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Shear Bond Strength of Three Adhesive Systems to Bovine Dentin

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Resumo: O objetivo desta pesquisa foi avaliar a resistência de união ao cisalhamento de um sistema adesivo autocondicionante e compará-lo a dois sistemas adesivos de quinta geração. Foram utilizados trinta incisivos bovinos hígidos, cuja superfície vestibular foi desgastada até a exposição da dentina. Os espécimes foram divididos em três grupos (n = 10) de acordo com o sistema adesivo utilizado: Optibond Solo Plus (OPBS); Prime & Bond NT (PBNT) e Clearfil SE Bond (CSEB). Confeccionou-se um cilindro de resina composta (Z - 100) pela técnica incremental, utilizando-se uma matriz em teflon. Após a confecção dos corpos-de-prova, os espécimes foram termociclados (300 ciclos: 5°C e 55°C). Realizou-se o ensaio de cisalhamento em máquina universal Instron (célula de carga = 500 kg, velocidade = 0,5 mm/min). Os dados (MPa) foram submetidos aos testes paramétricos ANOVA (1 fator) e de comparação múltipla de Tukey ($\alpha = 0,05$). Os resultados obtidos demonstraram que o grupo OPBS (21,20 ± 3,32) apresentou um desempenho superior aos demais grupos, seguido pelo grupo PBNT (14,37 ± 2,62). O grupo CSEB (autocondicionante) apresentou média de resistência adesiva (11,00 ± 2,78) significantemente menor do que os outros dois adesivos testados. Baseado nestes resultados concluiu-se que o sistema adesivo autocondicionante apresentou o menor desempenho em relação à resistência mecânica quando comparado aos adesivos de quinta geração avaliados.

Palavras-chave: *Resistência adesiva ao cisalhamento; sistemas adesivos.*

Abstract: The aim of this study was to evaluate the shear bond strength of a self-etching primer Clearfil SE Bond (CSEB) and to compare it to two five generations adhesive systems Optibond Solo Plus (OPBS) and Prime & Bond NT (PBNT). Thirty noncarious bovine incisors were embedded in acrylic resin boxes. After the exposure of dentin, the specimens were randomly divided into three groups (n = 10) according to the adhesive systems. It was constructed a cylindrical restoration in composite resin (Z - 100) by incremental insertion technique. The samples were thermal cycled (300 cycles: 5°C and 55°C) and subsequently the shear bond strength tests were performed on an Instron Universal machine, at 0.5 mm/min speed. The data (MPa) were submitted to analysis of variance using ANOVA one-way test and multiple comparison range ($\alpha = 0.05$). The adhesive system OPBS presented the best mean value (21.20 ± 3.32) followed by PBNT (14.37 ± 2.62). The self-etching adhesive CSEB showed mean shear bond strength (11.00 ± 2.78) significantly lower than the other two adhesive systems tested. It can be concluded that the 5th generation adhesive systems tested showed higher bond strength that the self-etching adhesive system.

Keywords: *Shear bond strength; adhesive systems.*

Introduction

One of the most important requirements of a restorative material is its bond capacity to the dental tissue, which can preserve a healthy dentin structure and substitute biologically, aesthetically and functionally what was lost due to carious lesions, trauma or other pathologies. The search for this material has stimulated a number of studies about adhesive systems.

When Buonocore[3], in 1955, introduced the enamel acid etching technique, the restorative dentistry went different ways. Since then, there has been significant improvements in the marginal sealing of a restoration in which its margins were placed on the enamel, also the preparations became conservative once the need of mechanical retention was eliminated, preserving the healthy dental structure.

However, bonding to the dentin has become a great challenge for the researchers, because of the striking histomorphological differences presented in relation to the enamel, which justify the unique approach for each tissue treated with the same adhesive system.

Moreover, dentinal instrumentation results in a smear layer which covers the structural components of dentin and penetrates into the tubules to form smear plugs.[13]

There are different mechanisms of adhesion currently used, depending on the clinical approach of the smear layer: it can be removed by acids or dissolved by self-etching primers.[37]

The concepts of total acid etching and dentin hybridization, proposed by Fusayama et al.[7], in 1979 and Nakabayashi et al.[20], in 1982, respectively, constituted the base of the fourth and fifth generations adhesive systems, which differ only by the number of clinical steps, because for the fifth generation adhesives, primer and hydrophobic resin are combined in a single bottle.

