INFLUENCE OF PRE-SILANIZED GLASS MICROFIBERS ON BOND STRENGTH AND NANOLEAKAGE OF TWO DENTIN-BONDING AGENTS

Celso Afonso Klein-Junior[1], Ariane da Silva Camargo, Daniel Machado de França, Maria Carolina Guilherme Erhardt[2], Fábio Herrmann Coelho-de-Souza[2]

1 Department of Dentistry, Lutheran University of Brazil, Canoas, Brazil.
2 Department of Conservative Dentistry, Federal University of Rio Grande do Sul, Porto Alegre, Brazil.

CORRESPONDING AUTHOR: fabio.herrmann@yahoo.com.br

ABSTRACT

Aims: The aim of this study was to evaluate the microtensile bond strength (µTBS) and nanoleakage of two dentin-bonding agents (Adper Single Bond 2 (SB) and Adper Prompt L-Pop (PLP) (3M ESPE), with addition of 10% of pre-silanized glass microfibers.

Materials and methods: Twelve extracted third molars were used, which had the occlusal enamel removed. The modified dentin-bonding agents were applied on the occlusal surface of the teeth, following instructions of the manufacturers. Thus, four groups were obtained: G1 - control SB, G2 - Modified SB, G3 - control PLP and G4 – modified PLP. A 4mm composite restoration was built on the entire area of dentin (Z250-A2 3M ESPE) by incremental technique. The restored teeth were sectioned into sticks (1.2 mm x 1.2 mm 7.0 mm). Two sticks of each group were used for nanoleakage analysis by scanning electron microscope (SEM). The µTBS test was conducted with a crosshead speed of 0.5 mm/min. Data were statistically analyzed by Student's t test, with a significance level set at 5%.

Results: The results showed statistically significant differences between G1 and G2 (G1-11.21 MPa +/- 2.69; G2 - 18.21 MPa +/- 7.31) and G3 and G4 (G3- 6.13 MPa +/- 1.62; G4 -18.95 MPa +/- 7.69). The descriptive analysis of SEM images showed less nanoleakage in G2.

Conclusion: It is concluded that the addition of 10% of pre-silanized glass microfibers increased the bond strength to dentin of the adhesive systems used and improved the quality of the hybrid layer for Adper Single Bond 2.

KEYWORDS: Dentin-bonding agents. Composite resins. Bond strength.
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INTRODUCTION

The evolution of adhesive systems has brought important changes to restorative practice, making the cavity preparations more conservative by utilizing the dental substrate for chemical or micromechanical retention[1]. While adhesion to enamel is generally more stable, dentin is still considered a challenging substrate, particularly in terms of long-term degradation[2-5].

Several techniques have been proposed to improve the performance of adhesive systems, including the active application of primer/adhesive, heating the air to volatilize solvents, applying multiple layers, utilizing different smear layer treatments, incorporating particles and fibers into adhesives, and introducing bioactive ions for dentin remineralization within the hybrid layer[6-10].

Various mineral compositions have been used in adhesive systems4 and to produce fibers. Glass fibers, for instance, are made from silica (SiO_2) with the addition of calcium oxide, boron, sodium, and aluminum. These fibers are amorphous materials, and their crystallization occurs after prolonged treatment at high temperatures[11]. The use of fiber tissue must be accompanied by an adhesive, creating a composite that combines the properties of both materials to create a product with enhanced characteristics beyond those of each individual component. The fibers contribute to the composite's strength while the matrix binds them together, facilitating the transmission of forces. Therefore, the primary purpose of using reinforcements in polymers is to increase the rigidity and strength of structural parts. Controlled incorporation of fibers into a material leads to improved resistance[12-14].

Even though dental adhesives have a good bond strength, these are susceptible to degradation overtime[4,5] and still lack cohesive strength, because their polymeric components have a small number of particles, as well as, in some cases, having solvents. The structural components of adhesives in general suffer polymerization of HEMA and BISGMA molecules and some co-polymers. During this polymerization, particles could be encapsulated into the adhesive polymer to improve mechanical, cohesive and rheological properties. Thus, the addition of glass microfibers compensates the low resistance of the BISGMA and HEMA, changing its mechanical properties. An adhesive polymer with fibers in its composition could provide gains in mechanical properties, bringing benefits to the bonding process[10].

Thus, the aim of this study was to evaluate the microtensile bond strength and nanoleakage (scanning electron microscope) of two dentin-bonding agents (Single Bond 2 and Prompt L-Pop-3M ESPE) with addition of 10% of pre-silanized glass microfibers.

