Bonding Potential of All-in-One Adhesives to Ground Enamel

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Abstract

Objectives: The study was aimed at assessing the bonding potential of recently introduced all-in-one adhesives to ground enamel. **Materials & Methods:** The microtensile test was used to evaluate the bond strength of the one-bottle self-etch adhesives Bond Force (Tokuyama), AdheSE One (Ivoclar Vivadent) and Xeno V (Dentsply), in comparison with the etch-and-rinse adhesive Prime&Bond NT (Dentsply). Four extracted human molars were assigned to each group for microtensile bond strength testing; two molars per group were used for SEM observations of the adhesive-enamel interface. The bond strengths in MPa, including pre-test failures as 'zero' values, were statistically analyzed (Kolmogorov-Smirnov test, Kruskall-Wallis Non Parametric Analysis of Variance, Dunn's Multiple Range test; $p<0.05$). **Results:** The following bond strengths were recorded in MPa (mean±standard deviation): Bond Force (29.99±21.23), AdheSE One (25.93±25.92), Xeno V (18.76±17.36) and Prime&Bond NT(43.48±26.67). The statistical analysis demonstrated that the bond strength achieved by Bond Force was similar to that of the control adhesive, whereas for AdheSE One and Xeno V the bond strength was significantly lower than for Prime&Bond NT. **Conclusions:** Within the limitations of the study it can be concluded that the milder self-etch adhesive has given higher bond strength values. **Clinical Significance:** To recommend to dental practitioners the adhesive with higher bonding potential to ground enamel.

Key Words *Microtensile, Adhesive, Self-etch, Enamel.*
Short title: *All-in-one adhesives on ground enamel.*

Introduction

The development of dental materials has resulted in the introduction of self-etch, all-in-one adhesive systems that combine etching, priming and bonding into one step.[1,2] One-bottle systems are easier to use and handling errors are reputed to be limited.[3, 4]

In single-component adhesives acidic monomers, hydrophilic monomers, water and/or organic solvents are combined into one solution.[5] The self-etching mechanism of these systems is based on acidic monomers that are able to partially demineralize and infiltrate the dental hard tissues.[2] Unlike etch-and-rinse systems, they use the smear layer as a bonding substrate, by incorporating it into the hybrid layer.[6] Post-operative sensitivity, a potential risk related to incomplete resin infiltration, is supposed to be limited.[7]

Apart from these attractive advantages, there are still some concerns surrounding one component systems. Self-etch adhesives are less effective in demineralizing the enamel surface in comparison with phosphoric acid,[8] while the acidity of self-etching monomers can be buffered by the mineral components of the smear layer.[6] Some studies have reported a limited bonding ability of self-etch adhesives to enamel.[9, 10]

The clinical success of a restoration depends on the quality and the durability of the adhesion between tooth substrate and the restoration.[11] As the literature does not provide conclusive information on the reliability of bond strength to enamel of self-etching adhesives, it seemed of interest to assess the bonding potential of newly introduced one-bottle systems to ground enamel in comparison with an etch-and-rinse adhesive tested as control.

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Table1
Adhesives, their chemical composition, pH values, Batch numbers and application indications

ADHESIVE	CHEMICAL COMPOSITION	APPLICATION
Bond Force **Batch** 4T10787	Phosphoric acid monomer, Bisphenol A di(2-hydroxy propoxy) dimethacrylate (Bis-GMA), Triethylene glycol dimethacrylate, 2-Hydroxyethyl methacrylate (HEMA), Camphorquinone, alcohol, purified water. **pH=2.3**	Application and agitation for 20s; Air drying firstly indirectly until there is no water movement and directly for 5s; Light-curing for 10s;
AdheSE One **Batch** K10655	Derivatives of bis-acrylamide, water, bis-methacrylamide dihydrogen phosphate, amino acid acrylamide, hydroxy alkyl methacrylamide, silicon dioxide, catalysts, stabilizers. **pH=1.5**	Application and agitation for 30s; Air dispersing until there is no water moving; Light-curing for 10s;
Xeno V **Batch** 0703004017	bifunctional acrylates, acrylic acid, acid phosphoric functionized ester, acid acrylicate water, tertiary butanol, phosphine oxide initiator, stabilisator **pH<2**	Application twice; Agitation for 20s; Air-drying for 5s; Light-curing for 20s;
Prime&Bond NT **Batch** 0509001773	Di- and Trimethacrylate resins, PENTA, Nanofillers-Amorphous Silicon Dioxide Photoinitiators, Stabilizers, Cetylamine Hydrofluoride, Acetone	Application of tooth conditioner gel for at least 15s; Rinsing for at least 10s and drying with cotton; Application of P&BNT and leaving the surface wet for 20s; Air-drying for 5s; Light-curing for 10s;

Adhesion was to be assessed quantitatively with microtensile bond strength testing, as well as qualitatively through SEM imaging of the adhesive-enamel interface.

