

## Etching time evaluation on the shear bond strength of two adhesive systems in primary teeth

### Avaliação do tempo de condicionamento ácido na resistência da união de dois sistemas de união em dentes decíduos

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#### ABSTRACT

**Purpose:** to evaluate the effect of the etching time and adhesive systems on the shear bond strength in primary teeth. **Methodology:** 48 sound primary teeth (USP teeth bank) were used. They were longitudinally sectioned (mesio-distal direction) and embedded in epoxy resin, leaving the buccal or lingual surface externally. The specimens were ground flat until dentin. They were randomly divided into 3 groups according to the etching time (7, 15 and 20 s) and the adhesive system used (Scotchbond Multipurpose Plus - SBMP and Prime & Bond 2.1 - PB): G1 - 7s + SBMP; G2 - 15 s + SBMP; G3 - 20 s + SBMP; G4 - 7 s + PB; G5 - 15 s + PB; G6 - 20 s + PB. Next the teeth were restored (Z100), on the dentin surface and stored in distilled water (37°C, for 72h). Then, the specimens were submitted to the SBS test in an Instron machine (0.5 mm/min). The fracture sites were analyzed in a Stereomicroscopic and SEM. The values for the SBS test were submitted to ANOVA and Tukey tests ( $p < 0.05$ ). **Results:** the SBS was higher for the 7s time groups. However, no statistical difference was observed when SB was used. The PB system showed higher values of SBS at 7 and 15s ( $p < 0.05$ ). The adhesive failure (86.5%) was more frequent. **Conclusion:** 1 - The interaction material versus time showed that for the SBMP the performance of the system did not vary due to the etching time, while for the PB the best results were found for the lower times of acid etching; 2 - The analysis of fractured sites showed that the most frequently failure type found was the adhesive type (86.5%); 3 - The larger the etching time the lower the shear bond strength values for the PB adhesive system.

#### UNITERMS

Etching time, acid etching, dental; primary teeth; adhesive systems

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**Relevância clínica:** considerando-se dentes decíduos é possível alcançar bons resultados de resistência da união em menor tempo de condicionamento ácido.

#### INTRODUCTION

The effective adhesion of restorative composite resins to mineralized dental tissues has been the subject of research for the latest 40 years, regarding permanent teeth. Since 1955, when Buonocore<sup>2</sup> achieved significant bond strength between an

acrylic resin and the dental enamel after the acid etching of its surface using 85% phosphoric acid, the restorative dentistry concepts have been under modifications. Thus, the industry began to value the esthetic adhesive materials, which save healthy dental tissue in instead of using metals, which sacrifice sound dental structure.

Due to the success achieved by the use of the enamel etching, the dentin etching has also been studied. The acid etching of the dentin removes the smear layer, which is composed of debris and bacteria, produced by carious tissue removal when sharp instruments are used. This procedure provides disclosing and enlarging of the dental tubules, creating porosities in the inter-tubular area in which the dentin matrix is exposed<sup>7,21</sup>. Studies indicate that the removal or alteration of the smear layer increases the effectiveness in the adhesion between composite resin and the tooth<sup>10</sup>. Gwinnett<sup>5</sup> and Olmez et al.<sup>19</sup>, reported that the excessive removal of the smear layer would lead to a collapse of the collagen zone weakening the bonding between the composite resin and the tooth.

One factor that hinders the adhesion to the dentin is its inherent humidity. In order to minimize the effects of this humidity, adhesive systems with hydrophilic characteristics were developed. Increase in the bond strength values was achieved when dentin was intentionally moistened before the placement of these adhesive systems<sup>4,14</sup>.

The dental products available are indicated for simultaneous use in primary as well as permanent teeth. However, considering the adhesive process, changes in the substrate can determine decrease in the bond strength, increase in the microleakage, and consequently, affecting the restoration longevity<sup>8</sup>.

Primary teeth show peculiar characteristics due to its function in the oral cavity. The life cycle of those teeth is much shorter than their permanent successors. In comparison to the permanent ones, they are less mineralized, and according to some authors, they show different tubular density and permeability<sup>11</sup>.

