EFFICACY OF ALL-IN-ONE ADHESIVE SYSTEMS ON UNGROUND ENAMEL

MILOS BELOICA,[1] CARLOS AUGUSTO RAMOS CARVALHO,[2] IVANA RADOVIC,[3] MARIAM MARGVELASHVILI,[4] CECILIA GORACCI,[5] ZORAN R. VULICEVIC,[6] MARCO FERRARI [7]

Abstract

Objectives: The aim of this study was to determine the microtensile bond strength (TBS) of three all-in-one adhesives to unground enamel, using an etch-and-rinse adhesive system as control. **Methods and Materials:** Three all-in-one adhesives (Bond Force, AdheSE One and Xeno V) were compared to etch and rinse adhesive (Prime&Bond NT) as a control. Composite resin build-ups were made on the buccal enamel surfaces, approximately 6 mm in height, using the proprietary resin composite with each tested adhesive. All the bonded specimens were stored for 24h (37∞C and 100% humidity) before testing. In each group four specimens were further processed for TBS test and one specimen was used for microscopic evaluation of the adhesive interface. **Results**: The TBS was influenced by the type of adhesive system ($p<0.05$). The etch-and-rinse adhesive achieved the highest bond strength to unground enamel ($p<0.05$). No statistically significant differences in bond strength were found among the all-in-one adhesives ($p>0.05$). **Discussion:** The results derived from this study revealed the difference between self-etch adhesives and the etch-and-rinse system that was tested as control. The superior bonding potential exhibited by the etch-and-rinse system may be ascribed to the ability of the phosphoric acid gel to remove the layer of aprismatic enamel. Adhesion of self-etching adhesives relies on micromechanical retention as well as their chemical composition. **Conclusions:** The tested all-in-one adhesives did not differ in their bonding effectiveness to unground enamel. The etch-and-rinse adhesive tested as control resulted in significantly higher bond strength than all-in-one adhesives. **Clinical Significance:** Although all-in-one adhesives are more user friendly, their bond strength to unground enamel was lower in comparison to etch and rinse adhesive system.

Key words: All-in-one adhesives, unground, enamel, micro-tensile bond strength test

Short title: All-in-one adhesives on unground enamel

Introduction

A common trend in dentistry is to make dental materials and their use simpler for everyday's clinical practice. Following the introduction in 1955 of the acid etching technique,[1] various researchers have worked on improving it over the past years.[2]

1. Research Associate at Clinic for Pediatric and Preventive Dentistry, Faculty of Dentistry, University of Belgrade, Belgrade, Serbia
PhD student, Department of Dental Materials, University of Siena, Policlinico Le Scotte, Siena, Italy
2. PhD student, Department of Operative Dentistry, Bauru School of Dentistry, University of Sao Paulo, Brazil
PhD student, Department of Dental Materials, University of Siena, Policlinico Le Scotte, Siena, Italy
3. Teaching and Research Assistant at Clinic for Pediatric and Preventive Dentistry, Faculty of Dentistry, University of Belgrade, Belgrade, Serbia
PhD student, Department of Dental Materials, University of Siena, Policlinico Le Scotte, Siena, Italy
4. PhD student, Department of Dental Materials, University of Siena, Policlinico Le Scotte, Siena, Italy
5. DDS, MSc, PhD, Professor at Department of Dental Materials, University of Siena, Policlinico Le Scotte, Siena, Italy
6. DDS, MSc, PhD, Professor and Chair of Department of Pedodontics, Clinic for Pediatric and Preventive Dentistry, Faculty of Dentistry, University of Belgrade, Belgrade, Serbia
7.MD,DDS,PhD, Professor and Chair of Department of Dental Materials, University of Siena, Policlinico Le Scotte, Siena, Italy

Corresponding Author:
Milos Beloica
Research Associate at Clinic for Pediatric and Preventive Dentistry, Faculty of Dentistry, University of Belgrade, Belgrade, Serbia
PhD student, Department of Dental Materials, University of Siena, Policlinico Le Scotte, Siena, Italy
Address: Molerova 66, 11000 Belgrade, Serbia
Phone: +381/63/8106451
E-mail: beloica.milos@gmail.com