The formulated null hypothesis was that the tested adhesives exhibited similar bonding effectiveness to ground enamel.

Materials and Methods

Twenty four human, caries-free, third molars were collected and stored in 0.5% Chloramine T solution at 4°C, to prevent bacterial growth, for a maximum of 3 months before they were used in the study.

The teeth were randomly divided into four groups (n=6) and assigned to different materials.

Group 1: Bond Force + Estelite Sigma (Tokuyama Dental, Tokyo, Japan);

Group 2: AdheSE One + Tetric Evo Ceram (Ivoclar-Vivadent, Schaan, Liechtenstein);

Group 3: Xeno V + Esthet X (Dentsply DeTrey Kostanz, Germany);

Group 4: Prime & Bond NT + Esthet X (Dentsply DeTrey Kostanz, Germany), as a control group.

A flat enamel surface was created by grinding either the buccal or the lingual surface of the molars with wet 600-grit SiC-paper.

All the materials were handled strictly following manufacturers' recommendations (reported in Table 1), at room temperature (23.0° C ±1° C) and 50%±5% relative humidity.

Following the bonding procedure, the proprietary restorative resin composite of each adhesive was used to incrementally build-up a cylinder of about 5 mm in height. Each added 2 mm thick composite layer was singularly cured for 40 s with a halogen curing device (Astralis 7, Ivoclar Vivadent, Schaan, Liechtenstein, 500 mW/cm2).

Table 2
Descriptive statistics of microtensile bond strength values and frequencies of premature failures (PF). Different letters indicate statistically significant differences.

Adhesives	Sticks	PF%	Mean	Median	Standard Deviation	Significance ($p<0.05$)
Bond Force	59	20.34%	29.99	30.39	21.23	**AB**
AdheSE One	53	35.85%	25.93	21.25	25.92	**B**
Xeno V	70	31.43%	18.76	19.49	17.36	**B**
Prime&Bond NT	67	13.43%	43.48	40.42	26.67	**A**

The bonded specimens were stored for 24 hours in 100% humidity and 37ºC until the microtensile bond strength tests were performed.

Two randomly selected specimens per group underwent SEM evaluation.

The remaining four teeth in each group were used for microtensile testing. The teeth were fixed on a glass platform with sticky wax and sectioned vertically into 1 mm thick slabs with a slow-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) under water cooling. Each slab was then serially sectioned into 1x1 mm sticks, according to the "non-trimming" method of the microtensile test, yielding 10-15 beams per tooth. Each stick consisted of half of its length of the substrate and for the rest of the composite build-up. The two halves were joined together at the interface by the bonding material of interest.

Each stick was measured with a digital caliper (Orteam s.r.l, Milan, Italy), glued with cyanoacrylate (Super Attak Gel, Henkel Loctite Adesici s.r.l) to a Gerardeli's jig, and tested in a universal testing machine (Triax Digital 50, Controls, Milan, Italy; cross-head speed: 0.5 mm/min) until failure occurred. The load at failure was recorded in Newtons and bond strength was calculated in MPa.

Failure modes were evaluated by a single operator under an optical microscope (Nikon SMZ645, Nikon, Japan) at X40 magnification, and classified as cohesive (within the composite material or tooth substrate), adhesive (between composite or adhesive and enamel), and mixed (adhesive and cohesive fractures occurred simultaneously).

All the sticks which failed prematurely were included and considered in the statistical calculations as "zero" values. As bond strength data were not normally distributed according to the Kolmogorov Smirnov test, the Kruskall-Wallis Non Parametric Analysis of Variance was applied to assess the significance of the differences in bond strengths among the experimental groups. Dunn's Multiple Range test was used for post-hoc comparisons. The level of significance was set at $p<0.05$ in all the analyses.

