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Bond Strength of Self-Adhesive Restorative Materials Affected by Smear Layer Thickness but not Dentin Desiccation

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Purpose: To use shear bond strength (SBS) testing to determine the effect of surface moisture and smear layer thickness on the adhesion of self-adhesive restorative materials and a universal adhesive.

Materials and Methods: One single-step self-etch universal adhesive, Prime & Bond Active (PA), was used to bond Ceram.x Spectra ST HV composite resin to dentin and enamel using the self-etching technique. Three commercially available restorative materials and one newly developed material with self-adhesive properties, Activa (A), Fuji II LC(F), Equia Forte (E), and ASAR-MP4 (S), respectively, were also bonded to enamel and dentin prepared moist and dry and to dentin prepared with a thick smear layer. Shear bond testing was performed using an Ultradent bonding apparatus.

Results: The universal adhesive generated the highest SBS to dentin and enamel, followed by the newly developed material. None of the materials tested were significantly affected by the moisture conditions on enamel or dentin. The thickness of smear layer significantly affected SBS to dentin for S, F, and E. However, S and F still exhibited higher shear bond strength to dentin with the thicker smear layer compared to the other self-adhesive materials. Only the universal adhesive in self-etch mode was not affected by the thicker smear layer and maintained significantly higher SBS.

Conclusion: None of the materials tested were affected by bonding to overdried dentin or enamel. All of the self-adhesive materials exhibited lower SBS to specimens with a thicker smear layer. The newly developed material ASAR-MP4 compared favorably to the other self-adhesive materials tested under all test conditions.

Keywords: adhesion to dental hard tissues, shear bond testing, glass ionomers, self-adhesive restoratives.

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A durable interface between a restorative material and mineralized tooth structure is essential for long-term clinical success of the restoration.[7] Adhesion of a restorative material can be mediated with a dental adhesive or by employing a "self-adhesive" restorative material such as a glass ionomer, resin-modified glass ionomer or self-adhesive resin composite. Self-adhesive composite cements and restoratives have been introduced to simplify the placement procedure and thus reduce the time of application and technique sensitivity.[1,3] Using these materials in clinical situations where isolation is difficult may provide great advantages to the dentist in placing a high quality long-lasting restoration. The use of these materials may provide for adequate adhesion to mineralized tooth structure in clinical situations where moisture control and isolation is difficult.[6] While the selective enamel-etch technique made possible with so-called universal adhesives might be considered the best means of providing high bonding performance to both enamel and dentin,[2] even these materials may not allow sufficient time to navigate the enamel conditioning procedure (etch-and-rinse) without risking contamination of the bonding interface in some clinical situations.

Glass-ionomer and resin-modified glass-ionomer materials have been shown to generate sufficient adhesion to tooth structure to exhibit high retention rates in nonretentive cervical lesions.[13,14,17] The inherent mechanisms involved in promoting adhesion with these self-adhesive materials are based on the diffusion of polyalkenoic acid and other acidic monomers into the softened substrate and on the formation of ionic bonds with the mineralized components of the substrate surface.[19] As these materials involve an acid-base reaction within an aqueous environment,

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Table 1 Universal adhesives

Adhesive	Manufacturer	Main components	Code
Prime & Bond active Lot: 1712000006	Dentsply Sirona; Konstanz, Germany	Phosphoric acid modified acrylate resin, multifunctional acrylate, bifunctional acrylate, acidic acrylate, isopropanol, water, initiator; stabilizer	PA
Resin Composite			
Ceram.x Spectra ST HV Lot No. 1711001048	Dentsply Sirona	Barium-aluminium-borosilicate glass, methacrylate functionalized silicone dioxide nano filler, methacrylate modified polysiloxane, dimethacrylate resin, ethyl-4-(dimethylamino)benzoate, fluorescent pigment, UV stabilizer, stabilizer, camphorquinone, titanium oxide pigments, aluminium silicate pigments	

