

MICROTENSILE BOND STRENGTH OF SELF-ETCHING ADHESIVES TO DENTIN

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ABSTRACT

The aim of this study was to determine microtensile bond strength of two and one-step self-etching adhesive systems to dentin. 25 intact human third molars were used. Flat surfaces of mid-coronal dentin were exposed and the teeth were divided into 5 groups (n=5). Composite build-ups were made using the following self-etching systems / composite materials: 2-step self-etching system Contax / Ecusphere Carat (DMG), 2-step self-etching system AdheSE / Tetric Ceram (Ivoclar Vivadent), 1-step 2-components self-etching system Futurabond NR / Grandio (Voco) and 1-step 1-component self-etching adhesive G Bond / Gradia Direct (GC Corp.). Two-step etch-and-rinse system PQ Clear with Amelogen Universal composite resin (Ultradent) was used in the control group. Bond strength was assessed with the non-trimming microtensile technique. Statistical analysis was performed using the 1-way ANOVA, followed by the Tukey test for post-hoc comparisons (p<0.05). Investigated self-etching systems showed satisfactory values of microtensile bond strength to dentin after 24 hours. Two-step self-etching system AdheSE resulted in significantly higher bond strength than all the other groups (p<0.001). Microtensile bond strength of self-etching systems Contax, Futurabond NR and G Bond was comparable to the etch-and-rinse system PQ Clear.

Key words: microtensile bond strength, self-etching adhesives

APSTRAKT

Cilj rada bio je da se odredi jačina veze dvofaznih i jednofaznih samonagrizajućih adhezivnih sistema sa dentinom. Površine dentina su eksponirane na 25 intaktnih trećih molara i zubi su podeljeni u 5 grupa (n=5). Izradene su kompozitne nadogradnje korišćenjem sledećih samonagrizajućih adheziva / kompozitnih materijala: dvofazni samonagrizajući sistem Contax / Ecusphere Carat (DMG), dvofazni samonagrizajući sistem AdheSE / Tetric Ceram (Ivoclar Vivadent), jednofazni dvokomponentni samonagrizajući sistem Futurabond NR / Grandio (Voco) i jednofazni jednokomponentni samonagrizajući sistem G Bond / Gradia Direct (GC Corp.). Dvofazni adhezivni sistem sa potpunim nagrizanjem PQ Clear sa Amelogen Universal kompozitnim materijalom (Ultradent) korišćen je u kontrolnoj grupi. Jačina veze određena je testom otpornosti mikrouzoraka na

kidanje. Rezultati su statistički analizirani jednofaktorskom analizom varijanse (ANOVA) i Tukey testom ($p < 0.05$). Ispitivani samonagrizajući sistemi pokazali su zadovoljavajuće vrednosti jačine veze sa dentinom nakon 24 časa. Jačina veze dvofaznog samonagrizajućeg sistema AdheSE bila je značajno viša nego jačina veze u ostalim grupama ($p < 0.001$). Jačine veze samonagrizajućih sistema Contax, Futurabond NR i G Bond nisu se statistički značajno razlikovale od jačine veze sistema sa potpunim nagrizanjem PQ Clear.

Ključne reči: jačina veze, otpornost mikrouzoraka na kidanje, samonagrizajući adhezivi

INTRODUCTION

Since the beginnings of adhesive dentistry, scientists and manufacturers have been continuously challenged by a general trend to simplify the clinical procedures [1]. The most common approach was to shorten the adhesive system's application time and to reduce the number of steps [2, 3]. However, literature data shows that simplification does not always result in improved features and durability, regardless of the specific adhesive approach being etch-and-rinse or self-etch [4-6].

Three-step etch and rinse systems are considered to be a "gold standard" [6, 7]. Two step etch-and-rinse systems resulted in increased technique sensitivity [8, 9] compared to three-step etch-and-rinse systems, mostly as a result of joining the priming and bonding step into one, which required more hydrophilic formulations of solvents and monomers. Kanca [10] and Gwinett [11] have recommended to implement wet and dry bonding techniques in clinical procedures, depending on the type of solvent in etch-and-rinse adhesives. Nevertheless, the variables in the amount of wetness are hard to define in precise terms as a recommendation for the clinician, especially if wet bonding approach is used. Therefore, the true possibility for standardization of the etch-and-rinse procedure is questionable. There is a possibility for discrepancy between depth of etch and depth of monomer infiltration [12-14], which influences degradation processes [15, 16]. The above mentioned features have been recognized as the main drawbacks of etch-and-rinse technique.

