Shear Bond Strength of Acetone-Based One-Bottle Adhesive Systems

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The aim of this study was to assess the shear bond strength of four acetone-based one-bottle adhesive systems to enamel and dentin, and compare to that of an ethanol-based system used as control. Fifty human molars were bisected mesiodistally and the buccal and lingual surfaces were embedded in acrylic resin using PVC cylinders. The buccal surfaces were ground to obtain flat dentin surfaces, while the lingual surfaces were ground to obtain flat enamel surfaces. All specimens were polished up to 600-grit sandpapers and randomly assigned to 5 groups (n=20; 10 dentin specimens and 10 enamel specimens), according to the adhesive system used: One-Step (Bisco); Gluma One Bond (Heraeus Kulzer); Solobond M (Voco); TenureQuik w/F (Den-Mat) and OptiBond Solo Plus (Kerr) (control). Each adhesive system was applied according to the manufacturers' instructions. The respective proprietary hybrid composite was applied in a gelatin capsule (d=4.3mm) and light-cured for 40 s. The specimens were tested in shear strength with an Instron machine at a crosshead speed of 5 mm/min. Bond strengths means were analyzed statistically by one-way ANOVA and Duncan's post-hoc ($p \leq 0.05$). Shear bond strength means (MPa) (±SD) to enamel and dentin were: *Enamel*: One-Step=11.3(±4.9); Gluma One Bond=16.3(±10.1); Solobond M=18.9(±4.5); TenureQuik w/F=18.7(±4.5) and OptiBond Solo Plus=16.4(±3.9); *Dentin*: One-Step=6.4(±2.8); Gluma One Bond=3.0(±3.4); Solobond M=10.6(±4.9); TenureQuik w/F=7.8(±3.9) and OptiBond Solo Plus=15.1(±8.9). In enamel, the adhesive systems had statistically similar bond strengths to each other ($p > 0.05$). However, the ethanol-based system (OptiBond Solo Plus) showed significantly higher bond strength to dentin than the acetone-based systems ($p \leq 0.0001$). In conclusion, the solvent type (acetone or ethanol) had no influence on enamel bond strength, but had great influence on dentin bonding, which should be taken into account when choosing the adhesive system.

Key Words: shear bond strength, acid etching, adhesive systems, dentin bonding, enamel bonding.

INTRODUCTION

Bonding to enamel can be satisfactorily achieved using the acid-etching technique (1). On the other hand, dentin bonding is more difficult due to the wet tubular structure, permeability properties and organic composition of dentinal substrate (2).

For contemporary adhesive systems, dentin bonding requires removal or modification of the smear layer and superficial demineralization by acid etching (2). Although chemical reactions between adhesive systems and dentin have been reported, it is generally accepted that dentin bonding relies primarily on micromechanical interaction similar that of enamel bonding, mediated by the permeation of resin monomers into acid-etched dentin (3). The entanglement of the polymerized adhesive resin with collagen fibrils and residual hydroxyapatite crystals generates an interfacial structure called "resin-dentin interdiffusion zone" or "hybrid layer" (3).

Currently, most studies and developments in dentin bonding are focused on simplification of the

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adhesive process. Several contemporary total-etch adhesive systems combine the primer and bonding agent in one bottle. No mixing of components is required and these simplified or "one-bottle" adhesives are light-cured. Dentin adhesive systems contain high-vapor pressure organic solvents (acetone and ethanol). These chemical agents, known as "water-chases", increase dentin wettability and help replace the water on the acid-etched and rinsed dentin surface with hydrophilic resin monomers (4).

In vitro shear bond strength testing is commonly used to quantitatively analyze and rank the performance of adhesive systems on enamel and dentin (5). It has been proved adequate and effective for evaluation and comparison of different adhesive systems and restorative materials.

The aim of this study was to evaluate *in vitro* the shear bond strength of four acetone-based one-bottle adhesive systems to enamel and dentin, and compare the results to those of an ethanol-based system used as control. The tested hypothesis was that acetone-based and ethanol-based one-bottle systems would yield similar bond strengths to enamel and dentin.

