

## Bond Strength and Interfacial Morphology of Different Dentin Adhesives in Primary Teeth

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### Abstract

**Objective:** To evaluate the interfacial morphology and the bond strength produced by the three- step, two- step and single-step bonding systems in primary teeth.

**Materials and Methods:** Occlusal surfaces of 72 extracted human deciduous teeth were ground to expose the dentin. The teeth were divided into four groups: (a) Scotchbond Multipurpose (3M, ESPE), (b) Adh Se (Vivadent), (d) OptiBond All-in-One (Kerr) and (e)Futurabond NR (VOCO, Cuxhaven, Germany). The adhesives were applied to each group following the manufacturer's instructions. Then, teeth from each group were divided into two groups: (A) For viewing interfacial morphology (32 teeth), with 8 teeth in each group, and (B) For measurement of bond strength (40 teeth), with 10 teeth in each group. All the samples were prepared for viewing under SEM. The statistical analysis was done using SPSS version 15.0 software.

**Results:** Observational measurement of tag length in different adhesives revealed that Scotchbond had the most widely spread values with a range from 12.20 to 89.10µm while OptiBond AIO had the narrowest range (0 to 22.50). The bond strength of Scotchbond Multipurpose was significantly higher ( $7.4744 \pm 1.88763$ ) ( $p < 0.001$ ) as compared to Futurabond NR ( $3.8070 \pm 1.61345$ ), Adhe SE ( $4.4478 \pm 1.3820$ ) and OptiBond-all-in-one ( $4.4856 \pm 1.07925$ ).

**Conclusion:** The three-step bonding system showed better results as compared to simplified studied bonding systems

**Key words:** Bond strength; SEM, Bonding systems; Primary teeth

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### INTRODUCTION

The concept ins in restorative dentistry have been continually changing over the last decades and adhesive dentistry has steadily gained in importance. The concept of adhesive restoration has been essentially the most noteworthy development n this ever progress-

ing science [1]. There are two different ways by which current adhesive systems obtain acceptable micromechanical retention between resin and dentin. The first method is based on complete removal of the smear layer and demineralization of subsurface intact dentin using acid-etching with mineral or organic acids,

leaving a collagen rich, moist surface into which resin must diffuse to form a hybrid layer, called the “etch and rinse approach” [2, 3]. The second method uses slightly acidic monomers, which partially demineralize the smear layer and underlying intact dentin, incorporating the demineralized smear layer remnants and using them as bonding substrate, called the “self etch approach” [3, 4]. There has been a trend to move from the original type of multicomponent bonding systems toward simplified, consolidated adhesive systems that are more user- friendly [5]. In an effort to search for an effective dentinal bonding agent, a large number of bonding systems have been developed that provide a high clinical retention rate of the restorative materials [6]. The bonding efficacy of adhesive systems has been shown to be different for primary and permanent dentition. Studies have shown bond strength and sealing ability in primary teeth to be less than in permanent teeth [7,8]. Lower bond strength in primary teeth may be attributed to chemical, physiological and micromorphological differences of primary teeth such as decreased mineralization, small tooth size and less number of dentinal tubules with decreased permeability or more reactivity to acidic conditioner [9, 10, 11]. Moreover, several studies showed that the peritubular dentin was dematerialized rapidly during acid treatment in primary teeth and the hybrid layer was thicker for primary than permanent dentin; thus, decreasing the available bonding [12]. This study was done to evaluate the interfacial morphology and bond strength produced by the three-step, two-step and single-step bonding systems when applied to dentin of primary teeth.

## MATERIALS AND METHODS

Seventy-two caries-free, unrestored, extracted, primary molars were collected for the study. The teeth were stored in distilled water. The dentin of occlusal surfaces of all teeth was exposed using a hand piece and straight diamond

fissure burs (Shofu Inc, Kyoto, Japan) with water and air spray and then abraded using 600 grit abrasive papers [13].