In association to those characteristics, primary dentin is a dynamic tissue that undergoes alterations in its function with aging and external stimulus. As the substrate is different from the permanent teeth, related to the adhesion, this process should suffer adaptations in order to guarantee the physiological characteristics of the primary teeth<sup>18</sup>.

Etched primary teeth tend to have the dentin surface demineralized faster than permanent ones, and consequently, they can exhibit thicker hybrid layer, which can decrease the bond strength between the bonding agent and the dentin<sup>18</sup>. In addition, organic acids tend to be more efficient in the adhesion to the dentin structure; while the inorganic and more concentrated, acids tend to provide a deep

demineralization, leaving debris on the contact surface that can interfere with the adhesive process<sup>21</sup>.

Considering the different morphological characteristics of the primary teeth when compared to the permanent ones<sup>6,24</sup> and previous reports<sup>11</sup>, such characteristics would influence the action of the adhesive system used<sup>1,9,12,16,17</sup>. The use of adhesive systems should be evaluated regarding the time and type of etching agents<sup>25</sup>.

The aim of this study was to evaluate the shear bond strength of the adhesive system in primary dentin, concerning different etching times.

## MATERIALS AND METHODS

Forty-eight sound primary molars donated by the teeth bank of the São Paulo University were used. The teeth were stored in a 2.5% glutaraldehyde solution, until they were processed. The roots were sectioned at the cement-enamel junction (CEJ) and discarded, and the crowns were longitudinally sectioned mesio-distally in a saw machine (ISOMET 1000 - Buehler UK Ltd). Each tooth resulted into two specimens, which were embedded in epoxy resin, inside plastic cylinders (P.V.C), with 20mm of external diameter and 20mm of height, with the buccal or lingual surface turned externally and projected 2mm above the border of the P.V.C. cylinders. The specimens were randomly divided into three groups according to the etching time (7, 15 or 20 seconds) and the adhesive systems tested, being G1 - 7s + SBMP; G2 - 15s + SBMP; G3 - 20s + SBMP; G4 - 7s + PB; G5 - 15s + PB; G6 - 20 s + PB.

The specimens were positioned individually in the central area of a metallic round base, measuring 20.5mm of internal diameter X 75mm of external diameter, for 29mm in height and 500g weight. The insertion of the sample was made until the superior border of the P.V.C. cylinder was parallel to the surface of the metallic base, with the teeth face projected above the borders, maintained in that position by means of a knob inserted in one of the faces of the metallic base. The specimens were flattened in a horizontal machine (Minimet 1000, Buehler UK Ltd) with a sandpapers sequence from grit 240 to 600, under water cooling, using a metallic support, until next an area of 5mm in diameter was obtained at the dentin surface of all the samples. Next, the surfaces were examined through a Stereomicroscope (Model XLT30 - New

Optical Systems) with 25X magnification in order to verify any enamel spot remained on the surface.

### Adhesive procedures

After the preparation of the dentin surfaces, an adhesive tape (Contact<sup>®</sup>) with a central hole of 3mm in diameter was bonded to the dentin surface, in order to define the adhesion area. The bonding procedure was accomplished following the manufacturers' instructions, except the etching time.

After light curing, a split mold with a central perforation in 3mm of diameter and 5mm in height was positioned on the established area of the samples. Then, the set was taken to a metallic holder to facilitate the insertion of the Z100 composite resin, A2 shade. The composite resin was inserted in 1mm thickness layers, and light cured, with a blue light Elipar TriLight. - (ESPE America Co), for 40 seconds and which the light intensity was measured in a radiometer (470 mW/cm<sup>2</sup>), before each restorative procedure.

Next, the sample was carefully removed from the metallic holder and the split mold was separated using a surgical blade to avoid inducing tensions at the adhesion areas during its removal.

The specimens were stored in distilled water, for 72 hours, at  $37 \pm 1^\circ\text{C}$  and relative humidity of 100%.

The shear bond strength was accomplished in an Instron Testing Machine (model 4411) in a 0.5-mm/min crosshead speed. The specimens were horizontally placed in a metallic glove, with 20.5mm of internal diameter and 20mm height, fastened to the upper holder of the machine of universal testing. In the lower holder the extremities of stainless steel strip (5mm width x 10cm length) were fastened, forming a loop that involved the composite cylinder bonded to the dentin surface.

