Microtensile Bond Strength of Tooth-colored Materials to Primary Tooth Dentin

Preenan Suwatviroj, DDS, MDSc Louise B. Messer, BDSc, LDS, MDSc, PhD, FICD
Joseph E. A. Palamara, BSc (Hons), PhD

Dr. Suwatviroj was a postgraduate student in pediatric dentistry, University of Melbourne, and is currently pediatric dentist, Institute of Dentistry, Soi. Bamrajnaradul, Nothaburi Province, Thailand; Dr. Messer is the Elsdon Storey Professor of Child Dental Health and Director of Graduate Studies, and Dr. Palamara is lecturer, School of Dental Science, University of Melbourne, Victoria, Australia.
Correspond with Dr. Messer at ljbm@unimelb.edu.au

Abstract

Purpose: This study measured the microtensile bond strengths of 2 tooth-colored restorative materials with and without conditioning of primary teeth dentin, and examined the micromorphology of the debonded surfaces and material-dentin interfaces.
Methods: Cylindrical specimens of packable composite resin (PCR) and resin-modified glass ionomer cement (RMGIC) bonded to dentin of primary teeth were ground to an hourglass shape and tested for microtensile bond strength. The debonded surfaces and material-dentin interfaces were prepared and examined under a scanning electron microscope (SEM).
Results: The microtensile bond strength values (mean±SD, in MPa) of PCR (Filtek P60 with Single Bond) and RMGIC (Fuji II LC), with or without the application of Cavity Conditioner (14.8±5.36, 12.01±4.43, 11.94±4.60, respectively), did not differ significantly (P>.05). Partial adhesive and partial cohesive failures within the restorative material predominated. The distributions of failure modes did not differ significantly between groups (P>.05). Under SEM, each material was seen to be closely adapted to dentin. Dentinal tubules were enlarged with etching, and the depth of penetration of resin tags of PCR was greater than for RMGIC. Smear plugs were incompletely removed by cavity conditioning.
Conclusions: The bond strength of the PCR, Filtek P60 with Single Bond, to dentin of primary teeth was comparable to that of the RMGIC, Fuji II LC. Conditioning of the cavity preparation with Cavity Conditioner did not improve the bond strength of Fuji II LC. The distribution of failure modes did not differ between materials.
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Composite resin (CR) can be used successfully as a restorative material in primary molars, particularly in the late mixed dentition.[1] Packable composite resin (PCR) has been introduced recently for posterior restorations, and it may have a place in restorative dentistry for children. This material is less sticky than previous CRs, with a higher viscosity due to modification in filler loading and filler types.

Resin-modified glass ionomer cements (RMGICs) have been developed from conventional glass ionomer cement (GIC) by adding components such as bis-glycidyl methacrylate (bis-GMA), hydroxyethyl methacrylate (HEMA), or a water/HEMA mixture to replace some of the water in conventional GIC. Complex materials have been developed by modifying the polyacrylic acid with polymerizable side chains; light curing increases the working time and reduces water sensitivity.[2] Advantageous in pediatric dentistry, fewer clinical steps are used in placing RMGICs than for CR restorations.[3] Since the mechanical properties of RMGICs are improved by adding polymerizable monomers and the fluoride release is similar to that of a conventional GIC, these materials may benefit patients with high caries activity.[4]

Pretreatment of dentin with polyacrylic acid before the placement of GIC has been thought to increase the bond strength to dentin.[5] The conditioner, Cavity Conditioner (GC Corp, Tokyo, Japan) contains 20% polyacrylic acid and 3% aluminum chloride. Polyacrylic acid has functional carboxyl ion groups that form hydrogen bonds, promoting cleansing and wetting of substrates and thereby removing cutting debris and residual organic contamination such as saliva or gingival crevicular fluid on the tooth surface.[5] Since polyacrylic acid is also part of the glass ionomer system, any residual acid does not interfere with the setting reaction of the cement. Cement adaptation to the cavity walls is promoted by alteration of the surface energy of the tooth by the acid.[6] Pretreatment with polyalkenoic acid exposes surface collagen fibrils to a depth of about 0.5μm, which is less severe than treatment with phosphoric acid as the hydroxyapatite in the exposed collagen fibrils is not fully denuded.[7] Aluminum chloride is thought to strengthen the collagen fibers after demineralization.[8]