Table 2 Self-adhesive restorative materials

Material	Manufacturer	Main components	Code
Experimental Material ASAR-MP4 Lot No. 1711004202	Dentsply Sirona	Aluminum-phosphor-strontium-sodium-fluoro-silicate glass, water, highly dispersed silicon dioxide, acrylic acid, polycarboxylic acid, ytterbium fluoride, bifunctional acrylate, self cure initiator, iron oxide pigments, barium sulfate pigment, manganese pigment, camphorquinone (photoinitiator), stabilizer	S
Fuji II LC Lot No. 1707132	GC; Tokyo, Japan	Fluoro-alumino-silicate glass, water, polyacrylic acid, HEMA, urethane dimethacrylate	F
Equia Forte Lot No. 170807A	GC	Fluoro-alumino-silicate glass, water, polyacrylic acid, polybasic carboxylic acid, camphorquinone (photoinitiator)	E
Activa Lot No. 171102	Pulpdent; Watertown, MA, USA	Bioactive glass, silica, diurethane modified with hydrogenated polybutadiene, methacrylate monomers, modified polyacrylic acid, sodiumfluoride, camphorquinone (photoinitiator)	A

the amount of surface moisture can be critical in promoting successful bonding.[4] Most manufacturers suggest that desiccation of the substrate be avoided for promoting better adhesion, but often the instructions for achieving ideal surface moisture are too vague to be clinically useful.[9]

The smear layer produced by cutting instruments on tooth structure can also have an influence on the adhesive properties of self-adhesive materials. Although the smear layer thickness does not appear to significantly influence adhesion with self-etching adhesives,[15] there is evidence that resin-based self-adhesive cements generate lower adhesion values to thicker smear layers.[5] Unfortunately, there has been a relatively limited number of investigations on the adhesive performance of self-adhesive materials. Shear bond strength testing can be a useful tool to investigate the effects of different substrate conditions and application techniques, giving useful guidance on the handling of these materials.[8,10-12,15]

The purpose of this laboratory study was to investigate the effect of two moisture conditions on shear bond strength to enamel and dentin of one universal adhesive and four self-adhesive restorative materials. One of these materials, ASAR-MP4, is a newly developed restorative described as a self-adhesive composite hybrid. In addition, the effect of the thickness of the smear layer on dentin bonding with these materials will be evaluated. The null hypotheses tested were 1) there are no differences in shear bond strength (SBS) to dentin and enamel among the materials tested; 2) there are no differences in SBS between the moist and dry surface conditions for a given material; and 3) there are no differences in shear bond strength to dentin between the two smear layer conditions.

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MATERIALS AND METHODS

Study Materials

The universal adhesive materials used in this study are shown in Table 1. A single-step self-etch universal adhesive was used, Prime & Bond Active (PA) (Dentsply Sirona; Konstanz, Germany). The resin composite used was Ceram.x Spectra ST HV (Dentsply Sirona). The self-adhesive materials used in this study are shown in Table 2. These materials included: 1. Fuji II LC (F) (GC; Tokyo, Japan); 2. Equia Forte (E) (GC); 3. Activa (A) (Pulpdent; Watertown, MA USA); and 4. a new material coded ASAR-MP4 (Dentsply Sirona). The experimental material has now been commercialized as Surefil One.

Specimen Preparation

De-identified extracted caries-free human molars were selected for this study. The bonding sites were prepared by sectioning the teeth mesio-distally and then removing approximately two-thirds of the apical root structure. The buccal and lingual tooth sections were mounted with dual-curing acrylic resin (Triad DuaLine, Dentsply Sirona) in 12-mm-diameter brass rings. The enamel and dentin bonding surfaces were ground flat to 180 grit for the thicker smear layer specimens and 600 grit for all other test specimens using a water coolant and a sequence of carbide polishing papers (Struers; Copenhagen, Denmark). All test specimens were ground immediately before preparation of the bonded assemblies. To obtain optimal moisture conditions, the teeth were left moist using a blot-drying technique prior to bonding. Excess water was not removed with an air flow to avoid desiccation of the substrate. The overdried condition was created by vigorously air drying the dentin for at least 10 s, leaving a visibly dry surface devoid of surface moisture. For the dentin specimens ground to a 180-grit surface, the moist technique was employed prior to placement of the adhesive or self-adhesive restorative material.

Shear Bond Strength Test

Ten specimens each were used to determine the SBS to enamel and dentin for each surface condition. For the universal adhesive test group, for both substrates and all substrate conditions, specimens were prepared without phosphoric acid pre-treatment (self-etch technique). Following the treatment of enamel and dentin with the adhesive agent, the adhesive film was visible-light polymerized for 10 s with a SmartLite Focus (Dentsply Sirona) LED curing unit. The prepared specimens were then secured in an Ultradent bonding clamp (Ultradent; South Jordan, UT, USA) fitted with a polytetrafluoroethylene mold with a cylindrical cavity 2.38 mm in diameter and 4 mm in height. The restorative resin composite was placed in the rings and polymerized for 40 s.

