Microshear Bond Strength of Resin Composite to Teeth Affected by Molar Hypomineralization Using 2 Adhesive Systems

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Abstract

Purpose: When restoring hypomineralized first permanent molars, placement of cavosurface margins can be difficult to ascertain due to uncertainty of the bonding capability of the tooth surface. The purpose of this study was to investigate the adhesion of resin composite bonded to control and hypomineralized enamel with an all-etch single-bottle adhesive or self-etching primer adhesive.

Methods: Specimens of control enamel (N=44) and hypomineralized enamel (N=45) had a 0.975-mm diameter composite rod (Filtek Supreme Universal Restorative) bonded with either 3M ESPE Single Bond or Clearfil SE Bond following manufacturers' instructions. Specimens were stressed in shear at 1 mm/min to failure (microshear bond strength). Etched enamel surfaces and enamel-adhesive interfaces were examined under scanning electron microscopy.

Results: The microshear bond strength (MPa) of resin composite bonded to hypomineralized enamel was significantly lower than for control enamel (3M ESPE Single Bond=7.08±4.90 vs 16.27±10.04; Clearfil SE Bond=10.39±7.56 vs 19.63±7.42; P=.001). Fractures were predominantly adhesive in control enamel and cohesive in hypomineralized enamel. Scotchbond etchant produced deep interprismatic and intercrystal porosity in control enamel and shallow etch patterns with minimal intercrystal porosity in hypomineralized enamel. Control enamel appeared almost unaffected by SE Primer; hypomineralized enamel showed shallow etching. The hypomineralized enamel-adhesive interface was porous with cracks in the enamel. The control enamel-adhesive interface displayed a hybrid layer of even thickness.

Conclusions: The microshear bond strength of resin composite bonded to hypomineralized enamel was significantly lower than for control enamel. This was supported by differences seen in etch patterns and at the enamel-adhesive interface. (Pediatr Dent 2006;28:233-241)

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Hypomineralized first permanent molars are at risk of posteruptive enamel breakdown (PEB) after erupting into the oral environment, where masticatory forces and dietary challenges lead to enamel chipping, dentin exposure, and early dental caries.[1] The restorative management usually depends on the defect's severity and the child's cooperation and age.[2,3] In restoring such molars with resin composite, cavosurface margins can be difficult to place due to the surface's uncertain bonding capability.

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Literature on bonding to hypomineralized enamel is limited. Case reports describing bonding to the hypomineralized enamel of amelogenesis imperfecta ascribe bonding difficulties to decreased mineral content and increased protein content.[4-6] Some authors recommend pretreating the enamel with 5% sodium hypochlorite to de-proteinate the hydroxyapatite,[6,7] and others remove defective hypomineralized enamel prior to bonding resin composite restorations.[8-11]

Limited laboratory studies may reflect the scarcity of extracted hypomineralized teeth with suitable surface areas for bonding where the enamel is free of PEB and caries. One abstract reported that bond strength of a sealant to normal enamel (19.9±5.39 MPa) significantly exceeded that of sealant to noncarious defective enamel (9.06±5.39 MPa).[12] Laboratory tests can conveniently screen new materials and techniques. Results to date suggest that the higher the bond strength of an adhesive, the better it will withstand oral stresses.[13] Care must be taken when comparing data from different laboratories due to differences in storage media, testing apparatus, specimen preparation, bonded surface area, strain rate to debond specimens, and operator skills.[14]

A method for testing microtensile bond strengths of dental adhesives to small rectangular surface areas (1.6-1.8 mm^2) of enamel and dentin was developed by Sano et al.[15] Early problems of enamel separating from the dentin and technically demanding specimen production[16] were overcome with the development of a microshear bond strength test by McDonough et al[17] and Shimada et al.[18] This simpler test is less technically demanding, reducing the incorporation of defects in the bonded assembly during specimen production. The small specimen size permits many tests to be performed on the same substrate/tooth, conserving extracted teeth and reducing variability.[17] Higher mean bond strengths have been obtained using smaller bonded surface areas. This is attributed to fewer interfacial defects and variations within the tooth.[15,19]

Several adhesives are available for bonding resin composite to enamel; the 2-step single-bottle adhesive and 2-step self-etching primer adhesive have shown consistent and successful bonding to ground enamel.[20-29] The reasons for the high failure rate of resin composites to hypomineralized enamel are unclear. With the advent of the microshear bond strength test, however, bonding to hypomineralized enamel can be evaluated, allowing evidence-based decisions regarding cavity preparation and material choice.

The purpose of this study was to investigate the adhesion of a resin composite bonded to control and hypomineralized enamel with an all-etch single-bottle adhesive (3M ESPE Single Bond, 3M ESPE, St. Paul, Minn) or a self-etching primer adhesive (Clearfil SE Bond, Kuraray Medical Inc, Tokyo, Japan), with 3 objectives:

1. investigate the microshear bond strengths of a resin composite bonded with 3M ESPE Single Bond or Clearfil SE Bond;
2. examine under scanning electron microscopy (SEM) the enamel etch patterns produced by 2 different adhesive systems:
   a. 35% phosphoric acid etch from Scotchbond etchant; or
   b. self-etching primer from Clearfil SE Bond; and
3. examine under SEM the enamel-adhesive interface following the use of these adhesives on control enamel or hypomineralized enamel.

Methods

Study sample

A total of 120 extracted, erupted first permanent molars (FPMs) from children under age 18 years were obtained from the Royal Dental Hospital of Melbourne, Australia, the Dental Department of the Royal Children's Hospital, Melbourne, Australia, and from patients in private pediatric dental practices. The study was approved by the Human Ethics Committee of each hospital, and informed consent was obtained from all parents/guardians for the use of their child's teeth in research. The FPMs were extracted due to caries or for orthodontic purposes in children with molar-incisor hypomineralization (MIH); frequently more than one FPM was extracted per child. Molars were collected over a 10-month period and stored in 10% neutral buffered formalin at room temperature.

