OPERATIVE DENTISTRY

Jul/Aug 2021
Volume 46
Number 4
351-476

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

OPERATIVE DENTISTRY

Volume 46 /Number 4
July/August 2021

www.jopdent.org

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

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

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Parthenon at night, Athens, Greece. Photo provided by Kevin Matis of Indianapolis, Indiana, USA. Photo taken with a Nikon D5500, 300mm f/6.3 1/5 sec.

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Invited Paper

Revisiting Conservative Class II Gold Foils

Robert H Bridgeman, DDS

The purpose of this editorial is to reintroduce you to the American Academy of Gold Foil Operators (AAGFO). Our board selected this classic article by Bruce Smith, which was published in the AAGFO journal in 1967 to illustrate the similarities and differences in the approach to restoring teeth with direct gold today (Figures 1 and 2).

Although it should come as no surprise to any of us that there are more options for dental restorative materials today than ever before, it should also challenge us to be more disciplined in our selection of the most practical material for each clinical situation we encounter. In some situations, it may not yet be necessary, or the right time, to treat the patient with a restoration at all. With respect to material options, our obligation to our patients is to discuss the performance, aesthetics, and expense of each restoration, and to identify which one best aligns with our patients' goals and expectations. If we truly practice this, then direct compacted gold foil is still the premiere restorative material for appropriate niche lesions.

In his April 1967 article, "Conservative Class II Foils," Dr Bruce Smith describes with great detail the benefits of foil, case selection, preparation, compaction, and finishing of Class II Foils. For many years now, and for a myriad of reasons, foils, much less Class II foils, have not been taught in schools. In 1947, Dr Gerald Stibbs essentially predicted this would

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http:/doi.org/10.2341/GFH.1

Conservative Class II Foils

Bruce B. Smith, d.m.d.

Seattle, Washington

Presented at the Annual Meeting of the American Academy of Gold Foil Operators in San Francisco, California on November 6, 1964.

THE CLASS II GOLD FOIL RESTORATION can be one of the most beautiful, delicate and functional restorations placed in a human tooth. Its replacement of diseased structure may be so fine as to withstand the damaging effects of decades of oral service. Usually it requires a little above average skill, but with training, restorations can be made within a reasonable length of time and with a minimum of discomfort for the patient. Many sleep while the operation is being done.

To use the term "Conservative" Class II foil is almost like repeating oneself. For this restoration is conservative above all other Class II types. However, there are some specific locations where the operation may be more easily accomplished and there are some conditions under which one may work more rapidly and easily. In addition to this, there are factors which make a Class II foil the operation of choice over an inlay from purely the standpoint of conserving tooth tissue for the patient.

As Ferrier[1] has said, "Consider only the tooth as an organ not capable of regenerative processes, such as bone, muscle, and mucous membrane, that once any part of it is lost, it can never he restored in kind; and that any restoration in any material falls far short of the original."

Dr Smith is in private practice in Seattle, Washington, and is on the Operative Dentistry staff at the University of Washington. He is Chairman of the Operative Section of the American Dental Association and Director of the John Kuratli Crown and Bridge Seminar. He has been a member of the University Ferrier Gold Foil Club for 17 years. In his spare time, Dr Smith enjoys sailboat racing.

Naturally, logic tells us that the first thing we can do is to save and conserve all possible dental tissue for the patient. And in considering incipient decay, gold foil is far superior than any inlay, for we can adapt the material to the needs of the case and not cut the tooth to suit a technique, which depends ultimately upon the withdrawal of a wax pattern from either a tooth or a die to establish the completed restoration.

So as prime indications we find (1) crowded or rotated teeth where an inlay would waste structure (Figs. 1 & 2); (2) bell crowned teeth for the same reason; (3) mesials of mandibular first bicuspids where no occlusal extension is required; and (4) generally speaking, mesial Class II cavities, as they are much easier for the average dentist who may not be familiar with the work.

A few general points are well to consider. On any mesial surface, a Class II foil has greater esthetic benefits. An inlay nearly always will show some gold. Often the operator has cut off the so-called "ears" of the bicuspid in preparing the inlay cavity, and amalgam used in these areas almost always shows through as a slight darkening. It is much easier to learn to condense the gold well on the mesial preparations. The angle of force is more nat-

Figure 1. Condensed cohesive and noncohesive foil in a rotated second bicuspid.

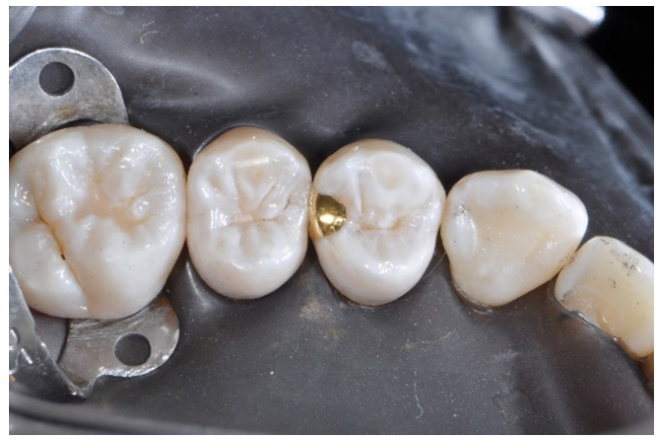

Figure 1. *Tooth restored with gold foil.*

happen, because even back then there was a certain disdain for the material.[2] He said poor case selection led to "a mistaken enthusiasm which engendered an era of sittings of many hours to insert huge fillings with the automatic mallet and which resulted in premature aging of the operator, premature death of the pulp, and a prayer for deliverance by the patient."[3] Because today's dentist heavily considers chair time when selecting filling materials, and because other more user-friendly and "forgiving" materials are now available, foil has taken a distant back seat, and there are very few doctors left who routinely, if ever, place gold foil in their practices.

Is There a Place for Gold Foil in Your Practice?

Gold foil has a place in the practice where the doctor believes in providing the best material for each specific case and is willing to dedicate the time necessary to learning and delivering each service they provide. Obtaining the necessary armamentarium is equally as

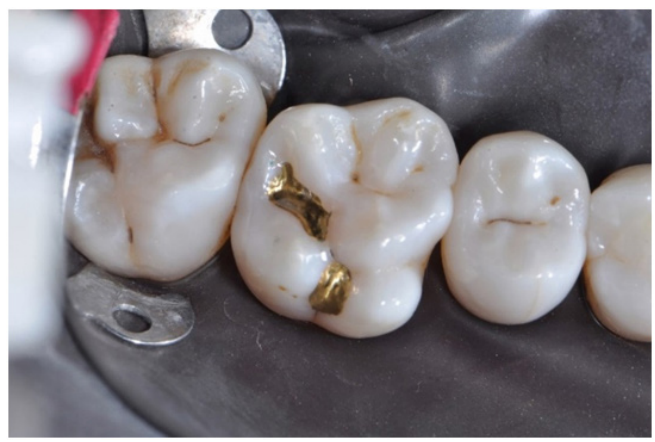

Figure 2. *Tooth restored with gold foil.*

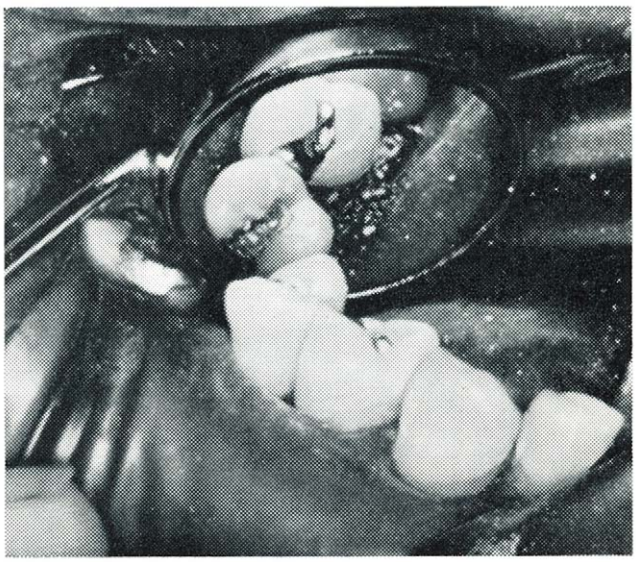

Figure 2. The completed restoration showing delicacy of form and minimal necessary extension.

ural and requires less use of highly offset bayonet condensers. In addition, when the work is done, it is more convenient to find any possible marginal or gingival angle deficiencies and to repair them with greater facility.

Distal Class II preparations, though slower and more awkward to fill, have one advantage in that the finishing strips and disks tend to lay in such a manner as to expedite finishing procedures. The use of the pneumatic or electromatic condensers render many of these areas highly accessible.

Condensation or compaction is the heart of all foil work - especially so in the Class II. The proximal gold should be layered and wedged toward each proximal wall. The vertical condensation should step out slightly beyond the cavosurface angle to give good wall adaptation, and the contact point should be well formed and condensed against the adjacent tooth using cohesive foil and not soft foil. A matrix has no place in this technique as lateral condensation later uses the excess gold for density and good coverage in finishing proximal and gingival margins. Minimum proximal extension often avoids great time waste. Over extension allows non-cohesive cylinders to slip out and makes it easier to add excessive amounts of foil

important as having practiced with it and the material prior to treating a live patient. Study clubs or, at the very least, dusting off the old dentoform, are probably the most reasonable places to gain proficiency with handling the delicate material prior to appointing one's first gold foil patient for a foil. Pursuing and receiving adequate mentorship early on will prove the smoothest road for learning the discipline and technique, and for avoiding pitfalls. Successfully identifying appropriate lesions and patients is essential. Selecting suitable first patients, such as relatives, friends, or employees, is an important consideration, as management of the operator's frustrations or procedural complications will be more easily navigated if the operator must veer from the original course of treatment. Having an experienced mentor present is also advantageous.

Case Selection and Case Presentation

If we are truly going to continue to offer all the best options in our practices, and if we are going to maintain vitality in dentistry, Class II foils are perhaps not the most appropriate place to begin. Therefore, consider straightforward Class I lesions with easy access, ie, buccal and occlusal pits on 1st molars and occlusal pits on premolars. In dental school, most of us first practiced on the dentoform with these teeth and surfaces. Suitable lesions need to be small because foil takes longer to handle, compact, and finish than the more modern options. Such "small lesions" include those where the enamel is slightly cavitated and chalky, an explorer barely sticks, transillumination reveals an underlying stain, our experience tells us a sealant or other nonrestorative approach will be inadequate, and the damaged tooth structure needs to be cut (Figure 3A). As operators, we should consider the caries management by risk assessment lesion classification, the patient, and all nonrestorative options prior to recommending a restoration. Other important considerations include: Can the patient tolerate a rubber dam and lengthy procedure? Will the patient accept a non-tooth-colored restoration? Is there presence of periodontal disease? Does the patient fear certain components of amalgam and composite, where the inertness of gold would be welcomed with more appeal? How old is the patient? How long does our patient expect to live? Does the patient value a slightly more expensive restoration to achieve better longevity?

Isolation and Preparation

With one valence electron, gold atoms adhere to one another very well, but any amount of any type of contaminant is catastrophic to the phenomenon of gold cohesion. Rubber dam isolation, therefore, is a must.[1] Furthermore, "superior results are obtained with less

on the lingual. Time is not only wasted in adding the excess gold, but often to a much greater extent in finishing it off.

Perhaps it is well to mention a few of the most common cause of difficulties or failures. One of the more frequent is inadequate condensation in the proximal gingival angles. This must be avoided in the placement and condensation of the three non-cohesive cylinders These are usually two 1/8 cylinders and one ¼ cylinder of No. 4 gold. They are swept powerfully into position with the No. 13, No. 14 parallelogram condensers in both a lateral and gingival direction, then condensed vertically with the large square bayonet condenser of the Ferrier study club set. Their final height when condensed should be about 2/3 rds of the height of the axial wall. This allows room for the following cohesive foil to aid in the retention of the proximal and to form the contact point.

Another common error is the use of an incorrect angle of force along the buccal occlusal walls of the preparations. To correct this tendency, a bayonet condenser or a right angle head in the pneumatic or electromatic condenser must be used. This is also frequently necessary on the mesial walls of distal cavities.

Proper layering of the gold bucco-lingually as described by Black[2] can be of great assistance in these situations. Yet from a biological standpoint, care should be exercised not to produce excessive wedging effects and pressures, as these can create hypersensitivity or even crack teeth.

These biological considerations are usually the normal ones we face in most operative procedures. There should be adequate pulpal protection from thermal shock during preparation procedures as well as suitable use of bases or medicaments to prevent post-operative complications. This may include prednisolone, calcium hydroxide and zinc oxide bases, or simply gum copal varnish. However, if sizable bases are necessary, the condensing pressures on the base should be considered. Sometimes a stronger base of zinc phosphate cement with alloy filings added is indicated. But the larger the cavity

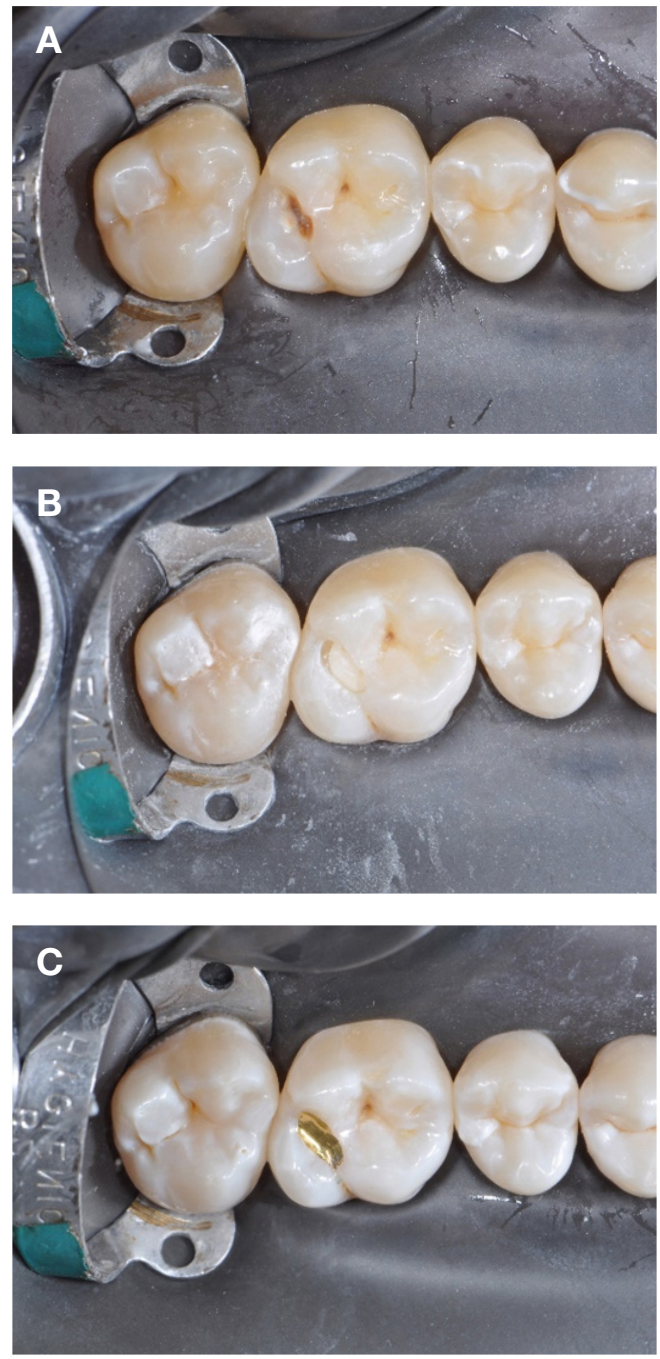

Figure 3. *(A): Preoperative #14-O caries detected visually and with slight explorer stick. (B): Completed preparation. (Note: Ionoseal light cured glass ionomer in pulpal floor to block out large void from caries removal, allow ideal conservative preparation, and minimize the amount of time placing and compacting the foil.) (C): Completed restoration with anatomic contours and grooves restored.*

effort," in less time, regardless of the material. Dr Smith reminds us that final outline form (convenience form) is ideal if it is just large enough for the operator to reach all areas of the preparation for adequate compaction

area the less the case is indicated for a foil restoration and the more an inlay or alternate procedure should be considered.

The separator can be a vicious instrument if care is not employed in its use. It should first be selected carefully to fit the case so that torsion effects are not incorporated. The jaws should be delicate and not impinge on the tissue. The screws should be free with a little "play" to avoid forceful wrench action and give more accurate control. Finally, the separator should be well stabilized with compound to avoid tissue damage and distribute pressures over four or five teeth.

CAVITY PREPARATION

With high speed a preparation can be cut very rapidly and efficiently, but the operator must have a clear picture of the preparation in mind to avoid overcutting or loss of detail. Fine cavity detail is of great importance in ensuring convenience of insertion of the gold and durability of the finished restoration.

The occlusal (Fig. 3) should be cut with a 700 series bur which has been broken and squared off to about 1/3 of its normal length. This automatically will set the proper depth and inclination of the walls. The walls must be slightly divergent in the isthmus area and at the occlusal wall distal to the proximal. This strengthens the marginal ridge. The only occlusal retention used should be gained at the expense of the buccal and lingual walls where they reach the distal. Proximal extension should be minimal to aid in supporting the non-cohesive foil and aids in a better esthetics. No bevels should be on any walls where non-cohesive foil is employed and only the fine finish of sharp cutting instruments is necessary to plane all walls to proper outline and completion.

FINISHING

One of the greatest aids to finishing procedures is a set routine. It is more than a convenience, it is a necessity. This is the one area where many men repeat and duplicate actions, wasting time, until they and finishing, yet small enough to minimize the amount of material to be compacted in a reasonable appointment. He goes on to describe the ideal characteristics of the Class II preparation (a textbook amalgam preparation). An occlusal Class I is simply a Class II without a proximal box, of course, but all of Dr Smith's recommendations of wall convergence or divergence should be followed to provide the strongest, full length, and dentin-supported enamel rod walls as possible. Sharp and crisp margins are obtained with sharp new burs and enamel hatchets (Figure 4A and 4B).

In the absence of occlusal caries extension, many Class II preparations can be completed as slot preparations with adequate approximal retention and resistance form (Figure 4). The use of a 169L to create opposing slots, entirely in the dentin of the buccoaxial and linguoaxial line angles to maximize retention. The slots can be shaped and internally sharpened with appropriate and sharp gingival margin trimmers to further improve retention of the initial increments of gold (Figure 4B).

Liners and Bases

Dr Smith recommended suitable bases that were available at the time, including calcium hydroxide and zinc oxide bases, to be utilized in cases where postoperative sensitivity was expected. Dentin desensitization with GLUMA (Kulzer, South Bend, IN, USA), followed by dentin block-outs with newer fluoride-releasing resin-modified glass-ionomers might prove more readily available, easier to place, sufficient to withstand condensing forces, and more effective in preventing postoperative sensitivity (Figure 3B). The operator should consider other restorative materials like cast gold, composite, or amalgam in larger preparations where excessive dentin substitution is necessary and the area is too large to apply foil in a timely manner. Although Dr Smith claims many patients sleep through the operation, encouraging the patient to begin 400 mg ibuprofen, four times a day, starting the day before and continuing for a few days after placing a gold foil is worth its weight in gold for both patient comfort and the operator not being bothered with follow-up visits for pulpitis. Taking ibuprofen preoperatively can significantly decrease the initial and total amount of inflammatory proteins that cascade, leading to a happier patient and doctor.

Gold Placement and Compaction

Conventional noncohesive sheets of foil (Jensen Dental, North Haven, CT, USA) and powdered gold (EZ Gold; courtesy of Dr Clyde Roggenkamp) are still available

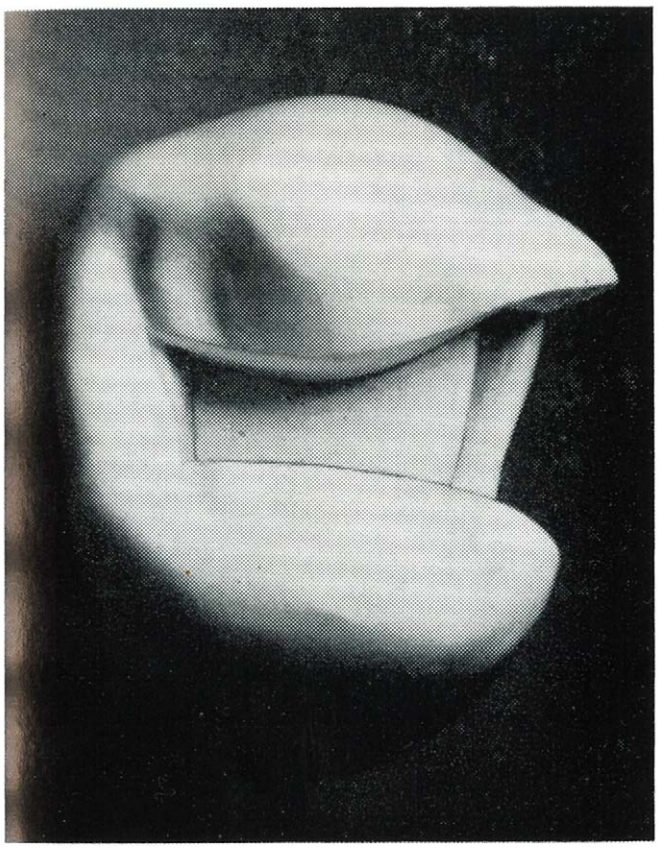

Figure 3. Occlusal view of normal Class II foil preparation. Note reverse curve on buccal to allow proper proximal boxing. Isthmus area and marginal ridge walls are slightly divergent. Retention areas are at the expense of the buccal and lingual walls toward the distal.

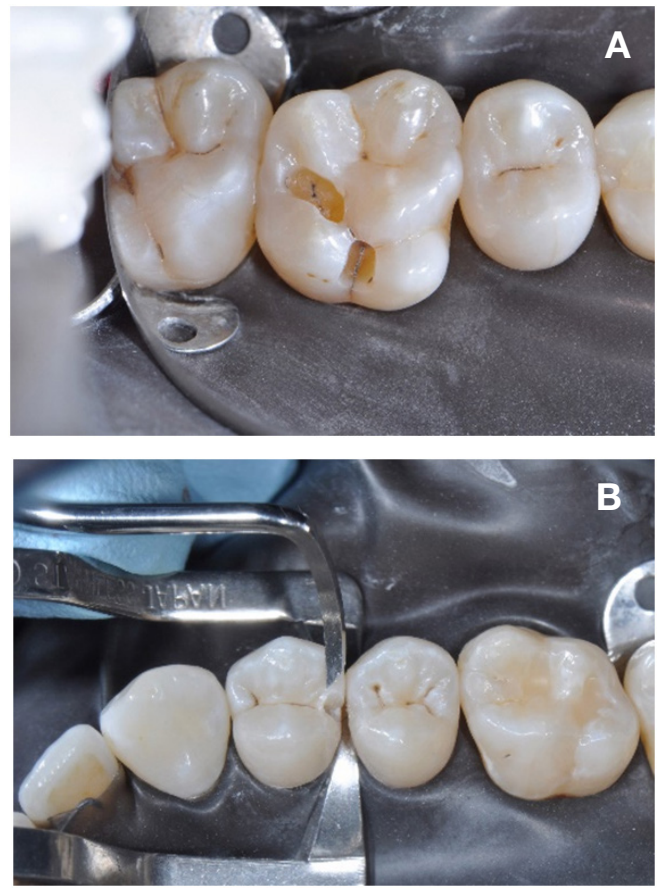

Figure 4. *Dry field established with rubber dam isolation in both images. Permanent orthodontic retainers and bridges are more challenging to perfectly isolate, but otherwise, dam margins should be inverted for optimal isolation. (A): Completed occlusal and palatal preparations, flat pulpal floor, and diverging distal wall to allow full length enamel rods supported by dentin, and smooth cavosurface margins and outline form. Internal line angles are sharpened with sharp explorer tine, and additional retention is cut with 33.5 inverse cone bur. (B): Completed Class II preparation with sharpened Tucker gingival margin trimmers #232 and #233, used to sharpen gingivoaxial, axiolingual, and axiobuccal internal line angles for retention (Tucker Institute Manual).*

eventually end up with a completed operation. The use of burs, files, gold knives and the Searl swagger (Suter Dental Instrument Co., Chico, California) should preceed [sic] the use of graded disks. An interesting miniature burnisher is of great convenience in finishing occlusals. The small instrument has short extensions which permit the operator to exert greater burnishing force with less tendency for the instrument to twist within his grasp. Also, the small burnishing surfaces are more suited to our present delicate cavity extensions. (Fig. 4)

Finishing burs may be moistened with water to prevent "leading." They usually consist of two types: one, a squared off 700 series bur, is very fast for use in dentistry today. Noncohesive foil becomes cohesive when degassed, or annealed, as the pellet is passed through a flame to remove the ammonia gas. At this point, the degassed pellets will readily stick to each other and compact more easily. Dr Smith recommends initially placing noncohesive gold cylinders into the proximal box of a Class II, reasoning that the gold will slip more easily into the sharp and retentive internal line and point angles, and to bulk up two-thirds of the box more quickly. Because cylinders and pellets are commercially unavailable, a member of the team must use sheets of noncohesive foil to roll their own cylinders and pellets, certainly a drawback for today's operator and team.[4]

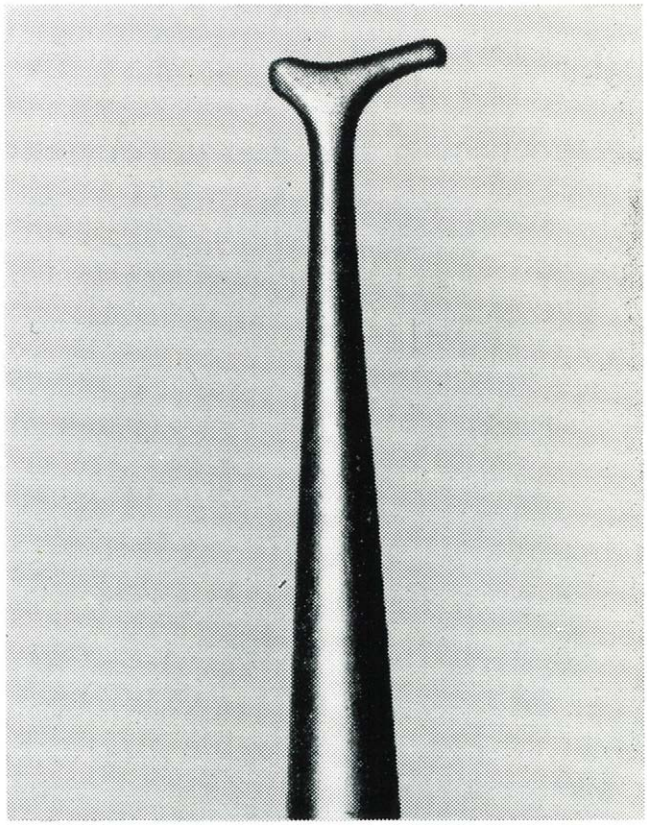

Figure 4. Extra-small burnisher with short leverage to prevent turning in the band.

and convenient in setting the inclined planes and central groove; the other - a round bur - may be right or left cutting, and is very helpful in trimming gold to margin, especially in the extensions. Finally, a dull number ½ round bur is excellent to accentuate and define previously established grooves.

The separator should be known by number; usually the Ferrier No. 4 is indicated for Class II foils. Occasionally, the No. 3 will be better on the angle of the arch for mesial restorations in first bicuspids. This depends upon the narrowness of the arch and the conformity of the teeth.

After all gross finishing is done, i.e., the gingival and all occlusal anatomy with the exception of the occlusal embrasure, the separator should be placed momentarily and a Gordon White saw passed through the contact area. A lightning strip and subsequent finer extra long finishing strips (Moyco) should be used with copious amounts of EZ Gold has also been shown to be a suitable material for bulking-up quickly, having ease of compaction (5 lbs), and doing so to a suitable denseness to survive the oral cavity, especially if veneered with cohesive (degassed) conventional foil. After bulking up the preparation to within 0.5–1.0 mm of the occlusal portions of the cavosurface margins, veneering with cohesive foil to finish out the anatomically correct contours of the restoration is preferred because it condenses more completely than powdered gold, leads to better marginal adaptation, and holds a better polish for hygienicity and aesthetics.

Dr Smith does not recommend the utilization of a matrix in Class II's, but it has been shown, particularly in deeper or wider boxes, or lesions not in contact with an adjacent tooth, that by better containment of the material, less gold is extruded (Figure 5). Thus, the benefits are 2-fold: less time is spent unnecessarily compacting excess gold, and less time is spent removing excess gold during finishing. A matrix can save precious time and material, and prevent frustration for both the patient and the operator.

Although one can conceivably place small Class I and Class II gold foils with hand condensation alone, malleting provides better density and polish. Obtaining an electromallet (Figure 6) or a pneumatic condenser will save considerable time in achieving adequate compaction. Because time is the real elephant in the room regarding the disappearance of gold foil and its operators, today's operator should treatment plan for ideally sized cases, utilize automated condensers for the bulk of the condensation, and consider high-speed finishing after final condensation.

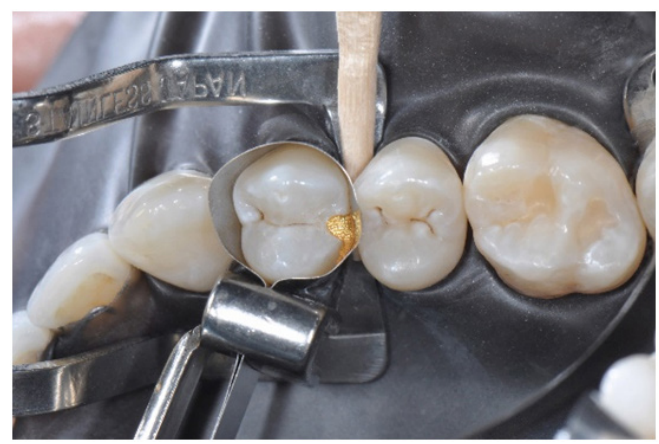

Figure 5. *EZ Gold quickly bulk filled to within 0.5 mm from the cavosurface margin, prior to cohesive foil veneer. Because the lesion was palatal to contact, the adjacent tooth could not act as the matrix; therefore, the operator utilized a Tofflemire matrix to better contain the gold and save time.*

air. This will leave a beautifully finished and polished interproximal surface.

The strips should be manipulated with care and relieved at either buccal or lingual surface to maintain proper contact point relationship and correct embrasures at this time. The occlusal embrasure should receive special consideration. A sharp gold knife or small cleoid swept across the marginal ridge while the separator is in place will set up the proper angulation for the embrasure and the escape gate. It is often convenient to mount a large but extra fine cuttle fish disk in the straight screwhead (small-size) mandrel. This will by-pass the separator frame and nicely round out and highly finish the embrasure.

A step by step logical finishing routine will reward the operator with consistently excellent results with a happy, rested patient.

An ideal Class II from the standpoint of ease of operation is the mesial of the lower first bicuspid. (Fig. 5) Because it occludes with the upper cuspid only, there is no stress on the occlusal surface and no occlusal extension is necessary. Both buccal and lingual proximal walls make acute angles with the gingival due to the shape of the adjacent mandibular cuspid. The interior has accentuated axial line angles to help retention. An excellent instrument for this delicate feature is the special gingival margin trimmer No. 28˚ and No. 29˚ (Suter Dental Instrument Co., Chico, California). These were designed by C. T. Fleetwood and are also of great convenience in lingual approach Class III foils. (Fig. 6)

Usually only three 1/16th non-cohesive gold cylinders are placed at the gingival. The cohesive gold placement is delicate and wedging should be carefully accomplished to ensure good wall adaptation. Finishing procedures are minimal and the operator can easily see and check his work.

The result is a delicate, beautiful and inconspicuous Class II restoration.

To cut across the large and solid transverse ridge would be a waste, both of time and tooth structure, for this tooth is much like an overgrown

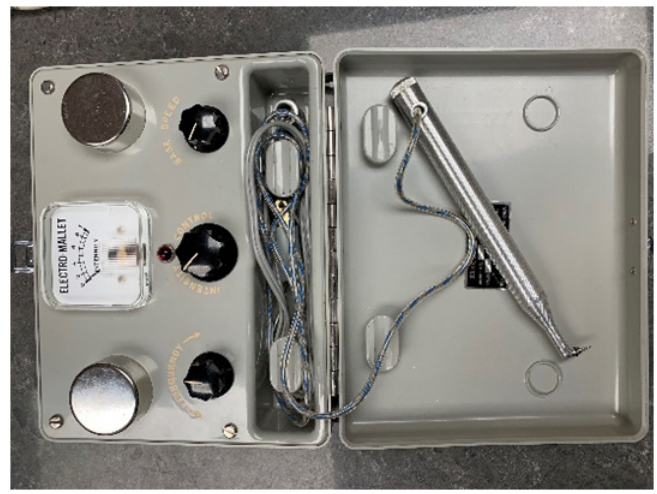

Figure 6. *Although locating one might prove challenging, an electromallet is a much more efficient instrument for condensing gold foil than a conventional mallet and foot condenser, and it frees the assistant of the burden of malleting (Roberson TM, Heymann H, Sturdevant CM, & Swift EJ (2002) Sturdevant's Art and Science of Operative Dentistry Mosby, Maryland Heights, MA).*

Finishing

The remaining tooth structure should dictate the final level, shape, and contours of the gold. Again, care should be taken not to overfill to save time. Once adequate contours have been achieved, Dr Smith recommends a system for finishing to save time. Finishing with hand instruments should still ideally be completed to trim flash, and burnishing should be completed prior to rotary finishing. Hand burnishing and carving the gold work hardens it, and it will polish more completely with less air porosity.

In Class II's the proximal contact should be adjusted first. Releasing the separator, the operator can gauge the weight of the contact prior to selecting interproximal saws, or which grit of finishing strips are necessary to avoid over- or underfinishing the contact area. The use of a swedge, either in the electromallet or by hand, helps to create the convex marginal ridge, forming proper embrasures on the occlusal, buccal, and lingual surfaces (Figure 7). Appropriate embrasures facilitate easier passage of floss into the proximal contact and for food to sluice off the occlusal table and around the proximal contact, appropriately stimulating the soft tissues. The interproximal contact weight is evaluated and adjusted to the appropriate contact weight with abrasive strips (Figure 8). Further finishing of the buccal and lingual margins is done with paper backed E. C. Moore's Discs (E. C. Moore, Dearborn, MI, USA).

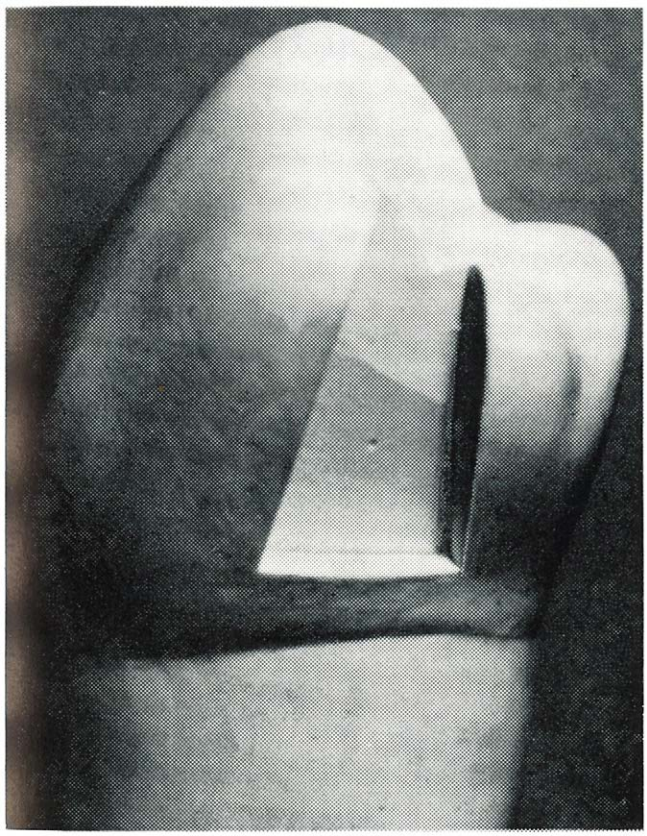

Figure 5. Cavity preparation for the mesial of the mandibular first bicuspid. No occlusal step.

cuspid. A central groove is almost never present. If a groove is present, it is nearly always in the distal portion of the occlusal. (Fig. 7) In addition, if an occlusal extension were made, the great size of the buccal cusp would tend to augment thermal shock because of gold being closer to the extension of the pulp.

One of the first questions asked by men tempted to try Class II foil work is, "How much time should this operation take?" Naturally, the correct answer is, "Enough time to do the case at hand properly." However, to quote averages which may be helpful, two to two and a half hours should be allowed in the beginning. Later, an hour and a half to two hours should be adequate. Ideal cases have been done in an hour or even 45 minutes by highly skilled men, and the most remarkable time of 40 minutes, including anaesthetic administration, has been witnessed.

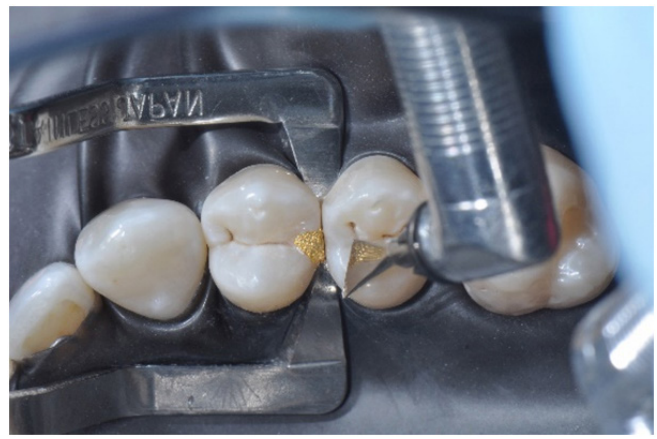

Figure 7. *After cohesive foil veneer is built up to properly restore contours, a swedge is used in an electromallet to properly create the embrasure form.*

With the better availability of burs and finishing materials today, many Class I's can be finished with high and slow speed rotary burs very quickly. Copious water spray should be used at lower speeds with the high-speed handpieces and with light pressure, and the assistant should keep air moving over the tooth while finishing dry with any handpiece. Shaping grooves and following ridges with dull, small, round- and football-shaped carbide burs is efficient and can be retraced with green and white stones, and with brownie, greenie, and super greenie points and cups, in both high- and slow-speed handpieces. Beginning in the grooves and working one's way outward, the higher spots seem to

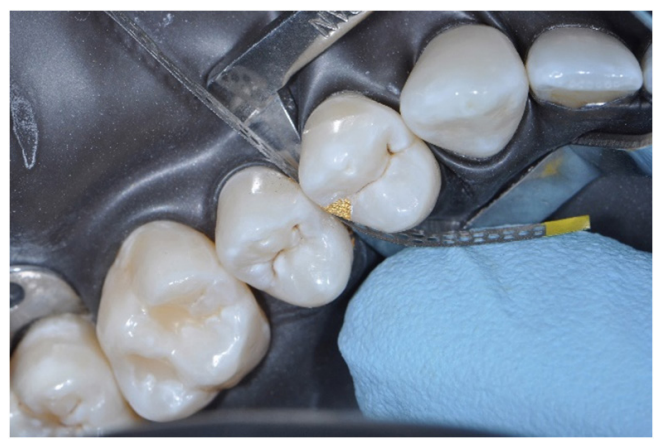

Figure 8. *After swedging, the separator is released, contact weight is evaluated with floss, and the proximal saw and the appropriate interproximal abrasive strip is selected to finish and smooth interproximally. Here, the operator felt the contact weight was slightly heavy and chose to begin with a medium grit strip (coarse = blue, medium = yellow, fine = red) to begin interproximal finishing.*

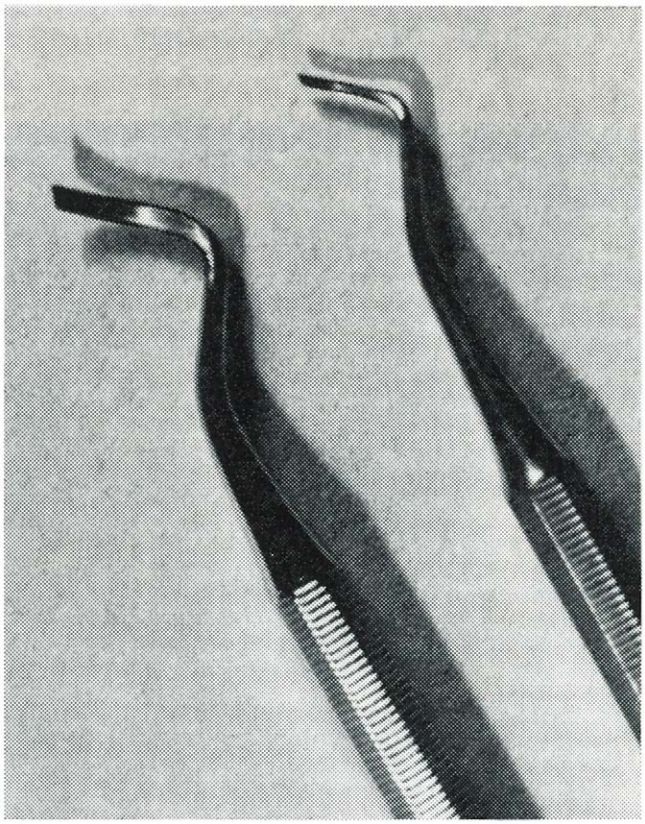

Figure 6. Instrument No. 28°, the extra- small gingival margin trimmer, compared with a normal size instrument.

To return to normal considerations however, it's safe to say that the time consumed to create a beautiful Class II foil is nearly always less than the time and effort required to produce and cement a Class II inlay. The operator should not think in terms of speed, but in terms of excellence, efficiency and service.

Lastly, let us consider contra-indications. It is proper here to quote the Latin legal phrase, *res ipsa loquitur*, the thing speaks for itself. For professional experience, training and judgment are almost perfect guides to the average man. A tooth without proper gingival support would certainly not be a likely candidate to receive condenser blows or even give good gold condensation. Large cavities imposing undue stress on the patient or the tooth are questionable to use. Devital teeth, or those with impaired circulatory protection, should be avoided if possible. Then, once in a great while, the unusual

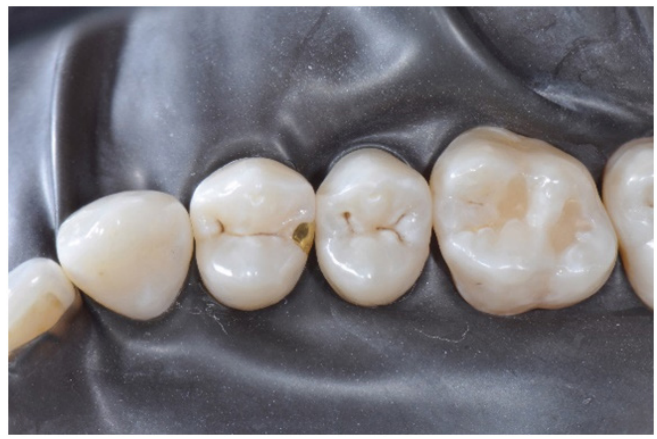

Figure 9. *A completed Class II gold foil.*

finish down without demanding much attention of their own.

Placing gold foils routinely in private practice, in a study club environment, or even on a dentoform will challenge even an experienced operator to improve their overall skillset as an operative dentist. As a material, gold foil arguably demands more patience and skill from the operator than any other restorative material. It continues to teach discipline in many of the principles we committed to memory in school and strive to practice today, including case selection, isolation, resistance and retention form, convenience form, material manipulation and condensation, and exquisite finishing. The acuity and attention to detail that direct gold restorations require are contagious and will likely spill over to positively impact every aspect of our clinical ability to provide better care and dentistry for our patients.

The AAGFO is perhaps the only source available today to obtain all the information regarding everything gold foil, in one place. Proficient mentors, courses, study clubs, instructional manuals, and lists of materials, instruments (AAGFO hand instrument kit; courtesy of G Hartzell Denmat), and supplies can all be found there. Our annual meeting is open to anyone interested in attending, and all the information needed to register for the meeting or to begin placing gold foil is available on our website, www.aagfo.org.

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.

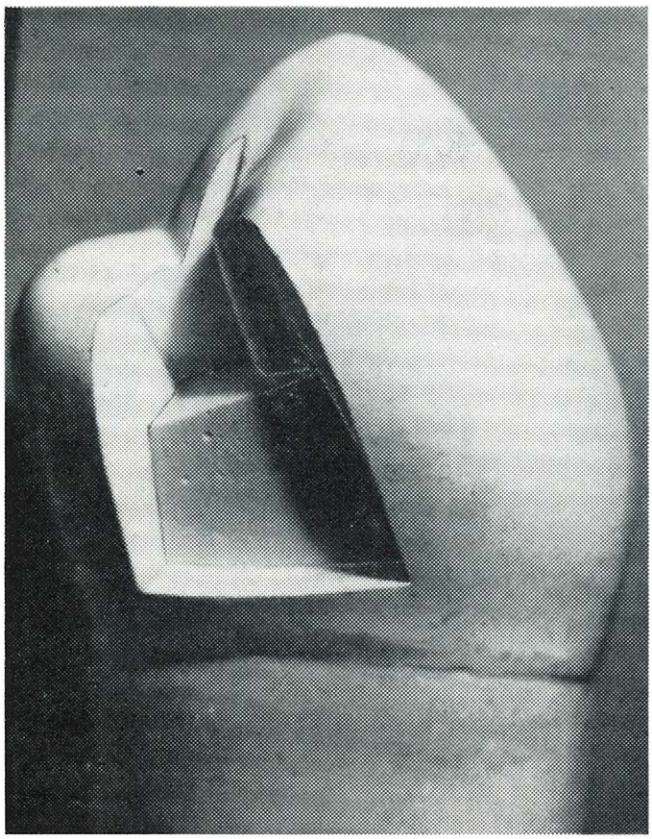

Figure 7. Cavity preparation for the distal of the mandibular first bicuspid. Occlusal step is sloped to avoid the large pulp horn.

patient will appear who is psychologically unsuited to stand the malleting or condensing blows. Fortunately, the usual patient, on the other hand, seems to actually enjoy his brief period of relaxation while the foil is placed.

In conclusion, it is hoped that some ideas and aids toward operational procedures will have been found here. If so, the author may have partially repaid his debt for some of the help and assistance gained from predecessors.

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Clinical Research

Glass Ionomer Versus Self-adhesive Cement and the Clinical Performance of Zirconia Coping/ Press-on Porcelain Crowns

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MC Mailart • R Di Nicoló • AB Borges

Clinical Relevance

The use of either GIC or self-adhesive resin cement has the same effect on the retention and durability of all-ceramic crowns.

SUMMARY

Objective: This split-mouth clinical study investigated the effect of luting cement on the performance of veneered yttrium-stabilized tetragonal zirconia polycrystal (Y-TZP) zirconia crowns.

Methods and Materials: A total of 60 crowns prepared with Y-TZP coping and press-on porcelain were made with a split-mouth design in 30 participants. The crowns were cemented either with glass ionomer cement (GIC) (Meron, Voco) or with self-adhesive resin cement (Bifix-SE, Voco).

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http://doi.org/10.2341/20-029-C

The restorations were assessed immediately after treatment and after 6, 12, 24, 36, and 48 months using the modified United States Public Health Service criteria. The parameters analyzed were retention, color stability, marginal discoloration, marginal adaptation, surface roughness, anatomic form, and secondary caries. The differences between the groups were analyzed by the Fisher exact test in each period of evaluation. The survival rate was analyzed with the Kaplan–Meier and log-rank test (α=0.05).

Results: After 48 months, 20 participants attended the recall. During the period of evaluation, 1 crown cemented with glass ionomer cement and 1 crown cemented with resin cement lost retention. Color match, marginal discoloration and adaptation, surface roughness, and anatomic form did not change in any of the periods evaluated, and no secondary caries was observed. No significant differences were found between the 2 luting cements for any of the clinical parameters analyzed, nor for the survival rates during the study.

Conclusions: The type of cement did not influence the performance of the crowns after 48 months of clinical use. Both cements resulted in adequate retention rates, aesthetic and functional outcomes, and biological response.

INTRODUCTION

Porcelain fused to metal (PFM) crowns are recommended as a strong and durable restoration for anterior and posterior teeth, and they are referred to as the gold-standard treatment when veneered with feldspathic ceramic.[1,2] However, the popularity of all-ceramic crowns (ACCs) has increased in recent years due to the demand for large reconstructions of damaged teeth with high aesthetic performance materials, even in challenging situations.[3-5] One of the most commonly used materials is yttria-stabilized tetragonal zirconia polycrystal (Y-TZP), which is biocompatible and aesthetic, and which has excellent mechanical properties, including flexural strength and fracture toughness.[3,6,7] It can be used as an infrastructure, veneered with ceramic (press-on or layering application), or as a monolithic crown. Survival rates up to 96.9% for Y-TZP all-ceramic single crowns have been reported.[8]

For conventional PFM crowns, mechanical retention and resistance are obtained by preparing a minimum practical convergence angle to allow for the micromechanical interlocking of the axial walls, cement, and intaglio of the crown[2] with nonadhesive cement such as zinc phosphate. However, with full feldspathic porcelain crowns, an adhesive system was recommended to avoid bulk fracture.[4,5] With the development of stronger ceramic materials, the use of adhesive cements became less essential, and even zinc phosphate or glass ionomer cements (GIC) could be used.[2,3]

Although Y-TZP ceramics, as coping or monolithically, have improved the performance of indirect restorations, a standardized adhesive cementation protocol has not been determined for this type of restoration.[9,10] The lack of a vitreous phase makes it resistant to the hydrofluoric acid etching generally used for adhesive purposes in ceramic restorations. The use of GIC for luting complete crowns has advantages in relation to the traditional nonadhesive cements, mainly the chemical bonding between the cement layer and the dentin through calcium chelation by the polyacrylic acid.[11] However, there is no chemical bonding between the cement and zirconia coping, and thus mechanical retention is also required. Another advantage is fluoride release, which can inhibit demineralization at the interface and close to the margins, preventing secondary caries.[11] Regardless of these favorable characteristics, there is no clear evidence of how glass ionomer cements influence the performance of Y-TZP ceramic restorations.

