Scientific Article

A comparison of three resin bonding agents to primary tooth dentin

Fernando Borba de Araujo, DDS, PhD Franklin García-Godoy, DDS, MS

Myaki Issáo, DDS, PhD

Abstract

This study determined the shear bond strength of resin composites to primary dentin using three dentin adhesives and the presence or absence of a hybrid zone. The buccal and lingual surfaces of 40 recently extracted noncarious primary teeth were ground flat with SiC paper ending with the 600 grit. The teeth were divided at randomly into eight groups of five teeth (10 surfaces) each: 1) Unetched dentin, dry dentin, All-Bond 2/Bis-Fil P; 2) Unetched dentin, moist dentin, All-Bond 2/Bis-Fil P; 3) Dentin etched for 15 sec with 10% phosphoric acid, dry dentin, All-Bond 2/Bis-Fil P; 4) Dentin etched for 15 sec with 10% phosphoric acid, moist dentin, All-Bond 2/Bis-Fil P; 5) Dentin etched with 10% maleic acid for 15 sec, dry dentin, Scotchbond Multi-Purpose/Z100; 6) Dentin etched with 10% maleic acid for 15 sec, moist dentin, Scotchbond Multi-Purpose/Z100; 7) Dentin etched with 10 citric acid/ 3% ferric chloride, dry dentin, Amalgambond Plus/Z100; 8) Dentin etched with 10 citric acid/3% ferric chloride, moist dentin, Amalgambond Plus/Z100. All teeth were thermocycled 1000x (5 and 55°C, 30-sec dwell time), and shear bond strength testing was conducted using an Instron™ (crosshead speed 0.5 mm/min). Failure sites after debonding were examined with the SEM. For each group, one additional tooth was used to prepare two class V cavities (one facial and one lingual) restored according to the specification in each group, sectioned buccolingually and examined with the SEM. The results, in MPa, were: 1) 12.55 ± 5.97; 2) 10.41 ± 6.16; 3) 9.94 ± 7.26; 4) 12.25 ± 4.70; 5) 13.02 ± 8.01; 6) 16.51 ± 8.62; 7) 12.51 ± 8.95; 8) 17.93 ± 6.44. ANOVA and Student-Newman-Keuls tests showed no statistically significant differences. SEM evaluation showed that the smear layer was removed in all groups exposing primary dentin tubules infiltrated by resin. A resin-reinforced hybrid layer was readily seen in all specimens. (Pediatr Dent 19:253–57, 1997)

Despite the vast number of research reports on the efficacy of bonding of resin adhesives to dentin of permanent teeth, very few have addressed resin bonding to primary dentin.[1–8]

Bonding to dentin has been difficult due to several factors, among them the use of hydrophobic materials, the hydrophilic nature of dentin (containing circa 20% water by weight), the achievement of pulpal biocompatibility, the development of a sufficiently high bond strength to overcome the polymerization shrinkage forces generated by light-cured resin-based materials, and until recently, a poor understanding of the presence and nature of the smear layer.[9]

One method to improve resin composite adhesion to dentin is through resin infiltration of the dentin (hybridization).[10] The primer resins diffuse into the outer few micrometers of the dentin rendering it more porous by acidic conditioning.

The newer bonding agents are hydrophilic and capable of forming a hybridization zone between resin and dentin. The bond strength of some of these hydrophilic primers and bonding agents or the formation of the hybrid zone in primary teeth has not been reported.

The purpose of this study was to evaluate the shear bond strength and the presence or absence of the hybrid layer when using three new adhesives in primary dentin.

Methods and materials

Forty noncarious primary molars extracted due to near exfoliation were used. Immediately after extraction, teeth were cleaned with curettes and placed in distilled water for no longer than 3 months. The buccal and lingual surfaces were ground to dentin immediately before the experiment using a series of wet SiC paper ending with 600 grit.

The teeth were divided randomly into eight groups of five teeth (10 surfaces) each: Group 1, 2 and 3 received five coats of the All-Bond 2 Primers A and B (Bisco, Itasca, IL) mix, applied to the dentin with a disposable brush and gently blown with air for 5 sec. For group 1, a thin layer of the All-Bond 2 Dentin Enamel Bonding Agent (Bisco, Itasca, IL) was then applied with a disposable brush and light-cured (Optilux 400, Demetron, Danbury, CT) for 20 sec. For group 2, the dentin was remoistened with a cotton pellet containing distilled water, and then five coats of the All-Bond

2 Primers A and B mix were placed and a thin layer of the All-Bond 2 Dentin Enamel Bonding Agent was then applied as for group 1. For group 3, the dentin was dried with oil-free compressed air for 5 sec, etched with 10% phosphoric acid (Bisco, Itasca, IL) for 15 sec, rinsed with distilled water for 5 sec, and the dentin air-dried with oil-free compressed air. Five coats of the All-Bond 2 Primers A and B mix and the All-Bond 2 Dentin Enamel Bonding Agent were then applied as for groups 1 and 2. For all three groups, a nylon cylinder filled with Bis-Fil P hybrid resin composite was placed over the treated dentin, the excess composite removed with a dental explorer, and the resin light-cured for 40 sec. Immediately, the teeth were placed in distilled water for 48 hr.