No surface conditioning or adhesive agent was used for the self-adhesive restorative materials. Following positioning of the bonding apparatus with the specimen-former insert, the restoratives were mixed for 10 s in a ProMix 2 mixing device (Dentsply Sirona) and placed directly onto the tooth substrate inside the polytetrafluoroethylene mold. In the light cured groups S, A, and F, the materials were allowed to self-cure at room temperature for 1 min to facilitate penetration and interaction with the substrate surface. For E and S self-cured, the specimens were allowed to self-cure for 6 min at room temperature. Following the curing protocols, all bonded assemblies were removed from the bonding apparatus, and the bonded specimens were stored for 24 h in distilled water at 37°C. Following this initial storage period, the specimens were thermocycled for 6000 cycles between water baths set at 5°C and 55°C.

After thermocycling, the specimens were loaded to failure at a crosshead speed of 1.0 mm/min using an MTS Insight machine and TestWorks 4 software (MTS Systems; Eden Prairie, MN, USA). An Ultradent custom shearing fixture was used to apply the load to the bonded assembly immediately adjacent to the flat ground tooth surface. Shear bond strengths (MPa) were calculated from the peak load at failure divided by the bonded surface area.

Statistical Analysis

A two-way ANOVA with factors restorative material and surface condition, followed by Tukey's highly significant difference (HSD) test ($\alpha = 0.05$), were used for analysis of the SBS data.

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RESULTS

The results of enamel bonding are shown in Fig 1.

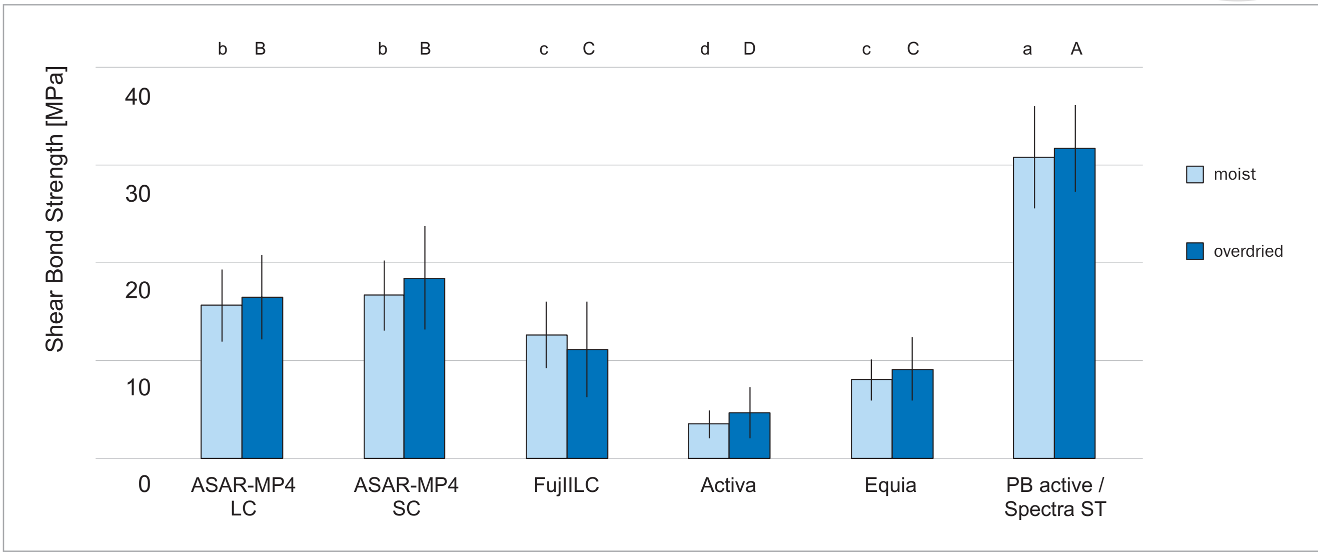

Fig 1 Results for shear bond strength (SBS) to moist and dry enamel. SBS groups comparing the moist surface condition marked with the same small letter were statistically similar ($p > 0.05$). SBS groups comparing the overdried condition marked with the same capital letter were statistically similar ($p > 0.05$). On enamel, the restorative material was a significant factor ($p < 0.05$), while the surface condition was not ($p > 0.05$). LC: light cured; SC: self-cured.

The dentin bonding results are exhibited in Fig 2.