Resin cements are often used to bond indirect restorations because of their adequate marginal adaptation, high flexural strength,[5] lower solubility, and superior esthetics compared with other luting agents. Additionally, they are reported to provide a durable bond between the ceramic and tooth structure, protecting the crowns from catastrophic fractures.[10] More recently, self-adhesive resin cements (SACs) have been developed to simplify the luting procedure. Instead of a two-step procedure with an adhesive system for bonding the resin cement to the tooth structure and a dedicated primer for bonding to the crown, a single material is used. The cement contains acidic phosphate monomers capable of chemically bonding to both the tooth structure and to zirconia.[12,13] Although some adhesion can be achieved, the bond strength values to enamel and dentin have been reported to be lower than with conventional adhesive systems.[9,14,15]

The different characteristics of the cements available for luting ACCs might affect their clinical performance, mainly with regard to retention and secondary caries. Clinical trials are thus necessary to evaluate the long-term behavior of the restorations and to establish which type of material and technique would provide the best results for luting such restorations. The aim of this

randomized clinical trial was to compare the use of GIC and SAC on the clinical performance of zirconia coping/press-on porcelain crowns. The null hypothesis tested was that the type of luting cement would not influence the performance of the ceramic crown.

METHODS AND MATERIALS

Study Design and Ethical Aspects

This study was a 48-month follow-up of a longitudinal, prospective randomized clinical trial designed as a split-mouth and double-blinded (participant and clinical examiner) study. Aiming to define the parameters of the study, a PICO question was stated: P, adult patients presenting the indication of two single-unit crowns, both in the anterior or both in the posterior area; I, cementation with glass ionomer cement; C, cementation with SAC; and O, clinical performance according to modified United States Public Health Service (USPHS) criteria. A research question was determined: Do single-unit ceramic crowns perform in a clinically similar way when cemented with GIC compared with SAC according to USPHS modified criteria? The reporting of the present study followed the Consolidated Standards of Reporting Trials guidelines.[16] All participants recruited for the study signed an informed consent letter containing the details and objective of the study, and the risks/benefits of the interventions. This study was approved by the Committee for the Protection of Human Participants of the local university and was registered at the website for clinical trials.

Calculation of Sample Size

Aiming to determine the sample size, an online software program (www.sealedenvelope.com) with sample size calculators was adopted. For the present study, the sample size was determined with a calculator for a binary outcome–equivalence trial. The clinical success rate of the all-ceramic crown reported in a previous study was adopted (98.5% at 36 months).[17] A significance level of 0.05, power of 80%, and equivalence limit of 10% were considered. Twenty-six restorations per group were required, and for the split-mouth design adopted, 52 restorations were required. Considering possible dropouts, 60 restorations were provided (30 per group). Thus, 30 participants were selected.

Participant Randomization and Allocation

An online software program was adopted to generate a randomization list (www.sealedenvelope.com). First, the treatment group (GIC–Meron and SAC–Bifix SE) block sizes and list lengths were entered into the software program. Each selected participant received an identification number, and a randomization list was created. For each participant, the 2 treatment options (first and second) appeared randomly in the list. For each option, a unique two-letter and one digit randomization code was created. For instance, for participant 1, the list could be (P1, Group GIC, XS2; P1, Group SAC, RQ4). To correlate the list with the prepared tooth, the ADA numbering system was used, and the first option in the list was applied to the tooth with the smaller ADA number. For example, if the treatment was performed on tooth 3 and 14, the first treatment option was applied on tooth 3 and the second treatment option on tooth 14. A person not involved with the clinical procedures assigned a sealed paper envelope containing the information about which tooth would receive which cement during the luting procedure, as well its randomization code to each participant. All records on the clinical charts used during the evaluation recalls contained just the randomization code. The codes were broken just after the statistical analysis.

Eligibility Criteria for Participants

The participants received a clinical examination. Inclusion criteria were as follows: at least 18 years of age with good general health, healthy gingival tissue, and an indication for two single crowns in the anterior or posterior area. The participants presented normal occlusion according to the Angle classifications, without posterior or anterior crossbite or functional crossbite. The force distribution among the teeth was equivalent, and the number of posterior teeth was the same on both sides. The occlusal force should be considered normal, without signs of bruxism and clenching. The exclusion criteria were as follows: poor oral hygiene, periodontal diseases, the use of a removable prosthesis or orthodontic appliances, and an implant-supported opposing tooth. Tooth mobility grade 1 (maximum horizontal mobility of 1 mm) was accepted (maximum grade). The prospective abutment tooth was vital or optimally endodontically treated. The antagonist and adjacent teeth were present. The condition of the opposing tooth (natural, restored with composite resin, amalgam, or ceramic), core material (dentin, glass fiber post, and composite resin or metal), and the reason for treatment were recorded at the beginning of the study. To compare the clinical performance of the luting agents, a split-mouth design was followed, and each selected participant received both treatments. Therefore, the 30 participants received 60 single crowns.

Blinding

Participants and examiners were blinded to the interventions. The operators were not blinded since the procedures involved in the cementation step could not be masked.

Tooth Preparation

The operators were fully informed about all the details of the study's protocol, as well as the rationale, objectives, and design. Additionally, the operators received training on dental manikins in an attempt to standardize the tooth preparation. All clinical procedures were monitored by the principal investigator. Before tooth preparation, the tooth shade was analyzed for each crown based on the shade of the neighboring teeth using the Vitapan Classical Shade Guide (Vita Zahnfabrik, BadSäckingen, Germany). Information about the type of tooth included in the study, the reason for performing the procedure, the kind of core, and opposing tooth are described in Table 1. The teeth were prepared according to the following parameters: preferably, placing the finishing line supragingivally or equigingivally; the margin design was a 1-mm-wide chamfer;[18] the axial reduction was 1.5 mm; the occlusal/incisal reduction was 1.5–2.0 mm; the total occlusal/incisal convergence was 6–15 degrees; and the line geometry was rounded.[19] For the axial and occlusal/incisal reductions and the definition of the chamfer margin, round-end taper diamond rotary instruments of appropriate sizes were used.

Table 1. *Distribution of Opposing Teeth, Core Material, Reasons for the Replacement, and Type of Teeth of all Groups*

Characteristics	Type	GIC	SAC
Core material	Metallic	7	6
	Glass fiber + composite	5	8
	Dentin	18	16
Opposing teeth	Enamel	25	25
	Composite	2	1
	Amalgam	1	2
	Ceramic	2	2
Reasons for replacement	Caries	7	9
	Fracture	10	9
	Esthetic	11	10
	Other	2	2
Type of teeth	Molar	12	12
	Premolar	8	8
	Incisors	10	10

Abbreviations: GIC, glass ionomer cement; SAC, self-adhesive resin cement.

Interim Restoration and Impression

After the tooth preparation steps, direct interim crowns were fabricated using an autopolymerizing bis-acrylic composite resin (Structur 2 SC; Voco) according to the manufacturer's instructions. To prepare the interim crown, a quadrant impression of each tooth before the preparation was made with irreversible hydrocolloid (Jeltrate; Dentsply Sirona). When the original crown had significant loss of structure, a wax-up was performed on a gypsum cast, from which the impression was made. After the tooth preparation, the bis-acrylic material was mixed and applied inside the impression and inserted in the mouth. After 90 seconds, the impression was removed from the mouth, and after 3 minutes the crown was removed from the impression, finished, and polished. Retraction cords (Ultrapack; Ultradent) were then placed according to the double-cord technique. For this purpose, a thin retraction cord (#000) soaked in aluminum chloride hemostatic solution (Hemostop; Dentsply Sirona) was placed into the sulcus, and a second retraction cord (#00) was placed on top of it. The outer retraction cord was removed after 5 minutes. Impressions were made using a simultaneous dual-mix technique and a polyvinyl siloxane material (Express XT; 3M Oral Care). The interocclusal registration was done with autopolymerizing polyvinyl siloxane (Registrado Clear; Voco). After that, the interim crowns were cemented with eugenol-free interim cement (Provicol; Voco). Casts were prepared with Type IV extra hard dental die stone.

Fabrication of Crowns

The casts were laser scanned with a Cercon Eye CAD module (Dentsply Sirona). The coping structure was milled out of a presintered Y-TZP block (Cercon Base; Dentsply Sirona) using a CAD/CAM System (Cercon Brain; Dentsply Sirona) and evaluated on the definitive die. The milled coping was subsequently postsintered in the Cercon Heat (Dentsply Sirona) high temperature furnace using the 6-hour sintering program at a maximum temperature of 1350°C. Following this, a wax pattern was made, and a press-on ceramic (Cercon Ceram Press; Dentsply Sirona) was pressed onto the coping. Cercon Ceram Press is composed of silica, alumina, potassium oxide, sodium oxide, and calcium oxide. The crowns were fabricated by the same laboratory technician.

Cementation of Crowns

The interim crowns were removed, and the tooth preparation was cleaned. The ceramic crowns were seated, and the occlusal and proximal contacts were evaluated. For occlusal evaluation, a thin articulation foil was placed between the opposing teeth during the maximum intercuspation position and during the protrusive and lateral movements of the mandible. Whenever necessary, the contacts on the ceramics were adjusted, and the surface was polished, resulting in an adequate interocclusal relationship. The proximal contacts were evaluated with dental floss and were also adjusted whenever necessary, resulting in physiologically acceptable proximal surface contacts. The marginal adaptation was checked with an explorer before luting. The definitive luting was performed with a GIC or an SAC (Table 2) according to the manufacturer's instructions. The intaglio surface of the crowns was airborne-particle abraded with aluminum oxide particles and cleaned in an ultrasonic water bath. The Bifix SE cement was provided in a self-mixing syringe. The cement was dispensed directly into the crown, which was seated, and any excess was removed. The buccal and lingual sides were light polymerized, followed by the chemical polymerization of the material. For Meron, 1 drop of liquid and 1 scoop of powder were dispensed on a suitable mixing pad. The powder was divided into 3 portions, and each one was mixed into the liquid with a solid plastic spatula. The cement was dispensed into the crown, which was seated on the prepared tooth and any excess cement was removed.

Clinical Assessment of Ceramic Crowns

All the crowns were evaluated by 2 independent, previously calibrated observers, who were blinded to the treatment, according to the modified USPHS criteria[20] and additional parameters. The criteria were retention, color stability, marginal discoloration, marginal adaptation, surface roughness, anatomic form, and secondary caries, as shown in Table 3. For each criterion, 1 of 3 or 1 of 4 scores were applied. In addition, occlusal contacts, gingival index, pulpal status, and chipping and wear of the opposing tooth were analyzed for each crown according to the parameters presented in Table 3. Baseline data were recorded 7 days after cementation. Follow-up appointments were made 6, 12, 24, and 48 months after insertion. The following criteria were selected to determine the need for replacement: fracture of the framework material, major chipping that is not repairable by composite resin material, caries in the abutment tooth, and tooth loss because of biological complications (eg, fracture of abutment tooth, irreversible pulpitis).

Statistical Analysis

For the statistical analysis, an intention-to-treat protocol was considered.[21] Data were analyzed using the Fisher exact test (α=0.05) for each individual parameter and the following periods of evaluation (7 days and 6, 12, 24, and 48 months). To determine the survival rate regarding restoration loss (failures), the Kaplan–Meier estimate was performed, followed by the log-rank (Mentel-Cox) test.[22] The tests were performed using Statistica for Windows (StatSoft) and GraphPad (GraphPad Software) software programs.

RESULTS

Eighteen of the 48 screened participants did not achieve the study eligibility criteria previously described. Therefore, 30 patients who needed 2 complete crowns were enrolled (Figure 1). The failure of treatment was considered as the loss or detachment of the crown for various reasons. The percentage of each score for all analyses is presented in Table 4. The Kaplan–Meier survival analysis was performed, and the survival curve is presented in Figure 2. The log-rank (Mentel-Cox) test showed nonsignificant differences between the curves (p=0.5619). Both groups presented a 95.8% survival rate after 4 years. Examples of the crowns after 4 years are presented in Figure 3.

Table 2. *Luting Agents Tested (Information Provided by Manufacturer)*

Cement	Type	Composition	Manufacturer
Meron	Glass ionomer luting cement	Powder: fluoro-aluminosilicate glass, polyacrylic acid Liquid: tartaric acid and distilled water	VOCO, GmbH, Germany
Bifix SE	Self-adhesive resin cement	Bis-GMA, UDMA, Gly-DMA, phosphate monomers, glass fillers, aerosil silica, initiators, stabilizers Filler content: 70% w/w	

Abbreviations: Bis-GMA, bisphenol A-glycidyl methacrylate; Gly-DMA; UDMA, urethane dimethacrylate.

Table 3. *USPHS Criteria Used for Clinical Assessment*	
Criteria	**Scores**
1. Retention	A) Cemented crown is retained.
	B) Cemented crown is partially retained.
	C) Cemented crown is not retained (released).
2. Color match	A) Crown remains equal to the adjacent tooth structure in color, shade, and translucency.
	B) Change on shade or translucency in tolerable values when compared to the adjacent tooth structure.
	C) Change on shade or translucency values outside the tolerable compared to the adjacent tooth structure.
3. Marginal discoloration	A) No evidence of discoloration at the margin between tooth and crown cemented.
	B) Surface discoloration at the margin between tooth and crown cemented.
	C) Discoloration penetrating deeper in the margin between tooth and crown cemented.
4. Marginal adaptation	A) No visible evidence of crevice along the margin, without penetration of the explorer.
	B) Visible evidence of cracks along the banks where the explorer can penetrate.
	C) The explorer penetrates deeply into the cracks between the crown and preparation.
5. Surface roughness	A) Visually the surface is well polished, glossy, with no detectable roughness.
	B) The roughness is visually detectable.
	C) Roughness is coarse, visible, and detectable. The surface is opaque.
6. Anatomic form	A) Crown is continuous and anatomically preserved. The anatomic form is ideal.
	B) Anatomic form differs from the homologous tooth but does not affect appearance. Other irregularities in the dentition allow this to be esthetically and functionally acceptable.
	C) Anatomic form is altered and unsatisfactory, and the esthetic result is unacceptable. Replacement of the crown is necessary.
7. Secondary caries	A) No evidence of secondary caries at the margins of the preparation.
	B) With evidence of secondary caries at the margins of the preparation.
8. Occlusal contacts	A) Adequate
	B) Excessive
	C) Absent
9. Gingival index	0) Normal gingiva
	1) Mild inflammation
	2) Moderate inflammation
	3) Severe inflammation
10. Pulpal status	A) Vital
	B) Irreversible inflammation/necrosis
	C) Endodontic treatment
11. Chipping	A) Minor chipping (the chipping is smaller or equal to 2 × 2 mm, and no coping material is visible).
	B) Major chippings (the chipping is larger than 2 × 2 mm, or the coping material is visible).
	C) Fracture of the framework material.
12. Wear of the opposing teeth	A) Absent.
	B) Wear just on enamel or with dentin exposition in one point.
	C) Wear reaching dentin until 1/3 of the crown.
	D) Wear reaching dentin > 1/3 of the crown.
Abbreviation: USPHS, United States Public Health Service.	

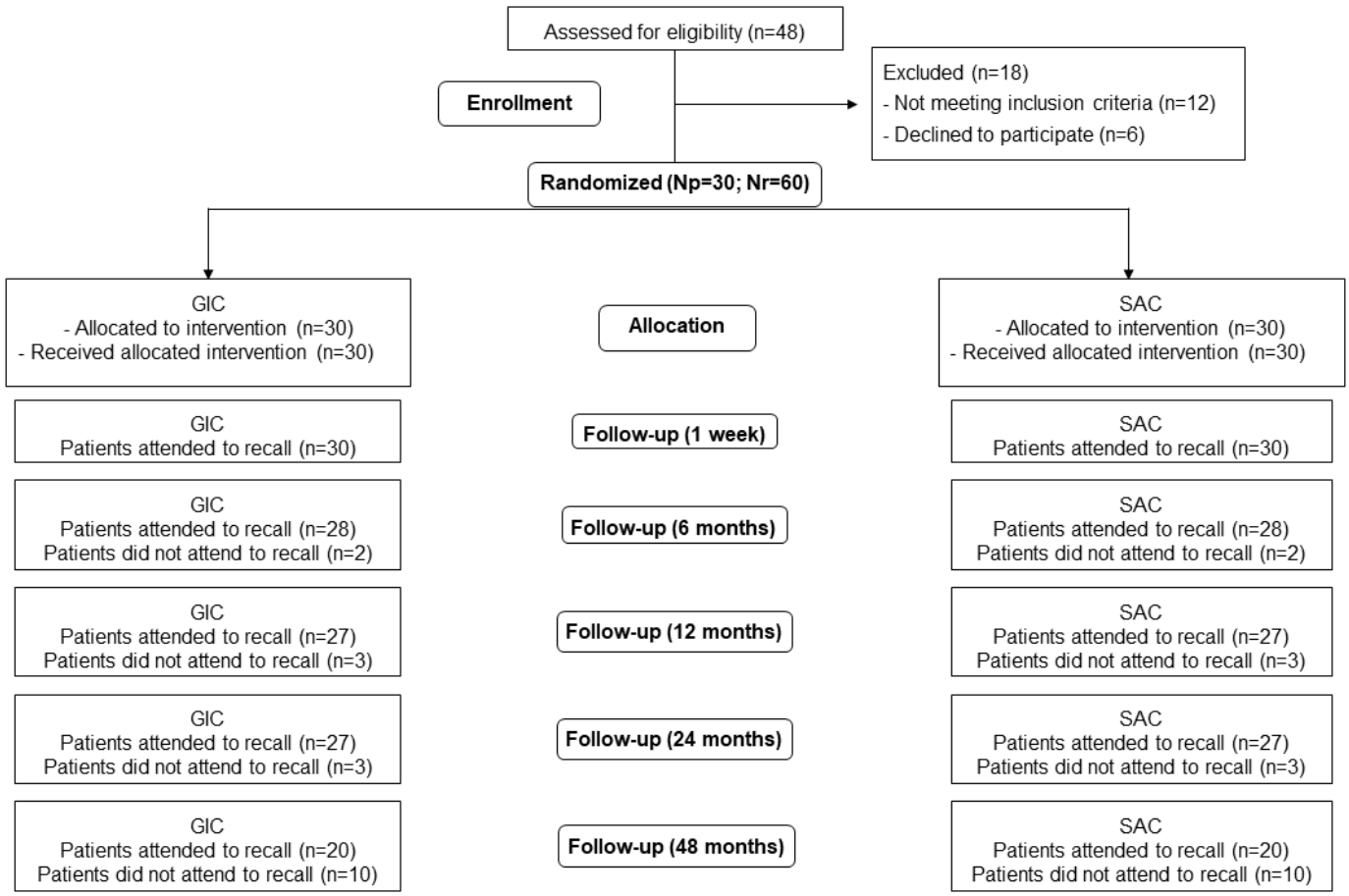

Figure 1. *Flowchart of the study (CONSORT). Abbreviations: CONSORT, Consolidated Standards of Reporting Trials; GIC, glass ionomer cement; SAC, self-adhesive resin cement.*

DISCUSSION

The clinical performance of the zirconia coping/press-on ceramic crowns cemented with either GIC or SAC presented similar clinical behavior after 48 months of evaluation. Both cements showed adequate retention rates and biological response for all the evaluated criteria, therefore the null hypothesis was accepted.

When considering the long-term clinical success of complete crown restorations, not only is the use of a strong material important but so is the retention to the remaining tooth structure produced by mechanical interlocking or adhesion of the cement to the tooth and the intaglio surface of the crown.[7] In addition, the thickness of the cementation interface, as well as the resistance of the cement to degradation in the oral environment is relevant. The presence of secondary caries around the margins is also a common cause of failure in indirect restorations,[11] and therefore the appropriate selection of cement becomes essential for clinical success. Although all-ceramic crowns, monolithic zirconia, or copings can be cemented with a variety of materials, differences in clinical outcomes of the different cements have not been fully demonstrated.[23]

A systematic review reported that some SACs have shown good clinical behavior similar to that of conventional resin cements[23] because the interaction between the cement acidic monomers and the hydroxyapatite form chemical bonds Oral Care, albeit with lower bond strength values, from a more superficial interaction with the tooth structure.[24,25] However, the self-adhesive material avoids a more complex bonding procedure, which is more susceptible to failures.[12,15] Various cement brands are available, with different formulations, which may have a great impact on their clinical behavior. The self-adhesive luting systems still need to be improved, since some in vitro studies reported that these materials presented issues regarding marginal adaptation and retention, which may be related to handling errors such as excessive drying of the cavity or residual moisture.[7,12,26]

The SAC used in the present study contained phosphate and phosphonate groups. An acidic pH provided by

Table 4. *Results of Clinical Evaluation*

Clinical evaluation		**Baseline**		**6 months**		**12 months**	
Criteria	Scores	n = 30	n = 30	n = 28	n = 28	n = 27	n = 27
		GIC	SAC	GIC	SAC	GIC	SAC
Retention	A	30 (100%)	30 (100%)	28 (100%)	28 (100%)	27 (100%)	26 (96.30%)
	B	–	–	–	–	–	–
	C	–	–	–	–	–	1 (3.70%)
Color match	A	30 (100%)	30 (100%)	28 (100%)	28 (100%)	27 (100%)	27 (100%)
	B	–	–	–	–	–	–
	C	–	–	–	–	–	–
Marginal discoloration	A	30 (100%)	30 (100%)	28 (100%)	28 (100%)	27 (100%)	27 (100%)
	B	–	–	–	–	–	–
	C	–	–	–	–	–	–
Marginal adaptation	A	30 (100%)	30 (100%)	28 (100%)	28 (100%)	27 (100%)	27 (100%)
	B	–	–	–	–	–	–
	C	–	–	–	–	–	–
Surface roughness	A	30 (100%)	30 (100%)	28 (100%)	28 (100%)	27 (100%)	27 (100%)
	B	–	–	–	–	–	–
	C	–	–	–	–	–	–
Anatomic form	A	30 (100%)	30 (100%)	28 (100%)	28 (100%)	27 (100%)	27 (100%)
	B	–	–	–	–	–	–
	C	–	–	–	–	–	–
Secondary caries	A	30 (100%)	30 (100%)	28 (100%)	28 (100%)	27 (100%)	27 (100%)
	B	–	–	–	–	–	–
Occlusal contact	A	30 (100%)	30 (100%)	28 (100%)	28 (100%)	27 (100%)	27 (100%)
	B	–	–	–	–	–	–
	C	–	–	–	–	–	–
Gingival index	0	21 (70%)	18 (60%)	23 (82.14%)	25 (89.30%)	23 (85.18%)	23 (85.18%)
	1	6 (20%)	10 (33.33%)	3 (10.71%)	2 (7.14%)	3 (11.11%)	3 (11.11%)
	2	3 (10%)	2 (6.67%)	2 (7.15%)	1 (3.56%)	1 (3.71%)	1 (3.71%)
	3	–	–	–	–	–	–
Pulpal status	A	18 (60%)	16 (53.33%)	18 (60%)	15 (53.57%)	17 (62.96%)	14 (51.85%)
	B	–	–	–	–	–	–
	C	12 (40%)	14 (46.67%)	10 (35.71%)	13 (46.43%)	10 (37.04%)	13 (48.15%)
Chipping	A	30 (100%)	30 (100%)	27 (96.43%)	28 (100%)	26 (96.30%)	26 (96.30%)
	B	–	–	1 (3.57%)	–	1 (3.70%)	1 (3.70%)
	C	–	–	–	–	–	–
	D	–	–	–	–	–	–
Wear of the opposite tooth	A	30 (100%)	30 (100%)	27 (96.43%)	28 (100%)	26 (96.30%)	26 (96.30%)
	B	–	–	1 (3.57%)	–	1 (3.70%)	1 (3.70%)
	C	–	–	–	–	–	–
	D	–	–	–	–	–	–

Abbreviations: GIC, glass ionomer cement; SAC, self-adhesive resin cement.

Table 4. Results of Clinical Evaluation (continued)

Clinical evaluation		**24 months**		**48 months**	
Criteria	Scores	n=27	n=27	n=20	n=20
		GIC	SAC	GIC	SAC
Retention	A	26 (96.30%)	26 (96.30%)	20 (100%)	20 (100%)
	B	–	–	–	–
	C	1 (3.70%)	1 (3.70%)	–	–
Color match	A	26 (100%)	26 (100%)	19 (95%)	19 (95%)
	B	–	–	1 (5%)	1 (5%)
	C	–	–	–	–
Marginal discoloration	A	26 (100%)	26 (100%)	17 (85%)	17 (85%)
	B	–	–	3 (15%)	3 (15%)
	C	–	–	–	–
Marginal adaptation	A	26 (100%)	26 (100%)	20 (100%)	20 (100%)
	B	–	–	–	–
	C	–	–	–	–
Surface roughness	A	26 (100%)	26 (100%)	20 (100%)	20 (100%)
	B	–	–	–	–
	C	–	–	–	–
Anatomic form	A	27 (100%)	26 (100%)	20 (100%)	20 (100%)
	B	–	–	–	–
	C	–	–	–	–
Secondary caries	A	26 (100%)	26 (100%)	20 (100%)	20 (100%)
	B	–	–	–	–
Occlusal contact	A	26 (100%)	26 (100%)	20 (100%)	20 (100%)
	B	–	–	–	–
	C	–	–	–	–
Gingival index	0	24 (92.30%)	25 (96.15%)	16 (80%)	16 (80%)
	1	2 (7.70%)	1 (3.85%)	2 (10%)	2 (10%)
	2	–	–	2 (10%)	2 (10%)
	3	–	–	–	–
Pulpal status	A	13 (41.85%)	14 (51.85%)	17 (85%)	16 (80%)
	B	–	–	–	–
	C	14 (51.85%)	13 (41.85%)	3 (15%)	4 (20%)
Chipping	A	26 (96.30%)	26 (96.30%)	19 (95%)	20 (100%)
	B	1 (3.70%)	1 (3.70%)	–	–
	C	–	–	1 (5%)	–
	D	–	–	–	–
Wear of the opposite tooth	A	26 (100%)	26 (100%)	20 (100%)	20 (100%)
	B	–	–	–	–
	C	–	–	–	–
	D	–	–	–	–

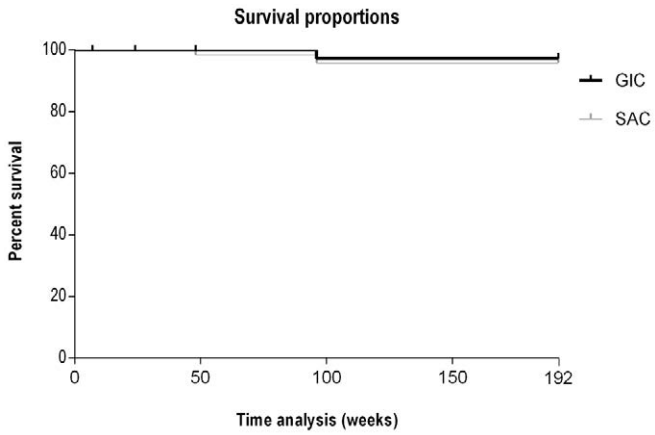

Figure 2. *Kaplan–Meier curve demonstrating survival rate of the cements for all-ceramic restorations with 95% confidence interval. Abbreviations: GIC, glass ionomer cement; SAC, self-adhesive resin cement.*

these molecules etched and simultaneously penetrated into the enamel and dentin.[24] These functional groups interact with calcium in the hydroxyapatite of enamel and dentin and can also react with the ceramic oxide surface without any pretreatment.[16,24,27] This could explain how the cement remained attached to the zirconia coping in the debonded crown in the present study (Figure 3F).

In the present study, most of the crowns (60% for GIC and 53.4% for SAC) were cemented on dentin. This substrate has been considered an adhesive challenge because of intrinsic moisture and its organic and inorganic composition. Nevertheless, SACs have shown stable adhesion to dentin because of their acidic monomers.[24,26-28]

The characteristics of higher viscosity and reduced wettability of the SACs in comparison with conventional adhesive systems explain the reduced bond strength of Bifix SE (15.28 MPa).[14,15] However, when analyzing the bonding of a cemented crown, the bonding of the cement to the tooth, and also to the intaglio surface of the ceramic coping, must be considered. The bond strength of the SACs RelyX U200 (3M Oral Care) and Bifix SE (Voco) to Y-TZP zirconia has been reported to be statistically similar.[27] However, the surface treatment of the ceramic before luting has a significant effect, and airborne-particle abrasion has been recommended.[27] The cement tested in the current clinical study (Bifix SE) showed a bond strength of 12.03 MPa to the Y-TZP zirconia after airborne-particle abrasion. The SAC seems to be an effective alternative for dentin substrate and also for zirconia.[28,29] However, these studies were in vitro, and clinical trials are scarce.

GIC is also expected to bond to the tooth structure by molecular interactions between the polyacrylic acid and the calcium of the mineralized tissues, thus providing a strong chemical bond to dentin and enamel with low solubility.[11] However, bonding to an artificial core or coping material is lacking.

The loss of retention rate observed in the present study of only 3% both for GIC and SACs was lower than the 7% previously reported in a practice-based retrospective study[30] with zinc phosphate and SAC. Only 2 of 60 crowns, 1 after one year (resin cement) and the other between 12 and 24 months (GIC) failed, evidencing the good performance of the tested materials during the study period.

Reducing the convergence angle and increasing the surface area of the tooth preparation increases

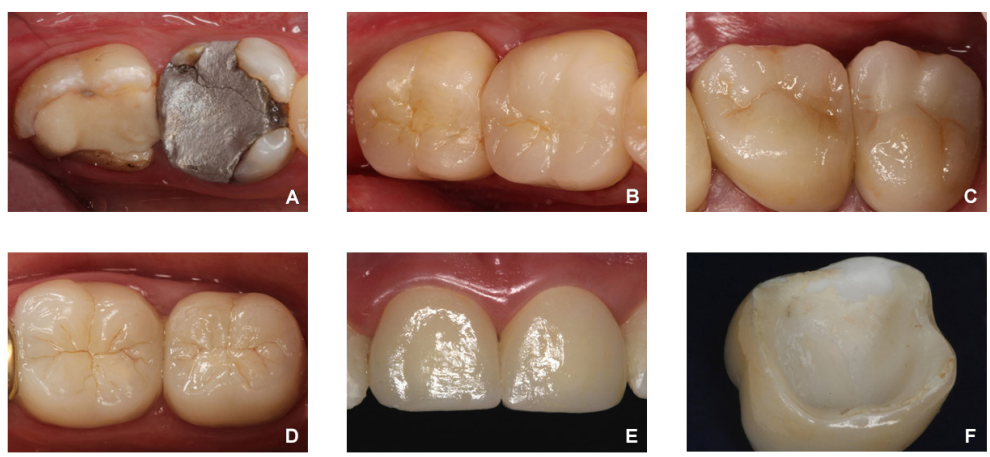

Figure 3. *Clinical pictures of two adjacent crowns. (A), (B): Clinical aspect before the treatment and after 4 years. (C)–(E): Aspects of the other patients with two adjacent crowns after 4 years. (F): Debonded crown showing that most of the self-adhesive resin cement remained bonded to the crown.*

mechanical retention for the crown.[18,19] An optimal tooth preparation, which is important in order to avoid debonding with conventional cements, is also acceptable with any other cement, including the adhesive ones.[25] In the present study, a similar clinical performance was observed for the 2 cements. The mechanical retention provided by the preparation may have been sufficient to retain the crowns despite the bonding characteristics of the cements and could explain the results. Perhaps, in challenging situations such as short crowns or excessively tapered preparations, the role of bonding would increase and have a greater influence on the results.[31]

Chipping is one of the most common clinical issues related to ceramics. This is a complex clinical situation, with little evidence about its mechanisms.[8] The crowns will still be clinically acceptable after polishing when the fracture is not severe.[32] In the present study, only minor chipping was observed, with a low incidence after 4 years (5% for only 1 crown cemented with GIC), although no relationship was found between the type of cement and crown chipping. Studies have been performed to investigate this occurrence, and several reasons have been suggested, such as surface defects of the ceramic, improper design of the zirconia framework,[33] overloading, fatigue, low fracture toughness,[34] low thermal conductivity,[32] and even parafunctional activity that was not detectable.[8] A previous study has also reported a low percentage of chipping (6.6%) in the first year.[8]

In the present study, no biological complication was encountered in the period of evaluation. Although some marginal discoloration was observed after 48 months (15% for both cements), proper marginal adaptation was observed. Secondary caries were also not detected, which could be associated with the reliable sealing provided by the luting materials and directly related to individual caries risk.[11,35] Although better caries inhibition would be expected for the GIC, nonsignificant differences were observed in relation to the resin cement. In fact, regardless of the material used, individual caries risk seems to be the major determinant for the development of secondary caries.[11] Moreover, marginal adaptation is considered one of the principal criteria for assessing the long-term success of tooth restorations.[36,37] Studies with more than 5 years of follow-up have shown the absence of secondary caries when good marginal fit and reliable sealing provided by cements are present.[8,17,30]

The present study showed that luting ceramic crowns with either GIC or SAC presented satisfactory performance. After 48 months, the survival rate was 95.8%, which is consistent with the rates of previous clinical follow-up studies.[35,38] Different results may be obtained in future recalls, because clinical factors such as aging and masticatory loads may influence the long-term behavior of the restorations.

CONCLUSIONS

Ceramic crowns cemented with GIC or SAC presented with similar behavior after 48 months of clinical use. Both cements showed adequate retention rates, aesthetic and functional outcomes, and biological response.

Regulatory Statement

This study was conducted in accordance with all the provisions of the human subjects oversight committee guidelines and policies of the Institute of Science and Technology. The approval code issued for this study is 20722713.0.0000.0077.

Conflict of Interest

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

(Accepted 29 May 2020)

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In Vivo Pulp Temperature Changes During Class V Cavity Preparation and Resin Composite Restoration in Premolars

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Clinical Relevance

Pulp cooling due to the Class V preparation in premolars and subsequent to etch & rinse bonding has a protective effect against the heat generated during the resin composite exposure to the curing light, regardless of the restorative procedure.

SUMMARY

Objective: This *in vivo* study evaluated the influence of the sequence of all restorative steps during Class V preparation and restoration in human premolars on pulp temperature (PT).

Methods and Materials: Intact premolars with orthodontic extraction indication of 13 volunteers received infiltrative anesthesia and isolation with rubber dam. An occlusal preparation was made with a high-speed diamond bur under air-water spray until the pulp was minimally exposed, then a thermocouple probe was inserted within the pulp. A deep, 2.0-mm depth Class V preparation was made using a high-speed diamond bur under air-water spray. Three restorative techniques were performed (n=7): Filtek Z250 placed in two increments (10-second exposure,

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http://doi.org/10.2341/20-098-C

shade:A2, 3M ESPE, St. Paul, MN, USA), Filtek Z350 XT (40-second exposure, shade:A3D, 3M ESPE) and Tetric N Ceram Bulk Fill (10-second exposure, shade:IVA, Ivoclar Vivadent, Schaan, Liechtenstein), both placed in a single layer. Bonding layer and resin composite were exposed to light from the same Polywave LCU (Bluephase 20i, Ivoclar Vivadent). The peak PT and the difference between peak PT and baseline (ΔT) values were subjected to two-way, repeated measures analysis of variance (ANOVA), followed by the Bonferroni post-hoc test (α=0.05).

Results: Cavity preparation and etch & rinse procedures decreased the PT values ($p<0.001$). The 40-second exposure of Filtek Z350 caused the highest peak PT values (38.7±0.8°C) and the highest ΔT values (3.4±0.8°C), while Tetric N Ceram Bulk Fill showed the lowest values (-1.6±1.3°C; $p=0.009$).

Conclusion: None of the evaluated procedures resulted in a PT rise near to values that could offer any risk of thermal damage to the pulp.

INTRODUCTION

Restorative procedures incorporating the use of light activated resin composites (RC) are widely used. In order to optimize clinical procedures with minimal chairside time, LED light-curing units (LCUs) with irradiance values over 2000 mW/cm² are available.[1] In this context, *in vitro* studies evaluating restorative procedures using RCs on extracted teeth have shown temperature increases within the pulp chamber or under dentin disks close to or even higher than 5.5°C, a temperature rise considered harmful to the pulp.[2-4] Such an elevation in pulp temperature (PT) has been attributed mostly to heat caused by the curing light itself and partly to the influence of the RC curing exotherm.[5-8] Therefore, the PT rise is influenced by preparation depth, LCU type, incident irradiance, and distance from light tip to the target, as well as by the thermal and exothermic properties of the RCs themselves.[3,4,9]

Although such *in vitro* studies brought important knowledge about the impact of high power LED devices and RCs on PT, the effects of a restorative procedure incorporating the use of light-activated RCs and the use of air-driven handpieces with water irrigation on PT rise is much more complex. For instance, some studies show that cooling methods, such as air-water spray or only air spray when using slow- and high-speed handpieces, can reduce heat generated during cavity preparation.[10,11] Indeed, *in vivo* studies performed in dogs and humans indicate an average PT drop of 8.1°C and 5.8°C, respectively, after a Class V cavity preparation was made on the buccal surface using a high-speed diamond bur under constant cooling water.[12,13] Moreover, PT values continue to drop, even after the cavity preparation had been completed.[12] Based on this evidence, it should also be expected that rinsing off phosphoric acid when applying an etch & rinse adhesive system to a cavity preparation would maintain low PT values, despite the lack of any evidence regarding this aspect. As a consequence, it is reasonable to assume that the pulp would already be in a cooled state at the moment when the tooth is first exposed to a curing light, and heat caused by light during a restorative procedure would not be as high as previously shown.[14,15] However, to the extent of our knowledge, there is no literature showing the impact of all preparation and restorative steps performed on *in vivo* temperature changes in human pulp during a typical Class V RC restoration placement in premolars.

The purpose of this *in vivo* study was to evaluate the influence of the sequence of all clinical steps performed during preparation and restoration of Class V cavities in human premolar teeth on the real-time intrapulpal temperature: high speed, water-cooled, instrumentation, etch & rinse bonding procedure and light-curing, and insertion and photocuring of RCs. The research hypotheses tested were: 1) immediately following tooth preparation and following bonding procedures, PT values will be significantly lower than the baseline, physiological values (prior to tooth preparation); and 2) the levels of PT decrease resulting from cavity preparation and bonding agent procedures will not prevent PT values from increasing higher than the baseline temperature during restorative procedures using different products and filling/curing techniques.

METHODS AND MATERIALS

Thirteen patients (seven males and six females) requiring extraction of first premolars for orthodontic reasons were recruited from the local orthodontic specialization program. Patient inclusion criteria included: 1) treatment plans indicating premolar extractions for orthodontic reasons, 2) the presence of healthy, intact, non-carious, and non-restored, fully erupted treatment teeth, and 3) patients with well-controlled health conditions that allowed all procedures involved in the research to be performed with minimal risk. Exclusion criteria included: 1) those patients who did not agree to volunteer for the study. The subjects, ranging from 12 to 30 years, went through an initial clinical exam at the State University of Ponta Grossa clinics, after which the research

methodology and study aims were explained. After informed consent was obtained, volunteers received both infiltrative and intraligamental injections using a local anesthetic (2% mepivacaine hydrochloride [36 mg], containing 1:100,000 epinephrine [18 microL]; Mepiadre, DFL Industria e Comercio, Rio de Janeiro, RJ, Brazil) when upper premolars were evaluated, while an inferior alveolar nerve block was used when procedures were performed on lower premolars.

The *in vivo* methodology to evaluate PT increase was described in previous studies,[12,16,17] and is illustrated in Figure 1. Prior to probe placement, two calibrated temperature probes were connected to a wireless temperature acquisition system (Temperature Data Acquisition-Thermes Wifi, Physitemp, Clifton, NJ, USA) and were kept immersed in 0.9% sterile saline solution at room temperature until pulp exposure was obtained. After receiving anesthesia and prior to the occlusal preparation and pulp exposure, the teeth were isolated with a rubber dam and a small amount of RC was bonded on the tip of the buccal cusp where the probe rested to adjust the probe position so the

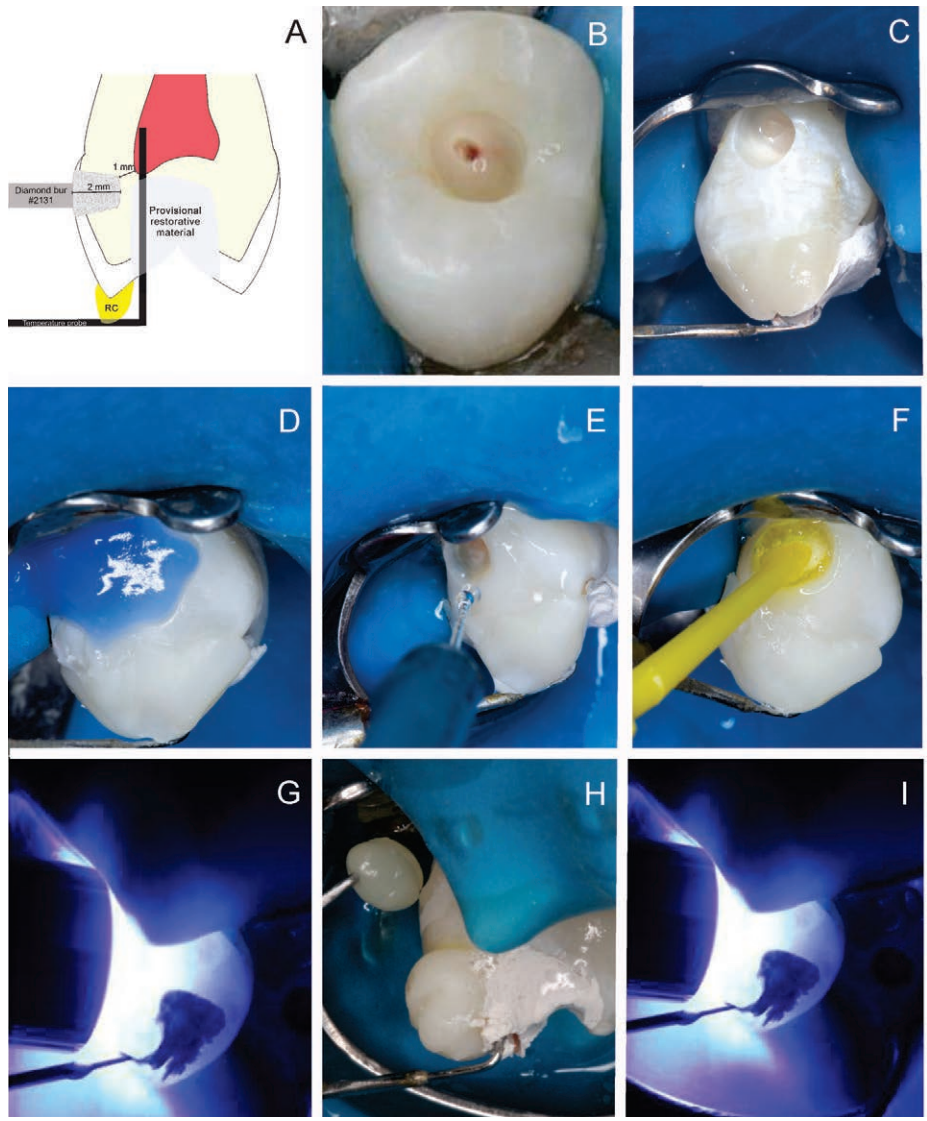

Figure 1. Representative images of temperature measurement in the dental pulp during restorative procedure and exposure to LED light. (A) Illustrative drawing showing the probe position and the shape of cavity preparation based on the diamond bur shape; (B) Deep, occlusal cavity preparation to produce a small pulp exposure; (C) Premolar with probe inserted into the pulp chamber through the occlusal cavity, sealed with a provisional restorative material and a deep Class V preparation; (D) Acid etching with 35% phosphoric acid gel; (E) Rinsing with water to remove acid; (F) Adhesive system applied according to the manufacturer's recommendations; (G) LED LCU tip positioned against the buccal surface of the tooth; (H) Increment of RC placed within the cavity preparation; (I) Polymerization of the RC layer. Abbreviations: LCU, light curing unit; LED, light-emitting diode; RC, resin composite.

probe tip could be as close as possible to the axial wall of the buccal, Class V preparation (Figure 1A). A small, occlusal preparation was made in the center of the premolar, using a round diamond bur (#1015, KG Sorensen, Cotia, São Paulo, Brazil) in a high-speed handpiece, with controlled air-water spray, until the pulpal floor was near the buccal pulp horn. A pulp exposure small enough for only the probe to fit into the pulp chamber through the buccal pulp horn (Figure 1B), with no pulp bleeding, was carefully obtained using a small, pencil-shaped diamond bur (#2134; KG Sorensen). One probe was removed from the saline solution and inserted immediately within the pulp tissue through the access hole. The probe was positioned to remain stable in a small groove previously created on the top of the cusp tip. The probe remained at a predetermined depth of approximately 4 mm within the pulp chamber, as close as possible to the axial wall of the buccal, Class V preparation, while PT was measured. The occlusal cavity was filled using a provisional restorative material (Cavitec, Caithec LTDA, Sao Jose dos Pinhais, PR, Brazil) to ensure probe stabilization and to reduce the heat that could dissipate through the cavity, as previously described (Figure 1A).[12,16] The other probe was maintained in the saline solution at room temperature (approximately 22.0°C) as a reference for validation of system operation. Room temperature was stabilized throughout the procedure, being controlled by air conditioning set to approximately 22°C. After probe stability was confirmed, real-time temperature data were continuously acquired for approximately 15 minutes, until a stable, baseline PT (approximately 35.3°C) was reached. Afterward, a controlled-size, Class V tooth preparation was made on the buccal surface (Figure 1C), using a diamond bur (#2131; KG Sorensen) in a high-speed handpiece with three water-emitting apertures (0.5 mm in diameter, Roll Air 3, KaVo Dental GmbH, Biberach, Baden-Württemberg, Germany), under controlled amounts of air-water spray (33 ml/min) at approximately 22°C. The preparation size was approximately 2.5 mm in diameter and 2.0 mm in depth, based on the bur dimensions, leaving approximately 1 mm thick dentin remaining between the axial wall and the pulp chamber (Figure 1A). A periodontal probe was used to confirm the preparation size of every cavity preparation.

In Vivo Restorative Procedures Using Light-activated RCs

Teeth were randomly assigned to groups according to the product (n=7). Manufacturer-recommended adhesive systems (Single Bond Universal, 3M ESPE, St Paul, MN, USA, and Tetric Nano Bond, Ivoclar Vivadent, Schaan, Liechtenstein) were applied and light-cured (Figure 1) according to the manufacturers' instructions (Table 1). In this regard, the Class V preparation was acid etched with 35% phosphoric acid for 15 seconds (Figure 1D), and the acid was removed with air-water spray for 15 seconds (Figure 1E). The bonding agent was applied according to the manufacturers' instructions (Table 1) (Figure 1F), and an air jet was gently applied to evaporate the solvent content until no adhesive accumulation was noticed. The adhesive layer was exposed to light curing for 10 seconds (Figure 1G) and the cavity was filled with RC composite according to each experimental group (Figure 1H). In order to simulate the effects of an incremental technique on PT values in a shallow cavity preparation such as that evaluated in the current study, Filtek Z250 (Shade: A2, 3M ESPE) was placed in two 1-mm thick increments, with each layer exposed to a dental curing light for 10 seconds (radiant emittance: 1230 mW/cm^2, Bluephase 20i, Ivoclar Vivadent). However, only PT values after the second exposure were compared to those obtained in the other products. A bulk-fill composite, Tetric N Ceram Bulk Fill (Shade: IVA, Ivoclar Vivadent), was placed in a single 2-mm thick increment, which was exposed to the same curing light for 10 seconds (Figure 1I). Finally, the cavity preparation was bulk filled (an approximtely 2-mm thick layer) with an opaque, darker shade composite comprising a longer, 40-second exposure following the manufacturer's instruction (Filtek Z350 XT, Shade: A3D, 3M ESPE) in order to observe pulp temperature change to an extreme in LCU exposure.

The PT values, expressed in Celsius degrees (°C), were obtained in real-time, every 0.2 seconds by a data acquision software program (DASYLab 11, Measurement Computing Corp, Norton, MA, USA). At the end of the analysis, the thermocouple probe was removed, and the teeth were atraumatically extracted. The probe was reinserted to simulate the intraoral position, and X-rays were taken to confirm placement as well as the thickness of the remaining dentin wall. The peak PT values during cavity preparation, bonding procedures, and exposure of bonding agents and RC to the curing light were recorded. In addition, the pre-preparation, physiologic baseline PT values were subtracted from the peak PT values to determine the PT range (ΔT) values during each restorative step. To determine the interval between each restorative step, the time into the data acquisition when each procedure was performed was recorded.

The radiant emittance values (mW/cm^2) of the LCU were obtained after spectral power analysis using a spectroradiometer (USB 2000, Ocean Optics,

Table 1: *Products, Manufacturer, and Instructions for Use*

Product Classification	Product (Shade)	Manufacturer	Composition (Supplied by Manufacturer)	Instructions for Use[a]	Filler Content/ Volume (%)
Dentin Bonding Agents	Single Bond Universal	3M ESPE	2-hydroxyethyl methacrylate; bisphenol A diglycidyl ether dimethacrylate (Bis-GMA); 2-propenoic acid, 2-methyl-, reaction products with 1,10-decanediol and phosphorous oxide (P2O5); ethanol; water; copolymer of acrylic and itaconic acid; camphorquinone; dimethylaminobenzoat(-4)	a; b	—
	Tetric N-Bond	Ivoclar Vivadent	Bis-GMA; ethanol; 2-hydroxyethyl methacrylate; phosphonic acid acrylate; urethane dimethacrylate; Aquatic Chronic 3, diphenyl(2,4,6-trimethylbenzoyl) phosphine oxide;	a; c	—
Incrementally Filled Copmosites	Filtek Z350 XT (A3D)	3M ESPE	Silane treated ceramic; silane treated silica; urethane dimethacrylate (UDMA); Bisphenol A polyethylene glycol; Diether dimethacrylate; Bisphenol A diglycidyl ether; Dimethacrylate (Bis-GMA); Silane treated zirconia polyethylene glycol dimethacrylate triethylene glycol dimethacrylate	d	63.3
	Filtek Z250 (A2)	3M ESPE	Silane treated ceramic; Bisphenol a polyethylene glycol; Diether dimethacrylate (Bis-EMA); Diurethane dimethacrylate (UDMA); Bisphenol A diglycidyl ether; Bis-GMA; Triethylene glycol dimethacrylate (TEGDMA); Aluminum oxide	e	60
Bulk-Fill Composite	Tetric N-Ceram Bulk-fill (IVA)	Ivoclar Vivadent	Bis-GMA; UDMA; ytterbium trifluoride; Bis-EMA; Aquatic Chronic	f	61

Abbreviations: Bis-EMA, ethoxylated bisphenol-A dimethacrylate; Bis-GMA, bis-phenol A diglycidyl ether dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, diurethane dimethacrylate.

