. *Representative TEM images of dentin beams with shortened application time (10,000×). Pointer indicates remaining fillers in GP after box-whisker plots storage. Asterisks indicate voids entrapped in adhesive layer in GP. A = adhesive resin; HL = hybrid layer; D = dentin; C = coated Pt-Pd.*

Figure 4. *Representative TEM images of dentin beams with manufacturer's recommended time (10,000×). Pointer indicates remaining fillers in GP after box-whisker plots storage. A = adhesive resin; HL = hybrid layer; D = dentin; C = coated Pt-Pd.*

revealed after 1 year in CU. Probably, the polymerization was compromised by residual water[20] and solvent,[21,22] which then could have accelerated the degradation of the resin-dentin bond over time.

In the case of SB, the μTBS was not affected by shortened application time or long-term storage. Since nano-layering formation of 10-MDP-Ca salts at the adhesive interface has been demonstrated with commercially 10-MDP-containing adhesives,[23,24] this nano-layering is thought to enhance a water-stable interface and contribute to bond durability.[23-26] So far, SB is the only universal adhesive for which the presence of nano-layering has been reported.[23] Conversely, it has been reported that nano-layering can be inhibited by the presence of 2-HEMA.[27] Moreover, the presence of nano-layering has not being identified at the resin-dentin interface created by most of the latest generation of universal adhesives.[28] Therefore, the claim that nano-layering of 10-MDP-Ca salts contributes to bond durability of the resin-dentin interface has recently been questioned.[28,29] However, from a previous report, the nano-layering formation in SB has been observed in a location

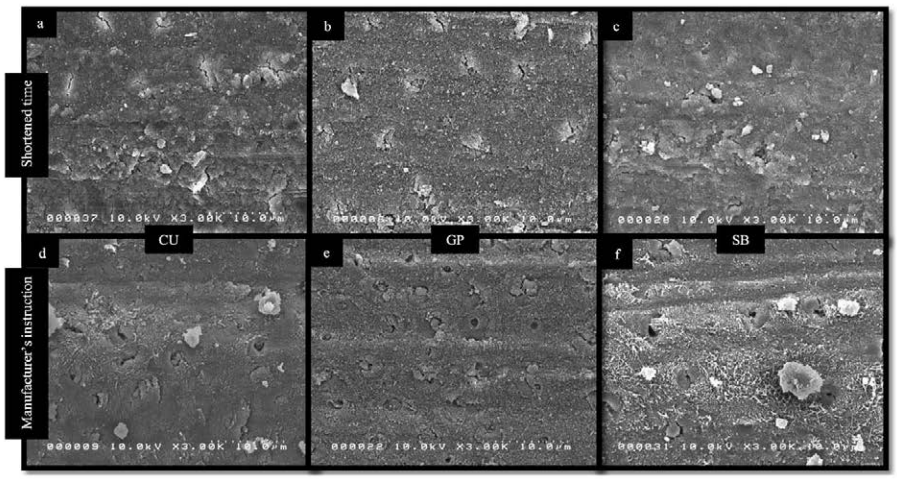

Figure 5. *Representative SEM images (3000×) of bur-cut dentin conditioned with each adhesive when applied with shortened time (a-c) and according to manufacturer's instructions (d-f).*

above the hybrid layer.[23] We speculate that this nano-layering might be responsible for improving the cohesive strength of the adhesive layer itself rather than prevent the degradation of the resin-dentin interface. In addition, chemical bonding between polyacrylic acid monomer (Vitrebond copolymer) and hydroxyapatite has also been reported.[30] Vitrebond copolymer is incorporated in SB (Table 1), and a recent study has demonstrated the chemical bond between Vitrebond copolymer containing adhesives and synthetic hydroxyapatite.[31] Therefore, it is possible that these two chemical bonding mechanisms might have contributed to the stable bond strength of SB in this study.

Another study demonstrated an increase in mineral dissolution from dentin when the application time of GP was increased to 10 seconds, compared with those of no waiting time.[17] This also supported the result of the current study since all adhesives demonstrated the ability to dissolve the smear layer more efficiently when applied with a longer time (Figure 5). However, the smear layer remained in all groups regardless of application time. This indicated that the smear layer was incorporated into the hybrid layer when this adhesive was applied in self-etching mode. Based on the pH (Table 1), GP was classified as an intermediately strong self-etching adhesive.[32] The etching ability of GP was more pronounced (Figure 5b) than the others, which are mild, self-etching adhesives (Figure 5a,c). Conversely, when adhesives were applied according to the manufacturer's instructions, the collagen fibrils were observed in CU (Figure 5d) and SB (Figure 5f) compared with GP (Figure 5e). This is possibly due to the different method of adhesive application. The GP was left undisturbed for 10 seconds according to the manufacturer's directions, whereas CU and SB were recommended to be rubbed for 10 and 20 seconds, respectively (Table 1). Previous studies demonstrated that rubbing application enhanced smear layer removal[33] and improve water evaporation, which led to less porosity in the adhesive layer[34-36] and improves resin monomer infiltration into dentin.[37] In the previous study,[6] resin tags had penetrated more deeply into the dentin when these adhesives were applied for the longer period, thus supporting the current findings. Regarding the smear layer, Pashley and Carvalho[38] suggested that the dentin smear layer impeded adhesion of the self-etching primer. Moreover, as the compromised bond strength from the bur-cut smear layer has already been reported,[6-8,15,39] the longer adhesive-smear–layer interaction time, combined with the rubbing application, could have induced the acidic primer to dissolve and penetrate through the smear layer to form a stronger bond to the underlying dentin.

In the present study, μTBS beams were cut and exposed directly to distilled water for 1 year to accelerate aging,[40] although this did not affect the dentin bond strength of the tested adhesives. Furthermore, microporosities of various sizes were observed on the fractured surfaces by SEM at high magnification (Figure 2). As previously discussed,[6] the porosities represent entrapped solvent and water that could not evaporate due to the limited amount of time allowed. Although porosities were observed for all adhesives in the SEM analysis, they were detected in GP only during TEM observation (Figure 3e). This might be due to the limited area of TEM observation and the larger-size porosities encountered with GP.

The presence of the porosities was confirmed from both SEM and TEM observations, and the porosity sizes matched (Figure 2c,d ,j and Figure 3e). In the current study, less porosity at the interface due to solvent evaporation was observed when the adhesives were applied for the longer time. For SB, however, the amount of residual porosity and related amount of residual solvent did not appear to have affected the resultant bond strength. A previous study[41] showed that bond strengths were also not different when SB was applied and experimentally had its solvent evaporated 50% versus 100%, suggesting that residual solvent had no effect on bond strength, such as were the findings of this study.

According to TEM observation, de-bonding of silica particles were detected in long-term storage groups (Figure 3d-f and Figure 4d-f). Some remaining filler can be observed in GP (Figure 3e and 4e). Filler de-bonding of hydrophilic adhesives was caused by hydrolysis of silane coupling agent.[14,42] GP is HEMA-free adhesive, thus expected to be less hydrophilic than the others and therefore, the filler detachment was less pronounced when compared to SB and CU (Figure 3d, f and Figure 4d, f). Surprisingly, according to μTBS test (Figure 1), filler de-bonding after long-term storage appears not to affect adhesive bond strength. Further study on the matter is warranted.

Our previous study[6] was the first to demonstrate the presence of such porosities within the adhesive layer of these universal adhesives. Because that study tested bond strength only after 24 hours, the findings prompt us to speculate that such a porous adhesive layer could favor water sorption and promote quick degradation of the adhesive, adversely affecting bond strength. Accordingly, it was expected in this follow-up study that the μTBS of the universal adhesives would be significantly decreased after long-term storage.[6] Nevertheless—and surprisingly—the 1-year storage did not influence the bond strength of the adhesives tested, regardless of the reduced application time of the experimental conditions. It is unclear whether the porosities in the adhesive layer actually increased water sorption, and if it did, why the expected softening and consequent weakening of the adhesive polymer did not become evident with resultant compromise of the bond strength. The higher percentage of mixed failures indicates that the bond to dentin was strong, even after the 1-year storage. Previous studies also evaluated the effect of aging on the bonding performance of universal adhesives and found that the bond strengths were stable after both 6-month storage[11] and 5000 cycles of thermocycling.[3] Based on the limitations of this study, one might not expect that 1-year storage would be sufficient to demonstrate the effect of bond degradation on universal adhesives. Further studies should be performed with a longer term to observe the effect of long-term storage.

CONCLUSIONS

Reduced application time caused significant reduction of bond strength of some adhesives and, therefore, is not warranted. Water storage for 1 year did not affect the bond strength of any of the adhesives to bur-cut dentin.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of Hokkaido University ethics committee. The approval code for this study is 2013-7.

Conflict of Interest

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

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Dynamic and Static Flexural Appraisal of Resin-based Composites: Comparison of the ISO and Mini-flexural Tests

AU Yap • AH Eweis • NA Yahya

Clinical Relevance: The mini-flexural test holds promise as a replacement for the ISO flexural test for the dynamic and static appraisal of dental resin-based composites.

doi: http://doi.org/10.2341/17-224-L

Original and Repair Bulk Fracture Resistance of Particle Filler and Short Fiber–Reinforced Composites

J Bijelic-Donova • S Uctasli • PK Vallittu • LVJ Lassila

Clinical Relevance: Longevity of repaired direct composite restorations may be improved by including a short E-glass fiber–reinforced composite with a semi-interpenetrating network matrix as the substrate material in bilayered restorations.

doi: https://doi.org/10.2341/17-207-L

Application of Calcium Silicate Materials After Acid Etching May Preserve Resin-Dentin Bonds

V Aggarwal • SS Bhasin

Clinical Relevance: Application of calcium silicate materials after acid etching can be a possible solution to preserve the resin-dentin adhesive interface.

doi: https://doi.org/10.2341/17-306-L

Chemical Interaction Characterization and Interface Analysis of Self-Etch Adhesives Containing 10-MDP and N-Methacryloyl Glycine Functional Monomers With the Dentin in Noncarious Cervical Lesions

BMB Oliveira • ALM Ubaldini • ML Baesso • LHC Andrade
SM Lima • M Giannini • L Hernandes • RC Pascotto

Clinical Relevance: The chemical interaction and morphology at the interface of self-etch adhesives and the dentin of noncarious cervical lesions depend on the functional monomer present in the adhesive. This fact is essential to evaluate the requirement for additional substrate preparation before commencing adhesive procedures.

doi: https://doi.org/10.2341/17-366-L

Linear Coefficient of Thermal Expansion Evaluation of Glass Ionomer and Resin-Modified Glass Ionomer Restorative Materials

G Pinto-Sinai • J Brewster • H Roberts

Clinical Relevance: Conventional glass ionomer materials overall exhibit linear coefficient of thermal expansion (LCTE) similar to tooth structure, while some resin-modified glass ionomer materials have LCTE similar to that reported for resin restorative materials.

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Dynamic and Static Flexural Appraisal of Resin-based Composites: Comparison of the ISO and Mini-flexural Tests

AU Yap • AH Eweis • NA Yahya

Clinical Relevance

The mini-flexural test holds promise as a replacement for the ISO flexural test for the dynamic and static appraisal of dental resin-based composites.

SUMMARY

The objective of this study was to determine the influence of specimen dimension and conditioning medium on the dynamic and static flexural properties of resin-based composites (RBCs). One conventional (Filtek Z350) and two bulk-fill RBCs (Filtek Bulk-fill and Beautifil-Bulk Restorative) were evaluated. Bar-shaped specimens with dimensions 25 × 2 × 2 mm (ISO flexural [IFT]) or 12 × 2 × 2 mm (mini-flexural [MFT]) were fabricated using customized stainless-steel molds, finished, measured, randomly divided into two groups, and conditioned in air or artificial saliva (SAGF) for seven days at 37°C. The specimens (n=10) were then subjected to dynamic and static three-point flexural testing. Data for storage modulus, loss modulus, loss tangent, flexural strength, and modulus were computed and subjected to *t*-test, analysis of variance/Tukey test, and Pearson correlation at a significance level of $\alpha = 0.05$. For both IFT and MFT, significant differences in dynamic and static flexural properties were more prevalent between materials after storage in saliva. For both conditioning mediums, the strongest correlation between IFT and MFT was observed for flexural strength. While significant positive correlations were observed for all flexural properties with saliva, no significant correlations were detected for loss tangent and flexural modulus with air. For both IFT and MFT, storage in saliva appeared to be more discriminative than storage in air. As moderate to strong positive relationships exist between IFT and MFT for dynamic and static flexural

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properties, the mini-flexural test holds promise as a replacement for the ISO 4049 in view of its clinical relevance and greater efficiency.

INTRODUCTION

The use of light-polymerized, resin-based composites (RBCs) in clinical dentistry has increased substantially over the past decade as a result of heightened patient esthetic demands, advancement in formulations, and simplification of clinical techniques. New RBCs are constantly being introduced to the dental market as manufacturers continue to improve upon their products.[1] RBCs are subjected to considerable flexural forces and need to withstand repetitive flexing and bending when used in anterior and posterior teeth.[2] The flexural properties of RBCs are usually assessed using destructive static tests that apply an escalating load until material failure.[3] As RBCs are visco-elastic in nature, static tests, which emphasize the elastic component, offer limited information on material structure.[4] Conversely, dynamic tests such as dynamic mechanical analysis (DMA), can define both elastic and viscous components of RBCs. These nondestructive tests have greater sensitivity to both macroscopic and molecular relaxation processes than do static techniques.[4,5] In addition, dynamic tests better simulate the cyclic masticatory and parafunctional loading to which RBCs are clinically subjected. The visco-elastic properties of RBCs have been studied[4,6,7] using DMA and were reported to be valuable in terms of predicting clinical performance.

DMA utilizes a range of frequencies and preset displacements that are within the elastic limits of the RBCs. Small oscillating "sinusoidal" stresses are applied isothermally or over a temperature range, and the resultant strains are measured as a function of time. Stress and strain of visco-elastic materials can be represented as follows:[8]

Stress:

$$\sigma = \sigma_0 \times \sin(\omega t + \delta);$$

Strain:

$$\varepsilon = \varepsilon \times \sin(\omega t)$$

where $\omega = 2\pi f$, where f is the frequency of strain oscillation; t is the time; and δ is the phase lag between stress and strain.

Parameters that can be derived from DMA include storage (elastic) modulus (E'), loss (viscous) modulus (E'',) and loss tangent (tan δ). Storage modulus embodies the stiffness of the material and indicates its ability to store elastic energy during a loading cycle. Loss modulus relates to the amount of energy loss by the material through viscous flow via conversion into heat.[9] Loss tangent is a dimensionless property that quantifies the material's ability to damp mechanical energy and is defined by the ratio of E'' to E'. When a sinusoidal stress is applied to a completely elastic or viscous material, deformation occurs precisely in phase or lags 90° behind the stress applied, respectively. For visco-elastic materials, the ensuing strain will lag behind the stress by an angle s, where s is $<90°$.

Bar-shaped specimens used in dynamic and static flexural tests[4,10-12] are often based on dimensions (25×2×2 mm) specified by the ISO 4049 standard.[13] Specimens measuring up to 50 mm long have also been employed in dynamic testing.[14] When flexural load is applied to the bar specimens, compressive, tensile, and shear stresses are evoked. Compressive stress occurs in the upper portion of the cross section, while tensile stress occurs in the lower. To simplify stress states, support spans (L) are planned long relative to specimen height (H) as shear stress and bending moments are independent and directly proportional to specimen length, correspondingly. In addition to changing support span at constant thickness, L/H ratios can also be modified by altering specimen height.[15] The large ISO bar-shaped specimens are, however, technically arduous to prepare without flaws. Several overlapping light irradiations are necessary as the exit windows of commercial curing lights are typically less than 25 mm. This leads to specimens that are not homogeneously cured with enduring stresses that may compromise flexural data reliability.[16,17] In addition to material and time wastage, these large specimens are also clinically irrelevant, as the mesio-distal diameter of molars is only about 11 mm and the cervico-incisal length of central incisors is around 13 mm.[18] Flexural tests involving smaller and more clinically relevant specimens are hence desirable.

Several studies have investigated the influence of specimen dimensions on the flexural strength of RBCs. While some reported similar flexural strength values, others observed higher strengths with shorter specimens.[16,17,19,20] Flexural strength correlation between the ISO and shorter mini-flexural specimens (12×2×2 mm) was found to be significant, positive, and very strong (r=0.95). Correlation for flexural modulus was also significant and positive, but strength of association was just moderate (r=0.53).[16] All preceding studies only focused on

Table 1: *Technical Profiles and Manufacturers of the Materials Evaluated*

Material (Abbreviation)	Manufacturer	Type	Resin	Filler	Filler Content, % by Weight/% by Volume	Lot No.
Filtek Z350 (FZ) A2 shade	3M ESPE, St Paul, MN, USA	Nanohybrid restorative	Bis-GMA Bis-EMA UDMA TEGDMA	Zirconia/silica cluster and silica nanoparticle	78.5/63.3	N771467
Filtek Bulk-Fill (FB) A2 shade	3M ESPE, St Paul, MN, USA	Bulk-fill restorative	AUDMA AFM DDDMA UDMA	Zirconia/silica cluster, ytterbium trifluoride	76.5/58.4	N789842
Beautifil-Bulk Restorative (BB) A2 shade	SHOFU Inc, Kyoto, Japan	Bulk-fill giomer restorative	Bis-GMA UDMA Bis-MPEPP TEGDMA	S-PRG based on F-Br-Al-Si glass	87/74.5	051623

Abbreviations: AFM, addition-fragmentation monomers; AUDMA, aromatic urethane dimethacrylate; Bis-EMA, ethoxylated bisphenol-A glycidyl methacrylate; Bis-GMA, bisphenol-A glycidyl methacrylate; Bis-MPEPP, bisphenol-A polyethoxy-dimethacrylate; DDDMA, 1,12-dodecanediol dimethacrylate; F-Br-Al-Si, fluoroboroaluminosilicate; S-PRG, surface-modified pre-reacted glass; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

static flexural testing, and none investigated the influence of specimen dimension on dynamic appraisal of RBCs. The objective of this study was to determine the influence of specimen dimension (specifically the ISO and mini-flexural test) on the dynamic and static flexural properties of RBCs. As the static flexural properties of RBCs had been shown to be dependent on storage medium,[20,21] the effect of conditioning in air and artificial saliva as confounding variables was also explored. Furthermore, correlations between the ISO and mini-flexural test results were performed for the various flexural properties. The null hypotheses were as follows: 1) there are no significant differences in dynamic and static flexural test values between the ISO and mini-flexural test; 2) there are no significant differences between conditioning in air and artificial saliva for the various flexural properties; and 3) there are no correlations between the ISO and mini-flexural tests for dynamic and static testing.

METHODS AND MATERIALS

Specimen Preparation and Conditioning

The materials evaluated and their technical profiles are shown in Table 1. They included a conventional composite (Filtek Z350 [FZ]) and two bulk-fill materials (Filtek Bulk-fill [FB] and Beautifil-Bulk Restorative [BB]). ISO flexural test (IFT) specimens of the various RBCs were fabricated according to ISO 4049 specifications (25×2×2 mm) using customized stainless-steel molds. The RBCs were placed in one increment and excess material was removed by compressing the molds between two polyester strips with glass slides. The top surface of the specimens was light polymerized with four overlapping 10 second irradiations using an LED curing light (Demi Plus, Kerr, CA, USA) with an output irradiance of 1330 mW/cm^2, a wavelength of 450-470 nm, and a light exit window of 8 mm. The glass slides and polyester strips were removed and the specimens were light-cured with another four overlapping 10 second irradiations. The specimens were detached from their molds, and any minor material excess was gently removed with fine polishing discs (Sof-Lex, 3M ESPE, St Paul, MN, USA). The final dimension of the specimens and their parallelism were verified with a digital caliper (Mitutoyo Corporation, Kawasaki, Japan). Procedures for fabricating the mini-flexural test (MFT) specimens (12×2×2 mm) were similar, with the exception that light polymerization was accomplished with two overlapping irradiations arising from their smaller specimen size, as compared to MFT specimens. Twenty IFT and MFT specimens were produced, randomly divided into two groups of 10 (n=10), and conditioned in air or artificial saliva (SAGF)[22] for seven days at 37°C in sealed containers. The pH of the artificial saliva was adjusted to 6.8 to resemble the pH of natural saliva and verified with a digital pH meter (Eutech ph2700, Singapore).

Dynamic Flexural Testing

After conditioning, the test specimens were subjected to dynamic flexural testing (DMA RSAG2, TA Instruments, New Castle, DE, USA) in their air or artificial saliva at 37°C. Specimens were loaded using a three-point bending configuration with span lengths of 20 mm for IFT and 10 mm for MFT inside

an immersion container enclosed in an environmental chamber.

Loading frequency was set to 0.1 to 10 Hz to represent the range from "close to static" to the upper limit of normal chewing frequency.[7,23] Storage modulus, loss modulus, and loss tangent data were recorded throughout the experiment and were computed as follows:[24]

Storage modulus:

$$E' = (\sigma^\circ/\varepsilon^\circ)\text{cosd} = (f_o/b\text{k})\text{cosd};$$

Loss modulus:

$$E'' = (\sigma^\circ/\varepsilon^\circ)\text{sind} = (f_o/b\text{k})\text{sind};$$

Loss tangent:

$$\text{Tan}\delta = E''/E',$$

where σ° is the maximum stress at the peak of the sine wave, ε° is the strain at the maximum stress, f is the force applied at the peak of the sine wave, b is the sample geometry term, and k is the sample displacement at the peak.

The sample geometry b for a three-point bending bar was calculated as follows:

$$4BH^3/L^3,$$

where B is the width of the specimen (in millimeters), H is the height of the specimen (in millimeters), and L is the distance between the supports (in millimeters).