MATERIAL AND METHODS

Fifty human molars stored in water for up to 3 months were bisected mesiodistally. The buccal and lingual surfaces were embedded in chemically-activated acrylic resin (AcryliMet, South Bay Technology Inc., San Clemente, CA, USA). The 50 buccal surfaces were mechanically ground with water-cooled sandpapers to obtain flat dentin surfaces, while the 50 lingual surfaces were ground to obtain flat enamel surfaces. An area of approximately 5mm x 5mm was demarcated on the surfaces and the specimens were polished with #240-, #400- and #600-grit sandpapers for 30 s. Ten dentin and ten enamel specimens were randomly assigned to 5 groups according to adhesive system used:

One-Step: Dentin/enamel was etched with a 32% phosphoric acid gel (Uni-Etch; Bisco Dental Products, Schaumburg, IL, USA) for 15 s and thoroughly rinsed for 5 s. Two coats of One-Step (Bisco) acetone-based one-bottle adhesive system were applied on tooth surface with a disposable brush tip, gently air-dried for 10 s and light-cured for 10 s.

Gluma One Bond: Dentin/enamel was etched with a 20% phosphoric acid gel (Gluma CPS Conditioner; Heraeus Kulzer, Dormagen, Germany) for 20 s and rinsed for 5 s. Gluma One Bond (Heraeus Kulzer) acetone-based one-bottle adhesive system was richly applied to the entire surface with a disposable brush tip, immediately followed by a second coat. The adhesive sytem was spread over with a gentle air-stream for 5 s to evaporate the solvent and residual moisture, keeping the air syringe 2 cm from the surface and light-cured for 20 s.

Solobond M: Dentin/enamel was etched with a 34.5% phosphoric acid gel (Vococid Gel; Voco, Cuxhaven, Germany) for 15 s and rinsed for 20 s. One coat of Solobond M (Voco) acetone-based one-bottle adhesive was applied homogeneously to the surface with a disposable brush and left undisturbed for 30 s. Solobond M was spread over with a faint air jet and light-cured for 20 s.

TenureQuik with fluoride: Dentin/enamel was etched with a 37% phosphoric acid, medium viscosity etchant (Den-Mat, Santa Maria, CA, USA) for 15 s and rinsed for 10 s. Three consecutive coats of TenureQuik w/F (Den-Mat) acetone-based one-bottle adhesive system were applied to the surface, left undisturbed for 15 s, gently air-dried with oil-free compressed air for 10 s, keeping the air syringe 2 cm from the surface, and light-cured for 15 s.

OptiBond Solo Plus (control): Dentin/enamel was etched with a 37.5% phosphoric acid gel (Kerr Gel Etchant; Kerr Co., Orange, CA, USA) for 15 s and rinsed for 10 s. OptiBond Solo Plus (Kerr Co.) ethanol-based adhesive system was brushed continuously onto the surface for 15 s, evaporated with a mild air stream for 3 s and light-cured for 20 s.

After acid etching and before adhesive application, the surfaces were air-dried with oil-free compressed air from an air syringe during 5 s for enamel specimens, or to remove water excess and keep surface visibly moist for dentin specimens. A heavy-viscosity micro-hybrid composite (Filtek P-60, 3M/ESPE, St. Paul, MN, USA) was condensed into a No. 5 gelatin capsule with 4.3 mm in diameter (Torpac Inc, Fairfield, NJ, USA) to fill two-thirds of the capsule and was light-cured for 80 s using UnisX visible light-curing unit (Heraeus Kulzer). Following the application of the adhesive system, an increment of a respective proprietary hybrid composite resin [Aelite (Bisco); Charisma (Heraeus Kulzer), Polofil Supra (Voco), Virtuoso (Den-Mat) and Prodigy (Kerr)] was inserted into the final third of the gelatin capsule and the capsule was seated securely against the flat enamel/dentin surface. Excess material was carefully removed from the capsule periphery and the resin layer was light-cured for 40 s (20 s at each side of the capsule) using XL 1500 light-curing unit (3M/ESPE). Light intensity, as monitored by a curing radiometer (Demetron/Kerr, Danbury, CT, USA), was over 450 mW/cm2 throughout the study.

After a 24-h storage in distilled water, the specimens were submitted to a thermocycling regimen (500 cycles) between water baths at 5 and 55ºC, with a 30-s dwell time in each bath and a 2-s transfer time. Shear bond strength was measured with an Instron Universal Testing Machine (Model 4444, Instron Corporation, Canton, MA, USA), using the Series IX Software System (Instron Corp.) for data recording. A knife-edge-shearing rod running at a crosshead speed of 5 mm/min was used to load the specimens until fracture.

Data were analyzed statistically by one-way ANOVA (independent variable: adhesive system; outcome variable: shear bond strength). Duncan's post-hoc test was used to identify statistically significant differences between pairs of means at a confidence level of 95% for each set of data. Statistical analyses were carried out using the SPSS 10.0 for Windows software package (SPSS Inc., Chicago, IL, USA).

RESULTS

Shear bond strength means are given in Figures 1 and 2.