Typical molars used in this study are shown:

1. a yellow-brown demarcated opacity from which specimens were prepared (Figure 1a); and
2. posteruption breakdown (PEB) of enamel and a failed restoration, common in MIH (Figure 1b).

Specimen preparation

Molar roots were removed perpendicular to the long axis with a 0.3-mm thick diamond blade (Struers A/S, Copenhagen, Denmark). The crowns were washed under running water and inspected visually when wet and air-dried to delineate areas of hypomineralized enamel and normal enamel. Hypomineralized enamel was defined as a visible enamel defect with a demarcated opacity.[30] The surface was smooth, cream or yellow-brown in color with a distinct boundary from adjacent normal enamel, and of apparently normal thickness[31,32] (Figure 1a). Normal enamel (hereafter termed control enamel) was defined as free from discoloration, caries, diffuse enamel opacities (eg, fluorosis), and hypoplasia.

Only teeth with at least a 2- to 3-mm diameter of control enamel or hypomineralized enamel were used. The 120 FPMs were utilized as follows:

1. microshear bond strength testing (55);
2. study of etch patterns (5);
3. study of enamel-adhesive interfaces;
4. study of enamel-adhesive interfaces (4); and
5. discarded due to insufficient enamel for study (56).

Enamel preparation for microshear bond strength test

Molar crowns were sectioned mesiodistally, perpendicular to the occlusal plane with a diamond blade, providing buccal or lingual sections. The sections were cut buccolingually, providing up to 4 specimens per tooth (control enamel total=44 and hypomineralized enamel total=56). Specimens were stored in 0.5% Chloramine-T (Sigma-Aldrich Co, St. Louis, Mo).

Specimens were ground with wet 600-grit silicon carbide paper (Struers A/S, Copenhagen, Denmark), to produce flat enamel surfaces 2 to 3 mm in diameter with a standardized surface roughness. This surface was oriented perpendicular to the enamel rods, placed in contact with a glass slab, and stabilized with sticky wax (Associated Dental Products Ltd, Wiltshire, UK). A plastic ring of 15-mm internal diameter was placed over each specimen and filled with Type III dental stone (Yellowstone, Gibling Stone, Thomastown, Victoria, Australia), to embed the specimen. Once the stone had set, the enamel surfaces were inspected and washed to ensure a clean, stone-free surface.

Bonding procedure

Two adhesives were used for bonding, following manufacturers' instructions:

1. 3M ESPE Single Bond (SB; batch no. 2GP, 3M ESPE, St. Paul, Minn) was applied to 22 control enamel specimens and 29 specimens of hypomineralized enamel; and
2. Clearfil SE Bond (SE; batch no. 309, Kuraray Medical Inc, Tokyo, Japan) was applied to 22 specimens of control enamel and 27 specimens of hypomineralized enamel.

After adhesive placement, a tube 1 mm high and of 0.975 mm internal diameter (Microtube Extensions, North Rocks, NSW, Australia) was placed on the enamel. The adhesive and tube were light cured together (XL3000 Curing Light, 3M ESPE). The tube was filled with resin composite (Filtek Supreme Universal Restorative; A3.5 Body Shade, batch no. 2AP, 3M ESPE) and light cured for 40 seconds. The tube was carefully removed with a scalpel blade, leaving a bonded resin composite rod perpendicular to the enamel. Specimens were stored in tap water at 37^0 C for 12 hours before testing.

Microshear bond strength test and failure modes

Each bonded interface was subjected to a microshear bond (µSB) test in an Instron testing machine (Model 5544 series, Instron Corp, Canton, Mass) using a blade to deliver a force parallel to the bonded surface, loaded at a crosshead speed of 1 mm/minute until failure. Group mean values were calculated. Fractured surfaces were examined under light microscopy at X20 magnification, and digital images were obtained. Failures were classified as:

1. adhesive failure at the enamel-adhesive interface;
2. cohesive failure in enamel;
3. cohesive failure in the adhesive; or
4. mixed failure (partial cohesive and partial adhesive failures).[33]

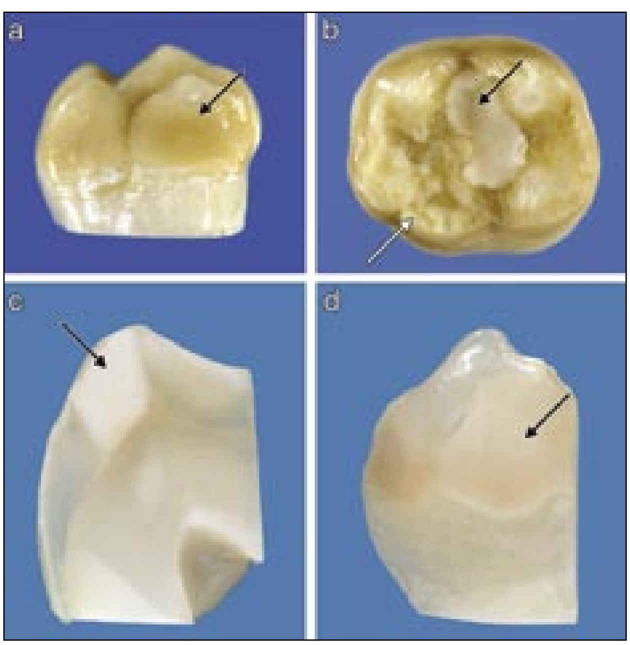

Figure 1a. Yellow-brown hypomineralized first permanent molars. Buccal view of a hypomineralized enamel defect (black arrow).