The formation of a high quality hybrid layer is achieved by the diffusion of the monomer among the collagen fibers, and this is the main bonding mechanism of the current adhesive systems that uses acid etching. However, this surface rich in collagen can collapse during rinsing and drying of the acid, interfering in the monomer diffusion. Another factor that can make the monomer diffusibility difficult is the overetching that alters the three-dimensional structure of the collagen fibers, by denaturating them.[18]

In addition, if dentin is overetched, the adhesive can be unable to fully penetrate to the base of the exposed collagen, forming a porous zone at the base of the hybrid layer.[29]

Due to the technique complexity and sensitivity, the innovations of the adhesive systems are directed toward a simplified application process. With the objective of avoiding the collapse of the collagen network and to simplify the clinical technique, the self-etching primer systems were developed and launched in 1994. They intent to dissolve the smear layer, and to incorporate it in the restoration, this leads to new discussions and studies about its use as a subtract to an adhesive system.[19,27] A hypothesis emerges: the self-etching adhesives provide equal or superior bond strength than the fifth generation adhesives? Based on this questioning, this investigation aims at evaluating the shear bond strength of two fifth generation adhesive systems and to compare them to the performance of a self-etching adhesive system.

Material and method

Thirty 3-year-old bovine incisors, erupted and intact, were extracted immediately after slaughter. The teeth were cleaned and their roots were sectioned in half with a low-speed diamond saw, allowing the pulp removal. By means of a round diamond bur, in a high-speed handpiece with copious water, a coronal access in the lingual face of teeth was prepared, until the pulp chamber exposition. The teeth were immersed in distilled water, frozen at -20 °C and used within 28 days of extraction.[8,16,36]

The labial enamel area to be ground was delimited with a graphite and a graduated ruler, to standardize the labial superficial dentine to be exposed for the test. The pulp chamber and the root canal were filled with gutta-percha and sticky wax, to avoid penetration of embedding media. The teeth were mounted in a silicone matrix (Rhodorsil - Classico Art. Odontológicos - Ind. Bras.), with self-cured acrylic resin (Classico Art. Odontológicos - Ind. Bras.), keeping the signed enamel area above the surface of the mounted blocks and parallel to the base plane. The acrylic resin blocks were placed into tap water to reduce the temperature rise from the exothermic polymerization reaction.[17]

The labial enamel was ground with a wet 80-grit silicon carbide (3M Brazil) on a water-cooled model trimmer (Kohl Bach S.A., Brazil) to expose the dentin. The pulp chamber access was cleared to allow the measure of the remaining dentin thickness, with a thickness spring caliper (Otto-Arminger & Cia Ltda. RS, Brazil). The dentin thickness was standardized in 2.0 ± 0.1 mm, remaining in superficial dentin, according to Nakamichi et al.[21], 1983. The dentin was regularized and polished with sequential 240, 400 and 600-grit sandpaper (3M Brazil), during 20 s each, to create an uniform smear layer.

The dentin was cleaned with distilled water for 10 s and dried with absorbent paper. To limit the area for the application of the adhesive systems, a special Scotchtape Mold (3M Brazil) with a standard central hole, 3 mm in diameter, was placed on each specimen.

The prepared teeth were divided into 3 groups (n = 10), according to the adhesive system. The adhesive systems used for this study are outlined in Table 1.

The specimens of Groups 1 and 2 were acid etched with 37% phosphoric acid for 15 s, rinsed, brief air dried and

Table 1. Materials used, with respective classification, batch numbers and manufacturers.

