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Effect of Enamel and Dentin Surface Treatment on the Self-Adhesive Resin Cement Bond Strength

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The aim of this study was to evaluate the effect of enamel and dentin surface treatment on the micro-shear bond strength of self-adhesive cement. Seventy-two extracted third molars had their crowns embedded in acrylic resin and worn to obtain a flat enamel or dentin surface. The enamel and dentin specimens were randomly assigned to 8 groups (n=12) that were based on surface treatment (11.5% polyacrylic acid solution or no treatment), substrate condition (wet or dry) and storage period (1 day or 90 days), and treated accordingly. Cylinders (1 × 1 mm) were fabricated using self-adhesive resin cement (RelyX U200) following the manufacturer's instructions. The specimens were stored in distilled water at 37 °C for either 1 day or 90 days and subjected to micro-shear bond strength test (EMIC DL 2000 at 0.5 mm/min). After this, the failure type of the specimens was determined. Data were subjected to statistical analysis (α=0.05). According to the results, the 11.5% polyacrylic acid application decreased the bond strength in both enamel and dentin samples. The moist groups showed higher bond strength than the dry ones, regardless of the substrate and surface treatment. Storage period did not influence bond strength. In conclusion, surface treatment with 11.5% polyacrylic acid and absence of moisture decreased the bond strength of the resin-cement (RelyU200), regardless of the storage period.

Key Words: dental materials, dental cements, shear strength, dentin, dental enamel.

Introduction

Resin cements have been widely used for indirect esthetic restorative procedures (1,2). These materials require prevous treatment of the tooth surface, like acid etching, to remove the smear layer, demineralize the underlying dentin and facilitate the penetration of cement into the exposed collagen fiber net to form a hybrid layer (2,3). Although efficient, it is a very sensitive technique considering the number of involved variables: operator, substrate quality, used material and the room temperature (1,2). In order to avoid this sensitive step and reduce clinical time, self-adhesive resin cements that do not require substrate surface pre-treatment have been developed (1,2,4,5).

Self-adhesive resin cements are composed of an acidic monomer (methacrylate phosphoric acid) that can partially remove and/or modify the smear layer, which enables penetration of the cement and produces micromechanical retention to the substrate (2,5). This suggests that a certain chemical bond occurs through a chelating reaction between the acid monomers and the hydroxyapatite present in dental substrates (2,6-10).

The bond strength achieved by self-adhesive resin cements is lower than those achieved by conventional resin cements (2,6,8,11,12) and adhesive systems for direct resins (1). This is due to 4 factors: 1. acidic monomers have low etching capacity, minimizing surface demineralization (2,5,6,8,13,14); 2. buffering effect of the minerals in the dentin that neutralizes the cement pH (15,16,17); 3. high viscosity of the cement that hinders its penetration into the interfibrillar space (4,5,9,18); and 4. non-removal or incomplete removal of the smear layer, which promotes a weak bond with the resin's intermediate layer (5,11,13,14,19).

In an attempt to increase the bond strength between resin cements and tooth surfaces, surface treatments with different conditioning agents have been suggested. Polyacrylic acid is a mild conditioning agent employed for cavity cleansing and surface conditioning in glass ionomer restorations (2,10,12,20). In these restorations, polyacrylic acid promotes the formation of irregularities on the surface of the substrate, forming an intermediate layer that facilitates ion exchange between the glass ionomer matrix and the calcium and phosphate in the partially demineralized smear layer (2,10,12,21). Furthermore, the carboxyl ions in the acid increase the cleaning power and wettability of the surface (2,10,12).

Therefore, the aim of this study was to evaluate the effect of enamel and dentin surface treatment (no treatment vs. polyacrylic acid solution) and different substrate conditions (moisture control) on the immediate (1-day storage) and long-term (90-day storage) micro-shear bond strength of self-adhesive resin cement. The hypotheses of this study were as follows: 1. acidic treatment of the substrate (enamel and dentin) increases bond strength values; 2. wet condition of the substrate (enamel and dentin) increases bond strength values; and 3. increase in storage period decreases bond strength values.

Material and Methods

Specimens of enamel and dentin were derived from caries-free, erupted third molars from patients of both genders, aged between 18 and 25 years. Seventy-two extracted third molar teeth were selected, cleaned and stored in a 0.5% chloramine-T solution for up to 2 months after extraction. Approval was obtained from the Ethics Committee (protocol #132.616) for carrying out this study.