[a] a) Pre-condition with phosphoric acid (35 - 37 %) of enamel and dentin for 15 s, rinse and air-dry without dehydration; b) Apply the adhesive with a microbrush, rubbing for 20 s. Apply air jet for 5 s, light cure for 10 s (Bluephase 20i – High Power); c) Apply the adhesive with the aid of a microbrush, rubbing gently for at least 10 s. Apply air jet until no adhesive accumulation is noticed, light cure for 10 s (Bluephase 20i – low mode: 656 mW/cm^2); d) Apply increment of up to 2 mm, adapt with a spatula and light cure for 40 s (Bluephase 20i – High Power); e) Apply increment of 1 mm, adapt with a spatula and light cure for 10 s (Bluephase 20i – High Power); f) Apply increment of up to 4 mm, adapt with a spatula and light cure for 10 s (Bluephase 20i – High Power)

Dunedin, FL, USA) connected to a 6-inch integrating sphere (Labsphere, North Sutton, NH, USA). The measurement system was calibrated using a light source traceable to NIST standards. The LCU tip end was positioned at the entrance of the integrating sphere, so all light emitted from the unit was captured. Wavelength-based, spectral power emission was recorded between 350 nm and 550 nm using software (SpectraSuite v2.0.146, Ocean Optics), which also provided a total emitted power value for that wavelength range and the spectral emission profile as well (Figure 2). The optical emitting area of the distal end of the light guide was calculated, and this value was divided into the integrated spectral power value to derive the total radiant emittance.

Statistical Analyses

The homogeneity test of variances and Shapiro Wilk normality tests were performed for the dependent variables peak PT, ΔT data, and interval between procedures, with both parameters passing. Thus, the peak PT and ΔT values were subjected to two-way, repeated measures analysis of variance (ANOVA) (independent variables "product" and "restorative steps"), followed by the Bonferroni *post-hoc* test. The interval between each restorative step was compared among products using one-way ANOVA followed by the Tukey *post-hoc* test. All statistical testing and *post hoc* power analysis were performed at a preset α of 0.05, using commercial statistical software (Statistics 19, SPSS Inc., IBM Company, Armonk, NY, USA).

RESULTS

For the number of evaluated teeth (n=7), the *in vivo* study was adequately powered for both peak PT and ΔT values (above 99.0%; α=0.05), which are shown in Tables 2 and 3, respectively. Cavity preparation caused a significant drop in PT values ($p<0.001$), and a further decrease in PT values was also noted after the etch & rinse bonding procedures ($p<0.001$). A significant increase in PT values was observed after the bonding agent and after the resin composite layers were exposed to LED light ($p<0.001$). The 40-second exposure of Filtek Z350 (38.7±0.8 °C) caused the highest peak PT values ($p<0.001$), while the PT values after the exposure of the second layer of Filtek Z250 were not different from the baseline, physiological PT values. The PT values after the exposure of Tetric N Ceram Bulk Fill to curing light were still significantly lower than the baseline PT values ($p=0.002$).

The ΔT values were negative after LED exposure in most scenarios (Table 3). No significant difference in ΔT values was observed among products either after cavity preparation or after the bonding procedures. The 40-second exposure of Filtek Z350 to curing light caused the highest ΔT values (3.4±0.8°C), which were significantly higher than those after the 10-second exposure of Filtek Z250 (0.3±0.5°C) ($p<0.001$), which in turn showed significantly higher ΔT values than did Tetric N Ceram Bulk-Fill (-1.6±1.3°C; $p=0.009$).

Figure 3 shows the representative profiles of PT changes during the restorative steps. The drop in PT values during cavity preparation and etch & rinse procedures was observed in all groups. A slow

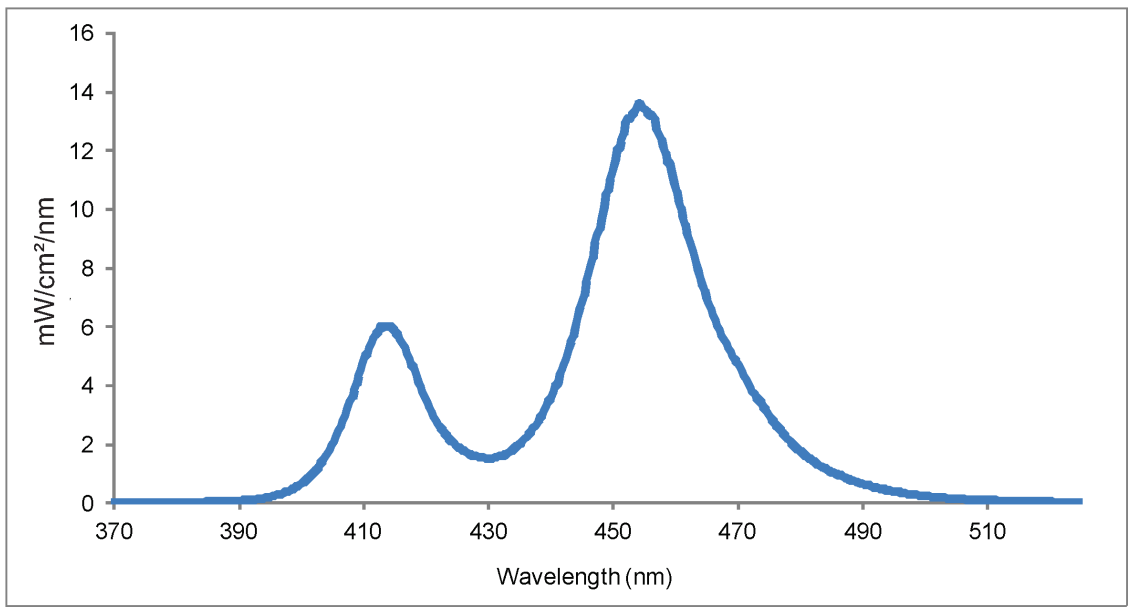

Figure 2. *Spectral emission profile of Bluephase 20i*

Table 2: *Mean (standard deviation) of* in vivo *pulp temperatures subsequent to specific restorative procedures*[a]

	Filtek Z250 Two Increments 10 s	**Tetric N Ceram Bulk-fill One Increment 10 s**	**Filtek Z350 One Increment 40 s**
Baseline temperature	35.0 (0.6) Aa	35.1 (0.9) Aa	34.9 (0.7) Ba
Cavity preparation	29.8 (1.2) Ca	27.9 (1.8) Ea	28.2 (2.1) Ea
Etch-and-rinse bonding procedures	30.2 (1.1) Ca	29.6 (1.6) Da	29.8 (1.4) Da
Exposure of bonding agent to LED light	32.2 (1.0) Ba	31.1 (1.6) Ca	32.2 (1.1) Ca
Exposure of RC layer to LED light	35.6 (0.6) Ab	33.9 (1.5) Bb	38.4 (1.0) Aa

Abbreviations: LED, light-emitting diode; RC, resin composites.

[a] *Means followed by similar letters (lower case: within row; upper case: within column) are not significantly different (α=0.05).*

Table 3: *Pulp Temperature (°C) Range with Respect to the Pre-preparation, Physiologic Baseline Temperature (ΔT) After Curing Light Exposure at Three Restorative Moments into the Restorative Sequence*[a]

	Filtek Z250 Two Increments 10 s Mean (SD)	**Tetric N Ceram Bulk-fill One Increment 10 s Mean (SD)**	**Filtek Z350 One Increment 40 s Mean (SD)**
Cavitypreparation	-5.3 (1.2) Aa	-7.2 (1.0) Aa	-6.8 (2.2) Aa
Etch & rinse bonding procedures	-4.9 (1.1) Aa	-6.0 (1.0) Aa	-5.4 (1.7) Ba
Exposure of bonding agent to LED light	-2.8 (0.8) Ba	-4.3 (1.1) Ba	-2.8 (1.1) Ca
Exposure of RC layer to LED light	0.3 (0.5) Cb	-1.6 (1.3) Cc	3.4 (0.8) Da

Abbreviations: LED, light-emitting diode; RC, resin composites.

[a] *Mean (SD) followed by similar letters (lower case: within row; upper case: within column) are not significantly different (pre-set α=0.05).*

PT increase was noted while the bonding agent was applied, followed by a small PT drop during the gentle airflow to evaporate the organic solvent. A quick but low rise in PT values was seen while the bonding agent layer was exposed to light curing. PT values increased rapidly during the exposure of RC layers to curing light. Small PT peaks were observed during short exposures such as a 10-second exposure (Figures 3A and 3B), while a higher PT peak was noted during the 40-second exposure (Figure 3C). When the Filtek Z250 was applied using the incremental technique and each RC layer was light-cured separately, the peak PT observed after the exposure of the first increment to curing light was apparently lower than that observed after the exposure of the second increment.

Figure 4 shows the comparison of intervals among products within each interval between restorative steps. Overall, no significant difference in most intervals between steps was noted among products. The interval between RC placement and light curing was shorter when Z250 was applied than when the preparation was filled with Tetric N Ceram Bulkfill (p=0.007).

DISCUSSION

To the extent of the authors' knowledge, this is the first *in vivo,* human study that evaluated the impact of PT drop caused by cavity preparation on the PT increase during restorative procedures with light-activated RCs. As previously shown *in vitro*[11] and *in vivo,*[12] cavity preparation under constant air-water spray caused a significant drop in PT values to approximately 29°C. A slow PT increase was then observed after cavity preparation, followed by another significant drop in PT due to the etch & rinse procedure (Figure 3). Therefore, the first research hypothesis was accepted. Although these results corroborate previous findings,[11,12,18] it is important to emphasize that heat production during cavity preparation depends on many factors, such as bur shape, type, and size; abrasiveness; wear of the diamond coating; pressure applied by the operator;

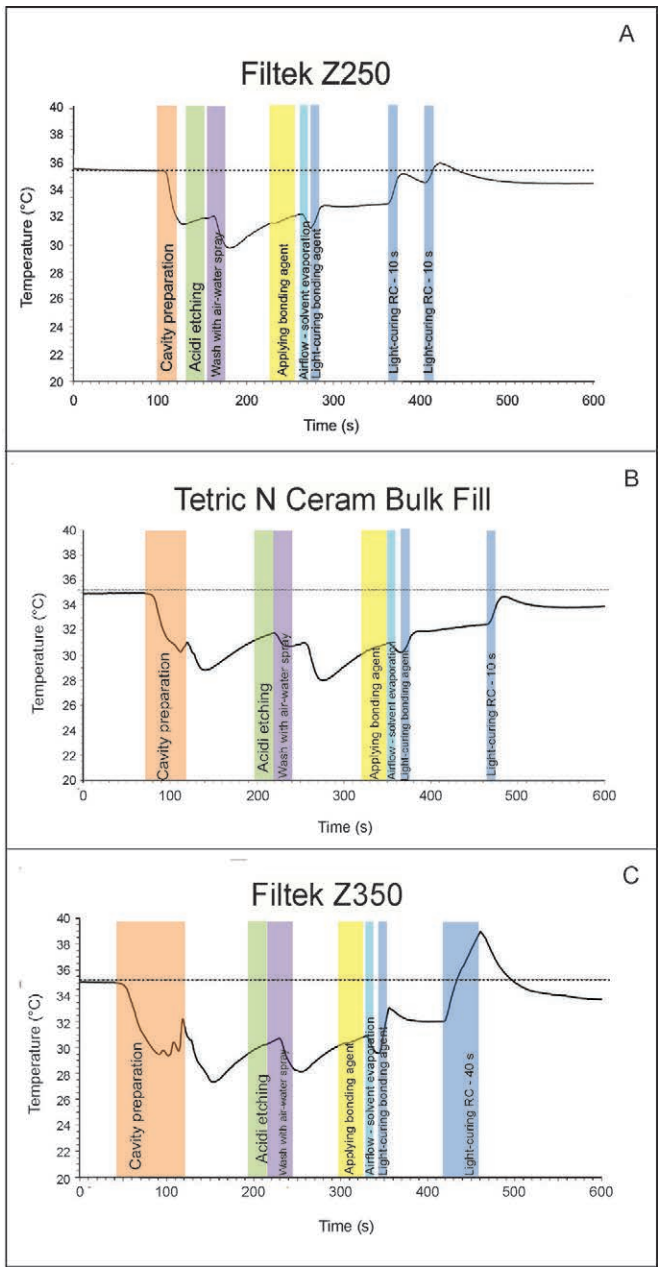

Figure 3. *Representative real-time profiles of PT changes during restorative procedures for each material examined: (A) Filtek Z250 (10 s); (B) Tetric N Ceram Bulk Fill (10 s; and (C) Filtek Z350 XT (40 s). Abbreviation: PT, pulp temperature.*

grinding time and rate; amount of debris clogging on the grinding surface; and speed and torque of the rotatory instrument.[18,19] However, it has also been shown that air-water spray has a major impact on the temperature within the pulp chamber when a high rate of air-water spray is applied.[11,18] Thus, water temperature[20] and flow rate[10] play an important role in controlling pulp temperature during tooth preparation. In this regard, the air-water spray used in the current study (33 ml/min) is within the range (30-50 ml/min) considered effective in reducing the temperature within the pulp chamber during the cavity preparation.[10,11] Therefore, because a high flow rate is applied, such outcomes should be expected, regardless of the bur type used.

Despite the increase in PT values after the etch & rinse procedure, due to the time lapse between that step and the subsequent light exposure of the bonding agent layer (Figure 4), the PT values were still lower than the baseline values, even after the first exposure of the bonding agent to curing light: approximately 32°C (Figure 3). In contrast to this finding, great concern has been shown by some authors as their results show that heat created during this step would be capable of inducing pulpal damage in deep preparations, due to the reported high temperature increase within the pulp when no RC layer was present to act as an insulator.[21-23] However, in those *in vitro* studies, the influence of cooling water during the cavity preparation or inclusion of an etch & rinse procedure, or the influence of airflow applied to evaporate the solvent were disregarded. Therefore, under clinical conditions, such as those used in the current methodology, light exposure of the bonding agent does not seem to offer any risk of pulp damage. Nonetheless, it is important to notice that the current methodology did not include the use of low-speed burs to remove caries, and only etch & rinse bonding agents were used. Because the use of low-speed burs without any cooling can increase PT to values near those considered harmful for the pulp[2,13] or even higher,[24] and the etch & rinse procedure is not applied when a self-etching bonding agent is used,[25] the current results should not be extrapolated to such other clinical scenarios. Further investigation is required to address those issues.

As clearly seen in the analysis of peak PT and ΔT values (Tables 2 and 3), only the 40-second exposure of Filtek Z350 increased PT to values exceeding the pre-preparation baseline PT, when high radiant exposure values were delivered (approximately 49.2 J/cm^2). Thus, the second research hypothesis was partially rejected. Such a great amount of energy delivered to an RC restoration is not usual in the daily clinical routine, as most manufacturers usually recommend shorter exposure intervals, such as 10 seconds or 20 seconds. The impact of the heat generated during an RC restoration on PT values depends on many factors: curing light irradiance, exposure time, RC properties (enthalpy of polymerization, thermal conductivity, density, heat capacity, reflectance, and light penetration depth), restoration size, amount of remaining dentin, presence of thermal barrier layers, and convective heat loss.[23] However, among these factors, the amount and

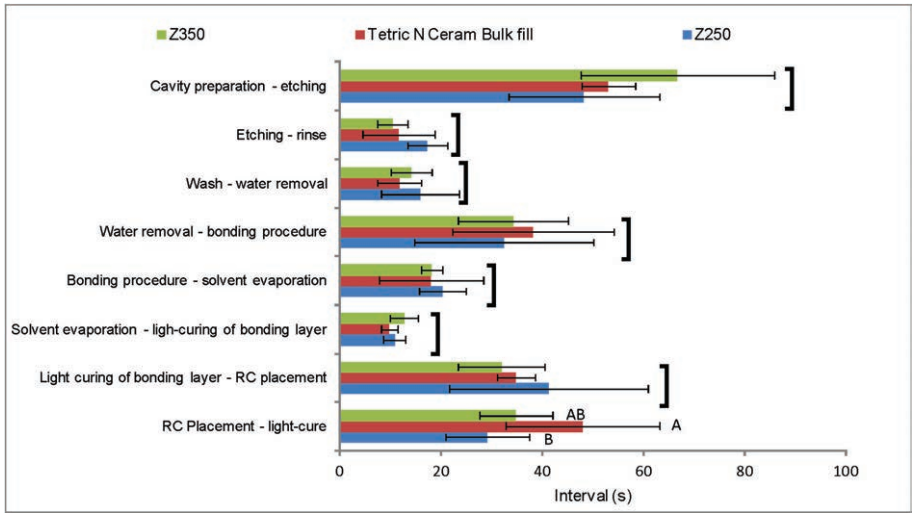

Figure 4. *Bar graph showing the average interval between restorative steps for each product. Means with different uppercase letters are significantly different (pre-set α=0.05). Means without significant difference are connected by brackets. Only comparisons among products within each interval were made.*

rate of the energy absorbed during irradiation have been considered the main factors for PT increase,[15,26] as also observed in this study. In addition, based on the current findings, the temperature of the tooth substrate and pulp also seemed to have a substantial impact on the influence of heat on PT values. In other words, any drop in dentin and enamel temperature due to the etch & rinse procedure[27] creates a gradient temperature between the cooled dentin walls and the pulp, so the cooled dentin acts as a heat sink that not only lowers the PT values but also absorbs some of the heat generated during light exposure, allowing the system to reach thermodynamic equilibrium. For this reason, a greater amount of energy, such as that delivered after 40-second exposure of Z350, was required to cause higher PT peak values than the physiological, baseline values, as noticed in the current study.

The influence of heat released from the RC layer has gained more attention with the introduction of bulk-fill RCs. Due to the exothermic nature of RC polymerization[28] and the greater volume of polymerizing composite when a thick layer of bulk-fill RC is placed within a deep cavity preparation, these resins generate more heat during light-curing than do conventional RCs.[14,29] Despite that aspect, no significant difference was noted in the peak PT values between teeth restored using Tetric N Ceram Bulk Fill and those restored using multiple thin increments of Filtek Z250. One could state that only a 2-mm thick layer of Tetric Ceram Bulk Fill was evaluated, so a greater volume of the bulk-fill RC would cause higher PT increase. In contrast to this assumption, previous research demonstrates that increasing the thickness of the RC layer resulted in a lower increase in temperature values within the pulp chamber.[30] The authors attributed those findings to the greater insulating effect provided by the thicker resin layers against the heat caused by the LCU. Thus, based on the current findings, the capacity of thicker RC layers to act as insulators and reduce the PT increase is greater than the influence of the heat from the exothermic polymerization on the PT values. Such a finding has also been seen *in vitro* when bulk-fill RCs were evaluated.[31]

Although the Class V preparations evaluated were considered deep because the axial wall was close to the pulp, cavity dimensions were not as large as those of Class I or Class II preparations. For this reason, the outcomes should not be extrapolated to those clinical scenarios, where multiple layers of resin composite and successive exposures to LCU are required. Moreover, because the thickness of remaining dentin plays an important role acting as a thermal insulator[32] due to its low thermal conductibility,[33] thicker or thinner remaining dentin layers could lead to different outcomes, as previously shown.[32] It should be also noticed that this study was conducted in young patients, so the evaluated premolars had larger pulp chamber volumes and higher pulp blood flow than do teeth of older patients.[34-36] Thus, the outcomes may vary according to the patient age. In addition, the current study was only performed on premolars, so differences in PT range may be expected in teeth with smaller or larger crown sizes. Therefore, caution should be taken when extrapolating these outcomes to clinical scenarios different from the one evaluated using this methodology.

CONCLUSION

Based on the limitations imposed by this *in vivo* study, it is possible to conclude that: 1) In the most clinically relevant scenarios, pulp chamber temperature decrease after cavity preparation prevented PT rise above the pre-preparation, physiologic baseline exposure conditions; 2) Longer exposure of a layer of resin composite to a high radiant emittance light curing unit caused higher peak PT and ΔT than did use of shorter exposure periods; and 3) None of the evaluated procedures resulted in a PT rise near to values that could offer any risk of thermal damage to the pulp.

Acknowledgements

The authors are indebted to the State University of Ponta Grossa for the support required to perform this study. The authors would also like to thank Ivoclar Vivadent Inc for the donation of the light-curing unit. This study was financially supported by grants #232/2014 and #488/2014 from Araucaria Foundation, Parana, Brazil.

Regulatory Statement

This study was approved by the Ethics Committee of the State University of Ponta Grossa (protocol #1.954.754).

Conflict of Interest

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

(Accepted 13 September 2020)

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Evaluation of At-home Bleaching Times on Effectiveness and Sensitivity with 10% Hydrogen Peroxide: A Randomized Controlled Double-blind Clinical Trial

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Clinical Relevance

At-home dental bleaching with 10% hydrogen peroxide used for 15 minutes can be considered an alternative to the 30-minute protocol, as it achieved effective tooth whitening and caused similar tooth sensitivity.

SUMMARY

Objectives: The aim of this randomized double-blind controlled clinical trial was to evaluate different protocols for at-home use of 10% hydrogen peroxide in whitening effectiveness and tooth sensitivity.

Methods: Seventy-two patients were selected according to the inclusion and exclusion criteria, with the upper central incisors having color A2 or darker according to the Vita Classical scale (VITA Zahnfabrik, Bad Säckingen, Germany) and randomized into two groups: 10% hydrogen peroxide applied once daily for 15 minutes (HP

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http://doi.org/10.2341/20-104-C

15) or applied once daily for 30 minutes (HP 30). Bleaching was performed for 14 days in both groups. The color was evaluated before bleaching, during bleaching (1st and 2nd weeks), and 1 month after the bleaching treatment using the Vita Classical, Vita Bleachedguide 3D-MASTER, and Vita Easyshade spectrophotometer (VITA Zahnfabrik). Dental sensitivity was recorded by the patients using the numerical rating scale (0-4) and visual analogue scale (0-10 cm). Color data were evaluated by two-way analysis of variance (ANOVA) of repeated measures (group vs. treatment time). The Mann-Whitney test was performed to contrast the means (α=0.05). Tooth sensitivity was assessed by Fisher's exact test (p=1.00) and intensity of tooth sensitivity was evaluated by the Mann-Whitney test (α=0.05) for both scales.

Results: A significant whitening effect was observed after 2 weeks of bleaching for all color measurements (p=0.01), with no difference between HP 15 and HP 30 (p>0.05). Also, the absolute risk and intensity of tooth sensitivity were similar (47%; p>0.05).

Conclusions: The effectiveness and tooth sensitivity of at-home bleaching carried out with 10% hydrogen peroxide applied for 15 minutes or 30 minutes are similar.

INTRODUCTION

Oral aesthetics have become increasingly important for many patients in recent years. The growing interest among the populace for whitening teeth and having a harmonious smile has led to higher demand for aesthetic procedures, including tooth whitening.[1-4] There are several bleaching methods, such as whitening strips, in-office, and at-home bleaching, and the combination of these two techniques.[5]

Among these modalities, the use of 10% carbamide peroxide (CP) for at-home bleaching has become popular, and several studies have shown quite satisfactory results.[5,6] Due to the low concentrations of CP applied at home when compared to in-office bleaching, the at-home technique has several advantages, including less irritation to tissues, low cost, and shorter office time.[7] However, some authors have shown concerns regarding the extended length of time required for using at-home bleaching trays (6-8 hours), mainly because this could increase discomfort to patients[8] during sleep[9] and increase the possibility of ingesting bleaching agents.[10,11]

Due to these concerns, the original technique has undergone modifications. Changes in tray material, tray design, active ingredient concentrations, time of custom-tray use, and type of active ingredients are examples of such modifications.[9,12] One modification was the introduction of hydrogen peroxide (HP) gels for at-home bleaching. As opposed to CP gels, HP bleaching gels undergo a faster degradation rate[13-15] and therefore should be used for shorter periods of time. As pointed out by Haywood (2003), the potential additional benefit to the "simplified regimen" is that it was perceived to be the most convenient and comfortable.[16]

However, in the literature consulted, clinicians and researchers have followed different application regimens with variations mainly in the time that the gel remains in contact with the teeth. For instance, 7.5%-10% HP was recommended for 1 hour/daily or two 30-minute treatments per day,[7,17,18] as well as shorter daily regimens such as 30 minutes.[19-22] Although those studies showed the same whitening effectiveness, since a minimal 14 days of daily use was applied, a high degree of tooth sensitivity has been reported, ranging from 58% to 80%.[7,17,18,20,21]

A potential way to mitigate tooth sensitivity is to apply HP-based bleaching gel for a shorter time, mainly because HP-based gels have fast degradation kinetics. It was shown that the amount of released HP was higher in the first 10 minutes, and up to 50% of the active ingredient was released within 15 to 20 minutes.[14,15]

Therefore, the objective of this randomized, parallel, double-blind clinical trial was to evaluate if there is equivalence among the bleaching effectiveness (primary outcome) of at-home bleaching performed with 10% HP applied for 15 min (HP 15) and 30 min (HP 30). Furthermore, the absolute risk and intensity of tooth sensitivity were evaluated as secondary outcomes. The null hypothesis was that at-home bleaching using 10% HP used daily for 15 or 30 minute intervals would have significant differences regarding: 1) tooth whitening, or 2) the absolute risk, or 3) intensity of tooth sensitivity.

METHODS AND MATERIALS

The present study was prepared using the protocol established by the Consolidated Standards of Reporting Trials statement.[23]

Trial Design, Settings, and Locations of Data Collection

This was a double-blind, controlled, parallel, randomized clinical trial, in which the evaluator and statistician were blinded to the group assignment. This study was

performed from November 2016 to June 2017 at the Clinics of the School of Dentistry of the State University of Ponta Grossa, PR, Brazil.

Recruitment

Two weeks prior to the bleaching procedures, all of the volunteers—who sought treatment at the clinic of the dental school—received a dental prophylaxis with pumice and water in a rubber cup and signed an informed consent form. Recruitment was carried out by placing printed ads throughout the university.

Eligibility Criteria

Patients included in this clinical trial were at least 18 years old and had good oral and overall health. All patients underwent an exam of teeth and soft tissues, and there could be no changes such as cavities, dental wear, friction, visible cracks, gingivitis, periodontitis, or injuries. Participants were required to have six caries-free maxillary anterior teeth, with absence of any restorations or periodontal disease. The maxillary central incisors had to be shade A2 or darker, as judged by comparison with a value-oriented shade guide (Vita Classical, VITA Zahnfabrik, Bad Säckingen, Germany). Two calibrated investigators performed this evaluation, and they were required to have at least 85% agreement (Kappa statistic). The calibration process was performed before beginning the study evaluation.

Participants with prostheses, anterior restorations or dental braces, or severe internal tooth discoloration (tetracycline stains, fluorosis, and pulpless teeth) were not included in the study. Additionally, participants with any other pathology that could cause sensitivity (such as dentin exposure, recession, or the presence of visible cracks in the teeth); pregnant or breastfeeding women; smokers; people with bruxism; or participants who had previously undergone tooth-whitening procedures were also excluded.

Sample Size Calculation

The calculation of the sample size was based on the color variation (ΔE). A minimum of 58 participants was required to exclude a mean difference of 2.66 in ΔE (50%:50% acceptability threshold with a power of 90% and an alpha of 5%, considering that the standard deviation of ΔE is approximately 3.[24] To account for possible dropouts by patients, an additional 25% of the sample was added; therefore, the final calculation was 72 participants. This limit of equivalence (difference of means) was based on the fact that only an ΔE greater than 2.66 is considered clinically perceptible.

Randomization, Allocation, Concealment, and Blinding

Blocked randomization was used (block sizes of 2 and 4), and an equal allocation ratio was used to form the allocation list for the two comparison groups. The randomization list was prepared using software freely available online (www.sealedenvelope.com). Opaque, sealed, and consecutively numbered envelopes containing the identification of the groups were prepared by a third person, who was not involved in the research protocol. The envelopes were only opened immediately before the beginning of the bleaching procedure.

Participants and operators could not be blinded to the study groups, as they could easily identify the two protocols used. However, the evaluator who performed the color assessments and the statistician were blinded to the treatments. To maintain evaluator blinding, the two protocols—15 minutes (HP 15) and 30 minutes (HP 30) per day with 10% hydrogen peroxide (White Class, FGM, Joinville, SC, Brazil)—were explained to the patients by a researcher who was not an evaluator; the protocols were coded as either A or B. Only the research coordinator knew the coding system.

Study Intervention

Alginate impressions (Avagel, Dentsply, Petrópolis, Rio de Janeiro, Brazil) of each subject's maxillary arch were made, and after disinfection with 2% glutaraldehyde for 10 minutes, these impressions were filled with dental stone (Asfer, Asfer Indústria Química Ltda., São Caetano do Sul, SP, Brazil). A 0.9-mm soft vinyl material (FGM, Joinville, SC, Brazil) was used to fabricate the custom-fitted tray that would hold the whitening gel in Plastivac P7 (BioArt, São Carlos, SP, Brazil). The excess material from the labial and lingual surfaces was trimmed to 1 mm from the gingival junction.

At this time, group assignments were revealed and each patient received the bleaching tray and their respective time-of-use protocol. All participants were instructed to wear the tray with the bleaching agent for 15 or 30 minutes (according to group allocation) once a day for 14 days. The participants were also instructed to remove the tray after each bleaching period and wash it with water.

As a measure of adherence to the experimental protocol, participants were given a diary in which they were asked to take note of the number of times they used the tray during the study. If they wore the bleaching tray 14 times, this would result in a 100% adherence to the protocol. Verbal instructions about oral hygiene were also provided, encouraging participants to brush their teeth regularly with fluoridated toothpaste containing

no whitening components. There was no restriction in the diet of the volunteers.[25]

Color Evaluation

Two calibrated evaluators recorded the shade of each subject's teeth at baseline, during treatment (after the first and second week of bleaching treatment), and at one-month post-bleaching. In the event of disagreements between the examiners during shade evaluation, a consensus was reached through discussion. The color evaluation was performed using two value-oriented shade guide units: Vita Classical (VITA Zahnfabrik, Bad Säckingen, Germany)[26] and Vita Bleachedguide 3D-MASTER (VITA Zahnfabrik)[27-29] and with the aid of a spectophotometer (Easyshade, VITA Zahnfabrik).[18,26]

For color evaluation with the Vita Classical scale, the 16 tabs of the shade guide were arranged from highest (B1) to the lowest (C4) value. Although this scale is not linear in the truest sense, for the purpose of analysis, the changes were treated as though they represented a continuous and approximately linear ranking.[30] The Vita Bleachedguide 3D-MASTER contains lighter shade tabs and is already organized from highest (0M1) to lowest (5M3) value.[31,32] The measurement area of interest for shade matching was the middle third of the facial surface of the anterior central incisor.[33] Color changes were calculated from the beginning of the active phase through to the individual recall times by calculating the change in the number of shade guide units (ΔSGU), which occurred toward the lighter end of the value-oriented list of shade tabs.

For color evaluation with the Vita Easyshade (VITA Zahnfabrik) spectrophotometer, an impression of the maxillary arch was taken with dense silicone paste (Speedex Putty, Coltene, Rio de Janeiro, Brazil). The impression was extended to the maxillary canine and served as a standard color measurement guide for the spectrophotometer. For each dental component evaluated, a window was created on the labial surface of the molded silicone guide using a metal device with a radius of 6 mm and well-formed borders.[7,29,31] The shade was determined using the parameters of the Easyshade device whereby the following values were indicated: L*, (a*), and (b*), where L* represented the value from 0 (black) to 100 (white) and a* and b* represented the shade, where a* was the measurement along the red-green axis and b* was the measurement along the yellow-blue axis. The color comparison before and after treatment was given by differences between the two colors (ΔE), which was calculated using the formula: $\Delta E=[(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$.[34] Despite the tooth color, measurements were performed for all maxillary anterior teeth (central and lateral incisors and canines); for statistical purposes, only the maxillary right central incisor was used. The measurement area of interest for shade matching was the middle third of the facial surface of the maxillary right central incisor.

Tooth Sensitivity Evaluation

All subjects were asked to keep a daily record of whether they experienced sensitivity. Each patient was asked to indicate the numerical value of the degree of sensitivity using a five-point Numeric Rating Scale (NRS) where 0 = none, 1 = mild, 2 = moderate, 3 = considerable, and 4 = severe.[29,35,36] The subjects were also asked to express their pain intensity using the Visual Analogue Scale 0-10 (VAS).[29,37,38] This scale is a 10-cm horizontal line with scores of 0 and 10 at each end, where 0 = no sensitivity and 10 = severe sensitivity. Patients marked a vertical line across the horizontal line of the scale that corresponded to the intensity of sensitivity. The distance in mm from the zero end was then measured with the aid of a millimeter ruler.

The worst score from the NRS scale and the highest numerical value obtained in the VAS scale during the entire bleaching treatment was considered for statistical purposes, in such a way that only a single value was taken from the two-week treatment period. The values were arranged into two categories: absolute risk of tooth sensitivity (TS), which represented the percentage of patients that reported TS at least once during treatment, and overall TS intensity.

Statistical Analysis

The analysis followed the intention-to-treat protocol and involved all participants who were randomly assigned (Figure 1).[23] In cases of missing data, the last observation was carried forward. The statistician was blinded to the study groups. The data were first analyzed using the Kolmogorov-Smirnov test to assess whether the data followed a normal distribution, as well as the Bartlett test for equality of variances to determine whether the assumption of equal variances was valid. After that, the absolute risks of bleaching-induced TS were determined using the Fisher exact test. The relative risk was calculated, as well as the confidence interval for the effect size. For comparison of TS intensity (NRS and VAS data) of the two groups, the worst score of the each group was calculated. After that, the Mann-Whitney test was applied. The color changes between groups (ΔSGU on both scales and ΔE) at each time point were compared using a Mann-Whitney U test. For the comparisons between times within each group, the Friedman test was applied. In all statistical tests, the significance level was 0.05. We performed all analyses

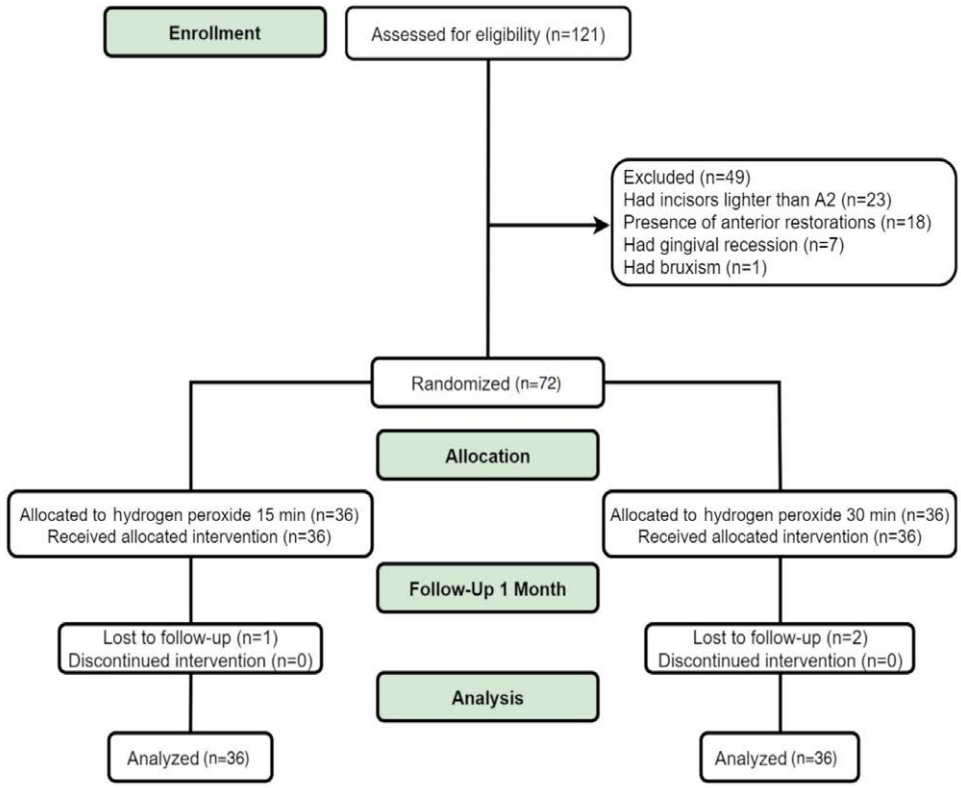

Figure 1. Flow diagram of study design phases including enrollment and allocation criteria.

by using SigmaPlot version 11.0 software (SPSS Inc, IBM, Armonk, NY, USA).

RESULTS

Characteristics of Participants

In all, 121 volunteers were evaluated for selection of the study sample, and 72 participants met the study inclusion criteria (Figure 1). The patients were randomly divided into HP 15 (n=36) and HP 30 (n=36). The mean age of all participants was similar, and the majority of participants included in the sample were female (Table 1).

Adherence to the Protocol

Adherence to the protocol was 86% for HP 15 and 78% for HP 30. Three participants did not attend the one-month recall visit—one in HP 15 and two in HP 30. For these participants, the last observation was carried forward for statistical purposes in order to maintain the intention-to-treat analysis.

Color Evaluation

Color change effectiveness of dental bleaching was verified through the mean and standard deviation of the ΔSGU using the subjective (Vita Classical and Vita Bleachedguide 3D-MASTER, VITA Zahnfabrik)

Table 1: *Demographic Characteristics of the Participants*

	HP 15 (n=36)	**HP 30 (n=36)**
Baseline color (SGU; mean ± SD)	6.1 ± 1.6	5.4 ± 1.0
Age (years; mean ± SD)	20.9 ± 3.0	22.8 ± 6.4
Gender (female; %)	78	58

Abbreviations: HP 15, hydrogen peroxide 15-minute protocol; HP 30, hydrogen peroxide 30-minute protocol; SD, standard deviation; SGU, shade guide unit measured by Vita Classical.

and objective (spectrophotometer) scales, and through the ΔE values (Table 2) in the different evaluation periods (p<0.05). Whitening of approximately 3.3 to 4.8 ΔSGU (Vita Classical) and 3.9 to 6.9 ΔSGU (Vita Bleachedguide), and ΔE of approximately 6.6 to 9.0 were detected for both groups at one month after bleaching (Table 2). Although a significant whitening effect was observed after only two weeks of bleaching (p=0.01), no statistically significant difference was observed between the study groups at the different periods (p>0.06). For all color measurements, the mean difference ranged from -0.6 to 1.0 and was not clinically important (Table 2).

Tooth Sensitivity

There was no significant difference in tooth sensitivity between the groups evaluated (p=1.00) (Table 3). Hydrogen peroxide 10% applied for either 15 or 30 minutes per day produced a 47% risk of tooth sensitivity. Regarding the intensity of dental sensitivity (Table 3), there were no significant differences between the two groups analyzed with the two pain scales used for this study (Table 4; p>0.36).

DISCUSSION

According to the results of the present study, the 10% HP-based bleaching gel applied for 15 or 30 minutes per day showed a significant whitening effect after two weeks. This is a noteworthy result, mainly because an application time as short as 15 minutes was enough to produce highly effective whitening.

Several manufacturers have launched HP-based gels on the market, due to the lengthy tray-wearing time of CP-based gel during at-home bleaching. The idea is that HP should bring about faster results than CP. This could occur during short active bleaching sessions, because unlike an HP-based gel, a CP-based gel first needs to break down into hydrogen peroxide and urea upon exposure to conditions of moisture before achieving the whitening effect.[17,19,39] The fast kinetics of degradation of HP when compared to CP seems to confirm this statement.[14,40]

However, as oxidization of an organic substance involves a series of consecutive steps and takes time to occur, there may be a limit as to how rapidly a chemical substance is released in HP-based gels. Therefore, part of the substance released from the

Table 2: *Color Change in Shade Guide Units (ΔSGU) and ΔE at Different Bleaching Periods for the 2 Bleaching Protocols Along with the Effect Size (95% Confidence Interval)*

	Periods	HP 15[a]	HP 30[a]	Mean Difference (95% CI)	p-value[b]
ΔSGU (Vita Classical)	After 1 week	3.3 ± 1.0 A	3.6 ± 1.0 A	-0.03 (-0.77 to 0.17)	0.09
	After 2 weeks	4.2 ± 1.3 B	4.8 ± 1.3 B	-0.06 (-1.21 to 0.01)	0.06
	After 1 month post bleaching	4.2 ± 0.8 B	4.7 ± 1.4 B	-0.05 (-1.04 to 0.04)	0.06
ΔSGU (Vita Bleachedguide)	After 1 week	3.9 ± 1.5 A	4.1 ± 1.8 A	-0.02 (-0.98 to 0.58)	0.72
	After 2 weeks	6.8 ± 2.5 B	6.9 ± 1.6 B	-0.01 (-1.09 to 0.89)	0.87
	After 1 month post bleaching	6.8 ± 2.5 B	6.8 ± 1.6 B	0.0 (-0.99 to 0.99)	0.97
ΔE (Vita Easyshade)	After 1 week	6.6 ± 3.8 A	7.2 ± 3.7 A	-0.6 (-2.36 to 1.16)	0.40
	After 2 weeks	7.9 ± 3.8 AB	8.4 ± 3.7 B	-0.5 (-2.26 to 1.26)	0.53
	After 1 month post bleaching	9.0 ± 3.8 B	8.0 ± 3.2 B	1 (-0.65 to 2.65)	0.28

Abbreviations: CI, confidence interval; HP 15, hydrogen peroxide 15-minute protocol; HP 30, hydrogen peroxide 30-minute protocol; SGU, shade guide unit.

[a] *Color change for different periods and evaluation methods. Means identified with the same capital letter are statistically similar values (Friedman test;* p>*0.05).*

[b] *For color change evaluation of different groups in each period (Mann-Whitney test;* p>*0.05).*

Table 3: *Comparison of the Number of Patients Who Experienced Tooth Sensitivity During the Bleaching Regimen in Both Groups Along with Absolute and Risk Ratio*

Treatment	Tooth Sensitivity (Number of Participants)		Absolute Risk (95% CI)	Risk Ratio (95% CI)
	Yes	No		
HP 15	17	19	47.2 (31.9-62.9)	1.0 (0.61-1.62)
HP 30	17	19	47.2 (31.9-62.9)	

Abbreviations: CI, confidence interval; HP 15, hydrogen peroxide 15-minute protocol; HP 30, hydrogen peroxide 30-minute protocol.
**Fisher exact test (p=1.00).*

HP-based gels may not even have time to contact the organic substance of the teeth, thereby decreasing the whitening effect.[41]

A recent systematic review of clinical studies showed that, even when HP-based products were used for a shorter period of time compared to CP-based gels, both at similar concentrations, there were no significant color changes in the shade guide units, with an equivalent level of gingival irritation and tooth sensitivity when comparing CP-based to HP-based gels for at-home bleaching.[41,42]

In this study, the HP 15 and HP 30 groups showed a significant whitening effect of approximately 4.2 to 6.8 shade guide units for different scales, and the ΔE varied by approximately 8 to 9 units, which corroborates with the findings of previous studies that applied more highly concentrated HP in at-home bleaching.[7,20,21,43] For example, in the study by Cordeiro and others[21] who evaluated the 10% HP wearing 30 minutes a day, a significant whitening effect of approximately 3 to 7 shade guide units for different scales was observed, as well as a whitening effect of 7 to 10 units of ΔE. Also, Chemin and others[7] evaluated a 10% HP applied twice a day for 30 minutes, and approximately 4 units on the Vita Classical scale and 6 units of Vita BleachedGuide were observed. When a color was measured with a spectrophotometer, an approximate ΔE 8 was observed, a significant whitening effect.

These results lead us to accept the first null hypothesis. It is worth noting that, even when applied for 15 minutes, 10% HP showed clinically important whitening when evaluated with the three instruments used for color evaluation. It is noteworthy that more than one color evaluation was performed, in order to provide data that can be compared with different clinical studies.

Regarding tooth sensitivity, HP has a low molecular mass, which is associated with its fast kinetics of degradation,[14,40] and helps it to diffuse rapidly into tooth substrates and reach the pulp chamber; it has already been observed that HP was found inside the pulp chamber after 5 minutes.[44,45] These factors may be responsible for the tooth sensitivity reported by most patients who have undergone bleaching procedures.[12] Considering that irritation to the pulp cells is proportional to the HP concentration used,[44,46] one would expect a lower tooth sensitivity in the HP 15 group compared to the HP 30 group. However, no significant differences were observed for either group, leading us to accept the second and third null hypotheses.

Actually, the risk of tooth sensitivity in this study was around 47% in both groups, a lower percentage

Table 4: *Means (Standard Deviations) and Medians (Interquartile Range) of Worst Score of the Tooth Sensitivy Intensity Using the NRS and VAS Scales*

Pain Scales	Means and Standard Deviations		*p*-value[a]	Medians and Interquartile Range		*p*-value[a]
	HP 15	HP 30		HP 15	HP 30	
NRS	0.7 ± 2.8	0.6 ± 0.7	0.55	0 (0–1)	0 (0–1)	0.78
VAS	0.9 ± 4.1	0.6 ± 0.3	0.36	0.1 (0–1.5)	0 (0–1.1)	0.54

Abbreviations: HP 15, hydrogen peroxide 15-minute protocol; HP 30, hydrogen peroxide 30-minute protocol; NRS, numerical rating scale; VAS, visual analogue scale.
[a] For each pain scale, the comparison was performed to different groups (Mann-Whitney test).

than that observed in clinical studies in which 10% HP was evaluated.[7,17,18] For instance, de La Pena[18] showed 58.3% tooth sensitivity and Chemin[7] indicates that the use of 10% HP causes around 80% tooth sensitivity. Nevertheless, a closer view regarding these studies showed that both of them applied HP once daily for one hour or twice daily for 30 minutes,[7,17,18] in comparison to the 15- or 30-minute applications in the present study. Therefore, it was assumed that the lower percentage of patients with tooth sensitivity, and the lower intensity thereof, in the present study is due to the shorter application time evaluated.

Also, when 10% HP-based gels are applied, gingival irritation is a very common finding.[21,22,39] This may occur because the 10% HP could react with soft tissue and cause injuries such gingival irritation and burning.[47] In contrast, none of the patients reported any problems related to gingival irritation in the present study (no data shown). Once again, the short application time of HP could justify the results herein.

The results of the present study show that application times as short as 15 minutes a day were enough to produce effective whitening, with a low number of adverse effects reported. This has an important clinical implication, as it reduces the total wearing time, favoring those patients who do not want to wear the bleaching tray for prolonged periods of time. However, only a one-month trial was performed. As color stability is considered a problem when bleaching gels are applied for short periods of time,[48] long-term follow-up should be performed in order to confirm the results of the present study.

CONCLUSION

The use of 10% hydrogen peroxide for 15 and 30 minutes daily is equally effective after two weeks of at-home tooth whitening, and produces equivalent absolute risk and intensity of tooth sensitivity.

Acknowledgements

This study was performed by Kaprice Chemin as partial fulfillment of her PhD degree at the State University of Ponta Grossa (UEPG), Ponta Grossa, PR, Brazil. This study was partially supported by the National Council for Scientific and Technological Development (CNPq) under grants 303332/2017-4 and 308286/2019-7 and Coordenação de Aperfeiçoameto de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001.

Regulatory Statement

This study was conducted in accordance with all the provisions of the human subjects oversight committee guidelines and policies of the State University of Ponta Grossa. The approval code issues for this study is 1.958.304.

Conflict of Interest

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

(Accepted 13 September 2020)

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Effect of Daily Usage Time of 4% Hydrogen Peroxide on the Efficacy and Bleaching-induced Tooth Sensitivity: A Single-blind Randomized Clinical Trial

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Clinical Relevance

Although color change was slightly lower in a 3-week 30-minutes/day protocol, than in the 120-minute protocol, this could be compensated by an extra week of bleaching. The advantage of the shorter protocol is the reduced daily application, making the procedure more comfortable for the patients.

SUMMARY

Objective: Compare the risk/intensity of tooth sensitivity (TS) and color change of a 30-minute vs. the recommended 120-minute application time of 4% hydrogen peroxide (HP) for at-home bleaching.

Methods: A single-blind, parallel, randomized clinical trial was conducted with 92 adult patients with caries and restoration-free anterior teeth A2 or darker, randomly allocated to two groups. Bleaching trays containing 4% HP were used for three-weeks. A four-week regimen was also

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http://doi.org/10.2341/20-058-C

offered to the patients for the 30-min group after the end of the 3-week protocol. The color change was assessed with the Vita Classical (VITA Zahnfabrik, Bad Säckingen, Germany) and Vita Bleachedguide shade guides (VITA Zahnfabrik) and the Vita Easyshade spectrophotometer (VITA Zahnfabrik) at baseline, weekly, and 30 days after the bleaching. The absolute risk and the intensity of TS were assessed daily using the 0-10 visual analogue scale (VAS) and 5-point Numerical Rating Scale (NRS) scale, and patient satisfaction was recorded with a Likert 0-7 scale. Risk of TS (Fisher's test), intensity of TS in NRS scale (Mann-Whitney test), VAS scale (*t*-test), and a color change (*t*-test) were compared.

Results: The 30-minute group saw color change of around 1 SGU inferior to the 120-minute group in all-time assessments ($p<0.05$). After an extra week of bleaching, mean color change was similar ($p>0.05$). Patient satisfaction was high for both groups ($p>0.05$).

Conclusions: A four-week protocol of at-home dental bleaching with 4% HP for 30 minutes/day whitened teeth similarly to the 120 minutes/day protocol, with low intensity of dental sensitivity and high patient satisfaction.

INTRODUCTION

The constant concern of contemporary society to obtain a bright and harmonious smile increased the popularity of dental bleaching, being nowadays one of the most requested clinical procedures of dentistry.[1] The bleaching process consists of reducing the saturation of the tooth, using bleaching gels based on peroxides, which provide a lighter-looking smile.[2] This treatment can be performed by two supervised techniques: at-home and in office.[3]

Among these techniques, at-home bleaching is the most used,[4] due to easy application, excellent esthetic result, lower cost than in-office bleaching, and full acceptance by patients, with efficacy extensively investigated in the literature.[2,5,6] The most frequently scrutinized at-home bleaching agent in the dental literature is 10% carbamide peroxide;[5,6] however, low concentrate hydrogen peroxide is also available for at-home use. Carbamide peroxide gels are used in concentrations between 10% and 22%,[7,8] both with 0.5% potassium nitrate and 0.11% fluoride ions while hydrogen peroxide is used in concentrations from 4% to 14%.[9–11]

Both dental bleaching agents are effective to whiten teeth,[12] but they vary in their recommended daily usage time. While carbamide peroxide is usually recommended for longer daily periods, hydrogen peroxide-based agents are recommended for shorter daily application times due to their different degradation kinetics.[13-15]

Despite the efficacy of at-home bleaching to whiten teeth, tooth sensitivity (TS) is a common and undesirable side effect. Tooth sensitivity results from the damage that hydrogen peroxide, which passes quickly through enamel and dentin, causes to the dental pulp.[16] Although for at-home bleaching TS is usually mild and transient, there are cases in which severe TS is reported, leading the patient to discontinue treatment.[16,17]

Higher gel concentration and contact time with the dental surface are known to be directly related to the higher prevalence of tooth sensitivity.[7,18-22] Indeed, Chemin & others[10] demonstrated that a 4% hydrogen peroxide bleaching gel had lower risk and intensity of TS than a 10% hydrogen peroxide product, even though approximately 40% of the participants from the 4% hydrogen peroxide group experienced some level of TS.