The bonding agents included in the study were:

Group I: Scotchbond Multipurpose (3M, ESPE, St. Paul, USA)

Group II: Adh Se (Vivadent), Ontario, Canada)

Group III: Futurabond (VOCO, Cuxhaven, Germany)

Group IV: OptiBond all- in-one (Kerr, Schweiz, Germany)

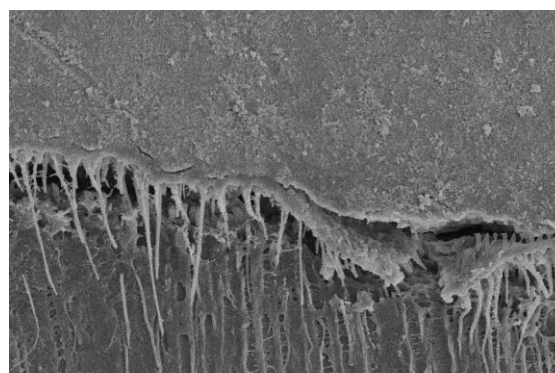
All the bonding agents were applied as per instructions given by the manufacturer; following which the teeth were divided into two groups:

### *Interfacial morphology assessment group:*

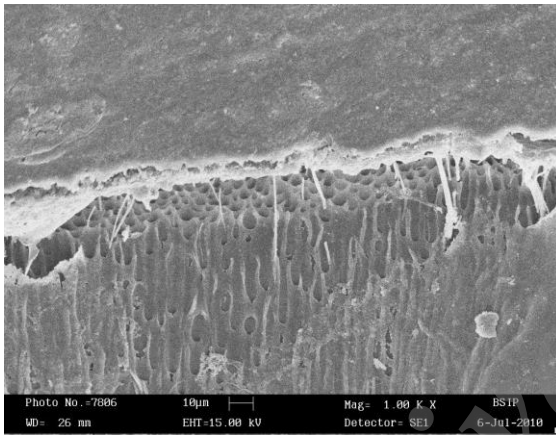
Thirty-two caries-free primary teeth were acquired and the occlusal surface of each tooth was ground to expose the dentin, following which bonding agents were applied; then blocks of composite resin (Esthet X HD, Dentsply) were built using custom-made hollow split molds. The sample teeth were divided into two equal halves vertically using a diamond disc. The sectioning was done under running water. The sectioned halves were then embedded into self-cure resin, keeping the resin-dentin interface exposed (for examination). The samples were sequentially polished with 600 and 1200 grit abrasive papers and Sof-lex finishing and polishing systems.

### *Preparation of samples:*

All the samples were immersed in 4% NaOCl (for deproteinization) for 20 min, and then in 20% hydrochloric acid (for demineralization) for 30sec. The specimens were rinsed with distilled water. All the samples were then sequentially dehydrated in ascending grades of ethanol i.e. 60%,70%, 80% and 90% alcohol for 20 min each and in 100% alcohol for 1 hr [14].

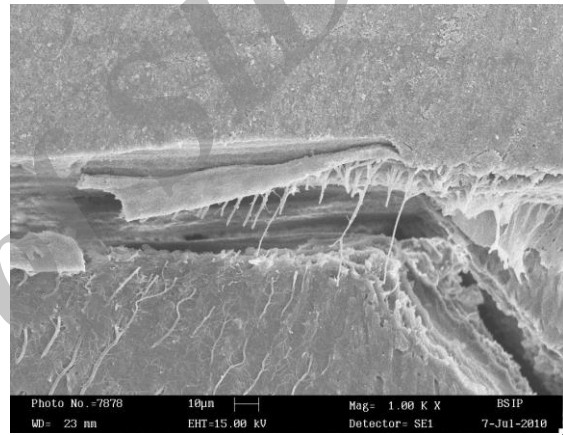


**Fig 1.** SEM photograph illustrating the interfacial morphology after treatment with Scotchbond (1000x)



**Fig 3.** SEM photograph illustrating the interfacial morphology after treatment with OptiBond all-in-one (1000x)

**Fig 2.** SEM photograph illustrating the interfacial morphology after treatment with Scotchbond (1000x)



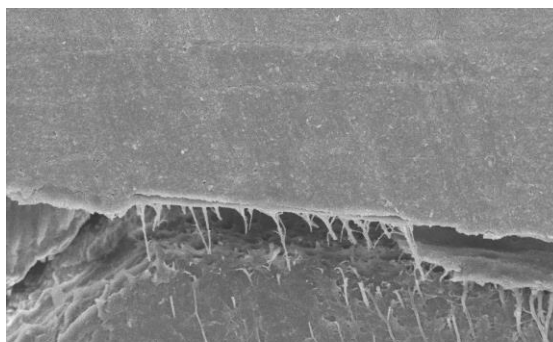
**Fig 4.** SEM photograph illustrating the interfacial morphology after treatment with Futurabond (1000x)

### **Sample preparation for scanning electron microscope (SEM) viewing:**

All samples were dried and mounted on aluminum stubs that were then placed in a vacuum chamber, sputter-coated with gold layer and observed under a SEM (LEO 430, Philips, England). The dentin-resin interface was observed under SEM. (LEO 430, Philips, England) at a magnification of 1000x and a series of photographs were taken.

