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

Mohamed Ahmed Wakwak, Eslam Hassan Gabr, Ahmed Mohamed Elmarakby

1Lecturer of Operative Dentistry, Faculty of Dental Medicine, Boys, Cairo, Al-Azhar University, Egypt; 2Assistant Lecturer of Operative Dentistry, Faculty of Dental Medicine, Boys, Cairo, Al-Azhar University, Egypt; 3Assistant Professor in the Department of Restorative Dental Sciences, Al-Farabi Colleges for Dentistry and Nursing, Riyadh, Kingdom of Saudi Arabia and Lecturer of Operative Dentistry Department, Faculty of Dental Medicine, Al-Azhar University, Assiut Branch, Egypt

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.

RESULTS: One-way ANOVA was used to compare between more than two non-related samples. The significance level was set at p ≤ 0.05.

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 carbondum 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 normal 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 ≤ 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 ≤ 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 system    | 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®, Methacryloxydicyclohexyl hydrogen phosphinate glycidyl methacrylate, HEMA®, Hydroxyethyl methacrylate, 4-META, 4-Methacryloxyethyl trimellitate anhydride, UDMA, Urethane dimethacrylate, TEGDMA, Triethylene glycol dimethacrylate, UV® Ultraviolet, Bis-GMA®, Bisphenolglycidyl methacrylate, BIS-EMA®, Ethoxylated bisphenol A glycidyl methacrylate.
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.

References

1. Lopes GC, Baratieri LN, Andrade MA, Vieira LC. Dental adhesion: Present state of art and future perspectives. Quintessence Int. 2002;33(3):213-24. PMid:11921770
2. Dey S, Shenoy A, Kundapur SS, Das M, Gunwal M, Bhattacharya R. Evaluation of the effect of different contaminants on the shear bond strength of a two-step self-etch adhesive system, one-step, self-etch adhesive system and a total-etch adhesive system. J Int Oral Health. 2016;3:1-7. doi:10.14815/jiodm.2016.06.38.2.141
3. Tay FR, Pashley DH, Garcia-Godoy F, Yiu CK. Single-step, self-etch adhesives behave as permeable membranes after polymerization. Part II. Silver tracer penetration evidence. Am J Dent. 2004;17(6):315-22. PMid:15575440
4. Werle SB, Stieglich A, Soares FZ, Rocha RO. Effect of prolonged air drying on the bond strength of adhesive systems to dentin. Gen Dent. 2015;63(6):68-72. PMid:26545278
5. Min JB. Drying adhesives. Restor Dent Endod. 2014;39(2):148. doi:10.5395/rde.2014.39.2.148 PMid:24790930
6. Salza U and Bockb T. Testing adhesion of direct restoratives to dental hard tissue a review. J Adhes. 2010;12(5):343-71. PMid:20978636
7. Andrade AM, Moura SK, Reis A, Loguercio AD, Garcia EJ, Grande RH. Evaluating resin-enamel bonds by microshear and microtensile bond strength tests: Effects of composite resin. J Appl Oral Sci. 2010;18(6):591-8. doi:10.1590/s1678-77572010000600010 PMid:21308290
8. Shah DD, Chandak M, Manwar N, Mani S, Mani A, Saini R, et al. Comparing shear bond strength of two step vs one step bonding agents on ground enamel and dentin: An in vitro study. Int J Experiment Dent Sci. 2014;3(1):1-3. doi:10.5005/jp-journals-10029-1058
9. Moszner N, Salz U, Zimmermann J. Chemical aspects of self-etching enamel dentin adhesives: A systematic review. Dent Mater. 2005;21(10):895-910. doi:10.1016/j.dental.2005.05.001 PMid:16038969
10. Abo-Alazm EA, Safy RK, Zayed MM. Solvent-free self-etch adhesive as a breakthrough in bonding technology: Fact or fiction? Tanta Dent J. 2016;3(2):83-8. doi:10.4103/1687-8574.188908
11. Khoroushi M, Shirban F, Shirban M. Marginal microleakage and morphological characteristics of a solvent-free one-step self-etch adhesive (B1SF). J Dent (Tehran). 2013;10(1):32-40. doi:10.5005/jp-journals-10024-1312 PMid:23724201
12. Umino A, Nikaido T, Sultana S, Ogata M, Tagami J. Effects of smear layer and surface moisture on dentin bond strength of a waterless all-in-one adhesive. Dent Mater J. 2006;25(2):332-8. doi:10.4012/dmj.25.332 PMid:16916237
13. Takai T, Hosaka K, Kambara K, Thitthaweerat S, Matsu N, Takahashi M, et al. Effect of air-drying dentin surfaces on dentin bond strength of a solvent-free one-step adhesive. Dent Mater J. 2012;31(4):558-63. doi:10.4012/dmj.2012-034 PMid:22864208
14. Chiba Y, Rikuta A, Yasuda G, Yamamoto A, Takamizawa T, Kurokawa H, et al. The effect of moisture conditions on dentin bond strength of single-step self-etch adhesive systems. J Oral Sci. 2006;48:131-7. doi:10.2334/josnusd.48.131 PMid:17023745
15. Lima GS, Ogliari FA, Moraes RR, Mattos ES, Silva AF, Carreño NL, et al. Water content in self-etching primers affects hydrolytic degradation of the resin dentine interface induced by the simulated pulpal pressure, direct and indirect water ageing. J Dent. 2010;37(3):155-9. doi:10.1016/j.jdent.2010.09.011 PMid:23429228
16. Feitosa VP, Leme AA, Sauro S, Correr-Sobrinho L, Watson TF, Sinhoreti MA, et al. Hydrolytic degradation of the resin dentine interface induced by the simulated pulpal pressure, direct and indirect water ageing. J Dent. 2012;40(12):1134-43. doi:10.1016/j.jdent.2012.09.011 PMid:23000523
18. Nassar AA, El-Sayed HY, Etman WM. Effect of different desensitizing adhesive systems on the shear bond strength of composite resin to dentin surface. Tanta Dent J. 2016;13:109-17. https://doi.org/10.4103/1687-8574.188913

19. Nagi SM. Durability of solvent-free one-step self-etch adhesive under simulated intrapulpal pressure. J Clin Exp Dent. 2015;7(4):466-70. https://doi.org/10.4317/jced.52307 PMid:26535091

20. Malacarne-Zanon J, Pashley DH, Agee KA, Foulger S, Alves MC, Breschi L, et al. Effects of ethanol addition on the water sorption/solubility and percent conversion of comonomers in model dental adhesives. Dent Mater. 2009;25(10):1275-84. https://doi.org/10.1016/j.dental.2009.03.015 PMid:19592083

21. Navarra CO, Cadenaro M, Codan B, Mazzoni A, Sergo V, De Stefano Dorigo E, et al. Degree of conversion and interfacial nanoleakage expression of three one-step self-etch adhesives. Eur J Oral Sci. 2009;117(4):463-9. https://doi.org/10.1111/j.1600-0722.2009.00654.x PMid:19627360

22. Nunes TG, Ceballos L, Osorio R, Toledano M. Spatially resolved photopolymerization kinetics and oxygen inhibition in dental adhesives. Biomaterials. 2005;26(14):1809-17. https://doi.org/10.1016/j.biomaterials.2004.06.012 PMid:15576155

23. Cadenaro M, Broschi L, Rueggeberg FA, Suchko M, Grodin E, Agee K, et al. Effects of residual ethanol on the rate and degree of conversion of five experimental resins. Dent Mater. 2009;25(5):621-8. https://doi.org/10.1016/j.dental.2008.11.005 PMid:19111335