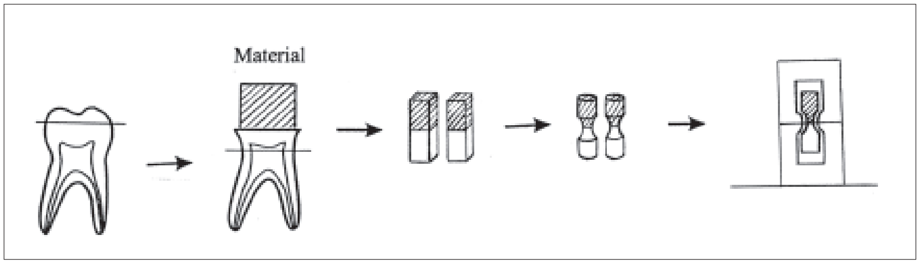


Figure 1. Preparation of hour-glass shaped specimens for microtensile bond strength test.

Important structural differences in primary tooth dentin compared with permanent tooth dentin may influence bonding characteristics.[9] The peritubular dentin of primary teeth is 2 to 5 times thicker than that of permanent teeth, and primary tooth dentin is less mineralized than dentin in permanent teeth.[10] Dentin tubules in primary teeth are less dense and of lower permeability than those in permanent teeth.[11] These factors may explain the lower bond strength reported for primary teeth than for permanent teeth under the same bonding protocol.[12] In occlusal and gingival areas, bonding agents showed greater inhibition of microleakage in permanent teeth than in primary teeth.[12]

The purposes of the present study were two-fold:

1. to evaluate the microtensile bond strength of a PCR and RMGIC with and without conditioning to dentin of primary teeth;
2. to observe the micromorphology of the debonded surfaces and material-dentin interfaces.

Methods

Specimen preparation and test groups

Thirty extracted human primary molars (caries-free or caries-limited to 1 proximal surface only) were stored in thymol solution. Occlusal surfaces were wet ground to expose a flat dentin surface and polished with wet 600-grit silicon carbide paper. Teeth were assigned randomly to 3 groups (10 teeth per group) and prepared as follows:

Group 1

Etchant (Scotchbond Multi-purpose, batch no.1 EP, 3M Espe Co, St Paul, Minn) was applied to the dentin surface for 15 seconds and rinsed; 2 consecutive layers of bonding agent (Single Bond, 3M Espe Co, St Paul, Minn) were applied, gently dried, and light cured for 10 seconds; then PCR (Filtek P60, 3M Espe Co, St Paul, Minn) was placed approximately 4 mm thick using 2 incremental curings of 20 seconds each (3M Curing Light XL 3000, wavelength 400-500 nm, 3M Espe Co, St Paul, Minn).

Group 2

RMGIC (Fuji II LC, shade A1, GC Corp, Tokyo, Japan) approximately 4 mm thick was placed on the dentin surface and light cured separately for 20 seconds for each capsule.

Group 3

The dentin surface was conditioned for 10 seconds (Cavity Conditioner, GC Corp, Tokyo, Japan), rinsed, and air dried. Then RMGIC (Fuji II LC, shade A1, GC Corp., Tokyo, Japan) was mixed for 10 seconds and placed as an increment approximately 4 mm thick with separate light curing for 20 seconds for each capsule.

The exposed RMGIC was coated with petroleum jelly. All teeth were stored for 1 week in tap water at 37°C before preparation for testing. A slow-speed saw under water coolant was used to remove the root and cut the bonded material and dentin into a bar-shaped specimen approximately 1.5 mm thick and 6 mm long. The material portion of the specimen was stabilized in a lathe, and a fine-finishing round diamond bur in a high-speed handpiece attached to a micrometer (to control movement) was used under air-water spray coolant to cut a cylinder. The interface area was ground to form an hour-glass shaped specimen 1.05±0.06 mm in diameter (Figure 1). The diameter at the adhesive interface was measured using a digital caliper.