Control over the application time can also be an alternative to reduce or prevent bleaching-induced TS. In a study conducted with carbamide peroxide for at-home bleaching, the authors showed that reducing the application time from 8 hours to 1 hour daily reduced the risk and intensity of TS by half without jeopardizing color change.[23] Although this effect has been demonstrated for carbamide peroxide gels, randomized clinical trials with variations in the at-home protocol, mainly application times, are still scarce in the literature.

Many manufacturers recommend the application of 4% hydrogen peroxide gel for two hours daily; however, the amount of active agent available to react with tooth structure is known to drop by half in just 20 minutes,[15] which suggests that the use of the tray for prolonged periods may not offer benefits and may potentially increase the risk of adverse effects.

Therefore, the objectives of the present study were to evaluate the impact of reduced application time of a 4% hydrogen peroxide product from 120 minutes/day to 30 minutes/day on the efficacy and tooth sensitivity of at-home bleaching in adults. The null hypotheses tested were that the application of 4% hydrogen peroxide gel for 30 minutes would produce similar: 1) color change and 2) bleaching-induced tooth sensitivity compared to the recommended 2-hour application.

METHODS AND MATERIALS

Ethics Approval and Protocol Registration

The report of this clinical trial followed the Consolidated Standards of Reporting Trials (CONSORT) statements.

This study was registered in the Brazilian clinical trials registry (ReBEC) under the number RBR-96QPWN.

Trial Design, Settings, and Locations of Data Collection

This was a single-blind, parallel, randomized clinical trial, in which the evaluator was masked to the group assignment. The treatments proposed in this study were performed in the School of Dentistry clinic at the state University of Ponta Grossa, PR, Brazil, from November 2018 to July 2019.

Recruitment

The volunteers were recruited through local advertisements. All participants signed an informed consent form before the beginning of the study and received dental prophylaxis to remove extrinsic stains.

Eligibility Criteria

Volunteers were selected for this study according to the pre-established criteria. To be included in the study, they were required to be at least 18 years old, be in good general and oral health, and have the anterior maxillary teeth without restorations or caries lesions. They were required to present canine shade A2 or darker according to a value-oriented Vita Classical shade guide (Vita Zahnfabrik, Bad Säckingen, Germany).

Patients who had undergone previous bleaching procedures, with dental prosthesis or visible cracks of enamel, history of spontaneous tooth sensitivity (TS), severe tooth discoloration coming from dental pathologies or use of medications, were not included in the study. Pregnant or lactating women and patients under orthodontic treatment or taking any anti-inflammatory or analgesic medicines were also excluded.

Sample size

The sample size calculation was based on the primary binary outcome (absolute risk of TS) for a superiority trial. The absolute risk of TS for at-home bleaching using 4% hydrogen peroxide gel (White Class, FGM, Joinville, Brazil) was reported by Chemin & others[10] to be 38%. Thus, a minimum sample size of 44 subjects per group was required to have an 80% chance of detecting, as significant at the two-sided 5% level, a decrease in the primary outcome measure from 38% in the control group to 13% in the experimental group. A 5% increase in the sample size, considering potential loss or refusal, gave a total sample size of 92 subjects.

Randomization, Allocation Concealment, and Binding

The randomization process was performed on a website freely available online (http://www.sealedenvelope.com). Blocked randomization (block sizes of two and four) with an equal allocation ratio was used to form the allocation list for both treatment groups. The sequence was inserted into cards placed sequentially in opaque and sealed envelopes numbered from 1 to 92, prepared by a third person who was not involved in the research protocol. These envelopes were opened only at the moment of the intervention, ensuring the random sequence's concealment.

Participants and operators could not be blinded to the study groups, as they could easily identify the two protocols used. However, the evaluator who performed the color assessments was blinded to the treatments. To maintain evaluator blinding, the two protocols—30 minutes and 120 minutes per day with bleacching gel were explained to the patients by a researcher who was not an evaluator; the protocols were coded as either A or B. Only the research coordinator knew the coding system.

Bleaching Procedure

Alginate impressions (Plastalgin, Septodont Healthcare India Private Ltd., Raigad, Maharashtra, India) were taken of each subject's maxillary and mandibular arch, and after disinfection, stone molds were prepared (Asfer, Asfer Indústria Química Ltda, São Caetano do Sul, Brazil). Custom bleach trays were fabricated by heating 0.9 mm soft vinyl material (FGM Dental Products, Joinville, Brazil) in a special device (Plastivac P7, BioArt, São Carlos, SP, Brazil). The excess material on the buccal and lingual surfaces was trimmed 1 mm above the gingival junction.

The bleaching gel used in this study for both groups was the 4% hydrogen peroxide agent (White Class, FGM). In the experimental group, the subjects used the bleaching tray for 30 minutes daily. In the control group, the tray was used for 120 minutes daily, following the manufacturer's instructions. Participants from both groups performed bleaching for three weeks. A four-week bleaching regimen was offered to the patients in the 30-minute group, after the one-month recall, if they did not reach the same bleaching efficacy of the 120-minute group. After the recommended daily period, the subjects were instructed to remove the trays, rinse with water and brush their teeth with a toothpaste free of bleaching agents or desensitizers. Both arches were bleached in the same way. All subjects received verbal and written instructions about the proper use of the bleaching agent and oral hygiene control.

Outcomes

Tooth Color Shade Evaluation—

The tooth color shade evaluation was recorded at baseline, weekly during the treatment, and one-month postbleaching, by two blinded calibrated evaluators (Kappa statistics measuring at least 85%). The evaluation was performed subjectively using value-oriented shade guide units and objectively using a dental spectrophotometer.[24] All recordings were made in the same room under D65 daylight fluorescent light with a luminescence intensity of between 1200 and 1600 lux.

Two shade guides were used for subjective evaluation: the Vita Classical (Vita Zahnfabrik) and the Vita Bleachedguide 3D-MASTER (Vita Zahnfabrik). The measurement area for shade matching was the middle third of the right upper canine's facial surface. If there were disagreements between the examiners during shade evaluation, a consensus was reached.

The Vita Classical scale was converted into numeric values arranged in 16 tabs from the highest (B1) to the lowest (C4) value. Although this scale is not linear in the most real sense, the scales' changes were treated as representing a continuous and approximately linear ranking, as already performed in prior published studies.[1,25,26] Thus, the lower the numeric value, the whiter the tooth. The Vita Bleachedguide 3D-MASTER contains lighter shade tabs, and it is already organized from the highest (0M1) to the lowest (5M3) value, by scores of 1 to 24. Shade guide units (SGU) were calculated by subtracting the individual recall times from those measured at baseline.

For objective evaluation, the Vita Easyshade spectrophotometer (Vita Zahnfabrik) was used. An impression of the maxillary arch was taken with high-putty silicone paste (Perfil, Coltene Holding, Altstätten, Switzerland), and a window on the labial surface of the silicone guide was created by using a metallic device with well-formed borders and 6-mm radius, which is precisely the diameter of the tip of the spectrophotometer, in the middle third of the right upper canine to standardize the area for color evaluation with the spectrophotometer.

The color parameters L*, a*, and b* were recorded. L* represented the luminosity, with values ranging from 0 [black] to 100 [white]), and a* represented color variations in the red-green axis, while b* represented color variations in the yellow-blue axis. Color change (ΔE) was given by the difference between baseline and each recall period, calculated by using the original CIELab (1976)[27] and also using the CIEDE2000, a formula based on the CIELab, which measures color difference and includes not only lightness, chroma, and hue weighting, but also an interactive term between chroma and hue differences for improving insight for small color differences.[28]

Tooth Sensitivity Evaluation—

The participants recorded TS daily during treatment using a Visual Analogue Scale (VAS) and a five-point Numeric Rating Scale (NRS).[22,25,26] For the VAS, the participants were instructed to place a line perpendicular to a 100 mm horizontal line with zero at one end indicating "no TS" and the other end indicating "severe TS." The distance between the marked line to the zero end was measured with the aid of a millimeter ruler.[1] For the NRS, the participants were instructed to indicate a numerical value (0 = none, 1 = mild, 2 = moderate, 3 = considerable and 4 = severe) that most represented their TS. These forms were returned to the researcher on the next clinical appointment.

The worst score from the NRS and the highest numeric value obtained in the VAS during the bleaching treatment were taken for statistical purposes. If the participant scored 0 (no sensitivity) in all-time assessments, this participant was insensitive to the bleaching protocol. We calculated the absolute risk of TS, representing the percentage of patients who reported TS at least once during treatment and the overall TS intensity for the two pain scales.

Assessment of Patients' Satisfaction—

Using a 7-point Likert Scale,[21] participants were asked to express how much they agreed to the statement that reflected their satisfaction with the bleaching treatment after the end of the treatment. For this purpose, the 7-point Likert scale was anchored by 1 (very dissatisfied) to 7 (very satisfied). Similarly, the participants used the same scale to express how much they would recommend the protocol to family and friends by anchoring 1 (would not recommend) and 7 (definitely recommend).

Statistical Analyses

The statistical analyses followed the intention-to-treat protocol according to CONSORT suggestion[29] and involved all the 92 participants who were randomly assigned. The statistical analyses were performed using the SigmaPlot for Windows version 12.0 (Systat Software) software, and in all statistical tests, the significance level was set at 5%.

The absolute risk of TS of both treatments was compared using Fisher's exact test. The relative risk and the confidence interval for the effect size were calculated. The comparison of the TS intensity (NRS and VAS) at the two different assessment points was performed

by using the Mann-Whitney test and the *t*-test for independent samples, respectively. Color changes (ΔSGU and ΔE) between groups at each assessment period were compared using a *t*-test. Comparisons between assessment periods and within groups were performed with the repeated measures one-way analysis of variance (ANOVA) and Tukey's test for pairwise comparisons. Data from the Likert scale from both groups were compared using a *t*-test for independent samples. In all statistical tests, the level of significance was set at 5%.

RESULTS

Characteristics of the Participants

A total of 146 participants were examined, but only 92 were eligible for the clinical trial (Figure 1). Reasons for exclusion are described in Figure 1. The demographic characteristics and the baseline color of the participants are described in Table 1. The distribution of the genders in both groups, as well as the baseline color parameters, were statistically similar, (p>0.05). The patients' ages ranged from 18 to 42 years, and most participants were White.

Adherence to the Protocol

Two participants from the 120-minute group did not return to the recall visits. We imputed the mean data of the group for the missing cells of these two participants.

One participant from the 30-minute group discontinued intervention because she was hospitalized for a reason not related to the intervention. The other two participants from the 120-minute group claimed lack of time and did not return for some recall visits. For these three participants, the last observation was carried forward for statistical purposes to keep the intention-to-treat analysis.[29]

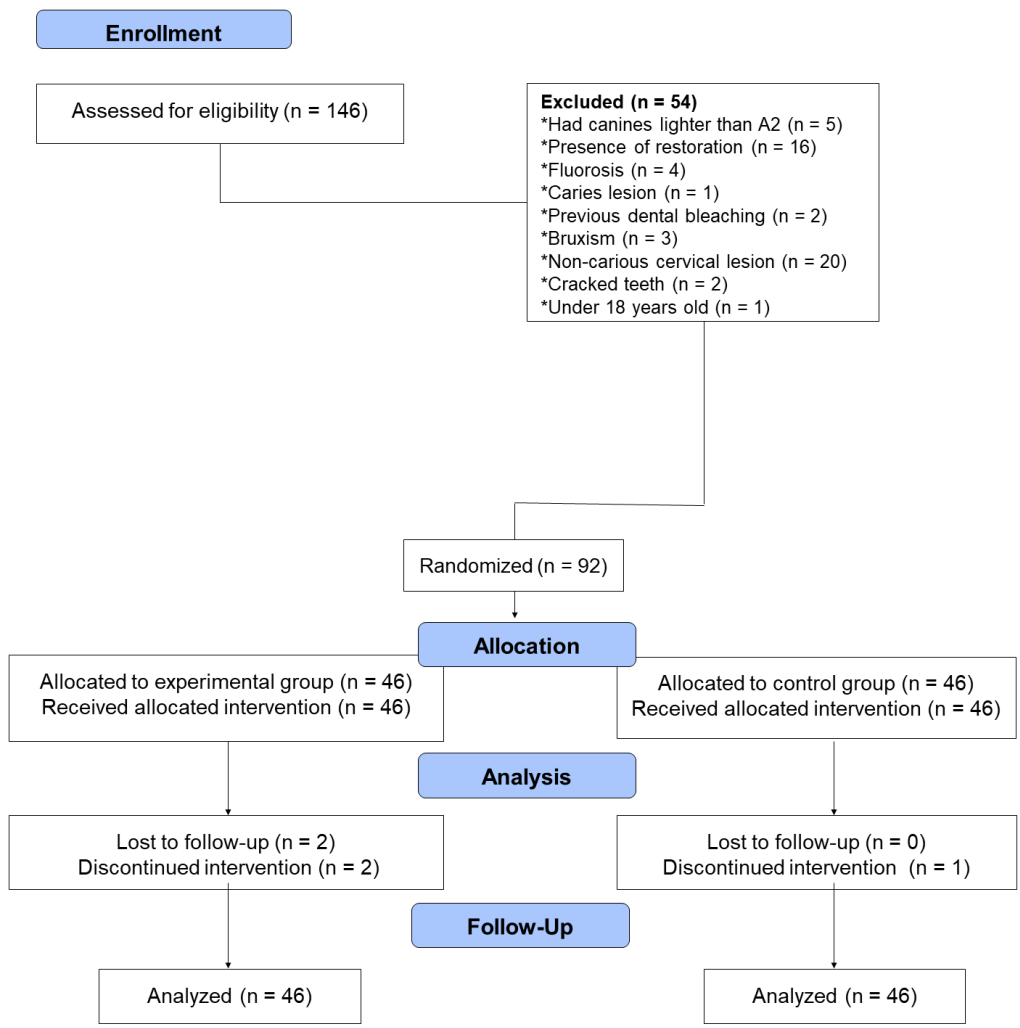

Figure 1. *Flow diagram of study design phases including enrollment and allocation criteria*

Table 1: *Baseline Characteristics of the Participants*

Characteristics		30-min	120-min	*p*-value
Baseline color, (mean ± SD)	SGU Vita Classical	11.0 ± 2.5	11.1 ± 2.4	0.417†
	SGU Vita Bleachedguide	20.3 ± 2.4	20.3 ± 2.8	1.0†
	L*	82.0 ± 3.4	82.7 ± 3.4	0.362†
	a*	-1.3 ± 1.7	-1.4 ± 2.3	0.793†
	b*	24.6 ± 3.9	24.8 ± 4.5	0.886†
Age (years; mean ± SD)		25 ± 6.4	23 ± 5.8	0.133†
Gender (female; %)		76.1	76.1	0.807††
Race	White (%)	73.9	78.2	0.879††
	Black (%)	2.2	2.2	
	Other (%)	23.9	19.6	

Abbreviations: a, color variations in the red-green axis; b*, color variations in the yellow-blue axis; L* luminosity, with values ranging from 0 [black] to 100 [white]); SGU, shade guide unit.*
†t-test for independent samples.
††Chi-square test.

All other participants, except those five, attended all recall visits up to one month after the treatment. Figure 1 depicts the participant flow diagram in the different phases of the study design.

Tooth Sensitivity

Table 2 demonstrates the number of participants who experienced TS during the bleaching treatment in each group. There was no significant difference between the two groups (Fisher test, p=0.34). The relative risk (95% confidence interval) crosses the null value 1, showing that reducing the application time of bleaching gel does not seem to reduce TS.

Table 3 shows data from the TS intensity obtained by NRS and VAS scales. No significant difference in the intensity of TS between groups was observed in any pain scale (p=0.32 and p=0.11 for NRS and VAS, respectively).

Color Evaluation

The descriptive data from bleaching obtained after three weeks of treatment can be seen in Tables 4 and 5. A higher degree of whitening was observed in participants from the 120-min in all-time assessments when the color change was evaluated with shade guide units (p<0.05). Participants from the 30-minute group performed an extra week of bleaching. The four-week 30-minute bleaching regimen achieved the same bleaching efficacy of the three-week 120-minute bleaching regimen (p>0.05).

For DE_{76}, significant differences between groups were observed (p<0.05) in some time assessments, while others were at borderline significance. For DE_{00}, non-significant results were obtained in all-time assessments, being borderline at the 1-month post- bleaching time. Similar results between the two groups were detected when the 30-minute group received an extra week of bleaching (p>0.05).

The mean L* values increased while the mean values of a* and b* decreased in both treatment groups (data not shown). These data suggest that the color change occurred towards an increase in lightness and reduction in the chroma, making teeth less yellow and less saturated.

Table 2: *Comparison of the Number of Patients who Experienced Tooth Sensitivity During the Bleaching Regimen in Both Groups Along with Absolute and the Risk Ratio*

Groups	Tooth Sensitivity (Number of Patients)		Absolute Risk (95% CI)†	Risk Ratio (95% CI)
	Yes	No		
30 min	32	14	70 (55-80)	0.9 (0.7-1.1)
120 min	37	9	80 (67-89)	

Abbreviations: CI, confidence interval.
†Fisher test (p=0.34).

Table 3. *Tooth Sensitivity Intensity Using Both Pain Scales, as well as the* p*-value from the Statistical Analysis*

Pain Scales	Means and Standard Deviations		Mean Difference (95% CI)	Medians and Interquartile Range		*p*-value
	30-min	120-min		30-min	120-min	
NRS (0-4)	0.9 ± 0.9	1.1 ± 0.9	0.2 (-0.2-0.6)	1 (0-1)	1 (0-1)	0.32†
VAS (0-10)	0.9 ± 1.3	1.5 ± 2.1	0.6 (-0.1-1.3)	0.4 (0-1.4)	0.8 (0.1-1.8)	0.11††

Abbreviations: CI, confidence interval; NRS. numeric rating scale; VAS; visual analogue scale.
†Mann-Whitney test
††t-test

Patient Satisfaction

Data from the Likert scale can be seen in Table 6. Patients in different bleaching groups gave similar scores (p=0.063) when evaluating their satisfaction after the end of the bleaching treatment (three-week regimen). They were quite satisfied with the overall bleaching treatment. The high and similar ratings between groups indicate that participants from both groups would undoubtedly recommend the same bleaching treatment to their family or friends (p=0.129).

DISCUSSION

A significant color change occurred after the end of the three-week protocol for both groups. However, this study showed that the recommended application time of 120-minutes produced a slightly higher whitening degree on tooth shade subjective evaluation (around one shade guide unit in all-time assessments) than that achieved with a daily 30-minute application when a 4% HP gel was employed. This is evidence that the color change occurring during bleaching is time-dependent,

Table 4: *Means and Standard Deviations of ΔSGU Obtained with the Vita Classical and Vita Bleachedguide 3D-MASTER at Different Periods*[a]

ΔSGU (Vita Classical)				
Periods	**30-min**	**120-min**	**Mean Difference (95% CI)**	***p*-value†**
One week	2.3 ± 2.0 A	3.1 ± 1.7 A	0.8 (0.03 to 1.57)	0.045
Two weeks	4.3 ± 2.2 B	5.9 ± 2.4 B	1.6 (0.65 to 2.55)	< 0.001
Three weeks	5.6 ± 2.4 C	6.8 ± 2.4 C	1.2 (0.21 to 2.19)	0.013
1 month post bleaching	5.3 ± 2.4 C	6.7 ± 2.5 C	1.4 (0.38 to 2.42)	0.006
3-week (120-min) vs. 4-week (30-min)	6.1 ± 2.6	6.8 ± 2.4	0.7 (-0.34 to 1.74)	0.157
ΔSGU (Vita Bleachedguide 3D-MASTER)				
Periods	**30-min**	**120-min**	**Mean Difference (95% CI)**	**p-value†**
One week	2.7 ± 1.9 A	3.7 ± 1.7 A	1 (0.25 to 1.75)	0.015
Two weeks	4.7 ± 2.1 B	5.8 ± 2.1 B	1.1 (0.23 to 1.97)	0.013
Three weeks	6.0 ± 2.3 C	7.1 ± 2.6 C	1.1 (0.08 to 2.12)	0.028
1 month post bleaching	5.6 ± 2.1 C	6.9 ± 2.8 C	1.3 (0.27 to 2.33)	0.013
3-week (120-min) vs. 4-week (30-min)	7.0 ± 2.5	7.1 ± 2.6	0.1 (-0.96 to 1.16)	0.870

Abbreviations: ΔSGU, change in shade guide units; CI, confidence interval.
[a]Comparisons within time assessments are valid only within each column. Different letters mean statistically different averages (repeated measures one-way ANOVA, p*<0.05).*
†t-test for independent samples.

Table 5: *Means and Standard Deviations of ΔE Obtained by Spectrophotometer (Vita Easyshade) at Different Periods*[a]

ΔE 1976				
Periods	**30-min**	**120-min**	**Mean Difference (95% CI)**	***p*-value**[†]
One week	5.8 ± 2.5 A	6.9 ± 3.1 A	1.1 (0.03 to 2.17)	0.061
Two weeks	7.6 ± 2.8 B	9.0 ± 3.5 B	1.4 (0.09 to 2.71)	0.039
Three weeks	8.4 ± 3.1BC	9.9 ± 4.2 C	1.5 (-0.03 to 3.03)	0.061
1 month post bleaching	8.9 ± 3.2 C	10.7 ± 4.2 D	1.8 (0.25 to 3.35)	0.030
3-week (120-min) vs. 4-week (30- min)	9.7 ± 3.4	9.9 ± 4.2	0.2 (-1.38 to 1.78)	0.816
ΔE 2000				
Periods	**30-min**	**120-min**	**Mean Difference (95% CI)**	**p-value**[†]
One week	3.8 ± 1.7 A	4.4 ± 2.0 A	0.6 (-0.17 to 1.37)	0.118
Two weeks	5.0 ± 1.9 B	5.7 ± 2.2 B	0.7 (-0.15 to 1.55)	0.077
Three weeks	5.5 ± 2.0 BC	6.2 ± 2.6 C	0.7 (-0.26 to 1.66)	0.134
1 month post bleaching	5.8 ± 2.1 C	6.8 ± 2.6 D	1 (0.02 to 1.98)	0.053
3-week (120-min) vs. 4-week (30- min)	6.2 ± 2.2	6.2 ± 2.6	0 (-1 to 1)	0.979

Abbreviations: ΔSGU, change in shade guide units; ANOVA, analysis of variance; CI, confidence interval.

[a]*Comparisons within time assessments are valid only within each column. Different letters mean statistically different averages (repeated measures one-way ANOVA,* p< *0.05).*

[†]t-*test for independent samples.*

since the 30-minute time is enough to bleach teeth but in a slightly whitening degree (approximately 1.2 units of ΔSGU and 1.5 units of ΔE 1976). Thus, the first null hypothesis was rejected.

Such results are consistent with previous studies demonstrating that the color change achieved during bleaching is directly related to the amount of time that the agent is in contact with the dental structure.[23,30,31]

However, the significant difference in color change in the 30-minute group was compensated by longer treatment time. Although not specified in the research protocol, we performed an extra week of bleaching in the participants from the 30-minute group (four-week protocol), and we observed that the color change reached the same level of that achieved in the three-week 120-minute protocol.

Regarding TS, literature findings led us to hypothesize that a shorter application time of a low concentrate product (4% HP) could reduce TS. A previous in vitro study has already demonstrated that diffusion of HP, cell viability, cell morphology alterations, oxidative stress, and cell membrane damage occurs in a concentration-time dependent fashion.[32] In agreement with those findings, clinical studies have also recommended reduced application times to minimize bleaching-induced TS.[20,23,33,34]

Contrary to our expectation, the present study did not show any difference in the risk and intensity of

Table 6: *Patient Satisfaction and Patient's Recommendation Using a 7-point Likert Scale*[a]

7-point Likert Scale	**Means and Standard Deviations**		**Medians and Interquartile Range**		***p*-value**[†]
	30-min	**120-min**	**30-min**	**120-min**	
Patient satisfaction	6.0 ± 0.9	6.4 ± 0.9	6 (5-7)	7 (6-7)	0.063
Patient recommendation	6.4 ± 0.9	6.7 ± 0.7	7 (6-7)	7 (7-7)	0.129

[a]*Maximum of 7 points.*

[†]Mann-Whitney statistical analysis.

TS between the two application times of 4% HP. Approximately 70% to 80% of the participants from both groups experienced pain at least once during bleaching, and the intensity of TS was very low in both cases. Thus, the second null hypothesis was not rejected.

The differences between the present and earlier clinical studies are ascribed to their use of high concentrate products and more extended application periods than those used in the present investigation. For instance, Kose & others employed a 35% HP in-office product[20] showing lower TS when the product was applied for 30 minutes rather than the recommended 45-minute protocol. Cardoso & others[23] employed a low concentrate 10% carbamide peroxide; however, they compared a short application time of one-hour versus an overnight use (at least 7-hour difference between the groups) showing a lower risk of TS for the shorter application period.

Unlike earlier studies, we used a very low concentration of HP and a slight difference in daily time regimen, which explains the similar TS risk and intensity of both time regimens in the present study. The extra amount of radical oxygen species (ROS) that may reach the pulp in the 120-minute regimen may not surpass the pulp defense system, contrary to what occurs when high concentrate products are applied for longer times.[32]

However, we could observe that even in the shorter application time of 30 minutes, the 4% HP product was capable of triggering TS. Due to its relatively low molecular mass (34 g/mol), HP crosses enamel and dentin very fast, and it takes approximately 15 minutes for the hydrogen peroxide to reach the pulp chamber.[31]

Clinical trials of bleaching protocols are essential as they allow the identification of optimum concentration and application times to keep HP penetration in the pulp chamber minimal without compromising bleaching efficacy. In this way, other time regimens than the one recommended by manufacturers should be investigated to find the shortest application time that yields similar color change.

We used shade guide units and a spectrophotometer for color change evaluation. The spectrophotometer is an objective method capable of quantifying differences in color and its dimensions.[24] The color dimensions can be quantified using either the CIELab 1976 formula (ΔE_{ab}) or the CIEDE2000 formula (ΔE_{00}). While shade guide units and the CIELab 1976 formula reached the same conclusions in most of the time assessments, the CIEDE2000 was less sensitive in detecting the slight changes between the groups under investigation. Kury & others[35] also found differences between the methods of an objective assessment; in their study, ΔE_{00} was also unable to detect a significant difference between groups.

The fact that some color change instruments detected significant differences while other instruments did not, cannot be seen as a limitation of the present study. These findings do reflect that the differences observed between the daily regimens were subtle and not substantial. Had they been expressive, all methods of a color change would yield statistically significant results. Similar results between the study groups were also observed with another recently developed index used to compare the bleaching efficacy of treatments, called whitening index (WI_D).[36] Its calculation is straightforward, and it is based on the L*, a*, and b* parameters from baseline and after bleaching. After an extra week of bleaching for the participants from the 30-minute group (4-week), these values were statistically similar to the three-week 120-min protocol.

Perceptibility (PT) and acceptability (AT) thresholds are very relevant in clinical dentistry.[37] Paravina & others[38] found that the PT and AT values were 1.2 and 2.7, respectively, for the CIELab system and 0.8 and 1.8, for the CIEDE2000 system. These values can be used as a reference for the evaluation of color changes after dental bleaching.

In our study, the ΔEab and ΔE_{00} values for whitening treatments were above the 50%:50% perceptibility thresholds and 50%:50% acceptability thresholds (AT) in dentistry for both groups, indicating that color change was perceptible for most observers.

The results of patients' satisfaction are in line with the subtle differences observed. Despite the statistical difference in the whitening degree between groups after three weeks of treatment for most tools employed, this difference was, in average, only one shade guide unit and did not affect patients' satisfaction or treatment recommendation. As important as using objective tools for color change evaluation is, the use of patient-centered outcomes to assess the efficacy of any given treatment[39] is just as important.

The literature has shown that there are differences between patients' and dentists' expectations with dental bleaching treatment,[40] but patients' opinion is more important than the dentists' perception. This is why patient-reported outcomes, which place patients' values, needs, and beliefs in the center of the healthcare field, seem to be of greater importance than evaluator-centered outcomes.[41]

The choice of the two application times investigated in this study did not impact patients' satisfaction. The chosen protocol for any patient may be based on other features, such as patients' time availability to wear the bleaching tray and patients' comfort.

In short, the reduction in daily use time of the whitening tray with a low HP concentrate gel produced a slightly lower whitening degree than the recommended 120-minute application time and the same risk and intensity of TS. However, both protocols resulted in high patient satisfaction.

CONCLUSIONS

The 30-minute application time of 4% HP for three weeks treatment can result in complete patient satisfaction. However, this treatment protocol does not produce the same whitening degree as the 120-minute protocol. This difference is not noticed when the treatment time of the 30-minute group is extended to four weeks. Tooth sensitivity was low in both protocols.

Acknowledgements

This study was performed by Renata Terra as partial fulfillment of his MS degree at the State University of Ponta Grossa (UEPG), Ponta Gross, PR, Brazil. The authors would like to thank FGM Dental Products for the generous donation of the bleaching products employed in this study. This study was partially supported by the National Council for Scientific and Technological Development (CNPq) under grants 303332/2017-4 and 308286/2019-7 and by the Coordenação de Aperfeiçoamento de Pessoal de Nivel Superior - Brasil (CAPES) - Finance Code 001.

Regulatory Statement

This study was conducted in accordance with all the provisions of the human subjects oversight committee guidelines and policies of the Scientific Review Committee from the State University of Ponta Grossa (protocol number 2.971.337).

Conflict of Interest

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

(Accepted 19 September 2019)

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Laboratory Research

Applicability of Exposure Reciprocity Law for Fast Polymerization of Restorative Composites Containing Various Photoinitiating Systems

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Clinical Relevance

The irradiation time of composite materials should not be shortened based on the exposure reciprocity law. Irradiation times shorter than 10 seconds increase the risk of insufficient polymerization at the bottom surface of a composite increment even when a high-power LED lamp is used.

SUMMARY

Objectives: The exposure reciprocity law (ERL) has been used to calculate the optimal irradiation time of dental composites. This study examined the applicability of ERL for fast polymerization of restorative composites containing various photoinitiating systems using a high-power multi-peak light-emitting diode (LED) lamp.

Methods: Three commercial composites differing in photoinitiating systems were tested: Filtek Ultimate Universal Restorative (FU) with a camphorquinone-amine (CQ-A) photoinitiating system, Tetric EvoCeram (TEC) with CQ-A and (2,4,6-trimethylbenzoyl)phosphine oxide (TPO), and Estelite Σ Quick (ESQ) with CQ and a radical amplified photopolymerization (RAP) initiator. Specimens 2-mm thick were polymerized using a high-power multipeak LED lamp (Valo) at 3 pairs of radiant exposures (referred to as low, moderate, and high) ranging from 15.8-26.7 J/cm^2. They were achieved by different combinations of irradiation time (5-20 seconds) and irradiance (1300-2980 mW/cm^2) as determined with a calibrated spectrometer. Knoop microhardness was measured 1, 24, and 168 hours after polymerization on specimen top (irradiated) and bottom surfaces to characterize the degree of polymerization. The results were

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http://doi.org/10.2341/20-112-L

statistically analyzed using a three-way analysis of variance and Tukey's post hoc tests, α = 0.05.

Results: Microhardness increased with radiant exposure and except for ESQ, top-surface microhardness was significantly higher than that on bottom surfaces. Combinations of high irradiance and short irradiation time significantly increased the top-surface microhardness of TEC at low and moderate radiant exposures, and the bottom-surface microhardness of FU at a low radiant exposure. In contrast, the microhardness of ESQ on both surfaces at high radiant exposure increased significantly when low irradiance and long irradiation time were used. With all tested composites, bottom-surface microhardness obtained at low radiant exposure was below 80% of the maximum top-surface microhardness, indicating insufficient polymerization.

Conclusion: Combinations of irradiance and irradiation time had a significant effect on microhardness, which was affected by photoinitiators and the optical properties of composites as well as spectral characteristics of the polymerization lamp. Therefore, ERL cannot be universally applied for the calculation of optimal composite irradiation time. Despite high irradiance, fast polymerization led to insufficient bottom-surface microhardness, suggesting the necessity to also characterize the degree of polymerization on the bottom surfaces of composite increments when assessing the validity of ERL.

INTRODUCTION

The shortening and simplification of composite restoration procedures is one of the main goals of contemporary dental materials research. Significant progress has been made with the development of self-etch adhesives that have a reduced number of application steps, which has decreased the time necessary for composite placement and the risk of errors compared with traditional etch-and-rinse systems. Further acceleration was accomplished by optimizing composite optical properties and the development of high-power light-emitting diode (LED) polymerization lamps. In addition, more efficient photoinitiating systems than camphorquinone-amine (CQ-A), such as (2,4,6-trimethylbenzoyl)phosphine oxide (TPO) or dibenzoyl germanium compounds[1-3] were incorporated in some products. This progress enabled the polymerization of so-called bulk-fill composites in 4-5 mm increments or the reduction of irradiation time of the standard 2-mm increments to 10 or even less than 5 seconds.

The theoretical basis for the reduction of irradiation time using high-power polymerization lamps is the validity of the exposure reciprocity law (ERL). ERL states that the degree of monomer double-bond conversion (DC) in light-cured composites depends on radiant exposure E (J/cm^2), ie, light energy delivered to the composite surface in the region of photoinitiator absorption wavelengths. Radiant exposure is controlled by 2 independent variables, irradiation time t (seconds) and irradiance I (mW/cm^2), which is the radiant flux incident on cm^2 of the composite surface per second. Based on this definition, the same DC could be achieved at a given radiant exposure either through a shorter irradiation time and proportionally increased irradiance, or a prolonged irradiation time and lower irradiance. However, ERL has its limitations in complex free-radical polymerization of (meth)acrylic monomers. Using simplified models of photoinitiated polymerization, it was derived that radiant exposure should correspond to irradiance to the 0.5-1 power, depending on the termination mechanism.[4,5]

The validity of ERL would allow clinicians and manufacturers to simply estimate the required irradiation time for various irradiances. Several studies[1,6-22] used infrared spectroscopy[1,5-9,12-14,16,17,19-22] or mechanical properties such as hardness[18,19,21] and elastic modulus[9,10,12,16,18,20] to verify ERL. Some of them confirmed its validity,[6-9,13,15,21] but often at clinically inappropriately long irradiation times and low irradiances. Other studies, however, reported that ERL validity depends on photoinitiating systems,[1,16] fillers,[1,14] or composite viscosity.[4,14] They showed a violation of ERL at short irradiation times combined with high irradiance, especially for composites with CQ-A initiating systems.[1,5,10-12,14,16,20] In such a case, the application of ERL could result in insufficient polymerization of the composites, which would adversely affect their mechanical, aesthetic, and biological properties. The majority of these aforementioned results were achieved using quartz tungsten halogen, plasma arc, or low-power LED polymerization lamps. Although modern high-power multipeak LED lamps were used in some recent studies,[18-21] their effect on the validity of ERL with fast curing composites containing various photoinitiating systems has not been fully clarified.

To examine ERL validity, several conditions should be met. Firstly, it is necessary to determine irradiance of the specimen using a calibrated spectrometer. In addition, if composite materials with various

photoinitiating systems are polymerized using multipeak LED lamps, it is important to measure not only the total irradiance but also irradiance at wavelengths corresponding to each peak to determine the effective light energy delivered in the light absorption range of the photoinitiators used. Secondly, the DC should be determined using, eg, infrared spectroscopy or a hardness measurement[23] on both the top (irradiated) surface and the bottom surface, because the incident light is substantially attenuated when passing through a composite. Thirdly, all measurements should be performed not only immediately after irradiation but also when the composite properties stabilize, as postirradiation polymerization[24,25] may alter outcomes.

The aim of this study was to investigate whether ERL could be applied to the polymerization of commercial composite materials activated by various photoinitiators and recommended for fast polymerization using a multipeak LED lamp. The first null hypothesis stated that the microhardness of tested composites measured on the top and bottom surfaces would not depend on radiant exposure, while the second null hypothesis assumed that microhardness at each radiant exposure would not depend on polymerization conditions, ie, combinations of irradiation time and irradiance, regardless of the composite material and photoinitiators used.

METHODS AND MATERIALS

Composite materials included the nanocomposite Filtek Ultimate Universal Restorative (FU, shade A2 dentin; 3M Oral Care, St. Paul, MN, USA), the nanohybrid composite Tetric EvoCeram (TEC, shade A2; Ivoclar Vivadent, Schaan, Liechtenstein), and the "supra-nano-filled" composite Estelite Σ Quick (ESQ, shade A2; Tokuyama Dental, Tokyo, Japan). According to the manufacturers, polymerization of FU is initiated by a CQ-A system, TEC contains a mixture of CQ-A with TPO, and ESQ is based on CQ and a newly developed radical amplified photopolymerization (RAP) initiator that allows a faster and more effective polymerization.[26,27] The composition of these materials is summarized in Table 1.

Irradiance Measurement

A corded multipeak LED polymerization lamp (Valo; Ultradent Products, South Jordan, UT, USA) was used in this study. Valo offers polymerization in 3 modes: standard, high power, and plasma emulation with claimed light intensities[28] of 1000, 1400, and 3200 mW/cm^2, respectively (Table 2). Irradiance measurements in each mode were performed using a USB2000+ spectrometer connected via an optical fiber with a CC-3 cosine corrector (all Ocean Optics, Dunedin, FL, USA). Its 3.9-mm diffuser diameter corresponded to the diameter of composite specimens. Before the measurement, the system was calibrated with a traceable

Table 1: *An Overview of Composite Materials Used*

Composite (Abbreviation; Manufacturer)	Monomers	Fillers	Filler (Load)	Photoinitiating System	Shade (Batch Number)
Filtek Ultimate Universal Restorative (FU; 3M ESPE, St. Paul, MN, USA)	Bis-GMA, UDMA, Bis-EMA, TEGDMA, PEGDMA	SiO_2 and ZrO_2 nanoparticles and their agglomerates	63.3 vol% (78.5 wt%)	CQ-amine	A2 Dentin (N747514)
Tetric EvoCeram (TEC; Ivoclar Vivadent, Schaan, Liechtenstein)	UDMA, Bis-GMA, Bis-EMA	Ba glass, YF_3, oxide mix, prepolymerized filler	53-55 vol% (75-76 wt%)	CQ-amine TPO	A2 (U26271)
Estelite Σ Quick (ESQ; Tokuyama Dental, Tokyo, Japan)	Bis-GMA, TEGDMA	SiO_2 and ZrO_2 particles, composite filler	71 vol% (82 wt%)	CQ RAP initiator	A2 (E654)

Abbreviations: Bis-EMA, ethoxylated bisphenol-A glycol dimethacrylate; Bis-GMA, bisphenol-A-glycidyl methacrylate; CQ: camphorquinone; PEGDMA, polyethylene glycol dimethacrylate; RAP: radical amplified photopolymerization; SiO_2, silicon dioxide; TEGDMA, triethyleneglycol dimethacrylate; TPO, diphenyl(2,4,6-trimethylbenzoyl)phosphine oxide; UDMA, urethane dimethacrylate; YF_3, yttrium(III) fluoride; ZrO_2, zirconium dioxide.

Table 2: *Comparison of Valo Light Intensity Declared by the Manufacturer and Irradiance Measured with a Cosine Corrector Under the Conditions of Specimen Polymerization, ie, Through a 1-mm Thick Microscope Slide and Transparent Plastic Foil*

Polymerization Mode	Declared Light Intensity (mW/cm^2)	Measured Irradiance Mean ± SD (mW/cm^2)		
		Total (380-515 nm)	380-420 nm	420-515 nm
Standard	1000	1300 ± 30	195 ± 5 (15.0%)	1105 ± 12 (85.0%)
High power	1400	1950 ± 28	333 ± 7 (17.1%)	1617 ± 10 (82.9%)
Plasma emulation	3200	2980 ± 30	613 ± 16 (20.6%)	2367 ± 15 (79.4%)

Abbreviation: SD, standard deviation.

light source HL-3P-CAL (Ocean Optics, Dunedin, FL, USA). The data were processed using the SpectraSuite Ocean Optics software. Irradiance was measured in the full range of wavelengths (380-515 nm) as defined by ISO/TS 10650:2019,[29] and in the violet (380-420 nm) and blue (420-515 nm) ranges, activating TPO and CQ, respectively. The measurements were performed 5 times per each mode under specimen polymerization conditions, ie, through a 1-mm thick microscope slide and transparent plastic foil. For all measurements, Valo was carefully centered over the cosine corrector using a custom-made guide and fixed using clamps to avoid any change in its alignment with the cosine corrector. The transmittance of composite specimens was calculated as the ratio of irradiance measured with and without a fully polymerized composite disc (6 mm diameter, 2 mm thickness) placed between the microscope slide and the cosine corrector. The Valo built-in timing intervals for each polymerization mode were measured 10 times with a calibrated stopwatch (Table 3).

Specimen Preparation and Microhardness Measurement

For each composite material, 6 experimental groups of cylindrical specimens (n=5) were prepared in Teflon molds (4 mm diameter, 2 mm thickness). The molds were placed on a microscope slide, laid on white filtration paper, and covered with a transparent plastic foil. The molds were slightly overfilled in 1 increment, covered with another transparent foil and microscope slide, and pressed with a finger to remove excess material. Polymerization at 6 predetermined modes was performed using Valo, with the tip placed concentrically and perpendicularly to the specimen's surface. Approximately 30 minutes after polymerization, top (irradiated) and bottom surfaces were slightly ground under water using P1000, P2500, and P4000 silicon carbide grinding papers and polished using the 3 μm MetaDi II diamond polishing paste on a nylon polishing cloth followed by the suspension of 1 μm MicroPolish II alumina oxide particles on the MicroCloth polishing cloth (all Buehler, Lake Bluff, IL, USA). The specimens were stored in the dark at room temperature.

Table 3: *Polymerization Conditions: Irradiance, Irradiation Time and Radiant Exposure*

Polymerization Setup	Irradiance (mW/cm^2)	Declared Irradiation Time	Real Irradiation Time(s)	Radiant Exposure (J/cm^2) (Uncertainty Interval)
2980/5	2980	5 s (3 s + 2 s)	5.3	15.8 (14.7-16.9) a
1950/8	1950	8 s (2×4 s)	8.5	16.6 (15.4-17.8) a
2980/6	2980	6 s (2×3 s)	6.5	19.4 (18.0-20.8) b
1950/10	1950	10 s (4 s + 2×3 s)	10.6	20.7 (19.2-22.2) b
1950/12	1950	12 s (3×4 s)	12.8	25.0 (23.2-26.8) c
1300/20	1300	20 s	20.5	26.7 (24.7-28.7) c

The uncertainty range of radiant exposures, which corresponds to a 95% confidence interval was calculated as the square root of the sum of squared standard deviations of irradiance (0.3%-0.7%), irradiation time (2%), uncertainties of the stopwatch (0.03%), and the traceable light source (3%) multiplied by 2. Different lowercase letters indicate statistically significant differences between radiant exposures.

Knoop microhardness was measured after 1, 24, and 168 hours (IndentaMet 1600-1105D; Buehler) at 5 locations on both the top and bottom surfaces of the specimen with a 25-g load and 5-second dwell time. The first location was in the center of the specimen, and the other locations on the perimeter in north, south, east, and west positions, approximately 1.5 mm from the middle of the specimen. With TEC and ESQ containing large prepolymerized filler particles of a size comparable with the length of indentations (up to approximately 100 μm), 2 or more indentations outside these particles had to be performed to obtain results varying less than 10%. If more indentations were performed at a single location, their arithmetic mean was calculated. The microhardness values from each location on a surface were then averaged to obtain mean surface microhardness, which was processed statistically.

Scanning Electron Microscopy Analysis

The structure of the composite materials was characterized using a scanning electron microscope ([SEM] JSM 5500-LV; Jeol Inc, Tokyo, Japan) in the backscatter electron mode. The composite surface was polished to a mirror gloss, as stated before, dried for 1 week at room temperature, and sputter coated with a thin layer of gold (JFC-1200 Fine Coater, Jeol Inc).

Statistical Analysis

The statistical analyses of microhardness data measured 1, 24, and 168 hours after irradiation were performed using a three-way analysis of variance (ANOVA), with factors of radiant exposure, postirradiation time, and specimen surface. Tukey's post hoc tests were used for pairwise comparisons. The analyses were performed at $\alpha = 0.05$ using the Statistica software (StatSoft 12, Tulsa, OK, USA). The expanded uncertainty of radiant exposures, which corresponded to a 95% confidence interval, was calculated as a square root of the sum of squared standard deviations of irradiance (0.3%-0.7%), irradiation time (2%), uncertainties of the stopwatch (0.03%), and the traceable light source (3%)[30] multiplied by 2. Differences between radiant exposures were assumed to be statistically significant if their values ± expanded uncertainty did not overlap.

RESULTS

Irradiance

Table 2 summarizes the light power intensities of Valo as declared by the manufacturer and irradiances measured with a cosine corrector under our experimental conditions in the wavelength range of 380-515 nm. The irradiances in standard, high power, and plasma emulation modes were 1300, 1950, and 2980 mW/cm^2, respectively. Based on the irradiances and irradiation times (Table 3), composite specimens were polymerized at clinically applicable conditions: 1300 mW/cm^2 for 20 seconds (referred to as 1300/20); 1950 mW/cm^2 for 12 seconds, 10 seconds, or 8 seconds (1950/12, 1950/10, and 1950/8, respectively); and 2980 mW/cm^2 for 6 seconds or 5 seconds (2980/6 and 2980/5, respectively). Radiant exposures corresponding to these polymerization conditions ranged from 15.8-26.7 J/cm^2. Given the 7.2%-7.4% expanded uncertainty in their determination, 3 pairs of statistically nonsignificantly different radiant exposures referred to as low (15.8 and 16.6 J/cm^2), moderate (19.4 and 20.7 J/cm^2), and high (25.0 and 26.7 J/cm^2) were achieved (Table 3). Due to the preset polymerization modes of Valo, the radiant exposures could not be matched more closely, which would have been optimal for the verification of ERL.

Table 2 shows that the radiant flux of individual diodes did not change proportionally with increasing irradiance but in favor of violet light. In the standard mode, violet light equaled 15% of total irradiance, while in the high power mode it rose to 17.1% and in the plasma emulation mode it increased to 20.6%. After passing through the composite specimens, the intensity of light decreased significantly, as shown in Table 4. The least amount of light was transmitted through FU (approximately 3% of the incident light), followed by TEC with 12% and ESQ with 13%. The attenuation was stronger in the violet region compared with the blue region. In the case of TEC, only ~1.5% of violet light was transmitted, while the transmittance of ESQ and FU for violet light was less than 1%.

Composite Microstructure

The SEM analysis revealed that FU contained a mixture of round polydisperse particles (up to approximately 5 μm in diameter), which are probably agglomerates of nanoparticles (Figure 1A). In contrast, large irregular particles (up to 30-50 μm) with a structure resembling microhybrid composites were observed in TEC (Figure 1B). Even larger filler particles (up to 100 μm) of a heterogeneous structure were found in ESQ (Figure 1C). Their amount seemed to be higher compared with that of TEC, and their distribution in the bulk material was less uniform.

Microhardness

In TEC and ESQ, the microhardness of large prepolymerized filler particles (70-80 KHN for TEC and 80-95 KHN for ESQ) was significantly higher than that of the surrounding composite (up to 51 KHN for

Table 4: *Irradiance Measured Through a 2-mm Thick Disc of Tested Composites (Mean±SD)*[a]

Polymerization mode	Total (380-515 nm)		Violet region (380-420 nm)		Blue region (420-515 nm)	
	Irradiance (mW/cm²)	Transmittance (%)	Irradiance (mW/cm²)	Transmittance (%)	Irradiance (mW/cm²)	Transmittance (%)
Filtek Ultimate Universal Restorative						
Standard	38 ± 2	2.9	0	0.0	38 ± 2	3.4
High power	55 ± 1	2.8	0	0.0	55 ± 1	3.4
Plasma emulation	91 ± 1	3.1	0.7 ± 0.5	0.1	90 ± 1	3.8
Tetric EvoCeram						
Standard	150 ± 9	11.5	2.6 ± 0.2	1.3	147 ± 8	13.3
High power	226 ± 4	11.6	4.9 ± 0.3	1.3	221 ± 4	13.7
Plasma emulation	380 ± 9	12.8	9.4 ± 0.1	1.5	371 ± 9	15.7
Estelite Σ Quick						
Standard	172 ± 3	13.2	0.1 ± 0.1	0.0	172 ± 3	15.5
High power	245 ± 5	12.6	1.2 ± 0.1	0.4	244 ± 5	15.1
Plasma emulation	405 ± 9	13.6	3.7 ± 0.6	0.6	401 ± 8	17.0

[a]*Transmittance was calculated as a ratio of irradiance measured with the disc to irradiance without the disc (see Table 2).*

both TEC and ESQ). If the Knoop indenter interfered with these particles, microhardness scatter increased markedly, which led to the loss of measurement sensitivity to the extent of matrix polymerization. For that reason, the tip of the Knoop indenter was directed outside these particles.

Tables 5-7 present top and bottom surface microhardness of the tested materials after 1, 24, and 168 hours from irradiation. The three-way ANOVA disclosed that all factors (radiant exposure, postirradiation time, specimen surface) were strongly significant ($p<0.001$). For FU and TEC, interactions among pairs of factors were also significant, whereas no significant interaction among factors was revealed for ESQ. Tukey's post hoc tests revealed that the microhardness of all tested composites increased with the time elapsed from light-curing. In the period between 1 and 24 hours, this increase was significant on both surfaces at all radiant exposures for FU and TEC, and at high radiant exposure for ESQ. Microhardness continued to increase between 24 and 168 hours, but the difference was not significant except for a few TEC and ESQ groups. Significantly higher microhardness was found on top surfaces compared with bottom surfaces in all groups of TEC ($p<0.001$) and in all groups of FU ($p<0.001$) except for the highest exposure of 26.7 J/cm² ($p>0.32$). In contrast, top and bottom surface microhardnesses of ESQ were not significantly different ($p>0.30$).

