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Enamel Wetness Effects on Bond Strength Using Different Adhesive Systems

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Clinical Relevance

The enamel surface should not be desiccated extensively when an all-in-one self-etching adhesive is employed.

SUMMARY

Objective: **To evaluate, through the application of different dentin bonding systems, the influence of wetness on shear bond strength in enamel.**

Methods: **This study evaluated three etch-and-rinse adhesive systems (Scotchbond MP [used with and without primer]; Singlebond; and Prime&Bond 2.1) and two self-etching adhesive systems (Clearfil SE Bond; and Xeno IV). Flat bovine enamel surfaces were either air-dried for 30 seconds or blotted with absorbent paper after acid-etching for the conventional bonding agents or before the application of self-etching bonding agents. The resin composite EsthetX was bonded to flat surfaces that had been treated with one of the adhesives, following the manufacturer's instructions. After being stored in water at 37°C for one week, bonded specimens were broken in shear. Data were evaluated with two-way analysis of variance (ANOVA) and Student-Newman-Keuls tests (α=0.05). For comparing each condition**

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individually, regardless of the adhesive or wetness condition, a one-way ANOVA and a Student-Newman-Keuls test (α=0.05) were applied.

Results: **The two-way ANOVA showed significant differences among adhesive systems. An interaction effect was also observed ($p<0.05$), but wetness did not influence shear bond strength (p=0.98). The one-way ANOVA showed that the all-in-one adhesive was the only material influenced by the presence of water at the enamel's surface.**

Conclusion: **The all-in-one adhesive behaved differently depending on whether the enamel surface was dry or wet.**

INTRODUCTION

Since the introduction of the enamel etching and adhesion technique, bonding agents have changed considerably from simple hydrophobic resin adhesives to complex hydrophilic dentin bonding types. This evolution has occurred mainly as a result of the large amount of water found in dentin. For this reason, adhesive techniques were specifically developed to interact with the moisture present in dentin, resulting in the "wet bonding technique" described by Nakabayashi and others.[1]

Numerous adhesive systems with different characteristics are available for the professional. Depending on whether etching was previously conducted with phosphoric acid or not, the adhesive can be respectively classified as etch-and-rinse or self-etching. Etch-and-rinse systems require a previous application of phosphoric acid in order to create microporosities on the enamel and to expose collagen fibrils on the dentin.[1] These bonding systems can be classified according to the number of operative steps in either a three-step process (phosphoric acid, primer, and adhesive) or a two-step process (phosphoric acid followed by the application of a single solution containing both primer and adhesive). On the other hand, self-etching adhesive systems contain acidic monomers that simultaneously acid-etch and infiltrate the demineralized surface. These systems can also be classified in a two-step process (acidic primer and adhesive) or a one-step or all-in-one process (all the components in a single solution). Though its goal is to reduce operative time, self-etching may also minimize the technique's sensitivity and the risk of application errors caused by the operator.[2] The enamel adhesion mediated by self-etching adhesives, however, is still a concern.[3] Some authors[4,5] have observed that pre-etching with phosphoric acid increases the bond strength. Additionally, the application time may also play a role.[6,7] These findings indicate that the application procedure of self-etching adhesives may still be technique-sensitive.

Since it is clinically difficult to maintain a dried enamel surface while simultaneously keeping the dentin wet, the use of hydrophilic bonding adhesives in enamel has been associated with the "wet bonding technique." The presence of water at the enamel surface may be of particular interest in orthodontics and pediatric dentistry because in these fields moisture control is sometimes difficult.[8] Saliva contains water, proteins, bacteria, and other organic components. Bond strength may be reduced if the tooth is contaminated with saliva during adhesive procedures.[9] However, the influence of water after enamel etching and before application of the adhesive has not been thoroughly defined,[10,11] especially in relation to self-etching adhesives. With self-etching adhesives, the composition includes not only hydrophilic monomers but also water and organic solvents such as acetone and ethanol. In these systems, water has the additional function of creating an ionization medium that allows the self-etching to occur.[12] After application, water and other organic solvents, if not properly removed by air-blowing, may remain trapped in the hybrid layer.[13,14] This could compromise both bond strength[15] and polymerization.[16] For this reason, it is hypothesized that the amount of water present at the enamel surface before the application of a self-etching adhesive may play a role.