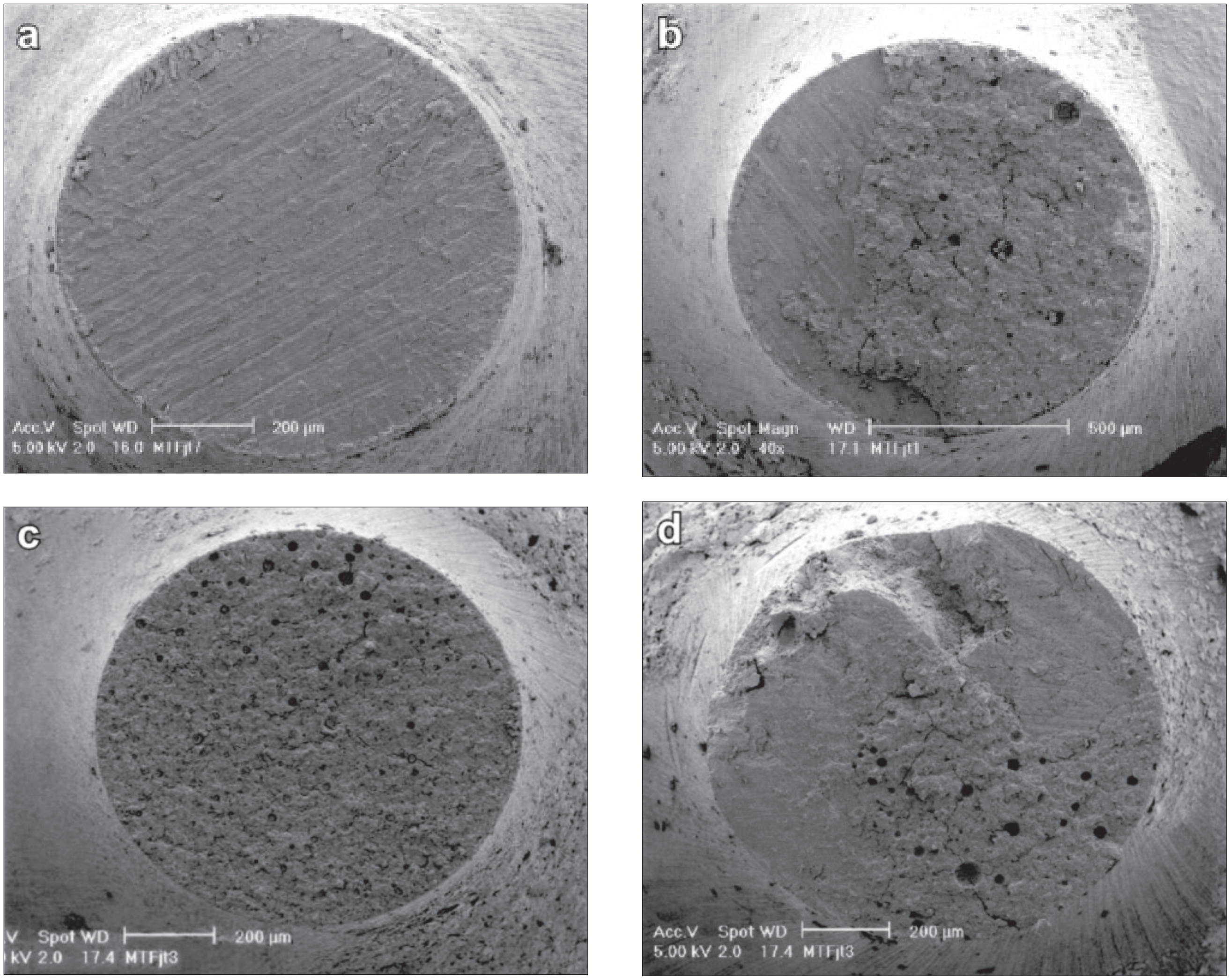
Microtensile bond strength test

Each specimen was tested for microtensile bond strength using a universal mechanical testing machine (Instron, Model 5544, Instron Corp, Canton, Mass) with the specimen in a holder and stressed in tension at a crosshead speed of 0.5 mm/minute.[13] The tensile force (in Newtons, N) at failure was recorded and converted to tensile stress (in megapascals, MPa) by the following formula:

