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Evaluation of Microtensile Bond Strength of Universal Self-etch Adhesive System to Wet and Dry Dentin

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Abstract

BACKGROUND: The durability of dentin-resin interfaces with the universal adhesive system is a crucial characteristic with chemical interactions between the exposed collagen and the adhesive monomers, but it is still compromised with wet and dry mode.

AIM: The present study evaluated the effect of dentin wetness and solvents containing of one-step self-etch adhesives on the microtensile bond strength (μTBS) of dentin at different storage times.

METHODS: Occlusal dentin of 54 extracted human molars was exposed. Each adhesive agent was applied according to manufacturer instructions to wet and dry dentin surfaces. Composite resin was incrementally built up. Bond strengths to dentin were determined using the μTBS test after water storage for 24 h, 1 month, and 6 months. One-way ANOVA was used to compare between more than two non-related samples. The significance level was set at $p \leq 0.05$.

RESULTS: Dryness of dentin increases the μTBS with solvent-containing adhesives while decrease the μTBS with solvent-free adhesive. There was an increase in microtensile bond strength values in the case of ethanol water-based self-etch adhesive over time. No statistically significant difference was found among different storage times regarding μTBS for solvent-free adhesive, while a statistically significant difference was found among different storage times in μTBS for solvent-containing adhesives.

CONCLUSION: Universal adhesive systems improve the durability and stability of dentin bond strength.

Introduction

The achievement of durable bonds with high bond strength in the tooth/restoration interface is the ultimate goal of dental adhesive systems [1]. Enamel and dentin bonding have progressed from multistep systems to simplification of the application procedure to reduce technique-sensitivity and working time. The most simplified adhesive system is the all-in-one type which includes all components in one bottle [2]. Self-etch adhesives contain a high concentration of solvents which must be eliminated after completing their function. Residual solvent may lead to deterioration of the adhesive interface between tooth structure and composite resin by interfering with resin polymerization [3]. The use of solvent-free adhesives may enhance the adhesion because it is free from the residual solvent. Solvent-free adhesives are hydrophobic and dense; these have less water sorption and less solubility than solvated resin blends. Complete elimination of solvent through air-drying is difficult to achieve. Consequently, some residual solvent remains trapped in the adhesive [4]. Bond strength testing remains a very important method used to screen new products and evaluate the influence of experimental variables. Adhesive performance on enamel and dentin may be quantified using several methodologically distinct approaches, roughly divided into macro or micro setups, depending on the size of the bonded area [5]. To improve stress distribution and the range of bond strength values, shear and tensile tests were almost completely replaced by the microtensile (μTBS) and microshear bond strength tests. A better stress distribution can be accomplished in smaller specimens since the number of voids and stress-raising factors is lower than that possibly occurred in larger areas such as in conventional shear or tensile bond strength tests [6].

Materials and Methods

A total of 54 freshly extracted human molars were selected. Each tooth was embedded vertically in the specially fabricated cylindrical plastic mold to the level of the cementoenamel junction of the tooth. Grinding machine was used to wet grind 2 mm from the occlusal surface to expose the dentin using a grit carborundum disc. The dentin surface was further abraded using a #600 grit wet silicon carbide abrasive paper for 60 s under running water to produce a polished surface. Teeth were divided into three main groups (18 teeth each) according to the type of one-step self-etch adhesive system; Group 1: Ethanol-water-based adhesive single bond universal (SB), Group 2: Acetone water-based adhesive G-aenial Bond (GB), and Group 3: Solvent-free adhesive Bond-1SF (SF). All materials used in this study are listed in Table 1. Each adhesive agent was applied according to manufacturer instructions to wet and dry dentin surface; then, resin composite was incrementally built up.

Wet dentin surface

Dentin surface was rinsed with distilled water and blot-dried with cotton pellet to remove the excess of water.

Dry dentin surface

Dentin surface was dried gently with oil-free compressed air for 10 s at 2 cm away from the dentin surface. The specimens were stored in distilled water at 37°C in an incubator with 100% humidity at different storage times (1 day, 1 month, and 6 months) until microtensile bond strength testing was performed. The specimens were sectioned using IsoMet 4000 microsaw device to produce multiple beam-shaped sticks with dimensions of 1 × 1 × 8 mm. The μTBS was assessed using a universal testing machine.

The mean and standard deviation values were calculated for each group. Data were explored for normality using Kolmogorov–Smirnov and Shapiro–Wilk tests and showed parametric (normal) distribution. Independent sample t-test was used to compare between two non-related samples. Repeated measure ANOVA was used to compare between more than two related samples. One-way ANOVA was used to compare between more than two non-related samples. The significance level was set at $p \leq 0.05$. Statistical analysis was performed with IBM® SPSS® Statistics Version 20 for Windows.