The shear bond strength data were analyzed by ANOVA and Tukey tests, at the 95% confidence level.

### Analysis of the fractured sites

The samples were examined in a Stereomicroscope at 25X magnification in order to observe the failure sites, and they were classified as cohesive (composite resin or dentin), adhesive or mix failure.

### SEM Analysis of the dentin/resin-bonding interface

The bonding interfaces were observed with SEM in order to illustrate the resin/dentin-bonding interface. Three primary molars were prepared for each group described previously. The teeth were ground flat by occlusal surface until reach the dentin surface. Then, the dentin surfaces were treated similar to those described for the experimental groups and they were longitudinally sectioned, in the mesio-distal direction into three sections. The sections were fixed with 2.5% glutaraldehyde in sodium cacodylate solution (pH=7.2) for 1 hour. After, they were washed in 0.2M sodium cacodylate buffer for 1 hour, which was changed for three times. The sections were immersed in distilled water for one hour and then, dehydrated in a graded series of ethanol (25-100%). The sections were etched with 50% phosphoric acid for three seconds, and ultrasonically washed for 25 minutes. Next, they were immersed in 1% sodium hypochlorite solution for five minutes, and ultrasonically washed for 25 min.

The sections were dried in hexamethyldisilazane (HMDS) for teen minutes, and left untouched for 12 hours in a room temperature. Next, they were placed on aluminum stubs and sputter-coated by gold/palladium for SEM examination.

## RESULTS

### Shear bond strength

The results of the shear bond strength test are displayed in Table 1 and Figures 1-2.

The seven sec etching time showed the highest values of SBS ( $p < 0.05$ ). There was no statistically significant difference between the adhesive systems studied. However, the interaction of adhesive system and etching time was significant and it was observed that the seven sec etching time showed the highest SBS values when PB adhesive system was used.

### Analysis of failure sites

The failure sites showed to all etching times and for both adhesive systems adhesive failure. However, the SBMP showed the higher percentual values of cohesive failure (18%) than PB (6%) as showed at Table 2.

**Table 1 - Shear bond strength values (MPa)**

	SBMP	PB	TOTAL
<b>7s</b>	2.25 a A	3.36 b A	2.81 a
<b>15s</b>	2.79 a A	2.41 a B	2.60 a b
<b>20s</b>	2.08 a A	1.77 a B	1.92 b
<b>Total</b>	2.37 a	2.51 a	

\* Different lower case letters on lines mean values with statistical significant difference (p<0.05).

\*\* Different upper case letters on columns mean values with statistical significant difference (p<0.05).

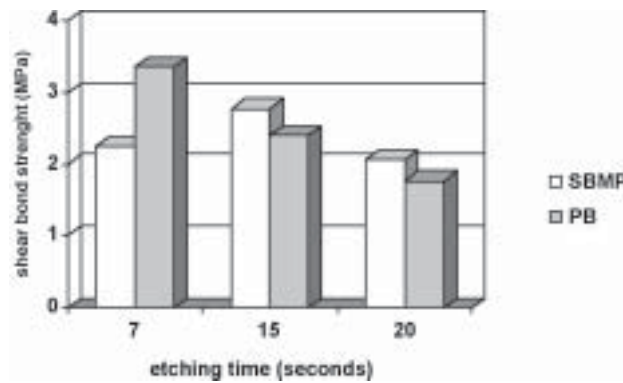


FIGURE 1 – Shear bond strength values (MPa) for the all etching times used in the study.

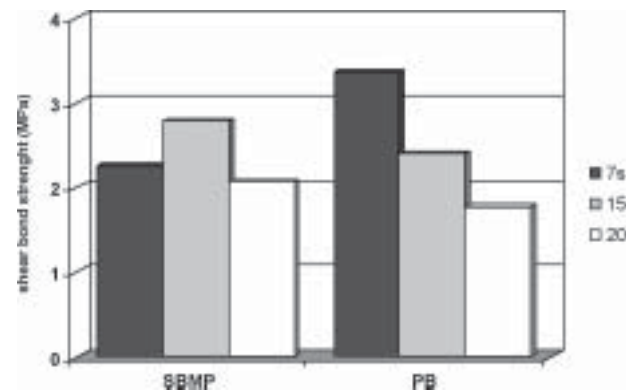


FIGURE 2 - Shear bond strength values (MPa) for all adhesive systems used in the study.