Tensile strength (MPa)=tensile force (N)x10^{-6}/cross-sectional area (m^2)

Examination of debonded surfaces

The debonded surfaces of the specimens were air dried, mounted on aluminum stubs with conductive silver liquid (Pro Sci Tech, Queensland, Australia), gold sputter-coated



Figures 2a-2d. Debonded surfaces of material and dentin.
Figure 2a. Adhesive failure.
Figure 2b. Partial adhesive failure and partial cohesive failure in material.
Figure 2c. Cohesive failure in material.
Figure 2d. Partial adhesive failure and partial cohesive failure in RMGIC and dentin.

(Gold Sputter Coater S150B, Edwards, England), and observed under field emission scanning electron microscopy (SEM, Philips XL 30 FEG, Eindhoven, The Netherlands) at ×40–50 magnification. The failure modes were classified into 5 types as follows:

1. Type 1—adhesive failure between material and dentin;
2. Type 2—partial adhesive failure between material and dentin and partial cohesive failure in material;
3. Type 3—cohesive failure in dentin;
4. Type 4—cohesive failure in material;
5. Type 5—mixed failure showing partial adhesive failure in dentin and partial cohesive failure in dentin and material.

Examination of material-dentin interfaces

Nine extracted, caries-free human primary molars were stored in thymol solution. A disc of dentin (approximately 1 mm thick) was obtained by cutting the occlusal enamel of each tooth to expose dentin and making a second cut parallel to the first cut using a low-speed diamond saw under water spray. The dentin surface was polished with wet 600-grit silicon carbide paper. The dentin discs were assigned randomly to 3 groups (3 discs per group), and specimens of PCR, RMGIC, and RMGIC with cavity conditioning were prepared as previously discussed.

Shallow grooves were cut into the surface of each specimen using a high-speed tungsten carbide pear-shaped no. 330 bur (Jet, ISO no. 237001008, LOT no. C6295, Beavers, Ontario, Canada) under water coolant, taking care to avoid cutting into the bonded interface. The specimens were fixed in 10% phosphate buffered formalin for 24 hours, rinsed in distilled water 3 times each for 15 minutes, dehydrated in an ascending ethanol series (10%, 20%, 30%, 50%, 70%, 90%) for 15 minutes in each and 3 times in 100% ethanol, and then critical-point dried (Samdri PVT-3, Tousimis Research Corp, Rockville, Md) to remove all moisture. The specimens were fractured

Table 1. Microtensile Bond Strength of Packable Composite Resin and Resin-modified Glass Ionomer Cement With and Without Conditioning of Dentin of Primary Teeth

Group and materials	Number of specimens	Mean microtensile bond strength (± SD) in megapascals
1. Packable composite resin (Filtek P60) with bond (Single Bond)	10	14.84 (5.36)*
2. Resin-modified glass ionomer cement (Fuji II LC)	10	11.94 (4.60)*
3. Resin-modified glass ionomer cement (Fuji II LC) with cavity conditioning (Cavity Conditioner)	10	12.01 (4.43)*

*No significant difference in bond strength between 3 groups of materials (ANOVA, F ratio=1.180, df=2, *P*=.323).

by hand along the prepared grooves, mounted on aluminum stubs with conductive silver liquid, and gold sputter-coated (Gold Sputter Coater S150B, Edwards, England). The material-dentin interfaces were observed under a field-emission SEM (Philips XL 30 FEG, Eindhoven, The Netherlands).

Statistical analysis

Descriptive statistics were performed and bond strength values for each group were compared using a one-way analysis of variance (ANOVA) and the least significant different multiple comparison test. The critical level of alpha was set at 0.05. The distribution of modes of failure was compared using the chi-square test.

Results

Microtensile strength of materials bonded to dentin

The mean microtensile bond strengths (± SD) of the materials bonded to dentin are shown in Table 1. Although the mean microtensile strength of PCR (Filtek P60) bonded to dentin using Single Bond exceeded that of RMGIC (Fuji II LC) with and without dentin conditioning, these values did not differ with statistical significance (ANOVA, F ratio=1.180, df=2, *P*=.323).

