Original Research Article

Bond strength of contemporary restorative systems to enamel and dentin

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Abstract

Introduction and objective: New aesthetic restorative systems have been recently introduced in dental market. The objective of this study was to evaluate the bond strength of contemporary restorative systems to ground enamel and medium-depth dentin.

Material and methods: Sixty bovine incisors were prepared by using silicon carbide papers to obtain samples of ground enamel and medium dentin (n=10). The following restorative systems were used: Adper SE Plus/Z350 [SE+Z350], P90 System Adhesive/Filtek P90 [SA+P90] and BeautiBond/Beautifil II [BB+BII]. They were applied to the surfaces, and Tygon tubes were positioned over each sample of hybridized enamel and dentin. The inner space of the tubes was filled in with composites. After light-curing, the tubes were removed and the samples were stored in distilled water at 37°C, for one week. Subsequently, the samples were positioned into a chisel-shaped test device and tested in a universal testing machine (EMIC), with a speed of 0.5 mm/min. Bond strength was calculated in MPa and data analyzed statistically by ANOVA and Tukey test (p <0.05).

Results: No statistical differences were found among groups in enamel. For dentin, [SE+Z350] group resulted in the highest bond strength.

Keywords: dentin; enamel; dentin-bonding agents; shear strength.
strength, while [BB+BII] group showed the lowest bond strength. The comparison between enamel and dentin, for each restorative system, showed that [SE+Z350] resulted in statistically higher bond strength to dentin than enamel. Conversely, [BB+BII] showed higher bond strength to enamel. For [SA+P90], no significant difference was found between enamel and dentin. **Conclusion:** Regarding bond strength, the materials showed different performance in dentin and the same performance in enamel. Only silorane restorative system presented a similar performance in both substrates.

**Introduction**

Although caries disease prevalence is decreasing in the last decades, due to factors as changing of a surgical-restorative paradigm to a health-promotion model and the collective procedures, secondary caries still constitute a problem to be resolved. Other questions inherent to composites, such as polymerization contraction and post-operative sensibility, also have been studied. Accordingly, dental companies have been developing some modified aesthetical restorative materials that present a lower polymerization contraction and/or antimicrobial action [16, 20].

The conventional (prior using of phosphoric acid gel) and self-etching adhesive systems contain, within their mechanism of action, a mechanical and/or retentive combination by chemical components to tooth’s substrates. The hybrid layer formed by the conventional adhesive systems is thicker, presenting resin tags; and the collagen fibrils are not always saturated by monomers. Self-etching adhesive systems are more complex formulations and contain water to facilitate the acid reaction on the tooth surface. It is observed a great pH variation among these materials, due to manufacturers’ specificities. In products showing mild acidity, the hydroxyapatite undergoing the interaction is not completely demineralized. It is believed that the retention could be partially obtained due to the chemical interaction between a more acid monomer and the hydroxyapatite crystal [6, 28, 30].

Currently, a new class of low polymerization contraction composites based on the silorane technology was introduced into market. The silorane composite substitutes the conventional matrix established on methacrylates, aiming to reduce the contraction [2, 13, 29]. Such material is still capable of providing a better hydrolytic stability, due to its hydrophobic property, and it is, therefore, less susceptible to water degradation [1, 12].

Also currently available, other restorative composite presents pre-reacted glass ionomer particles formed by an acid-base reaction of the fluoraluminosilicate glass with polyacrylic acid [11] and fluoride releasing capacity. Gordon et al. (2007) [8] reported that these products’ property of inhibitory biofilm formation (so-called glomers) is important and appropriate for some restorative treatments. However, their action mechanism and effectiveness have not already been clarified.

The aim of this study was to evaluate the bond strength (BS) of contemporary restorative systems to ground enamel and medium-depth dentin in bovine teeth.

**Material and methods**

Sixty freshly extracted bovine incisors, stored in freezer until sample construction, were used. The roots were sectioned by double-faced flexible diamond disc (#7016, KG Sorensen, Barueri, SP, Brazil), under refrigeration. Silicon carbide papers (#180 and #400) (Carborundum, Vinhedo, SP, Brazil) mounted in an electrical water-cooling polishing machine, were used for wearing the proximal, incisal, and lingual enamel surfaces. Following, the labial surfaces were also worn up to obtain 30 flat surfaces of ground enamel and 30 samples of flat medium-depth dentin. Samples presented approximately 100 mm$^2$ – 10 mm height x 10 mm width x 5 mm depth. In order to obtain standardized enamel surfaces and fresh smear layer at medium-depth dentin, prior to the adhesive procedures, each enamel and dentin sample was worn under refrigeration by silicon carbide papers (#600).