Considering the variations among available adhesive systems and different behaviors noted in the literature,[12,17,18] the presence of water at the enamel surface (which is associated with the "wet bonding technique") requires further investigation. Therefore, the aim of the present study was to evaluate the effect of wetness on the shear bond strength of bovine enamel prior to the application of various adhesive systems. Two working hypotheses were studied: 1) wetness influences shear bond strength and 2) the studied adhesive systems exhibit different behaviors.

METHODS AND MATERIALS

Five adhesives were evaluated: 1) a three-step etch-and-rinse (Scotchbond Multi-purpose Plus, 3M/ESPE, St Paul, MN, USA); 2) a two-step etch-and-rinse (Singlebond, 3M/ESPE); 3) a two-step etch-and-rinse (Prime & Bond 2.1, Dentsply/Caulk,

Table 1: *Adhesive Systems Studied and Application According to Manufacturers*

Adhesive and Manufacturer	Composition	Application Protocol
Scotchbond Multi-purpose Plus, 3M ESPE, St Paul, MN, USA	Primer: aqueous solution of HEMA and polyalkenoic acid copolymer Bonding: Bis-GMA and HEMA combined with initiation system	Phosphoric acid-etching for 15 s; rinse with water; dry gently for 2 s; apply primer and dry gently for 5 s; apply adhesive to enamel; light-cure for 10 s
Singlebond 2, 3M ESPE, St Paul, MN, USA	Bis-GMA, HEMA, polyalkenoic, acid copolymer, photoinitiators, ethanol, water	Phosphoric acid-etching for 15 s; rinse with water for 10 s; dry gently; apply two coats; air-dry for 5 s; light-cure for 10 s
Prime & Bond 2.1, Dentsply/DeTrey, Konstanz, Germany	UDMA, Bis-GMA, PENTA, butylated hydroxitoluene, 4-ethyl dimethyl aminobenzoate, cetilamine hydrofluoride, initiator, and acetone	Phosphoric acid-etching for 15 s; rinse with water within 15 s; dry gently; apply adhesive for 20 s; air-dry for 5 s; light-cure for 10 s
Clearfil SE Bond, Kuraray Medical Inc, Tokyo, Japan	Primer: MDP, HEMA, hydrophilic dimethacrylate, di-camphorquinone, aromatic tert-amine, water Bonding: MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, photoinitiator, aromatic tert-amine, silanated colloidal silica	Apply primer for 20 s; air-dry for 5 s; apply bond; air-dry for 5 s; light-cure for 10 s
Xeno IV, Dentsply/Caulk, Milford, DE, USA	Mono-, di- and trimethacrylate resins, PENTA, photoinitiators, stabilizers, cetylamine hydrofluoride, acetone, water	Grasp container at each end, or insert into holder, placing thumb along center score. Firmly apply pressure until container separates. Insert disposable applicator into opening to saturate applicator tip. Using the disposable microbrush applicator tip supplied, apply and scrub surfaces for 15 s. Apply a second layer with the microbrush, as above, scrubbing for 15 s. Air-dry for at least 5 s. Light-cure for 10 s.

Abbreviations: Bis-GMA, bisphenol A diglycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-Methacryloyloxydecyl dihydrogen phosphate; PENTA, dipentaerythritol pentacrylate monophosphate; UDMA, urethane dimethacrylate.

Milford, DE, USA); 4) a two-step self-etch (Clearfil SE Bond, Kuraray Medical Inc, Tokyo, Japan); and 5) a one-step self-etch (Xeno IV, Dentsply/Caulk). The three-step etch-and-rinse adhesive system was studied with and without application of the primer solution. When the primer solution was not applied, the bonding solution was applied according to the manufacturer's instructions. Table 1 displays the composition and manufacturers' instructions of the adhesives studied.