Distribution of failure modes

More adhesive failures (Type 1) between the materials and dentin occurred in the PCR group (4/10) than in either RMGIC group (1/10 in each group; Table 2). More partial adhesive failures and partial cohesive failures (Type 2) occurred in both RMGIC groups (8/10 and 7/10, respectively) than in the PCR group (6/10). There were no cohesive failures in dentin in any group, and very few cohesive failures in any material. The distribution of failure modes between groups did not differ significantly (x^2=6.286, df=6, *P*=.392).

Observations of debonded surfaces

The debonded tooth surfaces as seen under SEM are shown in Figure 2. Surfaces with adhesive failures showed no adherent material; dentinal tubules were exposed (Figure 2a). In partial adhesive failures and partial cohesive failures, some material was adherent to the dentin surface and the dentin was partly denuded (Figure 2b). The single specimen showing cohesive failure of RMGIC on conditioned dentin showed the dentinal surface fully covered with the material (Figure 2c). The specimens of RMGIC on dentin (with or without conditioning), demonstrating both partial adhesive failure and partial cohesive failure, showed areas of dentinal fragmentation, denudation, and partial material coverage (Figure 2d).

Observations of material-dentin interfaces

Cross-sectional views of the micromorphology of the material-dentin interfaces as seen under SEM are shown in Figures 3 to 5.

The interface of PCR and dentin, including an intermediate layer of adhesive resin (approximately 5-5.5 mm thick) forming a well-defined boundary with the resin, is shown

Table 2. Failure Modes Between Materials and Primary Teeth Dentin

Group and materials	No. of specimens	Type 1 failure*	Type 2 failure†	Type 3 failure‡	Type 4 failure§	Type 5 failure‖
1. Packable composite resin (Filtek P60) with bond (Single Bond)	10	4	6	0	0	0
2. Resin-modified glass ionomer cement (Fuji II LC)	10	1	8	0	0	1
3. Resin-modified glass ionomer cement (Fuji II LC) with cavity conditioning (Cavity Conditioner)	10	1	7	0	1	1

*Adhesive failure between material and dentin.
†Partial adhesive failure between material and dentin and partial cohesive failure in material.
‡Cohesive failure in dentin.
§Cohesive failure in material.
‖ Mixed failure (partial adhesive failure in dentin and partial cohesive failure in dentin and material).

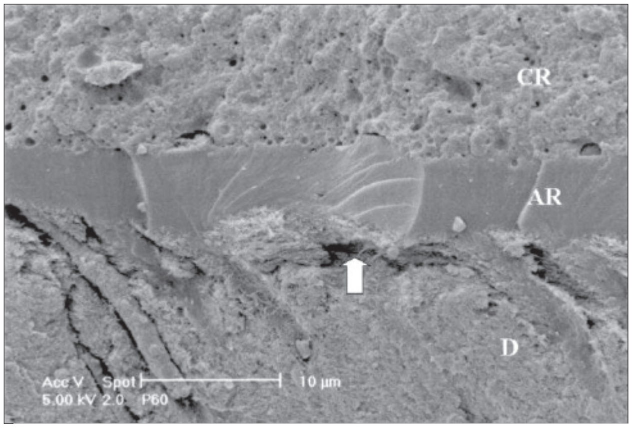


Figure 3. Interface of packable composite resin (Filtek P60 with Single Bond) and dentin.*
*CR=composite resin; AR=adhesive resin; D=dentin; white arrow=void from incomplete penetration of the AR.

