# Effect of Sodium Hypochlorite on Dentin Bonding in Primary Teeth

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**Purpose:** The aim of this study was to evaluate the effect of sodium hypochlorite (NaOCI) on the shear bond strength (SBS) using three bonding systems in primary dentin.

**Materials and Methods:** Forty-five sound extracted primary molars were selected. The crowns were longitudinally sectioned, embedded in polystyrene resin, and flattened until a dentin surface was reached. The samples were assigned to 6 groups (n = 15): G1, Single Bond (SB); G2, NaOCI + SB; G3, Prime & Bond 2.1 (PB); G4, NaOCI + PB; G5, Clearfil SE Bond (CSE); G6, NaOCI + CSE. All the adhesive systems were applied according to the manufacturers' instructions, except for the application of 10% NaOCI solution for 60 s in groups 2, 4 (after acid etching), and 6 (before applying adhesive system). The composite resin was placed in increments in a mold and light cured for 20 s. The samples were stored in distilled water at 37°C for 24 h and submitted to SBS testing with a crosshead speed of 0.5 mm/min. The failure sites were observed with SEM. The data were treated with ANOVA and Tukey's tests (p < 0.05).

**Results:** There was no statistically significant difference between the groups with or without treatment of the substrate with NaOCI, regardless the material used. The SBS averages in MPa ( $\pm$  SD) were: G1: 15.8(1.9)<sup>a</sup>; G2: 14.6(1.3)<sup>a</sup>; G3: 10.2(0.7)<sup>a</sup>; G4: 9.9(0.2)<sup>a</sup>; G5: 13.3(1.2)<sup>a</sup>; and G6: 10.7(1.0)<sup>a</sup>. There was a statistically significant difference between the materials (SB  $\geq$  CSE  $\geq$  PB). Mixed failure was the failure type most frequently observed for all groups.

Conclusion: Dentin surface treatment with NaOCI did not affect the resin-dentin bonding strength in primary teeth.

Key words: sodium hypochlorite, primary teeth, dentin bonding.

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The concept of dentin adhesion is based on micromechanical retention: monomers impregnate the exposed collagen network of demineralized superficial dentin, and upon polymerization, result in the formation of a hybrid layer.<sup>14,17,21,22,28</sup> For some authors, the for-

**Reprint requests:** Gisele Maria Correr, Piracicaba Dental School, UNICAMP, Av. Limeira, 901, 13414-0903, Piracicaba, SP, Brazil. Tel: +55-19-3412-5286, Fax: +55-19-3412-5218. e-mail: giselefop@yahoo.com mation of a hybrid layer is an essential condition for improving the bond strength between composite resin and dentin.<sup>5,6,10,21,23,26,30,33,42</sup> Although the hybrid layer has been described being responsible for a restoration's longevity, some studies<sup>27,37</sup> have shown leakage at the hybrid layer. This phenomenon might be due to hydrolytic degradation of the exposed collagen network. This has been related to the poor infiltration of monomers into the collagen remaining after dentin acid etching, creating a weak zone. This zone is vulnerable to degradation after long-term exposure to water.<sup>21,27,37</sup>

Dissolution of collagen fibrils with a deproteinizing solution after acid etching may result in better monomer diffusion by increasing dentin permeability and changing its composition.<sup>43</sup> Pioch et al<sup>33</sup> demonstrated that the use of NaOCI decreased leakage levels, although it does not improve the adhesion of bonding systems to NaOCI-treated dentin surfaces.