Ninety-six extracted bovine incisors were selected, cleaned, and embedded in acrylic resin. Flat enamel surfaces of approximately 5 mm were obtained by mechanically grinding the incisors with water-cooled sequential silicon carbide papers (Nos. 180, 320, 600, and 1000). The specimens were divided into two groups (n=48). Group 1 (dry) was air-dried for 30 seconds to desiccate the enamel surface. In group 2 (wet), surfaces were blotted with absorbent paper (Kimwipes EX-L, Kimberly-Clark, Roswell, GA, USA) to remove water excess, while still leaving the enamel moist and visibly shiny. The air-drying was performed at a standard distance of approximately 10 cm and at a 45° incline. The moisture control procedures were accomplished either after acid-etching the enamel (for the etch-and-rinse adhesives) or before the application of self-etching adhesives.

Specimens were then divided into six subgroups (n=8) according to the adhesives used. Because the three-step adhesive was applied either with or without primer solution, a total of six adhesives were studied for each wetness condition. Each adhesive system was applied, following its respective manufacturer's instructions (Table 1). Next the adhesive was light-cured continuously for 10 seconds with a halogen light source (Elipar Trilight, 3M ESPE, Seefeld, Germany), operating at approximately 500 mW/cm^2 irradiance, as measured by the incorporated radiometer.

A split Teflon mold (2.0 mm in diameter and 2.0 mm in height) was clamped to the treated enamel surfaces, filled with a resin composite (Esthet X, Dentsply/DeTrey, Konstanz, Germany), and light-cured continuously for 40 seconds (Elipar Trilight, 3M ESPE). The specimens were removed from the mould 10 minutes after the completion of the light-curing. Afterwards, specimens were stored in a 37°C water bath for seven days in dark canisters protected from light. All specimens were assembled at controlled levels of humidity (55%±5%) and temperature (23±1°C).

The shear bond strength test was conducted in a universal testing machine (EMIC, São José dos Pinhais, PR, Brazil) using a wire-loop method at a crosshead speed of 1.0 mm/min. The failure modes were evaluated at 18× magnification under a stereomicroscope. Failure was assessed as mainly adhesive or mainly cohesive within enamel or within resin composite. Data were submitted to two-way analysis of variance (ANOVA) and Student-Newman-Keuls tests (α=0.05). For the sake of comparing each condition individually, regardless of the adhesive or wetness condition, a one-way ANOVA and a Student-Newman-Keuls test (α=0.05) were also applied.

RESULTS

Mean values and standard deviations are presented in Table 2 and Figure 1. The two-way ANOVA showed significant differences among the adhesive systems, with an interaction effect between adhesives and enamel wetness also being observed ($p<0.05$). The enamel wetness showed a nonsignificant influence on shear bond strength (p=0.9842). In general, the etch-and-rinse bonding agents showed statically insignificant decreases on bond strength when the enamel was wet, whereas the self-etching bonding agents showed the opposite behavior. The results from the multiple comparisons obtained by the one-way ANOVA are displayed as superscripted letters in Table 2. The one-step self-etching adhesive showed a noticeable increase in shear bond strength when the enamel was kept wet before its application, as demonstrated by the one-way ANOVA. For the three-step etch-and-rinse adhesive, applying primer before the adhesive did not influence the shear bond strength.

The failure modes, evaluated at 18× magnification under a stereomicroscope, showed that specimens generally presented adhesive-enamel failure patterns. For this reason, no correlation between failure mode and shear bond strength could be found. These results showed that there was no relationship between failure mode and adhesive system or between failure mode and wetness.