Static Flexural Testing

After dynamic testing, the MFT and IFT specimens were loaded until fracture in a universal testing machine (Shimadzu Corporation, Kyoto, Japan) with a load cell of 5 KN and a crosshead speed of 0.5 mm/min until fracture occurred. Flexural strength, σ, in megapascals (MPa), was calculated using the following equation:

$$\sigma = \frac{3PL}{2BH^2},$$

where P is the maximum load exerted on the specimen (in Newtons), L is the distance between the supports (in millimeters: 20 mm for IFT and 10 mm for MFT), B is the width of the specimen (in millimeters), and H is the height of the specimen (in millimeters).

Flexural modulus, E', in megapascals (MPa), was calculated using the following equation:

$$E' = \left(\frac{F}{D}\right)\left(\frac{L^3}{4BH^3}\right),$$

where F/D is the slope (in Newtons per millimeter), measured in the straight-line portion of the load-deflection graph. L, B, and H were defined in the flexural strength equation. Flexural modulus was subsequently converted to Gigapascal (GPa).

Statistical Analysis

The SPSS statistical program (Version 12.0.1, SPSS Inc, Chicago, IL, USA) was used to analyze the flexural data obtained. Normality testing was done using the Shapiro-Wilk test. As data were found to be normally distributed, parametric analysis was permissible. The interaction effects between the independent variables (specimen dimension, conditioning medium, and material) and each of the dependent variables (storage modulus, loss modulus, loss tangent, flexural strength, and flexural modulus) were evaluated using factorial analysis of variance (ANOVA). One-way ANOVA/Tukey post hoc test and t-tests were used to compare material, specimen dimension, and storage medium differences. Correlations between IFT and MFT for dynamic and static testing results were done with Pearson correlation. All statistical analyses were carried out at significance level of $\alpha = 0.05$.

RESULTS

The mean storage modulus, loss modulus, loss tangent, flexural strength, and modulus values for the various RBCs, flexural tests, and conditioning mediums are reflected in Tables 2 and 3. Although interaction effects among material, specimen dimension, and conditioning medium were not significant, the influence on storage modulus and loss tangent of RBCs was medium- and specimen dimension–dependent.

Table 4 shows the intermaterial comparison of dynamic and static flexural properties when materials were conditioned in air and artificial saliva for IFT and MFT. When conditioned in air, no significant differences in flexural properties were observed between materials, with the exception of loss modulus and loss tangent for IFT and flexural strength for MFT. BB had significantly lower loss modulus, loss tangent, and flexural strength values than the other RBCs. When conditioned in artificial saliva, no significant differences in flexural proper-

Table 2: *Mean Dynamic and Static Flexural Values (Standard Deviations in Parentheses) for the Various Materials, IFT and MFT, When Conditioned in Air*

Materials	Air									
	ISO 4049 Flexural Test (IFT)					Mini-Flexural Test (MFT)				
	Storage Modulus, GPa	Loss Modulus, GPa	Loss Tangent, 10^{-3}	Flexural Strength, MPa	Flexural Modulus, GPa	Storage Modulus, GPa	Loss Modulus, GPa	Loss Tangent, 10^{-3}	Flexural Strength, MPa	Flexural Modulus, GPa
Filtek Z350 (FZ)	6.79	0.46	68	99.49	10.95	6.29	0.42	68	135.20	8.23
	(0.41)	(0.02)	(3)	(13.83)	(0.76)	(0.29)	(0.04)	(5)	(17.08)	(0.89)
Filtek Bulk-Fill (FB)	6.35	0.47	73	110.13	12	6.20	0.43	69	144	8.04
	(0.33)	(0.05)	(5)	(15.01)	(1)	(0.72)	(0.06)	(4)	(19.32)	(1.11)
Beautifil-Bulk Restorative (BB)	6.46	0.40	62	98.90	11.58	5.79	0.38	66	117.53	8.19
	(0.58)	(0.05)	(4)	(11.14)	(0.55)	(0.36)	(0.05)	(7)	(10.22)	(1.12)

ties between materials were observed only for storage modulus with IFT and loss modulus with MFT.

Significant differences in dynamic and static flexural values between the two flexural tests and conditioning mediums are indicated in Table 5 and Figures 1 and 2. Differences between IFT and MFT as well as air and artificial saliva were material-dependent. For all RBCs, MFT resulted in higher flexural strength than did IFT, regardless of the conditioning medium. Flexural modulus with IFT was greater than with MFT. For both IFT and MFT, exposure to artificial saliva resulted in higher loss tangent values, while conditioning in air led to greater flexural strength. In addition, conditioning in air gave rise to greater flexural modulus with IFT.

The results of correlation analysis are displayed in Tables 6 and 7. For both conditioning mediums, the strongest correlation between IFT and MFT was observed for flexural strength (r=0.85 and 0.97, respectively). While significant positive correlations were observed for all flexural properties with saliva, no significant correlations were detected for loss tangent and flexural modulus with air.

DISCUSSION

This study compared the static and dynamic flexural properties of RBCs between IFT and MFT with conditioning medium as a confounding variable. Based on the findings of this study, all three null hypotheses were rejected. Storage modulus, loss tangent, and flexural strength of the RBCs were found to be reliant on conditioning medium. For both IFT and MFT, storage modulus, flexural strength, and modulus when stored in air were significantly greater than when stored in artificial saliva, in which case significant differences in flexural properties exist. In contrast, loss modulus and loss tangent values were higher with artificial saliva. Conditioning in aqueous solutions has been reported[21] to decrease the flexural properties of RBCs, and this finding was supported by this study as well. The RBCs evaluated contain silica or silicate glass fillers that have irregularly distributed Si-O-Si bonds. When exposed to artificial saliva, the resin matrices

Table 3: *Mean Dynamic and Static Flexural Values (Standard Deviations in Parentheses) for the Various Materials, IFT and MFT, When Conditioned in Artificial Saliva*

Materials	Artificial Saliva									
	ISO 4049 Flexural Test (IFT)					Mini-Flexural Test (MFT)				
	Storage Modulus, GPa	Loss Modulus, GPa	Loss Tangent, 10^{-3}	Flexural Strength, MPa	Flexural Modulus, GPa	Storage Modulus, GPa	Loss Modulus, GPa	Loss Tangent, 10^{-3}	Flexural Strength, MPa	Flexural Modulus, GPa
Filtek Z350 (FZ)	5.97	0.50	83	64.82	9.44	5.48	0.47	88	91.71	6.58
	(0.31)	(0.07)	(9)	(5.39)	(0.67)	(0.56)	(0.06)	(9)	(10.10)	(0.76)
Filtek Bulk-Fill (FB)	6.19	0.54	88	90.34	10.36	6.09	0.46	84	122.39	7.64
	(0.20)	(0.03)	(5)	(9.54)	(0.46)	(0.49)	(0.06)	(10)	(16.63)	(1.07)
Beautifil-Bulk Restorative (BB)	6.17	0.42	68	64.10	10.35	5.51	0.43	73	86.6	7.34
	(0.52)	(0.06)	(7)	(5.33)	(0.64)	(0.53)	(0.06)	(5)	(3.57)	(0.92)

Table 4: *Results of Intermaterial Comparison of Dynamic and Static Flexural Properties When Conditioned in Air and Artificial Saliva for IFT and MFT*[a]

Test	Properties	Differences	
		Air	Artificial Saliva
IFT	Storage modulus	NS	NS
	Loss modulus	FB, FZ > BB	FB, FZ > BB
	Loss tangent	FB > FZ > BB	FB, FZ > BB
	Flexural strength	NS	FB > FZ, BB
	Flexural modulus	NS	FB, BB > FZ
MFT	Storage modulus	NS	FB > FZ
	Loss modulus	NS	NS
	Loss tangent	NS	FZ, FB > BB
	Flexural strength	FB > BB	FB > FZ, BB
	Flexural modulus	NS	FB > FZ

Abbreviations: BB, Beautifil-Bulk; FB, Filtek Bulk-Fill; FZ, Filtek Z350; IFT, ISO 4049 flexural test; MFT, mini-flexural test.
[a] *> Indicates statistical significance, while NS indicates no statistical significance. Results of one-way analysis of variance and post hoc Tukey test ($p<0.05$).*

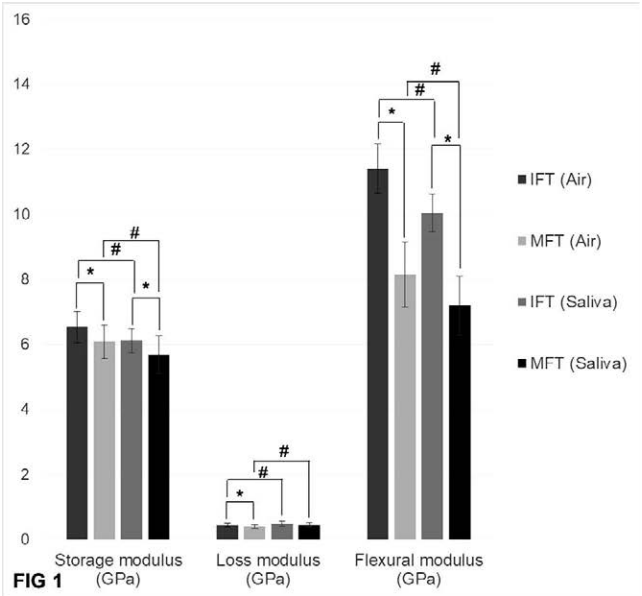
absorb water, swell, and radial stresses develop at the filler interfaces, thus straining the Si-O-Si bonds present. The high energy levels arising from the strained Si-O-Si bonds render the fillers more susceptible to stress corrosion attack ($Si_2O+H_2O=2$ SiOH).[25] Complete or partial filler debonding occurs and results in the decreased storage modulus, flexural strength, and modulus observed. The plasticizing effect of water on the resin matrices also explains the significantly greater loss tangent values (which are inversely proportional to storage modulus) when the RBCs were stored in artificial saliva.

For both IFT and MFT, storage in artificial saliva appeared to be more discriminative than storage in air. When conditioned in air, few significant differences in dynamic and flexural properties were observed between materials for both tests. For IFT, FB and FZ had significantly greater loss modulus and tangent than did BB. For MFT, the flexural strength of FB was significantly greater than that of BB. When conditioned in artificial saliva, significant differences between materials were observed for almost all flexural properties, with the exception of storage modulus for IFT and loss modulus for MFT. BB generally had lower loss tangent and flexural strength than did FZ and FB. BB, a bulk-fill giomer material, is based on pre-reacted glass ionomer (PRG) technology in which acid-reactive fluorosilicate glass is reacted with polyacids in the presence of water, freeze-dried, milled, silanized, ground, and utilized as fillers. Despite its higher filler content (74.5% as compared to 58.4% and 63.3% volume for FB and FZ, respectively), the lower flexural strength of BB may be attributed to the use of irregularly shaped PRG fillers that may serve as foci of stress concentrations.[26] Moreover, the degree of conversion of BB has been found[27] to be relatively lower than that of other bulk-fill RBCs.

Table 5: *Comparison of Static and Dynamic Flexural Properties Between IFT and MFT as Well as Air and Artificial Saliva for the Various Materials*[a]

Material	Properties	IFT vs MFT		Air vs Artificial Saliva	
		Air	Artificial Saliva	IFT	MFT
Filtek Z350 (FZ) [control]	Storage modulus	IFT > MFT	IFT > MFT	Air > saliva	Air > saliva
	Loss modulus	IFT > MFT	NS	NS	NS
	Loss tangent	NS	NS	Saliva > air	Saliva > air
	Flexural strength	MFT > IFT	MFT > IFT	Air > saliva	Air > saliva
	Flexural modulus	IFT > MFT	IFT > MFT	Air > saliva	Air > saliva
Filtek Bulk-Fill (FB)	Storage modulus	NS	NS	NS	NS
	Loss modulus	NS	IFT > MFT	Saliva > air	NS
	Loss tangent	NS	NS	Saliva > air	Saliva > air
	Flexural strength	MFT > IFT	MFT > IFT	Air > saliva	Air > saliva
	Flexural modulus	IFT > MFT	IFT > MFT	Air > saliva	NS
Beautifil-Bulk Restorative (BB)	Storage modulus	IFT > MFT	IFT > MFT	NS	NS
	Loss modulus	NS	NS	NS	NS
	Loss tangent	NS	MFT > IFT	Saliva > air	Saliva > air
	Flexural strength	MFT > IFT	MFT > IFT	Air > saliva	Air > saliva
	Flexural modulus	IFT > MFT	IFT > MFT	Air > saliva	NS

[a] *> Indicates statistical significance, while NS indicates no statistical significance. Results of* t*-test ($p<0.05$).*



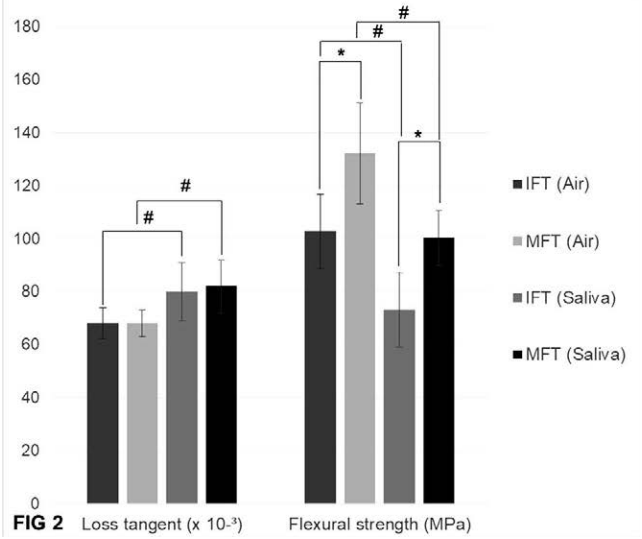


Figure 1. *Comparison of mean storage, loss, and flexural modulus between IFT and MFT as well as air and artificial saliva (pooled data for all materials). Results of* t*-test (*$p<0.05$*).*

Figure 2. *Comparison of mean loss tangent and flexural strength between IFT and MFT as well as air and artificial saliva (pooled data for all materials). Results of* t*-test (*$p<0.05$*).*

Storage modulus and loss tangent of the RBCs were also predisposed to specimen dimension variations. For both conditioning mediums, storage, loss, and flexural modulus obtained with IFT were higher than for MFT. In contrast, loss tangent and flexural strength associated with MFT were greater than for IFT. The difference in storage, loss, and flexural modulus between IFT and MFT, though statistically significant, was only marginal when compared to flexural strength (Figures 1 and 2). Among the aforementioned dynamic and static flexural properties, flexural strength was the most widely reported by other authors. The higher flexural strength observed with MFT corroborated the findings of earlier studies[16,20] and had been attributed to shear deformation with reduction of support distance. Calabrese and others,[17] however, reported significantly higher flexural strength with IFT. The incongruity in findings is attributable to variances in the RBCs evaluated.[15] Moreover, Muench and others[19] found that flexural strength was not influenced by length when specimens were light-cured on both surfaces. For stress-bearing restorations, RBCs with high flexural strength and modulus of elasticity are desirable to resist occlusal loads and sustain the tooth-restoration interface.[16]

All RBCs fulfilled the ISO flexural strength (IFT) requirement of 80 MPa when conditioned in air, but only FB achieved this value when conditioned in artificial saliva. As flexural strength values observed with MFT were higher than those observed with IFT, new minimum flexural strength values must be established for MFT, and conditioning should be carried out in artificial saliva or distilled water.

The modulus of elasticity quantifies a material's resistance to elastic (nonpermanent) deformation when a force is applied. It is defined by the slope of the stress-strain curve in the elastic region. Values for modulus of elasticity (elastic modulus) obtained with both dynamic and static testing should theoretically be similar. The dynamic and static

Table 6: *Correlations Between Dynamic and Static Flexural Properties When Conditioned in Air*

Properties	MFT Storage Modulus	MFT Loss Modulus	MFT Loss Tangent	MFT Flexural Strength	MFT Flexural Modulus
IFT storage modulus	**0.72****	0.59**	NS	0.59**	NS
IFT loss modulus	0.77**	**0.67****	NS	0.76**	NS
IFT loss tangent	0.44*	0.44*	**NS**	0.52**	NS
IFT flexural strength	0.81**	0.68**	NS	**0.85****	NS
IFT flexural modulus	NS	NS	NS	NS	**NS**

Abbreviations: IFT, ISO 4049 flexural test; MFT, mini-flexural test; NS, not significant.
** Correlation is significant at the 0.05 level,* $p<0.05$*; ** Correlation is significant at the 0.01 level,* $p<0.01$*. Results of Pearson correlation. Bold numbers indicate correlation between IFT and MFT for the same flexural properties.*

Table 7: *Correlations Between Dynamic and Static Flexural Properties When Conditioned in Artificial Saliva*

Properties	MFT Storage Modulus	MFT Loss Modulus	MFT Loss Tangent	MFT Flexural Strength	MFT Flexural Modulus
IFT storage modulus	**0.82****	0.50**	NS	0.42*	0.53**
IFT loss modulus	0.62**	**0.38***	0.44*	0.68**	NS
IFT loss tangent	NS	NS	**0.46***	0.57**	NS
IFT flexural strength	0.80**	NS	NS	**0.97****	0.47**
IFT flexural modulus	NS	NS	NS	NS	**0.45***

Abbreviations: IFT, ISO 4049 flexural test; MFT, mini-flexural test; NS, not significant.
** Correlation is significant at the 0.05 level, $p<0.05$; ** Correlation is significant at the 0.01 level, $p<0.01$. Results of Pearson correlation. Bold numbers indicate correlation between IFT and MFT for the same flexural properties.*

modulus of elasticity for RBCs for both IFT and MFT had not been compared in prior studies. The MFT geometry still permitted adequate deflection of the RBCs within the small load capacity of the DMA instrument employed. For both flexural tests, modulus of elasticity obtained with dynamic testing (storage modulus) was lower than that obtained with static testing (flexural modulus), regardless of conditioning medium. Sabbagh and others,[28] however, reported higher elastic modulus with dynamic testing for IFT. In their study, dynamic modulus of elasticity was determined through signal analysis with the Grindosonic instrument (Lemmens Elektronics, Haasrode, Belgium) and not DMA. The findings of the current study also contradicted those on other industrial materials in which dynamic modulus of elasticity was about 20% to 40% higher than static modulus.[29] The ratio of dynamic to static modulus of elasticity was relatively constant and ranged between 53% and 63% for IFT and between 70% and 83% for MFT for both conditioning mediums. As the ratios for MFT were more favorable (closer to 100%), flexural test specimens based on MFT dimensions are advocated. If possible, the modulus of elasticity of the RBCs should be similar to or higher than that of dentin, which is approximately 19 GPa.[30] None of the RBCs evaluated achieved this value for both IFT and MFT.

Correlations between IFT and MFT varied somewhat between conditioning in air and artificial saliva. To better simulate the wet oral environment, conditioning and testing in artificial saliva or distilled water is recommended. When conditioned in artificial saliva, significant positive correlations were observed for all dynamic and static flexural properties between IFT and MFT. Strength of the relationships ranged from moderate for loss modulus, loss tangent, and flexural modulus to strong for storage modulus and flexural strength. As MFT allows for the prediction of mechanical performance of RBCs under more clinically realistic conditions and is more efficient than IFT, it is a promising replacement for the ISO 4049 for both dynamic and static flexural testing.

CONCLUSIONS

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

1. Significant differences between IFT and MFT flexural test values were observed. With the exception of flexural strength, values for dynamic and static flexural properties were generally higher with IFT.
2. For both IFT and MFT, significant differences in dynamic and static flexural properties were observed between conditioning in air and conditioning in artificial saliva. As conditioning in artificial saliva was more discriminative and clinically relevant, it is the conditioning medium of choice.
3. Moderate to strong positive correlations were observed between IFT and MFT for all dynamic and static flexural properties when RBCs were conditioned in artificial saliva.
4. MFT holds promise as a replacement for IFT in view of its significant correlation to IFT, clinical relevance, and greater efficiency.

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Conflict of Interest

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

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Original and Repair Bulk Fracture Resistance of Particle Filler and Short Fiber–Reinforced Composites

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Clinical Relevance

Longevity of repaired direct composite restorations may be improved by including a short E-glass fiber–reinforced composite with a semi-interpenetrating network matrix as the substrate material in bilayered restorations.

SUMMARY

Objective: This study aimed to evaluate the original (OR) and repair (RR) fracture resistance of a semi-interpenetrating polymer network (semi-IPN)–based short fiber–reinforced composite compared to dimethacrylate-based composite materials by means of the *V*-notch test.

Methods and Materials: Circular specimens (5×2 mm) with a centrally machined 90° *V*-shaped notch were prepared. Four bulk fill (Filtek Bulk Fill, Venus Bulk Fill, TetricEvo Ceram Bulk Fill, SDR), three microfilled hybrid (GC-Anterior, GC-Posterior, Z250), one nanofilled (SupremeXTE), and two short fiber–reinforced (Alert, everX Posterior) composites were selected. EverX Posterior was the semi-IPN material. Specimens (n=12/group) were either dry or water stored for 7 and 30 days, respectively, at 37°C and then loaded in two-point load until fracture. One-half of each tested specimen was used for the repair procedure. Repairing surfaces were diamond-bur ground, etched, and treated with silane containing universal adhesive (Scotchbond Universal) before repair.

Results: Three-way analysis of variance revealed a significant statistical difference between the groups ($p<0.05$). The fracture resistance of dry-stored groups was greater than that of water-stored groups. The highest OR was observed for dry-stored Alert (23.4 N/mm), which significantly deteriorated in water (17.4 N/mm) ($p<0.05$). The highest RR was observed for everX Posterior (20.0 N/mm), which did not deteriorate in water significantly (19.0 N/mm) ($p>0.05$). The everX Posterior preserved the specimens' integrity at the final fracture load

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(ductile fracture), whereas all other materials fractured into two halves at the interface (adhesive failure).

Conclusions: The only material that provided enhanced repair strength that was close to the original cohesive strength of the material was everX Posterior. The endurance of repaired restorations can be improved by using semi-IPN-based filling material.