Self-etching adhesive systems offer reduced application time and lower technique sensitivity compared with etch-and-rinse systems. There is a possibility for chemical interaction between functional monomers of some self-etching systems and tooth tissue [17], which may be beneficial against hydrolytic degradation to which adhesive systems are exposed over a long period of clinical service [18]. The conventional two-step self-etching systems have been followed with simplified products [19]. Firstly, one-step two-component products have been introduced, since acidic monomers needed to be kept separate from water needed for ionization and subsequent demineralization. Afterwards adhesives in which all the components for etching, priming and bonding are supplied in a single bottle (commonly referred to as all-in-one adhesives), were presented. A lot of research has focused on one-step adhesives, showing potential shortcomings, such as incompatibility with chemical/dual cured composite resins [20]. Despite the fact that etching and resin infiltration occur simultaneously, which was believed to ensure that no discrepancies are possible between these two processes, nanoleakage was observed in some systems [21, 22]. It

was also pointed that all-in-one adhesives are highly hydrophilic polymers that are permeable to water movement after polymerization [23-26].

The aim of this study was to determine microtensile bond strength of two and one-step self-etching adhesive systems to dentin. The null hypothesis tested was that bond strength to dentin is not influenced by the adhesive system.

MATERIALS AND METHODS

25 intact human third molars were used. Teeth were extracted due to orthodontic reasons and stored in distilled water for no longer than one month. The roots were cut off approximately 2mm below the cemento-enamel junction with a low speed diamond saw under water cooling (Isomet, Buehler Ltd., Lake Bluff, IL, USA). Then each tooth was sectioned again, parallel to the first section, to expose a flat dentin surface of mid-coronal dentin. Pulp tissue was gently removed with an excavator. Pulp chamber was acid-etched, and after adhesive was applied (Excite – Ivoclar Vivadent, Schaan, Liechtenstein), filled with flowable composite (Tetric Flow – Ivoclar Vivadent). Each dentin surface was viewed under a stereo light microscope (Nikon SMZ645) to make sure that it is free of enamel. Smear layer was created by wet sanding with 320-grit silicon carbide paper, to create a clinically relevant smear layer [27]. Teeth were randomly divided into 5 groups with 5 teeth per group, and composite build ups were made using following materials:

Group 1: Contax, Ecusphere Carat (DMG, Hamburg, Germany)

Group 2: Futurabond NR, Grandio (Voco, Cuxhaven, Germany)

Group 3: AdheSE, Tetric Ceram (Ivoclar Vivadent)

Group 4: G Bond, Gradia Direct (GC Corp, Tokyo, Japan)

Group 5: PQ Clear, Amelogen Universal (Ultradent, Salt Lake City, UT, USA)

Adhesive systems were used strictly according to manufacturers' instructions (Table 1). Their composition is reported in Table 2. Following adhesive treatment, four 1.5mm increments of composite resin were built up and individually light cured with Dental Curing Light Model "VIP", with the light intensity of 600 mW/cm² (Bisco, Schaumburg, IL, USA). After the specimens had been stored in distilled water at 37°C for 24 hours, 1mm sections were made with a low speed diamond saw perpendicular to the adhesive interface. These sections were further sectioned to produce beams with adhesive area approximately 1mm², according to the non-trimming version of microtensile technique [28]. The exact dimensions of each beam's adhesive area were measured using a digital calliper, to the nearest 0.01mm. Beams were fixed to a testing unit with cyanoacrylate glue (Super Attak Gel, Henkel Loctite Adesivi S.r.l., Milano, Italy) and pulled to failure under tension using a universal testing machine (Triax, Controls S.P.A., Milano, Italy) at a crosshead speed of 0.5mm/min. Both halves of each stick were examined under stereomicroscope to determine the type of failure (adhesive, cohesive in dentin/resin or mixed). Microtensile bond strengths were determined by computing the ratio of maximum load (N) by the adhesion area of particular beam. Four pretesting failures occurred with Futurabond NR specimens and one pretesting failure was seen with PQ Clear specimens. For these sticks, bond

strengths of 0 MPa were recorded and included in the statistical analysis. As bond strength data were normally distributed (Shapiro-Wilk test) and homogeneous in variances (Levene's test), a one-way ANOVA was performed to examine the effect of different adhesive systems. Post-hoc multiple comparisons were performed using the Tukey test, with the significance level set at $p < 0.05$.

RESULTS

The results of microtensile bond strength testing are reported in Table 3. ANOVA test showed statistically significant differences between the groups. Post-hoc Tukey test indicated statistically significant differences between AdheSE and all the other groups ($p < 0.001$). No statistically significant differences were identified comparing Contax, Futurabond NR, G-Bond and PQ Clear between each other. The most frequent type of failure was adhesive in all investigated adhesive systems (Table 4).