The teeth roots were sectioned 1 mm below the cementoenamel junction. In order to prepare the enamel substrate specimens, the crowns of 48 teeth were sectioned in a mesio-distal direction with a slow speed diamond saw (Isomet 1000; Buehler, Lake Bluff, IL, USA) to obtain 2 sections of each tooth (96 enamel sections). In order to obtain the dentin substrate specimens, the crowns of other 24 teeth were sectioned perpendicular to the long axis of the tooth to remove the occlusal enamel and to expose the flat, middle-third dentin surface (24 dentin sections).

The tooth sections (enamel or dentin) were then cast in PVC rings using acrylic resin (Jet; Classic Artigos Dentais Ltda, São Paulo, SP, Brazil) and wet-sanded with 400- and 600-grit silicon carbide paper (Metaserv; Buehler). Thereafter, they were placed in an ultrasonic cleaner for 5 min, to expose the flat enamel area on the lingual, buccal or palatal surfaces of the molars and a flat dentin area on the occlusal surface.

To obtain the enamel substrate specimens, were selected teeth that presented the most flat enamel surfaces. Care was taken to remove only the minimum amount of tissue sufficient to expose an 8 mm (length) × 2 mm (width) flat enamel surface. Each specimen was examined both visually and under a stereomicroscope to ensure that no dentin exposure was present.

Enamel and dentin substrate specimens were then randomly assigned to 8 groups (n=12) based on the type of surface treatment (no treatment/control or application of 11.5% polyacrylic acid solution), substrate condition (wet or dry surface) and storage period (1 day or 90 days). At the time of specimen preparation, exposed enamel and dentin surfaces were wet-sanded with 600-grit silicon carbide paper for 1 min to standardize the smear layer. Next, enamel and dentin surfaces were subjected to the respective treatments. The control group received no surface treatment apart from smear layer standardization; the polyacrylic acid group was treated with a solution of 11.5% polyacrylic acid (Vitro Condicionador; Nova DFL, Rio de Janeiro, RJ, Brazil) for 10 s with a moistened micro-brush. Subsequently, the specimens were washed thoroughly.

The substrate condition (moisture control) was achieved as follows: for the wet condition, the moisture was moderately removed with absorbent paper, leaving a bright surface; for the dry condition, the surface was thoroughly air-dried for 30 s at a 5 cm distance.

Following the enamel and dentin surface treatments, a split silicone mold (diameter: 1 mm; height: 1 mm) was clamped to the enamel or dentin surfaces and filled with self-adhesive resin cement (RelyX U200; 3M ESPE, St. Paul, MN, USA). The self-adhesive resin cement was light-cured continuously for 20 s at 1,100 mW/cm^2 (Poly Wireless; Kavo, Joinville, SC, Brazil). Ten minutes after completion of the light curing, the bonded specimens were released from their molds and stored individually in dark canisters with distilled water at 37 °C for either 1 day or 90 days.

Shear bond strength was tested using a universal testing machine (EMIC DL2000; EMIC, São José dos Pinhais, PR, Brazil). The wire-loop method was used at a crosshead speed of 0.5 mm/min. Mean and standard deviation values were calculated for each enamel and dentin group.

The failure modes were evaluated at ×57 magnification under a stereomicroscope (SZX9; Olympus, Tokyo, Japan). Failure was assessed as mainly adhesive, mainly cohesive in the resin cement, cohesive in the enamel or dentin, or mixed.

The micro-shear bond strength values for the enamel and dentin substrates were subjected, separately, to analysis of variance (three-way ANOVA)—considering the factors of surface treatment (no treatment or polyacrylic acid), substrate condition (wet or dry), and storage period (1 day or 90 days) as well as Tukey's test (α=0.05).

The percentage and frequency of the types of failure in the enamel and dentin substrates were subjected to the chi-square test. All the statistical analyses were performed using Statistica version 10.0 (StatSoft South America Comércio de Software Ltda, São Caetano do Sul, SP, Brazil).

Results

Enamel Substrate

For the enamel substrate, analysis of variance detected significant differences in relation to treatment (p=0.003) and substrate condition (p=0.000005). An interaction effect was also found between treatment and substrate condition (p=0.0009). The control group (no treatment) showed higher bond strength values (7.57±4.28 MPa) than the treatment group (polyacrylic acid treatment) (5.64±2.17 MPa), regardless of substrate condition or storage period (1 day or 90 days). The wet substrate showed higher bond strength values (8.41±4.06 MPa) than the dry one (4.98±1.79 MPa), regardless of treatment or storage period. The association of control (no treatment) and wet condition showed higher bond strength values (10.57±2.50 MPa) than the other groups.

There was no significant difference in the interaction of storage period (p=0.944) with other factors. There was no significant difference between the two storage periods (1 day: 6.56±3.01 MPa and 90 days: 6.67±3.90 MPa), regardless of treatment and substrate condition.