INTRODUCTION

Replacement, repair, and refurbishment are types of interventions employed for restoring defective dental restorations. The least invasive treatment is refurbishment, which is used when an existing restoration has to be reshaped or refinished or has overhangs that need to be removed. A repair is more conservative than replacement; a repair is used to restore a defective part of a restoration, whereas replacement involves removal of an entire restoration.[1]

The bonding between the old and the new composite in repair is important. Although repair is a simple intervention, there is no consensus on the best course of treatment of the repairing substrate surface prior to repair. The most commonly used means for surface treatment of the repairing composite are macro- or micromechanical roughening with a bur, grinding paper, sandblasting with aluminum oxide particles or silica-coated particles, and etching with hydrofluoric acid.[2] Some of these, such as grinding paper or hydrofluoric acid, are unfeasible or unsafe methods for direct intraoral repair. In addition, the safety of sandblasting has been also questioned due to the aerosol contamination of the operating room.[3] Instead, clinically friendlier techniques are etching with phosphoric acid or grinding with diamond burs.[4-7] The last one was shown to provide equally good[6] or improved[4] repair bond strength compared to sandblasting with aluminum oxide particles. Surface roughening is followed by an application of an intermediate layer, which could be a silane agent alone or in combination with a bonding agent,[5,8] an unfilled or a filled bonding agent[5,6,9,10] or a low-viscosity flowable composite.[4,5] Usually, mechanical or chemical treatment used alone is ineffective in reestablishing the original strength of the material, but used together they improve the repair strength.[1,10] Lately, it has also been observed that bonding agent used alone as an intermediary layer aids the repair bond,[1] which is an important finding because a clinician would normally apply an adhesive layer to the whole cavity.

In order to establish a durable repair, both mechanical and chemical bonding must be achieved. The type of the composite to be repaired is crucially important, as different resin matrices (cross-linked, linear, or a combination of both) have different chemical and physical properties.[11,12] The bonding of a new composite to an aged thermoset matrix (dimethacrylate) in repairs is poor. This is because thermoset matrices are highly cross-linked and nondissolvable by monomers.[12-14] On the other hand, thermoplastic matrices (eg polymethylmethacrylates) have good reparability potential due to their linear chains. The combination of both phases, thermoset (cross-linked) and thermoplastic (linear), in one matrix system is known as a semi-interpenetrating polymer network (semi-IPN).[13] This matrix type has good reparability potential due to the presence of polymethylmethacrylate chains, which are easily dissolvable with monomers that have solubility parameters close to it.[13,15] Semi-IPN has been described for continuous fiber-reinforced composites (FRCs) and has been proven to be superior to cross-linked resin matrix in FRC repairs[11,12] because fresh monomers diffuse significantly deeper into semi-IPN than into cross-linked FRC.[14,16] Currently, however, only one semi-IPN-based composite material for filling applications is available on the market. This restorative material contains short fibers 0.85 to 1.09 mm in length,[17] and it therefore represents a semi-IPN-based short fiber–reinforced composite (SFRC). According to the fiber aspect ratio theory,[18-20] this material could be classified as high-aspect-ratio SFRC (short fiber length on a millimeter scale). This is important because only low-aspect-ratio SFRCs (short fiber length on a micrometer scale) have been presented on the market thus far.[21]

The semi-IPN-based high-aspect-ratio SFRC is commercially known as everX Posterior (EXP, GC Corp, Tokyo, Japan). It has been shown that the use of this SFRC obtains cohesive fractures between immediately incrementally placed layers of composite, even on removal of the oxygen inhibition layer.[22] This property could be advantageous in composite repairs, when this composite is the repairing substrate, that is, the underlying material. Furthermore, the mechanical and structural characterization of EXP revealed that protruding fiber ends and bridging fibers contribute to the fracture toughness of the material.[17] Based on these earlier findings, it could be hypothesized that the presence of both semi-IPN matrix and protruding fibers could enhance the repair strength of the EXP, even though the silane is removed from the fiber surface due to grinding. Consequently, an

Table 1: *Materials Used in the Present Investigation*

Material (Manufacturer)	Code and Lot Number	Type of Composite	Resin Matrix
Filtek Z250 (3M ESPE, Seefeld, Germany)	FZ250 N565032	Microhybrid	Bis-GMA, Bis-EMA, UDMA, TEGDMA
Filtek Supreme XTE (3M ESPE)	FS N649930	Nanofilled	Bis-GMA, Bis-EMA, UDMA, TEGDMA
G-aenial Anterior (GC Corp, Tokyo, Japan)	GA 1501151	Microfilled hybrid	UDMA, dimethacrylate comonomers (Bis-GMA free)
G-aenial Posterior (GC)	GP 1301161	Microfilled hybrid	UDMA, dimethacrylate comonomers (Bis-GMA free)
Smart Dentin Replacement (Dentsply, York, PA, USA)	SDR 151020	Flowable bulk fill	Modified UDMA, TEGDMA, EBPDMA
Filtek Bulk Fill (3M ESPE)	FBF N600679	Flowable nanofilled bulk fill	Bis-GMA, Bis-EMA, UDMA, procrylate
Venus Bulk Fill (Heraeus Kulzer, GmbH, Hanau, Germany)	VBF 010030	Flowable nanohybrid bulk fill	UDMA, EBPDMA
Tetric Evo Ceram Bulk Fill (Ivoclar Vivadent, Schaan, Liechtenstein)	TBF R72542	High viscosity (sculptable) nanohybrid bulk fill	Bis-GMA, Bis-EMA, UDMA
everX Posterior (GC)	EXP 1309111	High-aspect-ratio (millimeter-scale) SFRC[a]	Bis-GMA, TEGDMA, PMMA
Alert (Jeneric/Pentron, Wallingford, CT, USA)	Alert 3762763	Low-aspect-ratio (micrometer-scale) SFRC[b]	Bis-GMA, UDMA, TEGDMA, THFMA,
Scotchbond Universal (3M ESPE)	SU 589527	Filled phosphate dimethacrylate–based universal adhesive resin	Bis-GMA, HEMA, MDP, polyethylene glycol, water, initiator, silane

Abbreviations: Bis-GMA, bisphenol-A-glycidyl dimethacrylate; bis-EMA, bisphenol-A-dyethoxy dimethacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; EBPDMA, ethoxylated bisphenol A dimethacrylate, PMMA: polymethylmethacrylate; THFMA, tetrahydrofurfuryl-2-methacrylate; HEMA, hydroxyethylmethacrylate; MDP, methacryloyloxi-decyl-dihydrogen-phosphate.
[a] *High-aspect-ratio SFRC: short fiber length on a millimeter scale.*
[b] *Low-aspect-ratio SFRC: short fiber length on a micrometer-scale.*

objective in this study was to investigate the repair properties of the semi-IPN-based SFRC (EXP) in comparison to dimethacrylate-based composites. In addition, one low-aspect-ratio dimethacrylate-based SFRC (Alert, Jeneric/Pentron, Wallingford, CT, USA) was evaluated (Table 1). The original and repair strengths were evaluated using the V-notch test, which measures the bulk fracture resistance, known also as bulk toughness or torque to failure. This method was originally developed in 1995 by Uctasli and others.[23] The advantages of the method are that specimen size is approximate to the size of a dental restoration and that the test setup enables the assessment of stress in a dental restoration during occlusion. Indeed, the V-notch section resembles the fissure of a posterior tooth, whereas the roller fixed in the testing device resembles the antagonist.[23]

METHODS AND MATERIALS

Ten commercially available composites were investigated, including four bulk fill composites, three microfilled hybrid composites, one nanofilled composite, one low-aspect-ratio SFRC, and one high-aspect-ratio SFRC. The high-aspect-ratio SFRC was the semi-IPN-based composite material (EXP). The other materials were dimethacrylate-based composites, and all were shade A3. The restorative materials, their abbreviations, and codes are presented in Table 1.

Specimens (n=12/group) were prepared in a circular Teflon mold, 5 mm in diameter and 2 mm in thickness, with a centrally machined V-shaped notch. The angle of the central notch was accurately 90°. Original and repair fracture resistance (OR and RR, respectively) was evaluated by this test.

Monolithic Specimens (OR)

The material was packed into the V-shaped mold, pressed with a Mylar strip, and covered with a glass plate in order to exude excess material. Prior to polymerization, the glass plate was removed, and the specimen was light polymerized against the Mylar strip in intimate contact with the light-curing unit tip

Table 1: *Extended.*

Material (Manufacturer)	Filler Type	Filler, wt%/vol%
Filtek Z250 (3M ESPE, Seefeld, Germany)	Zirconia/ silica: 0.01-3.5 μm	78/60
Filtek Supreme XTE (3M ESPE)	Aggregated zirconia/silica cluster: 0.6-1.4 μm and nonagglomerated/nonaggregated silica filler: 20 nm	78.5/59.5
G-aenial Anterior (GC Corp, Tokyo, Japan)	Prepolymerized: silica and strontium and lanthanoid fluoride containing filler 16-17 μm; silica filler >100 nm and fumed silica filler <100 nm	80/63
G-aenial Posterior (GC)	Prepolymerized: silica and strontium and lanthanoid fluoride containing filler 16-17 μm; fluoro-alumino-silicate >100 nm and fumed silica filler <100 nm	81/65
Smart Dentin Replacement (Dentsply, York, PA, USA)	Barium-alumino-fluoro-boro-silicate and strontium-alumino-fluoro-silicate: 4.2 μm	68/44
Filtek Bulk Fill (3M ESPE)	Zirconia/silica: 0.01-3.5 μm Ytterbium trifluoride: 0.1-5.0 μm	64.5/42.5
Venus Bulk Fill (Heraeus Kulzer, GmbH, Hanau, Germany)	Barium-alumino-fluoro-silicate and ytterbium trifluoride, silica: 0.02-5 μm	65/38
Tetric Evo Ceram Bulk Fill (Ivoclar Vivadent, Schaan, Liechtenstein)	Barium-alumino-silica, spherical mixed oxide, prepolymer filler (ytterbium trifluoride, glass filler, and monomer 17 wt%)	80 (with 17% prepolymers)/60
everX Posterior (GC)	E-glass fiber (0.3-1.9 mm) Barium-boro-silicate	Filler: 74.2/53.6 E-glass: 8.6/7.2
Alert (Jeneric/Pentron, Wallingford, CT, USA)	E-glass fiber (60-80 μm) Barium-boro-alumino-silicate: 0.7 μm	Total, filler and fiber: 84/70
Scotchbond Universal (3M ESPE)	Colloidal silica nanofiller Vitrebond copolymer: fluoro-alumino-silicate glass powder based	Not applicable (trade secret)

(0-mm distance) for 20 seconds using a light-polymerizing unit (Elipar S10, 3M ESPE, Seefeld, Germany), having a 10-mm-diameter tip, an output intensity of 1600 mW/cm^2, and a wavelength range between 430 and 480 nm. On removal from the mold, specimens were additionally polymerized from the top side for 20 seconds and were then stored at 37°C either dry for seven days or in water for 30 days before testing. The thickness of each specimen was measured prior to testing with a digital caliper with an accuracy of ±0.002 mm (Mitutoyo Corp, Tokyo, Japan).

A fracture test was conducted in a universal material testing machine (Lloyd model LRX, Lloyd Instruments Ltd, Fareham, UK). The external force was applied with a cylindrical roller 3 mm in diameter in two-point opening (mode I) tensile load at a crosshead speed of 1.0 mm/min until failure. The cylindrical roller provided a two-point contact at the V-shaped notch, enabling equal distribution of the load with the purpose of breaking the specimens into two halves (Figure 1a and 1b).

Repaired Specimens (RR)

After testing, one-half of the fractured specimen was used for repair. The fracture surface of each half specimen was ground with a fine-grit diamond bur (40-μm mean grit size), etched with 37% phosphoric acid (Scotchbond Universal Etchant, 3M ESPE) for 15 seconds, washed with water spray for 15 seconds, and dried from a ~5-mm distance. A bonding agent (Scotchbond Universal adhesive, 3M ESPE) was then rubbed onto the surface using a microbrush applicator for 15 seconds, gently air-dried, and light cured for 20 seconds. The specimen was then returned to the mold, and new material was bonded to it by filling the vacant end with the same material used for fabrication of the original specimen in one increment. The repair specimens were thus a "sandwich" of old and new composite. In other words, the repairing substrate was the old composite, which was previously aged and tested. Preparation and light-curing steps were otherwise the same as for the monolithic specimens. Repaired specimens were made within seven days after testing the monolithic specimens. Following repair, storage mediums and storage times for the repaired specimens were identical with those of the original specimens; dry specimens were exposed to seven days of dry storage, and water-stored specimens were subjected to water storage for 30 days, both at 37°C. Specimens were

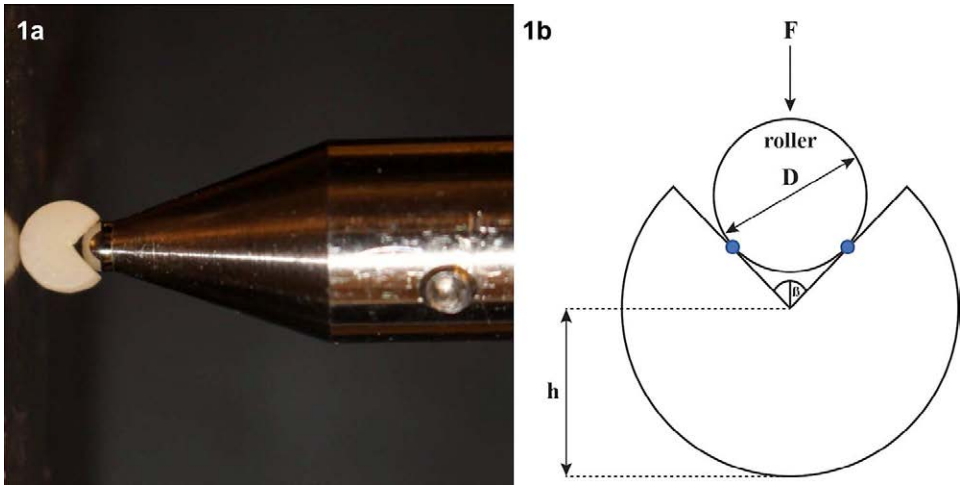

Figure 1. *Assembly of the V-shape test. (a): Specimen positioned under roller during testing. (b): Diagram. F, load applied; D, diameter of the roller (3 mm); h, height from notch to base; "β, half angle of the right-angled notch; solid circles, places of two-point contact.*

fractured as described earlier for the monolithic specimens.

OR and RR values were derived in accordance with the following equation:

$$T = F\,D\,0.71/2\,t\,h,$$

where T is the fracture resistance in newton millimeters (N/mm), F is the applied load in newtons (N), D is the roller diameter (3 mm), t is the specimen thickness (2 mm), and h is the height from notch to base (2.5 mm). When the specimen's notch is 90°, its half angle is $\phi = 45°$ and $\sin 45° = 0.71$, and this was the value used in the previous equation.

Fracture pattern analysis and scanning electron microscopy (SEM) investigations were conducted for EXP specimens, which were the only ones that did not break into two halves. The crack interface of three specimens (n=3) was analyzed. The specimens were ground wet (Stuers LabPol-21, Stuers A/S, Copenhagen, Denmark) with silicon carbide papers of decreasing abrasiveness (1000, 1200, and 4000 grit) and gold sputter coated prior to SEM examination.

Statistical Analysis

The data were statistically analyzed with SPSS version 23 (SPSS, IBM Corp, Armonk, NY, USA). Three-way analysis of variance (ANOVA) followed by the Tukey *post hoc* test (α=0.05) was used to determine if the fracture resistance was different for monolithic and repaired specimens (OR and RR), for the materials investigated (10 materials), and for the type of storage medium (dry or water). Material, storage type, and fracture resistance category (OR and RR) were independent variables, and fracture resistance (OR, RR results) was the dependent variable. Two-way ANOVAs followed by the Tukey *post hoc* test (α=0.05) were also conducted to evaluate the interactions between the material type and the storage medium individually for monolithic and repaired specimens (ie, for OR and RR separately).

RESULTS

OR was higher compared to RR for both dry- and water-stored specimens. Three-way ANOVA showed statistically significant interactions for factors "fracture resistance category (OR or RR)" ($p<0.05$), "material type," and "fracture resistance category" ($p=0.00003$) and for "storage medium," "material type," and "fracture resistance category" ($p=0.047$) but not for "storage" and "fracture resistance category" ($p=0.075$). However, there was a concern that the effect that each type of material had on the fracture resistance category (OR or RR) might have been different for dry and water storage media. Two-way ANOVA confirmed this, showing that the interaction between the material type and the storage medium was significant for OR ($p=0.014$) but not for RR ($p=0.332$). Statistical differences across the fracture resistance values and storage mediums for each material are presented in Figure 2.

Fracture resistance deteriorated in water. This was significant for the majority of materials in the OR category ($p<0.05$) but insignificant for the majority of materials in the RR category ($p>0.05$). The highest OR value was observed for dry-stored Alert specimens (23.4 N/mm), which significantly deteriorated in water (17.4 N/mm) ($p<0.05$). The highest RR value was observed for EXP specimens (20.0 N/mm), which did not significantly deterio-

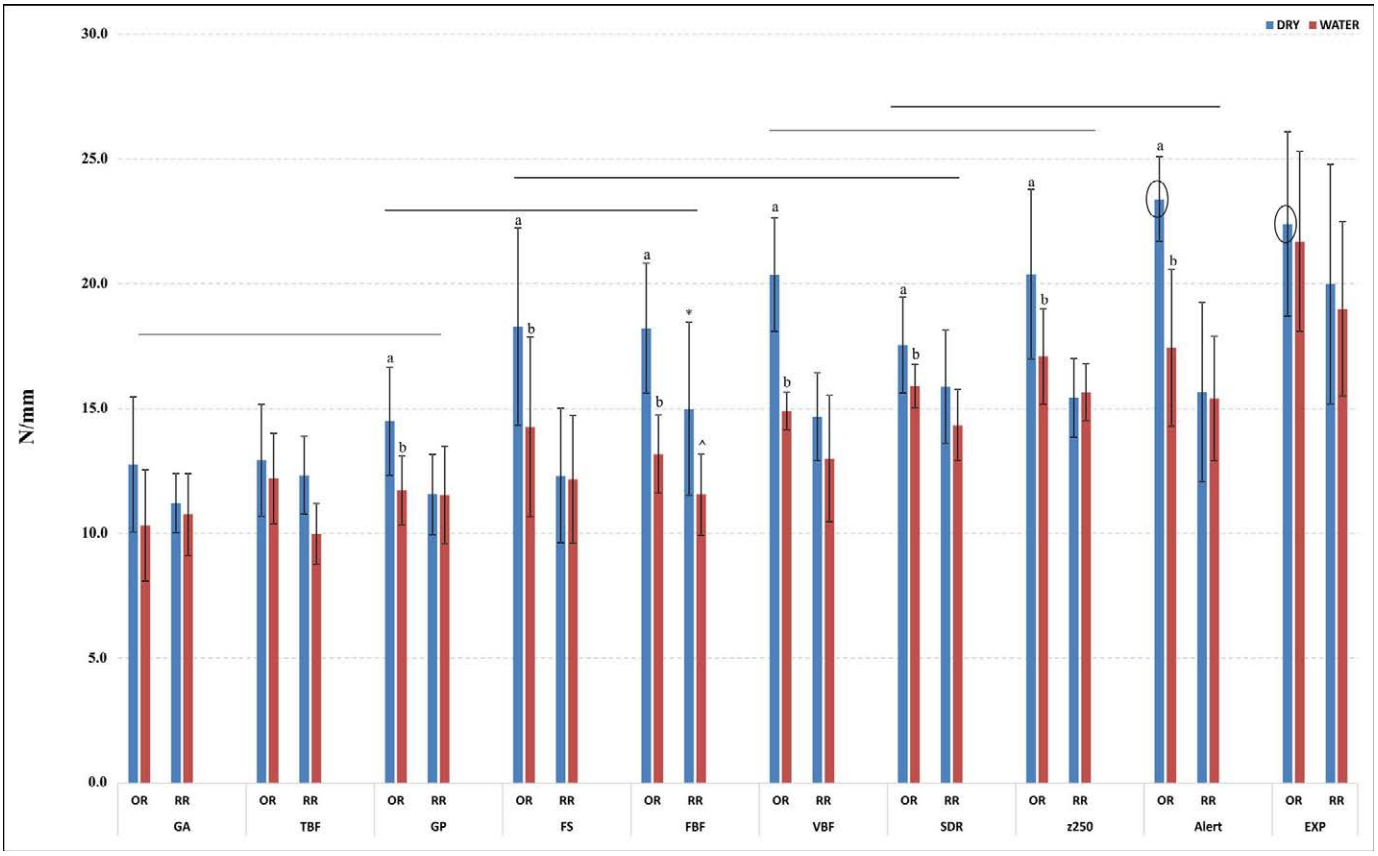

Figure 2. *Graphs showing original (OR) and repaired fracture resistance (RR) values of investigated materials. Horizontal lines above columns indicate groups that were statistically similar (*p*>0.05). For each material analyzed separately, small letters show the statistical difference between dry and water OR values, whereas symbols show the statistical difference between dry and water RR values. Circled areas show that for the SFRC materials, only dry groups were statistically similar (*p*>0.05). GA. G-aenial Anterior; GP, G-aenial Posterior; TBF, Tetric Evo Ceram Bulk Fill; FS, Filtek Supreme XTE; FBF, Filtek Bulk Fill; VBF, Venus Bulk Fill; SDR, Smart Dentin Replacement; Z250, Filtek Z250; EXP, everX Posterior.*

rate in water (19.0 N/mm) ($p>0.05$). Alert initially performed similar to EXP but significantly weakened on storage in water ($p<0.05$) and on repair ($p<0.05$), behaving in these cases as a microhybrid PFC (FZ250). Furthermore, EXP showed statistically higher OR and RR values ($p<0.05$) compared to all PFC materials investigated in this study.