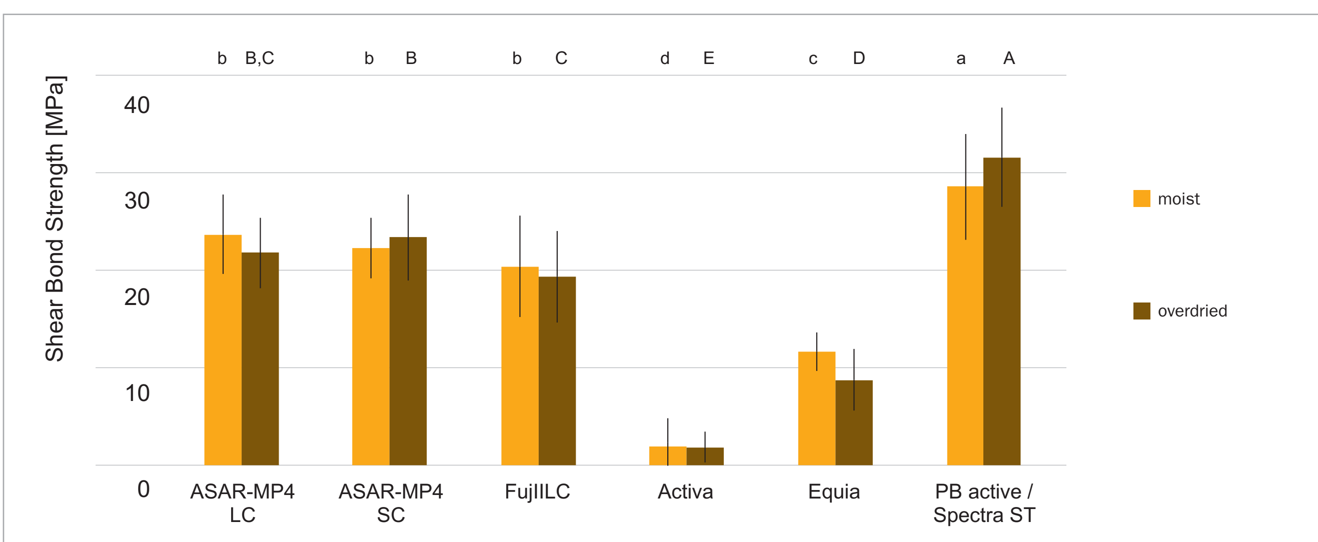

Fig 2 Shear bond strength (SBS) results to moist and dry dentin. SBS groups comparing the moist surface condition marked with the same small letter were statistically similar ($p > 0.05$). SBS groups comparing the overdried condition marked with the same capital letter were statistically similar ($p > 0.05$). On dentin, the restorative material was a significant factor ($p < 0.05$), while the surface condition was not ($p > 0.05$). LC: light cured; SC: self-cured.

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The results of dentin bonding after treatment with 600-grit vs 180-grit papers are illustrated in Fig 3.