**Table 2 - Type of failures found on the fractured sites for the analyzed samples**

Etching times	PB		SBMP	
	Adhesive	Cohesive	Adhesive	Cohesive
7 s	93.75%	6.25%	81.25%	18.75%
15 s	93.55%	6.45%	81.45%	18.55%
20 s	100%	0%	98.5%	1.5%

## DISCUSSION

In this study significant statistical difference was found in the interaction between the etching time and adhesive systems ( $p=0.02$ ) indicating that the success of the adhesive technique does not depend exclusively on the material employed, but also on its interaction with the substratum. On the other hand, this presents intrinsic characteristics that should be considered in the bonding process. The conditioning of the dentin substrate is an attempt of improving the effectiveness of the adhesive restorative materials when replacing the dental structure lost by the decay or trauma<sup>8</sup>. As the dentin is a dynamic substrate that suffers alterations with time, and from external stimuli, the adhesive process finds some barriers, which hinder the bonding process<sup>26</sup>.

Researches have been accomplished in order to verify the effectiveness of the etching acid in the modification of the substratum. The aim for using etching acids is to enable the penetration of resinous monomers inside the dentin; so resistant structures to bonding can be produced between restorative material and dental structures<sup>5, 15</sup>.

The modifications in the substrate are directly related to the concentration and application time of etching agents, which consequently will influence in the values of adhesion<sup>25</sup> depending on their effectiveness. However, the relationship between bonding strength values and clinical performance of the restoration is not well known, yet.

In permanent teeth, a shorter conditioning time than that for primary ones can lead to insignificant alterations in the structure of the substrate, and therefore to a decrease in the bond strength values. However, in primary teeth, which the amount of minerals seems to be smaller, and dentin tubules are less wide, the etching time may determine more intense alterations in extension that would decrease the bond strength values<sup>17</sup>.

The highest shear bond strength values were obtained for seven seconds (2.81 MPa), however, these values were not statistically different from the values obtained for 15 seconds (2.60 MPa), suggesting that a shorter exposure time of the substratum to the etching acid did not negatively influence the results. The significant interaction between adhesive system and etching time suggests that different adhesive systems may have different reaction according to alterations in the etching time.

The increase in etching time reduced the values of the SBS for the PB, though not interfering in the action of the SBMP system.

It could be observed that the larger the etching time the smaller the shear bond strength values; the results showed 1.92 MPa when 20 seconds of etching time was used.

These findings suggest that excessive demineralization, due to a longer etching time, could form deeply etched zones where bonding agents perhaps were not able to diffuse, and consequently produced weaker bonding areas due to the formation of a demineralized area not filled out by restorative material<sup>3, 8, 13, 16-17, 22</sup>.

It is important to point out that, the values obtained for primary teeth were much smaller than the ones found for permanent ones, as reported in the literature<sup>18</sup>, as well as for the nominal values of mechanical test that has demonstrated discrepancies depending on the methodology used<sup>27</sup>. In this study, the shear bond strength was accomplished using a steel strip whose width was similar to the thickness of the resin cylinder of the specimen. Based on mechanical laws this type of test provides smaller nominal values<sup>28</sup> because the load is distributed on the structure of the specimen, which would cause a sliding of the cylinder in relation to the bonding surface, what is not verified when a plain or round knife was used<sup>23</sup>. The specimen failed before the shear effort using a knife, due to the flexuring moment caused by the steel strip on the surface of the resin cylinder, resulting in smaller forces in the bonding surface, producing a real shear effort in the area<sup>23</sup>.

Considering the adhesive system, in all etching times tested, no statistically significant difference was observed between SBMP and PB, 2.37 MPa and 2.51 MPa, respectively.