Among the repair specimens, all specimens, except EXP, broke into two halves at the interface side. This adhesive mode of failure was a sign that the weakest link was the repair interface. Following testing, the EXP specimens (100%) were not detached. This was classified as a cohesive failure, that is, ductile fracture.

SEM analyses were conducted for unbroken EXP specimens. SEM revealed protruding fibers at the interface (pulled out or broken) and bridging fibers near the notch, where the fracture initiated (Figure 3a), and bridging fibers at the middle and end part of the fracture line (Figure 3b,c).

DISCUSSION

To date, no optimal or universal repair protocol has been yet established for composite repairs.[24] Principally all factors—the monomer,[12] the composite type,[10,24] and the intermediary agent type[25]—influence the repair strength. Generally accepted is that the macro- or micromechanical roughening is superior to chemical surface treatment.[4,6,10] Nonetheless, it has been also observed that an intermediate bonding agent layer alone[1,8] or in combination with a silane agent[8] could be sufficient in repairs as well. Currently, surface grinding with a diamond bur followed by an application of a bonding agent[4] or phosphoric acid etching followed by both silane and bonding agent application[24] are recommended as the safest, most efficient repair techniques. These methods combine effectiveness and safety and are

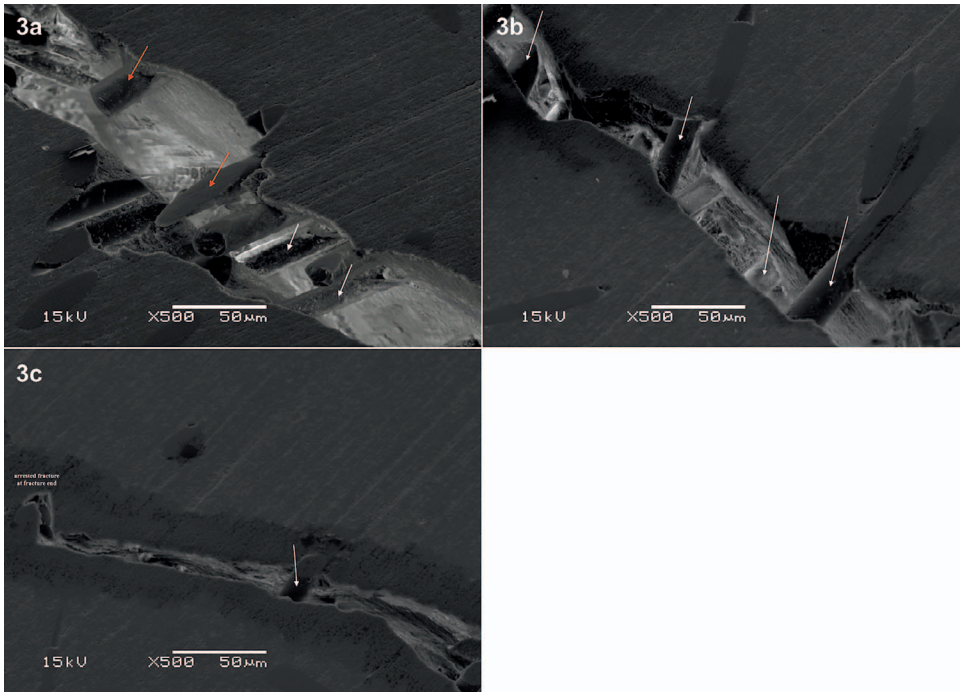

Figure 3. *(a-c): SEM micrographs showing typical fracture line segments. (a): SEM micrograph (500×) showing protruding fibers at the interface (red arrows) and bridging fibers (white arrows) at the beginning of the fracture line (near the notch). (b): SEM micrograph (500×) showing bridging fibers (white arrows) at the middle part of the fracture line. (c): SEM micrograph (500×) showing bridging fibers (white arrows) at the end part of the arrested fracture line.*

what all clinicians would most likely perform due to the simplicity of the technique. For these reasons, etching followed by an application of silane-containing universal adhesive of the previously diamond-roughened repair surface was the repair technique of choice for the purposes of the present study.

When repairing PFCs, the substrate is composed of thermoset matrix type and fillers. Fresh composite layers bond via the oxygen inhibition layer, but bonding of a new composite to aged thermoset matrix (dimethacrylate) is weak because of the cross-linked nature of the thermoset matrix.[12-14] If, however, the matrix is the semi-IPN type, the bonding possibilities during repair improve, and this is due to the secondary IPN bonding, which is possible only for the semi-IPNs.[13] More precisely, the principle of this type of bonding lies in the fact that the linear phase (polymethylmethacrylate) of the semi-IPN structure can be dissolved with monomers that have solubility parameters close to it. Examples of those are monomer solutions containing hydroxyethylmethacrylate (HEMA) or methylmethacrylate in combination with high-molecular-weight dimethacrylates.[14-16] The new resin swells and diffuses into the "old" non–cross-linked (linear) polymer matrix. On polymerization of the newly applied material, an *adhesive type* of bond, namely, the secondary IPN, is established.[13,15] In other words, the secondary IPN happens via dissolution of the semi-IPN substrate. In the present investigation, the secondary IPN was possible only for the EXP because it contains short E-glass fiber fillers embedded in a dimethacrylate resin matrix modified with polymethylmethacrylate linear chains. Moreover, the short fibers (0.85 to 1.09 mm in length[17]) protrude from the (substrate) surface and interlock with the newly applied composite layer, thus allowing a *mechanical type of bonding* for EXP. Indeed, SEM evaluation revealed protruding fibers at the fracture surface in this (Figure 3) and a previous study.[17] Consequently, because of the semi-IPN–promoted secondary IPN (adhesive bond) and the mechanical interlock (mechanical bond), a reliable repair was obtained, as observed in RR values. The results concur with findings of previous studies that illustrated the secondary IPN bonding mechanism in repairing aged semi-IPN–based continuous FRC[11] and showed that repair bond increases if resin matrix is semi-IPN.[12] To clarify, the difference between semi-IPN–based continuous FRCs and SFRCs is that continuous FRCs are used for reinforcing fixed dental appliances, whereas SFRC (EXP) is a filling material. Furthermore, protruding fibers were earlier shown to be responsible for the improved immediate shear bond strength and cohesive failure mode of EXP in the absence of an oxygen inhibition layer,[22] which was observed also in the present study (RR values). Indeed, protruding fibers allowed the ductile fracture types seen in this study, which is a sign of a durable adhesion. On the other hand, the resin matrix in Alert is thermoset, and for this reason the adhesive

type of secondary IPN bond is not achievable in repairs for this SFRC material.

In the absence of dissolvable matrix, the basis of PFCs' repair is roughening the repairing surface. Indeed, roughening the surface enlarges the bonding surface area and allows the keying with the newly applied material via the intermediate agent. This agent could be silane followed by adhesive application,[8,9,24] universal adhesive that contains silane,[8,9,26,27] or adhesive alone.[8,9] The intermediate agent wets the surface, absorbs to the roughened filler particle, and levels the surface irregularities by seeping into them.[28] Thus, it allows the interlocking mechanism between the adhesive and the repairing substrate and facilitates the diffusion of the new composite to the macromechanical porosities. It should be noted, however, that roughening alone is not an adequate surface treatment because it removes the silane layer from the exposed fillers, and the newly applied composite layer inadequately wets the surface.[29] In other words, adhesive treatment of the roughened surface enhances the repair.[28]

It should be noted that Alert has crushed fibers and that EXP has short fibers, which both already themselves enlarge the bonding surface. In addition, the areas between protruding fibers are gap-like and promote the interlocking mechanism between two successive composite layers. Consequently, short fibers behave as microretentive elements and aid better adhesion. Hence, it follows that SFRCs are naturally retentive, and in repairs two retentive forms could be achieved: *natural* microretention provided by the short fibers and macroretention *developed* by roughening the surface.

Several different types of PFCs were investigated in the current study. Although monomer types in various PFC brands are similar, there are some differences in the monomer concentrations; filler type and filler content are also different for different composite brands (Table 1). There is some evidence, however, that the presence of silica filler particles within the composite might be the key factor affecting the repair, which was related to the reactivity of the silica particles.[24] All PFCs investigated were silica based, which could explain the similar values. However, based on the accompanying filler type, materials in this study could be divided into 1) PFCs, either barium-alumino-fluoro-silicate based or zirconia-silica based, and 2) short-FRCs, both E-glass based. Thus, the differences could be explained by the variations in filler composition, content, and size. Namely, it has been shown that the etching effect depends on the filler particle composition; if the fillers are easily displaced by the etchant, as is the case with barium fillers, the possibility for silanization in re-restoration weakens because the etched surface loses fillers and matrix dominates.[30] Likewise, an efficient silanization is difficult also for prepolymerized fillers, but this is a direct consequence of their manufacturing technique.[31] The poor impregnation of the prepolymerized filler (by the resin matrix) leads to reduced mechanical properties,[31] and this was also observed in the present investigation. Namely, prepolymerized fillers containing composites (in this study GC-Anterior, GC-Posterior, and TetricEvo Ceram Bulk Fill), with ~63 vol% fillers, had similar values among them that were significantly lower than the other composites (Figure 2). This could also be attributed to the critical filler level (50 to 60 vol%) above which any further addition of filler could diminish the mechanical properties of the material.[32] Alert initially performed similar to EXP but significantly deteriorated on storage in water and on repair when behaving as microhybrid PFC (FZ250). This finding could indicate that the micrometer-scale crushed fibers acted as micrometer particulate fillers.

Furthermore, only one type of coupling agent was used in the present study. Scotchbond Universal adhesive is a prehydrolyzed silane containing adhesive with an acidic phosphate monomer 10-methacryloyloxi-decyl-dihydrogen-phosphate (MDP) and HEMA; that is, it is a phosphate-dimethacrylate–based silane-containing adhesive. It has been shown to be a good silanizer, which creates a consistent surface[27] and improves the repair strength of aged PFC substrates.[26,27] Clinically, its use could reduce the clinical steps because it eliminates the need for using separate silane and adhesive during the composite repair.[33] Furthermore, because of its low viscosity, the bonding agent could flow into the irregularities created during surface grinding and could improve bonding due to macroretention. Nonetheless, stability and effectiveness of the silane within this adhesive have been of some concern.[34] Namely, the low pH of the Scotchbond Universal adhesive might initiate premature hydrolysis and dehydration condensation, characteristics that make its silane unstable.[34] In the present investigation, the use of this agent led to an average RR of 84%, which was somewhat lower for water-stored specimens. A possible explanation could be that both acidic solution and water storage caused the hydrolysis of the silane agent that consequently led to

degradation. The aim of an intermediate layer is to make a transition interphase between the old and the new composite, but there is no consensus on whether any type of intermediate layer could be used for improving the repair interface[28] or whether the repair is bonding system dependent.[25] There was no counterpart for comparison included in this study design (group without the use of the agent), which makes further conclusions related to the use of the bonding agent inconclusive. Thus, it remains open whether another bonding agent, separate silane, or application of flowable composite would have improved the RR.

Mechanical properties of composites usually deteriorate in water,[35] which was also the case in this study. Both OR and RR deteriorated in water, but this reduction was more significant for OR than for RR. This finding could be due to the incomplete water saturation during short-term storage in water. Repaired specimens were prepared by adding new material to aged substrate, which contained absorbed water. This caused a significant decrease of RR and could be explained with the fact that repaired specimens were previously stressed and already aged. Clinically, this could mean that a repaired restoration is weaker because it was previously subjected to forces and aging conditions encountered in the oral cavity.

The interaction between the material type and the storage medium was significant for OR but not for RR. The only material that provided enhanced and almost comparable OR and RR was the semi-IPN–based SFRC (EXP). This finding indicates that while the original cohesive strength of the restorative materials is material dependent, the type of dimethacrylate-based substrate is not as important for the repair procedure. First, all repairs for dimethacrylate-based composite materials were considerably lower than their original counterparts, and, second, all failed at the repair interface. Consequently, the findings of this study reinforce the theory that the repair of dimethacrylate-based composite materials cannot be reliably achieved, whereas the semi-IPN matrix enhances the repair strength. The interaction between the material type and the storage medium was also significant only for OR. The absence of any interaction on repair could be attributed to the previously mentioned fact that the repairing substrate was already stressed and aged.

Fracture resistance (torque to failure) measured by the V-shape test is a rarely evaluated material characteristic,[23,36-38] particularly for FRCs.[38] The method was developed in 1995 by Uctasli and others, and, as the authors have stated, it resembles a clinical situation of stressing dental restoration in occlusion.[23] The V-notch is the fissure and is the side where stress concentrates, the sides of the notch are the cuspal inclines where the two-point contact occurs, and the cylindrical roller is the antagonist that generates the stress.[23] The role of the notch is to allow a sharp flaw in the material and facilitate crack initiation and propagation, which makes fracture resistance measurement more accurate. Therefore, creation of the notch is the most relevant step in this test type. The authors tested a few notch angulations and specimen dimensions and concluded that a circular 2×5-mm specimen with 90° notch angle approaches cavity size and is suitable for assessing the fracture resistance behavior of restorative materials.[23] To date, by this method, a number of PFCs were tested[23,36,37] but only one continuous FRC.[38] In the later study, unidirectional FRC was placed in different positions at the notch end, simulating placements beneath the cusp, at the fissure base or gingival side of a pontic.[38] The authors found it difficult to place the fibers in the right position in such a small mold cavity but showed that the fiber position was the most important factor affecting bulk fracture resistance. The results of the present study are consistent with the finding that fiber reinforcement enabled ductile fracture and impeded crack propagation.[38] Any difficulties with SFRC application were not experienced, however. Furthermore, accordant results were found for the mutual PFCs investigated earlier;[36,37] the small variances could be due to the different storage periods and media.

One limitation of the present study was that only one surface treatment was used. However, since other methods have not been proven to significantly recover the composite repair bond,[39] this disadvantage of the current study design is relative.

This *in vitro* study presents substantial potential of the semi-IPN–based SFRC (EXP) to improve the longevity of repaired restorations. However, only clinical studies of repaired EXP restorations could actually show the effectiveness of the semi-IPN and the millimeter-scale fibers in achieving this.

CONCLUSIONS

Within the limitations of the present investigation, it could be concluded that diamond-bur roughening followed by phosphoric acid etching and an application of silane-containing bonding agent is a simple treatment procedure and reliable repair method because the fracture resistance of the repaired

specimens was, on average, 84% of the original bulk fracture resistance.

The presence of millimeter-scale short E-glass fibers in the semi-IPN matrix enables durable bonding in composite repairs (absent an oxygen inhibition layer), and factors contributing to this are 1) the natural microretentive feature of the short fibers, 2) interface reinforcement by protruding and bridging fibers, and 3) semi-IPN–mediated secondary IPN. This is important because ductile failure and durable bonding are signs of clinical reliability of the adhesion. In other words, the longevity of the repaired restoration could be enhanced in this way.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of approval of the University of Turku, Institute of Dentistry.

Conflict of Interest

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

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Application of Calcium Silicate Materials After Acid Etching May Preserve Resin-Dentin Bonds

V Aggarwal • SS Bhasin

Clinical Relevance

Application of calcium silicate materials after acid etching can be a possible solution to preserve the resin-dentin adhesive interface.

SUMMARY

Introduction: The aim of the present study was to evaluate the effect of the application of calcium silicate materials (CSMs), after acid etching, on the longevity of the hybrid layer and marginal adaptation of composite restorations.

Methods and Materials: Eighty human permanent molars received an intrapulpal pressure of 15 cm H_2O. Sixty teeth received a mesial proximal slot preparation with the gingival margin extending 1 mm below the cemento-enamel junction. The samples were divided into two groups. Group 1 received restorations using two types of etch-and-rinse adhesives: ethanol based (Single Bond, 3M ESPE, St Paul, MN, USA) and acetone based (Prime & Bond NT, Dentsply, DeTrey GmbH, Germany). In group 2 samples, a commercially available CSM (ProRoot MTA) was allowed to set before grinding and placing into a distilled water solution. This solution was applied on the cavity floor after acid etching. The surface was washed after 30 seconds followed by application of adhesives and restorations as in group 1. The samples were stored in phosphate-buffered saline for six months, maintaining the intrapulpal pressure. An epoxy replica was made, and the marginal adaptation was evaluated using scanning electron microscopy. The percentage of continuous margin (CM) was recorded for each group. Another 20 samples were used for hybrid layer evaluation. The crowns were ground to expose dentin. Intrapulpal pressure was applied. The samples were divided into two groups and restored similar to samples restored for marginal adaptation evaluation. The samples were longitudinally cut in 1-mm slices. The slices were stored under 15 cm of phosphate-buffered saline to simulate the pulpal pressure. After six months, the adhesive interface was evaluated using a

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scanning electron microscope. Statistical analysis was done with two-way analysis of variance with Holm-Sidak's correction for multiple comparisons.

Results: Application of CSMs improved the marginal adaptation values in both adhesive groups. In group 1, there were areas of incomplete penetration of resins along with evidence of partial degradation of resin tags. Samples receiving CSM application after acid etching demonstrated long and regular resin tags with very few signs of degradation.

Conclusions: Application of CSMs after acid etching can be a potential avenue in preserving the resin-dentin bonds.

INTRODUCTION

Dental resin adhesives help in the bonding of restorations with dental hard tissues. The resin-enamel bonds are strong and durable; however, this is not true with resin-dentin bonds.[1-5] Traditional etch-and-rinse adhesives use acid etching to partially demineralize the dentin surface, exposing the collagen network.[5,6] This is followed by application of primer/adhesive, which penetrates into the spaces created by demineralization and encompasses the exposed collagen network.[6] This active zone, comprising resin adhesive and collagen fibers, is known as the hybrid layer.[3,7] The longevity of the resin-dentin bonds depends on the durability of the hybrid layer. Unfortunately, the hybrid layer is the most vulnerable area of the resin-dentin interface.[3]

Prior to primer/adhesive application, the etched dentin contains 60% to 70% water and approximately 30% collagen network.[3] In an ideal scenario, the entirety of the water content should be replaced by the penetrating resins of the dental adhesives.[3,8] However, the adhesive resins are not able to completely penetrate the full depth of demineralized dentin.[8-14] As a result, an area of uninfiltrated, demineralized dentin remains below the hybrid layer, which is composed of exposed collagen fibers. Hashimoto and others[9] demonstrated that the adhesive resins were not able to penetrate far enough to reach the depth of acid conditioning. This zone corresponded to the deposition of silver nitrate in the hybrid layer in later studies.[10-14] The exposed fibers are prone to the hydrolytic degeneration by activation of endogenous matrix metalloproteinases (MMPs) and cathepsins.[15,16]

To prevent degradation at the hybrid layer, it is desirable that hydrophobic adhesive resins should completely replace the water and penetrate to the full depth of the demineralized dentin. Various methods have been proposed to achieve this goal. Ethanol wet bonding replaced the water in the acid-etched dentin with ethanol.[14] This allowed increased penetration of hydrophobic resins dissolved in ethanol.[14] However, replacing water with ethanol is not easy and was not clinically feasible. Other methods aimed at deactivating the MMPs and cathepsins.[16]

Calcium silicate materials have been introduced in dentistry by Torabinezad and others,[17] who developed mineral trioxide aggregate (MTA). Calcium silicate materials (CSMs) are mainly composed of tricalcium silicates and dicalcium silicates with addition of other minerals.[18] The CSMs set through a hydration reaction producing calcium silicate hydrate (CSH) and calcium hydroxide.[19] The CSMs have shown an ability to remineralize the artificial demineralization.[20] When demineralized dentin slabs were placed in contact with set CSMs, zones of remineralization were reported.[20,21]

The present study hypothesized that the application of CSMs after acid etching may help to prolong the durability of the hybrid layer. The CSH particles may be able to enter the demineralized dentin, replacing and reducing the water content. Also, it may help to deactivate the proteinases by the caustic activity of alkaline pH. Composite restorations were placed with or without application of CSMs after acid etching. Hybrid layers and marginal adaptations were evaluated. The null hypothesis was that application of CSMs did not improve the resin-dentin bonds.

METHODS AND MATERIALS

The study was approved by the Institutional Ethics Committee (proposal 8/4/65/JMI/IEC/2016). The study involved the use of eighty freshly extracted, nonrestored, noncavitated human permanent molars. The teeth had similar crown dimensions. The samples were cleaned with a hand scaler and were stored in distilled water at 4°C until use. All samples were used within one week of their storage. The roots were trimmed perpendicular to the long axis of the tooth until the pulp chamber was exposed. The pulp tissue was gently removed from the chamber. A small hole was prepared in a plexiglass slab ($2 \times 2 \times 0.5$ cm) with a round bur (001/018; Mani Inc, Shioya, Tochigi, Japan). The tooth was glued to the plexiglass slab, in a manner that the hole was placed just over the pulp chamber opening. An 18-gauge needle was placed inside the hole so that it could penetrate

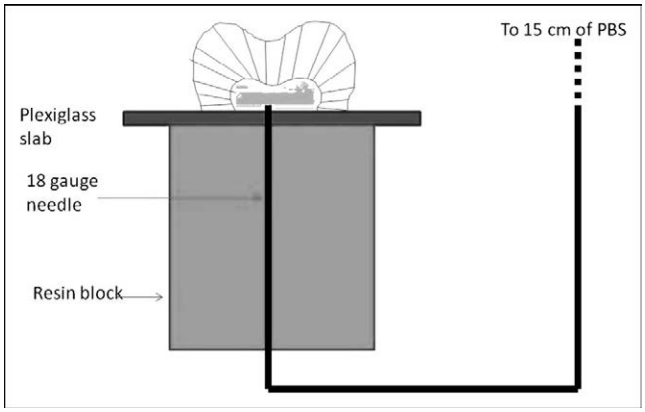

Figure 1. *Setup for simulated intrapulpal pressure. PBS, phosphate-buffered saline.*

into the pulp chamber. The tooth was sealed over the slab with the help of a cynoacrylate glue. The space between the needle and the hole was also sealed. The needle was attached to an intravenous set, which was attached to a bottle of phosphate-buffered saline. The bottle was attached to a pole and elevated 15 cm above the tooth to simulate intrapulpal pressure (Figure 1).

