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Influence of aging methods on push-out bond strength of adhesive systems to dentin cavities

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Abstract

This study evaluated the effect of aging methods on the bond strength of etch-and-rinse adhesive systems to dentin cavities. Eighty bovine incisors were used to prepare dentin cavities, which were bonded with different adhesive systems [Adper Scotchbond Multipurpose (SBMP), and Adper Single Bond 2 (SB)], and restored with composite resin. The specimens were stored in water for 24 h, and subjected to each aging method (n = 10): control group (not exposed to additional aging), thermal cycling (TC) (10,000 cycles; at temperatures of 5, 37, and 55 °C), mechanical loading (ML) (100,000 cycles; Hz; 60 N load), and TC + ML. The push-out bond strength test was performed using a universal testing machine. Failure modes were evaluated by scanning electron microscopy. Data were analyzed by two-way ANOVA and Tukey's test (α = 0.05). For SBMP, only TC + ML decreased the bond strength compared to control group. For SB, all aging methods decreased similarly the bond strength compared to control group. A high number of adhesive failures were observed. Therefore, SB was sensitive to all aging methods, whilst only TC + ML was able to decrease the bond strength for SBMP.

Keywords: Thermal cycling, Mechanical loading, Push-out, Bond durability

Background

The main challenge for dental adhesives is providing an equally effective bond to enamel and dentin. Bonding to enamel has been proven to be durable, while bonding to dentin is far more intricate and can apparently only be achieved when more complicated and time-consuming application procedures are followed [1, 2]. Consequently, researchers are focused on achieving protocols to increase the bond durability of adhesive system to dentin.

Microtensile bond strength and microshear bond strength tests are frequently used to investigate immediate and long-term dentin bond strength (after ageing methods) [3, 4]. However, these methods are routinely used in flat dentin surfaces in which C-factor is low, and the development of shrinkage stress is not directed toward the bonding interface [5], different from what it occurs in posterior tooth restoration in the oral environment. For this reason, push-out bond strength test has been used to evaluate adhesive performance in tooth cavities, where the composite shrinks into a high C-factor challenge and stresses generated directly to the bonding interface can jeopardize the bond strength [6].

Although the use of sodium hypochlorite has already been proposed to promote bond degradation in dentin cavities [7], thermal cycling (TC) and mechanical loading (ML) are methods commonly used to promote bond degradation [8–10], so that their ability to decrease the bond strength of dentin cavities bonded with etch-and-rinse adhesive systems need to be investigated. Thus, the present study aimed to evaluate the effects of TC, ML, and TC + ML on the push-out bond strength of dentin cavities bonded with different etch-and-rinse adhesive systems. The null hypothesis tested was that there would not be difference between aging methods and adhesive systems.

Methods

Samples preparation

A total of 80 bovine incisors, free from cracks and structural defects, were selected for this study. The teeth were disinfected in a 0.1% aqueous solution of thymol at 37 °C for no longer than 1 week. The roots were removed with a water-cooled diamond saw (South Bay Technology, San Clemente, CA, USA) coupled to a precision cutting machine (Isomet 1000; Buehler Inc., Lake Bluff, IL, USA). The buccal surface of the crown was wet ground with 400- and 600-grit SiC abrasive papers in a polishing machine (LaboPol-21; Struers, Copenhagen, Denmark) to obtain flat dentin surface.

Standardized conical cavities (2 mm top diameter × 1.5 mm bottom diameter × 2 mm height) were prepared with conical diamond burs (Komet Inc, Lemgo, Germany) at high speed under air–water cooling. A custom-made preparation device allowed the cavity dimensions to be standardized. The burs were replaced after every five preparations. To expose the bottom surface of the cavities, the lingual surfaces were ground in accordance to the procedure described for flattening the buccal surfaces. In this manner, a cavity with a C-factor magnitude of 2.2 was obtained [7].

The prepared specimens were randomly assigned to 8 groups (n = 10) according to adhesive system (Adper Scotchbond Multipurpose—SBMP and Adper Single Bond 2—SB, both 3M ESPE, St. Paul, MN, USA) and aging methods (24 h distilled water storage; thermal cycling—TC, mechanical loading—ML; and TC + ML combination).