### **Shear bond strength group:**

Forty caries-free primary teeth were acquired and mounted into self-curing acrylic resin.



The prepared samples were then randomly divided into four subgroups, according to the bonding system to be applied; specimens of each group were stored separately to prevent mixing between the groups. A custom made hollow split mold with an internal diameter of 2mm and height of 5mm was held on adhesive treated surface of the specimens and then composite resin (Esthet X HD, Dentsply, New York, USA) was placed inside the mold, condensed and light-cured (Gnatus, Brazil) for 40 sec. Following curing, the metal mold was split and removed. All the specimens were immersed in water for 24 hours.

Then tensile bond strength was measured using an Instron universal testing machine (Instron 5566, USA) at a crosshead speed of 0.5 mm/min.

All data were subjected to statistical analysis using SPSS (Statistical Package for Social Sciences) Version 15.0 statistical analysis software and the Kruskal-Wallis test and Dunn's test.

## RESULTS

### Interfacial morphology

Photographs of the resin/dentin interfaces were analyzed. The measurements were done on the photographs by means of a standard Vernier Caliper (Tresna, China) using the measurement scale given on the photograph and the following findings were revealed:

**Group I (Scotchbond Multipurpose)** had a thick hybridized complex and very long tags (12.20 to 89.10  $\mu\text{m}$ ); few small side branches were also seen in a large number of specimens.

There was good contact between the resin tags and the hybrid layer and the resin tags were conical. Conical shapes of the upper part of the tags ensured a good seal as the hybrid layer extended into the walls of the dentinal tubules, leading to hybridization of the walls (Fig 1).

**Group II (Adh SE)** had an irregular hybridized complex and thin numerous tubules were empty. Length of the tags varied between 0 to 23.30 $\mu\text{m}$  (Fig 2).

**Group III (OptiBond all-in-one)** showed a thin irregular hybridized complex and at some points of the interface the complex was absent. The tags' lengths varied between 0 to 22.50 $\mu\text{m}$ , broken in some places with numerous empty tubules (Fig 3).

**Group IV (Futurabond NR)** showed a thin but continuous hybridized complex with tag lengths that varied between 0 to 35.80 $\mu\text{m}$  (Fig 4). Evaluations were also done on two photomicrographs by randomly assessing the tag-length at five different locations.

Thus, for each specimen a total of 10 observations were made. The values of tag length showed extensive variability. Observations of tag length in different adhesives revealed that Scotchbond had the most widely spread values with a range from 12.20 to 89.10 $\mu\text{m}$  while OptiBond AIO had the most narrow range (0 to 22.50, Table 1); the intergroup differences were found to be statistically significant ( $P < 0.001$ ).

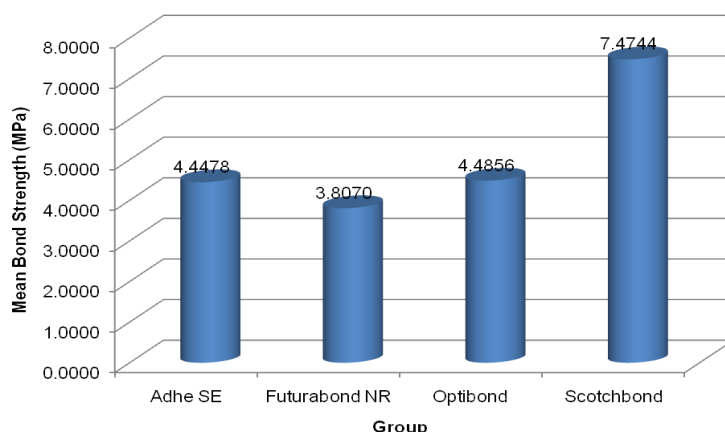
Table 2 shows that Scotchbond had significantly better results as compared to other three groups. Table 3 and Graph 1 show the mean bond strength of various groups. The bond strength of Scotchbond was significantly higher than the remaining three groups. The difference between the other three groups was not significant (Table 4).