Sixty teeth received a mesial proximal slot preparation. The cavities were prepared using standard (125 μm) and fine (63 μm) diamond burs (straight flat end 111/014, flat end tapered fissure 172/023, and tapered conical end 160/016; Mani Inc, Tochigi, Japan) in a high-speed turbine (KaVo, Germany). The occlusal and gingival dimensions of the isthmus were kept at 3.25 ± 0.25 mm and 4 ± 0.25 mm, respectively. The gingival margin was kept 1 mm below the cementoenamel junction. Each bur was replaced with a new bur after four cavity preparations. The samples were divided into two groups on the basis of CSM application. The groups were further divided into two subgroups on the basis of adhesive system used. The schematic methodology is depicted in Figure 2.

Group 1 (Control Group)

The samples were divided into two subgroups (n=15) on the basis of resin adhesive used. Two types of etch-and-rinse adhesive were used: ethanol based (Single Bond, 3M ESPE, St Paul, MN, USA) and acetone based (Prime & Bond NT, Dentsply, DeTrey GmbH, Germany). The composition and mode of application of the adhesive systems used in this study are presented in Table 1. The light-curing unit, (Bluephase G2, Ivoclar Vivadent, Schaan, Liechtenstein) was operated at an intensity of 1,200 mW/cm^2. The power intensity was regularly checked using a dental radiometer (LM-1 Light Meter, Woodpecker, Guilin, China). After application of adhesive agent, a 1-mm layer of flowable composite (Filtek Z350 XT for the Single Bond group and Esthet-X Flow for the Prime & Bond NT group) was placed and light cured. The remaining cavity

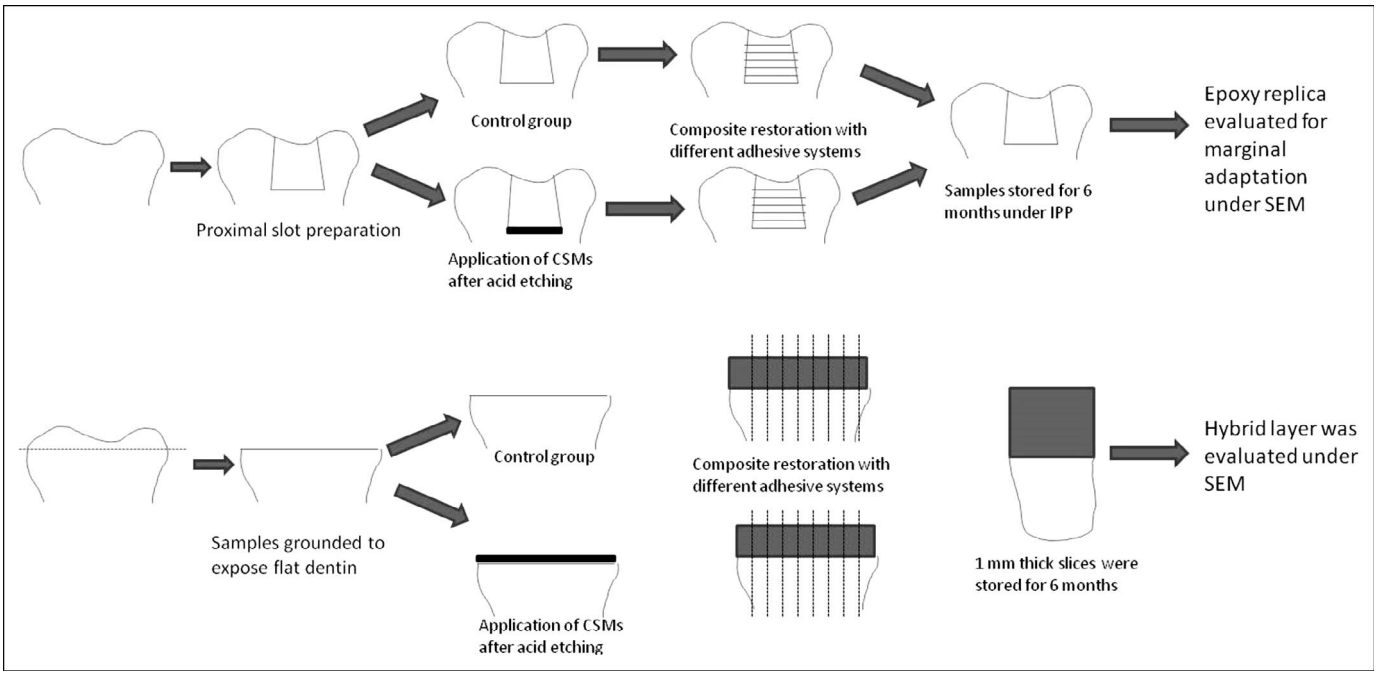

Figure 2. *Schematic methodology. CSM, calcium silicate material; IPP, intrapulpal pressure; SEM, scanning electron microscope.*

Table 1: *Composition and Mode of Application of Materials Used*

	Composition	Mode of Application
Single Bond (3M ESPE, St Paul, MN, USA) Batch No. N831690	Etchant: 35% phosphoric acid gel Adhesive: dimethacrylates, HEMA, polyalkenoid acid copolymer, 5 nm silane-treated colloidal silica, ethanol, water, photoinitiator	1. Apply etchant, wait 15 s, rinse 10 s 2. Blot excess water 3. Apply two coats of adhesive for 15 s 4. Gently air thin for 5 s 5. Light cure for 10 s
Prime & Bond NT Dentsply, DeTrey GmbH, Germany) Batch No. 1612000507	Adhesive: PENTA, TEGDMA, Bis-GMA, cetylamine hydrofluoride, acetone, nanofiller (amorphous silicon dioxide 8 nm), resin R5-62-1, T-resin, D-resin, CQ	1. Apply etchant, wait 15 s, rinse 10 s 2. Blot excess water 3. Apply two coats of adhesive for 15 s 4. Gently air thin for 5 s 5. Light cure for 10 seconds
Filtek Z350 XT flowable resin (3M ESPE, St Paul, MN, USA) Batch No. N33729	BISGMA, TEGDMA, silane-treated ceramic, EDMAB, Ytterbium fluoride	1. After application of bonding agent, 0.5-1 mm of resin applied 2. Light cure for 20 s
Esthet-X Flow (Dentsply, DeTrey GmbH, Germany)	BISGMA Adduct, BIGEMA Adduct, triethylene glycol dimethacrylate, barium floroborosilicate	1. After application of bonding agent, 0.5-1 mm of resin applied 2. Light cure for 20 s
MTA (ProRoot MTA white, Dentsply; Tulsa Dental Specialities, GmbH, Germany) Batch No. 144794	Powder: Dicalcium silicate Tricalcium silicate Bismuth oxide Liquid: Distilled water	1. Powder and liquid mixed with the help of a spatula 2. Material allowed to set for 7 d before use

was restored with a composite restorative material (Z350, 3M ESPE) in 2-mm increments using a clear matrix strip. After curing, the matrix strip was removed and gingival margins contoured with a composite polishing kit (Sof-Lex Finishing and Polishing System, 3M ESPE).

Group 2

The samples were subjected to application of CSMs before application of dental adhesive. A commercially available CSM (ProRoot MTA White, Dentsply; Tulsa Dental Specialities) was used. MTA was mixed according to the manufacturer's recommendations and was allowed to set for seven days at 100% humidity. The set MTA was crushed, and a fine powder was obtained. The cavity surface was etched with 37% phosphoric acid. The acid was washed after 15 seconds, and the cavity surface was kept moist. The set powdered MTA was mixed with distilled water, and a slurry was obtained. The slurry was copiously applied to the cavity surface and was gently rubbed for 30 seconds. The cavity surface was washed with distilled water for 30 seconds. Adhesive systems were applied, and cavities were restored as in group 1.

The samples were stored in phosphate-buffered saline for six months. The solution was changed every month. The intrapulpal pressure was maintained for the whole duration. For evaluation of marginal adaptation, the resin-dentin interface was first cleaned with 37% phosphoric acid for five seconds. Impression of the teeth was made using polyvinyl siloxane impression material (Honigum, DMG, Hamburg, Germany), and replicas were made using epoxy resin (Technovit Epox, Kulzer, Wehrheim, Germany). The replicas were mounted on aluminum stubs and were sputter coated. The gingival margins were analyzed at 65-1500× magnification in a scanning electron microscope (Zeiss, Oberkochen, Germany). The marginal adaptation was classified as "continuous margin" if the interface between the restoration and tooth was continuous and exhibited less than a 1-μm gap and as "gapped margin" if the interface had gaps more than 1-μm wide. The percentage of continuous margin (CM) was recorded for each group. Statistical analysis was performed using two-way analysis of variance (ANOVA) tests keeping the use of CSMs and type of adhesive systems as variables. The level of confidence was kept at 95%.

Evaluation of Hybrid Layer

To evaluate the resin-dentin interface, 20 teeth (n=5, four groups) were evaluated. Before application of intrapulpal pressure, the crowns of the teeth were ground to expose the dentin. The dentin surface

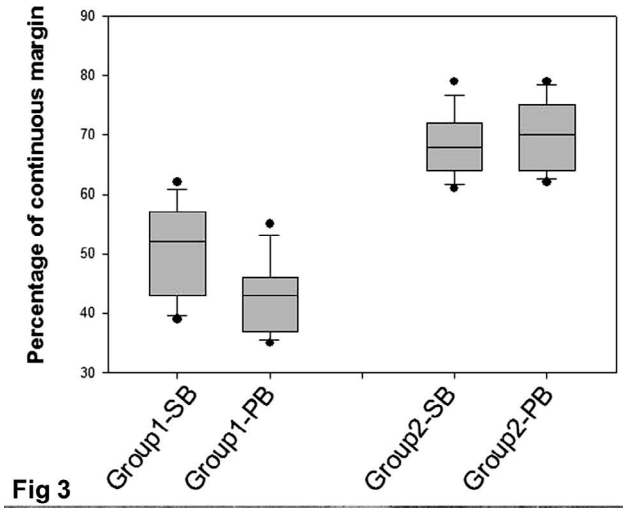

Fig 3

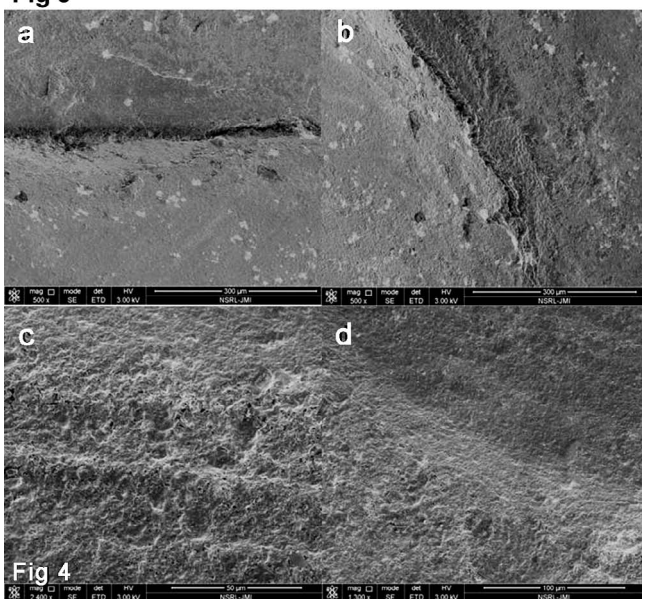

Figure 3. *Box plot graph of values of continuous margins in all groups.*

Figure 4. *Marginal adaptation in epoxy replicas at 500-2400× magnification. (a, b): Gapped margin. (c, d): Continuous margin.*

was ground on wet 600-grit SiC paper for 60 seconds. The samples were divided into two groups: the samples in the first group received acid etching, adhesive application, and restoration similar to the samples restored for marginal adaptation. A 2-mm composite buildup was done. In the second group, set MTA slurry was rubbed and washed after acid etching. The samples were restored using resin composites. The intrapulpal pressure assembly was removed, and the samples were cut into 1-mm slices to expose the resin-dentin interface. The slices were stored under 15 cm of phosphate-buffered saline to simulate the pulpal pressure. After six months, the adhesive interface was evaluated. The slices were immersed in 6N HCL for 30 seconds to demineralize the dentin, followed by rinsing with water for five minutes. Subsequently, the slices were immersed in 3% NaOCl for 10 minutes and were again rinsed with water for five minutes. The specimens were dehydrated and were mounted on aluminum stubs for sputter coating and scanning electron microscope analysis.

RESULTS

There was a significant difference between the values of percentages of CM obtained in different groups; hence, the null hypothesis was rejected ($p<0.05$, two-way ANOVA with Holm-Sidak's correction for multiple comparisons; Table 2). The overall marginal adaptation values in group 1 were 46.6% (Figure 3). There was a significant difference between the CM values of Single Bond (50.25±7.7%) and Prime & Bond NT (43%±6%) subgroups ($p=0.002$). Figures 4a,b show the marginal adaptation in the subgroups of group 1. A gross deterioration of the resin-dentin interface was noticed in most slices. Gaps as wide as 25 μm were noticed. Application of CSMs after acid etching significantly improved the CM values. Overall, the CM values were 69.2%, with no difference between the two subgroups (68.4%±5.2% and 70%±5.5% for Single Bond and Prime & Bond NT, respectively). Figure 4c,d shows some CMs in group 2. There was significantly less marginal deterioration as compared with group 1.

Five samples from each group were evaluated for resin tags and hybrid layer quality. A consistent hybrid layer was noticed in all groups. The thickness of the hybrid layer was 4-7 μm. In group 1, two types of defects were noticed: 1) areas of incomplete penetration of resin denoted by irregular diameter and length of the resin tags (Figure 4a,b) and 2) partial degradation of resin tags suggesting hydro-

Table 2: *Two-Way ANOVA*

Source of Variation	Degree of Freedom	Sum of Squares	Mean Squares	*F*	*p*
Application of CSM	1	7638	7638	199	<0.001
Different adhesive systems	1	120	120	3.1	0.082
Variable 1 vs variable 2	1	286	286	7.4	0.008

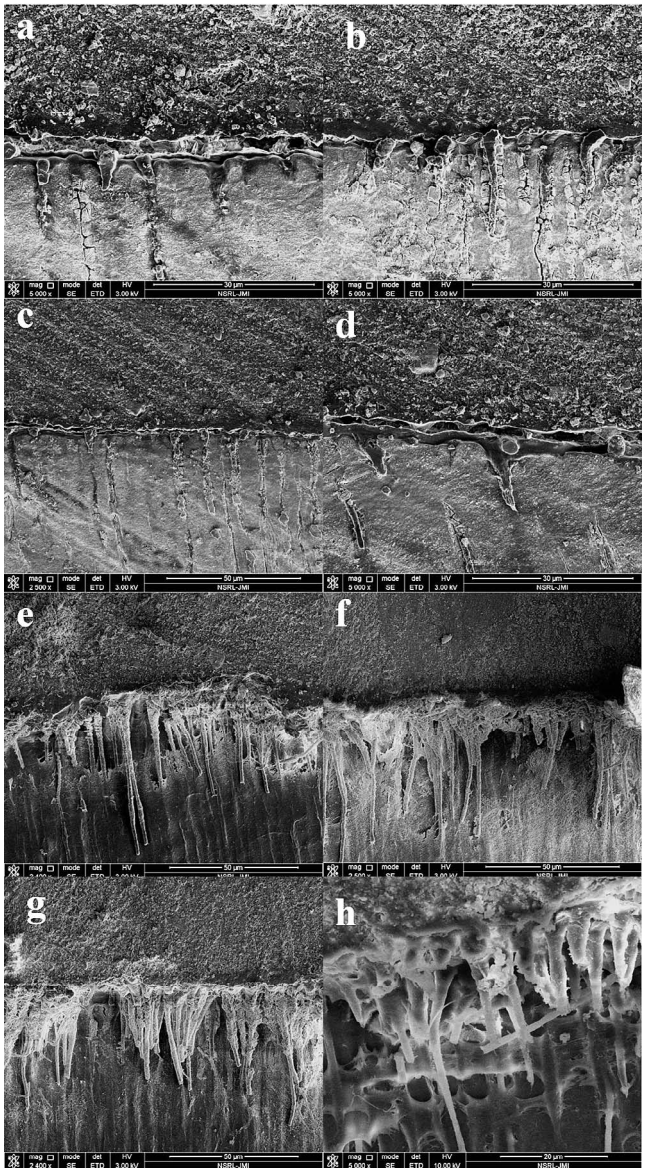

Figure 5. *Hybrid layer evaluation at 2500-5000× magnification. (a-d): Group 1 showing areas of incomplete resin penetration and partial degradation of resin tags. (a, c): Adhesive bond failure. (e-h) Long/ regular resin tags, with minimal sign of degradation.*

lytic degradation of resins (Figure 5a-d). Bond failure was seen at many places, with most of them being adhesive in nature (failure at the junction of the hybrid layer and dentin). In some groups, partial degradation of the hybrid layer, near the dentinal junction, was seen. Samples receiving CSM application after acid etching demonstrated long and regular resin tags with length ranging to tens of microns. The tags were initially funnel shaped and then subsequently tapered until they took a long, cylindrical form. The length and the diameter of the resin tags were consistent with very few signs of degradation (Figure 5e-g). Lateral branching of tags was noticed in some specimens (Figure 5h).

In group 2 specimens, deposition of particles was seen inside the dentinal tubules. An energy dispersive X-ray analysis (EDAX) was performed to ascertain their chemical composition. The analysis showed the presence of calcium, silicate, aluminum, and traces of chloride, phosphorous, and carbon (Figure 6).

DISCUSSION

To simulate intrapulpal pressure, the specimens received 15 cm of water pressure. The dentin is primarily a hydrated structure with dentinal fluid present in the dentinal tubules.[22,23] In a clinical scenario, the blood pressure inside the pulp chamber pushes the dentinal fluid outward toward the cavity floor. This impedes the penetration of resin into the dentinal tubules.[24] It is recommended to simulate the intrapulpal pressure in studies evaluating the resin-dentin interface. Application of simulated intrapulpal pressure has shown to reduce the length of resin tags and negatively affect the tensile bond strength.[24,25] Various authors have used pressures ranging from 5 cm to 36 cm of water.[25-27] Ciucchi and others[23] estimated the pulpal tissue pressure and reported a positive pulpal pressure of 14.1 cm of water in healthy teeth. An infiltration anesthesia with solutions containing epinephrine reduced the pulpal blood flow by 72%.[28] Purk and others[25] reported that application of different amounts of pulpal pressure (5 cm H_2O and 15 cm H_2O) reduced the microtensile bond strength compared with specimens restored without pulpal pressure. However, increasing the pulpal pressure from 5 cm to 15 cm had no effect on the bond strengths.[25]

Two-step etch-and-rinse adhesive systems were used in the present study. The systems differed on the basis of solvents. Prime & Bond NT was acetone based, while Single Bond used ethanol and water as solvents.[29] Acetone has a high vapor pressure and water-removing capacity, often termed a *water-chasing* ability.[29,30] However, while using acetone-based adhesives, care must be taken to evaporate the excess water before light curing the adhesive.[29] The ethanol-based systems usually contain water as a cosolvent. Combined with water, the ethanol solution is azeotropic in nature.[29] In comparing acetone- vs ethanol-based adhesive systems, the literature provides different results. Tay and others[31] observed a "complex phase separation pattern" in specimens restored using an acetone-based adhesive, in the presence of surface moisture. Transmission electron

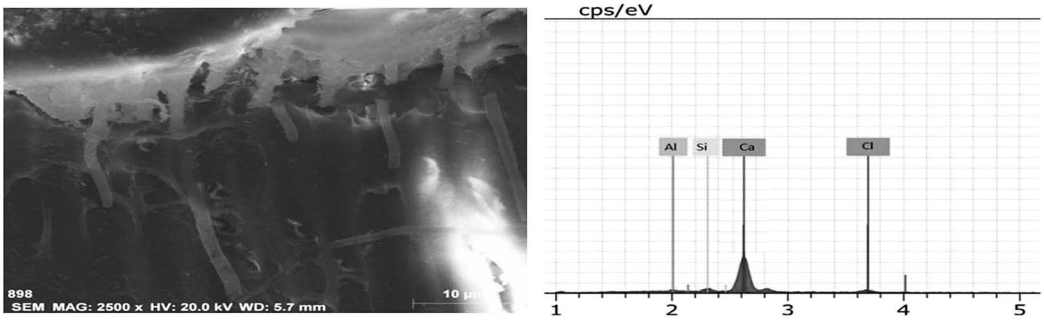

Figure 6. *EDAX analysis of particles deposited inside the dentinal tubules in group 2. The analysis showed presence of calcium, silicate, aluminum, and traces of chloride, phosphorous, and carbon.*

microscope examination revealed that in the presence of excess moisture, the intratubular resin infiltration was compromised.[31] The ethanol-based adhesive performed better in some *in vitro* studies.[24,25] The microtensile bond strength was higher than acetone-based adhesive in the presence of intrapulpal pressure. However, some studies have observed that a pressure of 15 cm H_2O does not compromise the bond strength of acetone-based adhesives.[32] The water-chasing ability was thought to be a reason for the better resin penetration despite perfusion through dentin. In the present study, the Single Bond (ethanol-based) adhesive presented with better marginal adaptation than the Prime & Bond NT (acetone-based) adhesive. The marginal adaptation was evaluated using an epoxy replica of the specimen. This method gives a reliable quantitative assessment of the marginal quality, in terms of continuous or gapped margins.[33,34] Since the specimens have to undergo a vacuum process during scanning electron microscopy, the use of epoxy replica rules out the possibility of formation of cracks, which can be formed on actual teeth if placed in a vacuum chamber.[34]