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Adhesive System	Manufacturer and Batch #	Components
Single Bond	3M Dental Products, St Paul, MN, USA Batch: 1105/7BB	Bis-GMA, HEMA, dimethacrylates, water, ethanol, polyalkenoic acid, acid copolymer, photoinitiator
Prime & Bond 2.1	Dentsply Indústria e Comércio Ltda. Petrópolis – RJ Brasil Batch: 64030	PENTA, UDMA, R5-62-1 resin, BPDM, butylated hydroxytoluene, 4-ethyl dimethyl aminobenzoate, cetylamine hydrofluoride, acetone, photoinitiator
Clearfil SE Bond	Kuraray America, New York, NY, USA Batch: 51207 #1975-KA	Primer: MDP, HEMA, hydrophilic dimethacrylates, camphorquinone, N, N-Diethanol-p-toluidine, water Bond: MDP, HEMA, bis-GMA, hydrophobic dimethacrylates, camphorquinone, N, N-Diethanol-p-toluidine, silanated colloidal silica

Table 1 Characteristics and main components of the adhesive systems used

Because of their nonspecific deproteinizing and disinfecting action,<sup>19</sup> sodium hypochlorite solutions (NaOCI) are widely used in various dental procedures, such as chemomechanical treatment in endodontic preparation, chemomechanical removal of carious lesions in dentin (eg, Carisolv),<sup>13</sup> and in dentin adhesion procedures.<sup>3,9,14,17,20,28,29,32,34,36,39,42.44</sup> Depending on the adhesive system used, dentin surface treatment with sodium hypochlorite can increase,<sup>3,14,20,32,34,36,39,43,44</sup> decrease,<sup>6,28</sup> or not interfere in<sup>17,35,42</sup> the bond strength between composite resin and dentin.

All of the studies mentioned above were performed on permanent teeth. However, little attention has been paid to the bonding mechanism in primary teeth. Due to the morphological and constitutional differences of primary vs permanent teeth, their bonding behavior differs. The dentin of primary teeth is more susceptible to acid etching,<sup>23-25</sup> and it is likely that the application of NaOCI solution to that substrate would produce different alterations than in permanent teeth.

The aim of this study was to evaluate the effect of 10% sodium hypochlorite solution applied to primary-tooth dentin for 60 s on the shear bond strength and interface morphology using three different bonding systems.

# **MATERIALS AND METHODS**

# **Specimen Preparation**

Forty-five recently extracted, sound human primary molars were selected, cleaned, and stored in a 0.5% chloramine T solution for up 2 months after extraction. The roots of the teeth were sectioned off 1 mm under the cementoenamel junction, and the crowns were sectioned in the mesio-distal direction using a double-face diamond saw (KG Sorensen, São Paulo, SP, Brazil). Then they were mounted on a 1.9-cm-diameter PVC ring, parallel to the base of the ring. The rings were filled with self-curing polystyrene resin and the embedded specimens were ground on a water-cooled mechanical polisher (Minimet 1000, Buehler, Lake Bluff, IL, USA) using 320-, 400-, and 600-grit silicon carbide abrasive paper (Carbimet Disc Set, #305178180, Buehler) to expose a flat dentin area of 3 mm in diameter on the lingual, buccal, or palatal surfaces.

## **Bonding Procedures**

The specimens were randomly assigned to 6 groups (n = 15). Before the surface treatment, the dentin surface was covered with a piece of adhesive tape with a 3-mm-diameter hole.

The adhesive systems used in this study, batch numbers, and components are described in Table 1. Composite resin Filtek Z-250 (3M Dental Products, St Paul, MN, USA) was used to complete the bonding procedure.

In all groups, the dentin was treated with the adhesive systems according to the manufacturers' directions. Additionally, 10% sodium hypochlorite solution (NaOCI) was applied in groups 2, 4 (after acid etching), and 6 (before application of the adhesive system) for 60 s. The groups received the following treatments:

- Group 1 (SB) Single Bond. The dentin surface was etched using 35% phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) gel for 15 s, rinsed with water for 10 s, and blot dried leaving a moist surface. Single Bond adhesive was applied and light cured for 10 s according to the manufacturer's instructions.
- Group 2 (SB/NaOCI) Single Bond + NaOCI. The same procedures as in Group 1 were followed, except that 10% sodium hypochlorite solution was applied for 60 s after acid etching, then rinsed for 30 s, and blot dried before the application of the adhesive.
- Group 3 (PB) Prime & Bond 2.1. The dentin surface was etched using a 37% phosphoric acid gel for 15 s, rinsed with water for 15 s, and blot dried leaving a moist surface. Prime & Bond 2.1 was applied and light cured for 10 s according to the manufacturer's instructions.
- Group 4 (PB/NaOCI) Prime & Bond 2.1 + NaOCI. The same procedures as in Group 3 were followed, except that 10% sodium hypochlorite solution was applied for 60 s after acid etching, then rinsed for 30 s, and blot dried before the application of the adhesive.
- Group 5 (CSE) Clearfil SE Bond. The dentin was treated with the self-etching Clearfil SE Bond system according to the manufacturer's instructions.
- Group 6 (NaOCI/CSE) NaOCI + Clearfil SE Bond. The same procedures as in Group 5 were followed, except that 10% sodium hypochlorite solution was applied for

60 s, then rinsed for 30 s, and dried before the application of the self-etching adhesive system.

A bipartite silicon ring mold (3 mm in diameter and 5 mm high) was then positioned over the treated dentin. The mold was filled with Filtek Z-250 (3M), shade A3, in two increments and light cured for 20 s per increment with an Elipar tri-light unit (ESPE, Seefeld, Germany). Light intensity was periodically measured in the unit, and it was found to range from 580 to 720 mW/cm<sup>2</sup>. The specimens were immersed in distilled water and stored for 24 h at  $37^{\circ}$ C.

## **Bond Strength Test**

Each specimen was submitted to the shear bond test in a universal testing machine (Instron model 4411, Canton, MA, USA). A stainless steel tape (5 mm in width and 10 cm in length) was placed around the composite cylinder in close contact with the dentin surface (Fig 1), and the specimens were loaded to failure at a crosshead speed of 0.5 mm/min.<sup>38</sup> Means and standard deviation were calculated with units expressed in MPa.

## **Statistical Analysis**

The data were submitted to ANOVA. The multiple comparison Tukey's test (p < 0.05) was chosen to examine significant differences of possible interactions (adhesive system vs treatment).

## **Failure Mode Evaluation**

All the specimens were observed with a stereomicroscope (Model XLT30, Nova Optical Systems, Novo Tempo Co. e Participações, Piracicaba, SP, Brazil) at 25X magnification to classify the failure sites as cohesive (in the dentin or in the composite), adhesive, or mixed failure. Three representative samples of each group were selected and observed with SEM (JEOL, JSM 5600LV, Tokyo, Japan).

#### SEM Evaluation of the Resin/Dentin Interface

To examine the effects on the dentin/composite interface from treatments employed in this study, 9 primary molars were selected. The crowns were sectioned in the mesiodistal direction, and the buccal, lingual, or palatal surfaces were ground until a flat dentin area was reached. After that, the same bonding procedures as in the 6 groups above were performed in all specimens. Next, the teeth were restored with composite resin Filtek Z-250, shade A3, in increments that were each light cured for 20 s. Each restoration was sectioned perpendicular to the bonded interface using an Isomet 1000 machine (Buehler). To prepare the specimens for SEM observation, the sectioned segments were wet abraded using a sequence of 400-, 600-, and 1200-grit silicon carbide abrasive paper (Carbimet Disc Set, #305178180, Buehler) on a flat surface. Then they were polished with a 1-µm and 0.5-µm diamond paste. The specimens were ultrasonically cleaned in distilled water for 30 min, followed by rinsing. The flat, polished surfaces were then fixed, demineral-



**Fig 1** Shear bond strength testing device. A: specimen; B: composite cylinder; C: stainless steel tape; D: metallic specimen holder.

ized, deproteinized, and dehydrated according to the method described by Perdigão et al.  $^{\rm 31}$ 

All specimens were sputter coated (SCD050 sputter coater, Balzers, Liechtenstein) and examined by SEM (JSM 5600LV, JEOL, Tokyo, Japan).