TABLE. SHEAR BOND STRENGTH AND FAILURE SITE FOR THE DIFFERENT GROUPS

Group	*Mean MPa*	*SD*	*Range*	*AR*	*RC*	*DC*
All-Bond 2/Bis-Fil P Dry dentin	12.55	5.97	4.48–22.12	8/10	2/10	0/10
All-Bond 2/Bis-Fil P Moist dentin	10.41	6.16	3.28–20.92	8/10	2/10	0/10
All-Bond 2/Bis-Fil P Acid-etched/dry dentin	9.94	7.26	2.69–25.71	7/10	2/10	1/10
All-Bond 2/Bis-Fil P Acid-etched/moist dentin	12.25	4.70	5.97–17.93	8/10	2/10	0/10
SMP/Z100 Dry dentin	13.02	8.01	5.23–29.89	4/10	4/10	2/10
SMP/Z100 Moist dentin	16.51	8.62	4.63–30.64	2/10	3/10	5/10
Amalgambond/Z100 Dry dentin	12.51	8.95	1.49–30.19	4/10	1/10	5/10
Amalgambond/Z100 Moist dentin	17.93	6.44	6.72–40.35	12	38	50

AR = adhesive-resin failure, RC = resin cohesive failure, DC = dentin cohesive failure.

In group 4, the dentin was dried with oil-free compressed air for 5 sec, etched with 10% phosphoric acid for 15 sec, and rinsed with distilled water for 5 sec. The dentin was air-dried with oil-free compressed air and remoistened with a cotton pellet containing distilled water. The All-Bond 2 Primers A and B mix and All-Bond 2 Dentin Enamel Bonding Agent were then applied as in groups 1, 2, and 3 followed immediately by the Bis-Fil P hybrid resin composite as in the three earlier groups.

In groups 5 and 6 the dentin was dried with oil-free compressed air for 5 sec and etched with 10% maleic acid (3M Co., St. Paul, MN) for 15 sec, rinsed with distilled water for 15 sec and gently air-dried with oil-free compressed air. For group 5, Scotchbond Multi-Purpose (SMP) Primer (3M Co., St. Paul, MN) was applied to the dentin with a disposable brush and gently air-dried. For group 6, this step was preceded by remoistening the dentin with a cotton pellet containing distilled water. For both groups 5 and 6, SMP Adhesive (3M Co., St. Paul, MN) was applied with a disposable brush and light-cured for 10 sec. Immediately, a nylon cylinder filled with Z100 (3M Co., St. Paul, MN) hybrid resin composite was placed over the treated dentin, the excess composite removed with a dental explorer, and the resin light-cured for 40 sec. Immediately, the teeth were placed in distilled water for 48 hr.

In groups 7 and 8 the dentin was dried with oil-free compressed air for 5 sec and treated with Amalgambond Plus Activator (Parkell, Farmingdale, NY) for 10 sec, rinsed with distilled water for 15 sec and the dentin gently air-dried with oil-free compressed air. For group 8, the dentin was remoistened as done earlier with distilled water. Both groups 7 and 8 had a thin layer of Amalgambond Plus Adhesive Agent (Parkell, Farmingdale, NY) applied over the dried dentin and left undisturbed for 30 sec followed by two drops of Amalgambond Plus Base (Parkell, Farmingdale, NY) and one of Amalgambond Plus Catalyst (Parkell, Farmingdale, NY) mixed and applied over the dentin with a disposable brush and left to dry for 60 sec before placing a nylon cylinder filled with Z100 hybrid resin composite. The excess composite was removed with a dental explorer and the resin light-cured for 40 sec. Immediately, the teeth were placed in distilled water for 48 hr.

Teeth in all groups were thermocycled for 1000 cycles in temperatures of 5° and 55°C with a 30-sec dwell time.

After thermocycling, the teeth were embedded in plaster so the resin composite cylinder was perpendicular to the knife-edge rod of the Instron testing machine (Instron Corp., Canton, MA) running at a crosshead speed of 0.5 mm/min.