The effects of polymerization conditions — As microhardness increased during the first 24 hours after irradiation, the effect of polymerization conditions was evaluated after 168 hours when the values of microhardness stabilized. With FU (Table 5), top-surface microhardness at low radiant exposures (15.8 and 16.6 J/cm²) was significantly lower than at moderate radiant exposure (19.4 J/cm²) and at high radiant exposures (25.0 and 26.7 J/cm²). Comparisons of FU microhardness on the top surface at various combinations of irradiance and irradiation time 2980/5 vs 1950/8 at low radiant exposures, 2980/6 vs 1950/10 at moderate radiant exposures, and 1950/12 vs 1300/20 at high radiant exposures did not reveal any significant effect of polymerization conditions ($p=1$). On the bottom surface, microhardness increased significantly with radiant exposure except for 15.8 and 20.7 J/cm². The effect of polymerization conditions was significant at low radiant exposures ($p<0.01$), as microhardness at increased irradiance and shorter irradiation time 2980/5 was higher than at 1950/8.

The microhardness of TEC (Table 6) was lower in comparison with FU. On the top surface, microhardness at the low radiant exposure of 15.8 J/cm² and moderate radiant exposure of 19.4 J/cm² was not significantly different from high radiant exposures ($p>0.17$). At low and moderate radiant exposures, significantly higher microhardness was measured for combinations of increased irradiance and shorter irradiation time, ie, microhardness at 2980/5 and 2980/6 was significantly higher than at 1950/8 and 1950/10, respectively. On the bottom surface, microhardness increased gradually with radiant exposure and increased significantly between low and high radiant exposures ($p<0.01$). The

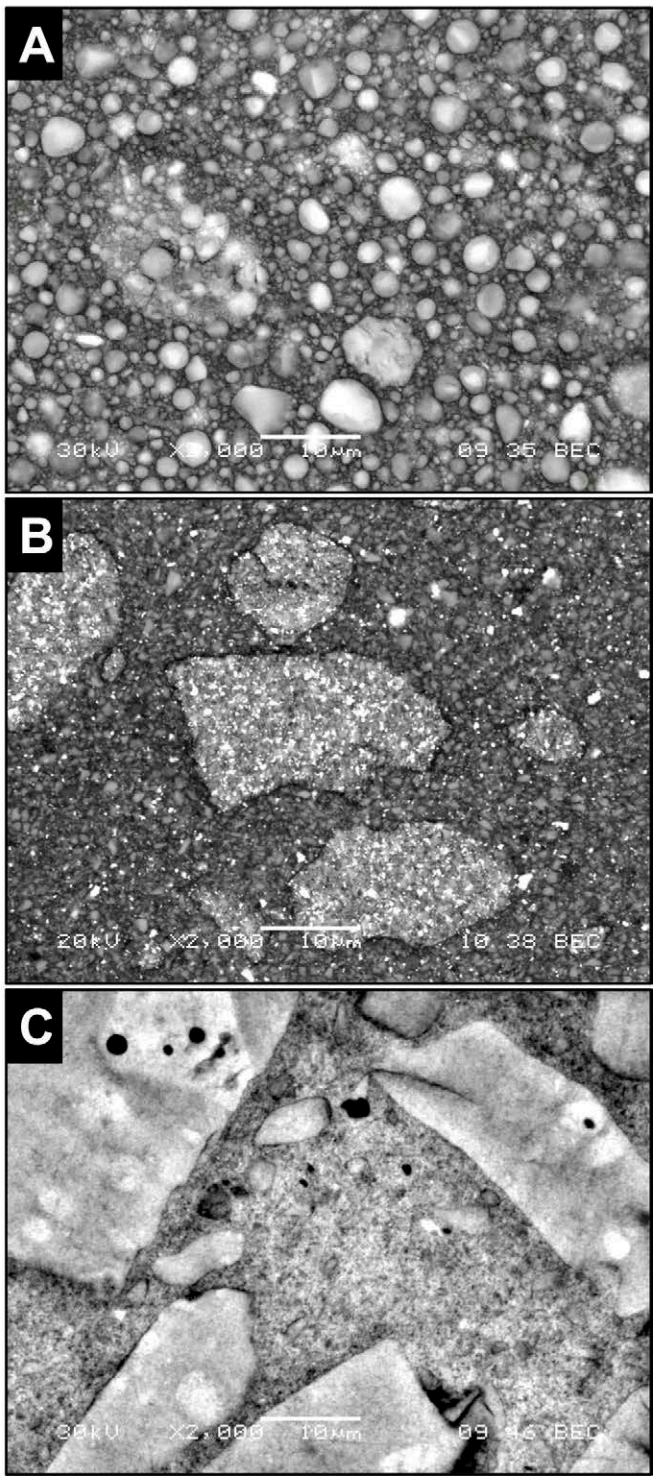

Figure 1. SEM analysis of composite microstructure. Backscatter electron images at 2000x magnification. (A): Filtek Ultimate Universal Restorative: agglomerates of ZrO_2 and SiO_2 nanoparticles. (B): Tetric EvoCeram: prepolymerized composite filler particles. (C): Estelite Σ Quick: heterogeneous prepolymerized composite filler particles. Abbreviations: SEM, scanning electron microscope; SiO_2, silicon di-oxide; ZrO_2, zirconium dioxide.

effect of polymerization conditions within each pair of radiant exposures was not significant ($p>0.9$).

The microhardness of ESQ (Table 7) increased gradually with radiant exposure on both surfaces, leading to significant differences between low and high radiant exposures ($p<0.01$). The effect of polymerization conditions was significant ($p<0.03$) at high radiant exposure on both surfaces, ie, significantly higher microhardness was obtained at mode 1300/20 compared with 1950/12.

Although the influence of radiant exposure on microhardness was material-dependent, none of the materials reached 80% of the maximal top-surface microhardness (62.7 KHN for FU, 40.7 KHN for both TEC and ESQ) or bottom surfaces at low radiant exposures (15.8 and 16.6 J/cm^2), suggesting insufficient polymerization.[31] The microhardness of ESQ at 15.8 J/cm^2 did not surpass the 80% threshold even on the top surface (Table 7).

DISCUSSION

The introduction of high-power polymerization lamps led to a discussion on whether irradiation time could be shortened when irradiance is proportionally increased. This would be applicable if ERL was upheld; however, in the opposite case, short irradiation times calculated using ERL may lead to a lower extent of polymerization, inferior mechanical properties,[24,32-35] an increased release of unreacted components,[36,37] or an increased susceptibility of composites to discoloration.

One of the essential requirements for the verification of ERL is the measuring of irradiance using a calibrated spectrometer or radiometer,[29] as noncalibrated spectrometers or even hand-held radiometers may provide misleading results. The measurement should be performed under the equivalent conditions as specimen polymerization to ensure the same amount of photons incident on the specimen surface as on the detector. Differences between the settings of irradiance measurements and specimen preparation, eg, in the distance of the lamp from the detector/specimen, may result in misinterpretation because irradiance varies with the distance between the tip of the lamp and specimen surface.[18] The detector and the specimen's irradiated surface should also be of a similar diameter, as spatial distribution of the emitted light may be heterogeneous.[38,39]

In this study, the irradiance of composite specimens was measured using a cosine corrector with a 3.9-mm diameter. This corresponded to the central area of the lamp's 9.6-mm tip diameter, where radiant flux is usually the highest.[38,39] In the standard and high power modes, irradiance surpassed the manufacturer's values

Table 5: *Filtek Ultimate Universal Restorative Microhardness Results*

Radiant Exposure (J/cm²)	Top Surface			Bottom Surface		
	1 h	24 h	168 h	1 h	24 h	168 h
15.8 (2980/5)	61.6 ± 2.4 ABa*	69.3 ± 1.9 Ab*	71.7 ± 2.9 Ab*	52.0 ± 1.1 Ba*	58.7 ± 0.5 Bb*	61.3 ± 1.0 Bb*
16.6 (1950/8)	61.2 ± 0.8 Aa*	70.3 ± 1.2 Ab*	72.4 ± 1.1 Ab*	46.0 ± 0.8 Aa*	54.4 ± 1.3 Ab*	56.1 ± 0.8 Ab*
19.4 (2980/6)	65.9 ± 1.2 Ca*	74.5 ± 1.0 Bb*	76.1 ± 0.5 BCb*	59.1 ± 1.5 CDa*	65.8 ± 2.0 Cb*	67.6 ± 1.6 Cb*
20.7 (1950/10)	65.0 ± 1.3 BCa*	72.5 ± 2.7 ABb*	74.5 ± 1.1 ABb*	56.3 ± 1.2 Ca*	63.8 ± 3.0 Cb*	63.9 ± 1.0 BCb*
25.0 (1950/12)	66.9 ± 1.1 Ca*	75.2 ± 1.2 Bb*	78.0 ± 1.4B Cb*	62.2 ± 2.2 DEa*	69.8 ± 1.8 Db*	72.8 ± 2.1 Db*
26.7 (1300/20)	64.0 ± 0.4 ABCa	75.2 ± 1.0 Bb	78.4 ± 1.4 Cb	63.6 ± 0.8 Ea	73.2 ± 0.7 Db	75.3 ± 1.9 Db

Different superscript letters indicate significant differences, uppercase letters in columns and lowercase letters in rows (among storage times within each surface). Asterisks indicate significant differences between corresponding groups on top and bottom surfaces. Polymerization was considered insufficient if bottom-surface microhardness after 168 hours did not reach 62.7 KHN, corresponding to 80% of the maximal top-surface microhardness (78.4 KHN). Abbreviation: h, hours.

by 30%-40% (Table 2), possibly due to the difference in the light-collecting area between the 3.9-mm diameter in our measurement and the 13-mm diameter of the Demetron LED hand-held radiometer used by the manufacturer.[28] In contrast, the irradiance of 2980 mW/cm² measured in the plasma emulation mode was slightly lower than the claimed value of 3200 mW/cm² measured using an integrating sphere with a 4-mm aperture.[28] These results demonstrate that calculations of radiant exposure should not rely on the manufacturer's data. Firstly, the information may be misleading, and secondly, manufacturers specify radiant exitance (mW/cm²) defined as radiant flux emitted by the entire lamp's tip surface per unit area, which is not equivalent to irradiance (mW/cm²), ie, the radiant flux incident on a composite surface per unit area.[29,40] Radiant exitance is therefore a characteristic of the lamp, whereas irradiance represents the light energy actually received by the specimen, and it varies with the distance from the lamp's tip and several other factors. Irradiance equals radiant

Table 6: *Tetric EvoCeram Microhardness Results*

Radiant Exposure (J/cm²)	Top Surface			Bottom Surface		
	1 h	24 h	168 h	1 h	24 h	168 h
15.8 (2980/5)	40.1 ± 0.4 BCa*	47.5 ± 0.9 Cb*	48.2 ± 0.8 BCb*	33.1 ± 0.6 Aa*	37.2 ± 1.1 ABb*	38.5 ± 0.4 Ab*
16.6 (1950/8)	35.6 ± 1.1 Aa*	40.9 ± 1.2 Ab*	44.1 ± 0.8 Ac*	31.8 ± 2.1 Aa*	36.4 ± 1.0 Ab*	39.2 ± 0.7 ABc*
19.4 (2980/6)	40.5 ± 1.3 BCa*	49.3 ± 1.0 Cb*	50.9 ± 0.6 Db*	36.0 ± 1.0 BCa*	40.5 ± 0.6 CDb*	42.1 ± 0.2 Cb*
20.7 (1950/10)	38.9 ± 0.9 ABa*	44.6 ± 0.9 Bb*	46.7 ± 0.2 Bb*	33.9 ± 1.2 ABa*	39.0 ± 0.5 BCb*	41.5 ± 0.1 BCc*
25.0 (1950/12)	41.5 ± 0.4 Ca*	48.5 ± 0.5 Cb*	50.3 ± 0.1 CDb*	37.6 ± 0.7 Ca*	41.7 ± 0.8 Db*	43.6 ± 1.6 Cb*
26.7 (1300/20)	41.7 ± 0.5 Ca*	49.4 ± 0.8 Cb*	49.3 ± 1.9 CDb*	36.3 ± 0.8 Ca*	40.7 ± 1.3 CDb*	42.2 ± 1.8 Cb*

Different superscript letters indicate significant differences: uppercase letters in columns and lowercase letters in rows (among storage times within each surface). Asterisks indicate significant differences between corresponding groups on top and bottom surfaces. Polymerization was considered insufficient if bottom-surface microhardness after 168 hours did not reach 40.7 KHN corresponding to 80% of the maximal top-surface microhardness (50.9 KHN). Abbreviation: h, hours.

Table 7: *Estelite Σ Quick Microhardness Results*

Radiant Exposure (J/cm²)	Top Surface			Bottom Surface		
	1 h	24 h	168 h	1 h	24 h	168 h
15.8 (2980/5)	32.7 ± 0.7 Aa	34.6 ± 0.8 Aab	37.9 ± 0.7 Ab	31.8 ± 1.0 Aa	33.7 ± 0.4 Aa	35.9 ± 0.9 Aa
16.6 (1950/8)	35.6 ± 0.5 ABa	40.6 ± 1.1 Bb	41.7 ± 0.5 ABb	35.5 ± 0.6 ABa	37.8 ± 0.5 ABa	38.8 ± 0.5 ABa
19.4 (2980/6)	40.4 ± 1.4 CDa	44.6 ± 1.5 BCab	45.7 ± 0.6 BCb	37.7 ± 0.8 BCa	41.9 ± 0.5 BCab	43.8 ± 0.6 CDb
20.7 (1950/10)	37.5 ± 1.5 BCa	43.2 ± 0.9 BCb	44.6 ± 0.8 BCb	37.2 ± 2.1 Ba	40.6 ± 1. 0 Bb	42.0 ± 1.1 BCb
25.0 (1950/12)	40.6 ± 2.0 CDa	44.6 ± 3.0 Cb	47.0 ± 3.1 Cb	40.4 ± 2.1 CDa	44.0 ± 3.7 CDb	45.8 ± 3.8 Db
26.7 (1300/20)	43.2 ± 1.4 Da	49.9 ± 1.1 Db	50.9 ± 1.0 Db	41.8 ± 0.3 Da	47.0 ± 1.6 Db	50.1 ± 0.6 Ec

Different superscript letters indicate significant differences: uppercase letters in columns and lowercase letters in rows (among storage times within each surface). Polymerization was considered insufficient if bottom-surface microhardness after 168 hours did not reach 40.7 KHN corresponding to 80 % of the maximal top-surface microhardness (50.9 KHN). Abbreviation: h, hours.

exitance only at zero distance, ie, when the detector/specimen are in contact with the lamp's tip.[40]

When using various radiant exposures in the examination of ERL validity, the uncertainty in the measurement of irradiance and irradiation time should also be taken into account. In this study, 3 pairs of radiant exposures obtained using different combinations of irradiance and irradiation time were selected to clarify the effect of polymerization conditions on the validity of ERL (Table 3). They included the conventional irradiation time of 20 seconds at irradiance 1300 mW/cm² and shorter irradiation times (down to 5 seconds) at irradiance of up to 2980 mW/cm². Variations in spectral irradiance at predefined radiant modes of the Valo lamp equipped with 3 kinds of diodes might be another issue affecting polymerization degree.[41,42] This is important because light emitted in the 420-515 nm region activates CQ, whereas the 380-420 nm wavelength range is effective for TPO. Irradiance measured in these ranges showed that that the proportion of violet light (380-420 nm) increased in the modes with a higher irradiance (Table 2). This increased the number of photons in the violet region, which could yield more radicals from TPO in TEC and contribute to improved polymerization.

When verifying the validity of ERL for dental materials, many studies focused only on the irradiated surfaces or thin films, but it is also essential to evaluate polymerization on the bottom surface of a composite increment where the risk of insufficient polymerization is increased. It is known that transmitted light energy decreases exponentially with increasing composite thickness due to light absorption and scattering.[43] The measurement of irradiance (through a 2-mm thick resin disc) of each composite revealed that the transmittance of ESQ and TEC was approximately 12%-13%, which corroborates previous reports with these materials.[44-46] The transmittance of FU was markedly lower, only around 3%, presumably because a more opaque dentin shade was used. Such an intensive decrease in irradiance was previously observed with some nanohybrid composites.[45] These measurements also revealed that the transmission of violet light (380-420 nm) was lower than that of blue light (420-515 nm), which corresponds to results of several previous studies.[45,47,48] This is due to the Rayleigh scattering of light by particles whose size is much smaller than the wavelength of the incident light, because the amount of scattering is inversely proportional to the fourth power of the light wavelength.

Microhardness is a suitable method for the evaluation of polymerization quality, because it is correlated with DC.[23,31] However, in the case of TEC and ESQ, the measurements were complicated by the presence of large irregular prepolymerized composite filler particles (Figure 1) whose hardness (70-80 KHN for TEC and 80-95 KHN for ESQ) was markedly higher than that of the surrounding composite and apparently independent of the polymerization conditions. These particles interfered with microhardness measurements and could conceal the effect of different exposures on matrix DC, so indentations were performed between them. Nevertheless, due to their high load, especially in ESQ, interference with the indenter could not always

be avoided, and it was necessary to perform multiple measurements to obtain symmetrical indentations and microhardness values, which did not vary by more than 10%. In the case of FU, the size of primary inorganic nanoparticles and their agglomerates was much smaller, up to approximately 5 μm in diameter, and they were homogenously distributed within the resin matrix, allowing for plastic deformation at indentation sites and thus more reliable microhardness measurements.

The microhardness of all tested composites increased significantly during the first day, indicating a high rate of postirradiation polymerization.[24,25] After 168 hours, the microhardness values stabilized and they were used to evaluate the influence of radiant exposure and polymerization conditions on the validity of ERL. Among the tested materials, the CQ-A-based nanocomposite FU exhibited the highest microhardness values. On the top surface, low radiant exposures resulted in significantly lower microhardness compared with higher exposures. In each pair of radiant exposures, the effect of different irradiance and irradiation time was not significant, suggesting the validity of ERL. On the bottom surface, microhardness was significantly lower compared with the top surface except for the highest radiant exposure (26.7 J/cm^2). This can be attributed to the aforementioned low transmittance of FU and hence fewer photons available for CQ photoactivation. Bottom-surface microhardness increased with radiant exposure as well. At low radiant exposures, however, significantly higher microhardness was obtained when higher irradiance was combined with shorter irradiation time, that is at 2980/5 compared with 1950/8, which contradicts ERL. A similar but nonsignificant difference was found between combinations 2980/6 and 1950/10 at moderate radiant exposure. Therefore, it can be speculated that higher irradiance resulted in faster photobleaching of CQ,[49,50] thus decreasing the absorbance of light and allowing an increased number of photons to reach deeper layers of the composite material. This result is contradictory to several previous studies that reported either a similar or lower quality of polymerization of CQ-based composites polymerized at higher irradiances and shortened irradiation times.[5,10-12,16,20] However, most of these studies did not examine polymerization quality in deeper layers of the composites, where the effect of photobleaching could manifest. The microhardness of ESQ increased gradually with radiant exposure on both surfaces, significantly between low and high radiant exposures. On the other hand, even at low exposures, there were no significant differences between top and bottom surface microhardness. This could be due to the transmittance of ESQ for blue light, which was the highest of all the tested composites, and the RAP initiating system, which is supposed to regenerate consumed CQ molecules and hence allow for a higher yield of radicals.[26,27] The regeneration of CQ could also prevent the photobleaching effect speculated for FU. As a consequence, combinations of higher irradiance and shorter irradiation time did not lead to higher microhardness. On the contrary, the influence of polymerization conditions was significant at high radiant exposures; microhardness obtained at 1300/20 was significantly higher than at 1950/12, suggesting that ERL was not upheld in this case.

Among the tested composites, TEC was the only one containing not only CQ-A but also TPO, a very efficient initiator with a high quantum yield. TPO does not require the presence of reducing agents (eg, tertiary amines), as it undergoes α-cleavage (Norrish type I reaction) and forms 2 free radicals.[51] Together with the increased proportion of violet light at the highest irradiance, highest irradiance, TPO presumably contributed to efficient polymerization of the top surface in the plasma emulation mode (2980 mW/cm^2). As a result, top-surface microhardness at low radiant exposure 15.8 J/cm^2 (2980/5) and moderate radiant exposure 19.4 J/cm^2 (2980/6) did not statistically differ from microhardness at high radiant exposures, where the proportion of violet light was lower. In the pairs of similar radiant exposures, microhardness values obtained at 2980/5 and 2980/6 were significantly higher compared with 1950/8 and 1950/10, respectively, which is contradictory to ERL. On the bottom surface, TPO could not be effectively activated because the proportion of violet light decreased to only about 2% of the transmitted radiant flux (Table 4). Consequently, microhardness decreased significantly compared with the top surface, which agrees with previous studies reporting a lower depth of cure of TPO-based composites. It was attributed to the increased scattering of violet light[45,47,48] and to the high molar absorptivity of TPO.[1,52] Microhardness on the bottom surface gradually increased with radiant exposure, and groups with low radiant exposure exhibited significantly lower values than groups with high radiant exposure, similar to FU and ESQ. No significant effect of polymerization conditions was observed in the pairs of similar radiant exposures, indicating that ERL was upheld. Overall, 5/18 groups (~28%) did not follow ERL, and based on these results, the first and second null hypotheses were rejected.

A ratio of bottom-to-top microhardness is an acknowledged criterion of polymerization quality of a 2-mm thick increment.[31] If this ratio is greater than 0.8, ie, the microhardness on the bottom surface surpasses 80% of the maximal microhardness measured

on the top surface, the polymerization is regarded as sufficient.[31] In this study, the criterion was not met at low radiant exposures of 15.8 J/cm^2 (2980/5) and 16.6 J/cm^2 (1950/8) with all tested composites (Tables 5-7). Moreover, the bottom-surface microhardness was just above the 80% threshold even in groups with moderate radiant exposures, especially 20.7 J/cm^2 (1950/10). These findings are noteworthy because radiant exposures used in this study were much higher than the manufacturers' minimal requirements (9 J/cm^2 for ESQ, 10 J/cm^2 for FU and TEC). In clinical practice where polymerization conditions are not ideal, the risk of insufficient polymerization is even higher. The distance between the lamp's tip and the composite surface is usually larger, and their mutual orientation is neither perpendicular nor concentric, thus decreasing the number of photons incident on the composite surface.

CONCLUSION

Within the limitations of this study, it was concluded that ERL cannot be used as a universal rule to calculate irradiation time for preset light intensities of polymerization lamps because composite polymerization was dependent not only on radiant exposure but also on combinations of irradiance and irradiation time. The composite layer thickness should be taken into account as well, because light is substantially attenuated when passing through the material. Despite high irradiance, insufficient polymerization of the bottom surface of a standard 2-mm increment was observed at short irradiation times, suggesting that it is necessary to examine the polymerization of both surfaces in the assessment of ERL validity.

Acknowledgments

Supported by Charles University, project PROGRES Q29/1LF.

Conflict of Interest

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

(Accepted 25 September 2020)

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Better Glass-fiber Post Preservation in Teeth with Ferrule When Subjected to Chewing

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Clinical Relevance

A 2.0-mm remaining dentin ferrule in root-canal–treated premolars helps to hold adhesively luted glass-fiber posts in place underneath crowns exposed to chewing.

SUMMARY

Objectives: To evaluate the influence of ferrule effect and mechanical fatigue aging on glass-fiber post push-out bond strength (PBS) to root-canal dentin at different root thirds of premolars.

Methods and Materials: Thirty-two sound maxillary premolar teeth were collected, and randomly assigned to two experimental groups (n=16): 'Remaining Dentin Ferrule' (RDF) = coronal crown cut 2.0 mm above the cemento-enamel junction (CEJ); 'Without Dentin Ferrule' (WDF) = coronal crown cut at the cemento-enamel junction. Teeth were endodontically treated, post spaces were prepared up to 10.0-mm depth from CEJ, and glass-fiber posts were cemented using a dual-cure self-adhesive composite cement. Standardized cores were built using a light-cure composite, upon which tooth cores were prepared using a 1.5-mm taper ogival-end diamond bur. Crowns were handmade using self-cure acrylic resin and cemented using the aforementioned composite cement. Half of the specimens were subjected to 1,200,000 cycles of mechanical fatigue in a chewing simulator (F = 'Fatigue'), while the other half were stored in distilled water at 37°C

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http://doi.org/10.2341/20-186-L

for 1 week (C = 'Control'). All specimens were horizontally sectioned into 1.0-mm thick slices prior to PBS test; the failure modes were assessed using stereomicroscopy and scanning electron microscopy (SEM). Data were analyzed for each root third using two-way analysis of variance (ANOVA) followed by Tukey HSD post-hoc test; frequency distribution was compared by Chi-square test (α=0.05) and post-hoc comparisons with Bonferroni.

Results: The mean PBS in MPa (SD) were = RDF_F = 10.4 (2.9); WDF_F = 6.9 (1.7); RDF_C = 14.5 (2.7); WDF_C = 14.2 (2.9). Similar PBS were found for the root thirds. For all root thirds, significant differences were found for both the factors Dentin Ferrule and Fatigue, and their interaction (p<0.05). The lowest PBS was found for specimens without dentin ferrule subjected to chewing fatigue (p<0.001). Most failures occurred at the composite cement/dentin interface, followed by mixed and composite cement/glass-fiber post interfacial failures. There was a significant increase in mixed failures for the WDF_F group (p<0.001).

Conclusion: Absence of 2.0-mm remaining dentin ferrule in premolars resulted in a higher decrease of the glass-fiber posts' PBS to dentin after mechanical fatigue, irrespective of root third.

INTRODUCTION

Clinicians routinely use intra-radicular posts to ensure retention of prosthetic crowns when restoring root-canal treated teeth with severe loss of coronal structure.[1-3] Prefabricated glass-fiber posts are intra-radicular posts that became popular among dentists during the last years because their placement requires a relatively short time without any dental lab involvement.[4-6] Cast metal posts may cause staining of the surrounding dentin due to metal corrosion. Even prefabricated stainless steel metallic posts are still unaesthetic compared to tooth-colored glass-fiber posts that enable placement of more translucent restorative materials with a better esthetic outcome.[7,8] Furthermore, the Young's Modulus (E) of glass-fiber posts is similar to that of dentin, which results in a more uniform stress distribution through the remaining tooth structure, along with a lower incidence of root fractures.[4,9,10] However, the excessive stress and strain accumulation at the adhesive interface may contribute to the clinical complication most frequently reported in literature, namely glass-fiber post de-bonding.[4,11-13]

Considering other factors that might have an influence on the long-term survival of post-and-crown restorations, a recent systematic review[14] concluded that posts with high E have better performance when there is no ferrule, as compared to posts with E similar to that of dentin. Thus, the traditional cast metal posts are still indicated in the absence of a ferrule.[14,15] Several finite element analysis (FEA) studies have also suggested that the presence of the so-called ferrule effect may change the distribution of occlusal stress and decrease its accumulation along the adhesive interface.[6,16-20] Among the limited number of long-term clinical trials published on glass-fiber posts, a 6-year follow-up conducted by Ferrari and others[21] reported a lower success rate (39%) for premolars restored with post-and-crown restorations without dentin ferrule. However, the number of glass-fiber posts debonding was exactly the same for both groups with and without dentin ferrule; the difference between success rates was hence related to other failures such as post/core fractures or endodontic failures. Laboratory studies reported immediate bond strength of different post-and-core systems luted using dual-cure composite cements to root-canal dentin.[22,23] However, damage associated with stress caused by occlusion and cyclic chewing in a clinical situation is often not simulated in laboratory studies.[24] Nevertheless, it is not (yet) clear whether absence of remaining dentin ferrule helps to maintain the dentin bond strength of glass-fiber posts cemented with self-adhesive composite cement in premolars restored with post-and-crown restorations that are exposed to a mechanical aging protocol. Thus, the aim of this *in vitro* study was to evaluate the influence of remaining dentin ferrule and mechanical fatigue aging on the glass-fiber post push-out bond strength (PBS) to root-canal dentin at different root thirds of premolars. The null hypotheses tested were that (1) absence of ferrule and (2) mechanical fatigue aging do not have a negative influence on fiber-post PBS to root dentin.

METHODS AND MATERIALS

Sound maxillary premolars extracted for orthodontic reasons were collected from donors. Thirty-two teeth having similar mesiodistal and buccolingual dimensions at the cemento-enamel junction (CEJ) and a root length of 15±1.0 mm were selected, cleaned with a hand scaler, examined for possible defects or cracks, and stored in 0.5% chloramine at 4°C. Teeth were used up to 3 months after extraction. They were embedded in self-cure acrylic resin (JET, Clássico, São Paulo, SP, Brazil) 2.0 mm apically to the CEJ. Impressions of teeth crowns were taken using polyvinyl

siloxane impression material (Express XT, 3M Oral Care, St. Paul, MN, USA) to further use the molds for subsequent provisional crown fabrication.

The experimental procedure is schematically illustrated in Figure 1. Half of the selected teeth had their coronal structure cut 2.0 mm above the CEJ to preserve a remaining dentin ferrule (RDF = Remaining Dentin Ferrule), while the other half were cut at the CEJ to not preserve a dentin ferrule (WDF = Without Dentin Ferrule); for the WDF, a double-faced diamond disk coupled to a precision-cutting machine (Isomet 1000, Buehler, Lake Bluff, IL, USA) was used under constant water cooling. Afterwards, teeth were endodontically treated by a single operator following the step-back technique. Each root canal was prepared up to size #30 (K-File, Dentsply Sirona, Ballaigues, Switzerland) at 1.0 mm from the apex. Preceding the use of each instrument, the root canal was irrigated with 2.5% sodium hypochlorite, upon which the root canal was dried with absorbent paper points. Root canals were obturated using the gutta-percha lateral condensation technique with an epoxy resin-based root-canal sealer (Top Seal, Dentsply Sirona). Coronal access was temporarily filled with a glass-ionomer filling material (Maxxion R, FGM, Joinville, SC, Brazil).

After storage in distilled water at 37°C for 24 hours, post spaces of 12.0-mm length were prepared for the RDF specimens versus the 10.0-mm length for the WDF specimens; this was done using a Gates-Glidden drill (Dentsply Sirona) followed by a standardized low-speed drill of the glass-fiber post system (Exacto, Angelus, Londrina, PR, Brazil). Translucent double taper glass-fiber posts (Exacto #1, Angelus) with 1.4-mm coronal diameter and 0.7-mm apical diameter were positioned in the post spaces and cut at 4.0 mm above the CEJ. The glass-fiber posts were removed from the root canals and cleaned with 96% ethanol. A silane primer (Silano, Angelus) was applied, left on the surface for 60 seconds, and dried with compressed air for 15 seconds. Post spaces were rinsed with distilled water and dried with absorbent paper points. A dual-cure self-adhesive composite cement (RelyX Unicem 2, 3M Oral Care) was handled following manufacturer's instructions, inserted into a nozzle needle tip (AccuDose 20ga NeedleTubes, Centrix, Shelton, CT, USA), and applied into the prepared post space. Glass-fiber posts were inserted into the post spaces and held in position under a constant weight of 1.0 kilograms, upon which the cement was light-cured for 40 seconds using a light-emitting diode light-curing unit (Bluephase 20i, Ivoclar Vivadent, Schaan, Liechtenstein) with an output of

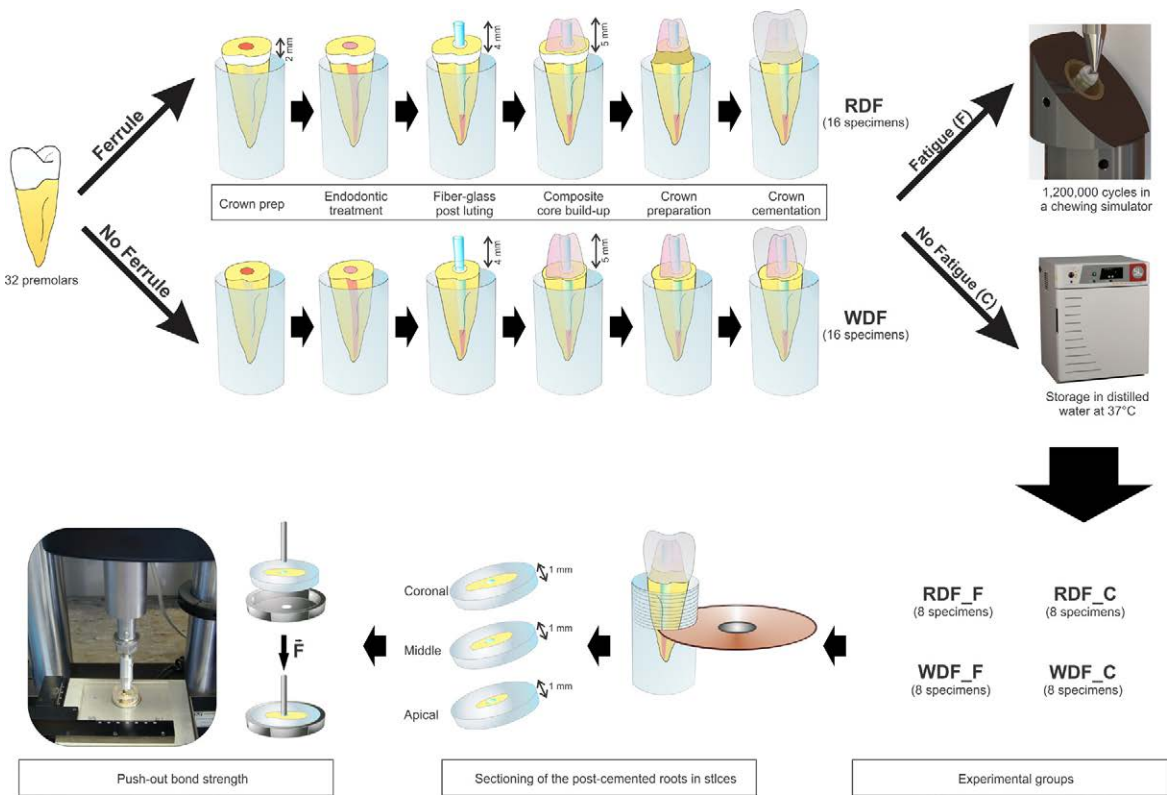

Figure 1. Schematic illustrating the study set-up.

1,200 mW/cm², measured with a light spectrometer (MARC-RC, BlueLight Analytics, Halifax, NS, Canada). After bonding procedures at the coronal area using a two-step etch-and-rinse adhesive (Adper Single Bond Plus, 3M Oral Care), standardized cores of 5.0 mm height were built with the aid of a transparent polyethylene core build-up matrix (CoreForms #11, KaVo Kerr, Brea, CA, USA) using a micro-hybrid light-cure resin composite (Filtek Z250, 3M Oral Care) in increments. Each 2.0-mm increment was light-cured for 20 seconds. A high-speed 1.5-mm taper ogival-end diamond bur (#3216, KG Sorensen, São Paulo, SP, Brazil) was used for crown preparation. The diamond bur was changed after five crown preparations. Tooth preparation involved a 1.5-mm chamfer finishing line at the buccal side and a 0.5-mm chamfer finishing line lingually. The composite cores were finished and polished with abrasive discs (Sof-Lex 2382C and 2382F, 3M Oral Care). Self-cure acrylic resin (JET, Clássico, Campo Limpo Paulista, SP, Brazil) was poured into each polyvinyl siloxane mold and positioned on the corresponding tooth, in order to produce an acrylic resin crown with the original tooth anatomy. After self-curing, the acrylic resin crowns were finished, polished, and cemented using a dual-cure self-adhesive composite cement (RelyX Unicem 2, 3M Oral Care).

After 1-week storage in distilled water at 37°C, half of the specimens were subjected to mechanical fatigue aging for 1,200,000 cycles (F = Fatigue) in a chewing simulator (SD Mechatronik Chewing Simulator, Willytec, Munich, Germany). The specimens were kept in distilled water, and a 6.0-mm diameter stainless-steel ball-shaped stylus was positioned in the center of each occlusal surface. A 50-N load was applied at an angle of 45° to the long axis of each tooth at a frequency of 1.6 Hz (Figure 1). The other half of the specimens were not submitted to mechanical fatigue (C = Control); teeth were stored in distilled water at 37°C for one week. After chewing simulation, all specimens were examined with an exploratory probe under a stereomicroscope with 3.5x magnification for clinical signs of failure such as crown and/or post dislodgment, marginal gap formation, and tooth and/or core fracture. Then, all specimens were sectioned perpendicularly to the long axis of the roots using a water-cooled low-speed diamond saw (Isomet 1000, Buehler). The first section was made at 5.0 mm from the apex, and the root apex was discarded. Then, two 1.0-mm-thick root slices were obtained from each third (coronal, middle, and apical). Specimens were examined under a stereomicroscope with 3.5x magnification to evaluate if the slices contained defects caused by sectioning. Each slice was positioned with its coronal aspect directed downwards on a push-out jig attached to a universal testing machine (Instron 4444, Instron, Canton, MA, USA). After positioning a cylindrical plunger on the center of the fiber post without any contact with surrounding dentin walls, a 0.5-min/mm force was applied in an apical-coronal direction until failure. PBS values were calculated by using the formula $R = L/A$, where L was the maximum load at failure (N) and A the bonded area (mm²), which was determined by using a formula to calculate the lateral area of a circular straight cone with parallel bases: $A = \pi \times g \times (R1 + R2)$, where $\pi = 3.14$, g = inclination, R1 = smaller base radius, and R2 = larger base radius. The inclination was calculated by using the formula $g = h^2 + (R2 - R1)^2$, where h = section height, measured with a digital caliper (Starrett 727, Starrett, Itu, Brazil), while R1 and R2 were obtained by measuring the internal diameters of the smaller and larger bases with an X-Y multipurpose modular measuring microscope equipped with a digital readout (Leitz VRZ-U, Wetzlar, Germany). The PBS data were analyzed for each root third separately. The Shapiro-Wilk test was used to check for data normal distribution (α=0.05), upon which the statistical software program SPSS 21.0 (IBM, Chicago, IL, USA) was employed to detect statistical differences using two-way analysis of variance (ANOVA) (dentin ferrule × mechanical fatigue), followed by applying Tukey's HSD *post-hoc* test (α=0.05).

The failure mode of each specimen was assessed using a stereomicroscope with 3.5x magnification. Failures were classified as adhesive between composite cement and dentin, adhesive between composite cement and glass-fiber post, cohesive fracture of the glass-fiber post, cohesive fracture of dentin, cohesive fracture of the composite cement, or mixed in case of a combination of at least two failure modes. The frequency distribution of failure modes among the experimental groups was statistically analyzed using the Person Chi-squared test (a=0.05) and *post-hoc* comparisons with Bonferroni. Representative specimens of the most frequent failures were observed under SEM (JSM-6610LV, JEOL, Tokyo, Japan).

RESULTS

No pre-test failures were recorded for all experimental groups tested. The PBS data for each root third and their respective pairwise comparisons are detailed in Table 1 and Figure 2. Both the factors Dentin Ferrule and Fatigue, and their interaction were significant for all root thirds (p<0.05). Irrespective of the root third, specimens not subjected to mechanical fatigue presented significantly higher PBS (RDF_C and WDF_C) (p<0.001) than specimens subjected to

Table 1: *Push-out Bond Strength (MPa) and Failure-mode Distribution (%) at Each Root Third (mean ± SD) (n=48)*[a]

Coronal				
Group	**PBS (MPa)**	**Failure mode (%)**		
		C/D	**C/P**	**M**
RDF_F	10.4 ± 2.7[b]	79	6	15
WDF_F	7.6 ± 1.6[c]	60	13	27
RDF_C	14.8 ± 3.7[a]	90	6	4
WDF_C	14.2 ± 3.3[a]	90	4	6
Middle				
Group	**PBS (MPa)**	**Failure mode (%)**		
		C/D	**C/P**	**M**
RDF_F	9.9 ± 1.6[b]	88	4	8
WDF_F	7.1 ± 1.9[c]	69	10	21
RDF_C	14.4 ± 2.5[a]	90	6	4
WDF_C	14.1 ± 3.2[a]	92	6	2
Apical				
Group	**PBS (MPa)**	**Failure mode (%)**		
		C/D	**C/P**	**M**
RDF_F	10.8 ± 4.5[b]	84	4	12
WDF_F	5.9 ± 1.7[c]	67	12	21
RDF_C	14.4 ± 1.8[a]	88	8	4
WDF_C	14.3 ± 2.3[a]	94	4	2

Abbreviations: C/D = adhesive failure at composite cement/dentin interface; C/P = adhesive failure at composite-cement/glass-fiber post interface; M = mixed failure; PBS = push-out bond strength; RDF_C = remaining dentin ferrule without (control); RDF_F = remaining dentin ferrule + fatigue; WDF_C = without dentin ferrule (control); WDF_F = without dentin ferrule + fatigue.

[a]*For each root third, different lowercase letters in the same column indicate significant difference* ($p<0.05$).

mechanical fatigue (RDF_F and WDF_F). For the experimental groups that were aged by mechanical fatigue, significantly lower PBS ($p<0.05$) was registered when there was no ferrule (WDF_F), as compared to when a dentin ferrule was remaining (RDF_F).

A similar failure-mode distribution was observed for all root thirds. Cohesive failures limited to the glass-fiber post, dentin, or composite cement were not observed. Most failures were adhesive between composite cement and dentin (Figure 3), followed by mixed failures (Figure 4) (Table 1). All observed mixed failures combined an adhesive failure at the composite cement/dentin interface and a cohesive fracture within root-canal dentin. The Pearson chi-square test revealed significant differences in failure modes among the experimental groups for all root thirds ($p=0.006$ coronal; $p=0.022$ middle; $p=0.013$ apical). A significant decrease of adhesive failures at the composite cement/dentin interface and an increase of mixed failures were found for WDF_F ($p<0.001$) concerning all root thirds.

DISCUSSION

The present research aimed to verify the 2.0-mm dentin remaining ferrule effect on glass-fiber post PBS to root dentin. A significant influence of the ferrule effect on PBS was only observed for the specimens that were subjected to mechanical fatigue aging. Thus, the first null hypothesis partially failed to be accepted. After chewing simulation, the presence of 2.0-mm remaining coronal dentin was not able to maintain the PBS to

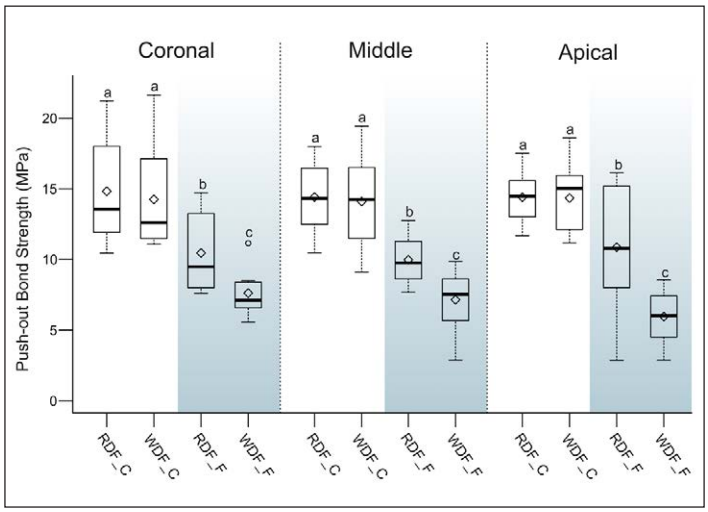

Figure 2. *Box plots of the push-out bond-strength results for each root third. The box represents the spreading of the data between the first and third quartile. The central horizontal line and the diamond symbol represents the median and mean, respectively. The whiskers extend to the minimum and maximum values measured, with the exception of the outliers that are represented with open dots. Abbreviations: RDF_C = remaining dentin ferrule (control); RDF_F = remaining dentin ferrule + mechanical fatigue; WDF_C = without dentin ferrule (control); WDF_F = without dentin ferrule + mechanical fatigue).*

root-canal dentin in comparison to the specimens that were not subjected to mechanical aging. Moreover, the absence of 2.0-mm remaining coronal dentin resulted in even significantly lower results. These results corroborate with previous FEA findings that observed a reduction in tensile stress at both the glass-fiber post/cement and the cement/crown interface when a ferrule was present.[6,17] Scientific literature shows that preservation of a dentin ferrule with sufficient height (1.5 to 2.0 mm) and thickness (1.0 mm) resulted in reduction of the wedging force of the glass-fiber post against the root structure. It consequently decreased stress at the adhesive interface, reducing the risk of failure during mastication.[3,16,19,20,25-27]

Regardless of the presence of ferrule, the present research showed that a significant lower PBS was only found for the specimens that were subjected to mechanical fatigue aging. Thus, the second null hypothesis failed to be accepted. These results indicate that mechanical fatigue aging of the specimens plays an important role when applying a PBS-test protocol and should be applied to specimens whenever possible. Although no glass-fiber post de-bonding was recorded for both aged groups, the significantly lower PBS recorded for the non-ferrule group (WDF_F) may be related to large micromovement of the glass-fiber post during chewing, thereby probably also increasing microleakage.[28] Chang and others[29] observed a dramatic increase in microleakage extension up to the glass-fiber post space after only 120,000 cycles of mechanical fatigue aging. This effect may be regarded as preceding PBS decrease and possible future glass-fiber post de-bonding. A six-year randomized controlled clinical trial performed by Ferrari and others[21] pointed out that preservation of dentin ferrule significantly reduced the failure risk of restored pulpless premolars. A possible explanation for the results reported by Ferrari and others[21] may concern the lower microleakage observed for those restored teeth presenting dentin ferrule and reduced post de-bonding and/or endodontic effects. A recent systematic review and meta-analysis[27] found that restorations exhibiting dentin ferrule showed a higher survival rate (88.4%) compared to those without dentin ferrule (78.1%). Nevertheless, no statistically significant difference was noted regarding general post failures and root fractures, although a higher number of failures were associated with non-ferrule restorations.[27]

The 45-degree oblique loading applied in the present study aimed to simulate the worst-case scenario, in which non-axial forces may induce bending moments and non-uniform stress distribution.[30] In addition, single-root and endodontically treated upper premolars have been shown to be more prone to root fractures, which is the justification for the selection of these teeth in this study.[1]

The predominance of adhesive failures between composite cement and root-canal dentin confirmed that this interface remains the weakest link of teeth restored with post systems.[31,32] The WDF_F group showed a significant increase in mixed failures that involved adhesive failure at the composite cement/dentin interface and cohesive fracture within root-canal dentin. Thus, repetitive forces exerted by the glass-fiber post towards the root-canal dentin during mechanical fatigue aging may cause minor dentin cracks that more easily propagated during testing, having resulted in lower PBS. On the other hand, the presence of dentin ferrule seems to attenuate, to a certain extent, crack initiation and propagation, since a lower percentage of mixed failures was observed for the RDF_F group. The fewer failures between composite cement and glass-fiber post indicated that silanization was able to provide

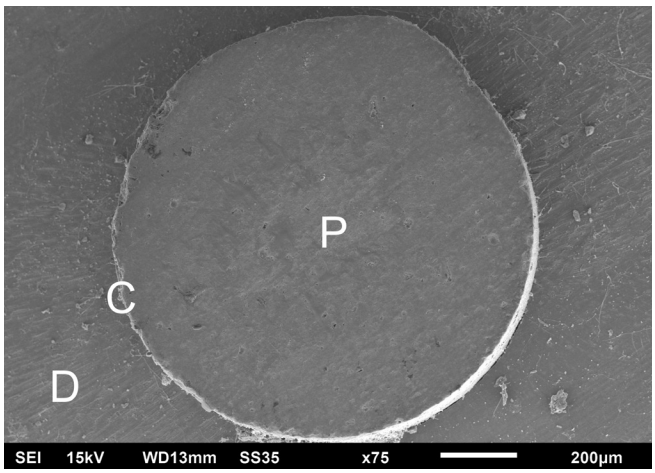

Figure 3. *Representative SEM photomicrograph of the most predominant failure mode for all groups (adhesive failure at the composite cement/dentin interface). Abbreviations: C, composite cement; D, dentin; P, glass-fiber post.*

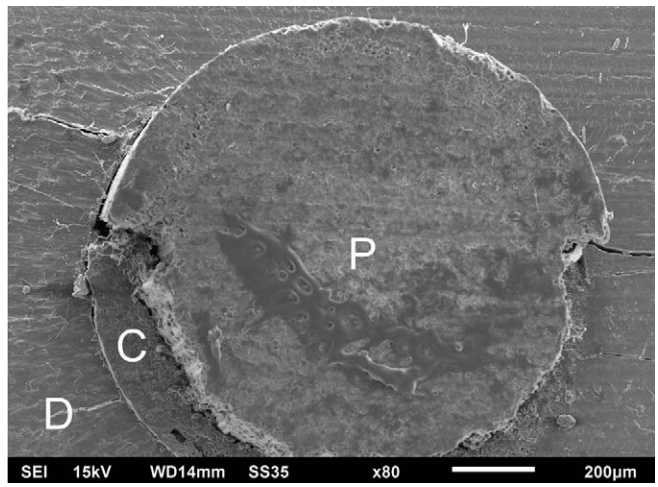

Figure 4. *Representative SEM photomicrograph of a mixed failure. Note the crack propagation within adjacent dentin. Note the crack propagation within adjacent dentin Abbreviations: C, composite cement; D, dentin; P, glass-fiber post.*

adequate chemical bonding and superior bond strength in comparison to the strength of the composite cement/dentin interface within the root canal. Nevertheless, even this type of cement/post failure can be reduced if additional surface pre-treatment of the post would have been conducted, such as hydrogen peroxide cleaning, air-abrasion with 30-mm (silica-coated) aluminum oxide particles.[33,34] Future research should address such additional fiber-post surface pre-treatments in order to evaluate their influence on PBS.