Materials	Classification	Batch No.	Manufacturer
Optibond Solo Plus (OPBS)	5th generation	009168	Kerr
Prime & Bond NT (PBNT)	5th generation	0008000480	Dentsply/Caulk
Clearfil SE Bond (CSEB)	Self-etching	51207	Kuraray America

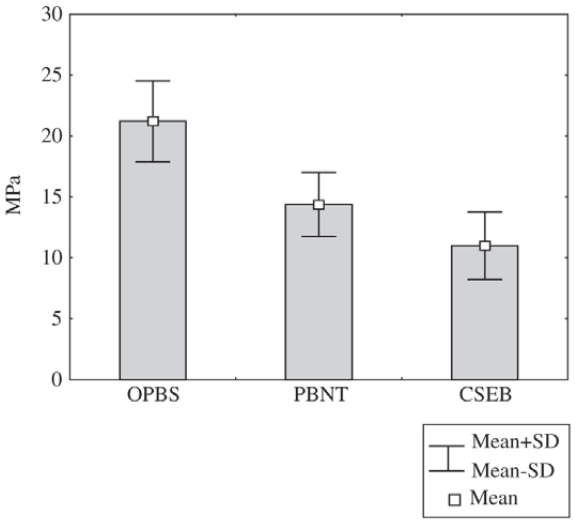

Figure 1. Graphic of the mean and standard deviation (MPa) for the three materials tested.

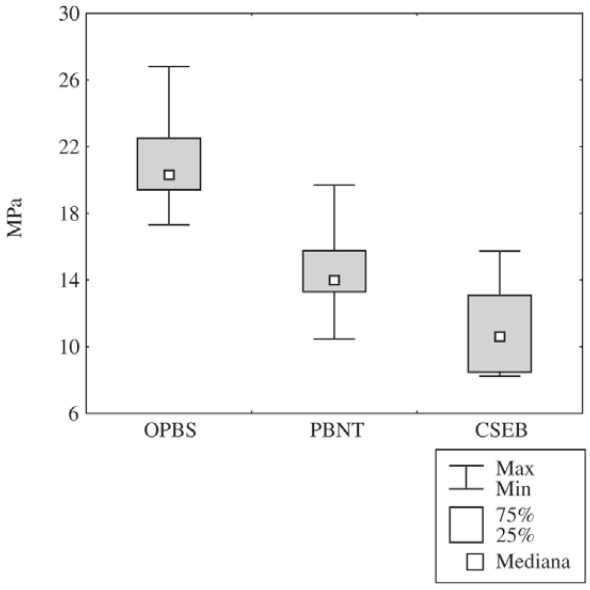

Figure 2. Box and Whisker Plot graphic of the data (MPa).

received the application of the adhesive systems OPBS and PBNT, respectively. In the Group 3, it was applied the self-etching adhesive system CSEB, according to the manufacturer's instructions.

A split Teflon mold was adapted to the specimens to insert the composite resin. With this mold it was possible to make the composite resin cylinders Z100 (3M Brazil) directly on the dentin surface. The insertion was done in 3 layers, each layer was light cured for 40 s (Optilux - Demetron Research Corp.; 550 mW/cm^2). Consequently, composite resin cylinders with 3 mm in diameter and 3 mm in height were bonded to dentin surfaces.

The specimens were identified and stored in 37°C distilled water for 24 h and then subjected to 300 thermal cycles, between 5°C and 55°C.

Each specimen was locked in a special device, designed by Pagani[25], 2000, to undergo laboratorial testing in an Instron universal testing machine (Instron Corp.), with a load cell of 500 Kg. The specimen were subjected to shear force, using a point with a 0.5 mm thickness edge. This point was applied in the base of the composite cylinder, parallel to the dentinal surface, with a cross-head speed of 0.5 mm/min, avoiding lever force.[6, 38, 35]

The data obtained in Kgf were transformed in MPa and then, were tabulated, codified and subjected to statistical analysis.

Result

The data of shear bond strength (MPa) obtained with the Instron machine are shown in Figuras 1 and 2. In these Figures, the values of central trend (mean and median) and dispersion (standard deviation, values range and interquartis range) are presented.

It was observed, as is noted in Figuras 1 and 2 that: (i) there is no overlapping of the interquartis range, showing the variability among the adhesive systems; (ii) the mean and median values behave the same way, that is, the OPBS overcomes the other materials and the PBNT occupies an intermediary position.

The mean shear bond strength obtained with OPBS (21.20 ± 3.32 MPa) is different from the PBNT (14.37 ± 2.62 MPa) and CSEB (11.00 ± 2.78 MPa). According to the one-way ANOVA, this difference is significant ($F_{2;\,27}$ = 31.59; p-value = 0.001) at 0.05 significance level.