Failure sites of both the resin cylinder and the dentin after debonding were examined visually and selected specimens were analyzed with the scanning electron microscope (JEOL JSM-840, JEOL, Tokyo, Japan).

For each group, one additional tooth was used to prepare two class V restorations (one facial and one lingual). The cavity preparations were 2.5 mm wide and 2.0 mm deep, and due to the small amount of root remaining on these exfoliating teeth, placed 1 mm above the CEJ. The cavities were restored according to the specifications in each group. After resin placement, the restorations were finished with water-cooled carbide burs and placed in distilled water for 24 hr. The teeth were sectioned buccolingually through the center of the restorations with diamond discs. The internal aspects of the two resulting sections were etched with 10% phosphoric acid for 5 sec to remove the smear layer, rinsed with distilled water, dried with oil-free compressed air, mounted on aluminum stubs, and coated with gold-palladium for SEM examination.

An ANOVA and Student-Newman-Keuls tests were used to evaluate the shear bond strength data.

Results

Shear bond strength

No statistically significant differences were observed between the groups (ANOVA $P = 0.179$). However, there was a tendency for higher bond strengths on the specimens bonded with moist dentin (Table) as well as higher dentin cohesive failures.

The bond strength value was not related to the failure mode recorded visually or with the SEM. For example, some samples recorded values of 6.27 MPa and resin cohesive failures, while others had mean values of 12.06 MPa and adhesive/resin failures. However, dentin cohesive failures were only recorded with mean values not lower than 14.10 MPa. The Table depicts the failure site for the different groups.

SEM evaluation

A resin-reinforced hybrid layer was readily seen in all specimens. Fig 1 depicts a resin cohesive and dentin cohesive mixed failure produced using SMP, etched, and moist dentin. Fig 2 reveals a specimen treated with Amalgambond Plus, etched, and moist dentin. A resin cohesive failure is evident. In the upper portion of the figure, a resin cohesive failure very close to the dentin surface is evident with resin penetrating the dentin tubules (Fig 3).

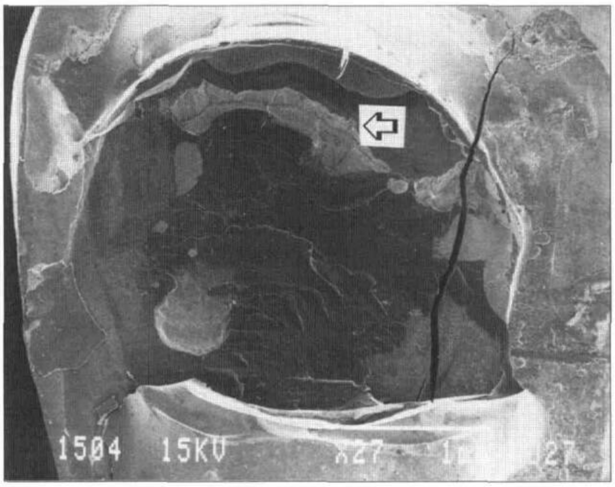

Fig 1. Resin cohesive and dentin cohesive failure (arrow; 22.53 MPa) with a specimen treated with SMP, etched and moist dentin. SEM 27x.

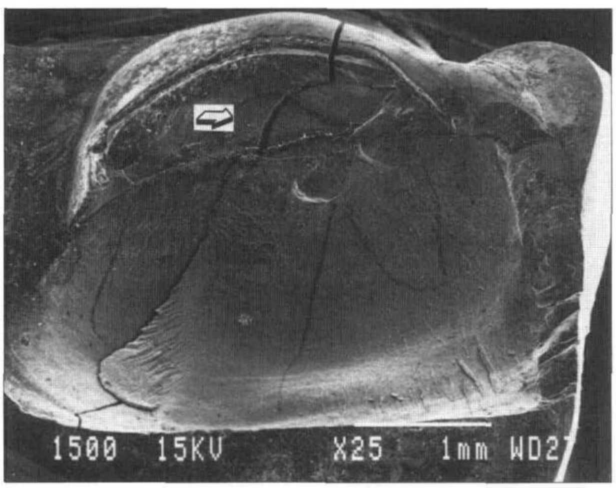

Fig 2. Resin cohesive failure (7.17 MPa) with a specimen treated with Amalgambond Plus, etched and moist dentin. In the upper portion of the figure, a resin cohesive failure very close to the dentin surface is evident (arrow). SEM 25x.