MATERIALS AND METHODS

Selection of teeth

Twelve healthy molars were used to carry out this research. The teeth were obtained through donations from patients who had the teeth extracted for therapeutic reasons and signed a term donation (approved by Lutheran University of Brazil ethical committee). The teeth were cleaned, disinfected in chloramine 0.5% for 7 days and stored in distillate water.

Preparation of the teeth

The teeth were included in PVC cylinders with 25mm of diameter and 20mm height, which were filled by acrylic resin (Jet Clássico, São Paulo, Brazil). Then, the teeth had their occlusal surface worn using an abrasive disk (80 rpm) under constant water-cooling. This wear was performed until the exposure of dentin (all enamel was removed), followed by the application of 600-grit sandpaper for 20 seconds (under water-cooling).

Modification of adhesives

Both adhesives tested (Adper Prompt L-Pop and Adper Single Bond 2 - 3M ESPE, St. Paul, MN, USA) were subjected to an addition of an amount of 10% by weight of glass microfiber, mixed into the adhesives (based on the amount of adhesives). These fibers had been previously silanized (Angelus – Londrina, PR) and had ground to smaller grain possible (an analytical balance was used to do the weighing of the adhesive and the microfibers).

Teeth restorations

The teeth were divided into 4 groups (n = 3 teeth): Group 1 - Adpter Single Bond 2 (SB), Group 2 - Adpter Single Bond 2 modified with pre-silanized glass microfiber, Group 3 - Adpter Prompt L-Pop (PLP), Group 4 - Adpter Prompt L-Pop modified with pre-silanized glass microfiber.

Group 1: A 37% phosphoric acid was applied for 15 seconds, then it was rinsed, and the dentin was softly drying with absorbent paper, keeping the surface slightly wet, then two coats of Adper Single Bond 2 was applied using a microbrush (Brush / KG Sorensen, Alphaville, São Paulo) on the surface of exposed dentine, followed by an application of a gentle blast of air to remove excess of solvent. The adhesive was lightcured for 10 seconds with Led unit (Smart Light, Dentsply). After the dentin-bonding agent, the composite resin was applied in small increments (2mm) and lightcured for 20 seconds to build a plateau of 4mm height.

Group 2: the same description of group 1, but with adhesive system Adper Single Bond 2 modified by glass microfibers.

Group 3: a layer of adhesive system Adper Prompt L-Pop was applied on the occlusal dentin surface, with a microbrush. Two coats of the adhesive were rubbed on the dentine, followed by a gentle air blast to evaporate the solvent, and lightcured for 10 seconds. After the dentin-bonding agent, the composite resin was applied in small increments (2mm) and lightcured for 20 seconds to build a plateau of 4mm height.

Group 4: the same description of group 3, but with adhesive system Adper Prompt L-Pop modified by glass microfibers.

Specimens' production

Restorations were sectioned into sticks, perpendicularly to the bonded interfaces, by a diamond saw (Isomet 1000, Buehler, Ilinois, USA).

Specimen bonding area was measured with a digital caliper

Table 1. Comparison of data among control groups and modified by fibers for both adhesive systems.

Group	*n*	*Mean*	*Standard deviation*	*t*	*p*
Single Bond2					
Control	10	11.21	2.69	-2.83	0,012
Fibers	9	18.21	7.31		
Prompt L-pop					
Control	9	6.13	1.62	-4.89	0.001
Fibers	9	18.95	7.69		

(Absolute Digimatic, Mitutoyo, Tokyo, Japan). The sticks had a cross-sectional area of 1.2 mm^2. Half of the specimens were tested in microtensile bond strength test (µTBS – n=10 for each group). Other sticks were analyzed by scanning electron microscope (SEM), in relation to nanoleakage (n=2 for each group). Sticks in the enamel-dentin junction were excluded.

Microtensile bond strength test (µtbs)

For (µTBS) test, each stick was attached to a testing device with cyanoacrylate (Zapit, Dental Ventures of North America, Corona, CA, USA) and subjected to a tensile force at a crosshead speed of 0.5 mm/min, performed on a universal testing machine (VERSAT 502M), using a cell of 500N.

Data obtained in the µTBS test were statistically analyzed by Student's t test, with a significance level set at 5%.

Analysis of nanoleakage

The sticks were subjected to a protocol of procedures to prepare them for viewing in a scanning electronic microscope (SEM). Two sticks of each group were immersed in silver nitrate for 24 hours. After, the sticks were washed in running water and submerged for 8 hours in a developing solution. After that, the sticks were polished with the sequence of water sandpaper 800, 1200, 1500, 2000 and 2500. Then, the sticks were subjected to a slow dehydration with silica gel and metallized with gold-carbon coverage on a sputter coater (BAL-TEC MED 020). Areas of adhesive-dentin union were analyzed in a scanning electronic microscope (XL 20 – PHILIPS) for nanoleakage qualitative evaluation.