However, it was observed that for the etching time seven seconds, PB system showed higher numerical values for shear bond strength (3.36 MPa) than for SBMP system (2.25 MPa). The numerical difference presented should be attributed to the different compositions of the adhesive systems. The PB system, has acetone as solvent, and possesses 37% phosphoric acid as etching agent, while the SBMP system, has the same acid the 35%, however has water as solvent.

These differences concerning the solvent can suggest that after the etching acid for 7s and rinsing of the dentin surface, the PB was more effec-



tive concerning the intrinsic dentin humidity, favoring a larger interlocking of the bond agent with the substratum, resulting in larger values of SBS.

There was not statistically significant difference between the adhesive systems for the etching times (15 and 20s). This corroborates the suggestions given above for the findings in the times of 7s. This way, a longer etching time (15 and 20s), in dentin of primary teeth, would produce larger amount of demineralized dentin. Consequently, after the rinsing of the surface, a greater amount of residual water is found, demonstrating that SBMP exhibited more homogeneous action among the different demineralization levels (Table 1), while PB, due to its composition, seems to be more vulnerable to the amount of residual water, exactly for interacting in a more effective way with the intrinsic dentin humidity.

These findings can be emphasized when the obtained patterns of failures are observed after SBS test (Table 2), because both systems presented adhesive failures. However, the PB presented predominantly a larger frequency of adhesive failures (95.76%) and smaller of cohesive failures (12.9%).

Concerning the performance of the materials studied, the interface material/dentin was observed using photomicrographs and it was found that the amount of demineralization produced by the etching acid of the dentin is related to the amount

of minerals and quality of their distribution on the surface. Thus, possibly a fast demineralization process is developed in primary teeth due to smaller mineralization of its dentin surface, leading to large demineralization zones as demonstrated by some authors who found a thicker hybrid layer<sup>18,19,20</sup>.

It was observed that both adhesive systems and all etching times studied showed the presence of resin tags in all the samples, presenting an interface with uniform adaptation, suggesting an appropriate adhesiveness, in the morphological point of view (Figures 3-8).

Specifically, for the seven seconds of etching time, the same characteristics at the resin/dentin interface were observed for both adhesive systems, suggesting that the reduction of the etching time should be applied in primary teeth (Figures 3 and 6), not interfering in the adaptation of the material on the dentin surface.

However, clinical and *in vitro* microleakage studies should be accomplished to evaluate and to substantiate the *in vitro* results of the bond strength and analysis of the interface, assuring the use of the adhesive process as an efficient restorative procedure, with a different protocol to primary teeth.

It could be concluded that the material versus time interaction of acid etching showed that larger bond strength values can be obtained when PB is used for a short period of time in primary teeth,

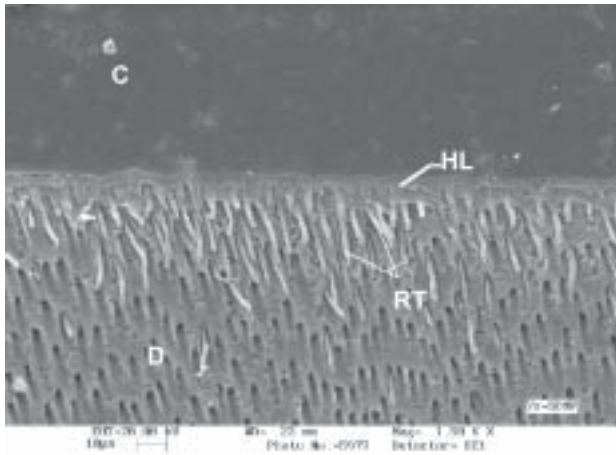


FIGURE 3 – SEM photomicrograph illustrating the resin-dentin interface reached in G1. Note: C (composite resin), HL (hybrid layer), D (dentin) and RT (resin tags).