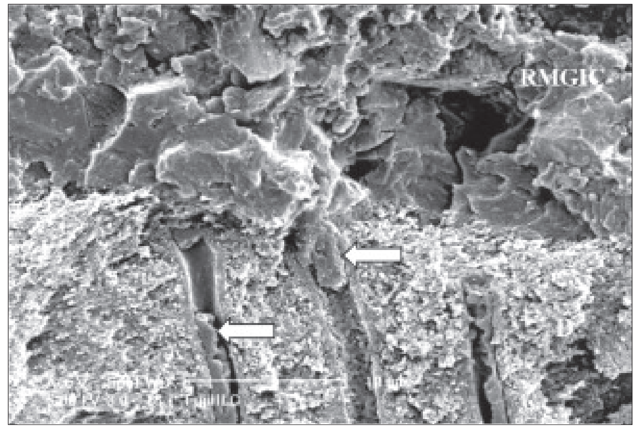
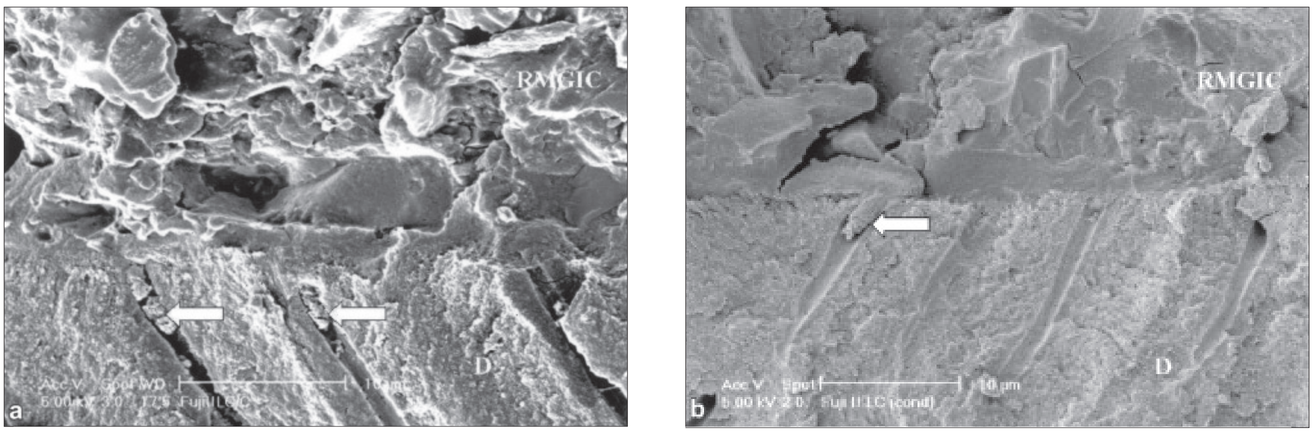


Figure 4. Interface of resin-modified glass ionomer cement (Fuji II LC) and dentin.*
*RMGIC=resin-modified glass ionomer cement; D=dentin; white arrow=penetration of RMGIC tags; open arrow=possible smear plugs.



Figures 5a-5b. Interface of resin-modified glass ionomer cement (Fuji II LC with the use of Cavity Conditioner) and dentin.*
*RMGIC=resin-modified glass ionomer cement; D=dentin; white arrow=smear plugs; open arrow=possible penetration of RMGIC to infiltrate smear plugs.

in Figure 3. Dentinal tubule enlargement (to a width of approximately 2-3 mm) by etching was noted, with penetration of resin tags into the tubules. Voids were seen at the dentin/adhesive resin interface (white arrow in Figure 3), possibly due to changes in orientation of the fracture plane to traverse lines of weakness during specimen preparation.

The interface of RMGIC and unconditioned dentin is shown in Figure 4. The material was closely adapted to the dentin, and the specimen showed smear plugs in the dentinal tubules (open arrow in Figure 4). The dentinal tubules appeared narrower (1-1.5 mm) than those in etched dentin in specimens prepared with PCR and bonding agent. Tags of RMGIC completely occluded the dentinal tubules (white diamond arrow in Figure 4), but penetrated only a short distance (approximately 4 mm) from the interface layer.

The interface of RMGIC and conditioned dentin is shown in Figure 5. The material was closely adapted to the dentin and a few dentinal tubules were occluded with smear plugs (white diamond arrows in Figure 5a). The dentinal tubules appeared narrower (1-1.5 mm) than those in etched dentin in specimens prepared with PCR and bonding agent. Tubules were occluded to variable extents with material penetration (open arrow in Figure 5b). In addition, there was partial infiltration into smear plugs remaining within the tubules due to incomplete conditioning.