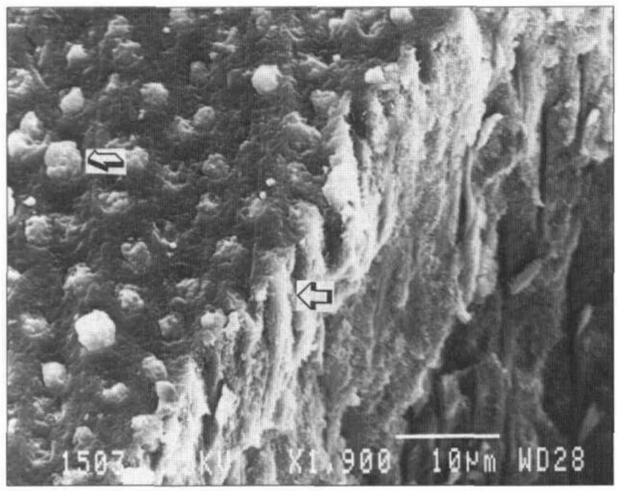

Fig 3. Higher magnification of Fig. 2. Resin failure near the dentin surface and resin penetration into the tubules is evident (arrows). SEM 1900x.

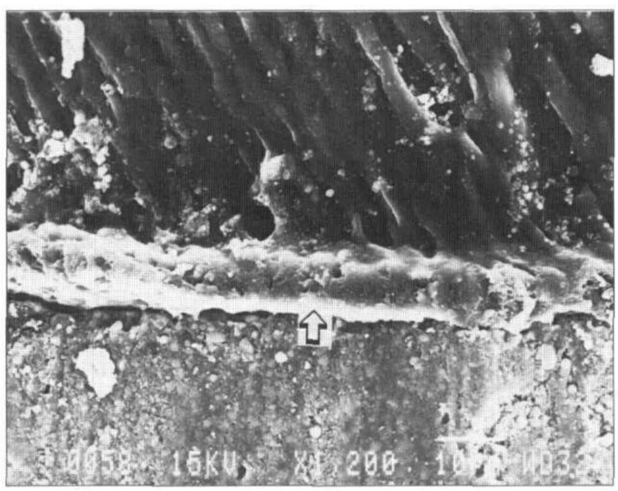

Fig 4. A hybrid layer (± 10 μm; arrow) was evident in most specimens. Specimen treated with All-Bond 2, etched and moist dentin. SEM 1200x.

Fig 4 displays a thick hybrid layer of approximately 10 µm evident with the specimens treated with All-Bond 2 regardless if the dentin was dried or moist. Similar patterns were observed with the other adhesives where the resin clearly penetrated into the dentin tubules.

Discussion

The results of our study are difficult to compare to the previous ones conducted on primary dentin, not only because of different methodologies, but also because of the different bonding agents used. However, Elkins and McCourt,[6] using All-Bond and Amalgambond, reported bond strengths of 11.60 and 12.62 MPa, respectively. Our results were very similar to these and to those of Hallett et al[11] using SMP in primary teeth enamel (11.18 MPa). The potential of these dentin bonding agents to equal or exceed the bond strength to enamel is evident in primary teeth. Another study[8] using a similar testing methodology as our study but employing Gluma 2000 adhesive system, reported a bond strength value of 8.2 MPa in primary dentin.

In this study, the bond strength value in primary dentin was not related to the failure mode recorded visually or with the SEM. For example, some specimens recorded values of 6.27 MPa and resin cohesive failures while others had mean values of 12.06 MPa and adhesive failures. However, dentin cohesive failures were only recorded with mean values not lower than 14.10 MPa.

In this study using SMP and Amalgambond, 50% of the failures were recorded as dentin cohesive failures; therefore, a higher bond strength was virtually impossible in these specimens. Malferrari et al[8], using Gluma 2000 in primary dentin, noted a similar trend: although the bond strength values were relatively low, evaluation with the SEM revealed that the true failure occurred in the resin, very near to the dentin surface. Hallett et al,[11] testing bond strength of SMP in primary enamel reported that most failures occurred at the adhesive-resin interface or were resin cohesive. The few cases they recorded as enamel-adhesive interface failures displayed some resin coating the enamel surfaces. García-Godoy and Finger,[12] using Gluma 2000 in permanent teeth, also reported that in most cases, the bond failure was cohesive in resin, close to the dentin. Due to this result they questioned the dye penetration technique for testing microleakage when using bonding agents capable of producing a hybrid layer.

With specimens treated with All-Bond 2, dry or moist dentin or etched or nonetched dentin seemed to be of no significance to obtain similar bond strength values. However, for SMP and Amalgambond, moist dentin produced a higher bond strength value although not statistically significant. Satisfactory or even enhanced adhesion to visibly moist dentin has also been demonstrated for other materials used in permanent teeth.[13–16] Research with the SMP[17, 18] and Optibond[g 18] in permanent teeth has indicated no statistical difference in shear bond strengths between specimens in which the dentin was left visibly moist and those in which it was dried.