As a result of their contribution, the adhesive systems have evolved from first and second to current sixth and seventh generation.[2]

Two categories of contemporary adhesive systems can be classified based on the interaction with the dental substrate: etch-and-rinse and self-etch adhesives. [3]

Since self-etch adhesives simultaneously condition and prime enamel and dentin, they do not require phosphoric acid etching as a separate step. Such a simplification allows to save chair time and makes the bonding procedure less technique sensitive.[4] Self-etch adhesives bond to ground enamel satisfactorily.[4-6] However, adhesion of these systems to unground enamel remains elusive and should be further investigated.[3,4]

The microtensile bond strength test (TBS) has had various improvements[7] since it was introduced by Sano et al. in 1994 .[8] The majority of investigations have focused on ground enamel and dentin rather than unground enamel. Since simplification in adhesive systems is appreciated in contemporary dentistry and common clinical procedures such as sealing procedures, bonding of splinting materials or of orthodontic brackets are performed on unground enamel, in order to predict their clinical behavior,[9] it is of interest to test the bonding potential of current adhesive systems also to the unground enamel.

The aim of this study was therefore to determine the bond strength of three different all-in-one adhesives to unground

Table I.
Chemical composition, batch number, type of material, pH value, instructions for use

Material/ Manufacturer	Composition	Batch Number	Type of material/pH	Manufacturers' instructions
Bond Force Tokuyama Dental Tokyo, Japan	Phosphoric acid monomer, Bisphenol A di(2-hidroxy propoxy) dimethacrylate (Bis - GMA), Triethylene glycol dimethacrylate, 2-Hydroxyethyl methacrylate (HEMA), Camphorquinone, Alcohol, Purified water	**4T10787**	one bottle self-etching adhesive /2,3	Apply the adhesive Rub under light finger pressure for 20s, or more Gently apply air for 5s, then strong air for 5s or more Light-cure for 10s or more
AdheSE One Ivoclar Vivadent Schaan, Liechtenstein	Derivates of bis-acrylamide, Water, Bis-mathacrylamide, dihydrogen phosphate, Amino acid acrylamide, Hydroxy alkyl methacrylamide, Highly dispersed silicon dioxide, Catalysts, Stabilizers	**K10665**	one bottle, self-etching adhesive /1,5	Apply the adhesive Brush onto surface for no less than 30s Disperse excess with a strong stream of air Polymerize for 10s
Xeno V Dentsply Konstanz, Germany	Bifunctional acryl resin with amide functions, Acryloyamino alkylsulfonic acid, 'inverse' functionalized phosphoric acid ester, Acrylic acid, Camphorquinone, Coinitiator, Butulated benzenediol, Water, Tert-butanol	**703004017**	one bottle self-etching adhesive /pH<2	Apply adhesive twice Gently agitate for 20s Air dry for 5s Polymerize for 20s
Prime&Bond NT Dentsply Konstanz, Germany	Di - and Trimethacrylate resins PENTA (dipentaerythritol penta acrylate monophosphate) Photoinitiators Stabilizers Nanofillers - Amorphous Silicone Dioxide Cetylamine Hydrofluoride Acetone	**509001773**	one bottle etch and rinse adhesive	Apply 36% phosphoric acid for at least 15s (enamel) Spray and rinse conditioned areas for at least 10s Blot dry conditioned areas Apply adhesive and leave the surface wet for 20s Gently dry for at least 5s Polymerize for 10s

enamel, using an etch-and-rinse adhesive system as control. The tested null hypothesis was that the different adhesive systems did not differ in their bonding potential to intact enamel.

Material and Methods

Specimen preparation

Twenty caries-free human third molars were randomly chosen for this study. Teeth were stored in 0.5% Chloramine T solution at 4oC for preventing bacterial growth before use in the investigation. Teeth were decoronated 2 mm beneath the cemento-enamel junction using a slow speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, Il, USA), under water-cooling so as to expose the pulp chamber.