Restorative procedures

The adhesive systems (Table 1) were used according to the respective manufacturers' instructions, as follows:

SBMP

Dentin was etched with 35% phosphoric acid (Scotchbond Etchant; 3M ESPE) for 15 s and washed thoroughly with water for 30 s. The excess water was blot dried with absorbent paper, leaving the dentin surface visibly moist (wet bonding). One coat of the primer was applied to dentin and air dried for 10 s at 20 cm with an air stream—2.6 bar (this distance was standardized using a millimeter ruler). One coat of the bonding agent was applied and light cured for 10 s using a LED (Coltolux; Coltène/Whaledent AG, Altstätten, Switzerland) at 1265 mW/cm^2 of irradiance monitored by a radiometer (model 100; Demetron/Kerr, Danbury, CT, USA).

Table 1 Materials used in this study

Commercial brand/manufacturer	Composition	Batch number
Scotchbond Etchant 3M ESPE, St Paul, MN, USA	Water, phosphoric acid 35%, synthetic amorphous silica	997505
Adper Scotchbond Multipurpose 3M ESPE, St Paul, MN, USA	Primer: water HEMA, copolymer of acrylic and itaconic, acids Adhesive: BisGMA, HEMA, triphenylantimony	1131400494 123050045
Adper Single Bond 2 3M ESPE, St Paul, MN, USA	Bis-GMA, HEMA, dimethacrylates, ethanol, water, photoinitiator system and a methacrylate functional copolymer of polyacrylic and poly(itaconic acid)	300770BR

HEMA 2-hydroxyethyl methacrylate, *Bis-GMA* bisphenol A diglycidyl ether dimethacrylate, *10-MDP* 10-methacryloyloxydecyl dihydrogen phosphate

SB

Dentin was etched with 35% phosphoric acid for 15 s and washed thoroughly with water for 30 s. The excess water was blot dried with absorbent paper, leaving the dentin surface visibly moist (wet bonding). Two coats of adhesive system were applied. The solvent was allowed to evaporate for 10 s at 20 cm and light cured for 10 s.

After applying the adhesive systems, the restorative procedure was performed using a microhybrid composite resin (Filtek Z100, shade A2; 3M ESPE) that was bulk inserted into the cavity from its wider side. Light curing was performed with the Coltolux LED for 20 s. The light tip was positioned directly on top of the restoration, which had been previously covered with a Mylar strip. The top and bottom surfaces of all restorations were finished with #1200 abrasive papers (Buehler Inc., Lake Bluff, IL, USA) coupled with a polishing machine (LaboPol-21; Struers). The specimens were stored in distilled water at 37 °C for 24 h, and subjected to aging methods: control group (not exposed to additional aging), TC, ML, and TC + ML [6].

Degradation procedures were performed using a thermal fatigue simulator device (MSCT-3; ElQuip, São Carlos, SP, Brazil), subjected to 10,000 thermal cycles; at temperatures of 5, 37, and 55 °C with dwell time of 30 s each and transfer time of 5 s; and a mechanical fatigue simulator (ER 37000, Erios, São Paulo, SP, Brazil), under 100,000 mechanical cycles, frequency of 4 Hz at 60 N load.

Bond strength analysis

The push-out bond strength test was performed using a universal testing machine (model 4411; Instron Corp., Canton, MA, USA). An acrylic device with a central orifice was adapted to the base of the machine. Each specimen was placed in the device with the top of its cavity against the acrylic surface. The bottom surface of the restoration was loaded with 1-mm-diameter cylindrical plunger at a crosshead speed of 0.5 mm/min until failure of the tooth-composite bond at the lateral walls of the cavity. The plunger tip was positioned in a way to touch only the filling material, thereby creating no stress at the surrounding walls. The load required for failure was recorded by the testing machine and later converted into MPa values.