Figure 1b. Yellow-brown hypomineralized first permanent molars. Occlusal view with evidence of posteruptive enamel breakdown (white arrow) and a failed restoration (black arrow).
Figure 1c. Yellow-brown hypomineralized first permanent molars, sectioned parallel to the enamel rods.
Figure 1d. Yellow-brown hypomineralized first permanent molars, sectioned perpendicular to the enamel rods.

Preparation for SEM

The digital images were studied, and 11 representative specimens were prepared for SEM. Each specimen was:

1. retrieved from the ring;
2. air-dried for 2 days on filter paper;
3. mounted on an aluminum stub with conductive silver liquid (Pro Sci Tech, QLD, Brisbane, Australia);
4. gold sputter-coated (Gold Sputter Coater S150B, Edwards, London, UK); and
5. examined under SEM (Philips XL30 FEG, Eindhoven, The Netherlands).

Examination of etch patterns

Five teeth were sectioned mesiodistally, and buccolingually to provide 16 specimens (8 control; 8 hypomineralized) which were allocated for either Scotchbond etching (35% phosphoric acid) or SE Primer, embedded in epoxy resin (Epofix Resin, Struers A/S, Copenhagen, Denmark), and serially polished with silicon carbide papers (600, 1200, 2400 and 4000-grit; Struers A/S, Copenhagen, Denmark) to create a 2-3 mm flat surface either parallel or perpendicular to the enamel rods (Figures 1c and 1d). After applying SE Primer, specimens were placed in an ultrasonic bath for 1 minute in 70% acetone, then for 1 minute in 100% acetone, to remove the resin from the SE Primer. Specimens treated with Scotchbond etchant were washed with water from a triplex syringe and then immersed in acetone. Specimens were air-dried and prepared for observation under SEM as above.

Examination of enamel-adhesive interface

Four teeth were sectioned mesiodistally and areas of control and hypomineralized enamel were sectioned buccolingually to provide specimens free of PEB or caries. Four buccal specimens (2 control; 2 hypomineralized) were abraded with a diamond bur (100-µm grit, ISO 806314107524, Komet, Lemgo, Germany) to remove surface enamel. Either SB or SE was used to bond a 0.5-mm thick layer of resin composite to the cut surface, which was polymerized for 40 seconds, placed in tap water at 37° C for 12 hours, and embedded in epoxy resin. Specimens were sectioned buccolingually and serially polished with wet silicon carbide papers (600-, 1,200-, 2,400-, and 4,000-grit), demineralized in 6M HCl for 30 seconds, rinsed under running tap water for 60 seconds, dried, and prepared for SEM observation.

Statistical analysis

Due to unequal variances, an analysis of variance (ANOVA) test was inappropriate and the Student's *t* test was used to compare µSB values. The critical level for ∝ was 0.01 (incorporating Bonferroni's correction for multiple statistical testing of the same data). Associations between failure modes, adhesive types, and enamel types were examined using the chi-square nonparametric statistic (∝=0.05). Adhesive strength was assessed by examining adhesive failures and mixed failures.

Results

Specimen failure during preparation for the microshear bond strength test

Four of the 27 specimens in group 3 (SE=hypomineralized enamel) failed due to rod fractures on tube removal. To maintain similar group numbers, these specimens were not resurfaced or rebonded. Thirteen of the 29 group 4 specimens (SB=hypomineralized enamel) also failed and were resurfaced and rebonded. Seven of these specimens failed again, leaving 22 specimens. More preparation failures occurred with SB than SE (24% vs 15%; Table 1); these were all on hypomineralized enamel.

Microshear bond strength test

The mean bond strengths for both adhesives were greater for control enamel than hypomineralized enamel (Table 1). The bond strengths of both adhesives to control enamel did not differ significantly (SB=16.27±10.04 MPa vs SE=19.63±7.42 MPa; *P*=.216), and the bond strengths of both adhesives to hypomineralized enamel did not differ significantly (SB=7.08±4.90

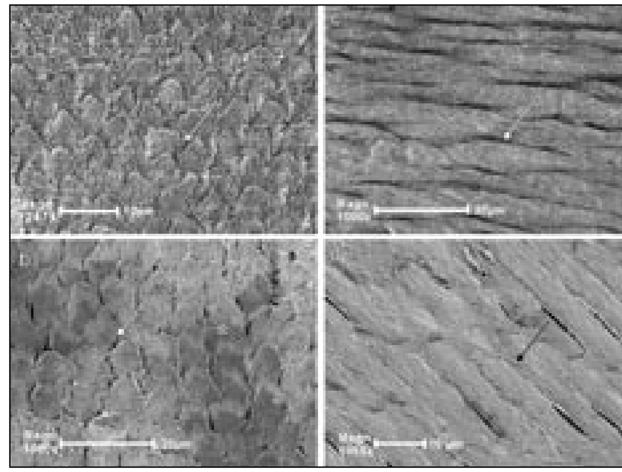

Figure 2a. Scanning electron microscope image of enamel etched with Scotchbond etchant. Control enamel sectioned perpendicular to the enamel rods, illustrating preferential inter-rod enamel dissolution (white arrow); and intercrystal porosity (white asterisk); scale=10µm.
Figure 2b. Scanning electron microscope image of enamel etched with Scotchbond etchant. Hypomineralized enamel sectioned perpendicular to the enamel rods, illustrating preferential inter-rod dissolution (white arrow) and poor intercrystal porosity (white asterisk); scale=20µm.
Figure 2c. Scanning electron microscope image of enamel etched with Scotchbond etchant. Control enamel sectioned parallel to enamel rods, illustrating preferential inter-rod enamel dissolution (white arrow); scale=20µm.
Figure 2d. Scanning electron microscope image of enamel etched with Scotchbond etchant. Hypomineralized enamel sectioned parallel to enamel rods, illustrating poor enamel etching with little preferential removal of inter-rod enamel (black arrow); scale=10µm.