DISCUSSION

In order to create smear layer in laboratory conditions, 600-grit silicon carbide paper is most commonly used. This procedure results in smear layer which is thinner than the smear layer created in clinical conditions by the use of diamond or carbide burs [27]. The adhesive strategy of self-etching systems relies on smear layer dissolution. Therefore, for laboratory testing of self-etching adhesives, it was recommended in the literature to form a thicker smear layer that more closely resembles the one created clinically, in order to avoid false positive results [29-31]. Oliveira et al. demonstrated that 320-grit silicon carbide paper creates such a clinically relevant smear layer, which is similar to a smear layer formed by a carbide bur. Having in mind that carbide burs are most frequently used in clinical conditions, smear layer on dentin surfaces in this study was created by wet-sanding with 320-grit silicon carbide paper.

The method utilized for bond strength testing was the microtensile bond strength test that was reported to be suitable for the evaluation of interfacial bond strengths on areas below 1 mm^2 [32]. In particular, the nontrimming variant of the technique was adopted to reduce the number of premature failures during specimen preparation, in comparison with the 'more aggressive' trimming variant of the microtensile bond test [33].

Adhesive systems in which primer and adhesive are joined together in one bottle are frequently referred to as "simplified adhesives". These are two-step etch-and-rinse systems and one-step self-etching systems. Three simplified adhesives were investigated in this study: two one-step self-etching systems (Futurabond NR and G-Bond) and two-step etch-and-rinse system PQ Clear as the control. Besides being easier to use, simplified adhesives are also more hydrophilic compared to two-step self-etching adhesive systems and three-step etch-and-rinse systems where adhesive is applied in a separate clinical step. Simplified adhesives usually result in lower bond strengths compared to adhesive systems in which primer and adhesive are applied in separate phases [4, 5]. Therefore, slightly lower bond strengths for G-Bond and Futurabond NR in comparison to AdheSE and Contax were expected. Futurabond NR

bond strength was the lowest, but not significantly different from Contax, G-Bond and from simplified etch-and-rinse adhesive PQ Clear in the control group.

Lower bond strengths of simplified adhesives are usually explained as a consequence of increased hydrophilicity due to joining primer and adhesive in one bottle [34]. The increased concentration of hydrophilic resin monomers in simplified etch-and-rinse adhesives may preclude the complete water removal after rinsing off the phosphoric acid and drying. Remnants of water may remain »trapped« in the adhesive layer. Studies have shown that water may exist in the adhesive layer as free, within the collagen matrix, or bound, if it forms hydrogen bonds with hydrophilic resin monomers [21, 24]. In areas where water remains polymerization may be incomplete. Also, these areas represent porous regions which may facilitate hydrolytic degradation of adhesive interface. Therefore, it is very important that water and solvents are completely removed following the adhesive application. In order to accomplish this goal, manufacturers recommend air drying of various durations and intensities. Yiu et al. have investigated the percentage of water and solvent that remains in the adhesive after drying, simulating clinical conditions [35]. It was reported that the percentage of remaining solvent increases with the increasing hydrophilicity of the adhesive.

On the other side, adhesive systems in which adhesive is applied in a separate step (three-step etch-and-rinse systems and two-step self-etching systems) were reported to provide better results [36-38]. Therefore, higher bond strengths were expected for two-step self-etching adhesive systems investigated in this study – AdheSE and Contax. AdheSE microtensile bond strength was the highest and significantly higher than bond strengths in other groups, which led to the rejection of the null hypothesis. Sensi et al reported that AdheSE bond strength was comparable to two-step etch-and-rinse system [39]. However, their study assessed shear bond strength of self-etching adhesives and the results cannot be directly compared to microtensile bond strength values. Goracci et al found AdheSE microtensile strength to dentin to be significantly lower in comparison to two-step etch-and-rinse system Excite (Ivoclar Vivadent). A different testing unit might account for the discrepancy with the results of the present investigation [40]. Two-step self-etching system Contax microtensile bond strength was significantly lower in comparison to AdheSE. Contax manufacturer states that »there is no need to dry the primer upon application«. Nevertheless, in the present study Contax primer was gently air dried to remove excess of water, taking care that this step leaves dentin surface covered with primer and shiny. Contax bond also contains water, unlike AdheSE adhesive (Table 2). It is possible that this difference in adhesive layer hydrophilicity resulted in significant difference in bond strengths of these two adhesive systems. AdheSE contains water only as a solvent in its primer for which manufacturer recommends strong air drying in order to completely remove water. AdheSE bond doesn't contain any solvent. It is possible that the hydrophobic adhesive layer that AdheSE forms stabilizes its interface with dentin and contributes to its high bond strength.

