Micro-tensile bond strength of adhesive systems applied on occlusal primary enamel

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The aim of this study was to evaluate the micro-tensile bond strength of adhesive systems (OptiBond Solo™, Kerr; Prime & Bond NT®, Dentsply) on occlusal surface of primary molars. The adhesives were tested under manufacturers' specifications and after contamination of the bonding site with saliva. Hour-glass cylindrical-shaped samples were obtained and subjected to a tensile force. No significant difference was observed among the groups. OptiBond Solo™ and Prime & Bond NT® showed similar values of bond strengths when applied on occlusal enamel of primary molar under either saliva contamination or not.
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INTRODUCTION

Although there has been a general decline in the prevalence of dental caries, mainly attributed to topical fluoride, in the last two decades, a concurrent increase in the proportion of pit and fissure caries has occurred.[1] The inherent morphology of pit and fissures allows plaque retention and makes difficult the beneficial effects of fluorides. Thus, the occlusal surfaces account for more than fifty percent of all surfaces affected by the disease[2], although they represent only 12.5 %.[1]

The enamel acid etching[3] along with the development of monomers with high molecular weight[4,5] have lead to the development of the fissure sealing technique.[6] The sealing obliterates pit and fissures with resinous materials and reduces the predisposition to caries progression. Innumerous articles have been published demonstrating that fissure sealing prevents occlusal surfaces from developing caries lesions.[7-9]

However, fissure sealing is mainly indicated for teeth in the eruptive stage. Under this circumstance, they are susceptible to inadvertent contamination by saliva and/or crevicular fluid during the clinical procedure.[10,11] Due to the fact that sealants (hydrophobic monomers) are not compatible to wet surfaces, several authors have suggested the use of hydrophilic adhesive systems between etched enamel and the sealant as a way of optimizing bond strength in face of moisture and salivary contamination.[12-15]

Although the above technique has shown to improve sealants retention[15] it increases the number of clinical steps and makes the clinical procedure time-demanding and more difficult. Therefore, other authors have evaluated the application of adhesive systems as sole material for fissure sealing.[16-19]

They have reported reduced microleakage either on dried or contaminated etched enamel and a higher retention rate when a filled adhesive system, OptiBond (dual cure and FL), were applied on permanent teeth. To the extent of our knowledge, there has been no bond strength evaluation of one-bottle filled adhesive systems neither to occlusal contaminated enamel nor on primary enamel.

Therefore the aim of this study was to evaluate the bond strength of two one-bottle adhesive systems to the occlusal surface of primary molars either on dried or contaminated etched enamel, by means of the micro-tensile bond strength test.

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Table 1. Adhesives systems: technical information and application mode.

Adhesive	Etchant	Composition	Sequence of use	Manufacturer
OptiBond SOLO™	H_3PO_4 37% gel	BIS-GMA, HEMA, GDM, GPDM (glycerol phosphate dimethacrylate), ethanol, fumed silica, barium glass, sodium hexafluorsilicate, camphorquinone	**a,b,c,d** (only group 2)**, e, f**	Sybron-Kerr Corp.
Prime & Bond NT®	H_3PO_4 37% gel	PENTA (dipentaerythritol penta acrylate monophosphate), R5-62-1, acetone, camphorquinone, cetylamine hydrofluoride, nanofillers (amorphous silicon dioxide) and two proprietary elastomeric resins	**a,b,c,d** (only group 4)**, e, f**	Dentsply

a) enamel etch (15s); **b)** rinse (15s); **c)** air-dry (5s); **d)** contamination of occlusal surface with 0,4 (l of saliva (10s); **e)** adhesive application – gentle agitation of first layer (15s), agitation of second layer (15s); **f)** light cure – *OptiBond SOLO* (20s) and *Prime & Bond NT* (10s each layer).

MATERIALS AND METHODS

This experiment used 16 exfoliated primary molars disinfected in 0.5% chloramines.[20] The study was approved by the Ethics Committee of the School of Dentistry - University of São Paulo.

The two filled one-bottle adhesive systems tested were: OptiBond Solo (Kerr) an ethanol based system and Prime & Bond NT (Dentsply) an acetone based system. The composition, application mode and batch number are described in Table 1.

The pulpal chamber of primary molars were conditioned with 35% phosphoric acid and filled with a composite resin in order to facilitate the specimen's preparation for micro-tensile test. The occlusal surfaces were cleaned with pumice and bristle-brush in the slow-speed hand-piece and randomly divided into four groups (n=4). Then, the occlusal surfaces were etched (37% phosphoric acid) for 15 s, rinsed for 15 s and air-dried for 5 s. The adhesive systems were applied, according to the manufacturers' directions of the manufacturer, in the specimens from groups 1 and 3. Teeth from groups 2 and 4, had the occlusal surface contaminated with 0.4 µl of saliva (stimulated, centrifuged and stored under refrigeration), before the adhesive application. The volume of saliva was measured by means of a micropipette (Gilson, USA). The light curing step was performed with a VIP light unit (Bisco, USA) under a light output of 600 mW/cm^2.