Discussion

Microtensile strength of materials bonded to dentin

Several primary teeth used in this study were carious, and the quality of dentin near caries may differ from that of caries-free teeth. Therefore, only caries-free surfaces were used in specimen preparation.

Other studies have investigated the bond strength of CR to dentin of primary teeth using several adhesive systems, reporting values ranging from 6.2 to 18.2 MPa.[12,14-17] These values are consistent with the findings of the present study (14.84 MPa). Variations in bond strength can reflect a number of factors, such as the specimen storage medium, adhesive system, bond strength test used, distance of the dentin studied from the pulp, location of the dentin studied (occlusal, proximal, or buccal), and the quality of the dentin.[18-20] Concerning the test used, the modified microtensile bond strength test of Phrukkanon et al (1998),[13] was used in the present study. This method uses a round cross-sectional bonding area that is considered preferable for uniform stress distribution.[13]

Other studies have investigated the bond strength of the RMGIC used in the present study (Fuji II LC, with Cavity Conditioner) to dentin of primary teeth, reporting values ranging from 16.0 to 22.26 MPa.[17,21] These values exceed those found in the present study (12.01 MPa and 11.94 MPa, with and without dentinal conditioning, respectively), but a different bond strength test was used.

In the present study, no significant difference in microtensile bond strength was seen between the PCR and the RMGIC, bonded with or without dentinal conditioning, to dentin of primary teeth. This supports the observations of others reporting bond strengths of CR and RMGIC bonded to primary teeth.[17] However, values for microtensile bond strengths in the present study were lower, possibly reflecting differing dentinal quality (carious teeth vs exfoliated teeth) and differences in extraoral storage time. Since no difference was observed with or without conditioning, the possibility of omitting this step should be studied further. If such studies confirm that conditioning is required for primary teeth, then further research should identify which type of conditioner provides the best bonding.

The mean microtensile bond strengths of RMGIC specimens with or without dentinal conditioning did not differ significantly in the present study, supporting previous observations on permanent teeth.[5,22] However, one particular study of RMGIC bonded to primary dentin (both carious and noncarious) reported that pretreatment with polyacrylic acid increased shear bond strength, except for Vitrebond on noncarious dentin.[23] Further research is needed to determine which type of conditioner provides the best bonding for each RMGIC.

The bond strength of adhesive resin to dentin is partly dependent on calcium content; calcium content decreases in dentin closer to the pulp, leading to lower bond strengths.[12] During specimen preparation in the present study, the occlusal surface was ground to a flat surface. Due to the large and prominent pulp in primary teeth, the bonded surface of each specimen obtained might represent various distances from the pulp. The higher moisture content and less intertubular material in deeper dentin are thought to interfere with bond strength.[18] Also, the variable water content and organic content of dentin may produce inconsistencies in bond strengths and influence the range of values.[23] Collectively, these variations could contribute to the wide range of values found in the present study, as indicated by the standard deviations shown in Table 1.

Several studies have compared the bond strength of adhesive restorative materials to dentin of primary and permanent teeth.[12,16-17,21] The variability in results may be due to the different materials and methods utilized. The bond strengths between materials and dentin of primary teeth were lower than those of permanent teeth, perhaps as a result of a thicker hybrid layer limiting complete penetration of adhesive resin in primary teeth.[12,17,24] However, a study of adhesion of a fluoridated light-activated resin bonded to primary and permanent dentin reported no significant difference in shear bond strengths.[16] For GIC and RMGIC, the differences in dentin of primary and permanent teeth appeared to have no effect on the shear bond strength.[21] With reference to the structural differences in dentin of primary and permanent teeth, the same conditioning and bonding process may affect the bond strength differently, and this remains to be elucidated.

Failure modes

There were no significant differences in the distribution of failure modes between the 3 groups of materials, with combined partial adhesive/partial cohesive failure in the material predominating. In contrast, a previous study on bonding of Fuji II LC to dentin in primary teeth reported mostly cohesive failures.[17] Of interest, the latter study also reported higher microtensile bond strengths which could allow the RMGIC specimens to fail cohesively prior to adhesive failure occurring. In the present study, lower bond strengths were found and adhesive failures were noted more frequently in the CR group, suggesting that the bond strengths observed may represent the strength of adhesive bonding per se.

