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Comparative Evaluation of Immediate Bond Strength to Enamel with One-Step Self-Etch Adhesives.

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ABSTRACT

Available knowledge in the literature regarding the immediate bond strength of one-step, self-etch adhesives to enamel surfaces covered with thick layers is limited. Therefore, the immediate bond strength of six one-step, self-etch adhesive systems to enamel surfaces covered with thick smear layers was evaluated. Flattened enamel surfaces were prepared and polished with 180-grit silicon carbide abrasive paper under running water for 20 seconds, to produce thick smear layers similar to clinical conditions. The following were applied to bonding surfaces: self-etch adhesives, six one-step, self-etch adhesives, Adper Prompt L-POP, iBond, Clearfil S3 Bond, AdheSE One F, G-Bond, Optibond All-in-one, one two-step, self-etch adhesive, and Clearfil SE Bond. Bonded teeth were sectioned into sticks immediately after bonding and subsequently subjected to microtensile bond strength testing. Differences in bond strength for enamel were determined using two-way ANOVA with Tukey tests. iBond, AdheSE One F, Optibond All-in-one failed to bond enamel surfaces covered with thick smear layers. However, Adper Prompt L-POP and Clearfil S3 Bond showed similar enamel bond strength to Clearfil SE Bond. It should be noted that the bonding effectiveness of the majority of one-step, self-etch adhesives tested to enamel surfaces covered with thick smear layers is poor.

Keywords: Adhesion; self-etch adhesive; immediate bond strength, microtensile

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INTRODUCTION

New materials are constantly being developed in an attempt to simplify clinical procedures and solve problems associated with resin bonding to enamel and dentin. Several tests should be performed in order to document their efficacy. Due to the high time and resource costs of clinical testing, laboratory evaluations are a reliable way to predict clinical efficacy [1].

In vitro bond strength tests are often relied upon as predictors of clinical performance for new generation adhesive-restorative materials. However, in most of the *in vitro* bond strength tests, measurements were performed 24 hours or more after the bonding procedures [2,3]. This experimental setting does not exactly reflect the clinical situation since the bonded interface is subjected to stresses seconds after being paced *in vivo*. These stresses may be caused by the polymerisation shrinkage of resin composites, occlusal and contouring adjustment, and finishing/polishing procedures that are performed immediately after the placement of the restorations [4].

Staninec and Kawakami reported that some dentin adhesives exhibited an increase in bond strength over a short period. The authors suggested that these increases may be due to further polymerisation at the interface or stress relaxation from hygroscopic expansion of the composite [5]. Burrow et al. found a steady increase in tensile bond strength over a period of 24 hours, suggesting that bonds mature during this period [4]. Barink et al. also reported that stress is expected to decrease during the first hour after polymerisation [6]. According to these authors, it may be advisable to instruct the patient not to load the restoration for a certain period [7].

Self-etching adhesive systems offer some advantages over total-etch adhesives, such as ease of use [8], reduced technique sensitivity [9], and limited postoperative sensitivity [10]. However, their etching potential may not be as aggressive as that produced by phosphoric acid; therefore, thick smear layers may present a challenge for self-etch adhesives [11]. The majority of 24 hour bond strength data available from published studies revealed the controversial bonding potential of simplified systems as compared with conventional two-step, self-etch adhesives. As it is imperative that the restoration supports initial masticatory loading and thermal fatigue, assessment of bond strengths immediately after the restoration procedure is important from a clinical perspective. Only limited information is available in the literature on the “immediate” bond strength of conventional two-step, self-etch adhesives. Moreover, this type of information is totally missing for the more recently introduced and simplified one-step, self-etch adhesives. Thus, it is of interest to examine the immediate bond strength potential of current one-step, self-etch adhesive systems in order to evaluate their performance before the onset of post-curing polymerisation, stress relaxation, or bond maturation.

The objective of the present *in vitro* study was to compare the enamel bond strength of immediately sectioned specimens produced from seven different self-etch adhesives, six of which were one-step, self-etch adhesive systems. The rationale behind the use of immediate sectioning was to simulate the creation of internal flaws within the bonded specimens in a way that may be comparable to the immediate stressing of bonded restorations *in vivo*. The null hypotheses tested was that the microtensile bond strengths of the adhesives ere comparable.

