Influence of Aging Treatments on Microtensile Bond Strength of Adhesive Systems to Primary Dentin

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ABSTRACT

Purpose: The purpose of this study was to evaluate the in vitro influence of aging treatments on the microtensile bond strength of 2-step total-etch and self-etch adhesive systems to primary dentin.
Methods: Class I cavities (4 mm x 4 mm x 2 mm) were prepared in 80 extracted human primary molars divided into 8 groups (N=5) per adhesive system: (1) Single Bond (SI); and (2) Clearfil SE Bond (SE). Restored teeth were exposed to individual and combined aging treatments: (1) thermal (2,000 x at 5-55°C; T); (2) mechanical load (100,000 x 80 N; M); (3) pH (mineralizing/demineralizing solutions; pH); and (4) control (24-hour water storage; C). Beam-shaped microtensile specimens were prepared (0.8 mm2 cross-sectional area) and loaded at a crosshead speed of 1.0 mm/minute in a universal testing machine. Fracture modes were examined by scanning electron microscope.
Results: Means (MPa) and standard deviations (±) were: (1) SI-T=24.17 (±1.99); (2) SI-M=25.64 (±9.75); (3) Si-pH=23.43 (±4.19); (4) SI-C=33.81 (±2.45); (5) SI-TM=18.60 (±3.57); (6) SI-TpH=23.90 (±4.28); (7) SI-MpH=20.96 (±4.06); (8) SI-TMpH=20.94 (±3.15); (9) SE-T=24.08 (±4.52); (10) SE-M=18.30 (±14.12); (11) SE-pH=19.22 (±8.93); (12) SE-C=37.80 (±7.28); (13) SE-TM=22.89 (±11.74); (14) SE-TpH=27.87 (±12.77); (15) SE-MpH=18.87 (±10.95); and (16) SE-TMpH=22.55 (±3.05). Both adhesive systems presented similar dentin bond strength (P>.05) which were significantly reduced when combined aging treatments were applied or when pH cycling (pH) was done.
Conclusion(s): Combined aging treatments and also pH cycling alone influenced negatively adhesive bond strengths. (J Dent Child 2007;74:109-12)

KEYWORDS: PRIMARY TEETH, MICROTENSILE TESTING, BOND STRENGTH

Dentin bond strength achieved with adhesive systems is largely studied. While several studies evaluated bond strength to permanent dentin, however, few studies that used primary dentin have been published.[1,6,14,22] Beyond the differences between primary and permanent teeth, variations regarding the chemical composition, tubular density, intrinsical humidity, and permeability[2,14,15,19] can affect the bond strength of adhesive systems in primary dentin.

Nör et al (1997),[19] observing that primary dentin is more reactive to acid etching than permanent dentin, suggested different application protocols for this substrate. On the other hand, contrary results have been reported concerning hybrid layer formation.[6,22] Bond strength to primary dentin is controversial, since some studies showed lower bond strength to this substrate compared to permanent dentin[7,20,22] while other studies demonstrated similar[8] or even higher values to primary dentin.[14] In pediatric dentistry, a self-etching system can be very useful since it requires fewer steps, thereby reducing application time, which is desirable with children.

While high initial bond strength is desirable, durability of resin-dentin over the time is an area of great interest once bond interface degradation can be related to microleakage

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and is detrimental to the longevity of the restorations.[3,12,13] Most studies are performed over short periods (eg, 24 hours). Additionally, other variables present in the oral environment— such as chemical attacks, temperature changes, and occlusal stresses—can affect the bond's interface, integrity, and durability. Long-term clinical trials are considered the best method to evaluate bond durability, however, high costs and long investments of time do not allow this kind of study to be routinely employed. Simulations of thermal and mechanical stresses have been used in some in vitro studies to simulate oral environmental conditions,[5,17,18] although chemical or pH cycling largely used in cariology studies9 are uncommon in bond strength evaluations.

The purpose of this study was to evaluate the in vitro influence of aging treatments on the microtensile bond strength of 2-step total-etch and self-etch adhesive systems to primary dentin.

METHODS

This study's protocol was reviewed and approved by the Institutional Review Board of the University of São Paulo, São Paulo, Brazil.

Eighty intact exfoliated second primary molars were used in this study. Prior to use, the teeth were stored in a refrigerator (4°C) for a maximum period of 3 months. The teeth were cleaned of debris with pumice paste via a slow-speed handpiece and stored in distilled water until they were used.

