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Assessment of resin-dentin interfacial morphology of two ethanol-based universal adhesives: A scanning electron microscopy study

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ABSTRACT

Objective: The objective of this study was to assess the resin-dentin interfacial morphology created by two universal adhesives using scanning electron microscopy (SEM). **Materials and Methods:** The occlusal surfaces of ten (n = 5) molars were reduced to expose a flat surface of dentin. Two universal adhesives, Scotchbond Universal Adhesive and Tetric N-Bond Universal, were independently applied to air-dried dentin. Light-cured resin-based composite restorative materials were used to incrementally build a composite "buildup." The specimen was sectioned mesiodistally to expose the resin-dentin interface. The inner surfaces of the specimens were polished. Samples were immersed in hydrochloric acid and then rinsed using distilled water. This was followed by immersion of the samples in 1% sodium hypochlorite solution. Then, samples were thoroughly rinsing with distilled water. Dehydration of samples was performed using ascending concentration of ethyl alcohol. Prepared samples were observed SEM at magnifications ×1500 and x4000. **Results:** Both universal adhesives could penetrate dentin-forming well-defined resin tags, lateral branches as well as a uniform hybrid layer. **Conclusions:** Two tested universal adhesives applied in self-etch mode can infiltrate into dentin-producing high-quality interfacial morphology. Similar interfacial morphology may be due to the similarity in composition and application mode.

Key words: Adhesive, hybrid layer, resin tags

INTRODUCTION

Current dental adhesive systems are categorized into two main classes on the basis of the technique of bonding to dental substrates: the etch-and-rinse adhesives that require the application of an acid etchant to the dental substrate before use and the self-etch adhesives (SEAs) for which the use of acid etchant is not mandatory.[1,2]

SEAs can either be two-step adhesives in which the application of an acidic primer is followed by that of a hydrophobic bonding agent, or one-step adhesives. The one-step SEAs can be further subdivided into two types: two-component adhesives that require mixing before use and single-component adhesives.[1] Thus, the single-component, one-step SEAs are considered as "all-in-one" adhesives, as they combine "conditioning," "priming," and "application of the bonding agent," and require no mixing.[1] Low technique sensitivity, ease of use, and simplicity

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have caused SEAs to be widely used in restorative practice.[3,4]

Based on micromechanical adhesion mechanism,[5] a strong bonding between adhesives and dental substrates may depend on the ability of adhesive monomers to penetrate the dental substrates.[6] The self-etch bonding strategy involves the penetration of acidic monomers beyond the smear layer into mineralized dentin by partially dissolving hydroxyapatite to generate a resin-infiltrated zone with minerals incorporated.[7] However, if SEAs are not able to penetrate to demineralize the dentin to form a hybridized zone, their bonding performance may be negatively affected.[8] Universal or multimode adhesives may represent the latest generation of dental adhesives.[9-11] Universal adhesives are designed to bond to tooth structures through the etch-and-rinse or the self-etch bonding strategies, using the same adhesive.[10] Despite their versatility, universal adhesives are primarily SEAs.[12] Several universal adhesives are currently available. However, detailed information on their ability to penetrate or interact with the dental tissues may be required. Therefore, the purpose of this *in vitro* investigation was to assess the resin-dentin interfacial morphological characteristics of two ethanol-based universal adhesives, namely, Scotchbond Universal Adhesive (SBU) and Tetric N-Bond Universal (TNU) using scanning electron microscopy (SEM).

MATERIALS AND METHODS

Specimen preparation

Ten permanent human third molars, extracted for surgical reasons, were used after approval from the Local Ethics Committee. The occlusal surfaces of molars were reduced using a diamond saw under water cooling, to expose a flat surface of mid-coronal dentin. The dentin surfaces were inspected using a magnifying loop to ensure that there were no remnants of enamel. Then, they were polished with # 600 grit silicon carbide paper under running water and were subjected to ultrasonic cleaning for 10 min.

The two universal adhesives, SBU and TNU, were applied, separately, to the air-dried dentin surfaces [Table 1] with scrubbing motion, using micro brushes, for 20 s. These were light cured for 10 s, using a light-emitting diode (LED) light-curing unit (SmartLite Max LED Curing Light, Dentsply Caulk) operated at 900 mW/cm^2. Immediately after light curing of adhesives, the resin-based composite Tetric N-ceram was used to incrementally build a composite "buildup" that was 3–4 mm in height, with peripheral borders maintained entirely in the dentin. Each increment (approximately 1 mm) was light cured for 20 s from approximately 1 mm. After composite buildup, each specimen was sectioned mesiodistally to expose the resin-dentin interface.