# RESULTS

The ANOVA test did not detect a statistically significant interaction between adhesive systems and treatment with or without 10% NaOCI treatment. Significant differences in SBS were found between adhesive systems: Single Bond showed significantly higher SBS values than did Prime & Bond 2.1, although neither system (SB or PB) was statistically different from the Clearfil SE Bond system, which yielded intermediate SBS values. The statistically significant differences are expressed in Table 2 by the vertical bars connecting the means. No statistically significant differences in SBS were found for a given adhesive system applied with or without 10% NaOCI (Table 2).

The percentage of each failure mode for all groups is presented in Fig 2. The mixed failure mode (cohesive in the composite resin and cohesive in the hybrid layer or in the adhesive) was the one most frequently observed in all the groups (Fig 3). No sample exhibited cohesive failure in dentin.

# DISCUSSION

Some studies have pointed out that the demineralized collagen zone (DCZ) may not directly contribute to bond strength.<sup>9,17,35,42</sup> The results of the present study corroborate this statement. It was verified that substrate treatment with a 10% NaOCI solution for 60 s did not significantly affect the resin-dentin shear bond strength in primary teeth.

## Correr et al

 Table 2
 Shear bond strength mean values (standard deviations) in MPa

Material	Treatment of the substrate		
	without NaOCI	with NaOCI	
SB	15.86 (1.97) a	14.66 (1.36) a	
CSE	13.36 (1.22) a	10.70 (1.10) a	
РВ	10.21 (0.27) a	9.90 (0.74) a	

Vertical bars in columns connect means which are not significantly different. Same letter in rows indicates no significant difference (p < 0.05).



Fig 2 Percentage of failure type found after the shear bond strength test.



**Fig 3** Top: Failure pattern found for Group 4 (PB + NaOCI). The figure shows apparently free material dentin (a); composite (b) and bonding agent (c) remained on the surface, representing a mixed failure. Bottom left: Mixed failure mode, showing regions with incompletely infiltrated tubules (a), and tubules completely infiltrated by the bonding agent (b). Bottom right: Mixed failure mode, showing a region where dentin tubules were completely infiltrated by the adhesive (a), and bonding agent remains on the surface (b).

These findings may be related to the particular characteristics of the substrate used in this study, as the largest amount of organic matter (type I collagen) and lower mineral content are found in primary compared to permanent teeth.<sup>1</sup> Perhaps the concentration and application time of the NaOCI solution used in this study were not sufficient to completely remove the demineralized collagen zone. Alterations in the substrate due to dissolved and destabilized residual collagen fibrils could cause changes in the energy of surface<sup>28</sup> and in the redox potential of the substrate,<sup>18</sup> and may have inhibited increased SBS values in this study. According to Daumer et al,<sup>4</sup> sodium hypochlorite disrupts the pyridinoline cross links that occur in collagen type I and II, with the formation of chloramines



**Fig 4** Photomicrograph of resin/dentin interface (2000X) found for Group 1(SB). C: resin composite; A: adhesive; HL: hybrid layer; T: resin tags.

and protein-derived radical intermediates.<sup>12</sup> These reactive residual free radicals in deproteinized dentin may compete with the propagating vinyl free radicals generated during light activation of the adhesive, resulting in premature chain termination and incomplete polymerization.<sup>18</sup>

The adhesive systems used in this study produced significantly different results. The PB system (acetone based) yielded lower SBS values than the groups that used the SB system (ethanol/water based), regardless of the substrate treatment, corroborating the results of Perdigao et al,<sup>28</sup> Prati et al,<sup>34</sup> and Vargas et al.<sup>43</sup> The difference between the materials' performances can be attributed to different compositions (solvent and monomer content),<sup>15,16,40</sup> the composition and depth of dentin,<sup>7,26</sup> and the relationship between the substrate wetness and adhesive system.