Results

a) For wet dentin condition
The highest mean values of microtensile bond strength (19.50 ± 0.97 MPa) were recorded in case of ethanol-water-based self-etch adhesive (single bond universal adhesive) at 1 day storage time, while the lowest mean values of microtensile bond strength (11.50 ± 0.61 MPa) were recorded in case of solvent-free self-etch adhesive (Bond-1 SF) at 6 months storage time.

b) For dry dentin condition
The highest mean values of microtensile bond strength (26.40 ± 0.57 MPa) were recorded in case of ethanol-water-based self-etch adhesive (single bond universal adhesive) at 1 day storage time, while the lowest mean values of microtensile bond strength (8.60 ± 4.23MPa) were recorded in case of solvent-free self-etch adhesive (Bond-1 SF) at 6 months storage time.

Comparisons between wet and dry dentin condition

A statistically significant difference ($p \leq 0.001$) was found in microtensile bond strength between wet and dry dentin conditions in 1 day, 1 month, and 6 months storage periods in Bond-1 SF, single bond universal, and G-aenial Bond. These are shown in Table 2. No statistically significant difference was found among different storage times in μTBS for SF. On the other hand, a statistically significant difference was found among different storage times in μTBS for solvent-containing adhesives. At 6 months storage time, no statistically significant difference was found among different adhesives agents regardless of the dentin condition.

Table 1: Materials used in this study

Materials	Specification	Composition	Manufacturer and batch number
Universal adhesive	Ethanol-water-based one-step self-etch adhesive system	MDP* phosphate monomer dimethacrylate resins, HEMA**, Vitrebond Copolymer, filler, initiators, silane, ethanol, water	3M ESPE St. Paul, MN, USA 692513 http://www.3m.com
G-aenial bond	Acetone water-based self-etch adhesive system	4-META#, anhydride 5-10% , acetone 30-40%, water 15-20% , dimethacrylate 15-20% , phosphoric acid ester monomer 15-20% , silicon dioxide 1-5% , photo initiator	GC CORPORATION, Tokyo, Japan 1410101 http://www.gcamerica.com
Bond-1 SF	Solvent-free, one-step self-etch adhesive	Mixture of UDMA±, TEGDMA±±, HEMA & 4-META resins, silane-treated barium borosilicate glasses, silica with initiator, stabilizers and UV+ absorber, organic and/or inorganic pigments, and opacities	Pentron Clinical, Orange, CA, USA 5603010 http://www.pentron.com
Filtek Z250 XT	Nanohybrid filled composite resin	Filler: zirconia/silica (82% by weight (68% by volume) Matrix: BIS-GMA≠, UDMA, BIS-EMA*#, PEGDMA and TEGDMA surface-modified zirconia/silica 20 nanometer	3M ESPE St. Paul, MN, USA 692513 http://www.3m.com

*MDP: Methacryloyloxydecyl dihydrogen phosphate glycidyl methacrylate, **HEMA: Hydroxyethyl methacrylate, #4-META: 4-Methacryloxyethyl trimellitate anhydride, ±UDMA: Urethane dimethacrylate, ±±TEGDMA: Triethylene glycol dimethacrylate, +UV Ultraviolet, Bis-GMA≠: Bisphenol glycidyl methacrylate, BIS-EMA*#: Ethoxylated bisphenol A glycol dimethacrylate.

Table 2: Comparison between microtensile bond strength of the adhesive agents with dentin condition and storage times

Dentin condition	Adhesive system / Storage time	Bond-1 SF		Single-bond universal		G-aenial bond		p value
		Mean	SD	Mean	SD	Mean	SD	
Wet	One day	13.60[a]	2.31	19.50[b]	0.97	14.40[c]	1.11	<0.001*
	One month	12.50[a]	1.85	17.20[b]	1.43	12.70[a]	0.76	0.005*
	Six months	11.50[a]	0.61	13.00[a]	0.86	11.50[a]	1.30	0.101ns
Dry	One day	10.80[a]	5.50	26.40[b]	0.57	21.40[c]	0.80	<0.001*
	One month	9.50[a]	4.05	20.10[b]	0.63	15.76[c]	1.47	<0.001*
	Six months	8.60[a]	4.23	16.00[b]	1.22	13.18[b]	1.64	0.020*

Mean with different letters in the same row indicate statistically significance difference *; significant ($p<0.05$) ns; non-significant ($p>0.05$).