Shear bond strength to enamel ranged from 11.3±4.9 MPa for One-Step to 18.9±4.5 MPa for Solobond M (Fig. 1). One-way ANOVA showed no statistically significant difference ($p>0.05$) among the enamel specimens . The solvent types of the one-bottle adhesive systems had no influence on enamel bond strength.

Shear bond strength means to dentin ranged from 3.0±3.4 MPa for Gluma One Bond to 15.1±8.9 MPa for OptiBond Solo Plus. One-way ANOVA showed that the adhesive systems differed at $p\leq0.0001$. Duncan's Post hoc test ranked these differences in three subsets at a confidence level of 95% (Fig. 2). OptiBond Solo Plus ethanol-based adhesive system differed statistically from the other bonding agents, while Solobond M, One-Step and TenureQuik w/F were ranked in the intermediary Duncan's subset. Although Gluma One Bond presented the lowest dentin bond strength among the adhesive systems, it was not statistically different from One-Step ($p>0.01$).

Student's t-test revealed higher shear bond strength to enamel than to dentin ($p\leq0.001$).

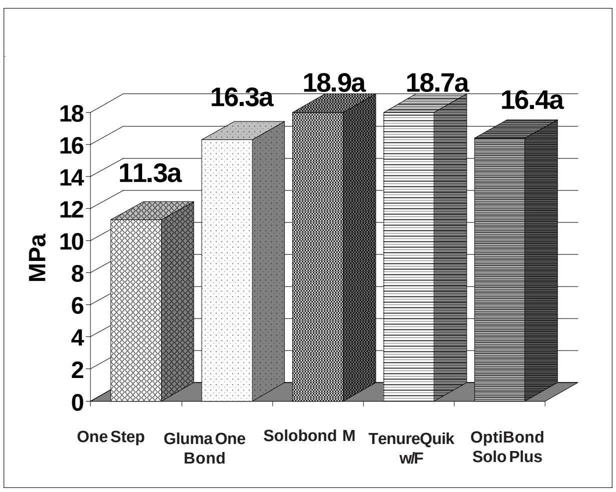

Figure 1. Shear bond strength means (MPa) to enamel. Different letters indicate statistically significant difference at $p<0.05$. OneStep, Gluma One Bond, Solobond M and TenureQuik w/F are acetone-based adhesive systems and Optibond Solo Plus (control) is an ethanol-based adhesive system.

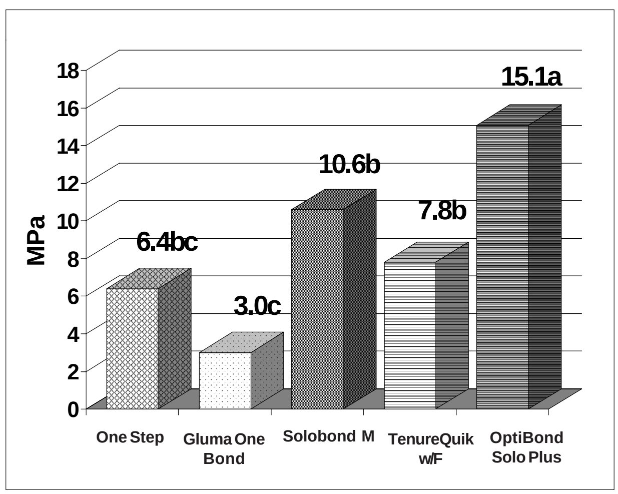

Figure 2. Shear bond strength means (MPa) to dentin. Different letters indicate statistically significant difference at $p<0.05$. OneStep, Gluma One Bond, Solobond M and TenureQuik w/F are acetone-based adhesive systems and Optibond Solo Plus (control) is an ethanol-based adhesive system.

DISCUSSION

Total-etch one-bottle adhesive systems combine the functions of primer and bonding agent in the same solution. Therefore, less viscous monomers are present diluted in solvents with a high volatile power such as ethanol and acetone. Regarding enamel bonding, this property seems to contribute to a complete interdiffusion of the system all over the etched area (6), resulting in high enamel bond strength (7), even under wet conditions (8). In the present study all total-etch one-bottle adhesive systems yielded similar bond strength to enamel.