Specimen preparation for scanning electron microscopy examination

The inner surfaces of specimens were polished using polishing discs (Flexi-D EVE Ernst Vetter GmbH) with a speed of 5000 rpm under water cooling. Samples were immersed in 6 mol/L hydrochloric acid for 30 s to remove minerals within the hybrid layer that was not protected by resin infiltration. The specimens were rinsed, using distilled water, for 1 min. This was followed by immersion of the specimens in 1% sodium hypochlorite solution for 10 min to dissolve all exposed collagen beneath the hybrid layer. Then, the specimens were thoroughly rinsed with distilled water for 5 min. Dehydration of the specimens was performed using ascending concentrations of ethyl alcohol (25% for 20 min, 50% for 20 min, 75% for 20 min, 95% for 30 min, and 100% for 60 min).[13] The samples thus prepared

Table 1: The materials' compositions, manufacturers, lot no., and manufacturers' instructions

Material/manufacturer	Composition	Manufacturer's instructions
SBU 3M ESPE, St. Paul, MN, USA	MDP, dimethacrylate, HEMA, vitrebond copolymer, filler, ethanol, water, initiators, and silane	Apply one drop agitate for 20 s Gently air dry for 5 s Photopolymerize for 20 s
TNU Ivoclar Vivadent; Schaan, Liechtenstein	MDP, MCAP, HEMA, D3MA water, ethanol, highly dispersed silicon dioxide, initiators, and stabilizers	Scrub for at least 20 s Disperse with oil- and moisture-free compressed air until a glossy, immobile film layer Light cure for 10 s
Tetric N-Ceram Ivoclar Vivadent; Schaan, Liechtenstein	Urethane dimethacrylate, Bis-GMA, ethoxylated Bis-EMA, triethylene glycol dimethacrylate, barium glass, ytterbium trifluoride, mixed oxide, silicon dioxide, prepolymers, additives, stabilizers, catalysts, and pigments	

SBU: Scotchbond Universal Adhesive, TNU: Tetric N-Bond Universal, HEMA: Hydroxyethyl methacrylate, MDP: Methacryloyloxydecyl dihydrogen phosphate, Bis-EMA: Ethoxylated bisphenol-A dimethacrylate, Bis-GMA: Bisphenol A-glycidyl methacrylate were observed with SEM (QUANTA FEG 250, FEI Company, The Netherlands) at magnifications ×1500 and ×4000.

RESULTS

SEM examinations (magnifications ×1500 and ×4000) of the resin-dentin interfaces created by the two universal adhesives showed similar interfacial morphological features [Figures 1 and 2]. SBU and TNU could penetrate the dentin-forming well-defined resin tags, lateral branches, and a uniform hybrid layer. Resin tags produced by the SBU showed anastomosis feature. The resin tags created by TNU were relatively shallower but more uniform compared to those of SBU. Predominant features of the two adhesive-dentin interfaces are listed in Table 2.

DISCUSSION

The ability of the adhesive systems to infiltrate into dental substrates and form high-quality hybridization is one of the requirements for adequate bonding.[14-16] Although interpretation of resin tag formation is controversial,[17] the morphology, length, and density of resin tags are used for evaluation of the efficiency of adhesive systems.[18,19]

Table 2: Predominant features of the respective adhesives interfaces observed by scanning electron microscopy

Adhesive	Feature		
	Resin tag formation	Lateral branches	Hybrid layer
SBU	Present Not uniform Deeper Anastomosed	Present	Uniform
TNU	Present Uniform Shallower Dense	Present	Uniform