Overwetting or overdrying the dentin may have influenced our results. Considering that acid etching more rapidly removes minerals from the dentin in primary than in permanent teeth, 23-25 the demineralized dentin layer in this study may have been thicker, perhaps leaving more water in the dentin tubules. Thus, more layers of PB (acetone-based) may have been necessary to completely penetrate the demineralized and deproteinized dentin, and completely remove the residual water. The drying method employed here may, however, have favored overdrying of the demineralized dentin. Jacobsen and Söderholm<sup>15</sup> observed that the acetone-based system (PB) was more affected by the drying process of the substrate than the water-based systems. Therefore, the lowest SBS values found for PB – could be related to blot drying producing slightly overdry dentin. In such a case, Single Bond would cause reswelling, while PB would make the surface even drier. In addition, Tay et al<sup>40</sup> demonstrated that these conditions could create weak zones due to the poor diffusion and conversion degree of the monomers. The highest SBS values, found for SB, may also be related to the presence



**Fig 5** Photomicrograph of resin/dentin interface (2000X) found for Group 2 (SB + NaOCI). C: resin composite; A: adhesive; T: resin tags.

of polyalkenoic acid copolymers causing a chemical reaction between the adhesive and the dentin.

The self-etching adhesive system (CSE) presented intermediate SBS values that were not significantly different from SB or PB, corroborating the results of other studies.<sup>2,8,11</sup> It is supposed that this system prevents the collapse of collagen fibrils, avoiding the exposition of the demineralized collagen zone not protected by the hydroxyapatite crystals.

Regarding the failure mode evaluation, mixed failure (cohesive in the composite resin and cohesive in the hybrid layer or in the adhesive) was the most frequently observed mode in all groups. There were minimal adhesive failures in all groups, and no cohesive failure in dentin was observed. Differences among the groups could be related to a variation in resin infiltration and resin tag integrity along with the fractured hybrid layer. For PB and CSE groups, which showed the lowest SBS values, more adhesive failures and regions with incompletely infiltrated collagen were observed.

There was no significant interaction of adhesive system and substrate, which agrees with the results of other studies.<sup>17,35,42</sup> The application of 10% NaOCI solution prior to the CSE system (self-etching) did not influence the SBS values, confirming the results of Marshall et al,<sup>19</sup> who observed no effects of NaOCI applied on the smear layer.

Although the 10% NaOCI did not affect the SBS values, the analysis of the dentin/adhesive interface of deproteinized samples showed an absence of hybrid layer, regardless the adhesive system used, as has also been demonstrated by other studies.<sup>5,14,34,43</sup> Figure 4 shows a hybrid layer at the dentin/adhesive interface in group 1 (SB), and Fig 5 depicts the dentin/adhesive interface for group 2 (SB + NaOCI) with no hybrid layer.

Examining the SBS values and the morphology of the resin/dentin interface, the results of this study indicate that the bonding mechanism does not solely depend on

the presence or absence of the hybrid layer. It may additionally depend on the roughness of the surface,<sup>41</sup> the adhesive infiltration into the treated dentin, the presence of hydroxyapatite crystals in the substrate, and – importantly – on the substrate's degree of wetness.<sup>32</sup>

# CONCLUSION

Because the results of this study showed no significant effect of NaOCI treatment of primary dentin on shear bond strength, the use of NaOCI solution – which represents an extra treatment step – should currently be discouraged in pediatric dentistry, where treatment time is often a critical factor for patients. Other studies should be conducted to observe whether NaOCI treatment affects the marginal adaptation and microleakage of restorations in primary teeth.

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**Clinical relevance:** The dentin bond strength in primary teeth does not seem to be affected by the use of sodium hypochlorite during the bonding procedures.