The available one-bottle total-etch adhesive systems became popular because they are easy to handle, convenient and more simple to use than multi-step adhesive systems, rather than due to a better bonding (9). For hydrophilic bonding systems, dentin bonding is optimized in the presence of high-vapor pressure organic solvents. These solvents facilitate deeper and more complete penetration of monomers into dentin to enhance micromechanical retention (10). It has been reported that to avoid the collapse of the exposed collagen network, adopting a moist bonding protocol is extremely important in acetone-based bonding systems due to the water-chaser effect of acetone (11). On the other hand, the excess of water can dramatically affect acetone-based shear bond strength to dentin (12). Although this study used wet bonding for all systems evaluated, the narrow window of opportunity to achieve adequate dentin bonding with acetone-based systems may explain the fact that all four acetone-based one-bottle systems yielded lower shear bond strength means to dentin than the ethanol-based system. These findings are consistent with those of previous studies (13,14). Another possible explanation to the low shear bond strength to dentin reached with acetone-based adhesive systems is its high percentage of acetone (near to 70% in some systems). This fact may not permit a monomer concentration that is high enough to allow the formation of a uniform film on dentin surface (15). This phenomenon might explain the great variations in bond strengths observed in the literature. For example, studies have reported bond strength means above 20 MPa for One-Step (16), while other studies using similar shear bond strength methodologies reached results around 7.3 MPa (17).

Some of the available one-bottle adhesive systems have filler particles. For this reason, a very favorable flexible intermediate zone is created whereby the interface stress induced by polymerization shrinkage, elasticity module of resins, water sorption and occlusal forces are transmitted and attenuated (18). In a three-dimensional cavity, the formation of this zone is considered very important. However, it has been reported that very viscous bonding agents have greater difficulty to penetrate the etched enamel surface to a depth equal to that of etching (19). Some *in vitro* studies have shown that filled bonding systems present lower bond strength to enamel (7,8), which seems to be related to the high viscosity of these adhesives (7) that make them unable to penetrate the interprismatic areas as profoundly as the unfilled bonding agents (20). However, this fact apparently depends on product specificities as in the present study the filled adhesive system (Optibond Solo Plus) did not have lower bond strength to enamel than the other systems. Further *in vivo* studies with total-etch filled adhesive systems are required to investigate whether their higher viscosity would increase long-term marginal infiltration under clinical conditions.

RESUMO

O objetivo deste estudo foi avaliar a resistência ao cisalhamento de quatro sistemas adesivos de frasco único com solvente acetona e um sistema com solvente etanol em esmalte e à dentina. Cinqüenta molares humanos foram seccionados ao meio no sentido mesiodistal e incluídos com resina acrílica em tubos de PVC. As superfícies vestibulares foram desgastadas até atingir dentina plana, enquanto as superfícies linguais foram desgastadas até atingir esmalte plano. Os espécimes foram polidos até lixa d'água de granulação 600 e divididos aleatoriamente entre os 5 grupos (n=20; 10 espécimes de dentina e 10 espécimes de esmalte), de acordo com o sistema adesivo utilizado: One-Step (Bisco); Gluma One Bond (Heraeus Kulzer), Solobond M (Voco), TenureQuik w/F (Den-Mat) and OptiBond Solo Plus (Kerr) (controle). Os adesivos foram aplicados conforme as instruções dos fabricantes. Uma cápsula cilíndrica gelatinosa (d=4,5mm), preenchida com compósito híbrido de cada fabricante foi posicionada sobre a superfície dental e fotopolimerizada durante 40 s. Após 24 h, os corpos-de-prova foram submetidos a ensaios de cisalhamento em uma máquina de ensaios Instron com velocidade de 5 mm/min. Os dados foram analisados estatisticamente pela ANOVA a um critério e teste de Duncan post hoc ($p \leq 0.05$). As médias de resistência ao cisalhamento resistência em esmalte e dentina (MPa) (±SD) foram: *Esmalte*: One-Step=11,3(±4,9); Gluma One Bond=16,3(±10,1); Solobond M=18,9(±4,5); TenureQuik w/F=18,7(±4,5) e OptiBond Solo Plus=16,4(±3,9); *Dentina*: One-Step=6,4(±2,8); Gluma One Bond=3,0(±3,4); Solobond M=10,6(±4,9); TenureQuik w/F=7,8(±3,9) e OptiBond Solo Plus=15,1(±8,9). Em esmalte, todos os agentes adesivos tiveram resultados estatisticamente semelhantes entre si ($p>0.05$). Entretanto, em dentina, o sistema adesivo a base de etanol

apresentou maior resistência de união que todos os sistemas a base de acetona (p≤0.0001). O tipo de solvente presente nos sistemas adesivos de frasco único não exerceu influência na sua capacidade de união ao esmalte, mas se mostrou um componente importante na adesão dentinária, o que deve ser levado em conta quando da escolha de um sistema adesivo.

ACKNOWLEDGEMENTS

The authors acknowledge the manufacturers for supplying the restorative materials used in this study.

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Accepted March 5, 2005