Since glass-fiber posts have a weak mechanical retention to root-canal dentin, their dislodgment resistance relies on the quality of adhesion of the composite cement to root-canal dentin.[27,35] A recent systematic review[23] showed that self-adhesive composite cements can provide adequate root-canal dentin bond strength, and that self-adhesive composite cements are less influenced by other variables, such as the composite-cement application method, operator experience, and fiber-post pre-treatment. When compared to multi-step resin-based luting systems, the single-step self-adhesive composite cement used in the present study is less technique-sensitive,[36] which may explain the similar PBS values recorded among the root thirds.[37] In principle, self-adhesive composite cements bond less effectively to dentin than adhesive-assisted composite cements. Because the root canal surface area to bond to is relatively large, the lower bonding potential is perhaps compensated for by the larger area. A recent multicenter randomized controlled 6-year clinical trial compared two glass-fiber post cementation strategies, namely using a two-step etch-and-rinse adhesive-assisted composite cement versus using a self-adhesive composite cement.[38] The survival rate of glass-fiber posts was not influenced by the cementation strategies.[38]

There still exists no consensus regarding the contribution of ferrule effect on survival of teeth restored with a glass-fiber post.[27,39] More clinical trials are still needed to determine the influence of remaining coronal dentin on survival of teeth restored with a glass-fiber post. Moreover, well-designed clinical trials to detect differences in survival, for example considering the variables "prefabricated post system" or "presence of post," are difficult to be executed, as the research design may require a sample size of over 500 per experimental group.[40] *In vitro* studies are simpler and more convenient and are thus useful in obtaining meaningful results. Further *in vitro* studies should, for instance, also investigate whether absence of ferrule has an influence on PBS when composite cements are used with different adhesive strategies (etch-and-rinse versus self-etch).

CONCLUSION

Absence of 2.0-mm dentin remaining ferrule in premolars restored with glass-fiber posts resulted in a higher reduction of the push-out bond strength to root-canal dentin after mechanical fatigue aging. PBS did not differ depending on the root third. The interface of composite cement with root-canal dentin appears to be the weakest link of glass-fiber post systems cemented with self-adhesive composite cement.

Regulatory Statement

This study was conducted in accordance with all the provisions of the human subjects oversight committee guidelines and

policies of the Federal University of Santa Catarina Ethics Committee for Human Research. The approval code issued for this study is #1.342.024.

Conflicts of Interest

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

(*Accepted 30 September 2020*)

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Effect of In-office Bleaching on Color, Translucency, and Whiteness Variations in CAD-CAM Monolithic Materials

RC Peña • R Simóes • AC Ramos • LN Dovigo • RG Fonseca

Clinical Relevance

The optical properties of previously stained Lava Ultimate (LU)—Vita Enamic (VE) and Vita Suprinity (VS)—benefited by a single session of in-office bleaching, without adversely providing subsequent clinically unacceptable variations on their color, translucency, and whiteness.

SUMMARY

Little is known about the impact of bleaching on the optical properties of computer-aided design and computer-aided manufactured (CAD-CAM) monolithic materials. The aim of the present study was to evaluate the effect of one session of in-office bleaching on stain removal, staining susceptibility, translucency, and whiteness variations of CAD-CAM monolithic materials. Disks were fabricated from Lava Ultimate (LU), Vita Enamic (VE), Vita Suprinity (VS), and IPS e.max CAD (IPS). A spectrophotometer was used to register Commission Internationale de l'Eclairage L*a*b* coordinates. For stain removal, 80 specimens from each material were assessed at baseline (R_0) and after immersion in deionized water or coffee for 36.5 days followed or not by bleaching with 40% hydrogen peroxide (R_1). For staining susceptibility, 80 specimens from each material were analyzed at baseline ($R_{0'}$), and after having been bleached or not and immersed in deionized water or coffee ($R_{1'}$). Both analyses were

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http://doi.org/10.2341/20-119-L

calculated as the color difference (ΔE_{00}) between R_1-R_0 and $R_{1'}$-$R_{0'}$, respectively. Differences in translucency (ΔTP_{00}) and whiteness (ΔWI_D) between R_1-R_0 and $R_{1'}$-$R_{0'}$ were also calculated. Data were analyzed by three-way ANOVA and the Games-Howell post hoc test (α=0.05). Clinical significance was based on 50%:50% perceptibility and acceptability thresholds for ΔE_{00}, ΔTP_{00} and ΔWI_D, respectively. Surfaces were analyzed by scanning electron microscopy. Coffee increased ΔE_{00} in LU, VE, and VS, and decreased their translucency and whiteness, whereas the IPS had only its whiteness affected. Bleaching after immersion in coffee decreased ΔE_{00} in LU and VE, and increased translucency and whiteness of LU, VE, and VS. No effect was observed on IPS. Bleaching before immersion in coffee decreased translucency of LU, but within the acceptable interval, while VE exhibited lower ΔE_{00}, and became more translucent and less dark. Both VS and IPS were not affected. One session of in-office bleaching benefited optical properties of the previously stained LU, VE, and VS, without increasing their susceptibility to staining or adversely providing clinically unacceptable variations in their translucency and whiteness. All variations exhibited by the IPS were below the perceptible threshold.

INTRODUCTION

The possibility of obtaining, in a single patient visit, well-adapted restorations that meet the esthetic demand of demanding patients was achieved, thanks to advances in computer-aided design and computer-aided manufacturing (CAD-CAM) technology.[1,2] In recent years, CAD-CAM monolithic materials have been introduced, including composite resin, polymer-infiltrated ceramic, zirconia-reinforced lithium silicate glass ceramic, and lithium disilicate glass ceramic. Despite some common indications, they exhibit very different compositions and microstructures,[3] and, consequently, different behaviors towards the same challenge.[4-6]

These materials are exposed in the oral cavity in constant contact with beverages and foods rich in pigments, which may affect the optical properties in a higher or lower degree, depending on the materials' structure and chemical composition.[7] Some of these materials, especially those containing triethylene glycol dimethacrylate (TEGDMA) [Lava Ultimate (LU) and Vita Enamic (VE)] and bisphenol A-glycidyl methacrylate (Bis-GMA) (LU) monomers absorb water to a potentially harmful extent,[8] which makes them susceptible to staining.[9-13] This fact can be observed in some studies,[9-12] such as that by Acar and others,[9] who found unacceptable and perceptible color differences after LU and VE were thermocycled in coffee. Other studies[10-13] also reported clinically unacceptable color differences for both materials after immersion in different staining solutions. In addition, ceramic materials may also be stained, as can be seen in two studies,[12,13] in which the IPS Empress CAD showed a clinically unacceptable color difference in cress and red wine, and in the study by Eldwakhly and others,[14] who found that the Celtra Duo and Lava Plus were significantly stained by cola and ginger, respectively.

Even with the proven susceptibility to staining, only four studies[11,15-17] were found that investigated the effect of bleaching on the color parameters of CAD-CAM monolithic materials, being that, in only two of them,[11,15] the materials were stained prior to bleaching. Karakaya and Cengiz[11] observed partial or total recovery of translucency and whiteness after LU and VE were bleached with 40% hydrogen peroxide. Alharbi and others[15] also found favorable results in color difference (ΔE) after using the same bleaching agent on the same materials. Both studies[11,15] stated that bleaching can be considered as an alternative method for the treatment of stained restorations. In addition to the scarcity of information, these two studies did not evaluate ceramic materials that are also susceptible to staining.[12,13]

Another question that arises is whether these materials, once bleached, become more susceptible to staining. It is known that porcelains[18-23] and composite resins[24-27] exposed to bleaching gels may undergo changes in their topography, as well as an increase in roughness, which might render the materials even more susceptible to changes in their optical properties when subsequently exposed to pigments. To date, no study with a similar purpose involving CAD-CAM monolithic materials was found. The lack of studies that have evaluated the behavior of CAD-CAM monolithic materials after bleaching leaves the dentist without knowing whether it really is necessary to protect these materials from the bleaching agents, or if they might benefit from bleaching. Therefore, the purpose of the present *in vitro* study was to evaluate the effect of an in-office bleaching agent on stain removal, staining susceptibility, as well as on translucency and whiteness variations of CAD-CAM monolithic materials. The null hypothesis was that their optical properties would not be affected by one single session of in-office bleaching performed before or after staining.

METHODS AND MATERIALS

Specimen Preparation

The materials tested are listed in Table 1. CAD-CAM blocks were milled into cylinders (Ø=7.0 mm) and sliced into 1.2 ± 0.02 mm disks with a precision saw (IsoMet 1000; Buehler). Some burrs of the disks were finished with a ceramic polisher (Exa Cerapol 0361HP; Edenta AG), and the IPS e.max CAD (IPS), and Vita Suprinity (VS) disks were crystallized (Programat P310; Ivoclar Vivadent AG) according to the manufacturer's instructions. For the IPS, the standby temperature was 403°C followed by a 6-minute closing time. The first firing temperature of 820°C was reached with a heating rate of 90°C per minute and held for 10 minutes, and the second firing temperature of 840°C was reached with a heating rate of 30°C per minute and held for 7 minutes. The first vacuum was held between 550°C and 820°C and the second vacuum between 820°C and 840°C. The long-term cooling was at 700°C. For the VS, the standby temperature was 400°C followed by a 4-minute closing time. The firing temperature of 840°C was reached with a heating rate of 55°C per minute and held for 8 minutes. The first vacuum was held at 410°C and the second vacuum at 839°C. The long-term cooling was at 680°C. Next, the disks were polished with silicon carbide papers (600, 1200, 1500 grits) under water irrigation.

Staining Solutions

The coffee capsules (Ristretto; Nespresso) were prepared in the long mode of the coffee machine Essenza Mini (Nespresso). To simulate a 1-year exposure, the specimens were immersed in the coffee for 30 minutes daily for 36.5 days, considering an intake frequency of 3 cups/day, and an exposure time of 60 seconds/cup.[28] Following each 30 minute coffee exposure, the specimens were washed and stored in deionized water until the next exposure. The specimens of the control group were stored for 36.5 days in deionized water, which was changed daily.

Bleaching Procedures

The in-office bleaching with 40% hydrogen peroxide (Opalescence Boost PF; Ultradent Products Inc, South Jordan, UT, USA) was performed in one single session, consisting of three applications of 20 minutes each, according to the manufacturer's instructions. After each one of the three applications of the bleaching gel, the specimen was washed with distilled water and gently dried with absorbent paper.

Color Analysis

The color analysis was assessed using a spectrophotometer (CM-2600d/2500d; Konica Minolta) coupled with the OnColor V5; CyberChrome

Table 1: *Materials Evaluated*

Material/Batch	Classification	Composition	Manufacturer
Lava Ultimate (LU)/ NA666576	Resin nanoceramic	Agglomerated nanoparticles of silica and zirconia (80% by weight), highly cross-linked polymer matrix composed of bisphenol A-glycidyl methacrylate (Bis-GMA), urethane dimethacrylate (UDMA), bisphenol A diglycidyl methacrylate ethoxylated (Bis-EMA), and triethylene glycol dimethacrylate (TEGDMA) (20% by weight). Particle sizes: 20 nm silica particles, 4-11 nm zirconia particles	3M Oral Care (St Paul, MN, USA)
Vita Enamic (VE)/ 42650	Polymer-infiltrated ceramic network	Fine structure feldspathic ceramic (86% by weight), resin polymer composed of UDMA and TEGDMA (14% by weight)	Vita Zahnfabrik (Bad Sackingen, Germany)
Vita Suprinity (VS)/ 48945	Zirconia-reinforced lithium silicate ceramic	56%-64% SiO_2, 15%-21% Li_2O, 8%-12% ZrO_2, 1%-8% other oxides	Vita Zahnfabrik (Bad Sackingen, Germany)
IPS e.max CAD (IPS) (control material)/ T28586	Lithium disilicate ceramic	57%-80% SiO_2, 11%-19% Li_2O, 0%-13% K_2O, 0%-11% P_2O_5, 0%-8% ZrO_2, 0%-8% ZnO, 0%-5% Al_2O_3, 0%-5% MgO	Ivoclar Vivadent AG (Schaan, Liechtenstein)

software, with UV excluded, D65 primary illuminant, d/2° (diffuse illumination, 2° viewing angle), in MAV mode so that the device reads the entire surface of the specimen. The intrinsic error of the device was ΔE_{00}=0.22. Before the readings, a reproducibility study of the color measurements was performed, in which four specimens from each material were submitted to color reading at two different times by one single operator who performed all the readings in the present study. Concordance was assessed using the intraclass coefficient (ICC) and 95% confidence intervals, with the following results: L^*ICC=0.998 (95% CI, 0.993-0.999) p<0.001; a^*ICC = 0.999 (95% CI, 0.997-1) p<0.001; b^*ICC=1 (95% CI, 0.999-1) p<0.001.

The Commission Internationale de l'Eclairage (CIE) L^*, a^*, and b^* color coordinates were measured three times for each specimen on black (L^*=27.80, a^*=-0.13, and b^*=-0.95) and white (L^*=99.46, a^*=-0.12, and b^*=-0.14) ceramic backgrounds. For the stain removal analysis, 80 specimens from each material were analyzed after storage in deionized water for 24 hours (R_0). Specimens were then immersed in deionized water (control group) (n=40) or coffee (n=40), and exposed (n=20) or not (n=20) to in-office bleaching before the second reading (R_1), which was also performed after storage in deionized water for 24 hours. Stain removal was calculated as the color difference between R_1–R_0. For the staining susceptibility analysis, the color of 80 specimens from each material was registered after storage in deionized water for 24 hours ($R_{0'}$). Next, the specimens were bleached (n=40) or not (n=40) and immersed in coffee (n=20) or deionized water (n=20) before the second reading ($R_{1'}$). The staining susceptibility was the color difference between $R_{1'}$ and R_0, that is ($R_{1'}$-$R_{0'}$).

Color differences were calculated by using the CIEDE2000 formula (1:1:1)[29]:

$$\Delta E_{00} = [(\Delta L'/K_L S_L)^2 + (\Delta C'/K_C S_C)^2 + (\Delta H'/K_H S_H)^2 + R_T (\Delta C'/K_C S_C)(\Delta H'/K_H S_H)]^{1/2}, \quad (1)$$

where ΔE_{00} defined the color difference between the two readings, and $\Delta L'$, $\Delta C'$, and $\Delta H'$ are the differences in lightness, chroma, and hue between the two readings. The parametric factors K_L, K_C, and K_H are correction terms for experimental conditions. S_L, S_C, and S_H are weighting functions for adjustment of the total color difference for variation in perceived magnitude, with variation in the location of the color coordinate difference between two color readings. R_T (rotation function) is a function that accounts for the interaction between the chroma and hue differences in the blue region. Since the oral cavity is dark, the L^*, a^*, and b^* color coordinates used to calculate the ΔE_{00} were those registered on a black background.[30] For color differences (ΔE_{00}), a 50%:50% perceptibility threshold and 50%:50% acceptability threshold were taken as 0.8 and 1.8,[31] respectively, being that: $\Delta E_{00} \leq$ 0.8 (imperceptible), $0.8<\Delta E_{00}\leq 1.8$ (acceptable), and $\Delta E_{00}>1.8$ (unacceptable).

The Translucency Parameter (TP_{00}) values were calculated according to the CIEDE2000 (1:1:1) formula[32]:

$$TP_{00} = \{[(L'_B - L'_W)/K_L S_L]^2 + [(C'_B - C'_W)/K_C S_C]^2 + [(H'_B - H'_W)/K_H S_H]^2 + R_T [(C'_B - C'_W)/K_C S_C][(H'_B - H'_W)/K_H S_H]\}^{1/2}, \quad (2)$$

where letters B and W represent the color factors over black and white backgrounds. TP_{00} changes between 0 and 100. Values close to 100 represent high translucency and close to 0 represent high opacity.

Differences in TP_{00} between R_1-R_0 and $R_{1'}$-$R_{0'}$ were evaluated considering the 50%:50% perceptibility and 50%:50% acceptability thresholds for translucency already determined in the literature, being: $\Delta TP_{00}\leq 0.62$ (imperceptible), $0.62<\Delta TP_{00}\leq 2.62$ (acceptable), and $\Delta TP_{00}>2.62$ (unacceptable).[32] A negative ΔTP_{00} means that the final TP_{00} was less translucent than the baseline.

The Whiteness Index for Dentistry (WI_D) values were calculated according to the following equation[33]:

$$WI_D = 0.511L^* - 2.324a^* - 1.100b^*. \quad (3)$$

As for the calculation of the ΔE_{00}, the L^*, a^*, and b^* color coordinates used to calculate WI_D were those registered on a black background.[34] Higher and lower WI_D values indicate whiter and darker specimens, respectively.

Differences in WI_D between R_1-R_0 and $R_{1'}$-$R_{0'}$ were evaluated considering the 50%:50% perceptibility and 50%:50% acceptability thresholds for whiteness determined in the literature: $\Delta WI_D\leq 0.94$ (imperceptible), $0.94<\Delta WI_D\leq 2.95$ (acceptable), and $\Delta WI_D>2.95$ (unacceptable).[34] A negative ΔWI_D indicates a decrease in whiteness compared to that of the baseline.

Scanning Electron Microscopy (SEM)

For the SEM analysis, two additional specimens from each material were obtained—one being bleached

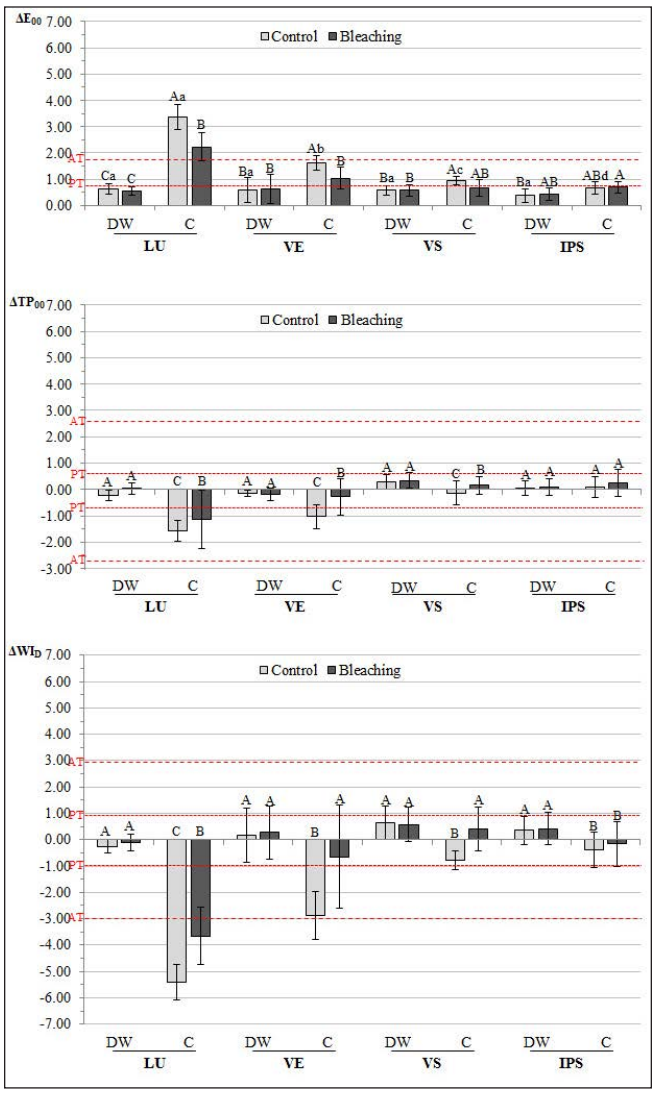

Figure 1. *ΔE00 (R_1-R_0), ΔTP_{00} (R_1-R_0), and ΔWI_D (R_1-R_0) mean values and statistical comparisons. Different uppercase letters indicate significant difference in bars within each material; different lowercase letters indicate significant difference (ΔE_{00}) within the control groups among the materials. Doted red lines: PT, perceptible threshold; AT, acceptable threshold; DW, deionized water; C, coffee.*

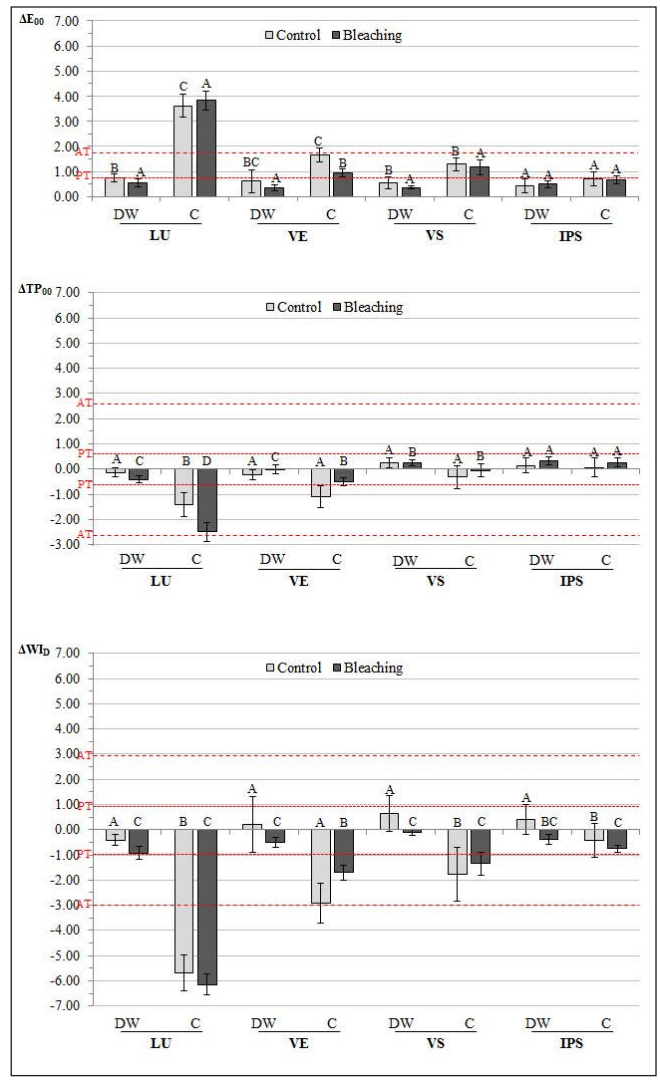

Figure 2. *ΔE_{00} (R_1-R_0), ΔTP_{00} (R_1-R_0), and ΔWI_D (R_1-R_0) mean values and statistical comparisons. Different uppercase letters indicate mean difference in bars within each material. Doted red lines: PT, perceptible threshold; AT, acceptable threshold; DW, deionized water; C, coffee.*

and the other not. The specimens were mounted on metallic stubs and analyzed under a high-resolution field emission scanning electron microscope (model JSM-6610LV), which operated at 25000× magnification with an accelerating voltage of 2.0 kV.

Statistical Analysis

Normality and homoscedasticity were verified using the Shapiro-Wilk and Levene tests. Some groups did not meet these assumptions. We chose to proceed with the three-way ANOVA, which is known to be robust for moderate deviations from normality/homocedasticity when a block design with balanced groups results in a sufficiently large sample size.[35] Thus, the data were submitted to a three-way ANOVA (material, immersion, and bleaching), followed by the Games-Howell post hoc test for multiple comparisons. The level of significance was set at 0.05. All statistical analyses were performed using the IBM SPSS Statistics, v22.0 statistical software (IBM Corporation).

RESULTS

The ΔE_{00} (R_1-R_0), ΔTP_{00} (R_1-R_0), and ΔWI_D (R_1-R_0) results are presented in Figure 1. For the ΔE_{00}, all isolated factors

and interactions were significant (p<0.001). The post hoc test presented p≤0.017. In deionized water, materials showed statistically similar stainability, which was below the 50%:50% perceptibility threshold. In coffee, the order of stainability was LU>VE>VS>IPS. When compared to deionized water, coffee increased the ΔE_{00} unacceptably for LU, and acceptably for VE and VS. Bleaching did not affect the optical properties of the groups immersed in deionized water. In coffee, bleaching decreased the ΔE_{00} in LU and VE, providing partial and total recovery in relation to the groups only immersed in deionized water. Bleaching did not significantly decrease ΔE_{00} in VS, but changed it from acceptable to imperceptible. No effect was found on IPS. For ΔTP_{00}, all isolated factors (p<0.001) and material*immersion (p<0.001) and immersion*bleaching (p<0.01) were significant. The post hoc test presented p≤0.014. In comparison with deionized water, coffee decreased the translucency in LU, VE, and VS. This change was acceptable for LU and VE, and imperceptible for VS. In the groups immersed in coffee, bleaching increased the translucency of LU, VE, and VS with partial recovery. In addition, ΔTP_{00} of VE changed from acceptable to imperceptible. For ΔWI_D, all isolated factors (p<0.001) and interactions (p<0.01) were significant. The post hoc test presented p≤0.032. In comparison with deionized water, coffee decreased the whiteness of all materials, unacceptably for LU, acceptably for VE, and imperceptibly for both ceramics. In the groups immersed in coffee, bleaching increased the whiteness of the LU, VE, and VS with partial (LU) or total recovery (VE and VS). In addition, ΔWI_D of VE changed from acceptable to imperceptible. In IPS, the imperceptible decrease in whiteness provided by coffee was not recovered by bleaching.

The ΔE_{00} ($R_{1'}$-$R_{0'}$), ΔTP_{00} ($R_{1'}$-$R_{0'}$), and ΔWI_D ($R_{1'}$-$R_{0'}$) results are presented in Figure 2. For ΔE_{00}, the isolated factors and interactions were significant (p<0.001), except for immersion*bleaching. The post hoc test presented p≤0.034. Among the groups immersed in deionized water, bleaching imperceptibly decreased ΔE_{00} in LU. In coffee, the only significant change was in VE, which exhibited lower ΔE_{00}, with total recovery. VS and IPS were not affected by bleaching. For ΔTP_{00}, the isolated factors and interactions were

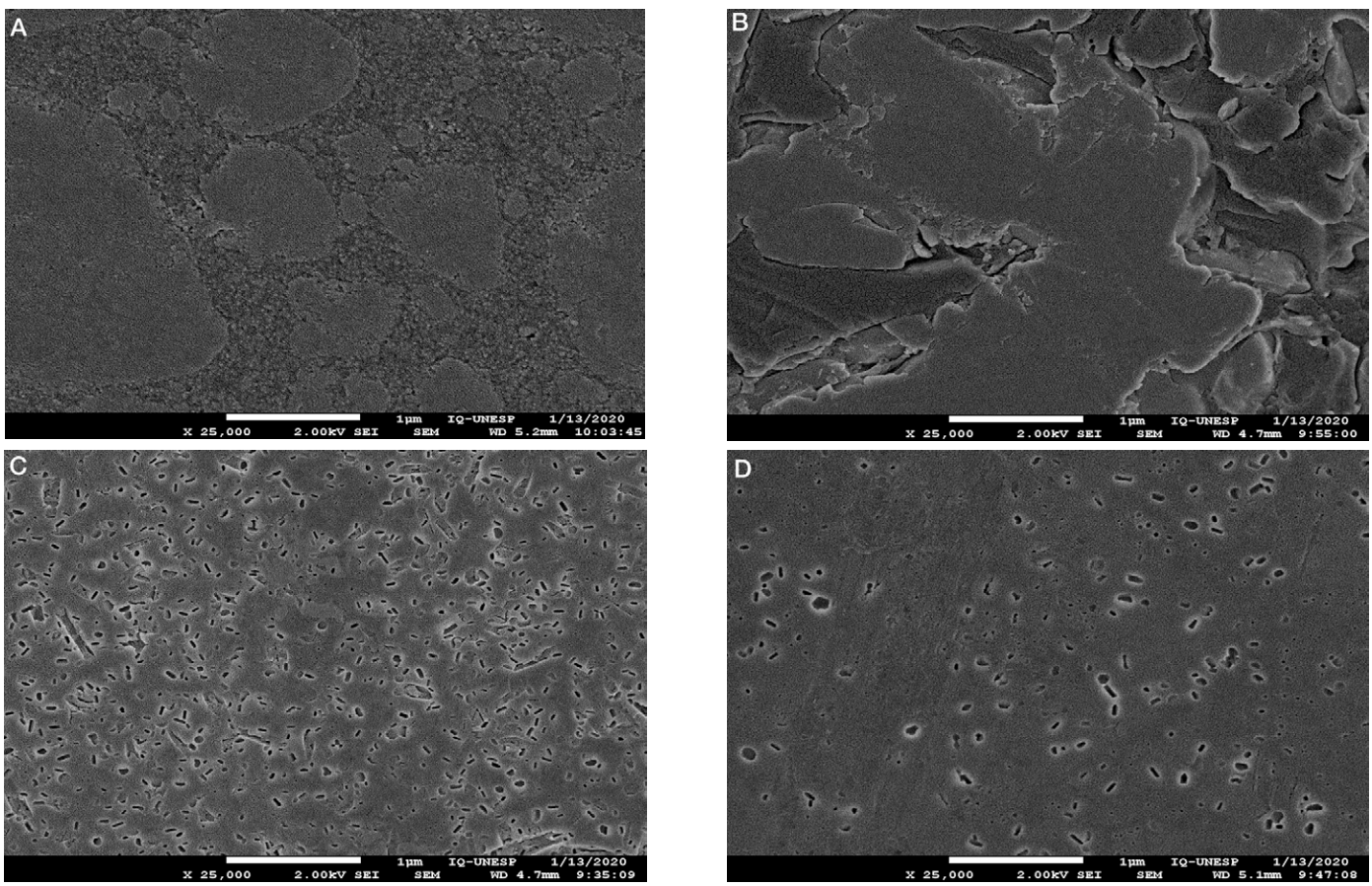

Figure 3. *Scanning electron microscope images (original magnification ×25,000) of the nonbleached materials. A, Lava Ultimate (LU); B, Vita Enamic (VE); C, Vita Suprinity (VS); D, IPS e.max CAD (IPS).*

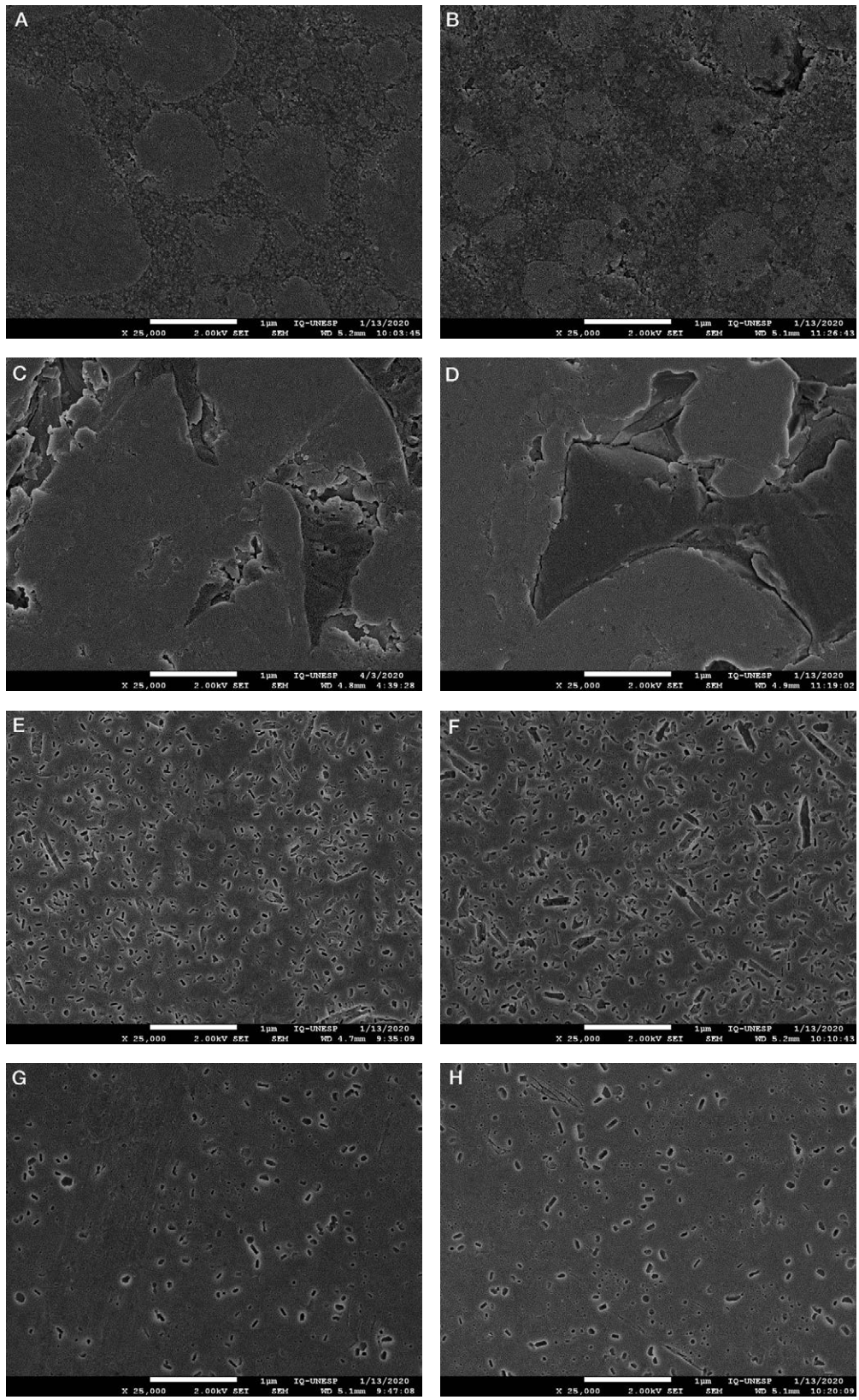

Figure 4. *Scanning electron microscopy images (original magnification ×25,000) of the, respectively, nonbleached and bleached materials. A and B, Lava Ultimate (LU); C and D, Vita Enamic (VE); E and F, Vita Suprinity (VS); G and H, IPS e.max CAD (IPS).*

significant ($p<0.001$), except for the immersion and the interaction immersion*bleaching. The post hoc test presented $p \leq 0.014$. Among the groups immersed in deionized water, bleaching imperceptibly decreased the translucency of LU. In coffee, LU became less translucent, but maintained the acceptability interval, while VE became more translucent, having ΔTP_{00} changed from acceptable to imperceptible, with partial recovery in relation to the groups only immersed in deionized water. VS and IPS were not affected by bleaching. For ΔWI_D, all isolated factors and interactions were significant ($p<0.001$). The post hoc test presented $p \leq 0.021$. Among the groups immersed in deionized water, bleaching imperceptibly decreased the whiteness of LU, VS, and IPS. In coffee, VE became less dark, with partial recovery, while LU, VS and IPS were not affected. SEM images are shown in Figures 3 and 4.

DISCUSSION

The present study was conducted to evaluate how one single session of in-office bleaching affects the optical properties of CAD-CAM monolithic materials that were previously or subsequently stained. The null hypothesis was rejected, since, in both situations, the optical properties were affected to a greater or lesser extent depending on the material.

In terms of color stability, the equivalent of 1-year exposure in coffee adversely and significantly affected the color, translucency, and whiteness of LU, VE, and VS, more pronouncedly in this order, while IPS had only its whiteness decreased. This is probably due to the hydrophilic nature of the ether linkage in the TEGDMA present in LU and VE, and of the hydroxyl groups in the Bis-GMA also present in LU.[8] Since water acts as a carrier for staining agents, materials with highly hydrophilic monomers tend to exhibit more discoloration,[11] as observed in the present study, whose results are in agreement with those of other studies.[9-13] In addition, the SEM images show that the surfaces of LU and VE are highly heterogeneous. The higher polymer content of LU (20%) in comparison with the polymer content infiltrated in the structure of feldspathic ceramic in VE (14%) explains their difference in color stability.[11,13] Regarding the ceramics, VS was affected more by the coffee than IPS, although the differences found in the optical properties of VS were imperceptible or acceptable. This finding is in line with a previous study[10] reporting higher color stability of IPS, when both materials were tested for the thickness of the laminate veneer (0.7 mm). However, considering that the mean crystal size of VS is four to eight times smaller than lithium disilicate crystallites in IPS,[7] and that the roughness of VS was similar[5,6] or lower[4] than that of IPS, it was expected that VS would behave similarly or better than IPS regarding the optical properties, which did not occur. Although no explanation is available in the literature, the SEM images in the study by Sen and Us[7] and in ours revealed that, when compared to IPS, VS exhibited a more porous and heterogeneous surface, which could explain its lower stability. In addition, it is not known if the presence of zirconia particles throughout the entire surface of VS might have some role in the behavior of VS.[3]

Bleaching after staining in coffee benefited the optical properties of LU, VE, and VS by providing partial or total recovery of their original color, translucency, and whiteness, and, in some situations, changing the differences from acceptable to imperceptible, while no effect was observed in IPS. These results found for LU and VE are supported by previous studies[11,15] in which a 40% hydrogen peroxide bleaching agent was also applied on both the materials, while no study with a similar scenario was found with VS and IPS for comparison. It is known that hydrogen peroxide releases OH• and H• free radicals, which are strong oxidizing agents[18] capable of oxidizing organic compounds such as chromophores present in the dental structure. Being small and highly reactive, the H• have a high capability to penetrate the surface of the materials, even the ceramic ones.[18] In resinous materials, hydrogen peroxide induces oxidative cleavage of polymer chains,[17,23] leaching of monomers and oxidation of surface pigments.[11] It is possible that IPS did not suffer significant changes, neither by coffee nor by bleaching, because it has a more homogeneous surface than VS.

In the comparison between the groups subsequently immersed in deionized water, although bleaching promoted significant differences in LU, VS, and IPS, all of them were below the imperceptible threshold. In coffee, the optical properties of VS and IPS were not affected, but the surface of VS seems to have been slightly affected by the bleaching when observing the SEM images. LU became less translucent, but still within the acceptability interval, and showed a tendency, although not statistically significant, to increase the staining susceptibility and to become more dark. Although no studies were found to compare with the results of the current study, the SEM images of LU suggest that bleaching affected its surface, which became more porous, and we wonder if additional bleaching sessions would render this material more susceptible to staining. On the other hand, bleaching before staining in coffee benefited all the optical properties of VE, making them closer to the groups only immersed in deionized water, for which no

explanation was found, not even in the SEM images. Due to the heterogeneous surface topography of the VE, it is difficult to determine if bleaching affected this material. One of the limitations of the present study is that roughness before and after bleaching was not presented, which is a property closely related to the optical properties. Another aspect is that the effect of just one single session of in-office bleaching was evaluated, which does not correspond to the high demand from patients. Our research group is evaluating both aspects in parallel studies. In addition, very little is known about how the mechanical properties of these materials, such as hardness, strength, among others, behave after in-office bleaching. Studies that investigate this issue are also essential in making a decision whether or not in-office bleaching is a safe procedure to be performed on these materials.

CONCLUSIONS

Within the limitations of the present study, one session of in-office bleaching benefited the optical properties of the previously stained LU, VE, and VS, without increasing their susceptibility to staining or adversely providing clinical unacceptable variations in their translucency and whiteness. The surface topography of the LU and VS suffered alterations after bleaching. All variations exhibited by the IPS were below the perceptible threshold.

Conflict of Interest

The authors of the present study 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 the present article.

(Accepted 23 January 2021)

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CAD/CAM Milled Glass Fiber Posts: Adaptation and Mechanical Behavior in Flared Root Canals

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Clinical Relevance

CAD/CAM milled glass fiber posts may be an alternative for the production of custom-made, well-adapted glass fiber posts for flared root canals. Adequate adaptation leads to a homogeneous post-cement-dentin interface that is mechanically stable for reliable rehabilitation.

SUMMARY

This study aimed to evaluate the cementation and mechanical behavior of flared root canals restored with CAD/CAM milled glass fiber post-and-core systems. Sixty-six endodontically treated human canines with a flared root canal were divided into three different groups according to the type of post: G_{PF} received prefabricated posts; G_{REL} received relined glass fiber posts, and G_{MILLED} received CAD/CAM milled glass fiber posts. Cementation was performed with self-adhesive resin cement. The samples were submitted to x-ray microcomputed tomography analysis for the analysis of voids and gaps. The roots were sectioned and submitted to the push-out bond strength test. The load-to-fracture was evaluated in post-and-core systems. G_{MILLED} presented lower void and lower gap volumes when compared to G_{PF} and G_{REL}. On the load-to-fracture test, G_{REL} presented statistically significant higher values than G_{MILLED}. G_{PF} values had no statistically

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http://doi.org/10.2341/20-198-L

significant difference from the two other groups. On the push-out bond strength test, G_{PF} presented statistically significant lower values when compared to G_{REL} and G_{MILLED}. The most common failure pattern was between dentin and cement in all groups. CAD/CAM milled glass fiber post-and-core systems presented an enhanced adaptation of glass fiber posts to flared root canal systems. Their results were comparable to relined posts in bond strength, while load-to-fracture-results for G_{MILLED} were lower than those for G_{PF}.

INTRODUCTION

The rehabilitation of endodontically treated teeth requires an intraradicular post cementation in cases where considerable coronal destruction is found.[1,2] Different materials are used to produce the posts,[2] and glass fiber-based posts have been widely used in clinical practice. The esthetic properties and mechanical behavior of these materials closely resemble those of dental hard tissues, contributing to their high acceptance and success for prosthodontic treatments.[3-6] The failure rate for such treatments ranges from 7% to 11% after up to 10 years.[3,4,7] Most of the failures are related to root fracture and loss of post retention.[2,7,8]

The clinical success of fiber posts depends on dental geometry, the position of the tooth in the arch, adaptation, cementation technique, residual coronal structure, and ferrule presence; an effective bonding is related to successful restorative treatments.[9-11] When glass fiber posts are used in these conditions,lower stress in the post-cement interface and a lower elastic modulus reduces the fracture risk.[12-13] The success of this technique may be impaired by the presence of flared root canals, where extensive loss of tissue is found[14-16] and the adaptation of posts is challenging.

In these cases, prefabricated glass fiber posts cannot match the canals' size and shape, and for this reason, a considerable amount of cement is needed for cementation.[16-17] A thick cement layer may be related to less retention due to impaired polymerization over the root canal,[18,19] reduced mechanical strength in the cement layer,[16,20-23] and increased formation of voids and gaps.[24] Custom-made posts may be produced by relining with composite resins to increase adaptation and reduce the cement layer's thickness.[17,25,26] In these cases, different interfaces are created, as several materials are used.[27,28] The manual adaptation may be time consuming and technique sensitive for clinicians, impairing its applicability and the long-term success of this procedure.

The computer-aided design/computer-aided manufacture (CAD/CAM) process may be applied to the production of individually, anatomically fitted, and monolithic glass fiber posts.[29,30] The possibility of scanning root canals or models allows the design of customized structures that match the shape and the size of the root canal system and may be installed with a thin layer of cement with better adaptation.[31,32] CAD/CAM milled glass fiber blocks are used to produce a monolithic post-and-core that avoids the use of different materials and the presence of multiple interfaces on the cemented structure.[29,33] As CAD/CAM manufacturing becomes more accessible and is increasingly used in dental applications, it is important to understand the ability of these methods to design and build posts for flared root canals. While several advantages have been observed for this manufacturing technique, few studies have attempted to understand monolithic glass fiber post-and-core behavior.[17,29,31] The effect of these posts on roots with extensive loss of tissue diameter should be investigated for the application of these structures in the clinical scenario. This study aims to evaluate the cementation and mechanical behavior of flared root canals restored with CAD/CAM milled glass fiber post-and-core systems.

METHODS AND MATERIALS

Tooth Selection

Sixty-six extracted, single-rooted permanent upper and lower human canines were used. Inclusion criteria were the absence of calcifications, cavities, cracks, and previous endodontic treatment. Teeth were measured and included when the distance between the cementoenamel junction and the apex was at least 15 mm. All teeth were stored in distilled water at 4°C for no more than six months.

Root Canal Preparation

The selected teeth were decoronated 2 mm above the cementoenamel junction using a double-sided diamond disc (KG Sorensen Ltda, Cotia, SP, Brazil) with low speed and under water cooling. All root canals were instrumented with reciprocating files (V-file TDK Blister, Eurodonto, Curitiba, PR, Brazil), under NaOCl 2.5% irrigation. The root canals were then filled with 1 ml of EDTA (Odahcam, Dentsply, São Paulo, SP, Brazil) for 3 minutes. Root canal obturation was performed using a lateral condensation technique with gutta-percha (Dentsply) and a resin-based endodontic sealer (AH Plus, Dentsply, Konstanz, Germany). All specimens were stored for seven days in distilled water at 37°C before further preparation.

Post Preparation

Before the post-cementation procedure, gutta-percha removal was performed with a stainless steel bur (Exacto #3, Angelus Indústria de Produtos Odontológicos S/A, Londrina, PR, Brazil), and 4 mm of filling material remained in the apical third. The root canals were enlarged with a sequence of Largo burs, washed with 5 ml of distilled water, and dried with paper points (Dentsply, São Paulo, SP, Brazil). The enlargement was performed to guarantee that dentin walls presented a 1 mm thickness to simulate flared root canals, to standardize the samples, and to minimize the anatomical differences between teeth. The prepared teeth were divided into three groups according to the different glass fiber posts used for reconstruction: prefabricated glass fiber post (G_{PF}), composite resin relined glass fiber post (G_{REL}) and CAD/CAM milled glass fiber post (G_{MILLED}). The preparation of posts for the different groups is summarized in Table 1. All specimens were water stored for 24 hours at 37°C.

For the G_{PF} group, posts (Exacto #3, Angelus) were cleaned with 70% ethanol. Silane (Angelus) was actively applied for 60 seconds and air-dried for 30 seconds. A self-etching resin cement (U200, 3M Oral Care, São Paulo, SP, Brazil) was mixed and dispensed into the post space with a syringe (Centrix, DFL Indústria e Comércio S.A., Rio de Janeiro, RJ, Brazil) and an endodontic tip. The post was inserted and polymerized for 40 seconds from the buccal and palatine faces with 1500 mW/cm^2 from a calibrated LED light-curing unit (Radii-cal, SDI Indústria e Comércio Ltda., São Paulo, SP, Brazil).

For the G_{REL} group, an adhesive resin (Adper Scotchbond Multi-Purpose, 3M Oral Care) layer was applied around the post and light-cured for 1 minute. For the relining, a composite resin (Filtek Z350, 3M Oral Care) was used directly around the post, inserted into the root canal, and light-cured for 5 seconds. The post was removed and light-cured for 60 seconds. After the relining, the post space was cleaned with distilled water and dried with paper cones. The post was

Table 1: *Description of Different Groups and the Cementation Protocol*

Groups	Cementation Protocol
G_{PF}	• Ethanol cleaning • Air drying • Silane application for 60 s • Air drying 30 s • Cement insertion with a syringe • Post insertion • Polymerization for 40 s
G_{REL}	• Ethanol cleaning • Air drying • Silane application for 60 s • Air drying 30 sec • Adhesive application • Adhesive polymerization for 60 s • Application of composite resin around post for post relining • Post modeling into the flared root canal • Composite resin polymerization for 60 s • Cement insertion with a syringe • Post insertion • Polymerization for 40 s
G_{MILLED}	• Ethanol cleaning • Air drying • Silane application for 60 s • Air drying 30 s • Cement insertion with a syringe • Post insertion • Polymerization for 40 s

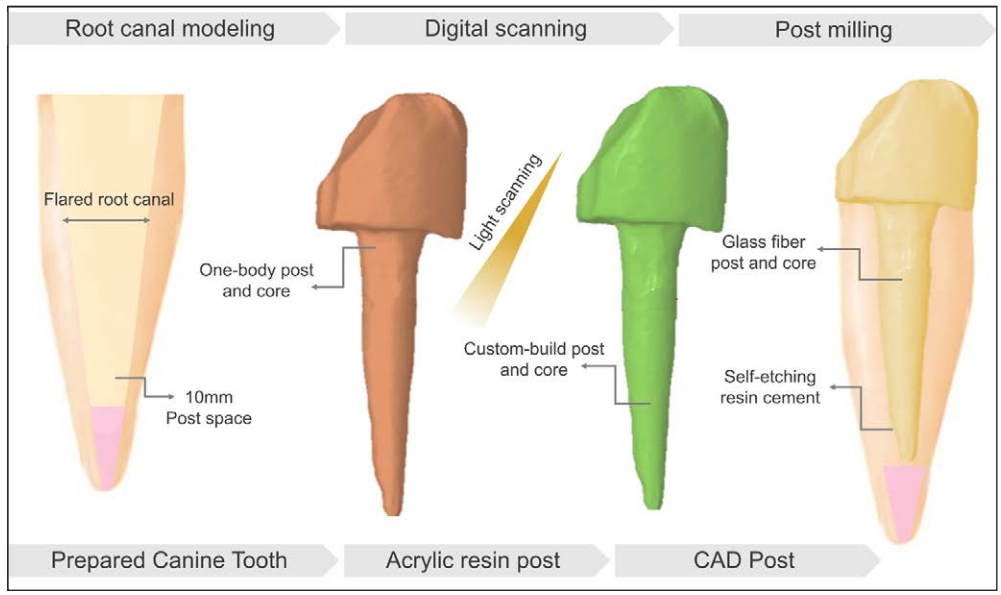

Figure 1. *Representative schematics of CAD/CAM milled glass fiber post production. After tooth preparation, the acrylic resin was used for canal root modeling. The modeled post was scanned for designing a custom-build CAD post that was milled and cemented on the flared root canal.*

washed with 70% ethanol, air dried, and cemented, as described in Table 1.

The G_{MILLED} group was prepared as described in Figure 1. The post space was filled with acrylic resin (Duralay-Reliance Dental, Alsip, IL, USA) for root canal modeling. The acrylic resin posts were scanned with a digital scanner (Shining3D DS-EX, Shining 3D Tech, Hangzhou, China) and computer-aided designed (CAD) posts were obtained with the root dimensions. Glass fiber discs (Fiber Cad - Post & Core, Angelus Indústria de Produtos Odontológicos S/A, Londrina, PR, Brazil) were used for milling on an industrial computer-aided machine (CAM) (DM5 Tecnodrill Indústria de Máquinas Ltda, Novo Hamburgo, RS, Brazil). The CAD/CAM milled posts were prepared and cemented as described in Table 1.

X-ray Micro-computed Tomography

After the cementation, five specimens per group were scanned by x-ray microcomputed tomography (inspeXio SMX-90CT Plus, Shimadzu, Kyoto, Japan). The X-ray tube was operated at 80 kv and 120 mA with a voxel size of 0.013 mm. Reconstruction was performed by inspeXio SMX-90CT software (Shimadzu) and resulted in a stack of 541 images. The Dicom files were used for tridimensional reconstructions with 3D Slicer software (https://slicer.org).[34] The quantification of the volume of voids and gaps was performed in 220 two-D images that were standardized in the samples to analyze the cement layer. The quantification was performed with imaging software (ImageJ, NIH, Bethesda, MD, USA) by the segmentation of the 2D image stack with a grayscale threshold selected between 0-140. The empty spaces in the cement layer were selected with an automatic selection tool, and the area was calculated on each image. The volume of empty spaces in the cement layer was calculated based on the measured area and the thickness of each slice in the 2D stack. All analyses were performed with the same threshold.