In both of the adhesive systems, there was a degradation of the hybrid layer. Hashimoto and others[35] observed two patterns of hybrid layer degradation: disorganization of collagen fibers and hydrolysis of resins. In the present study, hybrid layer and resin tags were evaluated after storage of slices for six months. In the present study, there were areas with incomplete penetration of resin into the dentinal tubules along with evidence of degradation of resin tags. This degradation was seen in both adhesive systems. Since the bonded interface was exposed by sequential rinses in hydrochloric acid and sodium hypochlorite, most collagen was removed and resin tags were exposed. Several mechanisms have been proposed to explain the degradation of the hybrid layer. During acid etching, the subsurface mineral is solubilized and extracted.[3] This part gets replaced with water, raising the water content of etched dentin to 70%.[3] In an ideal scenario, this water is replaced by the resin monomers. However, the resins are not able to fully replace the water. This condition gets worse by application of intrapulpal pressure that forces the fluid into dentinal tubules. This leads to presence of local water-rich regions within the hybrid layer, which can be identified as nanoleakage. The presence of water can cause hydrolysis of resins in the hybrid layer.[36,37] The second mechanism of the degradation of the hybrid layer relates to the activation of MMPs.[15] The MMPs are bound to the healthy dentin. During acid etching, the MMPs are released and activated.[15] These enzymes break the collagen fibers by virtue of their collagenase and gelatinase activity. As explained before, the resin does not penetrate fully to the depth of demineralization, exposing some collagen fibers surrounded by water.[8] These exposed fibers are attacked by the MMPs. Another mechanism relates to the presence of proteolglycan hydrogels, which interfere with the infiltration of resins.[16]

It is quite clear that any mechanism that protects the collagen fibers and restricts the movement of fluid inside the hybrid layer will make the adhesive interface more durable.[3] It was hypothesized that application of calcium silicate materials, after acid etching, will help to preserve the resin-dentin interface. The CSMs have many applications in dentistry because of their bioactive properties.[19] The first commercially available CSM was MTA, which consists of calcium silicates and aluminates along with bismuth oxide as a radiopacifier.[18] When set calcium silicate material was exposed to simulated body fluid for seven days, a layer of hydroxyapatite was formed over the CSM.[38] Tay and others[39] reported that when set Portland cement was immersed in phosphate-buffered saline, an

amorphous calcium phosphate (ACP) phase was formed that transformed into an apatite phase. These ACP precursors are subnanometer sized and have a potential to act as prenucleation clusters for the development of oriented apatite crystals.[40]

A commercially available CSM (ProRoot MTA) was used in the present study. The material was allowed to set for seven days and then finely ground. The set material was gently rubbed over the acid-etched dentinal surface. A pilot study revealed that using unset MTA over the dentin surface prevents resin infiltration since it tends to set within minutes of coming in contact with water. The marginal adaptation was better than the samples restored without CSM application. The hybrid layer evaluation revealed that the resin tags were long, parallel in shape, and had minimal signs of degradation. The hybrid layer was intact in most places. There was evidence of deposition of particles inside the dentinal tubules. EDAX analysis revealed that the particles had a chemical composition of calcium, aluminium, and silicate with traces of chloride, phosphorous, and carbon. Gandolfi and others[41] evaluated the effect of application of CSMs on the dentinal discs treated with EDTA and reported that the dentinal tubules were covered with particles having a chemical composition of calcium and phosphate and traces of aluminum, silicate, chloride, and magnesium.

As explained before, a major cause of degradation of the adhesive interface is the presence and flow of water through the dentinal tubules. Application of CSMs have been shown to reduce the permeability of dentin.[41] They also help to provide a precursor for apatite formation that helps to remineralize the dentin. This may also preserve the collagen fibers. The hydration of CSMs yield two main components: calcium-silicate-hydrate and calcium hydroxide.[18,19] When the set CSMs are in contact with phosphate-containing solutions, the calcium-silicate-hydrate leads to precipitation of ACP that transforms into carbonated apatite.[39] The calcium hydroxide has an alkaline pH, which can denature the enzymes.[42] It is possible that application of CSMs could have reduced the activity of MMPs by virtue of their alkaline pH.

There are a few limitations to the present study. The resin-dentin margins were not scanned before the aging process. This could have helped us in understanding the ill effects of aging on the resin-dentin interface. However, this phenomenon has been reported extensively in the literature. The aim of the study was to develop a strategy to negate this ill effect. Some researchers have raised doubts about the validity of scanning electron microscopy analysis. However, scanning electron microscopy has been a backbone of the research conducted on the analysis of resin-dentin interface.[1,2,6,7,8,10,14-16,27,28]

CONCLUSIONS

Application of CSMs after acid etching can be a possible solution to preserve the rein-dentin adhesive interface. Further studies, involving microtensile bond strength evaluation and future clinical trials, are required to evaluate the long-term effect of CSMs on demineralized dentin, and if promising results are obtained, a commercial formulation can be developed.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of approval of the Jamia Millia Islamia Institutional Ethics Committee. The approval code for this study is 8/4/65/JMI/IEC/2016.

Conflict of Interest

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

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Chemical Interaction and Interface Analysis of Self-Etch Adhesives Containing 10-MDP and Methacrylamide With the Dentin in Noncarious Cervical Lesions

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Clinical Relevance

The chemical interaction and morphology at the interface of self-etch adhesives and the dentin of noncarious cervical lesions depend on the functional monomer present in the adhesive. This fact is essential to evaluate the requirement for additional substrate preparation before commencing adhesive procedures.

SUMMARY

Objectives: To characterize the chemical interactions and analyze the interface of adhesive systems containing 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) and N-methacryloyl glycine (methacrylamide) functional monomers with the dentin in noncarious cervical lesions (NCCLs) compared with artificial defects (ADs).

Methods and Materials: Twenty human teeth with natural NCCLs on the buccal surface were used. Class V cavities, similar to NCCLs, were created on the lingual surface to serve as

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controls. Teeth were randomly allocated to two groups according to the functional monomer in the adhesive (N=10): G1, 10-MDP; and G2, methacrylamide. NCCLs and ADs were characterized by their mineral composition (MC) and degree of demineralization (DD) using micro-Raman spectroscopy, adhesive/dentin chemical interactions (CIs) were assessed with infrared photoacoustic spectroscopy, and interface morphology was evaluated with scanning electron and light microscopy. MC, CI, and DD data were submitted to Shapiro-Wilk and Student *t*-tests ($p<0.05$).

Results: Compared with ADs, dentin in NCCLs was hypermineralized ($p<0.05$). In G1, CI, and DD in the first 2 μm, and adhesive projections in NCCLs and ADs interfaces were similar. Additionally, a thin layer of dentin collagen was observed in ADs, while it was hardly present in NCCLs. In G2, although CI could not be identified, changes in the mineral components were observed. The DD in the ADs and NCCLs were statistically similar, while SEM showed a lack of adhesion at NCCLs interface. DD and collagen exposure in the ADs and NCCLs were more pronounced than in G1.

Conclusions: Results suggest that the G1 adhesive could be applied directly on the superficial sclerotic layer in NCCLs. In contrast, previous cavity preparation should be conducted to improve the micromechanical interaction of G2 with the dentin.

INTRODUCTION

Noncarious cervical lesions (NCCLs) form a group of lesions difficult to characterize in the dental practice because of their multifactorial etiology.[1,2] NCCLs result from the slow and progressive loss of mineralized dental structure caused by the association of different phenomena such as erosion, abrasion and abfraction.[3]

Laboratory studies have demonstrated that adhesion to dentin affected by NCCLs may lead to adhesive failures and compromise the longevity of restorations.[4-6] The main reason for this phenomenon is the molecular/chemical structural changes that occur at the interface, which result in less favorable adhesion to the substrate.[7] Because the dentin in NCCLs is sclerotic, the formation of a hybrid layer in the dentin/adhesive interface is compromised by irregular primer diffusion and reduced adhesive infiltration.[8]

Although the main adhesive mechanism to the dental substrate is based on the micromechanical retention resulting from the formation of a hybrid layer and resin tags, attention has recently been focused on the benefit of additional chemical interactions between the functional monomers present in adhesive systems and the components of the dental substrate.[9-12]

Chemical interactions can occur through ionic bonds established by acid monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) that react with the hydroxyapatite, forming monomer-Ca salts that are stable to degradation.[9,10] On the other hand, the interaction of adhesive systems containing methacrylamide can result in bonds with dentin collagen fibrils through the additional reactive groups present in monomeric acids. Since dentin collagen contains reactive groups such as amino or hydroxyl, the aldehyde or anhydride groups of the adhesive system can establish covalent bonds with collagen fibrils.[13]

The aim of this study was to analyze the chemical interactions of two self-etching adhesive systems, one containing the 10-MDP and the other methacrylamide functional monomers with the dentin in NCCLs and artificial defects (ADs) so that we could evaluate the requirement for additional substrate preparation before undertaking adhesive procedures.

METHODS AND MATERIALS

This *in vitro* study was approved by the local ethics committee (CAAE: 47305015.7.0000.0104). Teeth with natural NCCLs extracted for periodontal or orthodontic reasons were used. All teeth presented grade 4 of dentin sclerosis, according to the scale modified by Ritter and others.[14] Grade 4 is attributed to NCCLs with significant presence of sclerosis, in which the dentin is dark-yellow or brownish with a petrified appearance, significant translucency, or evident transparency.

Specimen Preparation

A total of 20 teeth with natural NCCLs located in the cervical region of the buccal surface were used in the experiment. They were randomly divided into two groups (N=10); G1, to be restored with an adhesive system containing 10-MDP, and G2, to be restored with an adhesive system containing methacrylamide. After extraction, the teeth were cleaned with sterile gauze and saline solution. Any remaining periodontal tissues were removed with the aid of

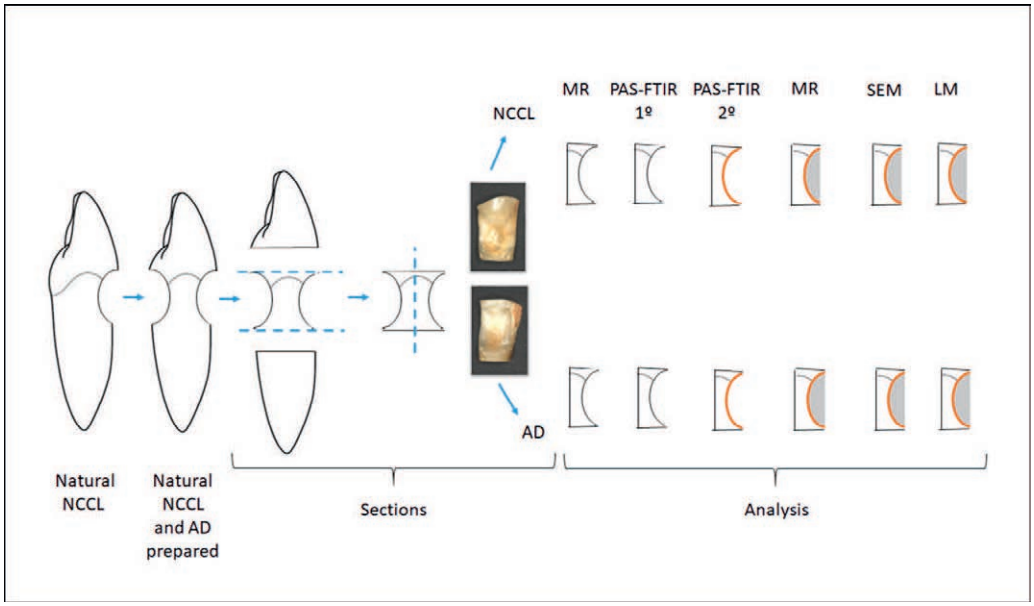

Figure 1. *Diagram illustrating preparation of the specimens and analysis sequence.*

periodontal curettes. After cleaning, the teeth were stored in saline at 4°C.

Artificial defects (ADs) in the shape of Class V cavities were created in the lingual surface of the same tooth with a CVDentus (C1, 1.0 × 4.0 mm) cylindrical diamond tip coupled with an ultrasound device (CVDent 1000, CVDVale, São Carlos, Brazil) under continuous water cooling. ADs were prepared in sound dentin with dimensions and shape approximately the same as those of the corresponding NCCL, serving as a control.[15]

Then, dental specimens containing the NCCLs and ADs were obtained from each tooth. The teeth were sectioned with a diamond disk at low speed under water cooling in the following sequence: first, just above the lesion to remove the crown, and then, just below the lesion to remove the remaining two-thirds of the root. Finally, a section was made along the long axis of the tooth to separate specimens containing the NCCLs from the ADs. Once the dental specimens were obtained, they were ready to be employed in the sequence of analyses described below (Figure 1).

Adhesive System Application

The composition of the adhesive systems used in this study and the recommended mode of application are described in Table 1. Light-curing was done with the Translux Power Blue unit (Heraeus Kulzer, Hanau, Germany) at 1000 mW/cm^2. The light-curing time for each adhesive system was according to the manufacturer's instructions.

Dentin Mineral Composition

Mineral composition analysis of the dentin in ADs (control) and NCCLs was performed with micro-Raman (MR) spectroscopy. The analysis was conducted with a micro-Raman spectrometer (Bruker Optik GmbH, Ettlingen, Germany), equipped with a Senterra confocal microscope, whose operation is based on infrared light scattering, ie, the source of light irradiation (laser with invisible wavelength) excites the studied matter. In this interaction, the Raman effect is obtained, which allows studying vibrations at the molecular level.

Specimens' spectra were measured at three different points on the dentin surface. All measurements were collected at a resolution of 4 cm^{-1} in the spectral region between 3500 and 450 cm^{-1}. Each spectrum was obtained from an average of 60 readings to decrease the signal-to-noise ratio, with a laser wavelength of 785 nm, power of 100 mW, and objective gain of 100×. In addition to the high number of readings, the signal pattern was improved by decreasing detector temperature to −84°C. In all readings, the surface area selected for measurement, the support mirror used, and manual focusing followed by autofocusing were performed following the same standardized procedures.

All spectra were placed on the same baseline and normalized with the aid of Opus spectroscopy soft-

Table 1: *Composition and Mode of Application of Adhesive Systems Used in the Experiment*

Group	System	Composition	Application Mode
G1	Clearfill SE Bond 2 (Kuraray Noritake Dental Inc., Tokyo, Japan)	*Primer:*	Apply primer for 20 s
		MDP	Gently air dry for 5 s
		HEMA	Apply adhesive
		Dimethacrylate	Photopolymerize for 10 s
		Camphorquinone	
		Water	
		Adhesive:	
		MDP	
		Bis-GMA	
		HEMA	
		Dimethacrylate	
		Camphorquinone	
		Initiators	
		Accelerators	
		Silanized colloidal silica (pH 2)	
G2	Xeno V+ (Dentsply Sirona, York, PA, USA)	Bifunctional acrylate	Actively apply adhesive for 20 s
		Acrylate acid	Gently air dry for 5 s
		Esters of phosphoric acid	Photopolymerize for 10 s
		Water	
		Tertiary butanol	
		Initiators	
		Stabilizers	
		(pH 1.38)	

Abbreviations: MDP, 10-methacryloxydecyl dihydrogen phosphate; Bis-GMA, bisphenol-A Bismethacrylate; HEMA, 2-hydroxyethylmethacrylate.

ware (Bruker Optics, Ettlingen, Germany). Additionally, origin software (OriginPro 8 Corp, Northampton, MA, USA) was used to obtain numerical quantifications of MR spectra by integrating each curve of the band at 961 cm^{-1} (phosphate) to calculate the respective area and mean of the three distinct points measured in the sound dentin of the AD specimens and in the sclerotic dentin of the natural NCCLs.

Adhesive/Dentin Chemical Interactions

Specimens' spectra were measured with Fourier transform infrared photoacoustic spectroscopy (FTIR-PAS) both before and after being submitted to the adhesive treatment. The technique provides the optical absorption bands of the sample, which are considered the fingerprint of specific molecules. Information on the chemical modifications within the specimen is expressed by means of changes and/or emergence of new peaks.

The experiments were performed with a Nicolet Spectrometer (MTEC Photoacoustics, Ames, IA, USA) equipped with a MTEC 200 photoacoustic cell model. This equipment allows monitoring the absorption of the substance of interest at specific specimen depths, providing the distribution profile of the substances along the thickness studied. All spectra were collected at a resolution of 8 cm^{-1}, with scanning speed of 0.5 cm/s. The spectral region of the measurements lies in the energy range between 4000 and 400 cm^{-1}. After the specimen was inserted, the photoacoustic cell was filled with helium to minimize interference on the optical absorption spectra of oxygen and water molecules present in the air and on the surface of the specimen.

To determine the depth of the analysis in the present experimental condition, FTIR-PAS test measurement depth was defined by the thermal diffusion length. The thermal diffusivity of the adhesive was measured by the thermal lens technique[16] as described by Oliveira and others.[15] The inspection depth of the technique for the readouts taken in this study was about 6 μm for G1 and 4 μm for G2. Since the adhesive film depth can range from 2-3 μm,[17,18] this technique enabled reading not only the hybrid region, but also the dentin under the adhesive.

To evaluate the spectrum of the adhesive system, a disc of pure adhesive was prepared, applying 1 mL of

the material on a histological glass slide. After photoactivation for 20 seconds, the disc was inserted into the measuring equipment. This is an important step to differentiate the composition of the adhesive from that of the dental structure and to verify the differences between photoacoustic absorption peaks of the adhesive and dentin.

Collected data were transferred to the origin software. Graphs were generated for each tooth individually, from which the average spectrum of ADs and natural NCCLs dentin specimens were calculated. Bands identified as chemical interactions between the dentin and the adhesive system were selected, and the respective intensities in the ADs and natural NCCLs before and after the application of the adhesive were compared.

Degree of Demineralization

After FTIR-PAS analysis, all cavities were filled with composite resin (Filtek Z250, 3M ESPE, St Paul, MN, USA) and subsequently sectioned for MR scanning analysis. Dental specimens were cut longitudinally with a sectioning machine (Isomet 1000 Precision Saw, Buehler, Lake Bluff, IL, USA) using a diamond disc (Diamond Wafering Blade, Series 15LC, Arbon size ½, 12.7 mm, Buehler) under water cooling.

MR spectra were obtained by scanning the inner region of the composite resin toward the deeper layers of the dentin in NCCLs and ADs. All spectra were obtained under the same conditions as previously described for mineral composition analysis. Raman spectra were acquired at 1-μm intervals in 20-μm long lines at a distance of 10 μm between lines. Scans were individually analyzed, and spectra reading presenting any flaws, possibly due to the presence of bubbles, were excluded from the analysis. All spectra were placed on the same baseline and normalized with the aid of the Opus spectroscopy software, and analyses were conducted with the origin software. Degree of demineralization (DD), as a function of location, was determined at the bands at 961 cm^{-1} (PO_4 of the dentin) in relation to the band at 1458 cm^{-1} (CH_2), according to the equation:

$$DD = \left(1 - \frac{961cm^{-1}intensityinterface/1458cm^{-1}intensityinterface}{961cm^{-1}intensitydentin/1458cm^{-1}intensitydentin}\right) \times 100\%$$

Interface Morphology

Analysis of the adhesive system/dentin substrate interface morphology was performed with scanning electron microscopy (SEM). Sample treatment was performed according to the protocol suggested by Monticelli and others.[19] After MR analysis, the samples were embedded in acrylic resin and polished with a wet sandpaper sequence (220, 400, 600, 1200, 1800, 2000 grit). Then the specimens were submitted to demineralization with 37% phosphoric acid for 10 seconds, deproteinization with 2% sodium hypochlorite for 1 minute, and dehydration with 100% alcohol for 2 minutes in an ultrasonic vat (Bio-Free-Gnatus, Ribeirão Preto, Brazil) and air jets. Specimens were sputter-coated with gold and evaluated with SEM (Shimadzu, Model SS-550 Superscan, Kyoto, Japan) with a magnification of 1000×.

Collagen Fiber Exposure

To examine collagen fiber exposure at the adhesive system/dentin interface, four additional teeth were prepared according to the protocol described above, up to the application of the adhesive system, and histologically analyzed under light optical microscopy.

Immediately after application of the adhesive system, samples were fixed in Karnovsky's solution (2.5% glutaraldehyde and 2% paraformaldehyde in 0.1 mol/L sodium cacodylate buffer, pH 7.3) for 48 hours and washed in running water for 4 hours. The samples were placed in a decalcifying solution (20% sodium citrate and 50% formic acid) for 25 days. After that, the specimens were washed in running water for 4 hours, dehydrated in an increasingly concentrated alcohol sequence, and embedded in paraffin. Histological sections, 6 μm thick, were serially made using a tungsten carbide blade coupled with a microtome (Leica RM2265, Leica Microsystems, Wetzlar, Germany).

Goldner-modified Masson Trichrome[19-21] was used to stain the specimens. Before staining, the samples were prepared according to the protocol proposed by Wang and Spencer.[20] The sections were first stained with Weigert's iron hematoxylin solution for 5 minutes, immersed in Masson solution for 10 minutes, and rinsed twice in 0.2% acetic acid solution. Afterward, they were kept in mordant solution for 5 minutes and washed again with 1% acetic acid. Finally, they were stained with a light green solution for 5 minutes and rinsed twice with 0.2% acetic acid. For sample dehydration, the sections were immersed in 96% and 100% alcohol twice for 1 minute. Specimens were immersed twice in xylol for 3 and 5 minutes and mounted within histological slides. Sections were examined and photographed at a magnification of 100× under a light optical microscope (Olympus BX41, Tokyo, Japan).

Statistical Analysis

Statistical analysis was performed using the R i386 3.0.2 software (R statistical software, R Foundation for Statistical Computing, Vienna, Austria). The areas of the band at 961 cm^{-1} (PO_4) for the phosphate present in the dentin, the intensities related to the chemical interactions found with FTIR-PAS, and DD were submitted to the Shapiro-Wilk and Student *t*-test ($p<0.05$).