MATERIALS AND METHODS

Twenty-one caries- and defect-free bovine incisors were stored in 0.02% sodium azide solution at 4°C to prevent bacterial growth. The teeth were cleaned; roots were severed using a slow-speed diamond saw under water cooling. Crowns were then embedded into self-curing acrylic resin. Enamel surfaces were flattened manually using 320-grit silicon carbide paper under running water. The surface of each tooth in the study was polished by hand with 180-grit silicon carbide abrasive paper under running water for 20 seconds in order to create thick smear layers similar to clinical conditions [12].

Samples were drawn to be randomly distributed into seven subgroups (n=3). Then, each group was randomly assigned one of the tested adhesives (Table 1): 2-step, self-etch adhesive, Clearfil SE Bond (Kuraray Medical Inc., Tokyo, Japan); one-step two-continent self-etch adhesive, Adper Prompt L-Pop (3M ESPE, St. Paul, MN, USA); one-step one-bottle self-etch adhesives, Clearfil S3 Bond (Kuraray Medical Inc., Tokyo, Japan);

iBond (Heraeus Kulzer, Hanau, Germany); AdheSE One F (Ivoclar-Vivadent, Liechtenstein, Schaan); G-Bond (GC, Tokyo, Japan); and Optibond All-in-One (Kerr, Orange, CA, USA).

Table 1: Materials used, components, and instructions for use

Materials	Components	Instructions for use
Clearfil Se Bond (Kuraray, Tokyo, Japan), LOT:01041A LOT:01552A	Primer: 10-MDP, HEMA, hydrophilic DMA, tertiary amine, water, photoinitiator Bond: 10-MDP, HEMA, bis-GMA, hydrophilic DMA, tertiary amine, silanated colloidal silica, photoinitiator	Apply Primer to tooth surface and leave in place for 20 s. Dry with a mild air stream to evaporate the volatile ingredients. Dispense the necessary amount of BOND into second mix well. Apply BOND to the tooth surface. After applying BOND, create a uniform film using a gentle air stream. Light cure for 10 s.
Adper Prompt L-Pop (3M ESPE, Seefeld, Germany) LOT:422280	Liquid #1: Methacrylated phosphate esters, bis-GMA, initiators based on CQ, stabilizer Liquid #2: Water, HEMA, polyalkenoic acid, stabilizer	Activate the L-Pop Unit Dose Dispenser to mix the adhesive. Apply mixed adhesive to entire surface, rubbing in with moderate finger pressure for 15 s. Use a gentle stream of air to thoroughly dry the adhesive into a thin film. Rewet the brush tip with adhesive and apply a second coat of adhesive to the tooth surface. The second coat does not require rubbing. Use a gentle stream of air to thoroughly dry the adhesive into a thin film. Light cure for 10 s.
iBond (Heraeus Kulzer, Hanau, Germany) LOT:010112	UDMA, 4-MET, glutaraldehyde, acetone, water, stabilizer, photoinitiator	Apply in three consecutive layers and massaged into the prepared tooth structure for 30 s. Following that, the solution is blown away with a gentle air stream. Light cure for 20 seconds.
Clearfil S3 Bond (Kuraray, Tokyo, Japan) LOT:041201	10-MDP, HEMA, bis- GMA, water, ethanol, silanated colloidal silica, camphorquinone	Apply adhesive to the tooth surface and leave for 20 s. Dry the entire surface sufficiently by high air pressure air for more than 5 s while spreading the bond layer thinly. Light cure for 10 seconds.
AdheSe One F (Ivoclar-Vivadent, Liechtenstein, Schaan) LOT:P31000	Bis-acrylamide, water , alcohol, bis-metacrylamide dihydrogen phosphate, amino acid acrylamide, highly dispersed silicon dioxide, initiators, stabilizers, potassium fluoride	Dry tooth surface. Apply an adequate amount of AdheSE One F is to the cavity with the VivaPen directly. Once the cavity surfaces have been completely coated, the adhesive must be brushed into the entire surface for 20 seconds. Dry with strong stream of air until a glossy, immobile liquid film results. Light cure for 10 s.
G-Bond (GC, Tokyo, Japan) LOT:1101141	4-MET, UDMA, phosphate monomer, DMA component, fumed silica filler, acetone, water, photoinitiator	Apply to the tooth surface using the micro-tip applicator. Leave undisturbed for 10 seconds. After application, dry thoroughly using oil free air under. High air pressure for 5 s. Light cure 10 seconds.
Optibond All-in-One (Kerr, Orange, CA, USA) LOT:3613640	Bis-GMA, GPDM, HEMA, ethanol, acetone, water, filler	Apply two coats with agitation, 20 seconds each. Mild air-blast for 5 s after gentle air-blast. Light cure for 10 s.