The fractured specimens were cut in half with a water-cooled low-speed diamond saw (Isomet 1000; Buehler) in order to obtain two specimens. Both specimens were fixed to aluminum stubs with the fractured interfaces upward. Specimens were sputter coated with gold (SDC 050 Sputter Coater; Baltec, Balzers, Liechtenstein) and evaluated by scanning electronic microscopy (JSM 5600LV; JEOL, Tokyo, Japan) to determine the failure mode. The failure modes were defined as adhesive failure, cohesive failure in composite, and mixed failure.

Bond strength data was subjected to two-way analysis of variance (ANOVA) and Tukey's test ($\alpha = 0.05$). Failure modes were descriptively analyzed.

Results

Bond strength

There were statistically significant differences between adhesive system ($p < 0.01$) and in the interaction adhesive systems × aging methods ($p < 0.01$). Comparison among the groups is shown in Table 2. For SBMP, no aging provided statistically similar bond strength mean to TC and ML, which were statistically higher than the mean provided by TC + ML. For SB, all aging methods provided statistically similar bond strength means, which were lower than the mean provided by no aging group (control group). No aging and TC + ML produced statistically similar bond strength between adhesive systems. On the other hand, SBMP showed higher bond strength values compared to SB in specimens aged by TC and ML.

Failure modes

Regardless of the adhesive system and aging method, adhesive failures were predominant (Fig. 1).

Discussion

In the present study, the push-out bond strength of etch-and-rinse adhesive systems after thermal/mechanical aging methods was evaluated. Based on the results obtained, the null hypothesis was rejected, since thermal/mechanical-aging methods provided bond strength reduction in different ways for each adhesive system tested.

The durability of adhesive bond between resin and tooth structure is of significant importance for the longevity of adhesive restorations [11]. Although long-term water storage is routinely used by researchers in bond durability studies, TC and ML approaches may be suitable methods to induce bonding degradation in a shorter period than 6–12 month water storage strategy. Indeed, thermal changes and mechanical fatigue are prone to occur in vivo rather than only hydrolytic degradation of the bonding interface. TC test is based on temperature changes that induce repeated stresses at

Table 2 Bond strength means (standard deviations) according to different adhesive systems and aging methods

Adhesive systems	Aging methods			
	No aging	TC	ML	TC + ML
MP	11.3 (4.1) Aa	10.9 (4.2) Aa	10.9 (4.4) Aa	6.4 (1.8) Ba
SB	11.2 (5.2) Aa	5.7 (2.4) Bb	6.5 (1.1) Bb	5.3 (2.6) Ba

MP Adper Scotchbond Multipurpose, *SB* Adper Single Bond 2, *TC* thermal cycling, *ML* mechanical loading

Different letters (uppercase for aging methods and lowercase for adhesive systems) indicate statistically significant difference ($p < 0.05$)