Scanning Electron Microscopy evaluation (SEM)

The teeth for SEM imaging of enamel-adhesive interfaces were fixed on a glass platform with sticky wax and sectioned vertically into two halves with a slow-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). Specimens were polished with 600-, 1000-, 1200-grit SiC paper under water cooling, etched with 32% phosphoric acid gel for 30 seconds, deproteinized with 2% sodium hypochlorite solution for 120 s. Each specimen was then dehydrated in an ascending ethanol series (25%, 50%, 70%, 90%, 95%,100%), dried, and mounted on metallic stubs prior to gold-sputtering (Polaron Range SC 7620, Quorum Technology, Newhaven, UK), and observing under Scanning Electron Microscope (SEM, JSM-6060LV, Jeol, Tokyo, Japan) at different magnifications (X500 & X1000).

Results

Mean and standard deviation values of microtensile bond strength, along with frequencies of premature failures in all the experimental groups are reported in Table 2.

Prime&Bond NT (43.48±26.67) demonstrated the strongest adhesion to ground enamel. Among the all-in-one adhesives, only Bond Force (29.99±21.23) achieved statistically similar bond strengths to the control material. Conversely, AdheSE One (25.93±25.92) and Xeno V (18.76±17.36) yielded significantly lower bond strengths than the etch-and-rinse system ($p<0.05$).

Premature failures occurred during specimen preparation in all the groups. Most of the failures were of the mixed type.

SEM evaluation

Following enamel etching with phosphoric acid in the control group, a thin hybrid layer with short resin tags

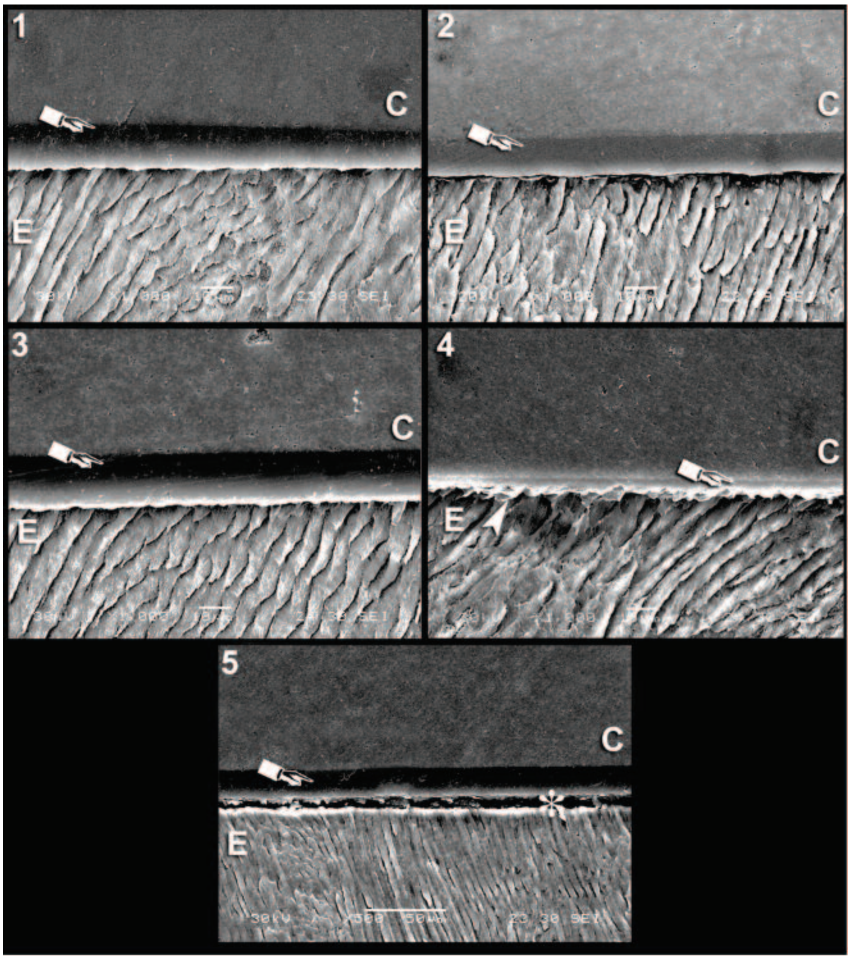

***Figure A** Scanning electron micrographs of the interfaces between the tested adhesives and ground enamel 1. Bond Force, 2. AdheSE One, 3. Xeno V . Pointers indicate adhesive layers. C-Composite, E-Enamel. 4. Prime&Bond NT. Thin hybrid layer with resin tags (arrowhead) was observed. Pointer indicates adhesive layer. C-Composite, E-Enamel (magnification X1000, bar 10 m). 5. Xeno V specimen with an evident interfacial gap (star). Pointer indicates adhesive layer. C-Composite, E-Enamel (magnification X500, bar 50m).*

could be detected (Fig A4). Conversely, all-in-one adhesives exhibited a very superficial interaction with the enamel substrate without evidence of a distinct hybrid layer (Fig A1,2,3). Nevertheless, satisfactory interfacial continuity was seen, except for Xeno V that showed some interfacial discontinuities (Fig A5).