Resin composite build-up "crowns" (Z 250, 3M ESPE, St. Paul, MN, USA) were constructed on the bonded surfaces in increments of 1 mm that were each light activated for 30s using the same light unit and light intensity. All the bonding procedures were done by a single operator at a room temperature of 24°C and 75% relative humidity.[21]

After storage in distilled water at 37°C for 24 h, the specimens were longitudinally sectioned perpendicular to the adhesive interface, by means of a diamond saw in a Labcut 1010 machine (Extec Corp., Enfield, CT, USA), yielding 1mm thick rectangular slices. Specimens were trimmed at the interface to a cylindrical hourglass shape (diameter of about 0.8 mm^2) using a fine diamond bur (3168F – KG Sorensen) in high-speed handpiece under air/water cooling.

The number of premature debonded sticks (D) per tooth during specimen preparation was recorded. The cross-sectional area of each hourglass specimen was measured with the digital caliper (Absolute Digimatic, Mitutoyo, Tokyo, Japan) to the nearest 0.01 mm and recorded for the calculation of the bond strength.

The hourglass specimens were individually attached to a modified device for microtensile testing[22] with cyanoacrylate resin (Zapit, Dental Ventures of North America, Corona, CA, USA) and subjected to a tensile force in a universal testing machine (Kratos Dinamometros, São Paulo, SP, Brazil) at a crosshead speed of 0.5 mm/min.

The bond failure modes were evaluated at X 400 (HMV-2, Shimadzu, Tokyo, Japan) and classified as cohesive (failure exclusive within enamel or resin composite, C), and adhesive or mixed (failure at resin/enamel interface or mixed with cohesive failure of the neighboring substrates, A/M).

Data treatment

For each tooth, a bond strength index[23] was calculated assuming the relative contribution of the possible mode of failures, according to the following formula (values in MPa):

Bond strength index:
$I_t = (B_{A/M} \times \%_{A/M}) + (C_D \times \%_D) + (C_R \times \%_R) + (B_D \times \%_D)$

Where:
- $B_{A/M}$ - Average bond strength of specimens with adhesive/mixed fracture pattern;
- $\%_{A/M}$ - Percentage of specimens with adhesive/mixed fracture pattern;
- C_D - Cohesive strength of enamel;
- $\%_D$ - Percentage of specimens that failed cohesively in enamel;
- C_R - Cohesive strength of resin;
- $\%_R$ - Percentage of specimens that failed cohesively in resin;
- B_D - Bond strength attributed to specimens that spontaneously debonded during preparation (4 MPa);
- $\%_D$ - Percentage of specimens debonded during preparation.

The formula assumes the cohesive strength of the resin composite (C_R) as the average value of all specimens (from a single tooth) that failed in that manner. Similarly it was calculated the cohesive strength of enamel (C_D). The value attributed to specimens that failed prematurely during preparation and could not be tested was 5 MPa. This value is arbitrary and corresponds to the minimum value that can be measured in the non-trimming microtensile test.[24] A bond strength index was calculated for each of the four teeth used per group. These were averaged to express the mean bond strength index for every material at each surface condition.

A two way ANOVA (material vs. surface condition) and Tukey's multiple test was used to compare the micro-tensile bond strength indexes. A one-way ANOVA was used to compare the cross sectional areas (mm^2) of the specimens. The level of confidence was established at $\alpha = 5$ %.

RESULTS

The mean cross-sectional area ranged from 0.79 to 0.91 mm^2 and no difference among the treatment groups was detected ($p<0.05$).

In this particular study, no cohesive failures within substrates were observed; therefore, this value was zero in the formula. The mean bond strengths and their respective standard deviations (MPa) are shown in Table 2. No statistical significant difference was detected among the experimental groups ($p<0.05$).

Table 2. Means and standard deviations (MPa) of bond strength values.

Groups	Bond strength (MPa)
OptiBond SOLO dry	28.7 ± 2.7
OptiBond contaminated	29.2 ± 3.1
Prime & Bond dry	34.2 ± 5.7
Prime & Bond contaminated	33.0 ± 1.8

DISCUSSION

The literature considers that the success of any adhesive procedure depends on the absence of gaps in the interface formed by the dental material and tooth substrate. Therefore, studies about the performance of such interfaces are an important tool for assessing a variety of dental products and techniques. Shear and tensile bond strength tests are the most used to assess tooth/dental material interface.[25]

Nevertheless, recent studies demonstrated that such tests, when used with the contemporary adhesive systems, have reached excessively high bond strength values as well as a great number of cohesive fractures either in tooth or restorative material. According to the authors, this fact is due to the manner in which the bond is stressed. A non-uniform stress concentrates in a localized region where it opens a flaw that finally fails. Thus, it would be impossible to measure the real value of resin-dentin or resin-enamel bond strength. [26,27]

The micro-tensile bond test became widespread provided that a reduced adhesive area, around 1 mm^2, has a better stress distribution during loading.[28,24] It has been widely used in bond strength tests both in dentin and enamel of permanent teeth.[29,30]

However, there are few micro-tensile studies assessing the adhesive interface in primary teeth. Moreover, all of them were performed in primary dentin.[31,32]

When the shear bond test was employed to assess the bonding interface of a restorative material on primary enamel, a high incidence of cohesive failure was found in the composite resin.[33,34] Meanwhile, the micro shear bond test showed similar values of bond strength for both the primary and permanent enamel.[35] This apparent controversy discloses a need of doing more studies with primary teeth in order to evaluate whether the data, obtained so far with permanent teeth, is valid for primary ones.

