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ORIGINAL ARTICLE

Influence of acid-etching or double-curing time on dentin bond strength of one-step self-etch adhesive

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Abstract This *in vitro* study was undertaken to investigate the effects of an optional etching step with 35% phosphoric acid or double curing time on the shear bond strength (SBS) of one-step self-etch adhesives to dentin. A significant improvement in shear bond strength was demonstrated when dentin was acid-etched with 35% phosphoric acid prior to the application of one-step self-etch adhesives. The use of curing time double that was recommended by the manufacturers of the respective adhesives was capable of increasing the bond strength of the Single Bond Universal Adhesive but not that of the other two adhesives (Xeno® V$^+$ and AdheSE®One F VivaPen), indicating that the bond strength is adhesive-dependent. Therefore, the use of phosphoric acid-etching on dentin is highly recommended, followed by one-step self-etch adhesives.

1. Introduction

The interaction of current adhesives with the dentin substrate generally involves one of two approaches, either etch-and-rinse or self-etch. The etch-and-rinse approach is still the most reliable and effective approach to achieve efficient and stable bonding to both enamel and dentin, while the self-etch approach is the most promising, with reduced steps during the application process, minimized technique-sensitivity, and fewer risk errors during application.[1,2] Self-etch adhesives do not require a separate etching step and are subdivided into two types: two-step self-etch adhesives and one-step self-etch adhesives (all-in-one adhesives). Two-step self-etch adhesives are comprised of separate etch-primer and adhesive steps, while one-step self-etch adhesives combine the etch-primer and adhesive into a single application.[3]

Self-etch adhesives are composed of aqueous mixtures of acidic functional monomers, generally phosphoric acid esters with a pH higher than that of phosphoric acid, and can be classified according to their acidity into: ultramild (pH $\geqslant$ 2.5), mild (pH $\approx$ 2), intermediate (pH $\approx$ 1.5) and strong (pH $\leqslant$ 1).[4–7]

Self-etch adhesives are a complex mixture of components including acidic functional monomers, an association of

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hydrophilic and hydrophobic monomers, co-monomers, cross-linkers, initiators, water and solvents. One of the co-monomers most widely used is hydroxyethylmethacrylate (HEMA), which acts as a solvent and prevents hydrophilic and hydrophobic phase separation. The application of self-etch adhesives to tooth structure prior to the placement of resin composite materials will ensure maximum adhesion by improving monomer penetration into the hydrophilic dentin substrate, and improve the wettability of this substrate with resin components.[8–11]

Controversial effects, including improved bond strength, no effect, and reduced bond strength, have been reported with the use of the phosphoric-acid-etching step in conjunction with one-step self-etch adhesives.[12–14]

The permeability of self-etch adhesives has been correlated with the incomplete polymerization of resin monomers, and these adhesives may be rendered less permeable by the use of curing times longer than those recommended by the respective manufacturers.[15]

Due to this controversial issue, and the limited information about the effects of longer curing times on the bond strength of one-step self-etch adhesives, this study was conducted to evaluate the effects of:

(1) an optional etching step with 35% phosphoric acid on the shear bond strength (SBS) of one-step self-etch adhesives to dentin; and
(2) curing time double that was recommended by the respective manufacturers on the shear bond strength (SBS) of one-step self-etch adhesives to dentin.

The null hypotheses tested were that:

(1) there would be no effect of phosphoric acid on the shear bond strength (SBS) of one-step self-etch adhesives to dentin; and
(2) there would be no effect of double curing time on the shear bond strength (SBS) of one-step self-etch adhesives to dentin.

2. Materials and methods

A pilot study was conducted at the beginning of our experiment to investigate the reliability of the tests used.

2.1. Tooth selection

Ninety recently extracted, caries-free human molar teeth were used. All teeth were cleaned with an ultrasonic scaler and polished with non-fluoridated pumice in a rubber cup mounted in a slow-speed handpiece (KaVo EWL, No. 6412500, Biberach, Germany). After being cleaned and polished, the teeth were stored in distilled water with 0.05% thymol solution in a dark container at room temperature until being mounted.

The teeth selected for study were free of caries, cracks, abrasion facets, fluorosis and damage due to extraction. Each tooth was examined under a stereomicroscope (Stereoscopic Zoom Microscope SMZ 1000, Nikon, Tokyo, Japan) to eliminate teeth with cracks or hypoplastic defects.

2.2. Preparation of specimens

To obtain a flat dentin-bonding surface, we ground the occlusal surfaces of the teeth at slow speed with a 180-grit silicon carbide paper mounted on an Automata Machine (Jean-Wirtz

Table 1 Materials used in the study with their compositions, manufacturers and application procedures.

Material	Manufacturer	Material composition	Application procedure
Single Bond Universal	3M Deutschland GmbH, Carl-Schurz-Strasse 1, 41453 Neuss-Germany and 3M ESPE, St. Paul, MN 55144-1000, USA	MDP phosphate monomer, dimethacrylate resins, HEMA, vitrebond copolymer, filler, ethanol, water, initiators, silane, pH ≈ 2.7	Apply adhesive and rub it in for 20 s, then direct a gentle stream of air for 5 s until the adhesive no longer moves and solvent has evaporated, and light-cured for 10 s
Xeno® V^{+}	DENTSPLY DETREY GmbH, 78467 Konstanz, Germany	Bifunctional acrylate, Acidic acrylate, Functionalized phosphoric acid ester, water, tertiary butanol, initiators, stabilizer, pH ≈ 1.3	Apply adhesive and gently agitate for 20 s, then direct a medium stream of air for 5 s until the adhesive no longer moves and solvent has evaporated, and light-cured for 10 s
AdheSE®One F VivaPen	Ivoclar Vivadent AG FL-9494 Schaan, Liechtenstein	Bis-acrylamide, water, alcohol, Bis-methacrylamide dihydrogen phosphate, Amino acid acrylamide, Hydroxyl alkyl methacrylamide, Alkyl sulfonic acid acrylamide, silicon dioxide, Initiators, stabilizers, Potassium fluoride, pH ≈ 1.4	Adhesive must be brushed into the surface for 20 s, followed by a direct strong stream of air until a glossy immobile liquid film results, then light-cured for 10 s
Filtek Z 250 Micro-hybrid resin composite	3M ESPE Dental Products, St. Paul, MN, USA	Bis-GMA, Bis-EMA, UDMA, Zirconia/silica (78% w/w), barium glass, Ytterbium trifluoride, mixed oxide prepolymer	•Place Filtek Z 250 –shade A2. •Light-cured each increment for 40 s