Applying the Tukey test ($\alpha = 0.05$), it was observed that the adhesive systems differ significantly.

Discussion

This research evaluated three adhesive systems available in the market, by shear bond strength test. The strength was applied on the base of composite resin cylinders, in a similar way as described by Okamoto & Nakabayashi[23] (1995), May et al.[14] (1997), Sinhoreti et al.[33] (1997). This assay is evaluated by International Standard Organization[9], in 1994, which rules mechanical tests to measure the bond strength to the dentin substrate.

The bond strength is an important data on the investigations about adhesion because it provides information about the bond capacity, represented by the hybrid layer, of absorbing the load energy.[11, 5, 38]

The use of bovine teeth is justified by the studies of Nakamichi et al.[21], 1983; Retief et al.[30], 1994; Coradazzi et al.[4], 1998 and Schilke et al.[32], 2000, who state that bovine dentin is a viable alternative to human dentin in adhesion studies.

The statistical analysis verified significant differences among the adhesive systems evaluated (Figuras 1 and 2). The two first, classified as belonging to the fifth generation, presented load particle to increase the hardness. The PBNT adhesive has extremely fine load, in nanometric scale (around 7 nm), to allow better permeation of the material by the collagen fibers of the demineralized dentin.[2, 39]

The values obtained for the adhesive PBNT agree with those of the researches performed by Perdigão et al.[28], 1999; Rodrigues et al.[31], 1994 and Tâmbara et al.[34], 2001. Among the adhesive systems evaluated, the OPBS presented shear bond strength statistically higher than the others, which agree with the results of Perdigão et al.[28], 1999 and of Lopes et al.[12], 2001 who found superiority in the OPBS in relation to the PBNT.

Besides the difference in the size of the particles, it should also be considered that both adhesives present distinct solvents, alcohol in the OPBS and acetone in the PBNT, which could partly explain the difference in diffusibility of the monomers and could affect the results.[18]

The high quality of the hybrid layer is related, among other factors, to its thickness, that is, the diffusion of the adhesive in the collagen network of the demineralized dentin. On the conventional hybrid layer, the demineralized collagen fibers remain continuous with the underlying mineralized collagen fibers. Therefore, the stress supported in this case is absorbed by the resin-collagen fibers.[18]

The thickness of the hybrid layer formed by the self-etching adhesive is smaller when compared to the one formed by the fourth and fifth generation adhesive systems, according to studies of Lamosa et al.[10], 2001; Miranda et al.[15], 2001 and Arrais and Giannini[1], 2001. This fact added to the lack of structural continuity between the smear layer and the underlying dentin, can explain the low bond strength presented by the self-etching adhesive evaluated in this research. Despite the value variation, the performance of this material agrees with studies which observed better results for adhesives of fourth and fifth generation, when compared to the self-etching ones.[24, 26]

The application technique of the self-etching adhesives is easier, but in order to be considered as high quality material, they must present a performance equal or superior to the fifth generation adhesives. However, this simplicity in its use, in which critical steps such as etching time and drying process are excluded, is associated to the formation of a thinner and less homogenous hybrid layer. This fact can be explained by the difference between the pH of the acid primer of the Clearfil SE Bond (2.04) and of the 35% phosphoric acid (0.6), which justifies the difference in the adhesive permeability and therefore the thickness of the hybrid layer.

As the results of this study are evaluated, it must be taken into consideration that the smear layer formed during the dentin preparation can interfere in the depth of the demineralized dentin when the self-etching adhesives are used, since the acid primer needs to be diffused through it. Researches about this kind of material have, thus, evaluated their behavior according to the selective use of burs considering that the dentin preparation determines the quality and thickness of the smear layer, and can interfere with the bond strength values.[22] It is recommended, therefore, that more researches be performed, in order to improve the self-etching adhesives, since it is a great advantage in reducing clinical steps.

Conclusion

Based on the experimental conditions used in this study, it follows that:

- there were significant differences among the shear bond strength of the three adhesive systems tested;
- the self-etching adhesive performed worse than the 5th generation adhesive systems;
- comparing the 5th generation adhesives tested, Optibond Solo Plus presented a significantly higher mean shear bond strength than Prime & Bond NT.

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