No report about bond strength of dental adhesive systems on intact primary enamel was found in the literature. However, it has been shown that the prismless layer has negatively influenced the etching pattern of both phosphoric acid and self-etching systems applied to intact primary enamel surface.[36]

The specimens for the micro-tensile test can be prepared in hour-glass shape or stick-like specimens of 0.7 – 1.0 mm^2 cross-sectional area.[24,37] It is usually easier to prepare sticks since a sequence of parallel serial sections from labial to lingual direction, plus another from mesial to distal face result in the sticks. But in experimental phase of this study while performing these sequences of sections many cohesive failures within enamel substrate were observed. This finding drastically reduced the number of sticks that could be obtained per tooth, probably, due to the attrition and eccentric movement of the cutting disc that induced stresses in the adhesive interface.[38]

A possible explanation is that the physiological resorption of primary teeth usually leads to such a loss of dental structure that sometimes it reaches the dentin bellow the pulp chamber. It creates a fragile tooth structure which makes difficult to obtain specimens in occlusal enamel for the micro-tensile test. This is somewhat true, that during specimen preparation, several tooth structure fractures were often seen. For these reasons, it was decided to fill up the inside pulp chamber with composite resin in order to reinforce the tooth structure before cutting it and performing the test. The use of specimen in hour-glass shape also reduced the number of specimens with premature failure. To use this method, each slab formed by composite resin/adhesive/tooth is trimmed to form a gentle curve at the interface aiming to concentrate the stress to the bonded area.

The values of bond strength found in the present study are similar to those obtained by other authors[30] using single-bottle adhesives (One Step - Single Bond) applied to the labial enamel of permanent teeth without contamination. These values show that one-bottle adhesive systems can effectively bond intact primary enamel, even when the enamel is contaminated with saliva after acid etching.

It is well known that the improvement of adhesive systems were based on dentin substrate, which is inherent moist and with higher organic content than enamel.[24] Such substrate has led to the development of hydrophilic monomers and organic solvents that are able to penetrate throughout the demineralized collagen network and simultaneously remove the residual water on this surface.[39] Therefore, resin-dentin bond strength reached higher values and additional success in adhesion technique was gained.[40,41]

Solvents present in many contemporary adhesive systems are able to mix to water and raise its vapor pressure allowing its evaporation. Water vapor pressure, used as solvent in some adhesives, is only 23 mbar, lower than ethanol and acetone that is 44 and 233 mbar respectively.[42,23] The performance of the adhesives assessed in this study may be attributed to the solvents compositions.

In one way, dentin moisture, that is more expressive after acid etching, can be compared to contamination of the etched enamel surface with crevicular fluid or saliva.[33] Thus, the behavior of adhesive systems in moist dentin seems to be similar in enamel, but in this case, carbonate hydroxyapatite crystals would be involved instead of collagen fibrils.[9]

The adhesive systems Tenure and ScotchBond Multi-Use were not efficient when applied as intermediate layer between enamel and sealant[15] probably because they have water as solvent which has low evaporation capacity.

El-Kalla and Garcia-Godoy[43] evaluated, through SEM, the labial surface of permanent teeth bonded with single-bottle adhesives (Prime & Bond 2.1; One Step and Tenure Quik with fluoride), under salivary contamination. They concluded that the pattern of adhesive penetration was not different between the contaminated and not contaminated groups. Saliva did not affect tags formation into enamel surface, probably due to the fact that the above adhesives are acetone-based systems. However, the groups contaminated with saliva did not show the same pattern of tags formation when a water-based adhesive system was used (Syntac Single Component).[43] It can be inferred that, etched enamel under salivary contamination should receive hydrophilic adhesives, with high vapor pressure solvents for fissure sealing, instead of hydrophobic adhesives.

The results obtained in this study are of clinical importance. Salivary contamination is a potential problem during adhesive procedures, especially in those cases where rubber dam can not be used, such in newly erupting tooth.[43]

Another relevant variable in the performance of an adhesive system, not assessed in this study, is the presence of filler particles. Grande *et al*.[16,17] and Witzel *et al*.[19] showed better results for OptiBond dual cure either in a clinical trial (retention rate) or in a laboratorial study (microleakage). The authors have attributed the better performance of this system to a higher mechanical strength and less polymerization shrinkage as it is filler loaded. On the other hand, this adhesive has ethanol as solvent what can partially justify the results in contaminated enamel surface with human plasma.

Additional studies should also be accomplished to evaluate the performance of self etching systems used as fissure sealants, either in permanent or primary teeth, since it has reduced clinical chair time, when compared to those systems that demand acid etching as a separate step.

CONCLUSION

The adhesive systems OptiBond Solo™ and Prime & Bond NT® showed similar values for bond strength, when applied on intact occlusal enamel of primary molar either under saliva contamination or not.

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