Table 1. Microshear Bond Strength and Failure Modes of Resin Composite Bonded to Control Enamel or Hypomineralized Enamel*

Measure	Control enamel: 3M Single Bond (n=22)	Control enamel: Clearfil SE Bond (n=22)	Hypomineralized enamel: 3M Single Bond (n=29)	Hypomineralized enamel: Clearfil SE Bond (n=27)
No. of specimens failed during specimen preparation (%)	0 (0%)	0 (0%)	7 (24%)	4 (15%)
No. of specimens tested	22	22	22	23
Mean microshear bond strength (MPa) (±SD)†	16.27±10.04‡	19.63±7.42§	7.08±4.90‡	10.39±7.56§
Adhesive failure (%)	10 (45)a	16 (73)a	3 (14)b	5 (22)b
Cohesive failure in enamel (%)	0 (0)a	0 (0)a	11 (50)b	12 (52)b
Cohesive failure in resin adhesive (%)	3 (14)a	1 (4)a	5 (23)b	3 (13)b
Mixed failure (%)	9 (41)a	5 (23)a	3 (14)b	3 (13)b

*Same letter superscripts denote columns with no association between failure mode and adhesive type (chi-square=4.0; df=3; *P*=.26). Different letter superscripts denote columns where the distribution of failure modes between control and hypomineralized enamel differed significantly (chi-square=37.1; df=3; *P*<.001).
†SD=standard deviation.
‡Significant difference (*P*=.001) between the microshear bond strength of composite bonded to control enamel vs hypomineralized enamel using SB adhesive.
§Significant difference (*P*<.001) between the microshear bond strength of composite bonded to control enamel vs hypomineralized enamel using SE Bond adhesive.

MPa vs SE=10.39±7.56 MPa; *P*=.088). The bond strengths differed significantly between enamel substrates for SB (control=16.27±10.04 MPa vs hypomineralized=7.08±4.90 MPa; *P*=.001), and for SE (control=19.63±7.42 MPa vs hypomineralized=10.39±7.56 MPa; *P*<.001).

Failure modes

Failures in control enamel were predominantly adhesive (SB=46%; SE=73%) or mixed (SB=41%; SE=23%; Table 1). No enamel cohesive failures were seen in either group. Failures in hypomineralized enamel were predominantly cohesive in enamel (SB=50%; SE=52%). All failure types, however, were noted in both hypomineralized groups. The distribution of failure modes between control and hypomineralized enamel differed significantly (chi-square=37.06; df=3; *P*<.001). There was no association between failure modes and adhesive type (chi-square=4.048; df=3; *P*=.256).

Adhesive strength of Clearfil SE Bond vs 3M Single Bond

Cohesive failures in enamel or adhesive reflect the direction of crack propagation and not the "true" adhesive strength. Therefore, the µSB values were re-examined, excluding specimens that failed cohesively. The mean bond strength values for both adhesives were then greater for control enamel than hypomineralized enamel. The mean values for resin composite bonded to control enamel did not differ significantly between SE and SB (20.07±7.31 MPa vs 17.31±10.45 MPa; *P*=.30). When bonded to hypomineralized enamel, the strength of SE adhesive was approximately double that of SB adhesive (10.65±4.01 MPa vs 5.23±3.77 MPa; *P*=.025). Although sample sizes were small (SE: N=8; SB: N=6) and the bond strengths did not differ significantly; SB's lower adhesive strength than SE corroborated the higher failure rate in specimen preparation (SB=13/29, 45% vs SE=4/27, 15%).

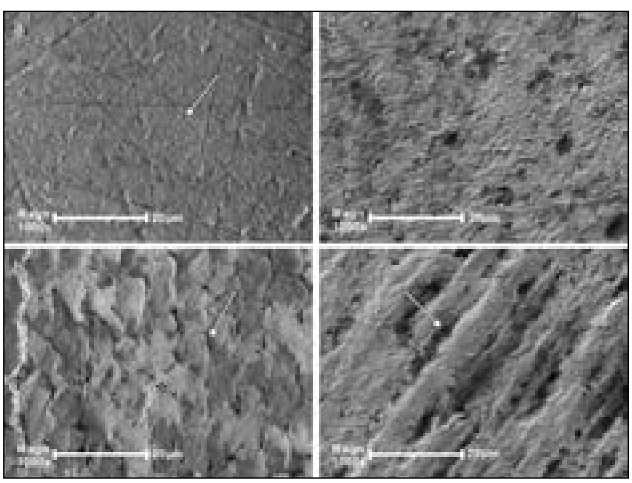

Figure 3a. Scanning electron microscope image of enamel etched with Clearfil SE Bond Primer. Control enamel sectioned perpendicular to enamel rods, illustrating minimal interaction with the enamel. Evidence of scratch marks associated with the 600-grit silicon carbide paper (white arrow); scale=20µm.
Figure 3b. Scanning electron microscope image of enamel etched with Clearfil SE Bond Primer. Hypomineralized enamel sectioned perpendicular to enamel rods, illustrating uneven, mild dissolution of inter-rod enamel (white arrow) and a precipitate on the surface (black asterisk); scale=20 µm.
Figure 3c. Scanning electron microscope image of enamel etched with Clearfil SE Bond Primer. Control enamel sectioned parallel to enamel rods, illustrating minimal interaction with the enamel; scale=20µm.
Figure 3d. Scanning electron microscope image of enamel etched with Clearfil SE Bond Primer. Hypomineralized enamel sectioned parallel to enamel rods, illustrating uneven, mild dissolution of inter-rod enamel (white arrow); scale=20µm.