## DISCUSSION

Better restorative materials promise better preservation of tooth structure.

**Table 1.** Dispersion of tag length in different adhesives

	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
			Lower	Upper		
Scotchbond	19.98	2.23	32.20	41.09	12.50	89.10
Adhe SE	5.99	0.67	3.69	6.35	0.00	23.30
OptiBond AIO	5.64	0.63	4.00	6.51	0.00	22.50
Futurabond	7.66	0.86	9.17	12.58	0.00	35.80
Total	19.53	0.98	17.05	20.89	0.00	89.10



**Graph 1.** Showing the mean bond strength of various groups

The clinical success of a restorative material such as composite resins depends on effective sealing of margins of the restoration, enhancing retention and preventing postoperative sensitivity and microleakage [15].

Shorter application time and lower number of applications may prove helpful in pediatric patients. Sound teeth were included in our study as in carious teeth the dentin is decayed and destructed because of the disease process which might influence the results [16, 17]. The preparations were performed on occlusal surfaces, following the methodology described by Van Meerbeek et al, [18] and Perdigao et al [19].

An important characteristic associated with occlusal surfaces used as a substrate for bonding is that a flat cut exposes dentin in different depths in relation to the pulp chamber and this method also allows standardization for the direction of the dentin tubules.

The evaluation of the resin tags in this study showed a great variance between the groups.

Three- step bonding agents showed increased number and density of the resin tags along with greater depth of penetration.

Bond strength of Scotchbond Multipurpose was significantly higher ( $p < 0.001$ ) compared to Futurabond NR, Adh SE and OptiBond-all-in-one.

**Table 2.** Multiple comparisons of digital evaluation

S. No.	Comparison	Lower 95%	Upper 95%	Result
2.	Scotchbond vs. Adhe SE	28.51	34.74	**
3.	Scotchbond vs. OptiBond AIO	28.20	34.58	**
4.	Scotchbond vs. Futurabond	23.03	28.51	**
8.	Adhe SE vs. OptiBond AIO	-0.31	-0.16	
9.	Adhe SE vs. Futurabond	-5.48	-6.23	
10.	OptiBond AIO vs. Futurabond	-5.17	-6.07	

\*\* denotes significantly different pair. Using Dunn multiple comparison

SBS (Shear bond strength) is influenced by the length and density of resin tags; which is a depiction of greater penetration. Better penetration results in greater bond strength of the bonding system. Courson also supported the above-mentioned finding; he found that the bond strength of Scotchbond Multipurpose was greater than the self-etch primers and one-step adhesives but the difference was not significant in primary teeth. While, when the same bonding agents were used on permanent teeth the difference was statistically significant. This difference is due to the compositional differences between primary and permanent dentin [20].

Confirmation to these results were shown by Bolanos- Carmona in 2008 who stated that the values of SBS were higher when a total-etch system was compared to other bonding agents. The SBS of Xeno III increased after pre-etching was included as an additional step [21]. The bond strength of three-step bonding agents is higher than the self-etching bonding agents or the total-etch bonding agents. Agostini et al. evaluated the bond strength of three self-etching primers (Prompt L-Pop- ESPE, Clearfil SE bond- kuraray, Etch and Prime-Degussa) and one adhesive with total etch technique (Prime and Bond NT- Dentsply) to deciduous teeth.

**Table 3.** The mean bond strength in four groups

Group	N	Range	Mean $\pm$ SD	SEM
Adhe SE	9	3.10-7.50	4.4478 $\pm$ 1.3820	0.46070
Futurabond NR	10	2.80-7.10	3.8070 $\pm$ 1.61345	0.51022
OptiBond	9	3.30-6.40	4.4856 $\pm$ 1.07925	0.35975
Scotchbond	10	4.83-11.00	7.4744 $\pm$ 1.88763	0.59692

SD: Standard Deviation; SEM : Standard error of mean

**Table 4.** Comparison between the groups

Comparison	Mean difference	P value
Adhe SE vs. Futurabond NR	0.64078	0.800 <sup>NS</sup>
Adhe SE vs. OptiBond	-0.03778	1.000 <sup>NS</sup>
Adhe SE vs. Scotchbond	-3.02662	0.001*
Futurabond NR vs. OptiBond	-0.67856	0.772 <sup>NS</sup>
Futurabond NR vs. Scotchbond	-3.00740	0.001*
OptiBond vs. Scotchbond	-2.98884	0.001*