10-MDP: 10-methacryloyloxy decyl dihydrogenphosphate; 4-MET: 4-methacryloxyethyl trimellitic acid; Bis-GMA: Bisphenol glycidyl methacrylate; DMA: Dimethacrylate
HEMA: 2- hydroxyethyl methacrylate; PENTA: Dipentaerythritol penta-acrylate phosphate
UDMA: Urethane dimethacrylate; GPDM: Glycerol phosphate dimethacrylate

All adhesive systems were strictly applied according to the manufacturers' instructions. A resin composite (Valux Plus, 3M ESPE, St. Paul, MN, USA) was used incrementally to form a composite buildup approximately 5 mm in height. Each composite layer was light-cured for 40 seconds at a curing intensity of 600 mW/cm^2.

Immediately upon completion of the restorative procedure, each tooth was fixed within an acrylic resin block that was attached on a low-speed cutting machine (Micracut 125; Metkon Instruments Inc., Bursa, Turkey) in such a way that the tooth remained parallel to the blade. By means of this water-cooled diamond blade, the tooth was sectioned in x and y directions into 0.9 x 0.9 mm composite-dentin or composite-enamel sticks using the "non-trimming" technique [13].

For the microtensile test, a cross-sectional area of each stick was precisely measured using a digital calliper and glued to a custom-made jig using cyanoacrylate. This jig is made of two parts that are kept together by a couple of post-hole joints. The jig was mounted on a Bisco Microtensile Tester device (Bisco, Schaumburg, IL, USA), and tensile forces were applied to the microtensile stick until failure. Failure modes were determined using a stereo microscope and recorded as adhesive, mixed or cohesive in resin or dentin/enamel.

The distribution of microtensile bond strength data was first checked for normality using the Kolmogorov-Smirnov test and then statistically analysed using one-way ANOVA design to examine the effect of the factor “adhesive” on bond strength, both by considering bond strength data per tooth (in order to take into account the tooth-related variance) and by pooling all sticks coming from different teeth of the same group. A Tukey HSD test was applied for post-hoc comparisons. For all the analyses, statistical significance was set at $p < 0.05$.

RESULTS

The microtensile bond strengths of the experimental groups are summarised in Table 2. Three of five one-step one-bottle self-etch adhesive systems (iBond, AdheSE One F, Optibond All-in-one) failed to bond enamel surfaces covered with thick smear layers during sectioning for the preparation of microtensile bond strength test specimens. Therefore, these adhesives were excluded from factorial design.

Adhesive significantly influenced the enamel bond strength (p=0.000). Clearfil SE Bond, a conventional two-step, self-etch adhesive, exhibited the highest microtensile bond strength (27.88 ± 9.2 MPa) of all the adhesives tested. Adper Prompt L-POP (27,26 ± 9,06 MPa) and Clearfil S3 Bond (22.15 ± 7.7 MPa), which are one-step, self-etch adhesives, showed enamel bond strength similar to that of Clearfil SE Bond, whereas G-Bond (17,96 ± 5,2 MPa) exhibited the lowest enamel bond strength, with significant difference from other adhesives ($p < 0.05$).

DISCUSSION

The microtensile test is considered a reliable adhesion testing technique [14] that can be used to assess the interfacial strength between an adhesive and a bonding substrate. To obtain microtensile specimens, a series of cuts are made on a single tooth that may induce the formation of internal defects. Similarly, under clinical conditions, flaws may develop because of the stresses acting on the newly created bonded interface. Some authors [15,16] believe that hygroscopic expansion caused by water sorption may help to relieve some of the internal stresses created during polymerisation shrinkage or close marginal leakage gaps. However, it is unclear whether this phenomenon can heal these internal flaws [17,18].