Etch pattern examination

Control enamel sectioned perpendicular to the enamel rods and etched with SB etchant showed uniform preferential dissolution of rod peripheries, intrarod etching, and increased intercrystal porosity (Figure 2a). Hypomineralized enamel prepared similarly showed preferential dissolution of rod peripheries and loss of inter-rod enamel resulting in enlarged inter-rod spaces; intercrystal porosity was minimal, possibly reducing the surface area available for bonding (Figure 2b). Control enamel sectioned parallel to the enamel rods and etched with SB etchant showed preferential dissolution of inter-rod enamel (Figure 2c), whereas hypomineralized enamel prepared similarly showed little preferential removal of inter-rod enamel and little increase in surface area (Figure 2d).

Figures 3a and 3b represent control and hypomineralized enamel, respectively, sectioned perpendicular to the enamel rods and etched with SE Primer. Control enamel showed little surface etching; the slight surface roughness and scratches were attributed to the silicon carbide paper (Figure 3a). Hypomineralized enamel showed uneven, preferential dissolution of peripheral inter-rod areas, increasing the surface area available for bonding; some enamel crystals showed an adherent precipitate (Figure 3b). Figures 3c and 3d show control and hypomineralized enamel, respectively, sectioned parallel to the enamel rods and etched with SE Primer. The etch patterns were similar to those in Figures 3a and 3b; the hypomineralized enamel appeared to have greater surface area than the control enamel, which showed only a rough surface.

Enamel-adhesive interfaces

The enamel-adhesive interfaces of hypomineralized enamel bonded with SB differed markedly from control enamel. The hypomineralized enamel was porous with cracks, whereas control enamel showed a uniformly thick hybrid layer (Figures 4a and 4b). Unlike control enamel, selective dissolution of inter-rod hypomineralized enamel was absent. Control enamel bonded with SE showed preferential inter-rod dissolution (Figure 4c), while hypomineralized enamel also showed porosity and enamel cracks (Figure 4d).

Discussion

Microshear bond strength test

The microshear bond strength test measures the bond strength of resin composite to ground enamel surfaces using adhesive systems. A recent abstract reported a mean bond strength (16.90±5.39 MPa) for fissure sealant to normal enamel, significantly exceeding that of the sealant to non-carious but defective enamel (9.06±5.39 MPa).[12] Although sealant type, specimen preparation, and bond test were not given, these values approximate the values obtained in the current study.

The microshear bond strength values of the 2 adhesives did not differ significantly on control enamel, confirming other reports.[22,23,29,34] Lopes et al[29] demonstrated similar bond strengths for SE (17.6±4.5 MPa) and SB (17.9±4.4 MPa) for resin composite bonded to bovine enamel. Although approximating the present values, data from different laboratories should be compared cautiously due to differences in specimens, substrates, storage media, specimen preparation, and techniques used.[14]

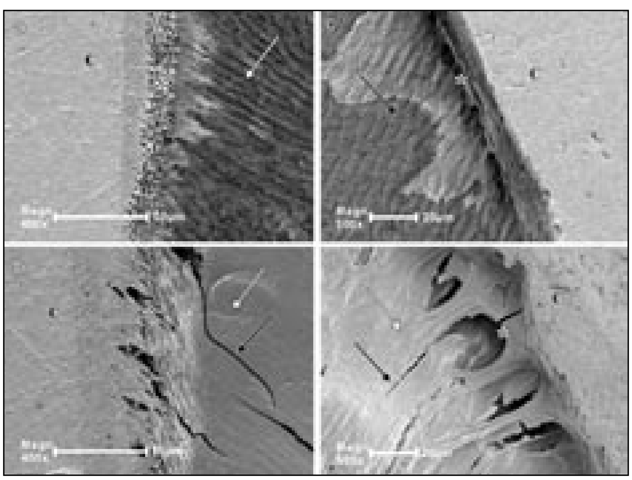

Figure 4a. SEM of bonded enamel using Single Bond or Clearfil SE Bond illustrating composite (C) and the adhesive interface (white asterisk). Control enamel bonded with Single Bond, demonstrating preferential inter-rod dissolution of the enamel (white arrow) and a uniform hybrid layer (white asterisk); scale=50µm.
Figure 4b. SEM of bonded enamel using Single Bond or Clearfil SE Bond illustrating composite (C) and the adhesive interface (white asterisk). Hypomineralized enamel bonded with Single Bond, demonstrating lack of preferential dissolution of the enamel (white arrow) and enamel cracks extending from the adhesive interface (black arrow); scale=50µm.
Figure 4c. SEM of bonded enamel using Single Bond or Clearfil SE Bond illustrating composite (C) and the adhesive interface (white asterisk). Control enamel bonded with Clearfil SE Bond, demonstrating preferential inter-rod dissolution of the enamel (black arrow); scale=20µm.
Figure 4d. SEM of bonded enamel using Single Bond or Clearfil SE Bond illustrating composite (C) and the adhesive interface (white asterisk). Hypomineralized enamel bonded with Clearfil SE Bond, demonstrating minimal preferential dissolution of the enamel (white arrow) and enamel cracks extending from the adhesive interface (black arrow); scale=20µm.

Studies by Shimada et al[22,23] and Wang et al[34] also found the microshear bond strengths of SE and SB to be similar when bonded to enamel, noting higher values (35-43 MPa). The differences from the present values may reflect enamel surface preparation with:

1. coarser silicon carbide paper (280-grit vs 600-grit);
2. smaller diameter rods (0.80 mm vs 0.975 mm); and
3. different shear test method (wire loop vs blade).

The present bond test was conducted at 12 hours (vs 24 hours), when a "maximum" bond strength may not have been achieved. Past studies, however, have shown little change in bond strength after about 10 minutes for most adhesives.[35,36] The lower microshear bond strength values noted may also reflect tooth storage (up to 10 months in 10% neutral-buffered formalin) and the mineral content of control enamel from MIH-affected molars. These molars may have 5% less mineral and lower calcium/phosphorous ratios than enamel from unaffected patients.[37,38] To reduce variation in the present study, the same molars were used for both control and hypomineralized specimens.