Studies with SMP,[19] when used with the 10% maleic acid etchant provided by the manufacturers, reported bond strengths to permanent teeth dentin of 23.9 MPa. This strength was found to increase to 26.2 MPa with the substitution of a phosphoric acid etchant. Swift & Triolo[17] also noticed that, with the SMP system, bond strengths to enamel were lower than those to dentin when the 10% maleic acid etchant was used, and suggested that longer etching times or alternative etchants should be investigated. Recently, the manufacturers of SMP include phosphoric acid to substitute for the original maleic acid provided with the kit. Most recently, in vitro shear bond strengths to dentin have been tested,[20] indicating that the current generation of dentin adhesive systems approached or exceeded the theoretical threshold value to resist contraction stresses during polymerization of resin materials, with SMP, All-Bond 2, and Optibond giving values of 23.1, 21.4, and 19.7 MPa, respectively. Others have reported shear bond strengths to dentin of 22.2,[21] and 20.2 MPa[22] using Optibond.

Because in this study there was no statistically significant difference among the shear bond strength values for any of the groups and because some specimens displayed dentin cohesive failures with relatively low shear bond strength values, reporting the bond strength values per se is not as significant as describing the failure site. Future studies should focus on the failure site while debonding the resin and correlate this information with the bond strength value.

Most studies agree that the bond strength is higher in permanent teeth.[4, 23] Other studies,[11] using newer generations of bonding agents, have recorded no significant difference. When comparing the results of bond strengths obtained in primary and permanent teeth dentin, dentin thickness difference must be taken into consideration. Also, the bond strength of some dentin adhesives decreases as the occlusal dentin approaches the pulp,[24–26] which was interpreted as the bond being dependent of the calcium level of the total area of solid dentin available, both of which decrease toward the pulp.[4] However, Hirayama et al.[27] found no difference in calcium or phosphorus content of the peritubular and intertubular dentin of primary and permanent teeth, but reported that the peritubular dentin, which is more mineralized but less crystalline than intertubular dentin[28] was two to five times thicker than permanent. As Bordin-Aykroyd[4] suggests, these differences could affect any chemical bonding of the adhesive or result in different effects of the pretreatment regimens on the dentin, which would also affect bonding. On the other hand, because of the thinner primary dentin, more dentin cohesive failures could be recorded in primary teeth even with lower bond strength values.

Another interesting observation in this study was that in all specimens, a hybrid layer was readily seen.

A recent study[29] has shown that when dentin is acid-etched, the subsequent moisture status of the collagen-rich outer zone is critical to achieving optimal shear bond strength. As a result of drying, the organic rich collagen phase at the surface of conditioned dentin is altered morphologically.[30] These morphological changes impair the penetration of the primer resulting in a reduction in bond strength.[16] In our study on primary dentin and the one by Barkmeier et al.[31] on permanent dentin, the bond strength to dentin was not statistically significantly altered whether the dentin was wet or dry. Perhaps, the time needed to rehydrate the dentin should be increased before the primer is applied. This is possible because in this study, although there was no significant difference in bond strength, there was a trend to obtain higher values with the moist dentin. Because the collagen-rich zone offers no direct quantitative contribution to bond strength,[30] dentin rehydration (moist dentin) would then be mainly important to achieve a maximum porous dentin surface so the resin components can diffuse through the outermost demineralized collagen-rich zone into the partially demineralized dentin below where it must polymerize.

Conclusions

1. There was no statistically significant difference in bond strength to dentin of primary teeth with the products and techniques used in this study, although there was a tendency for higher bond strengths and higher dentin cohesive failures on the specimens bonded with moist dentin.
2. The bond strength value was not related to the failure mode recorded visually or with the SEM.
3. Dentin cohesive failures were only recorded with mean values not lower than 14.10 MPa.
4. SEM evaluation showed that the smear layer was removed in all groups exposing dentin tubules infiltrated by resin.
5. A resin-reinforced hybrid layer was readily seen in all specimens.

Dr. Araujo is associate professor, Department of Pediatric Dentistry, Faculty of Dentistry, Federal University of Rio Grande do Sul, Porto Alegre, Brazil. Dr. García-Godoy is professor, departments of Pediatric Dentistry and Restorative Dentistry, University of Texas Health Science Center at San Antonio, Texas. Dr. Issáo (deceased) was professor and chairman, Department of Pediatric Dentistry, Faculty of Dentistry, University of São Paulo, Brazil

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