NS: P > 0.05; Not Significant; \* P < 0.05; Significant;  
Post-Hoc comparison using Tukey's HSD

When observations were done on enamel surface it was seen that Prime and Bond NT demonstrated significantly higher bond strengths. While Clearfil SE Bond showed the best results with dentin of primary teeth [11]. Clearfil SE belongs to the family of self-etching primers and contains 10-methacryloxydecyl dihydrogen phosphate (10-MDP) as functional monomer; which is dissolved in water. The excellent performance may be attributed to the additional chemical interaction of hydroxyapatite with the functional monomer 10-MDP. It has been shown to chemically interact with hydroxyapatite [22]. Apart from the total removal of smear layer, another factor which may be responsible for the better penetration of the resin monomers in the three-step bonding agents is that after phosphoric acid application on dentin for a brief etching period, due to the buffering action of the mineral phase of dentin a lower diffusion flux of hydrogen ions has been observed [23-25]. This restricts the extension of demineralization.

This phenomenon may contribute to better penetration of adhesives in the demineralized dentin, increasing the bonding efficacy [23]. Courson et al. stated that water occupying the interfibrillar spaces is lost by evaporation during air-drying after etching and rinsing, resulting in a collapse of the proteic network. These morphological changes can impair the penetration of the primer-adhesive resin combination. Thus, careful management of dentin moisture content is warranted [20].

Puppin- Rontani have confirmed the above results in deciduous teeth by testing Scotchbond Multipurpose and Prime and Bond which contains a similar concentration of phosphoric acid. The testing was done for varying time intervals and the results showed that higher values of SBS were reported at 7 and 15 sec of application as compared to a 20 sec application period [26].

Improved bond strength after application of three- step bonding agents may be a result of

more dense and long resin tags produced at the bonding interface; which was also shown in our study. Complete removal of smear layer occurs by means of etching which leads to better penetration of the adhesive; but at the same time the step of etching also leads to removal of water from in-between the collagen fibrils. Thus, precautions have to be taken to prevent collapsing of the collagen fibers. Prolonged drying of dentin should be avoided. However, more research is required to develop newer bonding agents with incorporation of properties of both total-etching systems and self-etching systems.

## CONCLUSION

1. The longest resin tags were seen in Scotchbond Multipurpose (12.20 to 89.10  $\mu\text{m}$ ), which was significantly greater than the other three groups.
2. The bond strength of Scotchbond Multipurpose was significantly higher ( $P < 0.05$ ) compared to Futurabond NR, Adh SE, and Opti-Bond all-in-one.

## REFERENCES

- 1- Neelima L, Santhish ES, Kandaswamy D, Bupesh. Evaluation of microtensile bond strength of total etch, self etch and glass ionomer adhesive to human dentin: an invitro study. *Indian J Dent Res* 2008, 19(2) 129-133.
- 2- Nakabayashi N, Kojima K, Masuhara E. The promotion of adhesion by the infiltration of monomers into tooth substrates. *J Biomed Mater Res.* 1982 May;16(3):265-73.
- 3- Pashley DH, Carvalho RM. Dentin permeability and dentin adhesion. *J Dent.* 1997 Sep;25(5):355-72.
- 4- Montes M, Goes MF, Sinhoreti M. The in vitro morphological effects of some current pre- treatments on dentin surface: A SEM evaluation. *Oper Dent.* 2005 Mar-Apr;30(2):201-12.
- 5- Tay FR, Pashely DH. Dental adhesives of the future. *J Adhes Dent.* 2002 Summer;4(2):91-103.