In this study, the bond strengths of seven current adhesive systems were evaluated following the “non-trimming” technique of the microtensile bond test method. This technique is currently considered to be a reliable adhesion test because it allows the loading stress to be more uniformly distributed by the testing of small specimens [14,19]. However, the higher means and standard deviations obtained from specimens of smaller diameter make the microtensile bond strength test discriminative enough to detect differences arising from treatment variables with the use of a smaller number of actual tooth specimens [20]. The trade-off of such an improvement in the reliability of adhesion testing is that the method is time consuming and technically demanding. Although the initial measurements of the “immediate” bond strength of each tooth were referred to in the present study, it should be noted that about three or four hours have elapsed between bonding and having the sticks ready for microtensile bond testing for each group.

Within the limits of this *in vitro* study, one of the one-step, self-etch adhesives (G-Bond) showed lower microtensile bond strengths than conventional two-step, self-etch adhesive when bonding to enamel among the one-step, self-etch adhesives with measured enamel bond strength. The poor performance of G-Bond may have been caused by the hydrolysis of the acidic monomer 4-META and the hydrophilic monomer HEMA in the presence of the water within the adhesive. It has been shown that these monomers can be readily hydrolysed by water upon storage at increased temperature [21]. As G-Bond is a water-containing, no-mix type 1-step, self-etch adhesive, the 4-META can hydrolyse very rapidly under elevated temperatures or prolonged storage. Even though all the materials tested were kept in the refrigerator after opening, one cannot be sure about the storage conditions during transportation or the time interval between production and delivery. However, Adper Prompt L-POP and Clearfil S3 Bond, one-step, self-etch adhesives exhibited adhesion properties for bonding to enamel comparable to a conventional two-step, self-etch adhesive (Table 2). This means that manufacturers of one-step, self-etch adhesives have successfully formulated the effective hybridisation of enamel covered with thick smear layers.

Table 2: Microtensile bond strengths (mean ± SD, MPa) and failure modes of each group

Adhesives	Bond Strength	Failure modes
Clearfil SE Bond	27,88 ± 9,2 ^{aA}	A>M>C
Adper Prompt L-POP	27,26 ± 9,06 ^{aA}	M>A>C
iBond	Na	-
Clearfil S3 Bond	22,15 ± 7,7 ^{aA}	A>M>C
AdheSE One F	Na	-
G-Bond	17,96 ± 5,2^{aB}	A>M>C
Optibond All-in-One	Na	-

Means (n=20) with the same lower superscript letters are not significantly different in the same row ($p > 0.05$), means with the same upper superscript letters are not significantly different in the same column ($p > 0.05$). Failures were classed as adhesive (A), cohesive either in composite or in dentin/enamel (C) or mixed (M). Na: Not available. Bonded specimens were destructed totally during sectioning.

The majority of one-step, self-etch adhesive systems failed to provide effective immediate bonding to enamel in this study. Three adhesives (iBond, AdheSE One F, and Optibond All-in-one) failed completely during specimen sectioning for microtensile bond strength testing. One possible explanation for this could be that the aggressiveness of these self-etch adhesives was not able to condition thick smear layers created on enamel surfaces polished with rough silicon carbide paper, which resulted in poor bonding performance. Previously, it was noted that thick smear layers hinder the enamel hybridisation of self-etch primers and adhesives [22]. Therefore, it can be concluded on the one hand that thick smear layers would impair some of one-step, self-etch adhesive systems tested in the present study. On another hand, it was reported that the performance of one-step, self-etch systems was inferior to the other adhesives in the literature [23-25]. However, other one-step adhesives, including Clearfil S3 Bond and Adper Prompt L-POP, exhibited enamel bond strength similar to that of Clearfil SE Bond in the present study. Therefore, it can be concluded that the bonding effectiveness of one-step, self-etch adhesives may depend on the product rather than the adhesive class.

The satisfactory results of two-step, self-etch adhesive were in agreement with previous studies [2,25]. For Clearfil SE Bond, the bonding component creates a hydrophobic coat that prevents the adhesive layer from behaving as a permeable membrane after polymerisation [20] that expedites water sorption within the adhesive layer [26]. In addition, the acidic primer of the Clearfil SE Bond is not based on UDMA and TEGDMA.

CONCLUSIONS

The assessment of bond strengths immediately after the restoration procedure is important from a clinical perspective. The finding of significantly lower immediate enamel bond strengths in some one-step, self-etch adhesives suggests that simplifying bonding procedures comes at the expense of reducing bonding effectiveness.

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