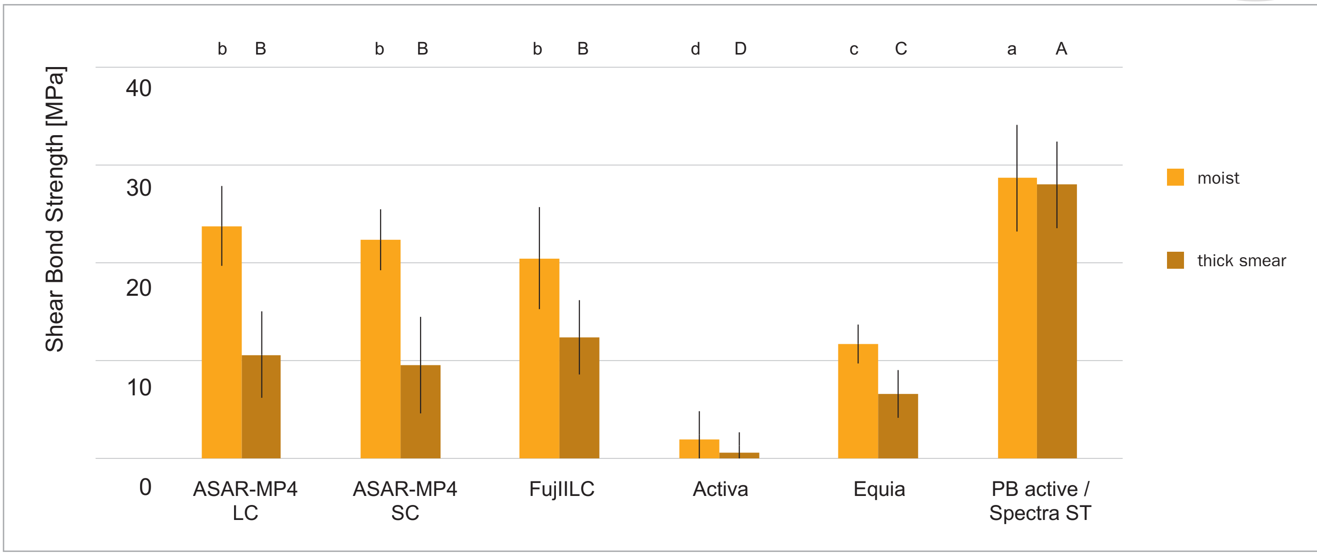

Fig 3 Shear bond strength (SBS) to 600-grit and 180-grit (thick smear layer) ground surfaces. SBS groups comparing the moist surface condition marked with the same small letter were statistically similar ($p > 0.05$). SBS groups comparing the thick smear layer condition marked with the same capital letter were statistically similar ($p > 0.05$). On dentin, the restorative material and the surface condition were significant factors ($p < 0.05$). LC: light cured; SC: self-cured.

DISCUSSION

The results of this investigation can provide valuable guidance on the best clinical technique to achieve optimal results with self-adhesive restorative materials. All the materials tested were insensitive to the extreme drying procedure used for surface preparation. In a clinical environment, it is visually and technically difficult to adjust the degree of surface moisture to a "moist" but not overwet condition. The ability to use an air syringe to remove excess water or other contaminants prior to placing the restorative material simplifies the procedure and eliminates ambiguity about the proper way to optimally prepare the substrate.

The significant effect of smear layer thickness on the shear bond strength of the self-adhesive restoratives also provides important insight into good clinical technique for these materials. While the use the abrasive papers in this study cannot be directly related to the smear layers left by carbide or diamond cutting tools used clinically, the results reported here suggest that minimizing the thickness of the smear layer on dentin is desirable. The significant reduction in SBS for the ASAR-MP4 material and Fuji II LC would suggest that with a thicker smear layer, these materials may not be able to fully penetrate that layer into the underlying dentin. From a practical standpoint, this might mean coarse diamonds should be avoided for final cavity preparation and fine diamonds or carbide burs be preferentially used to finish the cavity preparation.[16]

The results of this in vitro study are also consistent with the clinical results for Activa. The lower shear bond strengths are consistent with the relatively low retention rate of this material in nonretentive cervical cavities.[18] The manufacturer of this material now recommends its use in conjunction with an adhesive.

The universal adhesive, used in self-etching mode only, generated the highest shear bond strengths to all surfaces. This adhesive was insensitive to overdrying, as were the self-adhesive materials, but was also not significantly affected by the smear layer thickness. In both light- and self-cure modes, ASAR-MP4 generated the highest bond strength to enamel of all the self-adhesive restoratives. The dentin shear bond strengths for this material were similar to those of Fuji II LC and superior compared to the other self-adhesive materials. The present study suggests that ASAR-MP4 would generate similar or superior clinical retention values in Class V cavities compared to the glass ionomer and resin-modified glass ionomer evaluated here.

The first null hypothesis is rejected, as there were significant differences in SBS to both enamel and dentin among the materials tested. The second null hypothesis was ac-

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cepted, because there were no significant differences in SBS between the moist and dry surface conditions for a given material. Because significant differences in shear bond strength to dentin existed between the two smear layer conditions, the third null hypothesis was rejected.

CONCLUSION

The shear bond strengths of the materials tested were found to vary depending upon the material system. The newly developed self-adhesive composite hybrid compared favorably to the other self-adhesive materials with respect to adhesion to dentin and enamel. The materials tested were not adversely affected by overdrying the substrate, but the self-adhesive materials were significantly affected by the smear layer thickness.

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Clinical relevance: Based on shear bond strength testing, the clinical adhesive performance of a newly developed self-adhesive composite hybrid may equal that of glass-ionomer and resin-modified glass-ionomer restorative materials. Overdrying the tooth surface will not adversely affect the adhesion potential of these materials, but managing the thickness of the dentin smear layer is important for generating high bond strengths.