RESULTS

Adhesive/Dentin Chemical Interactions

Figure 2A shows the spectrum of the pure adhesive system containing the 10-MDP monomer (G1) with its organic and inorganic functional groups: methacrylate monomer (carbonyl C=O [1720 cm^{-1}], CH_2CH_3 [1457 cm^{-1}]), BIS-GMA (C=C [1638 cm^{-1}], C-O-C [1140 cm^{-1}], $[CH_3]_2$-C [1300 cm^{-1}], C_6H_4 [840 cm^{-1}]), and load (SiO_2 [1105 cm^{-1}]). Figure 2B shows the dentin spectra of the natural NCCLs and ADs. The bands associated with mineral and organic composition were observed at the following wavenumbers: phosphate (1179 cm^{-1}), amide I (1650 cm^{-1}), amide II (1550 cm^{-1}), amide III (1240 cm^{-1}).

Figure 3A and B illustrate the NCCL spectra before and after application of the adhesive system containing the 10-MDP monomer (G1). Circles and bars indicate changes in spectra, suggesting chemical interactions between the dentin and the adhesive system. Table 2 shows the bands and functional groups identified as possible chemical interactions between the dentin and the adhesive system containing 10-MDP monomer and the means and standard deviations of the intensities obtained from the ADs and natural NCCLs characterizing chemical interactions.

Although no statistically significant differences were observed between the two groups, mean values for natural NCCLs in all band intensities were numerically higher than those of ADs.

Figure 4A shows the photoacoustic absorption spectra of the pure adhesive containing the methacrylamide monomer (G2), with the following functional groups: CH_2 (1456 cm^{-1}), CH_3 (1373 cm^{-1}), PO_2 (1265 cm^{-1}), CH (1139 cm^{-1}), C-O-C (1040 cm^{-1}), and Al-OH (920 cm^{-1}). Figure 4B shows the dentin in the natural NCCLs before and after application of the adhesive system, suggesting a change in the inorganic components such as phosphate and calcium. However, no indication of chemical interactions can be observed.

Dentin Mineral Composition

Means and standard deviations of the measured spectra obtained from the integrated areas at the band at 961 cm^{-1} (PO_4) on the surface of the ADs and natural NCCLs are shown in Table 3. The Student *t*-test showed that the mineral content in dentin in natural NCCLs was significantly higher than that found in the ADs ($p<0.05$).

Degree of Demineralization (DD)

MR scanning analysis of the natural NCCLs and ADs with the adhesive systems used are represented by Figures 5 and 6. It can be observed that, in G1, the behavior in natural NCCLs (Figure 5A) and ADs (Figure 5B) was similar. However, in G2, the dentin in the ADs (Figure 6A) underwent deeper demineralization than did the natural NCCLs (Figure 6B).

Means of the DD (%) of the dentin using the systems G1 and G2 are presented in Table 4. The DD in G1 had similar behavior in the first 2 μm of the hybrid layer, whereas from 3 μm, a statistically significant difference ($p<0.05$) between the ADs and natural NCCLs was observed. In G2, DD presented no statistically significant differences between ADs and natural NCCLs.

Interface Morphology

Scanning electron microscopy analysis showed differences in the adhesive system/dentin substrate interface between G1 and G2 (Figure 7). In G1, images demonstrated similar projections of the adhesive within the demineralized dentin in ADs (Figure 7A) and natural NCCLs (Figure 7B), although the sclerotic aspect of the dentin with greater mineralization can be identified in the natural NCCLs. In G2, when applied to ADs (Figure 7C), the methacrylamide-containing adhesive provided greater inter- and peritubular demineralization, with well-defined projections within the dentin. On the other hand, the interface in the natural NCCLs (Figure 7D) was indefinite and more obliterated because of the higher degree of mineralization, compromising the formation of a hybrid layer due to irregular primer diffusion and reduced adhesive infiltration, leading to failure of the restoration (indicated by the arrow in Figure 7D).

Collagen Fiber Exposure

Photomicrographs of the samples stained with Goldner's Modified Masson's Trichrome are shown in

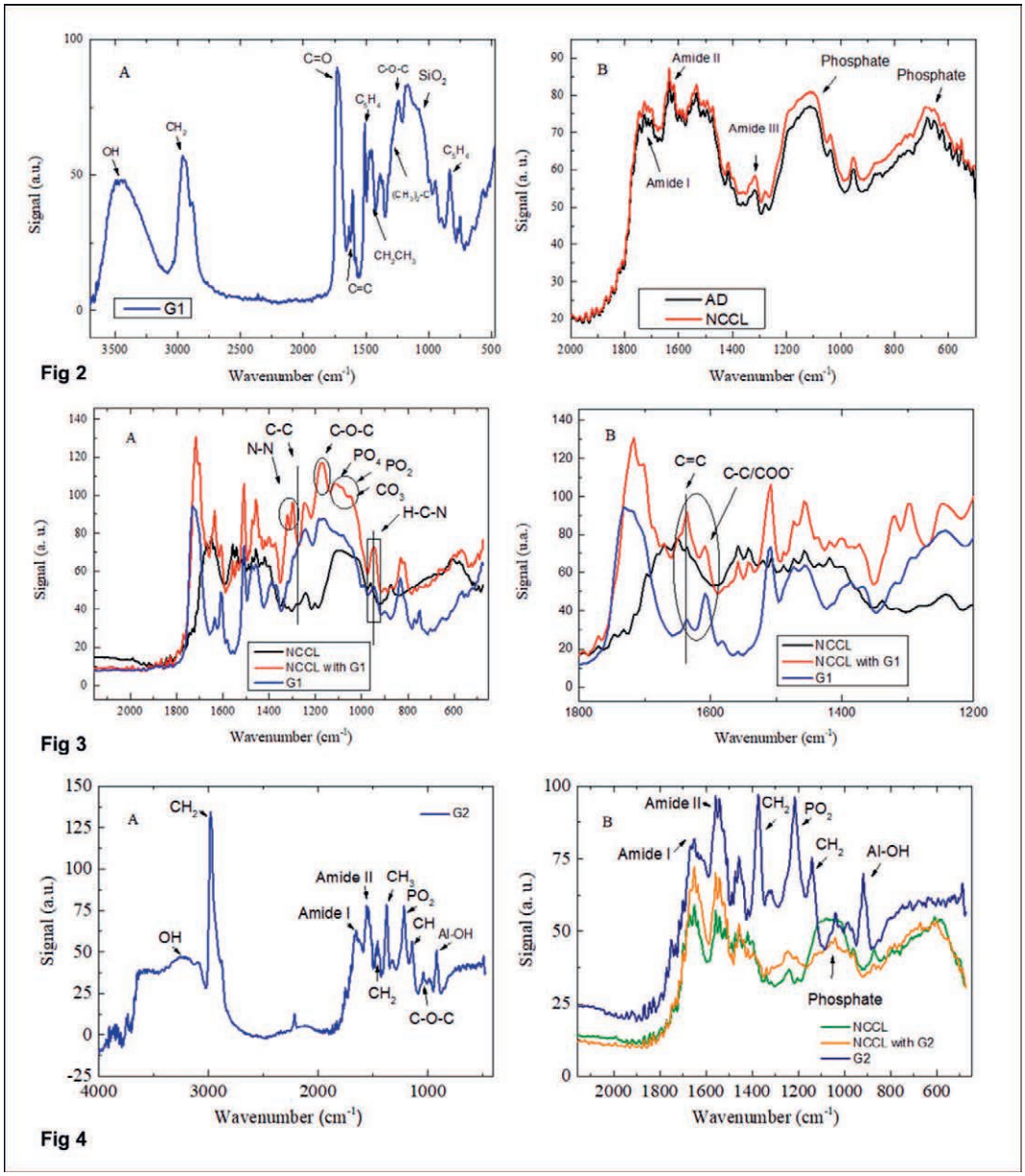

Figure 2. *Photoacoustic absorption spectra: (A) pure adhesive system containing 10-MDP (G1), and (B) dentin in ADs and natural NCCLs.*

Figure 3. *Photoacoustic absorption spectra obtained from the adhesive and the NCCL before and after application of the adhesive system containing the 10-MDP monomer (G1). (A) Spectral region 2100 to 550 cm^{-1}, and (B) spectral region between 1800 and 1200 cm^{-1}.*

Figure 4. *(A) Photoacoustic absorption spectra obtained from the pure adhesive containing the methacrylamide monomer (G2), and (B) NCCL before and after application of the adhesive system. Arrow indicates structural modifications in the region of the inorganic component of dentin.*

Figure 8. The dentin appears stained in green, the exposed unprotected collagen is evidenced in red/pink, while pure adhesive is unstained.[22,23]

In G1, histomorphological analysis demonstrated a thin layer (pink) of exposed collagen in the dentin of the ADs (Figure 8A). In the natural NCCLs (Figure 8B), collagen was hardly evident. In G2, the photomicrographs illustrate deeper demineralization of the dentin and more pronounced collagen exposure (dark red) in the ADs (Figure 8C) as well as natural NCCLs (Figure 8D).

DISCUSSION

Self-etching adhesive systems with various functional monomers were used in this study in an attempt to

Table 2: *Means and Standard Deviations (SDs) of the Intensities of the Bands That Characterize Chemical Interactions in ADs and Natural NCCLs With the Adhesive Containing the 10-MDP Monomer (G1)*

Assignment	Band	Group	Mean	SD	p*
C=C	1635 cm^{-1}	AD	60.82	3.83	0.25
		NCCL	63.24	3.8	
C-C/COO^-	1608 cm^{-1}	AD	49.66	3.67	0.19
		NCCL	52.47	3.92	
N-N stretching	1323 cm^{-1}	AD	59.86	6.22	0.52
		NCCL	62.45	8.26	
C-C stretching	1295 cm^{-1}	AD	64.28	7.23	0.58
		NCCL	66.82	9.55	
C-O-C	1169 cm^{-1}	AD	76.7	8.67	0.58
		NCCL	79.93	12.34	
PO_4	1112 cm^{-1}	AD	68.94	7.86	0.41
		NCCL	72.77	9.23	
PO_2 symmetric stretching	1081 cm^{-1}	AD	67.4	7.55	0.36
		NCCL	71.56	8.77	
CO_3	1045 cm^{-1}	AD	64	6.73	0.25
		NCCL	68.52	7.4	
H-C-N bending	946 cm^{-1}	AD	49.26	5.07	0.36
		NCCL	52	5.67	

** Student t-test ($p<0.05$).*
Abbreviations: AD, artificial defect; NCCL, noncarious cervical lesion

Table 3: *Means and Standard Deviations of the Integrated Areas of the Band at 961cm^{-1} (PO_4) of ADs and Natural NCCLs in arbitrary units (a.u.)*

Group	Mean (a.u.)	Standard Deviation	p
ADs	297,364.6	43,571.92	0.002*
NCCLs	387,726.7	80,574.17	

** Student t-test ($p<0.05$)*

identify strategies that might increase the longevity of esthetic restorations in natural NCCLs.

Controversy on the best strategy to restore NCCLs still exists. A fairly recent systematic review of the literature failed to find sufficient evidence to support a particular adhesive system or adhesive strategy for the restoration of natural NCCLs,[24] highlighting the necessity of investigating the adhesive interface in these situations.

In the present study, MR analysis showed that the areas corresponding to the phosphate band (961 cm^{-1}) in the dentin of ADs and NCCLs were statistically different, clearly demonstrating that the dentin in NCCLs was more mineralized than in ADs. This analysis was performed in our study with the objective of confirming the presence of sclerotic dentin and ensuring that specimens with natural NCCLs were in similar conditions. These findings corroborate previous studies that evaluated the molecular and structural differences in the mineral/organic components in the dentin of natural NCCLs and ADs using MR[7,15] and FTIR-PAS.[25]

It has already been demonstrated that the 10-MDP functional monomer present in G1 is capable of forming chemical bonds with the hydroxyapatite (calcium salts-MDP), improving the adhesion and longevity of restorations.[10,15,26,27] In the present study, FTIR-PAS analysis demonstrated that the peak intensities related to the chemical interactions (Table 2) in ADs were similar to those in natural NCCLs. The DD (Table 4) of natural NCCLs and ADs was also similar for the first 2 μm of the hybrid layer, becoming statistically different below the depth of 3 μm. However, SEM images (Figure 7A,B) demonstrated a similar behavior in terms of adhesive infiltration, despite the higher degree of dentin mineralization in natural NCCLs.

Histomorphological photomicrographs of the adhesive systems tested in this study revealed the presence of exposed and unprotected collagen (Figure 8). For the formation of an ideal hybrid layer, collagen should be fully protected by the monomer in the adhesive system, preventing any collagen labeling.[23] Studies show that fully exposed collagen is marked by a strong red color, and when partially coated by the adhesive, is marked by lighter colors.[22] In the present study, it is possible to observe a thin layer of light red/pink collagen in G1, indicating that the adhesive partially enveloped the demineralized dentin in the ADs (Figure 8A). In the natural NCCLs, the system was able to encapsulate collagen more closely to the ideal and was very little evidenced by the dye (Figure 8B).

The molecular/chemical alterations of the hypermineralized sclerotic dentin in NCCLs may result in a less favorable substrate for adhesion.[7] Some studies advocate removal of the superficial sclerotic layer to increase intratubular retention.[5,28-30] However, according to Tay and Pashley,[8] removal of the hypermineralized layer in NCCLs may not increase adhesive strength, since the sclerotic dentin might still contain crystals capable of preventing the infiltration of the adhesive into the dentinal tubules. Another systematic review failed to determine any differences in survival rates

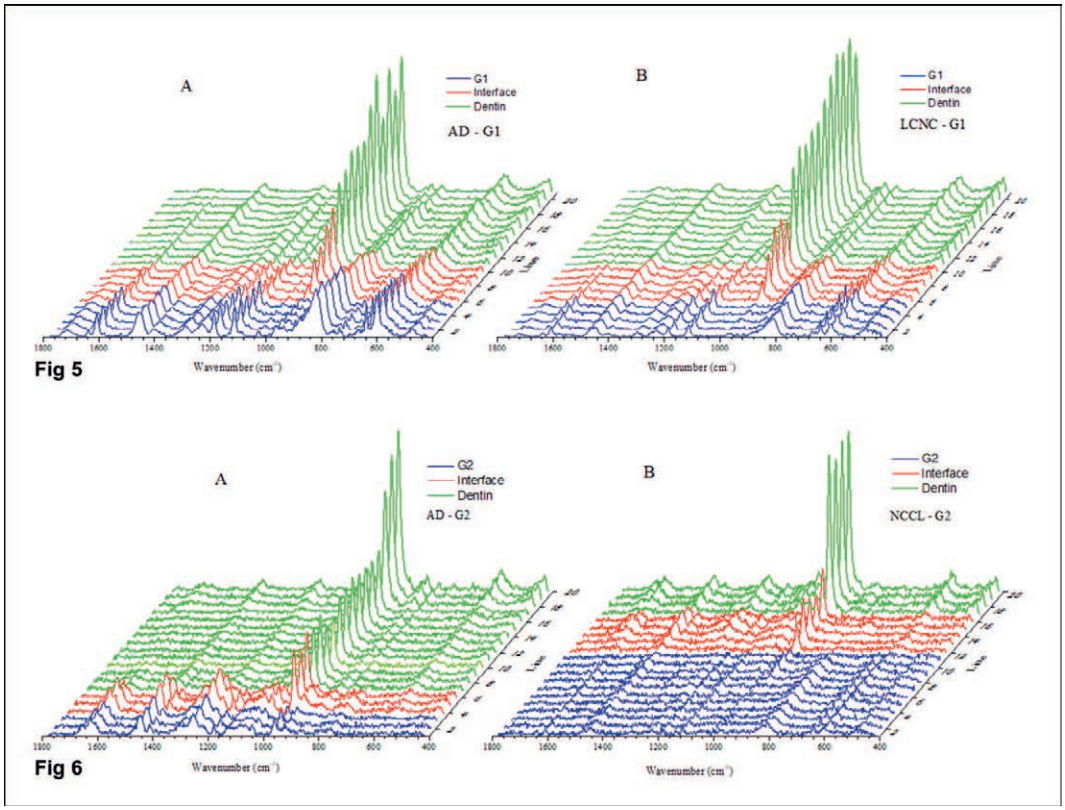

Figure 5. *MicroRaman spectra of the dentin/adhesive system interface in G1: (A) artificial defects (ADs), and (B) natural noncarious cervical lesions (NCCLs).*

Figure 6. *MicroRaman spectra of the dentin/adhesive system interface in G2: (A) artificial defects (ADs), and (B) natural noncarious cervical lesions (NCCLs).*

because of the small number of studies comparing the influence of dentin roughness on the retention of restorations in NCCLs.[31]

However, Luque-Martinez and others[32] demonstrated that the adhesive strength of self-etching systems containing 10-MDP in unprepared sclerotic bovine dentin was superior to the same surface prepared with diamond burs. Corroborating these findings, Oliveira and others,[15] using human teeth with natural NCCLs, also observed that adhesion of a self-etching system containing 10-MDP was stronger in natural NCCLs than in ADs, probably due to the chemical affinity of the monomer with the hydroxyapatite.

The results of the present study suggest that the adhesive system containing 10-MDP functional monomer (G1) can be applied directly on the superficial sclerotic layer in NCCLs since the intensity of chemical interactions and the degree of demineralization of natural NCCL and ADs are similar. This perception is reinforced by the fact that G1 was able to involve collagen in natural NCCLs more completely. As a

Table 4: *Degree of Demineralization (%) in ADs and NCCLs in Groups G1 and G2*

Group	Dentin	Degree of Demineralization							
		1 μm, %	*p*	2 μm, %	*p*	3 μm, %	*p*	4 μm, %	*p*
G1	AD	93	0.557	91	0.242	87	0.005*	81	0.016*
	NCCL	94		87		75		70	
G2	AD	91	0.514	91	0.515	89	0.202	83	0.076
	NCCL	89		87		80		67	

** Student t-test ($p<0.05$).*

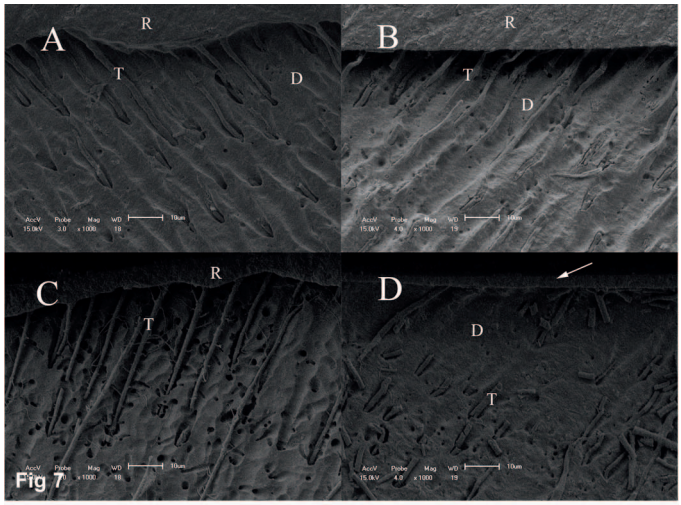

Figure 7. *The dentin/adhesive interface observed with scanning electron microscopy: (A) G1 in AD, (B) G1 in natural NCCL,* ***(C)*** *G2 in AD, and* ***(D)*** *G2 in natural NCCL. Arrow in Figure 7D indicates restoration failure. R: restoration; T: tags; D: dentin.*

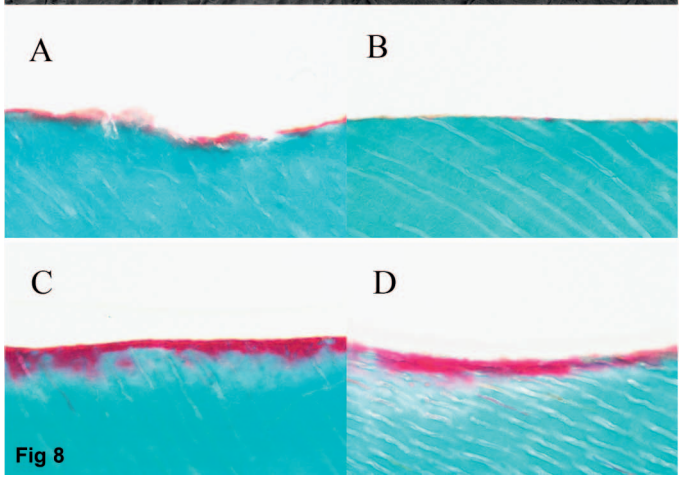

Figure 8. *Photomicrographs representative of natural NCCLs and ADs stained with Goldner's modified Masson's Trichrome, showing the dentin (green) and exposed collagen (red/pink): (A) G1—AD, (B) G1—natural NCCL, (C) G2—AD, and (D) G2—natural NCCL.*

result, apart from promoting unnecessary dental structure wear, surface preparation could actually hinder adhesion because of the presence of debris.