Tukey's multiple comparison test (α=0.05) was used to identify the significant differences among the tested groups for the enamel substrate. Mean and standard deviation (SD) values for bond strength (MPa) in the different groups of the enamel substrate are in Table 1.

The results of the present study demonstrated statistically significant differences among the groups. The control (no treatment)-wet-1-day group showed significantly higher bond strength values than the other groups except for the control (no treatment)-wet-90 days group. The polyacrylic acid groups did not differ from each other neither did they differ from the control (no treatment) groups in dry condition.

Regarding the failure mode in the enamel substrate, there were no significant differences between the groups (p>0.05), with predominant adhesive failures in all groups at both 1 day and 90 days. Cohesive and mixed failures were found in some samples, but had a random character (Fig. 1).

Dentin Substrate

For the dentin substrate, the analysis of variance detected significant differences for treatment (p=0.0000001), substrate condition (p=0.0000001), and storage period (p=0.022). An interaction effect was also found between treatment and substrate condition (p=0.005). The control group (no treatment) showed higher bond strength values (8.13±4.67 MPa) than the treatment group (polyacrylic acid treatment) (4.06±2.08 MPa), regardless of substrate condition or storage period (1 or 90 days). The wet substrate showed higher bond strength values (8.10±4.62 MPa) than the dry one (4.19±2.44 MPa), regardless of treatment or evaluation period. The 90-day group showed higher bond strength values (6.99±4.53 MPa) than the 1-day group (5.40±3.72 MPa), regardless of treatment or substrate condition. The association of control (no treatment) and wet condition showed higher bond strength (11.03±3.70 MPa) values than the other groups.

For the dentin substrate, Tukey's multiple comparison test (α=0.05) was used to identify significant differences among the tested groups. Mean and standard deviations (SD) values for bond strength (MPa) in the different groups of the dentin substrate are in Table 2.

The results of the present study demonstrate statistically significant differences among the groups. These differences followed the same pattern as those found for enamel. The control (no treatment)-wet-90-day group showed significantly higher bond strength values compared to the other groups except for the control (no treatment)-wet-1-day group. The polyacrylic groups did not differ from each other neither did they differ from the control (no treatment) groups in dry condition.

Regarding the failure mode in dentin substrate, there were no significant differences among the groups (p>0.05), with predominantly adhesive failures in all groups at both 1 day and 90 days. Cohesive and mixed failures were found in some samples, but had a random character (Fig. 2).

Discussion

The acidic treatment significantly decreased the shear bond strength in both enamel and dentin substrates, rejecting the first hypothesis. There were significant differences in shear bond strength between wet and dry

Table 1. Mean (MPa) and standard deviation (SD) values for micro-shear bond strength in the different enamel groups (n=12)

Substrate treatment	Substrate condition	Period (days)	Mean (±SD)*
No treatment (control)	Wet	1	10.67 (±5.05)[a]
		90	10.42 (±3.23)[ab]
	Dry	1	5.05 (±2.24)[c]
		90	4.72 (±1.34)[c]
Polyacrylic acid	Wet	1	6.59 (±2.65)[bc]
		90	5.75 (±2.10)[c]
	Dry	1	4.36 (±1.40)[c]
		90	5.96 (±1.96)[bc]

*Mean values followed by different superscript letters indicate statistically significant difference (p<0.05).

Table 2. Mean (MPa) and standard deviation (SD) values for micro-shear bond strength in the different dentin groups (n=12)

Substrate treatment	Substrate condition	Period (days)	Mean (±SD)*
No treatment (control)	Wet	1	10.01 (±4.21)[ab]
		90	12.30 (±4.83)[a]
	Dry	1	4.11 (±1.81)[c]
		90	6.61 (±2.84)[bc]
Polyacrylic acid	Wet	1	4.49(±1.83)[c]
		90	5.60 (±2.09)[c]
	Dry	1	2.92 (±1.08)[c]
		90	3.46 (±2.49)[c]

*Mean values followed by different superscript letters indicate statistically significant difference (p<0.05).

conditions, but not between the storage periods. The samples with retained humidity had a better performance than those with a dry substrate, supporting the second hypothesis. In contrast, the increase of the storage period did not influence the shear bond strength as expected, rejecting the third hypothesis. The self-adhesive cement demonstrated similar behavior in both enamel and dentin, regardless of the treatment protocol and substrate condition.