Discussion

The finding of significantly lower bond strengths for AdheSE One and Xeno V in comparison with the control adhesive requires rejection of the null hypothesis.

Numerous studies have been carried out in order to assess the bond strength of all-in-one adhesives, but results are diverse. In some of the studies it was concluded that self-etching adhesive systems provide a weaker bond to the enamel substrate than etch-and-rinse systems.[3,9,10] Conversely, other studies revealed that self-etching adhesives can represent a satisfactory alternative, even on enamel.[12,13]

The incomplete demineralization of enamel produced by self-etch systems has raised some concerns.[8] According to the classification proposed by Tay et al. based on the solution pH, self-etch adhesives can be categorized as mild, moderate, and aggressive.[6] It was noted that even the most aggressive self-etch adhesives are not as acidic as 35% phosphoric acid and less effective in modifying the enamel surface to make it more receptive to the adhesive resin.[8,14] A correlation was found between the pH of self-etch adhesives and length of the produced enamel resin tags.[15]

In the present study only Bond Force established enamel bond strengths that were comparable to the control material. Bond Force, having a pH of 2.3, can be considered as a mild self-etch adhesive.[6] As a possible explanation, the ability of milder self-etch adhesives to react chemically with residual hydroxyapatite crystals within the partially demineralized smear layer can be considered.[16] Besides, the self-reinforcing monomers of Bond Force are claimed to contribute to the development of an intrinsically stronger adhesive layer that may have accounted for the relatively higher recorded microtensile bond strengths [Tokuyama Technical Information].

Furthermore, this study's findings support the evidence provided in a previous investigation that the length of enamel resin tags does not impact the bonding performance.[15]

Solvent is an important component of single-bottle systems. Apart from keeping the adhesive as a homogeneous solution, it enhances the penetration of hydrophilic functional monomers into the enamel surface.[17] Bond Force contains ethanol, while Xeno V contains tertiary butanol. In the chemical composition of AdheSE One the only solvent is water. It was assumed that neither organic solvent type, nor the inclusion or exclusion of organic solvent in all-in-one adhesives impact on the bond strength.[18,19] The same indication emerges from the present study. In fact, although containing different solvents, the three investigated all-in-one's obtained similar conditions of adhesion to enamel. It is possible that the influence of the solvent type may be more effective on dentin. Further research could be undertaken to verify this aspect.

Concern has also been raised regarding the chemical stability of all-in-one solutions. Some formulations have showed limited shelf-life and refrigerator storage has been recommended to avoid temperature effects.[2,20] To overcome these possible limitations, new formulations of all-in-one adhesives incorporate monomers that are expected to be more stable. For example, refrigeration is no longer required for Xeno V, due to the inclusion of tertiary butanol.

However, since a recently opened bottle of adhesive was tested in this study, no inference can be made on this issue from the collected data. It seems worth directing future research to analyzing the effect of temperature changes and repeated bottle opening on the bonding performance of these new all-in-one adhesives.

Moreover, the present investigation assessed the immediate bond strength established on enamel. However, data on the long-term adhesion of these new materials need to be collected as such information is lacking on the newly introduced all-in-one adhesives, despite its relevance for the clinical use. There are indications in the literature that the acidity possibly retained by all-in-one adhesives, even after polymerization, may expose the adhesive layer to hydrolytic degradation over time.[9,21] Although such an issue is of more concern for bonding to dentin as a more hydrated substrate, it should also be clarified with regard to enamel adhesion.

Conclusions

Within the limitations of the study it can be concluded that the tested all-in-one adhesives differ in their bonding potential to ground enamel. The etch-and-rinse system demonstrated the strongest adhesion to ground enamel, although bond strengths achieved by the milder self-etch adhesive were comparable to those of the control system.

Future studies should be carried out in order to assess the bonding durability and shelf-life of the tested all-in-one adhesives.

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