Push-out Bond Strength

For the push-out bond strength, cemented root canals (n=12) were sectioned on a low-speed, water-cooled, double-sided diamond disc. For each tooth, nine slices were made with 0.7 mm ± 0.1 mm thickness. Before the analysis, the slices were stored in distilled water at 30°C for 24 hours. The push-out bond strength test was performed on each slice, placed with the apical side downward on a universal testing machine (EZ-SX Shimadzu, Kyoto, Japan). A compressive load was applied to the center of the slice at a crosshead speed of 0.5 mm/min until failure. The bond strength results were based on the maximum force (N) applied and the area of the section. The area was calculated using the formula:

$$\text{Area} = (\pi R + \pi r) \times L,$$

where R, r, and L are cervical radius, apical radius, and adhesive area.

The adhesive area (L) was calculated with the thickness (h) of each slice based on the following formula:

$$L = \sqrt{(R - r)^2 + h^2}.$$

A single operator performed all measurements. Failure pattern was classified as adhesive, between dentin and cement interface (Ac/d) and between cement and post interface (Ac/p) or cohesive in dentin.

Load-to-Fracture

For the load-to-fracture analysis, ten samples per group were submitted to a core reconstruction. For G_{PF} and G_{REL} groups, the coronal portion of the posts was etched with phosphoric acid 37% (Scotchbond etchant, 3M Oral Care) for 30 seconds in enamel and 15 seconds in dentin. A commercial primer (Adper Scotchbond Multi-Purpose, 3M Oral Care) was applied to the dentin. Solvent evaporation was performed, and an adhesive layer was applied. After light-curing, the core was built with a composite resin (BISCORE; Bisco Inc, Schaumburg, IL, USA) with a 5 mm height. Polymerization was performed for 60 seconds on four sites equally distributed in vestibular, bucal, distal, and mesial regions of the core. For G_{MILLED}, the core was modeled with acrylic resin (Reliance Dental Manufacturing LLC; Alsip, IL, USA) in the moment of root canal modelling. The dimensions of the core were the same used for G_{PF} and G_{REL}. The scanning and milling were performed at the same time, and thus a monolithic post-and-core structure was obtained.

The restored root canals were embedded in acrylic resin, and simulation of the periodontal ligament was performed with silicone rubber. Specimens (n=10) were then fixed in a metallic device, and a load was applied to the palatal surface at a 45° angle in a testing machine (Servopulser, Shimadzu Servohydraulic Fatigue Testing System, Kyoto, Japan) with a crosshead speed of 1 mm/min until fracture. Mode of fracture was recorded and classified as repairable in the tooth where debonding, core fractures, or oblique fractures in the cervical third were identified, or non-repairable in the tooth where vertical fractures or oblique fractures in middle and apical third were observed.

Statistical Analysis

A descriptive analysis was performed for microCT images. Data were submitted to equal variance analysis, and the normality of the data was assessed by Shapiro-Wilk. The volume of voids and gaps on microCT, as well as the load-to-fracture values, were compared by one-way ANOVA and Tukey. Repeated measures were used as dependent samples. Each tooth was sliced in nine samples, and thus a dependent analysis was performed for this factor. For the push-out test, two-way repeated-measures ANOVA and Tukey were used. The significance level was set at 0.05.

RESULTS

MicroCT images (Figure 2) were analyzed, and the presence of voids and gaps was evaluated in the cement layer of all specimens tested. Reduced volume of voids and gaps was observed for G_{MILLED} when compared to G_{REL} and G_{PF} (Figure 2; $p<0.05$). The presence of voids and gaps was observed mainly in the cervical third for all groups, while for G_{PF} (Figure 2A) and G_{REL} (Figure 2B), voids were observed in the apical third. The representative slices of microCT analysis are shown in

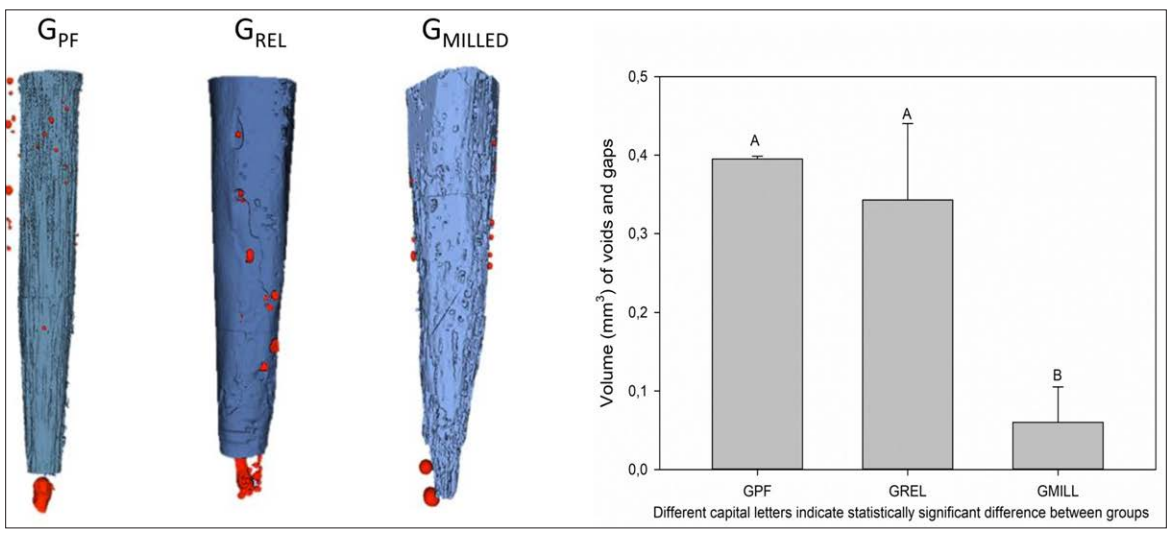

Figure 2. *Representative images of different posts used in each experimental group. The red structures surrounding the posts (blue) shows the presence of voids on the cement layer. Mean and standard deviation for void and gap volume measurements in microCT images. The voids and gaps were calculated in mm³.*

Figure 3. The cement layer thickness and the presence of voids and gaps are shown for the three groups. Fiber alignment is similar for G_{PF} (Figure 3A) and G_{REL} (Figure 3B), while a different pattern is observed for G_{MILLED} (Figure 3C).

For the push-out bond strength analysis, the obtained values ranged from 4.80 ± 2.15 MPa to 2.71 ± 1.81 MPa on G_{REL} and G_{PF}, respectively. No statistically significant difference was found between different thirds in any of the groups (p=0.103). There was a statistically significant difference between groups (p=0.023), and there was no interaction between the two factors (p=0.424). G_{MILLED} showed no statistically significant difference from G_{REL} and G_{PF} (Table 2; p>0.05). The failure modes are shown in Table 2. Adhesive failure was found on at least 58% of the samples. G_{REL} group presented a higher percentage of adhesive failure (69.66%), while G_{MILLED} presented the lowest percentage of adhesive failure (58.9%).

The load-to-fracture results are shown in Table 3. The average load-to-fracture on G_{REL} was 738.13 ± 151.19 N. G_{MILLED} values were statistically significantly lower than G_{REL} (544.83 ± 135.80 N; p<0.05). G_{PF} results were not statistically significantly different than G_{REL} and G_{MILLED} (p>0.05). The failure mode analysis showed increased core fractures (reparable) for G_{MILLED}. Non-reparable fractures were observed for G_{REL} (1) and G_{MILLED} (3).

DISCUSSION

The rehabilitation of teeth with extensive loss of hard tissues is a challenge in clinical practice,[7] and different techniques have been studied for the reinforcement of flared root canals.[26] In the present study, CAD/CAM milled glass fiber posts were used to restore flared root canals *in vitro*. A monolithic system was produced, and when compared to prefabricated and relined glass fiber posts, the tested posts presented a lower amount of voids and gaps, with comparable push-out bond strength values. The load-to-fracture was lower as compared to the other groups.

The internal adaptation of the post influences the cement needed for the procedure.[20,28] The formation of voids and gaps is found due to air entrapment during the cement manipulation, and it is known that a thin layer of cement is related to the formation of fewer voids and gaps.[17] When flared root canals were considered, the amount of cement used on prefabricated posts was obviously increased, and the space between the root canal shape and the fiber post may reduce the amount of cement and the formation of these voids and gaps.[17,33] Although the custom-build posts (G_{REL} and G_{MILLED}) are known to use a small amount of cement, the volume of empty spaces on the cementation region was higher for G_{REL} when compared to G_{MILLED} (Figure 2; p<0.05). This finding may be attributed to the space found on the apical third, because the shape of the post in this

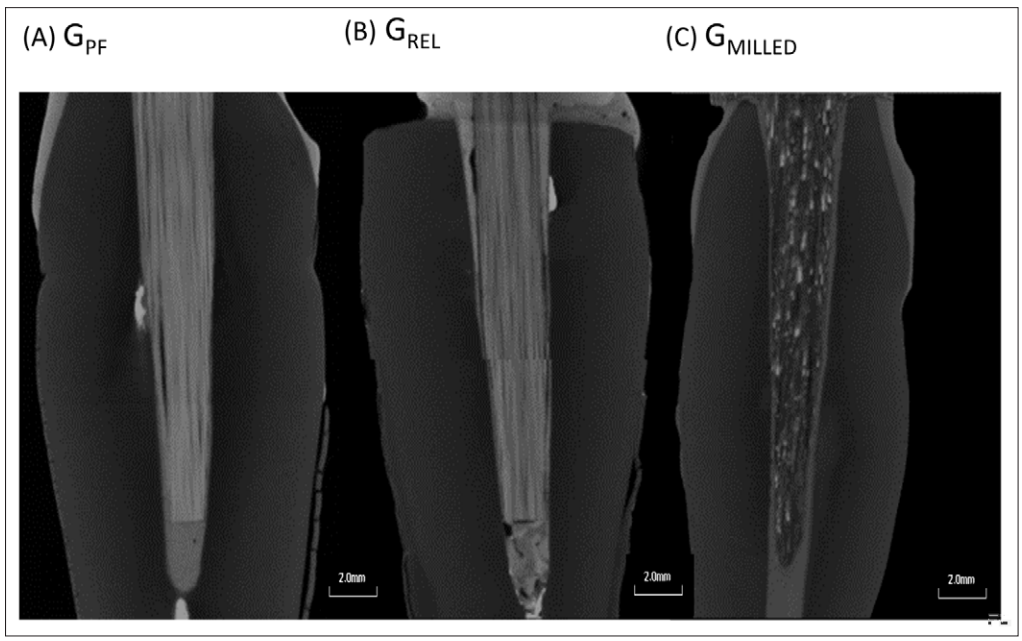

Figure 3. *Representative images of microCT analysis where the thickness of the cement and the fiber orientation is observed for different posts. The prefabricated posts (A) and relined posts (B) present similar fiber alignment, while the cement layer on these specimens is different due to the presence of composite resin used in relining and the higher number of interfaces. (C) CAD/CAM milled glass fiber posts show a different fiber alignment pattern maintaining the parallel alignment found in A and B. A homogeneous and thin cement layer is observed.*

Table 2: *Push-out Bond Strength (MPa) and the Failure Modes After Testing*[a]

Groups	MPa	Adhesive		Cohesive
		Ac/d	Ac/p	
G_{PF}	2.71 ± 1.81A	61.53%	3.37%	28.08%
G_{REL}	4.80 ± 2.15B	69.66%	5.61%	24.71%
G_{MILLED}	4.22 ± 2.58B	58.9%%	3.44%	38.35%

[a]*Different uppercase letters indicate a statistically significant difference (p< 0.05).*
Failure modes: Ac/d: adhesive between dentin and resin cement interface; Ac/p: adhesive between post and resin cement interface; Cohesive = cohesive in dentin.

region is not as closely adjusted to the conical shape of the root in the G_{PF} and G_{REL} as it is for G_{MILLED} (Figure 2). The CAD/CAM milled was able to reproduce the apical portion's conicity, leading to a reduced amount of cement and a reduced volume of voids in this third (Figure 2 and Figure 3). This shows that the accuracy of the scanning and milling process was guaranteed by the chosen method. It is important to highlight that an industrial milling machine was used in the present study, and thus the results may not be directly translated for the production of posts in dental milling machines. Despite this limitation, the benefits in post adaptation may be further explored for other CAD/CAM systems such as the ones designed exclusively for dental applications. The relationship between the presence of voids and gaps and the bond strength has been studied in different posts, and conflicting results have been found. While some reports related lower bond strength to void-containing structures,[22,28] other studies did not find a statistically significant difference.[23,24] Although the effect of voids and gaps is not always present, a thin and uniform cement layer is desired for stability of bonding and the mechanical interlocking of the cemented post.[28]

The cement layer may play an important role in push-out bond strength. Because cement is known to present lower mechanical strength than glass fiber posts, the higher the amount of cement in the root canal, the lower the force needed for dislodgement of the root slices. The lower volume of voids found for G_{MILLED} may explain these results because of the CAD-controlled cementation layer and the amount of cement used for cementation. Polymerization was performed for 60 seconds on 4 sites equally distributed in vestibular, bucal, distal and mesial regions of the core.[7] The push-out bond strength results were not affected by the tooth third in each group, showing that the cementation procedure was effective and that polymerization was achieved along the full extent of the root, excluding the possible effect of a low degree of conversion in the bond strength results.[18,35] Adhesive failures between the cement and the dentin were the most common failure mode found for all the groups, showing that dislodgement is more prone to happen in this interface and that the mechanical properties of luting agents were maintained.

The mechanical behavior of glass fiber posts may be influenced by the production method, and this is directly related to the orientation of the fibers, the adaptation of the post, and the stress dissipation on the post-cement-dentin interface.[5,29,36] The milling process is shown to result in decreased resistance for glass fiber posts alone.[29] In contrast, previous studies found comparable results for load-to-fracture when cemented prefabricated and milled posts were tested.[30,32,33,37] In the present study, static mechanical testing was

Table 3: *Load-to-fracture Results (N) and the Frequency of Reparable and Irreparable Modes of Fracture*[a]

Groups	Load-to-fracture	Reparable		Irreparable
		C	F	F
G_{PF}	610.58N ± 99.21AB	6	4	0
G_{REL}	738.13N ± 151.19A	6	3	1
G_{MILLED}	544.83N ± 135.80B	7	0	3

[a]*Different uppercase letters indicate a statistically significant difference (p>0.05).*
Reparable failures: core (C) or cervical third (F). Irreparable failures in the middle and apical third (F).

performed on cemented posts, and the G_{MILLED} group showed the lowest load-to-fracture values (Table 3), without a statistically significant difference from G_{PF}. The fibers' direction is a key factor for adequate stress dissipation, and milling the glass fiber blocks with vertical fiber orientation is required to guarantee adequate mechanical properties.[29] The alignment of fibers during milling must be obtained by the adequate positioning of the glass fiber block, avoiding different fiber orientation in the post. The representative slices in Figure 3 show that fiber orientation was kept parallel to the root's long axis in all tested groups. The analysis of failure modes in the G_{MILLED} group shows that most of the samples showed chipping failures on the core, and that fracture occurred parallel to the fiber orientation, showing that milling was performed in the right direction (Table 3). Although lower values were found, the core chipping indicates easily reparable fractures, but nonreparable failures were also found. Some studies suggest that stiff materials with high load-to-fracture values may result in increased catastrophic failure.[6] The possibility of repair of the fractured post and core structures is desirable for tooth maintenance, and the impact of the nonreparable failures found in the CAD/CAM milled group must be further evaluated.

The CAD/CAM milling process emerges as a machinable custom-build option to increase the adaptation of glass fiber posts on flared root canal systems. The application of a laboratory CAD/CAM system may contribute to the translation of these results to clinical practice, as access to this technology may be easier for dentists. CAD/CAM is known to present a high startup cost and the need for training, potentially limiting its application in clinical practice, especially for chair-side CAD/CAM.[38,39] Laboratory systems may be more accessible, and, in the results of this initial analysis, were shown to result in well-adapted posts for flared root canals. Using the findings of the present study, it is possible to design further analysis to compare the accuracy of different CAD/CAM systems in the design and fabrication of these posts. The production of a one-body monolithic system avoids the need for in-office adaptation of post and core production and prevents the formation of a multiple-interface system in the post system. The CAD/CAM manufacturing used in this study offered adequate adaptation with reduced cement layer and a lower amount of voids and gaps, evidenced by the high bond strength obtained in the G_{MILLED} group compared to the group with prefabricated posts. This adaptation is essential, especially for flared root canal systems, and may represent an alternative for reliable rehabilitation with lower technique sensitivity.

CONCLUSIONS

CAD/CAM milled glass fiber post-and-cores presented an enhanced adaptation of glass fiber posts to flared root canal systems. Their results were comparable in bond strength but lower in load-to-fracture when compared to relined posts.

Acknowledgments

The authors would like to thank CAPES "Coordenação de Aperfeiçoamento dePessoal de Nível Superior - Brasil - Finance Code 001".

Regulatory Statement

This study was approved by the research ethics committee from Universidade Federal do RioGrande do Sul. The approval code issued for this study is 3.617.474

Conflict of Interest

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

(Accepted 23 January 2021)

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Flexural Properties of Bioactive Restoratives in Cariogenic Environments

AU Yap • HS Choo • HY Choo • NA Yahya

Clinical Relevance

The strength of some bioactive materials can be compromised by cariogenic challenges. This may impact the clinical longevity of restorations, especially in stress-bearing areas.

SUMMARY

This study determined the mechanical performance of bioactive restoratives in cariogenic environments and compared the flexural properties of various bioactive materials. The materials evaluated included a conventional resin-based composite (Filtek Z350 [FZ]) and 3 bioactive restoratives, namely an alkasite (Cention N [CN]), a giomer (Beautifil-bulk Restorative [BB]), and an enhanced resin-modified glass ionomer (Activa Bioactive Restorative [AV]). Beam-shaped specimens (12 x 2 x 2 mm) were produced, randomly allocated to 4 groups (n=10), and conditioned in deionized solution, remineralizing solution, demineralizing solution (DE), or pH cycled for 14 days at 37°C. After conditioning/pH cycling, the specimens were subjected to 3-point flexural testing. Flexural data were subjected to statistical analysis using analysis of variance or Tukey's test (α=0.05). Mean flexural modulus and strength ranged from 3.54 ± 0.33 to 7.44 ± 0.28 GPa, and 87.07 ± 8.99 to 123.54 ± 12.37 MPa, respectively. While the flexural modulus of the bioactive restoratives was not affected by cariogenic/acidic conditions, flexural strength usually decreased, with the exception of CN. The strength of BB was significantly reduced by DE and

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http://doi.org/10.2341/20-045-L

pH cycling, while that of AV was lowered by DE. For all conditioning mediums, AV had a significantly lower modulus than the other materials. Apart from conditioning in DE, where differences in flexural strength was insignificant, FZ and AV were generally significantly stronger than BB and CN. The effect of cariogenic environments on flexural strength was found to be material dependent, and aside from the alkasite material (CN), cariogenic conditions were observed to significantly decrease the strength of bioactive restoratives.

INTRODUCTION

Resin-based composites (RBCs) have become the restorative material of choice in both anterior and posterior teeth, due to their excellent aesthetics, ability to bond to the tooth structure, and conservative cavity preparations.[1] Despite the advances made in photoinitiator, polymer, and filler technology, RBCs are still limited by polymerization shrinkage/contraction stress, incomplete monomer-to-polymer conversion, and limited toughness.[2] Polymerization shrinkage and contraction stress had been associated with several negative clinical outcomes, including cuspal deflection, crack propagation of teeth, decreased bond strength, internal and marginal gap formation, marginal leakage, and secondary caries.[3] Accordingly, systematic reviews of clinical trials have determined secondary caries and composite fracture to be the most common reasons for RBC restoration failure.[4-5] Alvanforoush and others compared the failure rates of direct RBC restorations in posterior teeth over 2 decades and presented some interesting discoveries.[6] Although the failure rates for the years 1995–2005 (10.59%) and 2006–2016 (13.13%) were comparable, the causes of failure differed. In 1995–2005, the reasons for failure were primarily secondary caries (29.47%) and composite fracture (28.84%), with few tooth fractures (3.45%). In 2006–2016, secondary caries (25.68%) decreased slightly, but the incidence of composite fracture (39.07%) and tooth fracture (23.76%) increased considerably. The authors ascribed their findings to the growing use of RBCs for extensive restorations and material variations.[6]

Secondary or recurrent caries (tooth decay at the margins of restorations) accounts for up to 59% of direct restoration replacements.[7] Secondary caries with RBCs can be attributed to patient (caries-risk profile), operator (placement procedures), and material-related factors.[8] More specifically, RBCs appear to favor cariogenic bacterial growth due to their surface characteristics, the components released, and the lack of antibacterial activity.[8] Bioactive restorative materials that replace tooth tissues and possess "therapeutic functions" have been developed to minimize secondary caries.[9] The concept of bioactive restorative materials is not entirely new, and materials were available for several decades in the form of fluoride-releasing materials such as glass ionomer cements (GICs).[10] Commercially available bioactive restorative materials are usually hybrids of RBCs and GICs containing antimicrobials as well as calcium phosphate, silicate, and/or aluminate.[9,10] Their "therapeutic functions" include the potential for suppressing biofilms and acid production, deterring proteins, diminishing secondary caries, and neutralizing acids, as well as replacing lost minerals through ion and other chemical release.[9]

Information pertaining to the mechanical characteristics of contemporary bioactive restorative materials is still limited. Findings have been equivocal, with studies reporting both comparable and lower strengths when compared with conventional RBCs.[11-13] The disparity can be attributed to differences in the materials evaluated, testing methodology, and the conditioning mediums used.[13] Ion release from bioactive restorative materials intensifies under acidic conditions and was associated with significant degradation of calcium-based glasses.[14] Cariogenic environments may well compromise the strength of bioactive restorative materials, leading to material fracture and early restoration failure. Moreover, some bioactive materials are capable of forming apatite-like phases in saliva that might enhance their mechanical properties.[14,15]

The effect of cariogenic challenges on the mechanical properties of bioactive restorative materials has not been explored. This is clinically pertinent, as these materials are advocated in patients with a high caries risk and are often utilized in large cavities.[16] Thus, the objectives of this study were to determine the mechanical performance of bioactive restorative materials in cariogenic environments and compare the flexural properties of various bioactive materials. The null hypotheses were as follows: (1) cariogenic challenges do not degrade the flexural modulus and strength of bioactive restorative materials, (2) environmental calcium phosphate does not enhance flexural properties, and (3) there is no difference in the flexural properties between the various bioactive materials.

METHODS AND MATERIALS

Specimen Preparation and Conditioning

The materials evaluated included a conventional RBC (Filtek Z350 [FZ]; 3M Oral Care, St. Paul, MN, USA) and 3 bioactive restorative materials, namely an alkasite (Cention N [CN]; Ivoclar Vivadent Inc, Amherst, NY,

USA), a giomer (Beautifil-bulk Restorative [BB]; Shofu, Kyoto, Japan), and an enhanced resin-modified glass ionomer (RMGIC; Activa Bioactive Restorative [AV]; Pulpdent, Watertown, MA, USA). The manufacturers and technical profiles of the materials are listed in Table 1. While the alkasite and giomer materials are essentially resin-based composites with alkaline and prereacted glass ionomer fillers, respectively, the "enhanced" RMGIC was augmented with other bioactive glasses. The minimum sample size was determined using the G*power software (version 3.1.9.4)[17] based on the analysis of variance test with an effect size of 0.5,13 alpha error of 0.05, and power of 80% for 16 material-medium combinations. Forty bar-shaped specimens (12 x 2 x 2 mm) of the various materials were fabricated for the mini-flexural test (MFT) using custom-made stainless-steel molds.[13] The materials were mixed according to the manufacturers' instructions (where applicable) and/or placed into the molds in a single increment. The material-filled molds were compacted between 2 polyester strips with glass slides to remove excess material. All materials were light polymerized with 2 overlapping 10-second irradiations from the top and bottom surfaces using a light-emitting diode curing light (Demi Plus; Kerr, Brea, CA, USA) and were left undisturbed in their molds for 5 minutes. The curing light had an output irradiance of 1330 mW/cm^2, a wavelength of 450–470 nm, and an exit window of 8 mm. The curing light was re-charged after every 10 specimens and a radiometer (Bluephase Meter II; Ivoclar Vivadent, Schaan, Liechtenstein) was used to verify the consistency of its performance. The polyester strips or glass slides were subsequently removed, and the materials were further polymerized with another 2 overlapping 10-second irradiations from the top surfaces. Specimens were then separated from their

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

Material	Manufacturer	Type (Curing methods)	Resin	Fillers	Filler content % by weight/ volume
Filtek Z350XT	3M Oral Care, St Paul, MN, USA	Nano-filled Composite (light cured)	Bis-GMA, Bis-EMA, UDMA, TEGDMA, PEGDMA	Prepolymer fillers containing barium glass, ytterbium fluoride, and spherical mixed oxides. Nonaggregated silica and zirconia Aggregated silica/ zirconia clusters	78.5/59.5
Cention N	Ivoclar, Vivadent Inc, NY, USA	Alkasite (self-cured with optional light curing)	UDMA, DCP, aromatic aliphatic-UDMA, PEG-400 DMA	Br-Al-Si glass, ytterbium and trifluoride Isofiller Calcium barium aluminium fluorosilicate and calcium fluorosilicate glass fillers	75/61
Beautifil-bulk Restorative	Shofu, Kyoto, Japan	Giomer (light cured)	Bis-GMA, UDMA, Bis-MPEPP, TEGDMA	S-PRG (based on fluoroboroaluminosilicate glass) and nano fillers	83.3/69
Activa Bioactive Restorative	Pulpdent, Watertown, MA, USA	Enhanced resin-modified GIC (dual-cured/light-cured)	Blend of UDMA and other methacrylates with modified polyacrylic acid	Bioactive glass, sodium fluoride	55.4 wt

Abbreviations: Aromatic aliphatic-UDMA, tetramethyl-xylylendiurethane dimethacrylate; Bis-EMA, ethoxylated bisphenol-A-glycidyl methacrylate; Bis-GMA, bisphenol-A glycidyl methacrylate; Bis-MPEPP, 2,2-Bis (4-methacryloxypolyethoxyphenyl) propane; DCP, tricyclodecan-dimethanol dimethacrylate; PEG-400 DMA, polyethylene glycol 400 dimethacrylate; PEGDMA, polyethylene glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; wt, weight.

molds, and any minor material flash was eliminated with fine polishing discs (Sof-Lex; 3M Oral Care). The finished specimens were inspected for defects and a digital vernier caliper (Mitutoyo Corporation, Kawaski, Japan) was used to establish the parallelism, as well as the final dimensions, of the specimens.

The measured specimens were randomly allocated into 4 groups (n=10) and conditioned in deionized solution (DI), remineralizing solution (RE), demineralizing solution (DE), or pH cycled (PC) for 14 days at 37°C in an incubator (IN-450, Memmert, Schwabach, Germany). The composition of the RE and DE solutions and their pH are displayed in Table 2.18 Each pH cycle involved exposure to DE (pH 4.8) for 8 hours and RE (pH 7.0) for 16 hours per day. The conditioning solutions (10 ml) were changed daily, and pH was checked with a digital pH meter (pH2700, Eutech, Singapore). Air exposure and evaporation of the solutions in the incubator was minimized with the use of sealed containers. Following conditioning/pH cycling, the specimens were rinsed with DI, gently air-dried, and subjected to 3-point flexural testing.

Flexural Testing

The conditioned/PC specimens were loaded in a universal testing machine (Shimadzu Corporation, Kyoto, Japan) with a load cell of 5 KN and crosshead speed of 0.5 mm/minute until fracture occurred based on the MFT. The distance between the supports for the 3-point bending set-up was fixed at 10 mm. Flexural strength, σ, in megapascals (MPa) was calculated using the following formula:

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

where P is the maximum load exerted on the specimen in newtons; L is the support span in 10 millimeters; B is the width of the specimen in mm; and H is the height of the specimen in mm.

Flexural modulus, E', in 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 had been defined in the flexural strength equation. Flexural modulus was converted to gigapascals (GPa).

Table 2: List of Conditioning Mediums, Their Composition and pH

Group	Conditioning Medium(s)	Constituents	pH
DI (Control)	Deionized water	H_2O	7.0
RE	Remineralizing solution	0.9 mM NaH_2PO_4, 1.5 mM $CaCl_2$, 0.15 mM KCl	7.0
DE	Demineralizing solution	2.2 mM NaH_2PO_4, 2.2 mM $CaCl_2$, 50 mM acetic acid	4.8
PC (pH cycled)	Demineralizing solution for 8 hours	2.2 mM NaH_2PO_4, 2.2 mM $CaCl_2$, 50 mM acetic acid	4.8
	Remineralizing solution for 16 hours	0.9 mM NaH_2PO_4, 1.5 mM $CaCl_2$, 0.15 mM KCl	7.0

Abbreviations: $CaCl_2$, calcium chloride; DI, deionized solution; DE, demineralizing solution; H_2O, water; KCl, potassium chloride; NaH_2PO_4, monosodium phosphate; PC, pH cycled; RE, remineralizing solution.

Statistical Analysis

Statistical analyses were carried out using SPSS software (Version 23; SPSS Inc, Chicago, IL, USA) and parametric statistical methods were employed, as data was found to be normally distributed (Shapiro-Wilk's test). Interactions between the independent variables (materials and mediums) for the 2 dependent variables (flexural modulus and strength) were assessed using two-way analysis of variance (ANOVA). One-way ANOVA and Tukey's post hoc test was performed at significance level α=0.05 for intergroup comparisons.

RESULTS

Mean flexural modulus (GPa) and strength (MPa) of the various materials after conditioning/pH cycling are shown in Table 3. As two-way ANOVA showed significant interactions between the mediums and materials, additional statistical analysis was performed based on individual materials and mediums. The results of intermedium and intermaterial comparisons are reflected in Tables 4 and 5, respectively.

The mean flexural modulus ranged from 3.5 ± 0.3 GPa for AV conditioned in DI to 7.4 ± 0.2 GPa for BB exposed to RE. While the difference in flexural modulus was statistically insignificant for CN and BB, significant differences were observed between conditioning mediums FZ and AV (Table 4). For FZ, the modulus after pH cycling was significantly higher

Table 3: *Mean Flexural Modulus (GPa) and Strength (MPa) of the Various Materials*

Materials	Conditioning Mediums	Flexural Modulus Mean (SD)	Flexural Strength Mean (SD)
Filtek Z350	DI	6.5 (0.6)	123.5 (12.3)
	RE	6.6 (0.3)	111.2 (9.5)
	DE	6.2 (0.5)	96.3 (8.7)
	PC	7.3 (0.5)	115.8 (8.8)
Cention N	DI	7.2 (0.5)	98.3 (9.0)
	RE	7.1 (0.7)	98.6 (6.8)
	DE	6.5 (0.6)	98.1 (7.6)
	PC	6.5 (0.6)	97.7 (9.7)
Beautifil-bulk restorative	DI	7.3 (0.5)	110.5 (6.5)
	RE	7.4 (0.2)	99.5 (6.8)
	DE	7.4 (0.2)	93.0 (7.8)
	PC	7.0 (0.5)	87.0 (8.9)
Activa bioactive restorative	DI	3.5 (0.3)	113.6 (4.3)
	RE	3.9 (0.2)	114.3 (7.4)
	DE	3.6 (0.3)	97.5 (9.6)
	PC	3.8 (0.3)	112.0 (7.3)

Abbreviations: DI, deionized solution; DE, demineralizing solution; PC, pH cycled; RE, remineralizing solution.

than conditioning in DI and DE. For AV, storage in RE resulted in significantly higher modulus values than in DI. No significant difference in flexural modulus was observed between DI and DE/PC (acidic environments). For all conditioning mediums, AV had a significantly lower modulus than the other materials (Table 5). When conditioned in DI, RE, and DE, BB had a significantly higher modulus than FZ. In addition, BB was also significantly stiffer than CN after exposure to DE. When PC, FZ had a significantly higher modulus than CN.

The mean flexural strength varied from 87.0 ± 8.9 MPa for BB when PC to 123.5 ± 12.3 MPa for FZ conditioned in DI. The flexural strength of CN was not affected by conditioning mediums and pH cycling (Table 4). For FZ and AV, storage in DE presented significantly lower strength values than all other mediums, including PC. For BB, exposure to RE, DE, and PC resulted in a lower strength than DI. Moreover, conditioning in RE gave higher strength values when compared with PC. No significant difference in flexural strength was observed between materials after conditioning in DE (Table 5). When stored in DI, CN was significantly weaker than all other materials and FZ was stronger than BB. After conditioning in RE and pH cycling, FZ and AV presented higher strength values than BB and CN.

Table 4: *Comparison of Flexural Properties Between Conditioning Mediums for Each Material*

Materials	Flexural Modulus	Flexural Strength[a]
Filtek Z350	PC > DI, DE	DI, PC, RE > DE
Cention N	NS	NS
Beautifil-bulk Restorative	NS	DI > RE, DE, PC RE > PC
Activa Bioactive Restorative	RE > DI	RE, DI, PC > DE

Abbreviations: DE, demineralizing solution; DI, deionized solution; PC, pH cycled; NS, no statistical significance; RE, remineralizing solution.

NS denotes no statistical significance while > indicates statistical significance. Results of one-way analysis of variance and post hoc Tukey test (p<0.05).

DISCUSSION

Key Findings and Methods

This study examined the effect of cariogenic environments on the flexural properties of bioactive restorative materials and compared the flexural modulus and strength between different bioactive materials. As cariogenic challenges and environmental

Table 5. *Comparison of Flexural Properties Between Materials for Each Conditioning Medium*

Conditioning Medium	Flexural Modulus	Flexural Strength
DI	BB, CN > FZ > AV	FZ, AV, BB > CN FZ > BB
RE	BB, CN, FZ > AV BB > FZ	AV, FZ > BB, CN
DE	BB > CN, FZ > AV	NS
PC	FZ, BB, CN > AV FZ > CN	FZ, AV > CN, BB

Abbreviations: AV = Activa Bioactive Restorative; BB = Beautifil-bulk Restorative; CN = Cention N; FZ = Filtek Z350.

NS denotes no statistical significance while > indicates statistical significance. Results of one-way ANOVA and post-hoc Tukey test (p<0.05).

calcium phosphate influenced the flexural properties of some materials—and significant differences in both modulus and strength were observed between materials—all 3 null hypotheses were duly rejected. A conventional composite (FZ) and DI that is void of all ions served as the controls for the present study. The RE (pH 7.0) mimicked saliva that is saturated with calcium phosphate, while an acidic cariogenic environment (pH 4.8) is represented by the DE. The alternating demineralization and remineralization of teeth was developed by ten Cate and Duijsters to produce artificial caries.[19] This pH cycling model attempts to replicate the periodic alternation of pH that occurs intraorally when sugars are metabolized by cariogenic bacteria, imitating the mineral lost and gained during caries formation.[20] The pH-cycling approach is especially applicable given the hypothetical interactions between bioactive restorative materials with tooth tissues and their surrounding environments.[9,10]

The technical and clinical significance of flexural testing has been detailed in earlier studies.[13,21] Briefly, flexural modulus defines material stiffness/rigidity, while flexural strength characterizes the maximum bending stress that can be applied before the material fractures. More recently, laboratory fracture strength was found to be correlated to material wear in clinical trials.[22] It was opined that for Class I, II, III, and IV cavities, materials with high modulus and strength are necessary to minimize deformation and fracture under occlusal forces during function and parafunction. In Class V cavities, materials with a low modulus that are capable of flexing with teeth are favored, as they are believed to reduce stress and debonding at the adhesive interface of restorations.[23,24] The MFT was selected over the traditional International Standards Organization (ISO) flexural test due to its strong correlation with the latter, greater clinical relevance, and laboratory efficiency (less specimen fabrication flaws and materials are required).[21]

Flexural Modulus

The flexural modulus of CN and BB was not significantly influenced by conditioning mediums/pH cycling, while that of FZ and AV was. For FZ, modulus after storage in DI and DE was observed to be lower than that for RE and PC. The reduced resistance to elastic deformation of RBCs after exposure to DI and other aqueous solutions is well reported in the literature and has been attributed to water sorption.[23,25] In addition to the plasticizing effect of water on the resin matrix, complete or partial debonding of the fillers also occurs arising from "stress corrosion"; this arises from the diffusion of water and the swelling of the resin matrix that generates radial tensile stresses at the resin-filler interfaces.[26] Nano-filled composites utilizing nanoparticles, and nanocluster fillers like FZ, appear to be more susceptible to degradation by acidic solutions.[27] The modulus of AV, as with the other bioactive restorative materials CN and BB, was not significantly decreased by acidic environments (ie, DE and PC). When conditioned in RE, a significantly higher stiffness was noted when compared with DI. AV is an RMGIC that is enhanced with a resin matrix that releases and recharges calcium, phosphate, and fluoride.[14,28] As with other GICs, they may be capable of surface apatite formation when environmental calcium phosphate is high.[15] This has been shown to increase the physico-mechanical properties of GICs and could likewise increase the flexural modulus of AV in the present study.[15]

Despite the aforementioned potential impact on flexural modulus,, AV had a significantly lower modulus than FZ, BB, and CN regardless of conditioning mediums. This may be attributed to its lower filler content when compared with the other materials (55.4 vs 75.0%–83.3% weight).[29]

The flexural modulus of BB was generally significantly greater than that of the conventional composite FZ. BB is a bulk-fill giomer restorative that employs the use of prereacted glass ionomer (PRG) technology. Fluorosilicate glass is reacted with polyacids, freeze-dried, milled, silanized, and ground to attain the PRG fillers, which are incorporated into nano-filled resin copolymers. Given its greater filler content, the higher modulus of BB when compared with FZ was anticipated. Findings of this study corroborated those of Eweis and others, who ascertained that BB had a similar or greater modulus than conventional and bulk-fill restorative/flowable RBCs.[13] CN is a powder-liquid–based alkasite material. The powder is comprised of a wide array of calcium/barium glasses, ytterbium trifluoride, and isofillers, while the liquid is a mixture of 4 dimethacrylate resins. The lower modulus of CN, when compared with BB and FZ after exposure to DE/pH cycling, could be partly contributed by the degradation of the array of basic glass fillers employed in CN when exposed to acids.[14]

Flexural Strength

Flexural strength, like flexural modulus, is determined by filler characteristics (composition, content, size, and geometry), as well as the constitution of the resin matrix.[24,29,30] Collectively, these factors can also influence the degree of conversion and water sorption that may also have bearing on flexural properties.[31] With the exception of CN, conditioning of all materials in DE significantly lowered flexural strength. Furthermore,

BB was also significantly weakened after exposure to RE and PC. The decrease in flexural strength observed with FZ, BB, and AV after storage in DE may be attributed to the susceptibility of their fillers to acid degradation.[14,25,27] Moreover, the polysalt matrix of AV may also be prone to acid attack.[32] The strength of FZ and BB was also found to be reduced after storage in citric acid.[13,25] BB was also weakened after RE/PC, and this may be contributed by its lower degree of conversion and conceivable increased environmental interactions.[33] The alkasite (CN) was the only material that was not affected by both cariogenic challenges or environmental calcium phosphate. This was in spite of its high ion release and may be contributed in part by the formation of an apatite-like phase in neutral artificial saliva.[14] Fluoride ion release and alkalizing potent in acidic conditions was found to be even greater if CN was not light-cured.[34]

When conditioned in DE, no significant difference in strength was observed between the materials. This could be ascribed to the deterioration of all the materials evaluated, including the conventional composite FZ when exposed to acidic environments.[14,27] When conditioned in DI, CN was significantly weaker than FZ, BB, and even AV despite its high filler content. The high release of ions associated with calcium fluorosilicate glass degradation in neutral pH might be an explanatory factor.[14] PRG filler degradation and ion release may also contribute to the significantly lower strength of BB when compared with FZ in DI. This could be further heightened by BB's relatively low degree of conversion.[33] In addition to fluoride, other elements released by PRG-fillers include aluminum, boron, sodium, silicon, and strontium.[35] Despite its relatively low filler content, no significant difference in strength was observed between FZ and AV when stored in DI. The low modulus of AZ could theoretically offer greater resilience and enhance its ability to withstand higher stress prior to fracture.[13]

After storage in RE and pH cycling, BB and CN were significantly weaker than FZ and AV. Filler content by itself cannot explain this observation, as both BB and CN had comparable or higher filler loading than FZ and AV. Both RE and PC involved exposure to calcium phosphate-rich solutions. Unlike GICs, the calcium glass fillers used in the giomer and alkasite are partially (ie, surface) reacted or unreacted. As they elute ions that play a role in mineral induction,[36] it is conceivable that these glass fillers are more reactive and interact considerably with environmental phosphate, leading to lower strength values. The formation of surface apatite phases on CN after conditioning in artificial saliva provides some evidence for this phenomenon.[14]

Limitations of the Study

Despite its positive findings, the present study had some limitations. First, the materials were exposed to the various mediums or pH cycling continuously for 14 days. While this conditioning period may appear extensive and might exaggerate the effects of DE/PC, it may occur in vivo at the margins and contact areas of restorations where dental plaque accumulates. As bioactive materials are capable of releasing ions over long periods of time,[37] the duration of exposure could also be extended to establish the long-term effect of the mediums or pH cycling on flexural properties. Second, the titratable acidity of the conditioning solutions, including DI, which is known to absorb atmospheric carbon dioxide to produce carbonic acid, should be examined in addition to pH. Titratable acidity was reported to play a more crucial role in surface degradation of RBCs than pH.[38] Third, bioactive materials are not a homogenous group of materials and, as such, more products from different manufacturers must be evaluated before definitive conclusions on the effect of cariogenic challenges on bioactive restorative materials can be made. In the meantime, caution must be exercised when indicating these materials in large cavities of patients with a high caries risk. Additionally, the assessment of flexural properties should be coupled with ionic and polymeric release of the various materials to better explain any observed changes. Finally, the present study utilized a static MFT that does not yield identical results to the ISO flexural test and provide an understanding of material structures. As most contemporary bioactive restorative materials are hybrids of RBCs and GICs, they are anticipated to undergo both viscous and elastic deformation when loaded. Dynamic mechanical analysis (DMA) should be conducted to examine the visco-elastic behaviors of these bioactive materials.[39] Moreover, DMA can be performed with different temperature loading frequencies and displacements to simulate variations in intraoral conditions.

CONCLUSIONS

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

1. The effect of conditioning/pH cycling on flexural properties of bioactive restorative materials was material and medium dependent.
2. The flexural modulus of the bioactive materials was not affected by cariogenic conditions. Environment calcium phosphate appeared to increase the rigidity of the enhanced RMGIC (AV).
3. With the exception of the alkasite material (CN), flexural strength of the giomer (BB), enhanced

RMGIC (AV), as well as the conventional RBC (FZ) was negatively impacted by cariogenic conditions.

4. AV had a significantly lower modulus than all the materials evaluated, regardless of conditioning environments.
5. Apart from conditioning in DE where differences in flexural strength was insignificant, the conventional composite (FZ) and enhanced RMGIC (AV) were significantly stronger than the giomer (BB) and alkasite (CN) restoratives.

Acknowledgements

This research work was funded by research grant GPF011E-2018, Faculty of Dentistry, University of Malaya.

Conflict of Interest

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

(Accepted 25 May 2020)

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Fluoride Release from Glass Ionomer Cement and Resin-modified Glass Ionomer Cement Materials under Conditions Mimicking the Caries Process

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Clinical Relevance

Glass ionomer cement and resin-modified glass ionomer cement release different amounts of fluoride when subjected to conditions simulating the process of caries development; consequently, these materials have different potentials to control caries.

SUMMARY

The anticaries potential of restorative ionomeric materials should be evaluated under a pH-cycling regime that simulates the caries process of demineralization and remineralization. Ten glass ionomer cement (GIC) materials and five resin-modified glass ionomer cement (RMGIC) materials were evaluated. A resin composite was used as a negative control. Six discs of each material were immersed for 6 and 18 hours each day in demineralizing (De-) and remineralizing (Re-) solutions, respectively. The solutions were changed daily over 12 days, during which the fluoride concentration was determined using an ion-specific electrode. The results were expressed as (1) the daily fluoride concentration in the De- and Re- solutions (μg F/ml), (2) the amount of fluoride released daily in the De- + Re- solution per area of specimens (μg F/cm²/day), and (3) the cumulative release over the 12-day period (μg F/cm²). During the first days, all materials showed a surge in fluoride release, followed by a gradual decline; however, three distinct patterns were observed, specifically: (1) greater fluoride release in the De- solution compared to the Re- solution during the study period; (2) an initial higher release in De- solution; and (3) a similar release in both solutions over the whole period. The materials differed statistically ($p<0.05$) with respect to daily and cumulative fluoride release. One GIC (Maxxion R) and one RMGIC (Resiglass

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http://doi.org/10.2341/19-296-L

R) had the highest and lowest ability to release fluoride, respectively. In conclusion, the GICs and RMGICs evaluated exhibited distinct qualitative and quantitative patterns of fluoride release under conditions simulating the caries process, which might reflect their anticaries potential.

INTRODUCTION

Despite efforts to control dental caries, 2.5 billion people have untreated caries lesions.[1] Glass ionomer cement (GIC) and resin-modified glass ionomer cement (RMGIC) are biocompatible restorative dental materials that also release fluoride.[2] Fluoride release is considered an important property of GIC and RMGIC[3] because it could prevent caries lesions in enamel and dentin adjacent to restoration areas, something shown by in situ studies.[4-6] The anticaries effect of these materials is facilitated because they release fluoride constantly in the mouth over long time frames and the mechanism of action involved has been discussed.[7]

Fluoride release from GIC and RMGIC probably mimics the known effect of fluoride on the caries process that occurs in the oral cavity.[7] Thus, the *in vitro* evaluation of this process could provide insights into the mechanism of how these materials contribute towards controlling caries.[7-10] At present, several GIC and RMGIC materials are available on the market; however, their anticaries potential, in terms of their ability to release fluoride, has only been evaluated in conditions that do not mimic the caries process.[11-18] In fact, just one evaluation has been made under conditions mimicking the caries process.[19] *In vitro* models could be used to simulate what happens in the oral cavity; however, it is important to use appropriate media to evaluate the release of fluoride from dental materials under laboratory conditions. Furthermore, the amount of fluoride released by different GIC and RMGIC materials changes with the media being used, which might lead to misleading interpretations of their anticaries potential.[7]

Here, we hypothesized that different types of ionomeric materials (ie, GICs and RMGICs) would present different (quantitative and/or qualitative) patterns of fluoride release when tested under conditions modeling the caries process.

METHODS AND MATERIALS

Experimental Design

Fifteen restorative glass-ionomer cement materials (10 GICs and five RMGICs), found in the market place, were used (Table 1). The fluoride release of these materials was evaluated in demineralizing (De-) and remineralizing (Re-) solutions (pH-cycling regime).[9,10] A resin composite was used as the negative control. The model used here simulated a high cariogenic challenge,[20] and it has been used to evaluate *in vitro* how GIC affects enamel demineralization adjacent to a restoration.[21]

Disk-shaped specimens (n=6) were immersed daily in De- and Re- solutions for 6 and 18 hours, respectively. These solutions were changed daily over a 12-day period. Fluoride concentrations in the De- and Re- solutions were analyzed with a fluoride ion-specific electrode (FI-SE). Fluoride concentrations in the De- and Re- solutions for each material were compared descriptively. The amount (μg) of fluoride released daily in the De- and Re- solutions was summed. The materials were statistically compared using μg F/cm^2 per day based on (1) the 7th day, when release had stabilized, and (2) the cumulative release over the 12-day period. The materials were coded, which allowed for blind analysis with FI-SE.

Preparation of Specimens

Six cylindrical specimens (7.8 mm in diameter and 2.2 mm in thickness) were prepared at room temperature (23°C±2°C) and (50%±20%) relative humidity, according to ISO 9917-1[22] and 9917-2[23] recommendations and the manufacturer's instructions. Materials were mixed and placed in Teflon molds using a Centrix system (Nova DFL, Rio de Janeiro, Brazil). Conventional glass ionomers were allowed to set under pressure between a polyester strip and glass plate for 15 minutes. Resin-modified glass ionomers and resin composite were placed on a glass plate between polyester strips and were light-activated on both the upper and lower surfaces. One multipeak light curing unit (Valo Cordless, Ultradent Products Inc., South Jordan, UT, USA), with a 9.4 mm active diameter and 1477 mW/cm^2 irradiance, was used for all light curing procedures. The light curing unit was positioned 1 mm above the surface strip, and it was activated for 20 or 40 seconds, according to the manufacturer's specifications. No protection was applied to the surface of the specimens. After hardening, specimens were stored at 37°C and 100% relative humidity for 24 hours,[9,10] and excess material was removed with a scalpel blade, coarse/medium Opti-Disc™ contouring discs (KaVo Dental, Brazil), and a low-speed handpiece.