SBU: Scotchbond Universal Adhesive, TNU: Tetric N-Bond Universal

Despite significant improvements in adhesive systems, the resin-tooth interface remains the weakest area of composite resin restorations.[20] In this study, the interfacial morphological characteristics of two ethanol-based universal adhesives, used in the self-etch mode, were evaluated using SEM. Both universal adhesives could penetrate the dentin-forming well-defined resin tags and lateral branches. Universal or multimode adhesives are primarily SEAs.[12] Both tested adhesives are one-step SEAs; SEAs demineralize and infiltrate the tooth structure simultaneously.[1] During substrate penetration, the acidic monomers of adhesives are gradually buffered by the mineral content of the substrate, losing their ability to further etch dentin.[8,21] The bonding ability of one-step SEAs depends on their specific composition. SEAs consist of resin monomers with functional groups, hydrophilic monomers, hydrophobic monomers, solvents, fillers, and initiators.[22,23] Interfacial morphology of both adhesives may also be affected by similarity of their compositions. Both tested adhesives were water-containing, ethanol-based, and hydroxyethyl methacrylate (HEMA)-containing adhesives. Water is essential to ionize the acidic monomers and trigger the demineralization process.[24] However, due to the low vapor pressure of water, it is usually combined with organic solvents of higher vapor pressure, in the adhesives, to ensure its evaporation.[24] Water-ethanol combination in both adhesives may also help to dilute the viscous monomers and facilitate its infiltration into dentin.[25]

HEMA is a conventional hydrophilic methacrylate monomer, incorporated into dental adhesive compositions[24] due to its hydrophilicity, which makes it an excellent adhesion promoter.[5] HEMA may enhance wetting properties and dentin penetration efficacy of adhesives.[26-28] The presence of HEMA in both adhesives plays a key role in their similar interfacial morphology. The tooth interfacial morphological features of SEAs may depend on their pH.[29] Based on their aggressiveness or acidity, SEA can be classified

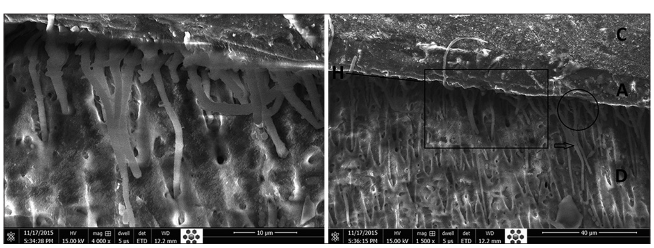

Figure 1: Scanning electron microscopy micrograph of the resin-dentin interface created by the Scotchbond Universal Adhesive. C: Composite, A: Adhesive, D: Dentin, H: Hybrid layer, Arrow: Resin tags (anastomosed), Rectangle: Section shown at magnification ×4000. Rounded: Lateral branches (×1500 [right side] and ×4000 [left side])

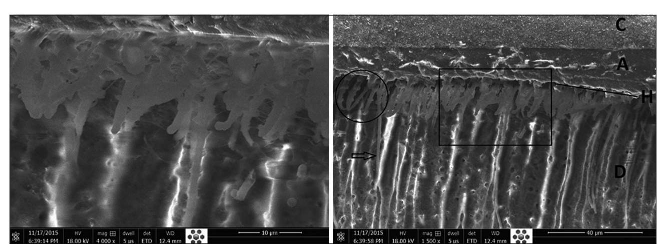

Figure 2: Scanning electron microscopy micrograph of the resin-dentin interface created by the Tetric N-Bond Universal. C: Composite, A: Adhesive, D: Dentin, H: Hybrid layer Arrow: Resin tag, Rectangle: Section shown at magnification ×4000. Rounded: Lateral branches (×1500 [right side] and ×4000 [left side])

into strong (pH ≤ 1), intermediately strong (pH 1–2), mild (pH ≈ 2), or ultra-mild (pH > 2.5).[1,25] In this study, SBU and TNU have pH of 2.7 and 2.5–3.0, respectively. Thus, the two tested adhesives can be mild or ultra-mild SEAs. This may also explain the similar interfacial morphology created by both adhesives.

In addition to the compositional factors, the application mode may also have influenced the interfacial morphology of adhesives. As recommended by manufacturers of the adhesives, scrubbing or vigorous application was performed for 20 s. It is believed that active application of adhesive improves infiltration of resin monomers into the dental substrates.[7,30,31] Active agitation is essential for SEAs as it may assist smear layer removal and improve the contact of the acidic monomers with the tooth surface.[31] In contrast, the interfacial morphological differences may be attributed to the differences in the resin content of the two universal adhesives. However, further investigations may be required to confirm this speculation.

CONCLUSIONS

The two tested universal adhesives, applied in self-etch mode, can infiltrate into dentin-producing high-quality interfacial morphology. The similar interfacial morphology may be due to similarity in composition and application protocol.

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Conflicts of interest
There are no conflicts of interest.

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