Buccal surfaces were cleaned with a pumice/water slurry, then with a fluoride free paste for composite resin (Universal Polishing Paste, Ivoclar Vivadent, Schaan, Liechtenstein), applied by a brush mounted in a low speed handpiece, under water cooling.

Bonding procedure

Teeth were divided into three experimental groups and one control group (n=5), according to the used adhesive system/composite resin (Table I):

1. Bond Force + Estelite (Tokuyama Dental, Tokyo, Japan)
2. AdheSE One + Tetric Evo Ceram (Ivoclar Vivadent, Schaan, Liechtenstein)
3. Xeno V + Esthet X (Dentsply, Konstanz, Germany)
4. Prime&Bond NT + Esthet X (control) (Dentsply, Konstanz, Germany)

Composite resin build-ups were made on the prepared buccal enamel surface by layering three 2 mm-thick increments, thus creating a build up of approximately 6 mm in height using with each tested adhesive the proprietary resin composite. Each increment was light cured for 40 s with a halogen curing device (VIP, Bisco Inc., 600 mW/cm^2 output

Table 2
Microtensile bond strength. Different superscript letters indicate statistically significant differences; SD = standard deviation; Failure mode: adhesive (A), cohesive (C), mixed (M), premature failures (PF)

Material	Bond strength in MPa	Failure mode in %	Number of specimens
	Mean ± SD	A/C/M/PF	
Bond Force Tokuyama Dental Tokyo, Japan	7,87±10,71a	44,83/3,45/0/51,72	58
AdheSE One Ivoclar Vivadent Schaan, Liechtenstein	5,65±9,45 a	28,13/3,13/1,56/67,18	64
Xeno V Dentsply Konstanz, Germany	7,84±11,14a	38,96/2,60/0/58,44	77
Prime&Bond NT Dentsply Konstanz, Germany	23,53±17,05 b	30/22/ 20/28	50

density). All the materials were applied according to the manufacturers' instructions. The chemical composition, batch numbers and the application modes are reported in Table I.

All the bonded specimens were stored for 24h (37°C and 100% humidity) before testing. In each group four specimens were further processed for microtensile bond strength test and one specimen was used for microscopic evaluation of the adhesive interface.

TBS test

Teeth were sectioned vertically into 0.9x0.9 mm thick slabs with a slow speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). Each slab was fixed on a glass platform with sticky wax and serially sectioned into 0.9x0.9 mm sticks, according to the 'non trimming' method of the microtensile test.[10-12] Each stick was measured with a digital caliper (Orteam s.r.l., Milan, Italy), and glued with cyanoacrylate (Super Attak Gel, Henkel Loctite Adesivi s.r.l) to a Gerardeli's jig. Afterwards, the specimens were stressed 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 (N). Bond strength was calculated in MegaPascals (MPa) by dividing the load at failure by the bonded surface area.

Failure modes were evaluated by a single operator under an optical microscope (Nikon type102, Tokyo, Japan) at 40x magnification, and classified as cohesive (within enamel), adhesive (between composite and enamel) or mixed (adhesive and cohesive fractures occurred simultaneously).

Statistical analysis

Sticks that had failed prior to testing were included in the statistical calculations as 'zero' values.

A preliminary linear regression analysis showed that the tooth of origin did not have a significant influence on the measured bond strength; therefore, the sticks were considered as independent within each subgroup. As bond strength data were not normally distributed, the Kruskall-Wallis Analysis of Variance was applied to assess the significance of the differences in bond strength among the groups. A series of Mann-Whitney tests with Bonferroni correction was used for the post-hoc comparisons.

In all the analyses the level of significance was set at $p<0.05$.