Three restorative systems were used according to the manufacturers’ instructions. The experimental groups were divided, respectively, in accordance with the ground enamel and medium-depth dentin samples: [1] and [4] Adper SE Plus + cylinders of Filtek Z350; [2] and [5] P90 System Adhesive + cylinders of Filtek P90; [3] and [6] BeautiBond + cylinders of Beautifil II (n = 10).

Board I shows the employed materials, manufacturers’ names, batch number, and expiration dates.
<table>
<thead>
<tr>
<th>Name, acronym, and pH</th>
<th>Manufacturer</th>
<th>Batch number</th>
<th>Expiration date</th>
</tr>
</thead>
<tbody>
<tr>
<td>One-step self-etching adhesive system (all-in-one) – BeautiBond (BB) pH=2.4</td>
<td>Shofu</td>
<td>100807</td>
<td>9/2011</td>
</tr>
<tr>
<td>Restorative composite Filtek P90 – A3 – silorane (P90)</td>
<td>3M ESPE</td>
<td>9ER</td>
<td>11/2010</td>
</tr>
<tr>
<td>Restorative composite Beautifil II – A3 – fluoride particles (BII)</td>
<td>Shofu</td>
<td>100872</td>
<td>9/2011</td>
</tr>
</tbody>
</table>

For preparing the samples for the microshear bond strength test, we used the methodology developed by McDonough et al. (2002) [15] and Shimada et al. (2002) [22]. Three transparent cylindrical tubes (Tygon Tubing, TYG-030, Saint-Gobain Performance Plastic, Maimi Lakes, FL, USA) were positioned on the ground enamel and medium-depth dentin of each previously hybridized sample. The tubes inner space were filled in (0.7 mm) with the composites through dental probe number #5 (SSWhite/Duflex, RJ, Brazil). After light-curing, the tubes were removed by sharp scalpel (Gillette, SP, Brazil) to expose the small composite cylinders (0.7 mm width x 1.00 mm height), with bond area of 0.38 mm² (obtained by the following formula: $\pi R^2$) bonded to ground enamel and medium-depth dentin. All light-curing procedures were executed by a light-cured device LED Radii Cal (SDI), at 1200 mW/cm². Thus, three composite cylinders were bonded to each ground enamel and medium-depth dentin sample. Samples were kept in distilled water, at 37°C, for one week. Following, samples were tested in universal testing machine (Emic, Sao Jose dos Pinhais, PR, Brazil). The compression load resulting in the shear bond strength was performed at the cylinder bases by chisel-shaped test device, at 0.5 mm/min speed, up to bond disruption. BS was calculated by microshear bond strength and the values were expressed in MPa. Data were evaluated through two-way ANOVA and Tukey’s test, at level of significance of 5%.

**Results**

Two-way ANOVA found statistical significant differences. Tukey’s test identified differences at 5% level of significance. Means and standard deviation are seen in table I.
Table I – SB means (MPa ± SD) – Tukey’s test (p < 0.05)

<table>
<thead>
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<tbody>
<tr>
<td>Ground enamel</td>
<td>[1] 6.6 (2.2) A b</td>
<td>[2] 5.2 (1.6) A a</td>
<td>[3] 5.5 (2.7) A a</td>
</tr>
</tbody>
</table>

Line – Same capital letters indicate means without significant differences by Tukey’s test (p<0.05)
Column – Same minuscule letters indicate means without significant differences by Tukey’s test (p<0.05)

There were no statistically significant differences among the materials in ground enamel. In medium dentin, [SE+Z350] resulted in a higher SB mean, while [BB+BII] showed the lowest one, with statistically significant differences for all groups. When ground enamel and medium dentin were compared, [SE+Z350] showed the highest BS mean for dentin, with significant statistically difference. [SA+P90] did not present expressive statistically significant difference at both ground enamel and medium dentin. When [BB+BII] was applied to enamel, SB result evidenced the highest mean values, showing statistically significant difference.

