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In vitro evaluation of bonding effectiveness to dentin of all-in-one adhesives

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ABSTRACT

Objectives: The study was aimed at assessing the bonding potential of all-in-one adhesives to dentin.

Methods: The microtensile bond strength of the all-in-one adhesives Bond Force (Tokuyama), AdheSE One (Ivoclar-Vivadent), and Xeno V (Dentsply) was measured in comparison with the etch-and-rinse system XP Bond (Dentsply). The ultrastructural characteristics of dentin–adhesive interface were observed under scanning electron microscope (SEM). Twenty human extracted third molars had their mid-coronal dentin exposed and ground with wet 600-grit SiC paper in order to create a standardized smear layer. Bonding procedures were performed according to the manufacturers' instructions and microtensile beams were obtained with the ''non-trimming'' technique. The bond strengths in MPa were statistically analyzed including pre-test failures as ''zero'' values (Kolmogorov–Smirnov test, Levene's test, One-Way ANOVA, Tukey's test $p < 0.05$).

Results: The following bond strengths were recorded in MPa (mean $\pm$ standard deviation): AdheSE One 31.7 $\pm$ 21.3; Xeno V 42.8 $\pm$ 26.4; Bond Force 43.3 $\pm$ 22.1; XP Bond 51.9 $\pm$ 18.6. The statistical analysis demonstrated that the bond strengths achieved by Bond Force and Xeno V were similar to that of the etch-and-rinse adhesive, whereas the bond strength of AdheSE One was significantly lower. A distinct hybrid layer with resin tags was seen only in XP Bond specimens. All-in-one adhesives demonstrated a rather superficial interaction with the dentin substrate.

Conclusions: Although the strongest bond to dentin was established by the etch-and-rinse system, however the all-in-one adhesives containing organic solvents reached bond strength levels that were comparable from a statistical point of view.

1. Introduction

The latest innovative efforts of dental adhesives manufacturers have been directed toward the reduction of handling steps, which allows to save chairtime and limits the chance for operator errors in the bonding procedure.[1–8] Thus, all-in-one adhesives have been introduced. Their adhesion is founded on the self-etch approach and involves a single-step application as the etching, priming, and bonding actions are implemented by the same solution.[1,2,4,6,7] The latter is a mixture of hydrophilic and hydrophobic monomers in water and organic solvents such as acetone and ethanol.[2,4–8] Carboxylic and phosphate groups in hydrophilic monomers act as proton donors in the partial demineralization of smear layer and have the potential to bond ionically with calcium of residual hydroxyapatite.[1–8] Hydrophobic monomers such as bisphenol A diglycidyl ether dimethacrylate (bis-GMA) and urethane dimethacrylate (UDMA) are responsible for the mechanical properties of the cured adhesive.[1,2,9,10]

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Table 1 – Chemical composition and instructions for use of the tested adhesives.

Adhesive	Chemical composition	Application
AdheSE One	Derivatives of bis-acrylamide, water, bis-methacrylamide dihydrogen phosphate, amino acid acrylamide, hydroxy alkyl methacrylamide, silicon dioxide, catalysts, stabilizers.	Application and agitation for 30 s; air dispersing until there is no water movement; light-curing for 10 s.
Batch K10655	pH 1.5	
Bond Force	Methacryloyloxyalkyl acid phosphate, HEMA, bis-GMA, TEGDMA, camphorquinone, purified water, alcohol.	Application and agitation for 20 s; air-drying firstly indirectly until there is no water movement and directly for 5 s; light-curing for 10s.
Batch 4T10787	pH 2.3	
Xeno V	Bifunctional acrylic amides, acrylamido alkylsulfonic acid, 'Inverse' functionalized phosphoric acid ester, acrylic acid, camphorquinone, butylated benzenediol, water, tertiary butanol.	Application twice; agitation for 20 s; air-drying for 5 s; light-curing for 20 s.
Batch 0703004017	pH < 2	
XP Bond	PENTA, TCB resin, UDMA, TEGDMA, HEMA, nanofiller, camphorquinone, DMABE, butylated benzenediol, tertiary butanol.	Application of DeTrey Conditioner 36 gel syringe for at least 15 s; rinsing for at least 15 s and drying with cotton; application of XP Bond and leaving the surface undisturbed for 20 s; air-blowing for at least 5 s; light-curing for 10 s.
Batch 0802001651		

Abbreviations: HEMA: 2-hydroxylethyl methacrylate; bis-GMA: bisphenol A diglycidyl ether dimethacrylate; TEGDMA: triethylene-glycol dimethacrylate; PENTA: dipentaerytritolpentacrylate; TCB resin: butan-1,2,3,4-tetracarboxylic acid, di-2-hydroxyethylmethacrylate ester; UDMA: urethane dimethacrylate; DMABE: 4-(dimethylamino)benzethyne.

While bonding to enamel of all-in-one adhesives remains a concern, their adhesion to dentin has achieved satisfactory levels.[8]

Nevertheless, several aspects in the formulation of these adhesives can still be improved, with a bearing on their shelf-life, etching potential, and bond durability. As a result of a constant improving effort by the manufacturers, the category of all-in-one adhesives is continuously supplemented by new products, that first require a pre-clinical *in vitro* screening.[2] In this perspective bond strength tests have recognized the potential to provide useful information, based on the concept that the stronger the bond, the better it will oppose to functional stress.[1]

Beside the quantitative assessment of adhesion contributed by bond strength tests, an appreciation of the quality of tooth–biomaterial interaction can be gained with microscopic observations of the interface.

The present study was indeed conducted with the aim of investigating the bonding potential to dentin of newly introduced all-in-one adhesives. Microtensile bond strength tests and scanning electron microscope imaging of the dentin–adhesive interface were performed for three all-in-one adhesives and one etch-and-rinse system. The latter was tested as control.

The null hypothesis that the adhesives measured similar microtensile bond strengths to dentin was subjected to statistical testing.

2. Materials and methods

Twenty human caries-free third molars, collected following informed consent approved by the Ethical Committee of the University of Siena, were stored in 0.5% Chloramine T solution at 4 °C for preventing bacterial growth for no longer than 3 months before they were used in the study.

Molars were randomly divided into four groups ($n = 5$). Groups were defined as follows, based on the adhesive/resin composite combination to be tested:

- Group 1: Bond Force/Estelite Σ (Tokuyama, Tokyo, Japan).
- Group 2: AdheSE One/Tetric EvoCeram (Ivoclar-Vivadent, Schaan, Liechtenstein).
- Group 3: Xeno V/Esthet-X (Dentsply DeTrey Kostanz, Germany).
- Group 4: XP Bond/Esthet-X (Dentsply DeTrey Kostanz, Germany).

Roots were cut off 2 mm below the cement–enamel junction with a water-cooled slow-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). On each tooth the occlusal portion was also removed, so as to expose a mid-coronal dentin substrate. In order to produce a standardized smear layer, dentin was ground with #600-grit wet SiC paper for 1 min.

Bonding procedures were performed following the manufacturers' instructions that are reported in Table 1.

The proprietary restorative resin composite of each adhesive was then used to incrementally build-up a crown of about 5 mm in height. Each added layer of resin composite was maximum 2 mm in thickness. It was adapted onto the flat dentin surface or onto the previously added increment with a clean plastic filling instrument, and singularly cured for 40 s with a halogen curing device (VIP, Bisco Inc., Schaumburg, IL, USA, 600 mW/cm^2).

The bonded specimens were stored for 24 h in 100% humidity at 37 °C prior to microtensile bond strength testing and SEM observations.