The pH Cycling Regime

The specimens were immersed separately in polyethylene tubes containing 1.0 mL De- solution (50 mM acetate buffer [pH 5.0] containing Ca 1.12 mM and

Table 1: *Restorative Materials Used and Specifications*

Material (Type/Name)	**Composition (Powder; Liquid)**	**Batch**	**Manufacturer**
1. Conventional glass ionomer cements			
Bioglass R	Calcium, barium, and aluminum fluorosilicate, polyacrylic acid, inorganic filler; polyacrylic and tartaric acid; deionized water	012/1	Biodinâmica (Brazil)
EQUIA Forte Fil	Aluminum fluorosilicate glass, carboxylic acid, ferrous oxide; polybasic carboxylic acids	1708112	GC Corporation (Japan)
Fuji 9 (Gold Label High Strength Posterior Restorative)	Aluminum fluorosilicate glass (95%), polyacrylic acid powder (5%); Polyacrylic acid (40%), polybasic carboxylic acids(10%), distilled water (50%)	1612091	GC Corporation (Japan)
Ionglass R	Calcium, sodium and aluminum fluorosilicate, polyacrylic acid; tartaric acid, water	579317	MAQUIRA (Brazil)
Ionofil Plus	Aluminum fluorosilicate glass, polyacrylic acid powder; polyacrylic and tartaric acid, water	1702476	VOCO GmbH (Germany)
Ketac Molar Easymix	Aluminum-lanthanum-calcium fluorosilicate glass, tartaric acid, sorbic acid, benzoic acid, pigments; acrylic acid copolymer, tartaric acid, maleic acid, water	642344	3M ESPE (Germany)
Maxxion R	After mixing: aluminum fluorosilicate glass, polycarboxylic acid, calcium fluoride, tartaric acid, water	010917	FGM (Brazil)
Riva Self Cure	Aluminum fluorosilicate glass, polyacrylic acid, pigments; tartaric acid, water, polyacrylic acid	11058422V	SDI (Australia)
Vitro Fil R	Aluminum-strontium fluorosilicate, dehydrated polyacrylic acid, ferrous oxide; polyacrylic acid, distilled water, tartaric acid	17100760	NOVA DFL (Brazil)
Vitro Molar	Barium and aluminum silicate, dehydrated polyacrylic acid, ferrous oxide; tartaric acid, polyacrylic acid, water	17090644	NOVA DFL (Brazil)
2. Resin-modified glass ionomer cements			
Fuji 2 LC (Gold Label Light-Cured Universal Restorative)	Aluminum fluorosilicate glass (85-95%), polyacrylic acid (5-15%); Polyacrylic acid, 2-HEMA, distilled water, UDMA, camforquinone	17020118	GC Corporation (Japan)
Resiglass R	Calcium, barium and aluminum fluorosilicate, polyacrylic acid, inorganic filler; di-methacrylate groups, deionized water, catalyst	102/18	Biodinâmica (Brazil)
Riva Light Cure	Aluminum fluorosilicate; polyacrylic acid, tartaric acid, 2-HEMA, acidic monomers, di-methacrylates	1105259	SDI (Austrália)
Vitremer	Aluminum fluorosilicate, potassium per-sulfate, ascorbic acid, pigments; polyalkenoic acid, acrylic acid copolymer, methacrylate groups, camphorquinone, water, 2-HEMA	N780313	3M ESPE (Germany)
Vitro Fil LC	Aluminum-strontium fluorosilicate, filler, initiators, ferrous oxide; 2-HEMA, aqueous solution of polyacrylic and tartaric acids, benzoyl peroxide, camphorquinone	18010035	NOVA DFL (Brazil)
3. Resin composite			
Filtek Z250 XT	Bis-GMA, Bis-EMA, UDMA, TEGDMA, Canforquinone, Zirconia-Silica filler (0.01-3.5 Micrometers)	911387	3M ESPE (Brazil)

Abbreviations: 2-HEMA, hydroxyethyl methacrylate; Bis-GMA, bisphenolglycidyl methacrylate; Bis-EMA, bisphenolglycidyl ethyl-methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

Pi 0.73 mM). This solution was 50% undersaturated when compared to human enamel/dentin (see References 24-26 for details on preparation). After 6 hours, a 0.1 ml volume of TISAB III (Orion 940911, Thermo Fisher Scientific, Cambridge, MA, USA) was added to the tubes. The specimens were then removed, washed with purified water, dried with laboratory absorbent tissue, and transferred to another tube containing 1.0 mL Re- solution (Tris buffer 0.1 M [pH 7.0] containing Ca 1.5 mM, Pi 0.9 mM, and KCl 150 mM).[21] After 18 hours of immersion in Re- solution, the same procedure as described for the De- solution was repeated. The tubes were maintained at 37°C under agitation (90 rpm) over the 12-day study period. After each daily cycle, fresh De- and Re- solutions were used. TISAB III was used to buffer the De- and Re- solutions.[10] The sources of Ca ions and Pi ion salts used to prepare the De- and Re- solutions were $CaCl_2$ and KH_2PO_4, respectively, and the pH was adjusted.

Determination of Fluoride

Fluoride concentration in the De- and Re- solutions was recorded daily using FI-SE (Thermo Scientific Orion 96-09, Orion Research Incorporated, Cambridge, MA, USA) coupled to the ion-analyzer Star A214 (Thermo Scientific Orion). The electrode was calibrated with the standards of fluoride solutions.[10] The fluoride concentration was determined by performing a linear regression analysis of the logarithm of fluoride concentrations of the standards with the respective mV readings (r^2=0.9999) on an Excel spreadsheet (Microsoft, Redmond, WA, USA). The results obtained from the De- and Re- solutions were expressed in μg F/ml. The amount of fluoride released by the materials each day in the De- + Re- solutions, the amount of fluoride on day 7 after the stabilization of all materials, and the cumulative amount of the 12-day period was expressed as μg F/cm^2 of the exposed surface area of the specimens.

Statistical Analyses

The daily fluoride concentrations of the De- and Re- solutions were analyzed descriptively. Data of daily fluoride release in the De- + Re- solutions was adjusted to a log-normal distribution. Three outliers were removed, and the remaining data were statistically analyzed by repeated measures for differences between the materials. Data on the cumulative difference of fluoride released by the materials evaluated over the 12-day period were log-transformed. One outlier was removed to homogenize the variance and normally distribute the measurements. All data were analyzed by analysis of variance (ANOVA), followed by Tukey's test (α=0.05), using SAS software 8.01 (SAS Institute Inc., Cary, NC, USA).

RESULTS

Qualitative Evaluation of Fluoride Release in De- and Re-solutions

The general pattern of daily fluoride release by the ionomeric materials in De- (Figure 1A) and Re- (Figure 1B) solutions over the 12-day period involved an early surge of fluoride release on the first five days, followed by a gradual decline, until stabilization throughout the remaining period. In general, more fluoride was released by the De- solution compared to the Re- solution.

However, when the pattern of fluoride release from each material in the De- and Re- solutions was compared, we detected three distinct patterns (Figure 2A, 2B, and 2C). Three GICs (Maxxion R, Vitro Fil R, and Ionglass R) exhibited pattern A of fluoride release, with more fluoride being released in the De- solution compared to the Re- solution over the 12 days, while none of the RMGICs exhibited pattern A of fluoride release (Figure 2A). Pattern B (Figure 2B) was exhibited by seven GICs (Bioglass R, EQUIA Forte Fil, Fuji 9, Ionofil Plus, Ketac Molar Easymix, Riva Self Cure, and Vitro Molar) and four RMGICs (Fuji 2 LC, Resiglass R, Riva Light Cure, and Vitro Fil LC). Pattern B was characterized by an initial higher release of fluoride in the De- solution compared to the Re- solution. However, this difference was not maintained over time. Pattern C (Figure 2C) was exhibited by one RMGIC (Vitremer), which showed a very similar pattern of fluoride release in the De- and Re- solutions over the 12-day period.

Quantitative Evaluation of Fluoride Release

Daily Release in De- + Re- Solutions — The amount of fluoride released daily in the De- and Re- solutions was summed. All ionomeric materials exhibited a surge in fluoride release during the first five days, followed by a decline and stabilization (Figure 3). ANOVA showed statistically significant differences for materials, time, and interaction (p<0.0001). The materials differed regarding the time period between the surge of fluoride release and stabilization. All GICs and RMGICs differed from the control group (see Supplementary Material, available online).

Certain GIC materials (Ionglass R, Ionofil Plus, Vitro Fil R, and Vitro Molar) exhibited a very fast surge in fluoride release that stabilized after day 2 of pH-cycling. Other GIC materials (Bioglass R, Fuji 2 LC, Ketac Molar Easymix, and Maxxion R) and two RMGIC

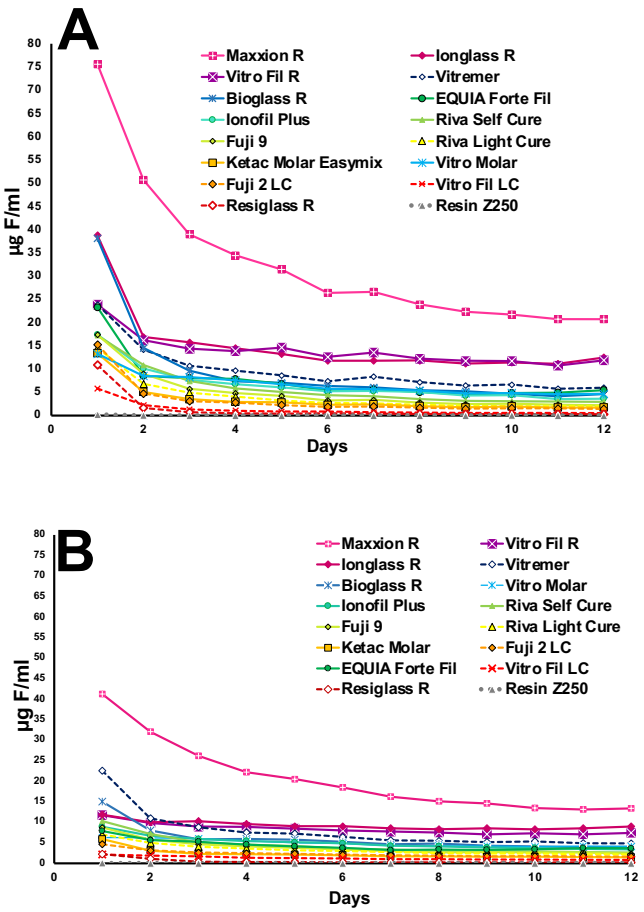

Figure 1. *Mean (n=6) daily fluoride concentration (µg F/ml) in demineralizing (A) and remineralizing (B) solutions by glass ionomer cements (continuous line) materials, resin-modified glass ionomer cements (dotted line), and the control (Resin Z250) over the 12-day period of the pH-cycling regime to which the specimens were subjected.*

materials (Riva Light Cure and Vitro Fil LC) stabilized after day 3. Two GIC materials (EQUIA Forte Fil and Fuji 9) and two RMGIC materials (Resiglass R and Vitremer) stabilized after day 4. In comparison, one GIC (Riva Self Cure) stabilized after day 5.

When the materials were compared on each day, one GIC (Maxxion R) exhibited the greatest fluoride release over all periods, while one RMGIC (Resiglass R) exhibited the lowest fluoride release. The statistical difference among the materials on each day is shown in the Supplementary Material (available online).

By day 6 of evaluation, the burst effect of fluoride release ceased for all materials, after which all materials released a constant amount of fluoride daily. Figure 4 shows the statistical difference among the materials on day 7 of evaluation. All fluoride materials differed from the resin composite negative control ($p<0.05$) on fluoride release. The GIC Maxxion R released the highest amount of fluoride ($p<0.05$), and the RMGIC material, Resiglass, released the lowest amount. Between the highest fluoride release by Maxxion R and the lowest by Resiglass R, groups of materials, irrespective of being GIC or RMGIC, were found by decreasing order of fluoride release; the statistical significance of fluoride release among these materials is depicted in Figure 4.

Cumulative Fluoride Release Over the 12 Days — Figure 5 shows the cumulative amount (µg F/cm^2) of fluoride released in the De- + Re- solutions over the 12 days. ANOVA showed a statistically significant difference among all materials. All ionomeric materials significantly differed ($p<0.0001$) from the resin composite negative control with respect to fluoride release. Out of all of the

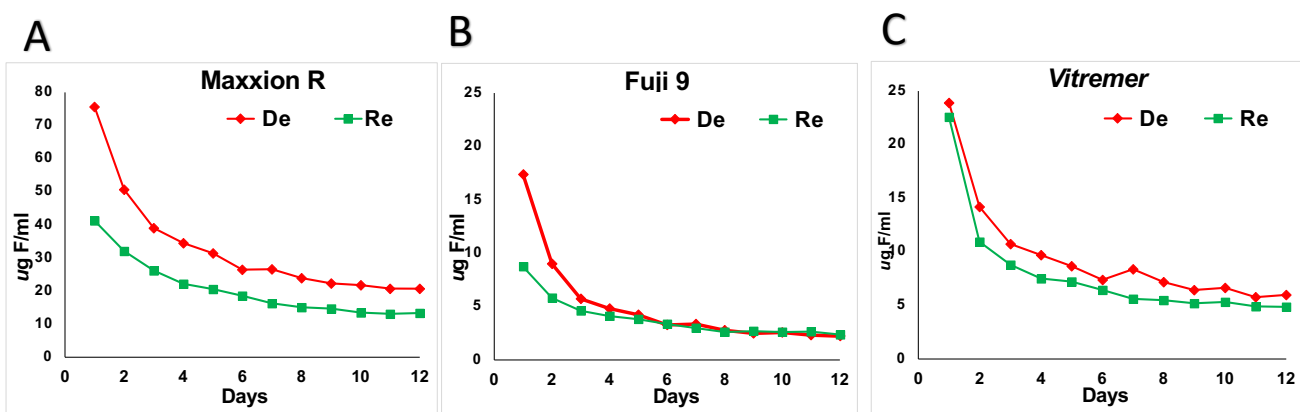

Figure 2. Representative patterns (A, B, and C) of fluoride release in De- and Re- solutions (µg F/ml) by glass ionomer cements and resin-modified glass ionomer cement materials over the 12 days of pH-cycling (resin-modified glass ionomer cements in italic).

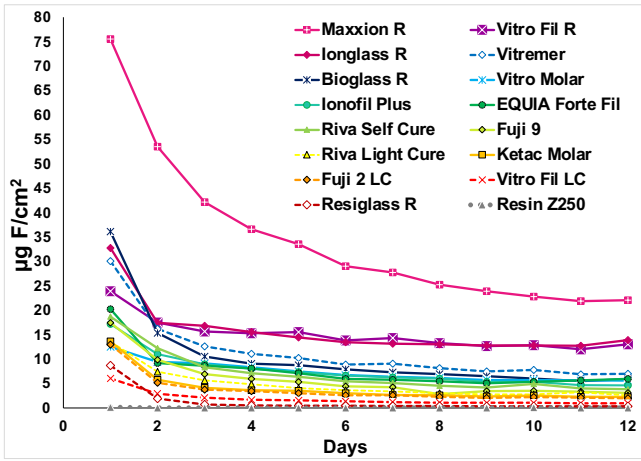

Figure 3. Daily fluoride released (mean, n=6) (µg F/cm²) in demineralizing + remineralizing solutions by glass ionomer cement materials (continuous line), resin-modified glass ionomer cement materials (dotted line), and the control (Resin Z250) during the 12 days of pH-cycling.

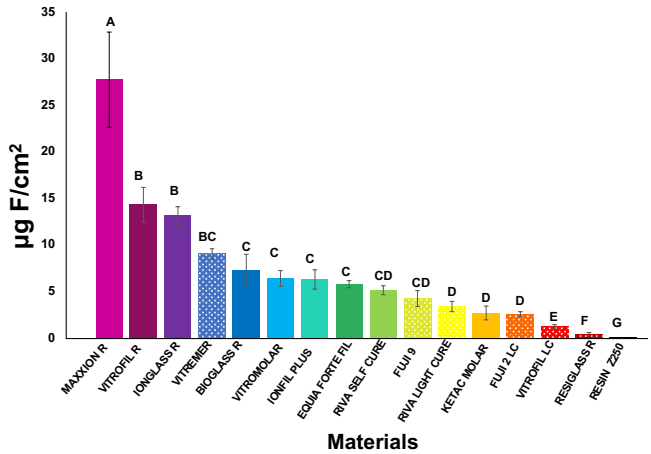

Figure 4. Mean (standard deviation; n=6) of fluoride release (µg F/cm²) by the glass ionomeric cements (solid fill) and resin-modified glass ionomer cement materials (textured fill), and the control (Resin Z250) in De- + Re- solutions on day 7 of the pH-cycling regime. Capital letters show differences among the materials (p<0.05).

GIC and RMGIC materials, one GIC material (Maxxion R) released the most fluoride, while one RMGIC material (Resiglass R) released the least fluoride. The other GICs and RMGICs released intermediate amounts of fluoride between these two materials.

Summary of the Results — The main results described above are summarized in Table 2, facilitating comparisons among all materials and among the GIC and RMGIC materials.

DISCUSSION

To have some validity, models should simulate real life. Thus, the mechanisms of fluoride release by GICs and RMGICs, and their anticaries potential, must be evaluated under conditions that simulate the physicochemical process of caries development.[7] Through mimicking the caries process of demineralization and remineralization, to which teeth restored with these materials are subjected daily, we evaluated the ability of ten conventional GICs and five RMGICs to release fluoride.

Our findings clearly showed qualitative (Figures 1-2) and quantitative (Figures 3-5) differences in fluoride release among the evaluated materials. However, these differences might not be due to any common or specific components reported by the manufacturers described in Table 1. For example, when the 10 GICs were compared with the five RMGICs based on the qualitative results of fluoride release, nothing differentiated the type of material (Table 2). The same was true when comparing GIC and RMGIC materials from the same manufacturer; thus, resin and aluminum fluorosilicate doped with other ions (Table 1) do not confer specific patterns in terms of qualitative fluoride release from RMGIC materials or GIC materials, respectively.

When evaluating the quantitative data of fluoride release and composition of the materials (Table 1), we did not identify any distinct or common element. No differentiation was detected between conventional GICs (doped or not with ions) and RMGICs during the early surge of fluoride release[27] nor for the time required to stabilize and sustain release. The early surge of fluoride release among all tested materials ranged from 48 to 120 hours (Figure 3; Supplementary Material, available online), irrespective of the composition of the material

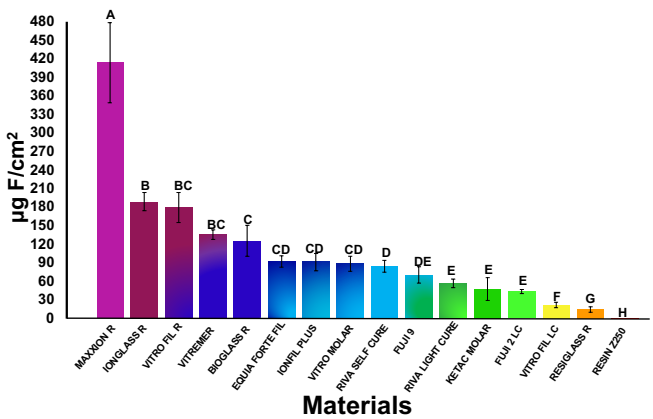

Figure 5. Mean (standard deviation; n=6) cumulative fluoride release (µg F/cm²) by the glass ionomer cements, the resin-modified glass ionomer cement materials (dotted lines), and the control (Resin Z250) over the 12 days of pH-cycling in De- + Re- solutions. Capital letters show differences between the materials (p<0.05).

Table 2: *Qualitative and Quantitative Fluoride Release Considering all Materials Evaluated (GICs and RMGICs) and by Type of Material (GIC or RMGIC)*

Material	**Fluoride Release (Qualitative patterns)[a]**			**Fluoride Release (Quantitative Ranking)**
	A	**B**	**C**	
ALL	Maxxion R VitroFil R Ionglass R	Bioglass R EQUIA Forte Fil Fuji 9 Ionofil Plus Ketac Molar Fuji 2 LC Riva Light Cure Riva Self Cure VitroFil LC Resiglass R VitroMolar	Vitremer	1- Maxxion 2- VitroFil 2- Ionglass 2-3 Vitremer 3- Bioglass R 3- Vitro Molar 3- Ionofil Plus 3- EQUIA Forte Fil 3-4 Riva Self Cure 3-4 Fuji 9 4- Riva Light Cure 4- Ketac Molar 4- Fuji 2 LC 5- VitroFil LC 6- Resiglass R
GIC	Maxxion R VitroFil R Ionglass R	Bioglass R EQUIA Forte Fil Fuji 9 Ionofil Plus Ketac Molar Easymix Riva Self Cure Vitro Molar	None	1- Maxxion 2- VitroFil 2- Ionglass 3- Bioglass R 3- Vitro Molar 3- Ionofil Plus 3- EQUIA Forte Fil 3-4 Riva Self Cure 3-4 Fuji 9 4- Ketac Molar
RMCIG	None	Fuji2 LC Resiglass R Riva Light Cure VitroFil LC	Vitremer	1- Vitremer 2- Fuji 2 LC 2- Riva Light Cure 3- VitroFil LC 4- Resiglass R

Abbreviations: GIC, glass ionomer cement; RMCIG, resin-modified glass ionomer cement.

[a]A = greater fluoride release in De- solution compared with Re- solution during the study period; B = initial higher release in De- solution; C = similar release in both solutions over the whole period.

(Table 1). The early surge is induced by a superficial rinse that causes an initial higher dissolution of the complexes of aluminum or phosphate with fluoride, which form during the acid-base reaction.[16] This process is mainly regulated by glass particles inside the cement that have not reacted.[27] After the higher release of fluoride on the first day, sustained release during the remaining period might be the result of diffusion through pores, fissures, and bulk diffusion.[28,29]

This study also ranked fluoride concentration on day 7 of the experiment and over the cumulative 12-day period (Table 2). However, no association between the composition of the materials and their ability to release more or less fluoride was detected. Conventional aluminum fluorosilicate GICs tended to release more fluoride compared to RMGICs (Figures 4 and 5). Consequently, the GIC Maxxion R and the RMGIC Resiglass R released the greatest and lowest amounts of fluoride, respectively. However, one RMGIC (Vitremer) was still among the four materials that released the most fluoride, and there was also one conventional GIC (Ketac Molar Easymix) (Figures 4 and 5).

Although we could not explain the qualitative and quantitative patterns of fluoride release based on the composition of the evaluated materials, the results could be used to indicate their anticaries potential. Fluoride interferes with the physicochemical process of caries development, reducing the demineralization of enamel and dentin, and enhancing remineralization during restoration.[7] Therefore, maintaining constant low fluoride concentrations in the oral cavity for prolonged periods are more important than maintaining high concentrations for short periods of time. Thus, the burst effect of fluoride release found for all materials might be less relevant in terms of efficacy on caries control compared to the sustained release shown by all materials after day 6 (Figure 3; Supplementary Materials, available online). However, the early burst of fluoride release might have some clinical relevance for repairing caries lesions on the surface of teeth located close to areas of GIC or RMGIC restorations. This possibility is remote, even for the GIC material Maxxion R, which had the highest ability to release fluoride during the first three days of the burst effect. During the first three days, this material released a mean of 55 μg F/cm^2. Thus, restoration of a 1 x 1 cm surface area would release 55 μg of fluoride in the mouth, which could be relevant in terms of remineralization, if it was not diluted very fast by salivary clearance.[30] Indeed, the biofilm formed around the restoration site is the only area where fluoride that is released in the oral cavity by GIC or RMGIC is retained and shows an anticaries effect.[6] Thus, the capacity of this particular GIC's fluoride release might be balanced with its mechanical properties.

The experimental data showed that GICs and RMGICs interfere with caries development in the enamel or dentin adjacent to restoration sites by releasing fluoride. This phenomenon reduces demineralization and enhances remineralization.[7] However, the key mechanisms of this process have not been identified. Our findings showed three patterns of fluoride release in De- and Re- solutions (Figures 1 and 2). If reducing demineralization is more relevant, the GICs and RMGICs that showed pattern A of fluoride release would be more effective at controlling caries. If fluoride is as effective at reducing demineralization as it is at enhancing remineralization, pattern C (as exhibited by Vitremer) would be highly effective at controlling caries. For the anticaries effect of toothpaste, the effect of fluoride on the remineralization of enamel and dentin might be more relevant than reducing demineralization.[31] Thus, further studies are required to test these potential pathways in GIC materials.

Dental caries is a chronic disease and caries lesions progress each time sugar is ingested.[7] Consequently, fluoride must be constantly available in biofilm to interfere with the caries process. Thus, the anticaries potential of the tested GIC and RMGIC materials should be reflected by their ability to sustain fluoride release, as shown in the quantitative results for daily (Figure 4) and cumulative (Figure 5) fluoride release. From day 6 of evaluation, all materials presented sustained fluoride release, differing statistically on day 7 (Figure 4). Maxxion R released the most fluoride (27.7 μg F/cm^2), whereas Resiglass R released the least (0.41 μg F/cm^2). Only a very low physicochemical fluoride concentration is needed to interfere with caries,[7] and it could be achieved in biofilm fluid by all materials;[6] however, the 10-fold difference in fluoride release between Maxxion R and Resiglass R might be relevant in mitigating caries, but this requires further evaluation. In comparison, the high release of fluoride by Maxxion R could provoke the erosive dissolution of the restoration site.[32] Thus, the persistence of restoration might be balanced by the anticaries property of this material when considering the cost-effectiveness of this intervention.

The distinct qualitative and quantitative patterns of fluoride release by these materials might reflect their anticaries properties. Thus, future studies should evaluate how different doses of fluoride release influence enamel-dentin demineralization and/or remineralization in validated models.[7]

CONCLUSION

In conclusion, the GICs and RMGICs evaluated exhibited distinct qualitative and quantitative patterns of fluoride release under conditions simulating the caries process, which might reflect their anticaries potential.

Acknowledgments

The manuscript was based on the Master of Science thesis of the first author at the Graduate Program in Dentistry, Cariology Area, Piracicaba Dental School, University of Campinas, Brazil.

Funding

The first author received a scholarship from University of Costa Rica (N° 048-2017, Costa Rica). This manuscript is the outcome of a research project that was also supported by Funcamp (Proc 4887.1, Brazil).

Conflict of Interest

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

(Accepted 13 June 2020)

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Protocols for Mechanical Cleaning of the Post Space on the Bond Strength Between Root Dentin and Cementation System

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Clinical Relevance

Mechanical protocols can be promising alternatives to chemical protocols for cleaning the root dentin surface before cementation of fiberglass posts.

SUMMARY

Objective: To evaluate the effects of mechanical versus chemical cleaning protocols for cleaning the root dentin surface before cementation of fiberglass posts for their effect on the bond strength, failure mode, and dentinal penetration of the cementing agent using an etch-and-rinse adhesive system on dentin prepared to receive a fiberglass post.

Methods: Forty roots of bovine teeth were endodontically treated and prepared for fiber post cementation. The specimens were randomized into 4 groups of 10: Control group (CO) - irrigation with

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http://doi.org/10.2341/20-194-L

2.5% NaOCl; DW group- irrigation with distilled water; RB group - rotating brush for cleaning root canals, and CUI group - continuous ultrasonic irrigation. The fiberglass posts were cemented, and the specimens were immersed in distilled water for 6 months. A push-out test was performed on the cervical, middle, and apical thirds of the samples. Dentinal penetration of the cementing agent and the fracture pattern were evaluated by laser confocal microscopy. Statistical analysis was performed using analysis of variance (ANOVA) and post hoc Tukey tests (α=0.05). Results: The RB and CUI groups showed significantly higher bond strength values when compared to the Control and DW groups ($p<0.05$). In addition, in the control and DW groups, the apical third presented lower bond strength values when compared to middle and cervical thirds.

Conclusion: While DW showed the highest incidence of adhesive type failure, CUI resulted in the highest dentinal penetration of the cementing agent ($p<0.05$). RB and CUI resulted in the highest bond strength between cementation system and root dentin. In addition, CUI favored greater dentinal penetration of fiberglass post cementing agent.

INTRODUCTION

A residual-free dentin surface is crucial for the formation of a suitable adhesive interface when an etch-and-rinse adhesive system is used.[1-4] However, during the preparation of the post space for cementing a fiberglass post, heat is generated in the root canal that leads to plasticization of the endodontic filling materials used.[5,6] These materials precipitate on the dentin surface to form a layer comprising gutta-percha residue, endodontic cement, and dentin debris.[7]

Several chemical protocols have been proposed for cleaning and disinfecting the intraradicular space prior to cementation of the fiberglass post.[8-10] Certain substances such as peroxides that are routinely used in the irrigation of root canals generate oxidative radicals.[9,11] These peroxides have a negative impact on the degree of conversion of the resinous compounds, thereby impairing the adhesion of the etch-and-rinse adhesive system on the dentin substrate.[12-14] It was previously reported that the use of chemical protocols without mechanical agitation showed less removal of dentin residues compared to irrigation associated with mechanical agitation.[15,16]

Mechanical protocols using ultrasonic agitation, lasers and intracanal brushes combined with irrigation solutions have shown promising results in cleaning intraradicular dentin.[17,18] However, the use of irrigation solutions, such as NaOCl, polyacrylic acid, chlorhexidine, or silver nanoparticles influenced the bond strength and interface permeability of adhesive material to dentin substrate.[19] To our knowledge, no studies to date have investigated the effectiveness of mechanical methods without chemical irritants for dentin post space cleaning prior to cementing fiberglass posts using etch-and-rinse adhesive or conventional dual resin cements.

In addition, although ultrasonic agitation of the irrigant is considered effective in cleaning root dentin,[20,21] it is an expensive alternative due to the need for specific equipment and supplies. A root canal cleaning brush that is activated in continuous rotational movement can be a cheaper alternative. Due to direct contact with the dentin surface, it easily removes debris from the plasticization of the materials used in endodontic filling.[17,22]

Therefore, the aim of this study was to evaluate the effects of cleaning the post space using 2.5% NaOCl (Control), distilled water (DW), rotating brush (RB) in continuous motion with distilled water, and continuous ultrasonic irrigation (CUI) with distilled water on the bond strength, fracture pattern, and dentinal penetration of the cementation system at the cervical, middle, and apical thirds of the root canal. The null hypothesis was: There is no difference in bond strength, fracture pattern, and the extent of cementation system penetration among the tested intra-root dentin cleaning protocols.

METHODS AND MATERIALS

Sample Preparation

The study was approved by the animal research ethics committee of the local university (Protocol No. 1,201,002). Forty healthy bovine incisors with similar root anatomy and devoid of structural changes were selected; they were stored in 0.1% thymol solution at 4°C for a maximum of 1 month.

The roots were standardized for length and measured 17 mm from the root apex. The root canals were prepared using the Universal ProTaper System (Dentsply Maillefer, Ballaigues, Jura-Nord Vaudois, Switzerland). Chemical-mechanical preparation and root canal filling were performed according to the protocol proposed by Aranda-Garcia and others[23] Vertical condensation of the root canal filling material was done and the root canals were provisionally restored with a temporary cement (Coltosol; Coltene, Rio de Janeiro, RJ, Brazil). The roots were kept in 100% relative humidity, at 37°C for seven days. The post space (length of 11 mm) was prepared with a #1

wide bur (Dentsply) supplemented by a #1 bur (White Post DC; FGM, Joinville, SC, Brazil). The preparation was performed without coolant. Periapical radiographs were performed to verify that the root filling was correctly removed.

Treatment With Cleaning Protocols

Following post space preparation, the specimens were randomly allocated into three groups based on the cleaning protocol. These included:

- Control group: The post space was irrigated with 10 mL of 2.5% sodium hypochlorite using a 30 G endodontic irrigation cannula (NaviTip; Ultradent, South Jordan, UT, USA). Cervical to apical movements were performed at an amplitude of 3 mm. Following a 1-min irrigation, aspiration was performed with the aid of a vacuum tip (Capillary tips; Ultradent). After that, 17% EDTA was applied to the root canal for 3 min and then dried with absorbent paper tips (Maillefer, Dentsply Sirona, York, Pennsylvania, USA).
- Distilled water (DW): The post space was irrigated with 10 mL of distilled water using a 30 G endodontic irrigation cannula (NaviTip; Ultradent). Cervical to apical movements were performed at an amplitude of 3 mm. Following a 1-minute irrigation, aspiration was performed with the aid of a vacuum tip (Capillary tips: Ultradent).
- Rotating brush (RB): The post space was initially filled with distilled water followed with a root canal cleaning brush (MKLife, Porto Alegre, RS, BR) (Figure 1A), which was used in continuous rotational movement (1200 rpm, 4 N) for 15 seconds using a specific device (XSmart Plus, Dentsply Sirona). The same operation was repeated three times.
- Continuous ultrasonic irrigation (CUI): The ultrasonic tip (AE12; Adiel, Ribeirão Preto, SP, BR) (Figure 1B) adapted in an ultrasonic unit (Ultrawave XS; Ultradent) was used at a power level of 5. The insert was introduced 1 mm short of the length of the post space and moved from cervical to apical direction for 1 min under continuous irrigation with distilled water.

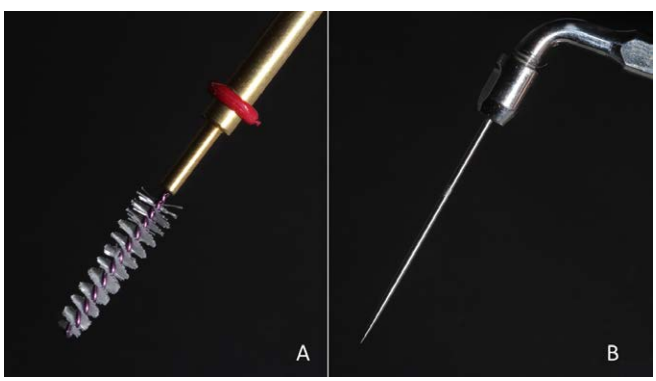

Figure 1. *(A): Root canal cleaning brush. (B): The ultrasonic tip.*

After completion of the cleaning protocols, the post space was irrigated with 5 mL of distilled water and dried with absorbent paper tips (Maillefer, Dentsply Sirona). The external surface of the fiber post (White Post DC # 1; FGM) was cleaned with 95% alcohol and conditioned with 37% phosphoric acid (Condac 37; FGM) for 1 minute. Silane (Prosil; FGM) and an etch-and-rinse adhesive (Ambar; FGM) were subsequently applied, and light cured for 60 seconds (Valo, Ultradent).

Fiber Post Cementation

The dentin post space was conditioned using 37% phosphoric acid for 15 sec and irrigated with distilled water for 30 sec. Subsequently, the dentin was gently dried with absorbent paper tips (Maillefer, Dentsply Sirona). The etch-and-rinse adhesive system Ambar; FGM) was applied to the post space and photoactivated for 20 seconds. The conventional dual resin cement (AllCem Core; FGM) was manipulated according to the manufacturer's recommendations and inserted into the root canal using specific intracanal tips of the cementation system. Thereafter, the fiberglass post was positioned and photoactivated for 40 seconds on each side of the specimen. The specimens were stored in distilled water for 6 months at 37°C; the water was renewed every 15 days.

Bond Strength and Failure Mode Evaluation

Subsequently, the specimens were included in polyester resin (Maxi Rubber, Diadema, SP, Brazil) and sectioned into apical, middle, and cervical thirds of 2.0 ± 0.1 mm in thickness. Irregularities generated during the sectioning procedures were removed with 1200 water-wet sandpapers (3M Oral Care, Saint Paul, MN, USA) in a polishing machine (Buehler, Lake Bluff, IL, USA). A universal testing machine (EMIC, São José dos Pinhais, PR, Brazil) was used for the push-out test at a crosshead speed of 0.5 mm/min. The slices were inserted into a metal device with a central hole (Ø=3 mm) larger than the diameter of the canal. The push-out test was performed as outlined by Magro and others.[24]

Failure mode was assessed using a confocal laser microscope (LEXT OLS4100; Olympus, Shiniuki-ku, Tokyo, JP) as described by Ramos and others.[8] An image of each quadrant of the perimeter of the post space was obtained (four images per specimen). The

failure mode was classified as type 1 (adhesive) when it occurred between the fiber post and the cement; type 2 (adhesive) when it occurred between the dentin and the cement; type 3 (cohesive) when it occurred inside the cement; and type 4 (mixed) when several failure modes occurred in combination.[8,25] Figure 2 shows adhesive 1 and 2, cohesive and mixed failure modes.

Intratubular Cement Penetration

Intratubular cement penetration was examined using an Olympus FluoView Confocal Laser 1000 microscope (Olympus Corporation, Tokyo, Japan) at 20× magnification. Prior to fiber post cementation, 0.01% of rhodamine dye was incorporated into the resin cement, as described by Ramos and others.[8] Cervical, middle, and apical samples were assessed. The images were recorded as quadrants; 10 measurements were taken in each quadrant of the dentin post space. Dentin samples were analysed using a 10× oil lens at 100× magnification. The image depth was 70 μm and the dimension was 800 × 800 pixels. Image J software (Softonic International S.L., Barce, Spain) was used to analyse the images as described by Ordinola-Zapata and others.[26] The average (in μm) of the 40 measured values was calculated to measure the extent of dentinal penetration of the cementing agent.

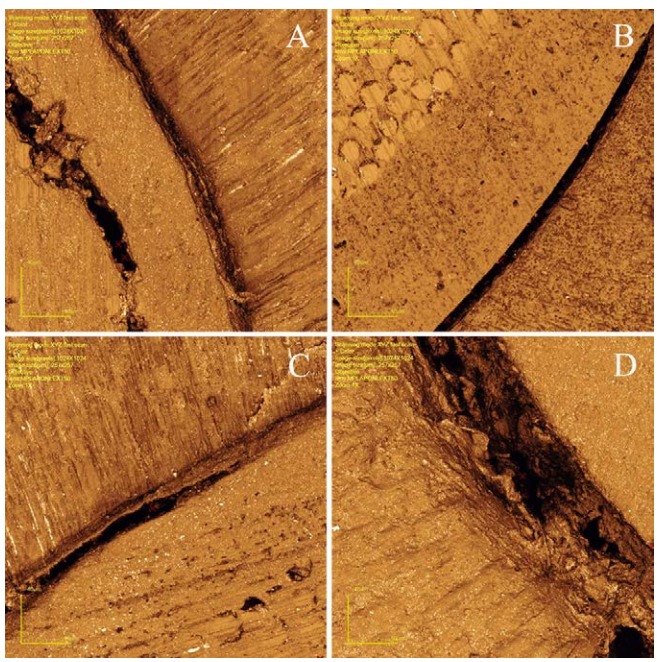

Figure 2. *Representative image of the failure mode: type 1 (A), adhesive, between post and cementation system; type 2 (B) adhesive, between dentin and cementation system; type 3 (C) cohesive, inside the cementation system; type 4 (D) mixed, combination of fracture pattern. Scale: 1024×.*

Statistical Analysis

The data obtained were initially subjected to a Shapiro-Wilk normality test. The results of the push-out test and that of the dentinal penetration of the cementation system were analysed using the analysis of variance (ANOVA) and Tukey tests. All tests were evaluated with a significance level of 5%.

RESULTS

Bond Strength and Failure Mode Evaluation

Table 1 shows the arithmetic mean and standard deviation of the bond strength values (in MPa) of the cementing system, after using the cleaning protocols in all the thirds of the post space. There was no statistically significant difference between the CO and DW groups, regardless of the root region ($p<0.05$). There was no difference in the bond strength values between the RB and CUI groups ($p>0.05$). The RB and CUI groups showed significantly higher bond strength values when compared to the CO and DW groups ($p<0.05$). In addition, in the CO and DW groups, the apical third showed lower bond strength values when compared to middle and cervical thirds.

Regarding the failure mode, the CO and DW groups presented the highest incidence of type 2 failure. However, the RB and CUI groups presented the highest incidence of type 3 failure. Figure 3 shows the fracture pattern distribution for the different groups.

Intratubular Cement Penetration

Figure 4 shows representative images of the cementation system penetration inside dentin based on the protocol adopted for cleaning the post space (500×). In all post space thirds, the CO and DW groups showed the lowest and the CUI group showed the highest penetration of the cementation system ($p<0.05$) (Table 2). In addition, in the CO and DW groups, the apical third showed less cementation system penetration when compared to the middle and cervical thirds.

DISCUSSION

In this study, all the cleaning protocols resulted in different values of bond strength, failure mode, and dentinal penetration of the cementation system using the etch-and-rinse adhesive. The null hypothesis was thus rejected. Bond strength values were higher after mechanical cleaning of the intraroot space compared to the irrigation protocols alone. Possibly, the superiority of mechanical cleaning is due to the absence of interaction between irrigators for chemical cleaning with cementation systems, which could negatively

Table 1: *Bond Strength Values of the Cementing System by Dentin Cleaning Protocols and Thirds of the Post Space*

Root Region	Groups Mean in MPa (SD)			
	CO	DW	RB	CUI
Cervical	20.14 (6.95) Ab	21.46 (4.37) Ab	36.71 (2.77) Ca	35.20 (3.37) Ca
Middle	19.64 (3.62) Ab	19.87 (4.93) Ab	34.08 (3.71) Ca	32.18 (3.13) Ca
Apical	15.07 (1.96) Bb	14.43 (4.45) Bb	31.14 (2.24) Ca	31.23 (5.09) Ca

Note: Different uppercase letters in the same column indicate a statistically significant difference ($p<0.05$); Different lowercase letters on the same line indicate a statistically significant difference ($p<0.05$). Abbreviations: CO, cleaning protocol with 2.5% sodium hypochlorite; DW, cleaning protocol with distilled water; RB, cleaning protocol with rotating brush; CUI, cleaning protocol with continuous ultrasonic irrigation.

interfere with adhesion to the dentin substrate.[19,27] The preparation of the post space after endodontic treatment generates the formation of a smear layer on the instrumented dentin. This smear layer is impregnated with residues of materials such as endodontic sealants and pasted gutta-percha. This residue has specific characteristics different from a standard smear layer generated during endodontic treatment.[7,28,29] In the present study, endodontic treatment was performed followed by the post space preparation to simulate this condition.

Obtaining clean dentinal surfaces after mechanical preparation of the post space is a critical step towards the ideal retention of the fiberglass post when resin cement is used. Therefore, an adequate removal of the residual layer is necessary.[30] Sodium hypochlorite solution (NaOCl) is the most recommended irrigant for root canals[24] and it is known for its antibacterial and proteolytic actions, dissolution capacity, and debridement properties.[31] However, it degrades into sodium hydroxide and hypochlorous acid, thereby promoting the release of *singlet* oxygen.[32] Sodium hypochlorite thus acts as an oxidizing agent that creates an oxygen-rich layer on the dentin wall that inhibits resin polymerization and increases marginal microleakage.[33,14] This effect hinders polymerization of the methacrylate chain of resinous compounds.[2,6] Furthermore, NaOCl can cause collagen dissolution by

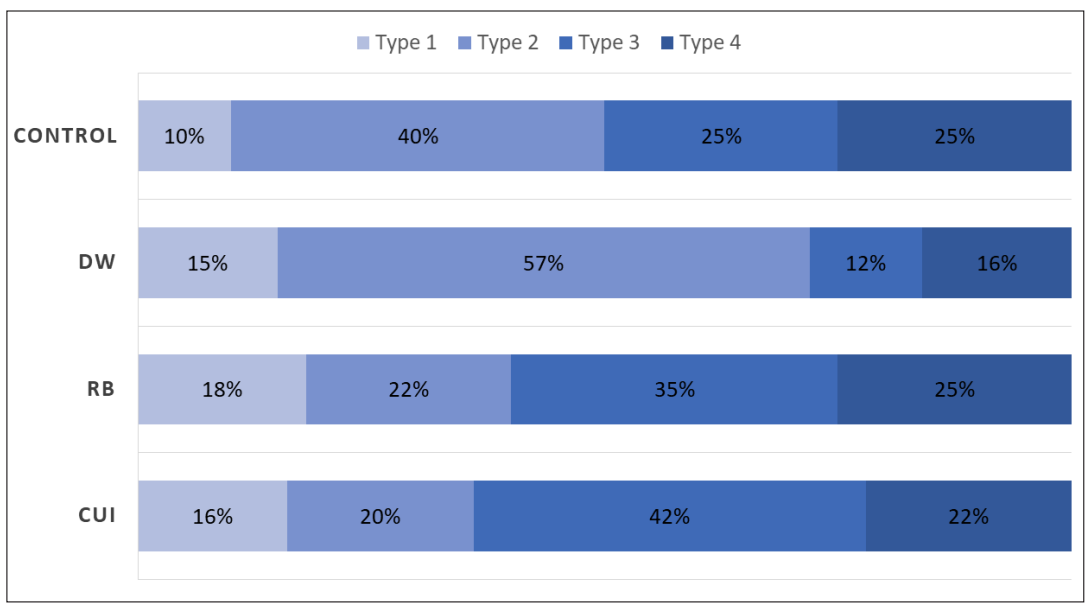

Figure 3. *Incidence of the failure mode demonstrated after using the cleaning protocols. Type 1 (adhesive, cement/post); Type 2 (adhesive, cement/dentin); type 3 (cohesive); type 4 (mixed). Abbreviations: CI, conventional irrigation; CUI, continuous ultrasonic irrigation; RB, rotating brush.*

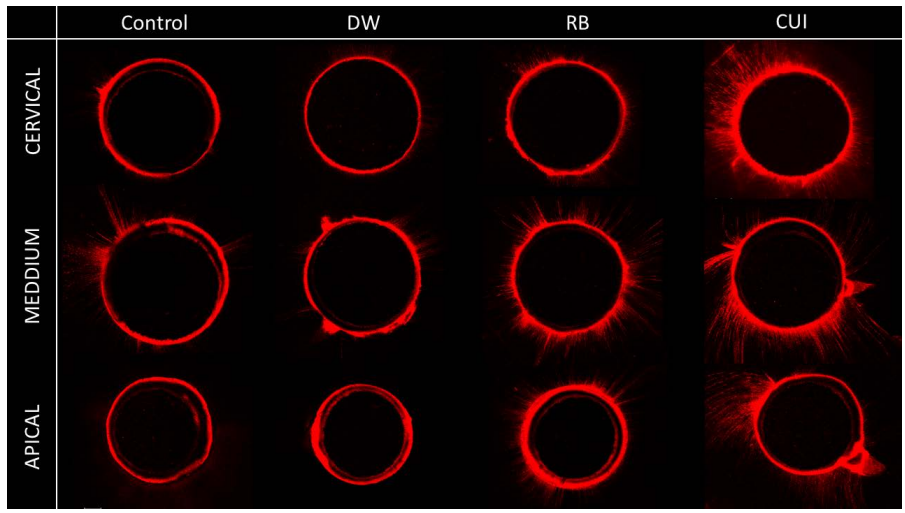

Figure 4. *Representative image of dentinal penetration of the cementation system, depending on the thirds and cleaning protocols of the post space. Abbreviations: CI, conventional irrigation; CUI, continuous ultrasonic irrigation; RB, rotating brush. Scale: 100x. 600 µm.*

breaking carbon bonds and disorganizing the protein's primary structure, which results in degradation of the dentinal tissue.[34] Therefore, only distilled water was used as an irrigator in the mechanical cleaning protocols of this study.

In the present study, the groups that used mechanical cleaning showed the best results, both for bond strength and penetrability of resin cement in intratubular dentin, even when compared to irrigation with NaOCl. These results indicate that mechanical cleaning with ultrasonic irrigation and rotating brushes can be an effective alternative for dentin cleaning without the interference of chemical solutions. It is likely that the ultrasonic agitation of the distilled water and the brush attrition over the dentin surface resulted in a greater removal of dentin debris. Consequently, there was greater exposure of intertubular dentin in these groups. Previous studies showed that continuous ultrasonic irrigation showed better dentinal cleaning of the post space when compared to conventional irrigation using only irrigation solutions.[35,36] However, the effectiveness of continuous ultrasonic irrigation combined with distilled water or saline solution has been little investigated to date.

A previous study conducted by Jamleh and others[35] showed that the CUI can effectively clean the root canal system. However, it was not possible to avoid combining it with irrigators. On the other hand, Spoleti and others[37] found that ultrasonic activation with sterile saline was able to disinfect the root canal effectively. This issue should be further investigated in future studies, seeking alternatives for cleaning the post space, without the interference from chemical irrigation substances.

In this study, the two-step etch-and-rinse adhesive system was used after acid conditioning of the

Table 2: *Dentinal Penetration of the Cementation System Depending on the Dentin Cleaning Protocols and Thirds of the Post Space*

Root Region	**Dentinal Penetration by Method Used µm (SD)**			
	CO	**DW**	**RB**	**CUI**
Cervical	36.01 (1.46) Ac	39.63 (5.61) Ac	98.94 (11.63) Cb	191.27 (11.34) Ea
Medium	39.14 (9.82) Ac	42.69 (6.40) Ac	93.50 (9.76) Cb	199.82 (19.58) Ea
Apical	29.85 (6.81) Bc	23.04 (5.09) Bc	73.23 (4.80) Db	107.52 (9.34) Ca

Note: Different uppercase letters in the same column indicate a statistically significant difference (p<0.05); Different lowercase letters on the same line indicate a statistically significant difference (p<0.05). Abbreviations: CO, cleaning protocol with 2.5% sodium hypochlorite; DW, cleaning protocol with distilled water; RB, cleaning protocol with rotating brush; CUI, cleaning protocol with continuous ultrasonic irrigation.

dentin. This system renders a more effective form of hybridization, which allows efficient cleaning of smear layer and higher adhesion values.[38] Due to this cleaning potential of the etch-and-rinse adhesive system, it can be indicated to assist the mechanical cleaning of the root canal during the preparation of the post space. A study conducted by Saraiva and others[39] showed that only the pretreatment with NaOCl did not improve the bond strength of the adhesive cement to the root canal dentin. However, it suggested that the use of 37% phosphoric acid for 60 seconds presented a beneficial effect on bond strength in the apical third of the root. For this reason, this type of adhesive system was used in this study.

Regarding the bond strength in the different root regions, it was found that the CO and DW groups showed lower values in the apical third compared to the middle and cervical thirds. This result can be expected due to the greater difficulty of access and polymerization in this region.[40] In addition, the penetrability of the cementation system was also lower in the apical third when compared to the cervical and middle thirds. This may possibly be related to poor cleaning in the apical third.

Confocal microscopy makes it possible to ascertain the depth and extent of penetration of materials into the root dentin.[27] In this study, in all thirds of the post space, the cleaning protocol with CUI resulted in the greatest penetration of the adhesive system into the dentinal tubules. This result suggests that the CUI effected greater dentinal cleaning. A recent study conducted by Neelakantan and others[41] showed that ultrasonic irrigation was able to significantly remove more smear layer and yield open dentinal tubules when compared to the sonic activation. However, the endodontic literature is inconsistent with regard to the effects of ultrasonic irrigation on smear layer removal.[41,42] On the other hand, a greater dentinal penetration depth obtained when CUI was used did not appear to have interfered with the bond strength values. This question can be justified due to the reduced extent of penetration of the adhesive system in the dentinal tubules, approximately 15%.[2,43]

Concerning the failure mode, it was observed that in the CO and DW groups the most frequent type of failure was the type 2, wherein the rupture occurred more frequently at the interface between dentin and adhesive system. It is likely that a greater amount of dentin residue on the surface reduced the micro-retention following hybridization.[3] Thus, the significance of the results of this study was to demonstrate the relevance of mechanical protocols in assisting the cleaning of the dentinal surface and its potential contribution in improving adhesion of the fiberglass post to root dentin.

Although it is difficult to reproduce *in vivo* conditions *in vitro*, the present study was designed based on several parameters that were previously established in the literature. However, variations in the temperature of the oral cavity and the impact of masticatory forces are factors that were not considered in this study. Therefore, randomized clinical trials analysing the longevity and quality of these protocols must be encouraged. In addition, new materials and mechanical cleaning devices for the root canal must be developed and analyzed to elucidate the impact of cleaning protocols on the bond strength of the resin.

CONCLUSIONS

Following post space preparation, cleaning the dentin surface with a rotational brush in continuous motion or continuous ultrasonic irrigation yielded the highest bond strength of the cementation system to the root dentin. However, ultrasonic agitation resulted in a greater penetration of the adhesive system in dentin when compared to the other modalities. In addition, groups with mechanical treatment had the highest incidence of cohesive failure when compared to the others.

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 presented in this article.

(Accepted 04 October 2020)

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Volume 46 /Number 4
July/August 2021

www.jopdent.org
351-476

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