Discussion

Currently, the adhesive systems have been classified into two categories: those in which acid-etching is performed separately by phosphoric acid gel (at concentrations varying from 35 to 37%) to demineralize tooth’s substrates; and self-etching adhesive systems of two- or one-step (all-in-one), according to the time of application onto tooth. It is possible to find the one-step product displayed in two flasks – two liquids to be combined – or in one flask. Due to the increasing demand for less complex procedures for bonding, and also aiming to reduce the technique sensibility, there is a trend towards using one-step self-etching adhesive systems. These are composed of a complex mixture of primers, acids, and bond agents, among others [4, 9]. In our research, two two-step adhesive systems and one all-in-one was used. [SE+Z350] restorative system was used as control because such material class (self-etching adhesive systems and nanoparticulated composites), regardless their manufacturer, have been very used, mainly in developing countries of the Northern Hemisphere [5].

All-in-one products, although very modern because of the technology employed in their manufacturing (complex multifunction monomeric molecules), are related to the concept of nanoleakage, i.e., the visualization of areas impregnated by silver in electronic microscopy analysis [21]. The high degree of nanoleakage at the hybrid layer (proposed by Nakabayashi et al. [17], in 1982) could be attributed to the areas not reached by the adhesive system, showing a high concentration of solvent and, mainly, water which is indispensable to ionize the product and start the demineralization process after its contact with tooth’s substrate. Nevertheless, nanoleakage is still higher in the conventional adhesive systems that demand a moist dentin for their application [23, 27].

We used bovine teeth due to several studies, such as Reis et al. (2004) [19], which proved that there are no statistically differences in BS when human teeth are compared to bovine, both for enamel and dentin. The analysed products were called “contemporary restorative systems” in order to state that some of these materials are not compatible with those employed in both daily clinic practice and in vitro investigations.

Recently available in the Brazilian market, Adper SE Plus self-etching adhesive system comprises the concept of nanotechnology in its composition, i.e., it seeks to increase BS, facing the several physical-chemical challenges that occur in oral environment. The product eliminates the acid-etching step with phosphoric acid gel, rinsing, and maintenance of a moist dentin. Therefore, the risk of post-operative sensibility is reduced, because both action mechanisms occur simultaneously, and thus, dentin is saturated by the resin monomers. A product’s differential is the warranty of the exact place where the adhesive was applied, through changing its color at the moment of the bond application. Besides that, it is radiopaque, which decreases the risk of diagnosis errors [14].

Used with the aforementioned adhesive, the restorative composite Filtek Z350 contains a combination of non-agglomerated silica nanoparticle fillers of 20 nm with zircon / silica nanoagglomerates of 5-20 nm, varying about 0.6 to 1.4 µm. Such properties are related to handling, smaller polymerization contraction (main problem of the products based on methacrylate, such as Adper SE Plus and Z350), higher mechanical resistance, and physical-chemical properties, specially, polishing maintenance that is very important for aesthetics.
This influences on the marginal leakage, and promotes smaller biofilm retention on tooth surface [10].

In our study, the described restorative system did not show expressive statistically significant differences in ground enamel in relation to the other studied groups. According to García et al. (2007) [7], the ground substrate, as described in the methodology, generates a higher BS when compared to sound enamel. We suspected that pH 1.0 was not sufficiently acid to demineralize the ground enamel, because this product has a performance similar to the others whose pH were less acid. In medium-depth dentin, however, there was a higher BS, with statistically significant differences among the restorative systems used and regarding to enamel. Unlike what occurred in enamel, we believed that pH 1.0 was able to demineralize such substrate, which contains less mineral, is more heterogeneous and physiologically more dynamic [6]. The findings of Yoshida et al. (2004) [30] justified the relationship between the tooth substrate demineralization and its pHs, as well as, the complex formulations of the contemporary adhesive systems. Although phosphoric acid mean pH is 0.6 (not very distant from pH 1.0 of the adhesive), it is worth noting that the adhesive system contains in its formulation, hydrophilic and hydrophobic components, while phosphoric acid gel actions separately on tooth's substrates.

Concerning to silorane-based materials, its resin matrix is very different from conventional materials based on methacrylates [16]. Consequently, a new adhesive system was made for promoting these materials bonding to enamel and dentin. Filtek P90 is sold with a self-etching adhesive system – P90 System Adhesive -, which has the features of methacrylate-based adhesive regarding to the mechanism of bonding to tooth tissues. However, adaptations were necessary to make P90 System Adhesive compatible with the high hydrofobicity of the silorane matrix. The aforementioned adhesive system comprises the application of two resin solutions: the first one is hydrophilic and bonds to the substrate; the second, hydrophobic, bonds to Filtek P90. On the other hand, the first solution needs to be separately light-cured, which does not occur to other adhesive systems.