Occlusal Class I cavities were prepared using diamond burs (1092 KG Sorensen, São Paulo, Brazil) mounted in a high-speed handpiece under water irrigation and replaced every 2 preparations. Cavities were prepared in a special device to standardize cavity dimensions: 4 mm x 4 mm x 2 mm deep. A commercially 2-step, total-etch adhesive system (Single Bond, 3M ESPE Dental Products, St Paul, Minn) and a 2-step self-etching primer (Clearfil SE Bond, Kuraray Medical Inc, Osaka, Tokyo, Japan) were used in this study. Half of the prepared cavities were treated with 37% phosphoric acid gel (Scotch Etchant, 3M ESPE Dental Products) for 15 seconds and then rinsed for 15 seconds with distilled water. The excess water was removed with a cotton pellet, leaving the surface visibly moist. Two consecutive coats of Single Bond were applied, gently air dried, and light cured for 10 seconds. The other half (40 teeth) were restored with Clearfil SE Bond. Primer was applied for 20 seconds, lightly air dried followed by the bond application, and cured for 10 seconds. Cavities were restored using composite resin (Z100, 3M ESPE Dental Products). Each of the 2 1-m composite layers was light cured for 40 seconds using a light curing unit with the intensity of 600 mW/cm2 (QHL75 Curing Lite, Dentsply, Petrópolis, RJ, Brazil). After being stored in distilled water at 37°C for 24 hours, the specimens were randomly divided into 8 groups per adhesive system (N=5) according to the aging treatment:

1. C=control group (no treatment);
2. T=thermal cycling only (2,000 cycles, 5°C and 55°C);
3. pH=pH cycling only (10 cycles, 8 hours immersion on demineralizing solution, and 16 hours on remineralizing solution);
4. M=mechanical load cycling only (100,000 cycles, 80 N);
5. TpH=thermal and pH cycling;
6. TM=thermal and mechanical load cycling;
7. MpH=pH and mechanical load cycling;
8. TMpH=thermal, pH, and mechanical load cycling.

Before being subjected to pH cycling, the specimens had their cervical portions sealed with epoxy resin based adhesive (Araldite, Brascola, São Bernardo do Campo, São Paulo, Brazil) and then covered with 2 coats of nail varnish. Those subjected to mechanical load cycling were embedded in vinyl tubes with acrylic resin by the root portion to maintain occlusal surfaces perpendicular to the tooth's long axis.

THERMAL CYCLING

Thermocycling was performed in a thermal cycling machine with 2 baths at 5°C and 55°C, with a dwell time of 60 seconds and a transfer time of 5 seconds between each bath.

PH CYCLING

Specimens were placed separately in a container with 10 ml of demineralizing solution (2.2 mM (milimolar) of $CaCl_2$, 2.2 mM of NaH_2PO_4, 0.05 M of acetic acid, and 1M of KOH at 4.5 pH) for 8 hours and in remineralizing solution (1.5 mM of $CaCl_2$, 0.9 mM of NaH_2PO_4 and 0.15 mM of KCl at 7.0 pH) for 16 hours, according to that proposed by Featherstone (1996).[9] After every immersion, specimens were rinsed with deionized water. Solutions were changed every day for 10 days.

MECHANICAL LOAD CYCLING

Mechanical load cycling was done in a special machine (Ética Scientific Equipaments, São Paulo, Brazil). A load of 80 N was applied on the restorations using polyacetal tips attached to the machine for 100,000 cycles (~4Hz).

For groups 5, 6, 7, and 8, aging treatments were done following the same protocol previously described.

MICROTENSILE BONDING TEST

Specimens were longitudinally sectioned into 2 axes (mesiodistal and buccolingual) across the bonded interface (pulpar floor) with a diamond wafering saw under water refrigeration in a Labcut 1010 machine (Extec, Enfield, Conn) to obtain dentin (pulpal floor)/resin beam-shaped sticks with a cross-sectional area of approximately 0.8 mm.[2]

The sticks were attached with cianoacrylate adhesive gel (Super Bonder, Henkel Loctite, São Paulo, Brazil) to a modified caliper for microtensile testing and subjected to microtensile testing at a crosshead speed of 1.0 mm/minute, until fracture.

All fractured sticks were mounted on metallic stubs, gold sputtered, and observed with a scanning electron microscope (SEM; 44 Oi, LEO, Cambridge, UK) for fracture mode analysis. The fracture mode was classified as an adhesive/mixed failure if debonding occurred between the resin and dentin and as a cohesive failure if it occurred in composite resin or dentin.

Only the specimens that showed adhesive/mixed failures were considered in the calculation for bond strength means and standard deviations. Bond strength data (MPa) were analyzed by 2-way analysis of variance (adhesive system; treatment aging) and Tukey's test at a significance level of 0.05.

Table 1: Failure Mode Analysis According to the Adhesive System

	Failure mode (%)		
System	Adhesive / mixed	Cohesive in resin	Cohesive in dentin
Single Bond	94	3	3
Clearfil SE Bond	98	1	1

RESULTS

Failure modes of tested sticks are shown in Table 1. Adhesive/mixed failure was predominant to all groups independent of the treatment or adhesive system.