RESULTS

Microtensile bond strength test (µtbs)

To check the normality of the data, the nonparametric Kolmogorov-Smirnov test was used. To compare the adhesive systems (Adper Single Bond 2 and Adper Prompt L-Pop) and between groups (control and fibers) the Student t test (**Tables 1** and **2**) was used, with a significance level set at 5%.

The results showed a statistically significant difference among groups for both adhesive systems, and the fibers group (modified adhesives) showed the best performance.

The results of **Table 2** showed a statistically significant difference between the adhesive systems only for the control group (original adhesive), being the adhesive system Adper Single Bond 2 with the higher mean than Adper Prompt L-Pop.

Nanoleakage

The specimens of adhesive systems Adper Single Bond 2, Adper Prompt L-Pop and Adper Prompt L-Pop modified by glass microfiber presented nanoleakage along the hybrid layer (**Figure 1**). It is possible to notice the water trees in the base of the hybrid layer. The adhesive system Adper Single Bond 2 modified by glass microfiber did not present nanoleakage in the hybrid layer (**Figure 2**).

DISCUSSION

The results obtained in this study, about a total-etch and a self-etching all-in-one dentin-bonding agents, both modified by addiction of 10% of pre-silanized glass microfibers, showed that the fibers improved the microtensile bond strength when compared to control groups (original adhesives). The main reason to use reinforcements in polymers is to increase rigidity and structural strength of materials. Munchow et al[10] (2020) also revealed a better performance for fiber-modified bonding agents. A higher degree of conversion was found and a stable bond strength after one-year storage, for adhesives with 5 or 25% of fibers.

Bond strength is one of the most important in vitro information regarding to adhesion, analyzed by either tensile or shear bond strength tests[3-5]. Osorio et al.[9] (2016) reported a bond strength in dentin of 25-30 MPa, immediately, keeping the values around 22-24 MPa after 6-months with helping of nanoparticles addiction. Dentin bond strength is required to resist the polymerization shrinkage stresses in the composite resin restorations, avoiding the formation of marginal gaps and microleakage.

Table 2. Comparison between the two adhesive systems Adper Single Bond 2 and Adper Prompt L-Pop.

Group	*n*	*Mean*	*Standard deviation*	*t*	*p*
Control					
Single Bond2	10	11.21	2.69	4.91	0.001
Prompt L-pop	9	6.13	1.62		
Fibers					
Single Bond2	9	18.21	7.31	-0.21	0.837
Prompt L-pop	9	18.95	7.69		

The glass microfibers, when inserted in adhesives Adper Single Bond 2 and Adper Prompt L-Pop, have probably generated a gain of cohesive resistance, which is still a little deficient in these materials. The Adper Single Bond 2 presents around 5% weight of colloidal silica, which is required to produce cohesive resistance. When the microfibers were added, values of particles were increased, 5% of silica and the new 10% of glass microfibers (hybrid fillers). The nanoparticles presented by Osorio et al.[9] (2016) had another aim besides bond strength, zinc-loaded nanoparticles helped the process of remineralization the dentin in the hybrid layer, to avoid long-term degradation.

To turn microfibers chemically compatible with silica and monomers, this study used previously a silane coupling agent to prepare the surface of the glass microfibers for bonding. Silane has the function of being a linker chemical agent to the inorganic filler and organic polymers[15]. Therefore, it seems that there was an acceptable bonding between the glass microfibers, the silane, and the monomers present in the adhesive. However, these results are short-term (immediate), it is necessary to carry on this research, to evaluate long-term results and other methods, especially clinical trials.

An amount of 10% of fiber fillers was employed in the current study. Larger or uncontrolled quantities of fiber could lead to saturation of the adhesive, making it more fragile and with alteration of color, opacity, and performance. Fibers generate an increasing in strength of the material, when added in controlled amounts. Munchow et al.[10] (2020) showed a good performance for adhesives with 5 or 25% of fibers only, while there was more nanoleakage for adhesives with 50% of fibers. Nanotechnology could also help the process of getting the fibers (nanofibers), being an additional variable for future studies[9].

The results of this study are an important step in the optimization of bond strength of adhesive systems. The benefits presented here are applied to either total-etch or self-etching adhesives, regarding microtensile bond strength. Cardoso et al.[16] (1998) showed that the self-etching adhesive had lower values for microshear bond strength, as well as seen in our study for μTBS. Goracci et al.[17] (2004) showed that the self-etching adhesive Prompt-L-Pop presented the lowest values of shear bond strength, when compared to other adhesives.