Table 2: *Mean Shear Bond Strengths (MPa) and Standard Deviations. Lowercase Superscripted Letters Indicate No Statistical Differences Between Groups (α=0.05)*

Adhesive	Dry Technique	Wet Technique
Scotchbond (primer + bonding)	18.3 ± 4.1 bc	15.7 ± 3.4 abc
Scotchbond (bonding only)	18.3 ± 3.7 bc	13.3 ± 3.4 ab
Singlebond	19.8 ± 3.4 c	18.6 ± 3.9 bc
Prime & Bond 2.1	12.7 ± 4.9 ab	10.8 ± 5.3 a
Clearfil SE Bond	15.7 ± 3.1 abc	17.8 ± 3.1 bc
Xeno IV	11.0 ± 5.0 a	19.7 ± 1.9 c

DISCUSSION

The study's first hypothesis was partially rejected since only the all-in-one adhesive system was statistically affected by the presence of water at the enamel surface. The second hypothesis was accepted since the evaluated adhesive systems showed different behaviors. In general, etch-and-rinse adhesives were negatively affected by the presence of water, and self-etching adhesives were positively affected by it. As demonstrated in Table 2, the all-in-one adhesive requires more water at the dental surface in order to achieve higher bond strength values. This finding may be explained by the fact that self-etching adhesives need an ionizing medium for the chemical reaction to get started.[12] Thus, the presence of water at the enamel surface could create a better ionizing medium for the all-in-one adhesive, which would increase the adhesive's ability to interact with the enamel. In addition to facilitating an adequate bonding procedure, especially for the self-etching adhesives, water may also remain trapped in the hybrid layer,[13,14] which could impair both polymerization and bond strength.[16,19] For this reason, the air-blowing procedure following adhesive application should be cautiously executed.

Regarding the method, some points should be addressed. The present study evaluated various dental adhesives, testing their sensitivity to surface moisture via shear bond strength tests using bovine enamel as a bonding substrate. Although bovine enamel presents structural differences from human

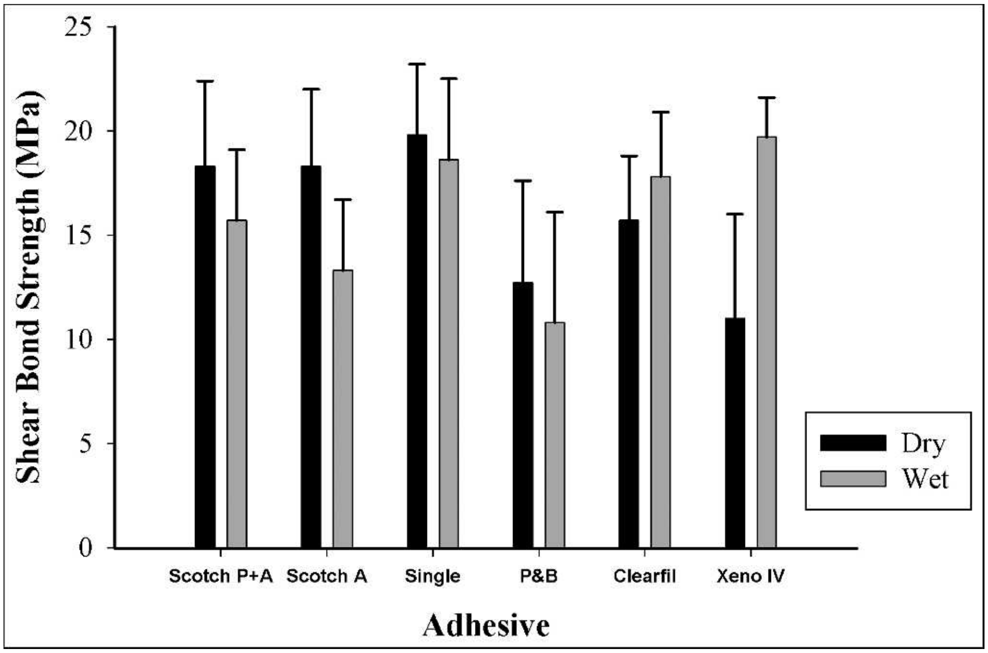

Figure 1. *Mean shear bond strengths (MPa) and standard deviations for different adhesives.*

enamel, its use in this research concurs with previous studies.[20,21] Bovine dentin has been proposed[22] as a suitable alternative for human dentin. Krifka and others,[23] for example, compared the shear bond strengths of enamel and dentin derived from both bovine and human primary teeth, showing that bovine primary teeth may be used as alternatives to human teeth. Additionally, no significant differences in enamel microleakage have been observed between human and bovine substrates.[24]