The material-dentin interface

The adhesive resin or bonding agent has a major role in stabilizing the hybrid layer and forming resin extensions into the dentinal tubules.[7] The bonding agent Single Bond is an adhesive system which also removes the smear layer, combining the etching primer and adhesive resin into a single solution. During etching, dentin is demineralized by phosphoric acid so that the smear layer is removed and the collagen fibrils are exposed. The inclusion of water and ethanol in Single Bond may facilitate resin penetration into the collagen network.[25] In the present study, the SEM observations suggest that planes of weakness may have been formed where the adhesive resin failed to infiltrate areas of dentin which had been etched excessively. According to Nor et al, smear layers are more easily removed from primary teeth than from permanent teeth, suggesting greater reactivity to acidic dentin conditioners.[26] Although resin tags were noted in the present study, the adhesive resin did not penetrate completely to the base of the exposed collagen and voids were noted. This porous zone is thought to be susceptible to degradation by oral fluids or bacterial invasion.[27] Permeation of oral fluids into this demineralized dentin zone may be the source of postoperative discomfort.[28] Enlargement of the tubules was also noted in the present study as the result of etching which removed inorganic material from the peritubular dentin.

Close adaptation of the RMGIC to dentin was noted for both conditioned and unconditioned specimens. The adhesion of this material to enamel and dentin is due to ionic interaction between the cement and the tooth substrate and a mechanical interlocking of the polymer to the dentin.[29] The penetration of short tags of RMGIC into the dentinal tubules confirms the observations of others who have noted that long tags are not characteristic of this material.[8,29-31] The conditioning process did not completely remove the smear layer, as smear plugs were found occluding some dentinal tubules and dentinal tubules were not enlarged. No differences were apparent between conditioned and unconditioned specimens, as residual smear plugs were found in both groups. Consequently, it is not surprising that the microtensile bond strengths of the 2 groups did not differ significantly.

Conclusions

An in vitro study of the microtensile bond strengths of a PCR (Filtek P60) and RMGIC (Fuji II LC) bonded to dentin of primary teeth showed that:

1. The microtensile bond strength of Filtek P60 with the use of Single Bond was comparable to that of Fuji II LC; conditioning of dentin with Cavity Conditioner did not improve the bond strength of Fuji II LC. In addition, conditioning was not able to remove the smear layer completely.
2. Although both materials adapted closely to the dentin, incomplete penetration of the adhesive resin into the demineralized dentin was observed in some specimens of PCR.

Acknowledgments

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Abstract of the Scientific Literature

Influence of Site Preparation Methods on the Pain Reported During Palatal Infiltration Using the Wand Local Anesthetic System

This study compared the effect of different site preparation methods on the pain response to palatal injections using the slow rate flow of the Wand Local Anesthetic System through a 30-gauge needle. Fifteen adults with previous experience with local anesthetics volunteered to participate in the clinical trial. A questionnaire assessing prior pain experience with dental injections and the level of anxiety was obtained. The methods of tissue preparation were: topical anesthesia (20% benzocaine gel); pressure anesthesia (cotton-tipped applicator firmly pressed); both; and neither. Each participant received 4 palatal injections in 2 separate appointments (2 injections per session) during which the tissues sites prepared in a random sequence, with the left side always receiving the first injection. Forty-eight seconds were necessary to administer 0.3 ml of local anesthetic. Patients, as well as the operator, rated the pain intensity postoperatively immediately after the procedure. All the recorded pain measurements failed to demonstrate statistical differences for the various site preparation methods when the slow rate of local anesthesia was given.

Comments: This study provided important information regarding site preparation and pain response. Extrapolation of the results to the pediatric patient is difficult to make due to the tremendous importance of the distraction technique whenever administering local anesthesia, especially to the palatal site. **MG**

Address correspondence to Robert E. Primosch, DDS, MS, MEd, Department of Pediatric Dentistry, University of Florida, PO Box 100426, Gainesville, FL 32610-0426. rprimosch@dental.ufl.edu.

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