The most frequent type of failure was adhesive in all groups (Table 4). Cohesive failures in dentin were observed in three AdheSE specimens and one PQ Clear specimen, at bond strengths between 30 and 42 MPa. Cohesive failures in dentin during microtensile bond strength testing occur much less frequently than during

conventional (shear and tensile) bond strength tests [32]. Dentin tensile strength varies with dentin depth [41]. In superficial dentin it is approximately 61.6 MPa, in mid-coronal dentin 48.7 MPa, while in deep dentin tensile strength is 33.9 MPa [41]. Based on these results, cohesive failures in dentin may be expected at bond strength values that are higher than dentin tensile strength in the specific region. In the present study an effort was made to prepare samples so that the adhesive interface lays in mid-coronal dentin. It is possible that in some specimens the adhesive interface was formed in somewhat deeper dentin which resulted in the rare occurrence of cohesive dentin fractures. Cohesive fractures in composite occurred at high bond strength values that were most frequently above 40 MPa.

CONCLUSIONS

The investigated self-etching systems showed satisfactory values of microtensile bond strength to dentin after 24 hours. The highest values of microtensile bond strength were obtained with a two-step self-etching system AdheSE. Simplified one-step self-etching adhesive systems used in this study showed lower values of bond strength, but without a statistically significant difference compared to the etch-and-rinse system PQ Clear.

Table 1 - Adhesive systems used and application method

Adhesive system	Etchant	Primer	Adhesive	Manufacturer	Batch number
AdheSE	Apply primer and, when thoroughly coated the surface, brush into for 15 sec (total reaction time: 30 sec). Disperse excess amounts with a strong stream of air.		Apply bond, beginning at dentin. Disperse with a very weak stream of air. Light cure.	Ivoclar Vivadent	G 05739
Contax	Work primer into tooth structure for 20 s		Work adhesive into primed tooth structure for 20 s, thin out, light cure.	DMG Hamburg	529426
Futurabond NR	Mix 1 drop of liquid A and 1 drop of liquid B for 5 sec. Apply mixture to tooth and massage for 20 sec. Dry with a faint air jet for 5 sec. Light cure.			Voco	0482
G-Bond	Shake bottle well. Apply to tooth surfaces. Leave undisturbed for 10 sec. Dry thoroughly for 5 sec under maximum air pressure, to form a thin film. Light cure.			GC Corp.	0405241
PQ Clear	Etch enamel and dentin for 15 seconds (UltraEtch). Rinse. Blow excess water off, without desiccating.		Apply adhesive and air thin. Maintain a high glossy surface with no dry spots. Light cure	Ultradent	64QM

Table 2: Composition of adhesive systems used

Adhesive system	Etchant	Primer	Adhesive
AdheSE	(primer) Phosphonic acid acrylate Bis-acrylamide Water Initiators Stabilizers		(adhesive) Dimethacrylates Hidrohy-ethyl methacrylate Highly dispersed silicon dioxide Initiators Stabilizers
Contax	(primer) Water Maleic acid Sodium fluoride		(adhesive) Hydrophilic, acidic Bis-GMA-based resin matrix Water Additives, catalysts
Futurabond NR	Liquid A: Polyfunctional adhesive monomers (Methacroyl-Phosphorus-Acid-Ester, Methacroyl-Carbon-Acid-Ester), Dimethacrylates, Functionalized SiO ₂ -nano-particles, Initiators Liquid B: Ethanol, Water, Hydrophilic adhesive monomers, Fluorides		
G-Bond	4-MET Phosphate ester Urethane-dimethacrylate Acetone Water Silica fine powder Catalyst		
PQ Clear	35% orthophosphoric acid		Methacrylate resins Ethanol solvent Silicate fillers (filler content 8%) Camphorquinone Initiator (proprietary)

Table 3: Microtensile bond strength to dentin (μ TBS). Numbers are means. Values in brackets are standard deviations. Different superscript letters indicate statistically significant differences.

Adhesive system	μ TBS (MPa)	Surface area (mm ²)	Number of sticks per group
1. Contax	30.35 [9.11] ^a	1.07 [0.08]	37
2. AdheSE	47.44 [15.89] ^b	1.07 [0.08]	36
3. Futurabond NR	24.90 [14.67] ^a	0.95 [0.10]	35
4. G-Bond	31.00 [14.03] ^a	1.11 [0.11]	32
5. PQ Clear	32.69 [13.63] ^a	0.99 [0.12]	33

Table 4: Failure distribution

Adhesive system	Adhesive	Cohesive in dentin	Cohesive in resin	Mixed
1. Contax	75.67%	0%	10.81%	13.51%
2. AdheSE	47,22%	8,33%	19,44%	25.00%
3. Futurabond NR	71.43%	0%	2.86%	25.71%
4. G-Bond	56.25%	0%	12.50%	31.25%
5. PQ Clear	60.61%	3.03%	6.06%	30.30%

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