- 6- Meerbeek BV, Munk JD, Yoshida Y, Inoue S, Vargas M, Vijay P, et al. Adhesion to enamel and dentin: Current status and future challenges. *Oper Dent.* 2003 May-Jun;28(3):215-35.
- 7- Swift EJ. Dentin/enamel adhesives: Review of the literature. *Pediatr Dent.* 2002 Sep-Oct;24(5):456-61.
- 8- da Silva TPD, Aparecida MA, Machado M, Eduardo JN. SEM study of a self etching primer adhesive system used for dentin bonding in primary and permanent teeth. *Pediatr Dent.* 2001 Jul-Aug;23(4):315-20.
- 9- Sumikawa DA, Marshall GW, Gee L, Marshall SJ. Microstructure of primary tooth dentin. *Pediatr Dent.* 1999 Nov-Dec;21(7):439-44.
- 10- Noonan VK, Horner JA, Simpson MD, Mathews WG, Pashley DH. The effect of dentin depth on the permeability and ultrastructure of primary molars. *Pediatr Dent.* 1994 Jan-Feb;16(1):29-35.
- 11- Agostini FG, Kaaden C, Powers JM. Bond strength of self-etching primers to enamel and dentin of primary teeth. *Pediatr Dent.* 2001 Nov-Dec;23(6):481-6.
- 12- Malek afzali B, Ghasemi A, Torabzadeh H, Hamedari R, Tadayon N. Effect of multiple adhesive coating on microshear bond strength to primary tooth dentin. *J Dent (Tehran).* 2013 Mar;10(2):169-74. Epub 2013 Mar 31.
- 13- Miranda C, Maykot Prates LH, de Souza Vieira R, Calvo MCM. Shear bond strength of different adhesive systems to primary dentin and enamel. *J Clin Pediatr Dent.* 2006 Fall;31(1):35-40.
- 14- Perdigao J, Lambrechts P, Van Meerbeek B, Vanherle G, Lopes AL. Field emission SEM comparison of four postfixation drying techniques for human dentin. *J Biomed Mater Res.* 1995 Sep;29(9):1111-20.
- 15- Casgrande L, Brayner R, sarmiento Barata J, Borba de Araujo F. Cervical microleakage in composite restorations of primary teeth- in-vitro study. *J Dent.* 2005 Sep;33(8):627-32. Epub 2005 Mar 5.
- 16- Shashikiran ND, Gunda S, Subba Reddy VV. Comparison of Resin-Dentine interface in primary and permanent teeth for three different durations of dentine etching. *J Indian Soc Pedod Prev Dent.* 2002 Dec;20(4):124-31.
- 17- Nor JE, Feigal RJ, Dennison JB, Edwards CA. Dentin Bonding: SEM comparison of the resin-dentin interface in primary and permanent teeth. *J Dent Res.* 1996 Jun;75(6):1396-403.
- 18- Van Meerbeek B, Inokoshi, S, Braem M, Lambrechts P and Vanherle G. Morphological aspect of resin-dentine interdiffusion zone with different adhesive system. *J Dent Res.* 1992 Aug;71(8):1530-40.
- 19- Perdigao J, Swift EJ, Denehy GE, Wefel JS, Donly KJ. In vitro bond strength and SEM evaluation of dentin bonding systems to different dentin substrates. *J Dent Res.* 1994 Jan;73(1):44-55.
- 20- Courson F, Bouter D, Ruse ND, Degrange M. Bond strength of nine current dentin adhesive systems to primary and permanent teeth. *J Oral Rehabil.* 2005 Apr;32(4):296-303.
- 21- Bolanos-Carmona V, Gonzalez-Lopez S, De Haro-Munoz C, Briones-Luja MT. Interfacial morphology and bond strength of self-etching adhesives to primary dentin with or without acid etching. *J Biomed Mater Res B Appl Biomater.* 2008 Nov;87(2):499-507. doi: 10.1002/jbm.b.31135.
- 22- Moura SK, Pelizzaro A, Bianco KD, de Goes MF, Loguerico AD, Reis A, Grande RHM. Does acidity of self-etching primers affect bond strength and surface morphology of enamel. *J Adhes Dent.* 2006 Apr;8(2):75-83.
- 23- Osorio R, Aguilera FS, Otero PR, Romero M, Osorio E, Garcia-Godoy F, Tolendano M. Primary dentin etching time, bond strength and ultra-structure characterization of dentin surfaces. *J Dent.* 2010 Mar;38(3):222-31. doi: 10.1016/j.jdent.2009.11.001. Epub 2009 Dec 5.
- 24- Sardella TN, Alves de Castro FL, Sanabe ME, Hebling J. Shortening of primary dentin etching time and its application on bond



- strength. *J Dent.* 2005 May;33(5):355-62. Epub 2004 Dec 13.
- 25- Camps J, Pashley DH: Buffering action of human dentin in vitro. *J Adhes Dent.* 2000 Spring;2(1):39-50.
- 26- Puppin- Rontani RM, Caldo- Teixeira AS, Sinhoreti MAC, Correr Sobrinho L. Etching time evaluation on the shear bond strength of two adhesive systems in primary teeth. *Cienc Odontol Bras* 2004; 7(3); 6-14.

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