Currently, polyacrylic acid is under study to explore the possibility of this weak acid providing further demineralization to enhance bonding. However, the results of this study show that the use of 11.5% polyacrylic acid resulted in lower shear bond strength than the controls. While the control-enamel-dry-1-day and control-dentin-wet-1-day groups showed bond strength values of 10.67 MPa and 10.01 MPa, respectively, the acid-enamel-dry-1-day and acid-dentin-wet-1-day groups showed values of 5.05 MPa and 4.11 MPa, respectively. Thus, the removal of the smear layer by this acid promotes demineralization that could damage the interaction between the resin and the collagen network on the dentin surface, because the bonding mechanism is similar to that of glass ionomers with an intermediate interfacial layer incorporating partially dissolved smear layer. This finding is in agreement with data from previous studies (12,18). However, different results were found in a study that used higher concentrations of polyacrylic acid (14). In the study of Pavan et al. (2), the used concentration of polyacrylic acid was 25%, which promoted a demineralization more proportional to the degree of penetration of resinous compounds into the dentin surface irregularities. Therefore, more studies are necessary to further the understanding on this subject.

Possibly, some acidic monomers in the self-adhesive cements are able of bonding chemically with the hydroxyapatite in the dental structure. As demonstrated in the present study, the self-adhesive cement requires more water in its interaction on the dental surface in order to achieve higher bond strength values. This finding may be explained by the fact that self-adhesive materials need an ionizing medium for the chemical reaction to get started (5). Thus, the presence of water on the enamel or dentin surface could create a better ionizing medium for the tested material, which would then increase the adhesive's ability to interact with enamel or dentin. Thus, when moisture control is difficult to obtain, as in indirect restorations where the use of rubber dams is complicated, the present study shows that contemporary self-adhesives

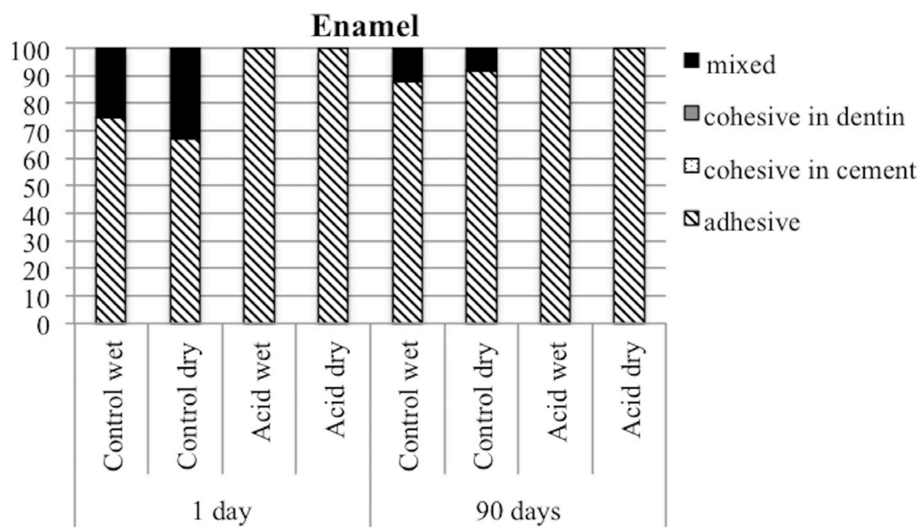

Figure 1. Distribution of failure modes found after micro-shear bond strength test for the enamel groups.

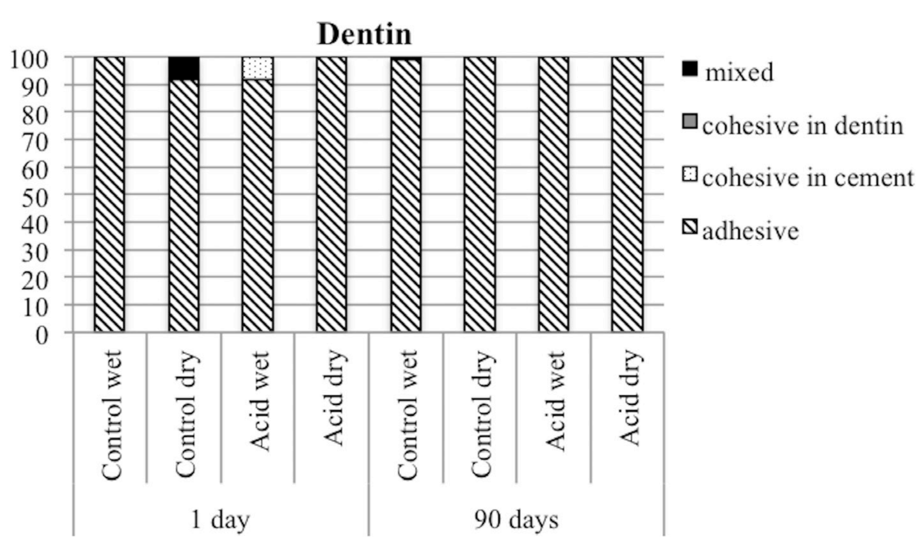

Figure 2. Distribution of failure modes found after micro-shear bond strength test for the dentin groups.

cements could be a better option since they are not negatively affected by moisture on the enamel or dentin surfaces.