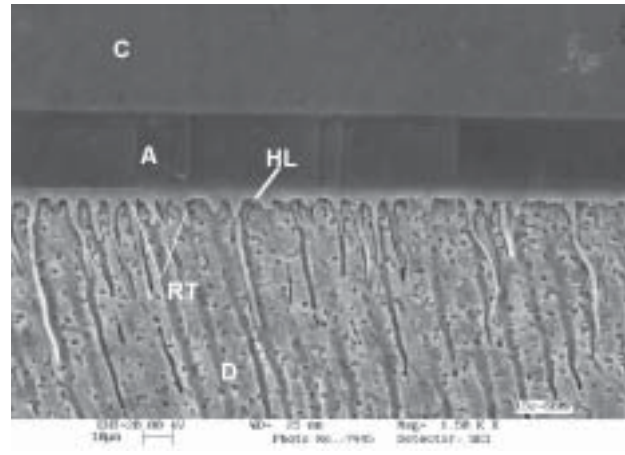


FIGURE 4 – SEM photomicrograph illustrating the resin-dentin interface reached in G2. Note: C (composite resin), A (bond agent), HL (hybrid layer), D (dentin) and RT (resin tags).

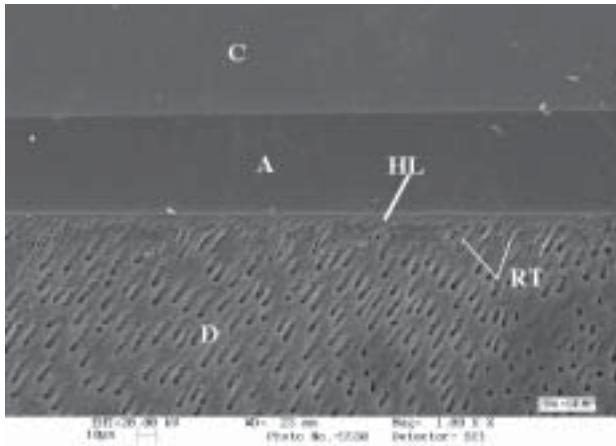


FIGURE 5 – SEM photomicrograph illustrating the resin-dentin interface reached in G3. Note: C (composite resin), A (bond agent), HL (hybrid layer), D (dentin) and RT (resin tags).

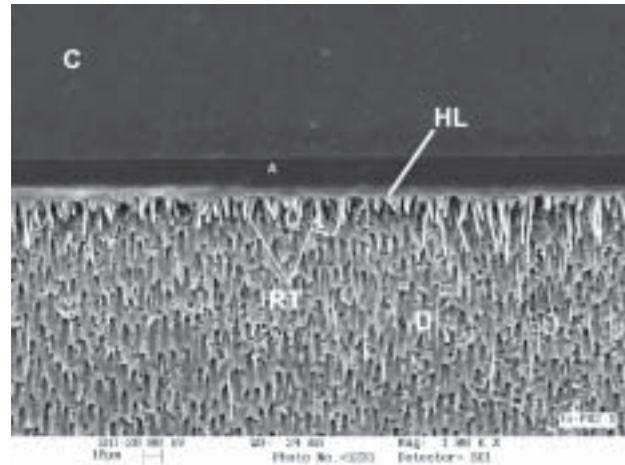


FIGURE 6 – SEM photomicrograph illustrating the resin-dentin interface reached in G4. Note: C (composite resin), A (bond agent), HL (hybrid layer), D (dentin) and RT (resin tags).

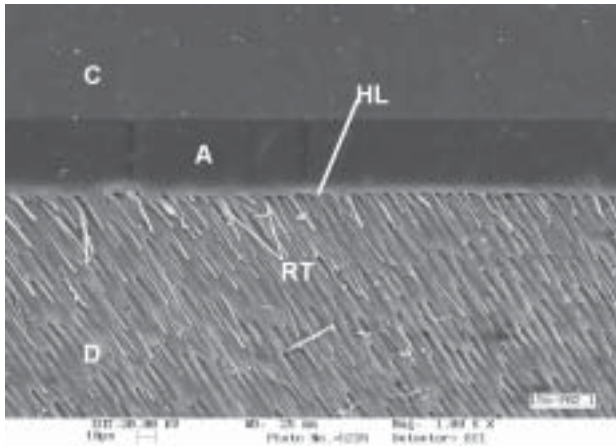


FIGURE 7 – SEM photomicrograph illustrating the resin-dentin interface reached in G5. Note: C (composite resin), A (bond agent), HL (hybrid layer), D (dentin) and RT (resin tags).