The methacrylamide monomer present in the G2 system interacts with the collagen present in the dentin,[33] showing high hydrolytic stability of the amide portion[34] as well as the ability to demineralize the dentin,[35] providing good long-term adhesive strength in healthy dentin compared with methacrylate-based adhesive systems.[36]

In G2, MR analysis demonstrated that the DD in ADs (Figure 6A) was probably caused by the high demineralizing power of the system (pH 1.38) and the absence of a smear layer. In contrast, in natural NCCLs, the demineralization was less intense due to the hypermineralized characteristic of the surface (Table 4). SEM images (Figure 7C,D) confirmed these findings and demonstrated adhesive failure in the NCCL restoration. A clinical study using the Xeno Select adhesive system, which has the same functional monomer in G2, demonstrated that the adhesive was not able to fulfill the ADA criteria for the restoration failure rate of less than 5% after 6 months of clinical performance in natural NCCLs.[37]

In the natural NCCLs of G2, the DD in the first 4 microns of the hybrid zone was similar to that of the ADs (Table 4). Furthermore, FTIR-PAS analysis demonstrated changes in the dentin spectra after adhesive application. These modifications suggest a change in the inorganic components, such as phosphate and calcium, but do not indicate the occurrence of chemical interactions (Figure 4). Zhou and others,[38] when testing adhesives with 10-MDP (Clearfil S3 Bond), 4-META (GBond), and methacrylamide (Xeno V) in sound and deproteinized dentin, also observed that the -C=C-COO- chemical bonds could not be identified in the spectrum of the methacrylamide-based adhesive. The absence of a signal in the infrared spectrum of the Xeno V adhesive group indicated low affinity with the

dentin. The band at 1718 cm^{-1} of Xeno V was much weaker than that of the other adhesives, which may explain the absence of a signal after being applied to the dentin surface.[38]

In the present study, a thick band of dark red collagen could be observed in the histomorphometric analysis of specimens in G2, indicating that the adhesive system demineralized dentin at a greater depth than in G1, both in the ADs and natural NCCLs. Additionally, they revealed that the functional monomer did not involve the collagen in either dentin substrates tested with G2. This result confirms the FTIR-PAS and SEM findings, which also demonstrated that no hybrid layer was optimally formed to provide the expected chemical interaction between the collagen in the natural NCCLs and the ADs with G2.

Thus, the results of the present study demonstrated that infiltration of the methacrylamide-based adhesive in the NCCLs was limited because of the presence of a more obliterated dentin, compromising hybrid layer formation at the interface. Considering that in the present study, G2 demineralized the dentin more deeply in the ADs than did the natural NCCLs, we recommend that the superficial sclerotic layer be removed by means of cavity preparation to provide micromechanical interaction and effectively bond to the dentin in the NCCLs.

In vitro studies, which attempt to simulate actual clinical conditions, present some important limitations. Despite that, the cavities were tested in a paired way, ie, the same tooth received the control cavity on the surface opposite the natural NCCLs to allow direct comparison between groups and avoid possible differences in the permeability between teeth, which could interfere with the results obtained from the dentin in NCCLs and ADs. Further studies testing adhesive systems with various chemical compositions and dentin surface preparation modes are required to enable dentists to increase the longevity of esthetic restorations in natural NCCLs.

CONCLUSIONS

The use of a self-etch adhesive system in NCCLs requires different substrate preparation strategies according to the functional monomer present in its composition. Because the G1 adhesive containing the 10-MDP monomer was shown to react chemically with the mineral component present in the sclerotic dentin, it can be applied directly to the surface of NCCLs. On the other hand, the G2 adhesive containing methacrylamide demonstrated that the superficial sclerotic layer of NCCLs needs to be removed before adhesive application in order to obtain improved micromechanical interaction.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of approval of the local permanent research ethics committee. The approval code for this study is CAAE: 47305015.7.0000.0104.

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, or company presented in this article.

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Linear Coefficient of Thermal Expansion Evaluation of Glass Ionomer and Resin-Modified Glass Ionomer Restorative Materials

G Pinto-Sinai • J Brewster • H Roberts

Clinical Relevance

Conventional glass ionomer materials overall exhibit linear coefficient of thermal expansion (LCTE) similar to tooth structure, while some resin-modified glass ionomer materials have LCTE similar to that reported for resin restorative materials.

SUMMARY

Objective: The purpose of this evaluation was to evaluate the linear coefficient of thermal expansion (LCTE) of 12 conventional glass ionomer (GIC) and four resin-modified glass ionomer (RMGI) restorative materials.

Methods: GIC and RMGI specimens (2 mm × 5 mm × 5 mm) were fabricated (n=12) following manufacturer instructions and were placed in 0.2M phosphate-buffered saline and stored at 37°C and 98% humidity for one week. Specimens had LCTE determined with a thermomechanical analysis (TMA) unit using a 15°C-50°C heating cycle as well as a 50°C-15°C cooling cycle at a 5°C/min rate, using a 3-mm ball-point probe under 0.02 N probe pressure with all specimens kept saturated with PBS using a specially designed quartz container. Each specimen was tested three times, with the mean representing the specimen LCTE. Mean results between specimen heating and cooling were compared with paired Wilcoxon sign rank test, while results between materials were compared with Kruskal-Wallis/Dunn's (α=0.05).

Results: GIC LCTE ranged from approximately 5°C to 20°C ppm $^{\circ}K^{-1}$, while the RMGI LCTE ranged from approximately 25°C to 47°C ppm $^{\circ}K^{-1}$. With some exception, the LCTE during cooling displayed a greater trend.

Significance: Under moisture conditions similar to the oral cavity, GIC materials overall had LCTE values closer to that reported for tooth structure. RMGI materials displayed higher values, which was thought to be related to the amount of resin in the matrix. A generally greater LCTE trend with cooling for all materials was noted, but the small magnitude of the difference is presently thought to be of minor clinical significance.

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INTRODUCTION

The oral environment is subject to many challenges during daily function. Temperature changes fluctuate due to the ingestion of food and liquid, with one review reporting *in vivo* temperature ranges that varied as low as 0°C and as high as 70°C.[1] Restorative materials placed into this environment may show thermal expansion or contraction in response to these thermal changes. A high degree of difference in the thermal expansion characteristics between restorative materials and tooth structure may cause interfacial stress development, which has been implicated as one of the etiological factors in marginal deterioration and microleakage.[2-4]

Linear coefficient of thermal expansion (LCTE) determination methods have included thermochemical analysis,[5] thermomechanical analysis combined with moiré interferometry,[6] both beta radiation and theta dilatometry,[7,8] strain gauges,[9] and x-ray diffraction.[10] LCTE analysis of some conventional glass ionomer (GIC) and resin-modified glass ionomer (RMGI) restorative materials have been reported.[11-14] However, studies to date have investigated only a limited number of products, and evaluation conditions were not under intraoral humidity conditions. Furthermore, these investigations have not investigated material LCTE when subjected to a controlled cooling challenge. The purpose of this investigation was to investigate the thermomechanical properties of GIC and RMGI restorative materials using both heating and cooling challenges. The null hypothesis was that there would be no differences between materials and/or temperature challenge results.

METHODS AND MATERIALS

The GIC materials evaluated are listed in Table 1, with the RMGI products evaluated in Table 2.

Both GIC and RMGI specimens (2 mm × 5 mm × 5 mm) were fabricated using a polyvinylsiloxane mold. Materials were triturated/prepared and carefully injected into the mold and covered with a polyethylene strip and glass slide with digital pressure to prepare a flat surface. GIC materials were allowed to set for the recommended finishing time, while RMGI specimens were photoactivated for 20 seconds on both sides using a visible light curing unit (Bluephase G2, Ivoclar-Vivadent, Amherst, NY, USA). All manufacturer recommendations were followed. Specimens (n=12) were immediately placed in a 98% ± 2% 0.2M phosphate-buffered saline (PBS) environment at 37°C for one week under dark conditions. At the appointed testing time, specimens were placed in a thermomechanical analysis (TMA) unit (TMA841E, Mettler Toledo, Columbus, OH, USA) fitted with a 3-mm-diameter ball-point probe that maintained 0.02 N pressure against the specimen. During analysis, the specimens were maintained under PBS moisture using a specially designed quartz container (Figure 1) that was placed inside the TMA unit. The quartz container was subjected to the TMA thermal protocol (eg, "blank run") so that any effect from the container would be automatically subtracted from the TMA specimen results.

Figure 1. *Quartz container on TMA platform showing immersed sample.*

Specimens were subjected to a thermal challenge protocol that is graphically depicted in Figure 2.

The protocol consisted of an initial five-minute hold at 15°C to allow the sample to thermally equilibrate. The temperature was then raised to 50°C at a rate of 5°C/min, which was followed by a second five-minute hold to allow thermal stability. The specimen was then subjected to a cooling challenge back to 15°C at 5°C/min. LCTE was determined using the slope of each respective thermal challenge, with each specimen tested three times, with the mean representing the LCTE of each specimen. The Shapiro-Wilk and Bartlett's test identified discrepancies in both the data distribution and variance. Mean results between heating and cooling for each sample were compared with the Wilcoxon signed rank test, while results between materials were compared with Kruskal-Wallis and Dunn's post hoc test, with all analysis performed at a 95% level of confidence (α=0.05).

RESULTS

The LCTE results are listed in Table 3.

Table 1: *GIC Restorative Products*

Material	Manufacturer	Powder/Liquid Ratio, g/g	Powder Content	Liquid Content[a]
Chemfil Rock Capsules	Dentsply, Sirona Int. (York, PA, USA)	[b]	Polycarboxylic acid 10%-25%	Polycarboxylic acid 10%-25% Tartaric Acid 2.5-10%
Equia	GC America (Alsip, IL, USA)	0.40/0.12	Trade secret	Trade secret
Equia Forte	GC America	0.40/0.13	Trade secret	Trade secret
Fuji Triage Capsules	GC America	0.30/0.15	Trade secret	Trade secret
Ketac Fil Plus	3M ESPE (St Paul, MN, USA)	[b]	Trade secret	Acrylic acid–maleic acid copolymer 35%-55% Tartaric acid 5%-10% Water 45%-55%
Ketac Molar Quick Aplicap	3M ESPE	[b]	Oxide glass chemicals (nonfibrous) 85%-95% Copolymer of acrylic acid–maleic acid 1%-5% Dichlorodimethylsilane Reaction Product with Silica <2%	Water 60%-65% Copolymer of acrylic acid–maleic acid 30%-40% Tartaric acid 10%
Ketac Silver Aplicap	3M ESPE	[b]	Silver 45%-55% Oxide glass chemicals 40%-50% Titanium dioxide 1%-5% Copper <0.01	Water 40%-60% Copolymer of acrylic acid–maleic acid 30%-50% Tartaric acid 5%-15%
Ketac Universal	3M ESPE	[b]	Glass, oxide, chemicals >95%	Acrylic acid–maleic acid copolymer 30%-50% Tartaric acid 1%-10% Benzoic acid <0.2%
Riva Protect Fast Capsules	SDI Limited, (Bayswater, Victoria, AUS)	0.34/0.19	Fluoro aluminosilicate glass 90% Polyacrylic acid 10%	Polyacrylic acid 25% Tartaric acid 10%
Riva Self Cure Fast Capsules	SDI Limited	0.40/0.15	Fluoro aluminosilicate glass 90%-95% Polyacrylic Acid 5-10%	Polyacrylic acid 20%-30% Tartaric acid 10-15%
Riva Self Cure High Viscosity Capsules	SDI Limited	0.50/0.13	Fluoro aluminosilicate glass 90%-95% Polyacrylic Acid 5-10%	Polyacrylic acid 20%-30% Tartaric acid 10-15%
Riva Silver Capsules	SDI Limited	0.72/0.14	Fluoro aluminosilicate powder 40%-60% Polyacrylic acid <10% Alloy powder 30%-50%	Polyacrylic acid 30% Tartaric acid 10% Balance ingredient 60%

[a] *Remaining percent is assumed to be water*
[b] *Not available from manufacturer information; content information obtained from manufacturer information.*

Equia Forte demonstrated the lowest LCTE value, followed by Riva SC HV, Ketac Silver, Ketac Universal, Ketac Fil, and Fuji Triage. The RMGI products demonstrated higher LCTE values, with Ketac Nano having the highest LTCE value, followed by Riva LC, Riva LC HV, and Fuji II LC, all of which were statistically similar. In comparing heating and cooling LCTE, except for Riva Protect Fast, all materials demonstrated greater cooling LCTE. Chemfil Rock, Ketac Fil, Ketac Molar Quick, Ketac Silver, Riva SC Fast, and Riva Silver were the only materials that did not significantly differ from the heating challenge.

DISCUSSION

This study evaluated the thermomechanical characteristics of 11 GIC and four RMGI restorative materials. The determination of LCTE direct restorative materials is considered relevant as a large difference in the LCTE characteristics between interfaces may produce stress that could cause marginal deterioration and microleakage.[2-4] The LCTE of ceramic/metal-ceramic layers also must closely be matched to prevent interfacial stress development that may be the etiology of defects.[15-19]

Previous TMA studies for different materials have used protocols involving different ranges. Spierings

Table 2: *RMGI Restorative Products*

Material	Manufacturer	Powder/Liquid Ratio, g/g	Powder Content	Liquid Content
Fuji II LC Capsules	GC America (Alsip, IL, USA)	0.33/0.010	Trade secret	HEMA 25%-50% Polybasic carboxylic acid 5%-10% UDMA 1%-5% Dimethacrylate 1%-5%
Ketac Nano Quick Mix Capsules	3M ESPE (St Paul, MN, USA)	N/A	Paste A: Silane-treated glass 0%-55% Silane-treated zirconia 0%-30% PEGDMA 5%-15% Silane-treated silica 5%-15% HEMA 1%-15% BISGMA <5% TEGDMA <1 %	Paste B: Silane-treated ceramic 40%-60% Copolymer of acrylic and itaconic acids 20%-30% Water 10%-20% HEMA 1%-10%
Riva LC Capsules	SDI Limited (Bayswater, Victoria, AUS)	0.42/0.14	Fluoroaluminosilicate glass powder 95%-100%	Polyacrylic acid 15%-25% Tartaric acid 1%-5% HEMA 20%-30% Dimethacrylate cross-linker 10%-25% Acidic monomer 10%-20%
Riva LC HV Capsules	SDI Limited	0.47/0.14	Fluoroaluminosilicate glass powder 95%-100%	Polyacrylic acid 15%-25% Tartaric acid 1%-5% HEMA 15%-25% Dimethacrylate cross-linker 10%-25% Acidic monomer 10%

Abbreviations: BISGMA, bisphenol A diglycidyl ether dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; PEGDMA, polyethylene glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.
[a] Content information obtained from manufacturer information.

and others reported a 14°C to 56°C range[20]; Versluis and others, 26°C to 75°C[9]; Bullard and others, 5°C to 55°C[2]; Sideridou and others, 0°C to 80°C[12]; Sidhu and others 25°C to 50°C[11] Tezvergil and others, 23°C to 160°C[21]; Vaidyanathan and others, 30°C to 70°C[22]; Hashinger and Fairhurst, room temperature to 120°C[23]; and Kwon and others, 20°C to 80°C range.[24] Thermal conditions during intraoral function have been reported to occur over a wide range of temperatures, reported from −5°C to 76°C.[1,25,26] However, it has been suggested that the mean maximum intraoral temperature is approximately 46°C with fluids and 41°C with solid food.[27] As for the minimum range of intraoral functional temperatures, approximately 15°C was suggested by Youngson and Barclay,[28] and 0°C was reported by Palmer and others.[29] In addition, the reported TMA temperature change rate has also been variable, with values being reported of 10°C min^{-1},[3,10,13,20,24] 5C min^{-1},[11] 3°C min^{-1},[22] and 1°C min^{-1}.[23] The temperature range chosen for this study was 15°C to 50°C at a rate of 5°C min^{-1} and was based on the minimum temperature suggested by Youngson and Barclay[28] and the higher temperature slightly above the temperature suggested by Feuerstein and others.[27] The temperature change rate was chosen as a slow rate is recommended (1°C to 5°C min^{-1}) to allow sufficient heat penetration through the sample to prevent errors due to temperature lag. [30-32]

With restorative resins, LCTE has been reported to be influenced by the amount of filler contained in the resin.[21-23] Under the conditions of this study, the RMGI materials demonstrated higher LCTE values than the GIC materials. With the exception of Ketac Nano, the RMGI products do not contain fillers per se, and the LCTE in glass ionomer restorative materials may be influenced by the amount of resin contained within the matrix. Although tooth material LCTE have been accomplished with essentially 100% humidity[8] and GIC materials in a moist (65%) environment,[3] this study is the first, to the authors' knowledge, that has tested GIC materials totally immersed in PBS. Although this resulted in an environment with higher humidity than the range reported for the human oral cavity (78%-94%),[33] immersion in physiologic solution allowed a reproducible testing environment, as maintaining stable humidity values is difficult within the TMA device.

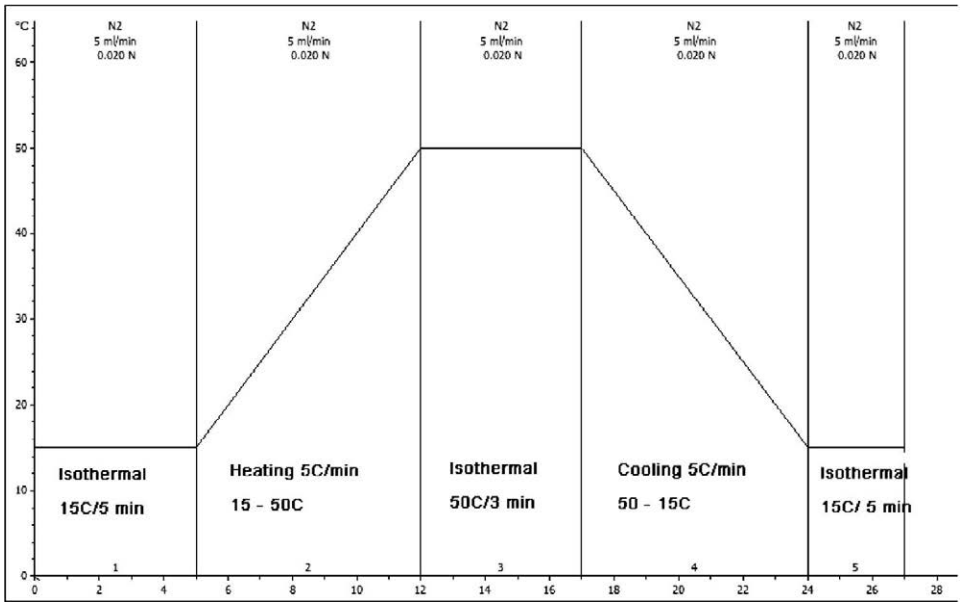

Figure 2. *TMA regimen.*

With the exception of Chemfil Rock and Riva Protect Fast, the GIC materials demonstrated a mean LCTE that was lower than 15 ppm $°K^{-1}$. The results of this study differ from those earlier reported for GIC restorative materials. Both Sidhu and others[10] and Yan and others[3] reported that GIC materials demonstrated shrinkage during thermal challenge, but these results did not involve maintaining moisture, and the resulting shrinkage was due to moisture loss. Yan and others[3] in the same study also maintained at least 65% humidity, which was reported to cause minimal dimensional change for GIC materials. The present study, which had humidity values closer to that of the oral environment, did demonstrate specimen dimensional change. Tooth structure LCTE has been reported to be approximately 17 ppm $°K^{-1}$ for enamel and 11 ppm $°K^{-1}$ for dentin.[8] With combined dentin and enamel, the noncarious molar occlusal surface has been reported to be approximately 16 ppm $°K^{-1}$ with the molar cervical area to be approximately 5 ppm $°K^{-1}$.[34] For restoration of cervical molar areas, it might be intuitive to reason that the conventional GIC materials would be indicated, but the reported longevity of RMGI materials in cervical restorations cannot be dismissed. [35-37]

Table 3: *Mean (SD) LCTE Results (ppm/°K)*[a]

GIC Materials	Heating LCTE	Cooling LCTE
Chemfil Rock	16.7 (2.0) D a	21.4 (2.7) ABCD a
Equia	12.7 (2.3) DE a	17.5 (3.8) ABCDE b
Equia Forte	5.6 (1.6) G a	7.6 (0.9) E b
Fuji Triage	9.0 (2.2) EFG a	13.7 (3.4) BCDE b
Fuji II LC	25.4 (3.5) BC a	30.0 (5.0) AB b
Ketac Fil	7.9 (2.4) EFG a	8.8 (3.4) E a
Ketac Molar Quick	6.9 (1.0) FG a	7.4 (1.4) E a
Ketac Nano	47.4 (8.5) A a	50.3 (10.7) A b
Ketac Silver	8.1 (1.8) EFG a	9.2 (2.6) DE a
Ketac Universal	8.5 (2.1) EFG a	11.1 (1.9) CDE b
Riva Protect Fast	20.6 (3.9) CD a	14.6 (3.9) ABC b
Riva LC	30.6 (6.4) B a	36.9 (7.2) A b
Riva LC HV	28.9 (5.6) B a	32.6 (6.7) AB b
Riva SC Fast	14.4 (4.0) D a	17.6 (5.1) ABCDE a
Riva SC HV	6.4 (1.9) FG a	8.4 (2.5) E b
Riva Silver	12.3 (3.1) DEF a	14.6 (3.9) BCDE a

[a] *n=12. Uppercase letters identify similar groups per column (Kruskal-Wallis/Dunn's; α=0.05); lowercase letters identify similar groups per row (paired Wilcoxon sign rank; p=0.05).*

This study is the first to report LCTE determination during a dedicated cooling challenge. The results found that the cooling challenge from 50°C to 15°C produced a higher LCTE trend as compared with the heating cycle. With the exception of Chemfil Rock, Ketac Fil, Ketac Molar Quick, Ketac Silver, Riva SC Fast, and Riva Silver, GIC and RMGI products demonstrated significantly more LCTE during the cooling cycle. The reasons for this hysteresis and its significance are presently not known, and further research is warranted to identify possible causes such as proscribed temperature rate, testing conditions, or artifact induced by hardware used. Nevertheless, although the results are interesting, the difference is of relatively little magnitude, and it is presently thought doubtful to be of clinical significance.

CONCLUSION

Under the conditions of this study, the LCTE of conventional GIC materials during heating challenge ranged from approximately 5°C to 20°C ppm $°K^{-1}$, while the LCTE of the RMGI materials ranged from approximately 25°C to 47°C ppm $°K^{-1}$. With some exception, during a cooling challenge, the LCTE of all materials displayed a trend of higher LCTE, with the reason and significance unknown. However, the magnitude of the difference was relatively small and is presently thought to be of minor clinical significance.

Note

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of approval of the USAF Post Graduate Dental School, Keesler Air Force Base, MS.

Conflict of Interest

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

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