According to Tezvergil-Mutluay et al. (2008) [24], the polymerization reaction of silorane is different. The oxygen, a polymerization inhibitor agent, links to the free radicals of the conventional methacrylate-based monomeric systems. The layer inhibited by oxygen is considered an advantage in the bond resistance among the following layers. It improves the monomeric net bonding by forming a covalent union. Silorane materials, however, undergo a cationic reaction, in which the layer inhibited by the oxygen does not exist on the polymerized surface.

The described restorative system showed the lowest BS for ground enamel, although without statistically significant difference regarding to other groups. Also, silorane did not present statistically significant differences when the substrates were compared between each other. Since the adhesive system presents pH 2.7 (the least acid system of all the systems studied), accordingly, we believed that this system did not severely etched both the ground enamel and medium-depth dentin. Because the bonding between successive layers depend only on the composite reactivity, as already observed, and corroborating the findings of García et al. (2008) [5], this system often presents a smaller BS when compared to methacrylate-based composites, which display, on the other hand, a higher polymerization contraction. This contraction is not very much intensified in silorane-based products (mean of less than 1% of volumetric contraction, in comparison with more than 2% of methacrylate-based resin materials).

By analyzing the behavior in dentin of the three restorative systems, silorane showed a good performance regarding BS, but intermediary and with statistically significant differences for all groups. Although a pH 2.7 is less acid than the pH 2.4 of the giomer's adhesive system, silorane presented a higher BS, almost twice the mean obtained in the giomer group.

According to Gordan et al. (2007) [8], Beautifil is a restorative system, so-called giomer, which uses a methacrylate-based resin and pre-reacted glass ionomer particles. This technology is subclassified into: F-PRG – fully-reacted pre-reacted glass; and S-PRG – surface-reacted pre-reacted glass. The in vitro researches of Okuyama et al. (2006) [18] revealed that fluoride releasing by this material was small, during a 21-day period, followed by a significant reduction after this time. Notwithstanding, there are interesting technologic evidences involved in the production of this material. These aforementioned particles are made through a reaction of acid-reactive glasses containing fluoride with polialcenoic acid, in water, prior to their incorporation into resin components. This technology (S-PRG) is different and more effective, considering the physical and chemical properties, than that employed in compomers'
manufacturing, for example. For these latter, the dehydrated polialcenoic acid is a part of the resin matrix and the reaction between the glass and acid does not occur since the water is absorbed by the material.

Most of self-etching adhesive systems contain phosphoric acid ester derivatives into a mixture of water and solvents. This acid component conducts the reaction during enamel and dentin’s demineralization. The formulation based on the all-in-one adhesive system, so-called BeautiBond, employs an interesting chemical approach for maximizing the union (mechanic) and bonding (chemical) to tooth substrates. BeautiBond contains a monomer of carboxylic acid that promotes bonding to dentin, and phosphonic acid to generate bonding to enamel [11]. This is a single approach, taking into account that one-step all-in-one self-etching adhesive systems often present very specific demineralizing properties in their compositions, both for enamel and dentin.

In our study, the fluoride-releasing restorative system showed a higher ground enamel BS, without statistically significant differences among groups. Although its pH (2.4) has been considered as mild for enamel's demineralization, product’s intrinsic formulation containing phosphonic acid was more effective than those responsible for dentin’s demineralization, which showed the smallest BS mean, with statistically significant differences among groups.

Although BS means are very below those found by Urabe et al. (2000) [25], we considered our results, both for enamel and dentine substrate, logical. Escrivano et al. (2003) stated that there is a controversy when simply comparing BS data among the studies in literature, due to variations in the techniques employed and in the several conditions under which the substrates are stored or used. According to the same authors, the results of several types of shear bond, tensile, or compression tests, depend on the samples' storage and tested area, and on the devices and machines used, among others.

**Conclusion**

According to the results and their statistical analysis, it can be concluded that, considering BS, the restorative systems show a similar performance for enamel and different for dentin. Only silorane-based material presented a similar performance for both tooth’s substrates.

**References**


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