In order to standardize the adhesion area, enamel surfaces were mechanically flattened. This procedure, however, could potentially influence the shear bond strength because differing bonding behaviors have been reported[25] in ground and unground surfaces. Because the adhesion to ground enamel may be facilitated by the absence of both aprismatic and hypermineralized substrates, some moisture-prone clinical situations, such as orthodontic bracket bonding or pit-and-fissure sealing, could lead to different bond strength results. The use of ground enamel in the present study may also explain why the self-etching adhesives showed bond strengths similar to those of "traditional" each-and-rinse adhesives.

A two-way ANOVA, using adhesive type and wetness condition as independent variables, is an appropriate statistical design for the present study. However, because the interaction effect can play a role in detecting the influence of wetness on a specific adhesive, a one-way ANOVA was applied as well. This one-way ANOVA showed results similar to those of the two-way ANOVA. As shown in Table 2, the one-way ANOVA detected that only the all-in-one adhesive was actually influenced by wetness, which explains why the two-way ANOVA's general comparison found no influence of wetness. Additionally, the one-way ANOVA showed where the differences among adhesives occurred.

Specimens generally presented adhesive-enamel failure patterns when evaluated under a stereomicroscope. These results may be related to the unequal stress distribution caused by shear bond strength tests, especially for specimens with relatively large bonded areas (usually 3–6 mm in diameter).[26] This issue, which has been a concern, was recently addressed.[27] Therefore, the present study used specimens with smaller bonded areas (3.1 mm^2). Additionally, the shear stress was developed by a wire-loop instead of a conventional knife-edge chisel, which may have created better stress distribution at the adhesive-enamel interface.[26,27]

The results found for the etch-and-rinse adhesives showed that its bonding to enamel was not affected by the presence of water, which agrees with the findings of previous studies.[11,28] The bonding solution present in Scotchbond contains bisphenol A diglycidyl methacrylate (Bis-GMA) and 2-hydroxyethyl methacrylate (HEMA), combined with an initiation system. The presence of HEMA gives this material a hydrophilic behavior, which explains why the water had little influence on the bond strength when the adhesive was applied without the primer solution. Although applying the primer before the Scotchbond bonding solution did not influence the shear bond strength, the primer's use on wet enamel showed a smaller decrease in bond strength values than when the bonding solution was used alone.

The degree to which self-etching adhesives demineralize dental structure may vary, as they present different pH levels, varying from mild to moderate to strong pH.[2,12] This difference in demineralization potential, however, is not always correlated with the depth of adhesive infiltration at the dental structure.[29] It is believed[30] that some acidic monomers present in self-etching adhesives are capable of chemically bonding with the hydroxyapatite present in the dental structure. In the present study, the samples were stored in water for one week before shear bond strength tests were performed. Perhaps chemical bonding could influence the results if the storage periods were lengthened. Because only one storage time was employed, it is impossible to discuss the influence of chemical bonding. Additionally, it is not clear to what extent this chemical bonding could benefit bond strength in comparison with conventional etch-and-rinse adhesives.[31] The study of the influence of wetness after different storage periods when several self-etching adhesive systems are employed could be of interest to future researchers.

It should be noted that the effect of various adhesive systems on bond strength might still be operator-dependant,[32] since the air-blowing duration after priming also influences the bond strength.[15] The results presented here should not be extrapolated to all one-step self-etching systems because only one material was evaluated and because various systems differ not only in pH, but also in hydrophilicity and comonomers.

When moisture control is difficult to obtain, the long-term behavior of adhesive-bonded restorations may be compromised. This is particularly true in orthodontics and pediatric dentistry, in which contamination with saliva could lead to premature debonding of brackets, thereby increasing the treatment duration or the loss of retention of pit-and-fissure sealants, respectively. The present study showed that contemporary adhesives are not negatively affected by moisture at enamel surfaces. Additionally, when a self-etching adhesive is used, the enamel surface should not be extensively desiccated.

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