The wide standard deviations noted may reflect enamel differences within specimens and between teeth. Hypomineralized areas can vary within a tooth, and color differences have been associated with variations in porosity and hardness. For example, calcium:phosphorous ratios decrease as the hypomineralization worsens in appearance,[38] and white defects are harder and less porous than yellow defects.[39,40] In selecting enamel for the present study, no color distinctions were made due to the subjectivity of color classifications and the few molars available. The wide standard deviations may also reflect bonding to microscopic interfacial defects in hypomineralized enamel, and difficulty in applying only a shear stress to the bonded interface.

The present study assumed that the hypomineralization defects involved the full enamel thickness; this may not always occur.[30,31,34] One very high value (33.92 MPa) was noted for resin composite bonded to hypomineralized enamel with SE Bond. It is suggested that the rod was bonded to superficial, fully mineralized enamel overlying deeper hypomineralized enamel located closer to the dentinoenamel junction.

Failure modes

The failure modes of control enamel bonded with either SB or SE adhesive were predominantly adhesive and mixed failures, supporting other reports.[22,23,34] In contrast, the dominant failure mode for bonded hypomineralized enamel was cohesive failure within enamel. Although no other studies appear to have examined failures of bonded hypomineralized enamel, such cohesive failures have been observed in enamel bonded parallel to enamel rods.[41-43] The overall structure of hypomineralized enamel is not as well organized as normal enamel. The high frequency of cohesive failure in hypomineralized enamel indicates its inherent weakness.

Adhesive strength of Clearfil SE Bond vs 3M Single Bond

When examining the bond strengths of specimens that failed either adhesively or by mixed failure, SE appeared to bond to hypomineralized enamel better than SB (10.65±4.01MPa vs 5.23±3.77 MPa; *P*=.025), consistent with the distribution of preparation failures where 45% (13/29) of hypomineralized specimens bonded with SB failed, and 15% (4/27) of those bonded with SE failed. Extrapolating from studies on bonding to deep dentin—where self-etching adhesives exhibited higher bond strengths than all-etch single-bottle adhesives[44,45]—it is suggested that lower bond strengths of the latter adhesives reflect intrinsic moisture within the porous enamel lattice and extrinsic moisture from rinsing. This results in overwet conditions that inhibit resin infiltration and dilute the water-soluble primer, thus lowering the bond strength. For the self-etching primer adhesive, 2 factors may promote its bonding to hypomineralized enamel:

1. rinsing is omitted, thus eliminating the interference of residual water on the bond; and
2. SE bonds both micromechanically and chemically to hydroxyapatite,[46] whereas the all-etch adhesive SB relies primarily on micromechanical retention, which may be limited as shown in Figures 2b and 2d.

Etch patterns

Enamel bonding relies on resin tag formation, where micromechanical retention is a function of the surface area and surface energy of the etched enamel.[47] The characteristic features of phosphoric acid-etched enamel are spicular enamel rods with inter-rod and intercrystal porosity, which increase surface area.[48] Following resin infiltration and polymerization, macro-tags form circumferentially around the enamel prisms and micro-tags form within the prism cores between the crystals, contributing most to total bond strength.[20,49,50] In the present study of control enamel, the etch patterns produced by phosphoric acid on perpendicularly sectioned enamel rods showed preferential dissolution of interprismatic enamel and prismatic etching, creating intercrystal porosities. In contrast, intercrystal porosity and micro-tag formation were minimal after etching hypomineralized enamel, perhaps explaining the lower adhesion of SB. The larger interprismatic spaces in hypomineralized enamel may promote moisture retention and structural weaknesses, allowing crack propagation.

Phosphoric acid etching of control enamel sectioned parallel to the enamel rods showed preferential inter-rod dissolution and prism etching, demonstrated by a coarse prism appearance. The increase in surface area appeared less than for enamel sectioned perpendicular to the enamel rods. Phosphoric acid etching of hypomineralized enamel did not preferentially dissolve inter-rod enamel and increase surface area, perhaps explaining the limited ability of the all-etch adhesive SB to bond to hypomineralized enamel sectioned parallel to the rods. Although not studied, others have found that the bond strength of phosphoric acid-etched enamel sectioned parallel to enamel rods is lower than for perpendicularly-sectioned rods. Hence, it is recommended to bevel enamel margins in cavities prepared for resin composite.[41-43]

Although the surface area on hypomineralized enamel appeared to increase more than on control enamel, irregular etch patterns were created by SE Primer. This minimal interaction was attributed to insufficient resin removal. These surface differences, however, did not affect bond strength values, where significantly higher strengths were achieved for control enamel than hypomineralized enamel. In addition to micromechanical retention, SE's adhesion may depend on the hydration and surface energy of porous hypomineralized enamel, the wetting ability of the primer and adhesive, and the enamel's chemical composition. A recent study noted that the 10-methacryloyloxydecyldihydrogen phosphate (MDP) in SE Primer forms a stable calcium salt within the hydroxyapatite lattice.[46] Hypomineralized enamel is lower in calcium content than control enamel,[38] perhaps reducing chemical adhesion and bond strengths.

Enamel-adhesive interfaces

Studying the enamel-adhesive interfaces highlighted the fragile nature of the bond to hypomineralized enamel. The interfacial porosities and enamel cracks may explain the many enamel cohesive failures (SE=52%; SB=50%), which were not evident in bonded control enamel. The cracks were observed following drying of the protein in hypomineralized enamel during SEM preparation.