No statistically significant differences were observed between adhesive systems (P>.05) (Table 2), so the material factor was discarded and the influence on the bond strength was done considering only the aging treatments.

Statistically significant differences (P<.05) were verified when comparing control to pH groups and groups that received combined treatments (except TpH). In other words, bond strengths were significantly lower in the pH, TM, MpH, and TMpH groups than in the control group, as described in Table 2.

Table 2: Microtensile Bond Strength Values of Bond Strength According to Adhesive System and Aging Treatment

Aging treatment	Single Bond (MPa±SD)*	Clearfil SE Bond (MPa±SD)
Control (C)	33.81 ± 2.45^{a}	33.81 ± 2.45^{a}
Thermal (T)	24.17 ± 1.99^{ab}	24.08 ± 4.52^{ab}
pH (pH)	23.43 ± 4.19^{b}	19.22 ± 8.93^{b}
Mechanical (M)	25.64 ± 9.75^{ab}	18.30 ± 14.12^{ab}
Thermal/pH (TpH)	23.90 ± 4.28^{ab}	27.87 ± 12.77^{ab}
Thermal/mechanical (TM)	18.60 ± 3.57^{b}	22.89 ± 11.74^{b}
Mechanical/pH (MpH)	20.96 ± 4.06^{b}	18.87 ± 10.95^{b}
Thermal/mechanical/pH (TMpH)	20.94 ± 3.15^{b}	22.55 ± 3.05^{b}

* Different letters show significant differences.

DISCUSSION

Resin-dentin bond durability is essential for mechnical, biological, and esthetical factors. High bond strength values are desirable, since the bond interface should bear the stresses caused by the composite polymerization shrinkage and avoid adhesion failure caused by the thermal, mechanical, and chemical demands of the oral environment.

Despite different application protocols, the bonding mechanism and compositions of both adhesive systems evaluated presented similar bond strengths, which was observed in previous studies.[3,22] The use of the total-etch approach can result in a discrepancy between demineralized and infiltrated dentin leaving unprotected collagen fibrils which are more susceptible to hydrolytic degradation and, consequently, bond compromising.[13,25] This phenomenon, however, is not associated with self-etch approach systems due simultaneously to mineral dissolution and monomers infiltration.[13,24] This helps explain how the hybrid layer is more stable for self-etching systems than for total-etch systems. Additionally, the self-etch approach does not require separate acid etching. Hence, the residual moist problem is overcome, simplifying the application technique.

Most of the current adhesive systems were evaluated under controlled conditions and shortly after application. Nevertheless, some variables can negatively influence bond strength test results.

The microtensile bond strength test presents many advantages compared to the typical tensile test, since it allows for obtaining specimens from cavities[16,21] that are closer to the clinical situation than classical flat surfaces with small c-factors used in conventional tensile strength test studies.

Thermal, chemical, and mechanical treatments have been used to simulate the aging effect that occurs in the mouth, which can negatively influence bond strengths and can be related to marginal deterioration microleakage. Regarding this, in vitro simulations can be useful to predict the longevity of the adhesive procedures. Thermal and mechanical treatments (T and M groups), however, did not influence bond strengths when performed alone, corroborating previous studies.[5,10]

Groups submitted to pH cycling presented lower bond strengths values compared to the control group, even applied separately. Such lower values should be explained by the mineral loss in enamel cavity margins that can result in gap formation and consequent microleakage. Thermal and mechanical (Group 6), pH and mechanical (Group 7), and thermal, pH and mechanical (Group 8) treatments adversely affected bond strengths. Previous studies[5,18] also showed reduced values to thermal and mechanical cycling combination. The lower results of groups submitted to combined aging treatments can be associated with the increased effect of combined treatments. Despite the fact that the control group had been exposed to water for less time (24 hours), the detrimental effect of combined aging treatments could be confirmed by obtained results.

According to the SEM observation, most of the specimens showed adhesive/mixed failures independent of treatment or material, which agrees with previous results[5,16,23] and is expected when microtensile testing is done. Cohesive failures in dentin respond for a lower percentage and can be related to the weak strength of depth dentin.[11]

Lower bond strength can be associated with: (1) microleakage; (2) secondary caries; (3) sensitivity; and (4) restoration failure. Although in vitro results cannot be directly extrapolated by clinical condition, they can predict that adhesive systems are negatively influenced by oral environment stressors.

CONCLUSIONS

The null hypothesis that thermal, pH, and mechanical stressors would not influence bond strength was partially rejected when 3 stressors were applied in the same specimens. Combined aging treatments and also pH cycling alone influenced negatively adhesive bond strengths to primary dentin.

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