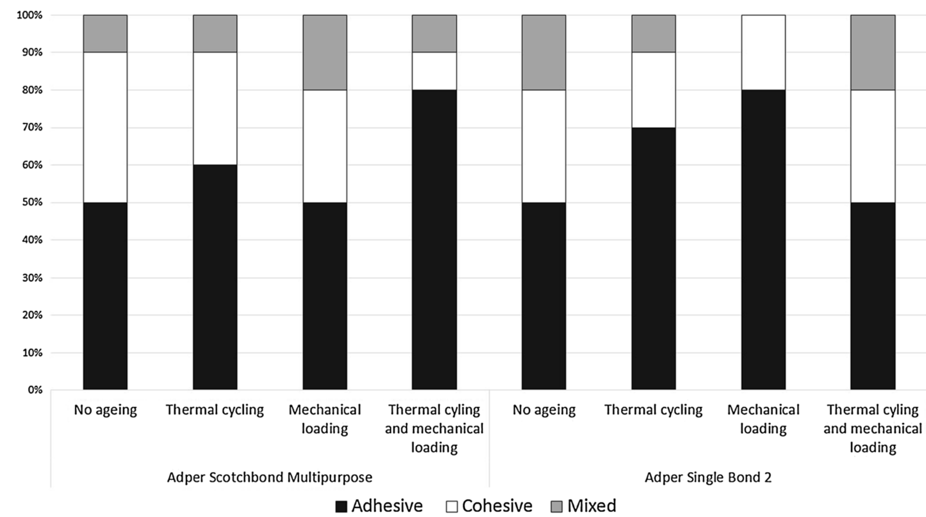

Fig. 1 Failure modes according to different adhesive systems

bonding interface. Such stresses arise due to differences in the thermal expansion coefficients of both materials and can lead to bonding failure [12]. ML test intends to simulate chewing conditions and is based on the incidence of repeated loads, producing alternate stresses [13]. The literature reports that simultaneous application of thermal and mechanical load cycling decreases the bond strength values; therefore, the sum of their effects generates more tension at the adhesive interface for micro tensile specimens [14].

SBMP had decreased bond strength only after TC + ML in comparison with non-aged samples. SBMP is a three-step etch-and-rinse adhesive system that contains a wet solvent-rich primer covered with a bonding agent that seals the primed dentin, rendering the interface less susceptible to water sorption and polymer hydrolysis [15]. The temperature changes induce different contraction/expansion behaviors in the dentin/composite interface due to differences in the expansion coefficient of dentin and composite [16]. The effect of mechanical stress occurs due to the different modulus of elasticity of the components of the adhesive interface, which determine different degrees of elastic or plastic deformation, thereby creating a gap between cavity and adhesive [17]. Due to a higher mechanical resistant hybrid layer formed after adhesive application, it is found that only treatment that generated more stress (TC + ML) was able to promote an effective reduction on bond strength values. On the other hand, thermal stress and mechanical loading solely were not capable to degrade it, so that SBMP would require the synergic effect of them to be susceptible to degradation.

In fact, conventional three-step adhesive systems are gold standard in terms of adhesive cavity sealing and mechanical properties [18]. However, the simplified adhesive system (two-step etch-and-rinse) combines the primer and the bonding agent into one single solvated solution. The solvent present in such adhesives is also more difficult to evaporate, frequently remaining entrapped within the adhesive layer after polymerization [8]. In this view, SB was capable to suffer bond strength decrease after all aging methods. The more hydrophilic nature of simplified adhesive systems render them more prone to water sorption and consequently more susceptible to the effects of hydrolytic degradation [19]. Solvated adhesive exhibits lower degree of carbon double bonds conversion and poorer mechanical properties than solvent-free adhesives [20], increasing the susceptibility of SB to degradation process.

Although bond strength results showed difference between ageing methods and adhesive systems that were not observed in the failure mode, we believe that the reason for this was the uniform distribution, which can have occurred during the test, favoring adhesive failures. In fact, classical studies have showed that non-uniform stress distribution across the interface is highly related to cohesive failures [21, 22].

Different testing methods, such as shear and tensile bond strength, have been used to measure dentin bond strength. One disadvantage of these methods is that they are generally performed on flat dentin surfaces, where the C-factor is very low and the shrinkage stress is not directed at the bonding interface. For these reasons, the push-out bond strength test was used in the present study. The advantage of using the push-out bond strength test is that adhesion and composite polymerization occur in a high C-factor cavity that was bulk filled with the composite, similar to clinical conditions, with increased stress generation directed to the bonding area [23].

It is likely that studies involving dentin bond durability in tooth cavities should be encouraged, since it would be more prone to reproduce clinical condition in in vitro adhesion researchers. However, the authors should be attempt to choose effective ageing methods to decrease bond strength of each adhesive system used in their investigations.

Conclusion

Based on the results of the present study, it can be concluded that two-step etch-and-rinse adhesive system was sensitive to TC, ML, and TC + ML, whilst only TC + ML was able to decrease the bond strength for the three-step etch-and-rinse adhesive tested.

Authors' contributions

BCDB, FHBA, AC, LVFC, and TAM participated in the study design, data collection, data interpretation, manuscript drafting and critical revision. MCAJM, LVFC, and TAM participated in the laboratory analyses and data collection. BCDB and FHBA performed the statistical analysis and participated in the data interpretation. FHBA and AC participated in the supervision of laboratory research, manuscript drafting, and critical revision. All authors read and approved the final manuscript.

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Competing interests

The authors declare that they have no competing interests.

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