The present findings indicate that bonding to hypomineralized enamel is less effective than bonding to "normal" enamel. Although both adhesives showed low microshear bond strength values, the all-etch technique may be more detrimental to bonding clinically, since moisture incorporation in porous hypomineralized enamel during rinsing may limit resin infiltration. This study stands as the first to explore the effect of self-etching adhesives on hypomineralized enamel. Since different self-etch adhesives may vary in acidity (pH), other self-etch adhesives need to be investigated. It is recommended that the clinical success of a resin composite restoration in a hypomineralized molar may be improved by:

1. removing all discolored hypomineralized enamel;
2. placing the cavity margins on apparently normal enamel; and
3. bonding with a self-etching primer adhesive.

Conclusions

Based on the results of this laboratory study of the adhesion of resin composite bonded to control and hypomineralized enamel with an all-etch single- bottle adhesive or a self-etching primer adhesive, the following conclusions can be made:

1. The microshear bond strength of resin composite bonded to hypomineralized enamel was significantly lower than control enamel.
2. The all-etch single-bottle adhesive and the self-etching primer adhesive did not differ significantly in their ability to bond to either enamel substrate.

3. The limited bonding to hypomineralized enamel of the all-etch single-bottle adhesive was attributed to inadequate micro-tag formation, consequential to the formation of little intercrystal porosity.
4. Although the self-etching primer adhesive etched hypomineralized enamel deeper than control enamel, the low microshear bond strength values suggest that factors other than micromechanical are involved in adhesion.
5. A high frequency of cohesive failures occurred when resin composite was bonded to hypomineralized enamel, reflecting the weakened structure of this form of enamel.

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References

1. Weerheijm KL, Jalevik B, Alaluusua S. Molar-incisor hypomineralization. Caries Res 2001;35:390-391.
2. Fayle SA. Molar incisor hypomineralization: Restorative management. Eur J Paediatr Dent 2003; 4:121-126.
3. Mahoney EK. The treatment of localized hypoplastic and hypomineralized defects in first permanent molars. N Z Dent J 2001;97:101-105.
4. Wright JT, Deaton TG, Hall KI, Yamauchi M. The mineral and protein content of enamel in amelogenesis imperfecta. Connect Tissue Res 1995; 32:247-252.
5. Takagi Y, Fujita H, Katano H, Shimokawa H, Kuroda T. Immunochemical and biochemical characteristics of enamel proteins in hypocalcified amelogenesis imperfecta. Oral Surg Oral Med Oral Pathol Oral Radiol Endod 1998;85:424-430.
6. Wright JT. The etch-bleach-seal technique for managing stained enamel defects in young permanent incisors. Pediatr Dent 2002;24:249-252.
7. Venezie RD, Vadiakas G, Christensen JR, Wright JT. Enamel pretreatment with sodium hypochlorite to enhance bonding in hypocalcified amelogenesis imperfecta: Case report and SEM analysis. Pediatr Dent 1994;16:433-436.
8. Li RW. Adhesive solutions: Report of a case using multiple adhesive techniques in the management of enamel hypoplasia. Dent Update 1999;26:277-282, 284, 286-287.
9. Yip HK, Smales RJ. Oral rehabilitation of young adults with amelogenesis imperfecta. Int J Prosthodont 2003;16:345-349.
10. Hunter L, Stone D. Supraoccluding cobalt-chrome onlays in the management of amelogenesis imperfecta in children: 12-year case report. Quintessence Int 1997;28:15-19.
11. Lygidakis NA, Chaliasou A, Siounas G. Evaluation of composite restorations in hypomineralized permanent molars: A four-year clinical study. Eur J Paediatr Dent 2003;4:143-148.
12. Whigham R, Salama F, Barenie J, Fairhurst C, Hanes C. Sealant bond strengths on dysplastic molars. Pediatr Dent 1989;11:246-247.
13. Van Meerbeek B, De Munck J, Yoshida Y, et al. Adhesion to enamel and dentin: Current status and future challenges. Oper Dent 2003;28:215-235.
14. Watanabe LG, Marshall GW, Marshall SJ. Variables influence on shear bond strength testing to dentin. Tagami J, Toledano M, Prati C, eds. *Advanced Adhesive Dentistry Proceedings Book of the Granada 3rd International Kuraray Symposium: December 3-4 1999*. Cirimido, Spain: Kuraray Co, Ltd; 2000:75-90.
15. Sano H, Shono T, Sonoda H, et al. Relationship between surface area for adhesion and tensile bond strength: Evaluation of a micro-tensile bond test. Dent Mater 1994;10:236-240.
16. Pashley DH, Sano H, Ciucchi B, Yoshiyama M, Carvalho R. Adhesion testing of dentin bonding agents: A review. Dent Mater 1995;11:117-125.
17. McDonough WG, Antonucci JM, He J, et al. A microshear test to measure bond strengths of dentin-polymer interfaces. Biomaterials 2002;23:3603-3608.
18. Shimada Y, Antonucci JM, Schumacher GE, McDonough WG, Tagami J. Effects of regional tooth structure and sectioning orientation on microshear bond strength. Tagami J, Toledano M, Prati C, eds. *Advanced Adhesive Dentistry Proceedings Book of the Granada 3rd International Kuraray Symposium: December 3-4 1999*. Cirimido, Spain: Kuraray Co, Ltd; 2000:91-103.
19. Phrukkanon S, Burrow MF, Tyas MJ. The influence of cross-sectional shape and surface area on the microtensile bond test. Dent Mater 1998;14:212-221.
20. Inoue S, Vargas M, Abe Y, et al. Microtensile bond strength of 11 contemporary adhesive to enamel. Am J Dent 2003;16:329-334.
21. Kanemura N, Sano H, Tagami J. Tensile bond strength to and SEM evaluation of ground and intact enamel surfaces. J Dent 1999;27:523-530.
22. Shimada Y, Senawongse P, Harnirattisai C, Burrow MF, Nakaoki Y, Tagami J. Bond strength of two adhesive systems to primary and permanent enamel. Oper Dent 2002;27:403-409.
23. Shimada Y, Iwamoto N, Kawashima M, Burrow JF, Tagami J. Shear bond strength of current adhesive systems to enamel, dentin and dentin-enamel junction region. Oper Dent 2003;28:585-590.