Scanning Electron Microscopy evaluation (SEM) of enamel-adhesive interface

One specimen from each group was sectioned vertically into 2 halves with a slow-speed diamond saw (Isomet, Luehler, Lake Bluff, IL, USA) under water cooling. Each specimen was polished with silicon carbide paper (300, 600, 1000, 1200 grit paper) under water cooling, etched with silica-free 32% phosphoric acid gel for 30 seconds (Bisco Inc., Schaumburg IL, USA), and immersed in 2% sodium hypochlorite for 120 seconds. The procedure was followed by dehydration in an ascending ethanol series (25%, 50%, 70%, 90%, 95%, 100%). Drying was performed with oil free air spray. Specimens were mounted on metallic stubs, gold-sputtered (Polaron Range SC 7620, Quorum Technology, Newhaven, England), and observed under a Scanning Electron Microscope (JSM 6060 LV, JEOL, Tokyo, Japan) at different magnifications in order to evaluate the morphologic characteristics of the enamel-adhesive interface .

Results

Microtensile bond strength

Microtensile bond strength data and failure modes distribution

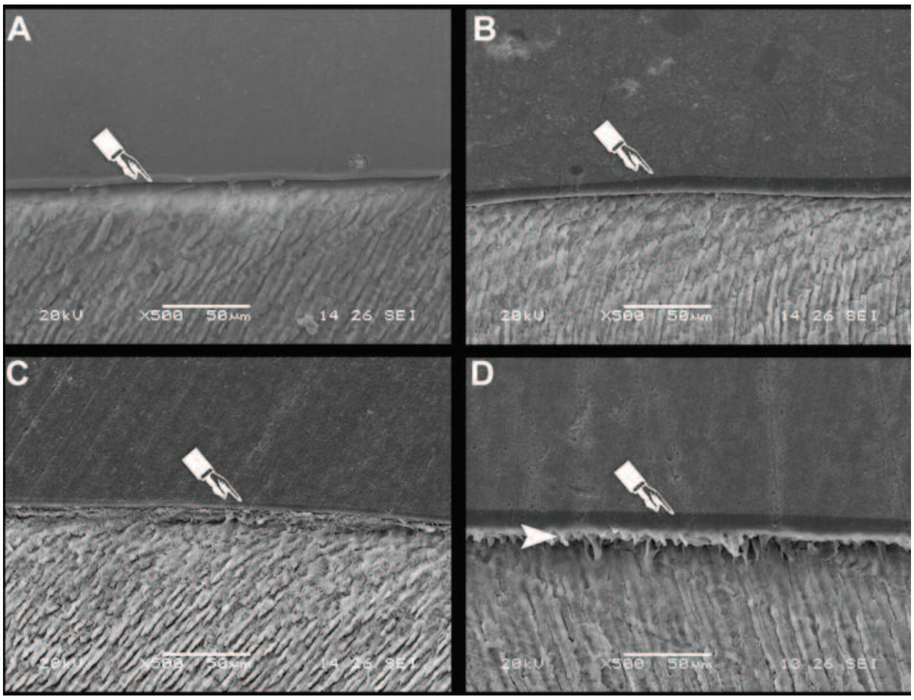

Figure I. SEM micrographs of the interface developed on intact enamel by the tested adhesives (500X, bar 50 µm). A superficial interaction was established by the all-in-one solutions, that yielded adhesive layers (pointers) of varying thicknesses (A: Bond Force; B: AdheSE One, C: Xeno V). With the use of phosphoric acid gel in the etch-and-rinse system (D: Prime&Bond NT), a thin enamel hybrid layer with short resin tags (arrow) was formed.

are reported in Table II.

The TBS was influenced by the type of adhesive system ($p<0.05$). The etch-and-rinse adhesive achieved the highest bond strength to unground enamel and the difference was statistically significant ($p<0.05$). No statistically significant difference in bond strength emerged among the all-in-one adhesives ($p>0.05$).

SEM evaluation

The all-in-one adhesives exhibited a rather superficial interaction with the enamel substrate, without evidence of formation of a distinct hybrid layer. AdheSE One yielded a thicker adhesive layer than Bond Force and Xeno V (Figure I A-C).

It was only with the use of the etch-and-rinse system Prime&Bond NT that a thin, yet homogeneous hybrid layer was developed, with resin tags penetrating into the enamel substrate to a limited depth (Figure ID).

Discussion

The results derived from this study revealed the difference between self-etch adhesives and the etch-and-rinse system that was tested as control. Therefore, the null hypothesis has to be rejected.