Many methods can measure bond strength for determining the effectiveness of adhesion between the cement and tooth structure, like micro-shear and micro-tensile tests (19). Micro-shear test has some advantages, as a more economic use of extracted teeth, better stress distribution at the interface, and facilitates microscopic examination of the failed bonds, owing to the smaller surface area (19). Different sample sizes have been used in similar studies. For example, Costa et al. (16) used 10 samples per group while Kasraei et al. (19) prepared 9 samples per group. In the present study, 12 samples per group were sufficient to provide statistical power.

Further, it is interesting to evaluate long-term behavior of the adhesive strength of the cement-tooth interface. *In vitro* aging may be laboratory-simulated by storage of specimens in aqueous solutions for long periods (3). Immersion in water at 37 °C has been frequently used to simulate aging of the adhesive interface and is a well-accepted procedure in the literature (6). Aqueous solution simulates accelerated aging of resin cements because it has a solubility parameter similar to that of the BisGMA and, therefore, results in maximum softening of the resin, which may be clinically relevant. On the other hand, artificial aging with 10% sodium hypochlorite or ethanol solution may also be performed (6,10). In the present study, immersion in water was for 90 days, but no difference was observed between long-term (90 days) and immediate results (1 day). This finding is positive in the clinical context, where it is expected that the adhesive cementation presents an enduring vitality, and prevent microleakage and maladjustment in prosthetic restorations. However, the variables confronted in this study are insufficient to draw a correlation with the clinical situation. For this purpose, samples should be subjected to changes in pH, thermal cycling, load switches and enzymatic challenges, among other tests (16).

The fracture patterns observed by microscopic analysis of the samples in this research were consistent with those found in several studies of bond strength, demonstrating a higher percentage of adhesive failures (11,21). Nevertheless, there was no significant difference in the type of failure between the control and treatment groups; mixed and cohesive failures found in some samples were related more to an occasional failure in the specimen confection than to poor cement default behavior.

Self-adhesive cements are still considered as new in dentistry. Nevertheless, the values of bond strength of self-adhesive cements, demonstrated in several studies, are similar to those presented by conventional resin cements, such as RelyX ARC (17). The main advantages presented by this system are ease of application and lesser sensitivity to procedures. Although it has been showing promising laboratorial and clinical results (15), further research on these materials is still required.

Based on the results of this study, it may be concluded that the application of 11.5% polyacrylic acid solution decreased bond strength in both dentin and enamel; the samples with retained humidity obtained better bond strength than those with dry substrate; and the storage period did not significantly influence the bond strength of the samples.

Resumo

O objetivo do estudo foi avaliar o efeito de tratamentos de superfície em esmalte e dentina na resistência de união (RU) de um cimento auto-adesivo. As coroas de setenta e dois terceiros-molares foram incluídas em resina acrílica e desgastadas para obtenção de áreas planas em esmalte e dentina. Os espécimes de esmalte e dentina foram aleatoriamente distribuídos em 8 grupos (n=12) de acordo com: tratamento de superfície (sem tratamento ou aplicação de solução de ácido poliacrílico a 11,5%), condição do substrato (seco ou úmido) e período de armazenamento (1 dia ou 90 dias). Após, cilindros (1 x 1 mm) foram confeccionados na superfície dos espécimes utilizando cimento resinoso auto-adesivo (Rely X U200) segundo as instruções do fabricante. Os espécimes foram armazenados em água destilada a 37 °C por 1 dia ou 90 dias e submetidos ao ensaio mecânico de microcisalhamento (EMIC DL 2000 a 0,5 mm/min). O tipo de falha de cada espécime foi determinado após ensaio. Os dados foram submetidos à análise estatística (α=0,05). De acordo com os resultados, a aplicação de ácido poliacrílico a 11,5% diminuiu a RU em ambos substratos, esmalte e dentina. Os grupos mantidos úmidos mostraram maiores valores de RU do que os secos, independentemente do substrato ou tratamento de superfície. O período de armazenamento não influenciou nos valores de RU. Concluiu-se que o tratamento de superfície com ácido poliacrílico a 11,5% e a ausência de umidade no substrato diminuíram os valores de RU para o cimento resinoso auto-adesivo, independentemente do período de armazenamento.

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