Discussion

One of the main problems regarding adhesive dentistry is the degradation of the resin-dentin bond by water over a period. Bonding to enamel remains the simplest and most reliable of all adhesive procedures, while bonding to the dentin is difficult mainly due to the heterogeneous composition of the dentin [7]. The results of the present study revealed that solvent-containing self-etch adhesives provided higher initial μTBS than solvent-free self-etch adhesive system. The presence of water as a solvent in the composition of self-etch adhesive systems is necessary to ionize the acidic monomers and trigger the demineralization process, while the other cosolvents like ethanol are added to form an azeotropic mixture with water. This mixture accelerates the removal of excess water by means of air-drying and also promotes the diffusion of monomers into the dentin [8]. On the other hand, the solvent-free adhesive system failed to penetrate the dentin microstructures and to form a sufficient hybrid layer, which affected the bond quality of resin composite to dentin [9]. Moreover, the solvent-free adhesive system does not have solvents in its composition so that the resin tags in solvent-free adhesive system seem to be less numerous and shorter than solvent-containing adhesives, which might be attributed to less chance of the adhesive to penetrate into the demineralized dentin [10]. These explanations are confirmed with the results obtained by the study of Moszner *et al*. [9] which concluded that elimination of the solvent from self-etch adhesive systems may be decrease or hindered the infiltration of adhesive components into dentin, which leads to debility of hybrid zone formation and decrease of the bond strength to the dentin.

Regarding the dentin hydration, the present results showed significant increase in the μTBS of SF with wet dentin, this may be due to the composition of SF which does not contain water, so in the dry dentin, the adhesive could not remove the smear layer effectively. There would be poor penetration of bonding resin into the underlying dentin, which eventually leads to poor hybrid layer formation [11]. Moreover, the water is required to dissociate these monomers to release the hydronium ions (H_3O+), which responsible for demineralization [2]. The result of this study is in agreement with the results obtained by the study of Umino *et al*. [12] which suggested that prolonged air-drying of the dentin surface removed water and decreased the bond strengths of SF.

While for solvent-containing adhesives, the measures of μTBS to dry dentin were significantly greater than to wet dentin. The moisture on the wet dentin surface may dilute the adhesives, thus decrease the etching effect of the adhesives, which might decrease the potential for hybridization and finally lead failure of the resin composite bond strength [13]. Moreover, the excess water could decrease the bond strength due to competition with monomers for infiltration into the substrate. Water might reduce the degree of conversion and interfere with polymerization. As a result, unpolymerized acidic monomers could continue to etch the dentin, which will lead to decrease the bond strength [14]. This is in agreement with the results obtained by the study of Lima *et al*., [15] where they found that the adhesives applied to dry dentin showed higher bond strength than blot dry dentin.

According to the storage, the μTBS of all adhesives agents decreased with time. This may be related to the ability of simplified resin bonding systems to absorb water that plays an important role in hydrolytic degradation of resin-dentin bonds after long-term water storage [16]. Furthermore, the water can infiltrate and decrease the mechanical properties of the polymer matrix by swelling and reducing the frictional forces between the polymer chains, a process is known as "plasticization" [17].

Whatever, the SF showed no significant decrease in the mean bond strength after aging. This may be due to the unique composition of this adhesive, which contains neither water nor organic solvents in the ingredients to eliminate technical issues in terms of evaporation of solvents and concerns for the durability of resin-dentin bond [18]. Moreover, the non-solvated adhesives are less hydrophilic and exhibited lower water sorption, solubility, and higher degree of conversion when compared to solvated ones [19].

While for solvent-containing adhesive agents, at 6 months, the dentin bond strength has a large drop. This could be attributed to the presence of water, a high concentration of hydrophilic domains and residual solvents affect the polymerization reaction, leading to the suboptimal degree of conversion and reduced bond longevity as a result of the elution of unreacted monomers. The final consequence of this process is the formation of a porous structure and permeable membrane. Therefore, simplified adhesives are characterized by increased water sorption, which promotes polymer swelling and other water-mediated degradation phenomena [20].

The air-drying is not able to accomplish significant solvent evaporation in the solvent-containing adhesives [21]. Ethanol containing adhesives have more affinity for chasing water than acetone containing done. Their evaporation increases the concentration of monomers in the adhesives, which lowers the vapor pressure of the remaining residual solvents, making it impossible to evaporate all solvents during the air-drying stage [22], [23]. The residual water and solvents are responsible for producing localized areas of incomplete monomer polymerization, which generating porosities within the bonded interfaces, in turn, may permit inward diffusion of water molecules during storage. Moreover, water may have diffused freely through the nanoporosities that were left after the evaporation of solvents/unreacted monomers [19]. This is confirmed by the results of the study of Nassar *et al*. [18], which concluded that the μTBS of the solvent-containing one-step self-etch decreased significantly after aging for 6 months, while in solvent-free self-etch adhesive, there was no significant decrease in the μTBS after aging for 6 months. Hence, further studies should be done to evaluate the durability of one-step self-etch adhesives more than 6 months of storage time.

Conclusion

1. The type of solvent may have an obvious effect on the dentin bond strength.
2. Ethanol-water-based one-step self-etch adhesives showed better bonding to dentin than acetone water-based self-etch adhesives.
3. Dentin wetness increases the bond strength with universal self-etch adhesives.

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