Nanoleakage is known as the leakage that occurs in the hybrid layer and can be present either between the adhesive layer and dentin or between adhesive and composite resin[3,18]. The total-etch Adper Single Bond 2 adhesive system modified by 10% of glass microfibers could prevent the development of nanoleakage, but the same benefit did not occur with the self-etching Prompt-L-Pop adhesive

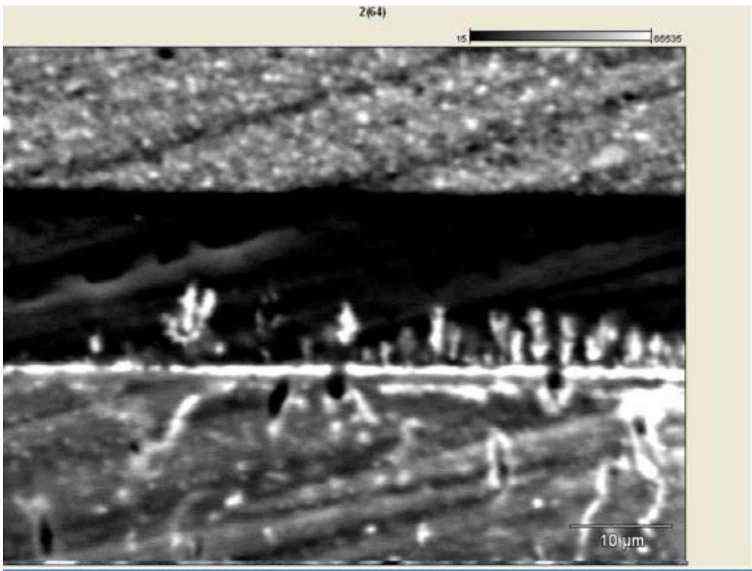

Figure 1. Nanoleakage within the hybrid layer.

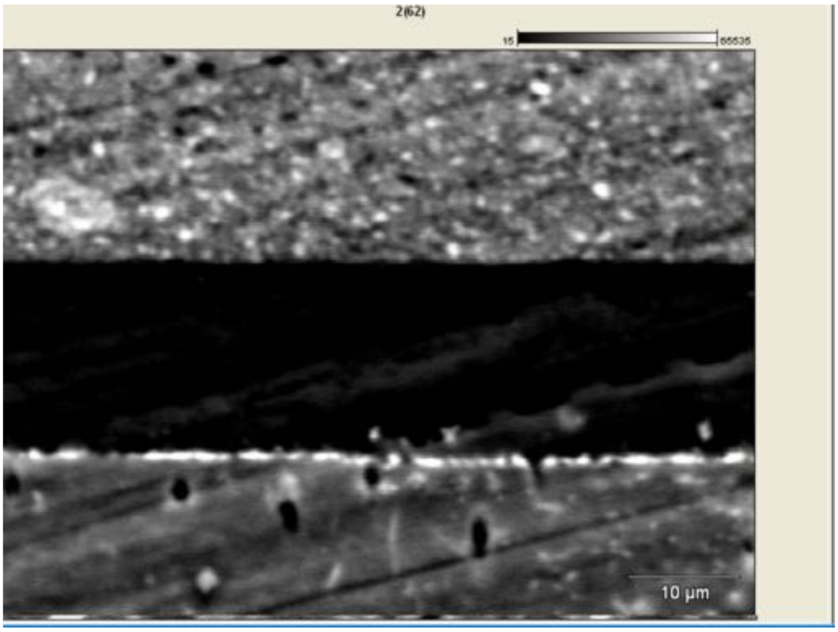

Figure 2. Absence of nanoleakage (Adper Single Bond 2 with microfibers).

system. The absence of nanoleakage in the hybrid layer was probably associated with the reduction of the stress of polymerization, polymerization shrinkage and elastic modulus by the microfiber net[19]. According to Ozel and Soyman[20] (2009), composites with microfiber nets showed significantly lower microleakage and decreasing the stress of polymerization. Munchow et al.[10] (2020) did not show any difference regarding nanoleakage among the modified adhesives.

Considering the limitations of this study, the incorporation of pre-silanized glass microfibers into dentin-bonding agents seems to be a promising fact for restorative dentistry, to optimize the performance of these systems, particularly in relation to the bond strength and quality of the hybrid layer. However, clinical trials must be conducted to confirm these results in a clinical situation.

CONCLUSION

Considering the limitations of this study, it is possible to conclude:

1) The addition of 10% of pre-silanized glass microfiber in the adhesive systems Adper Single Bond 2 and Adper Prompt L-Pop improved the bond strength to dentin.

2) A better quality of the hybrid layer was detected in Adper Single Bond 2 with the addition of 10% of pre-silanized glass microfiber.

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