The bond strength of the all-in-one adhesives investigated in the present study was lower than that of the control total-etch system. These findings are in agreement with the results of several studies in which self-etch and etch-and-rinse adhesive systems were compared.[3,13-15]

However, results retrieved in this study are not in line with the outcome of several other studies in which no significant difference was reported between self-etch and etch-and-rinse adhesives.[6,16] The difference between the groups was also evident when premature failures were taken into consideration. Prime&Bond NT had 28% premature failures while each of the investigated all-in-one adhesives had more than 50% premature failures

The present study was performed on unground enamel, whose composition along with the presence of the superficial prismless layer has been well documented.[17,18] The aprismatic layer in enamel is less conductive to bonding,[19] and its removal is recommended prior to the bonding procedure.[20]

The superior bonding potential exhibited by the etch-and-rinse system may indeed be ascribed to the ability of the phosphoric acid gel to remove the layer of aprismatic enamel. It has been shown that the bond strength to intact enamel of several self-etch adhesives greatly benefited from preliminary etching with phosphoric acid.[20-22] However, phosphoric acid etching, combined with all-in-one adhesives, should be limited to enamel as it may have a detrimental effect on bonding to dentin.[22]

It would be of interest to verify whether and to what extent the adhesion to intact enamel of the new all-in-one adhesives investigated in this study may be enhanced by preliminary phosphoric acid etching.

Etching pattern largely depends on the acidity of the conditioner,[4] and shallower etching pattern may jeopardize bond strength and durability.[21] Self-etching adhesives pH values may be 10-20 times higher than that of 37% phosphoric acid.[4]

According to their acidity, self-etching systems may be classified as mild (pH>2), moderate (2>pH>1), or strong (pH<1).[4]

In agreement with the findings of Pashley et al.,[4] also in our study the self-etch adhesives achieved relatively low and similar bond strengths to unground enamel, regardless of their pH values, ranging between 1.5 and 2.3 (Table 1).

Adhesion of self-etching adhesives does not rely solely on micromechanical retention. The functional monomers of some mild self-etch formulations are claimed the ability to chemically react with Calcium ions that residue within the partially demineralised dental substrate (Bond Force Technical Bulletin, Tokuyama). Despite the contribution of such chemical interactions to the overall bonding mechanism of mild self-etch solutions, the adhesive potential of etch-and rinse systems remains superior on intact enamel.

Solvents have an important role in bond strength as well. All-in-one adhesives investigated in the present study utilize different solvents. While Bond Force and Xeno V have alcohol as a solvent, AdheSE One uses water. The C2-4 alkyl contained in Bond Force is claimed the ability to enhance the chemical stability of the solution, in comparison with acetone-based formulations (Bond Force Technical Bulletin, , Tokuyama Dental, Japan). Conversely, Xeno V features tert-butanol as a solvent. According to the manufacturer's claims, as the alcohol group is shielded by surrounding methyl groups of the tertiary group, the adhesive should be more chemically stable than those containing ethanol or iso-propanol. The use of tert-butanol combined with acryl resins with amide functions and inverse functionalization should lengthen the materials' shelf life (Xeno V Technical Bulletin, Dentsply, Konstanz, Germany). AdheSE One on the other hand has water as a solvent. The idea behind putting water as a solvent is to limitate phase separation. In the adhesive solutions containing acetone, the latter evaporates quickly and the separation of water from the adhesive monomers follows (AdheSE One Technical Bulletin, Ivoclar Vivadent, Schaan, Liechtenstein).

Although high early bond strength values can be achieved with some self-etching adhesives, their resistance to thermal and mechanical stresses over time has raised some concern.[14] In our study the bond strength of the adhesives was assessed at an early time, and no inference can be made from these data on the durability of the bond.

Conclusions

Within the limitations of this study the following conclusions may be drawn. The tested all-in-one adhesives did not differ in their bonding effectiveness to unground enamel.

The etch-and-rinse adhesive tested as control resulted in significantly higher bond strengths than the all-in-one adhesives.

Further investigations are necessary in order to provide clinical recommendations on whether all-in-one adhesives may be safely used on unground enamel.

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