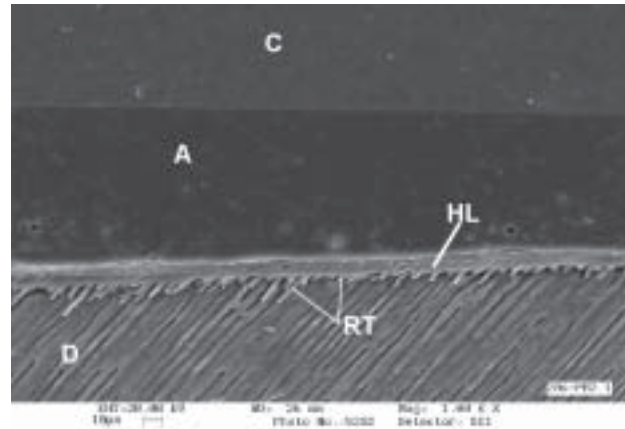


FIGURE 8 – SEM photomicrograph illustrating the resin-dentin interface reached in G6. Note: C (composite resin), A (bond agent), HL (hybrid layer), D (dentin) and RT (resin tags).

although, the clinical implication is unknown whether larger averages of SBS would be beneficial or not to the longevity of the restoration.

Thus, it must be emphasized that the smaller the time of work the smaller the chances of con-

tamination of the dentin and, therefore, the smaller the microleakage levels that would be found, mainly in a child's dental treatment, in which the time of work is a decisive factor for the child behavior control.

## CONCLUSIONS

According to the results, it can be concluded that:

1. The interaction material versus time showed that for the SBMP the performance of the system did not vary due to the etching time, while for the PB the best results were found for the lower times of acid etching;
2. The analysis of fractured sites showed that the most frequently failure type found was the adhesive type (86.5%);

3. The larger the etching time the lower the shear bond strength values for the PB adhesive system.

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## RESUMO

**Objetivo:** avaliar o efeito do tempo de condicionamento ácido e sistemas de união na resistência ao cisalhamento (RUC) em dentes decíduos. **Metodologia:** 48 molares decíduos, hígidos, doados pelo Banco de dentes da USP, foram seccionados longitudinalmente (mésio-distal) e embutidos em resina epóxica, deixando as superfícies V ou L expostas. As amostras foram lixadas até a obtenção de uma superfície plana em dentina e distribuídas em 3 grupos de acordo com o tempo de condicionamento ácido (7, 15 ou 20 s) e sistemas de união (Scotchbond Multipurpose Plus -SBMP e Prime & Bond 2.1-PB): G1 - 7 s + SBMP; G2 - 15 s + SBMP; G3 - 20 s + SBMP; G4 - 7 s + PB; G5 - 15 s + PB; G6 - 20 s + PB. Confeccionou-se restaurações com compósito Z100, sendo armazenados em água destilada a 37°C, por 72h. Os corpos-de-prova foram submetidos ao ensaio de RUC (Instron - 0,5 mm/min). Os sítios de fratura foram analisados em Microscópio Estereoscópico e MEV e os resultados submetidos à análise estatística ANOVA e Teste de Tukey ( $p < 0,05$ ). **Resultados:** os maiores valores de RUC foram obtidos por G1 e G4. Não houve diferença estatística entre G1 e G2, enquanto o G4 apresentou maiores valores em relação aos G1 e G2 ( $p < 0,05$ ). A falha adesiva foi a mais freqüente (86,5%). **Conclusões:** 1 – A interação material\*tempo de condicionamento demonstrou que para o SBMP o desempenho do sistema não diferiu em relação do tempo de condicionamento ácido, enquanto que para o PB os melhores resultados foram observados para os menores tempos de condicionamento ácido; 2 – A análise dos sítios de fratura demonstrou que a falha mais freqüentemente observada foi a do tipo adesiva (86,5%); 3 – Quando maior o tempo de condicionamento ácido, menor os valores de resistência da união para o sistema adesivo PB.

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Ataque ácido dentário, condicionamento; condicionamento ácido; dente decíduo, sistemas de união

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