24. Hannig M, Reinhardt KJ, Bott B. Self-etching primer vs phosphoric acid: An alternative concept for composite-to-enamel bonding. Oper Dent 1999;24:172-180.
25. Reifeis PE, Cochran MA, Moore BK. An in vitro shear bond strength study of enamel/dentin bonding systems on enamel. Oper Dent 1995;20:174-179.
26. Toledano M, Osorio R, De Leonardi G, Rosales-Leal JI, Ceballos L, Cabrerizo-Vilchez MA. Influence of self-etching primer on the resin adhesion to enamel and dentin. Am J Dent 2001;14:205-210.
27. Perdigao J, Lopes L, Lambrechts P, Leitao J, Van Meerbeek B, Vanherle G. Effects of a self-etching primer on enamel shear bond strengths and SEM morphology. Am J Dent 1997;10:141-146.
28. Hashimoto M, Ohno H, Yoshida E, et al. Resin-enamel bonds made with self-etching primers on ground enamel. Eur J Oral Sci 2003;111:447-453.
29. Lopes GC, Marson FC, Vieira LC, de Andrada MA, Baratieri LN. Composite bond strength to enamel with self-etching primers. Oper Dent 2004;29:424-429.
30. Weerheijm KL. Molar incisor hypomineralisation (MIH). Eur J Paediatr Dent 2003;4:115-120.
31. Commission on Oral Health Research and Epidemiology. A review of the developmental defects of enamel index (DDE Index). Commission on Oral Health, Research, and Epidemiology. Report of an FDI Working Group. Int Dent J 1992;42:411-426.
32. Weerheijm KL, Duggal M, Mejare I, et al. Judgement criteria for molar incisor hypomineralisation (MIH) in epidemiologic studies: A summary of the European meeting on MIH held in Athens, 2003. Eur J Paediatr Dent 2003;4:110-113.
33. Burke FJT, Shortall ACC, Combe EC, Aitchison TC. Assessing restorative dental materials: 1. Test methods and assessment of results. Dent Update 2002; 29:188-194.
34. Wang H, Shimada Y, Tagami J. Shear bond stability of current adhesive systems to enamel. Oper Dent 2004;29:168-175.
35. Burrow MF, Tagami J, Negishi T, Nikaido T, Hosoda H. Early tensile bond strengths of several enamel and dentin bonding systems. J Dent Res 1994;73:522-528.
36. Price RD, Hall GC. In vitro comparison of 10-minute vs 24-hour shear bond strengths of six dentin bonding systems. Quintessence Int 1999;30:122-134.
37. Fearne J, Anderson P, Davis GR. 3D X-ray microscopic study of the extent of variations in enamel density in first permanent molars with idiopathic enamel hypomineralisation. Br Dent J 2004;196:634-638.
38. Jalevik B, Odelius H, Dietz W, Noren JG. Secondary ion mass spectrometry and X-ray microanalysis of hypomineralized enamel in human permanent first molars. Arch Oral Biol 2001;46:239-247.
39. Jalevik B, Noren JG. Enamel hypomineralization of permanent first molars: A morphological study and survey of possible aetiological factors. Int J Paediatr Dent 2000;10:278-289.
40. Suckling GW, Nelson DG, Patel MJ. Macroscopic and scanning electron microscopic appearance and hardness values of developmental defects in human permanent tooth enamel. Adv Dent Res 1989;3:219-233.
41. Ikeda T, Uno S, Tanaka T, Kawakami S, Komatsu H, Sano H. Relation of enamel prism orientation to microtensile bond strength. Am J Dent 2002;15:109-113.
42. Shimada Y, Tagami J. Effects of regional enamel and prism orientation on resin bonding. Oper Dent 2003;28:20-27.
43. Munechika T, Suzuki K, Nishiyama M, Ohashi M, Horie K. A comparison of the tensile bond strengths of composite resins to longitudinal and transverse sections of enamel prisms in human teeth. J Dent Res 1984;63:1079-1082.
44. Sattabanasuk V, Shimada Y, Tagami J. The bond of resin to different dentin surface characteristics. Oper Dent 2004;29:333-341.
45. Pereira PNR, Okuda M, Sano H, Yoshikawa T, Burrow MF, Tagami J. Effect of intrinsic wetness and regional difference on dentin bond strength. Dent Mater 1999;15:46-53.
46. Yoshida Y, Nagakane K, Fukuda R, et al. Comparative study of adhesive performance of functional monomers. J Dent Res 2004;83:454-458.
47. Reis AF, Oliveira MT, Giannini M, De Goes MF, Rueggeberg. The effect of organic solvents on one-bottle adhesives' bond strength to enamel and dentin. Oper Dent 2003;28:700-706.
48. Goodman BD, Gwinnett AJ. A comparison of laser- and acid-etched human enamel using scanning electron microscopy. Arch Oral Biol 1977;22:215-220.
49. Peumans M, Van Meerbeek B, Yoshida Y, Lambrechts P, Vanherle G. Porcelain veneers bonded to tooth structure: An ultra-morphological FE-SEM examination of the adhesive interface. Dent Mater 1999;15:105-119.
50. Shinchi MJ, Soma K, Nakabayashi N. The effect of phosphoric acid concentration on resin tag length and bond strength of a photo-cured resin to acid-etched enamel. Dent Mater 2000;16:324-329.