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INTERFERENCE OF BONDING AGENT ON COMPOUND RESINS RESISTANCE TO MICRO SHEARING

*¹Thiago Santos Dantas Araújo, ¹Sthéfane Mendes Avelar, ¹Letícia Sampaio Antunes Pieroni,
²Polyana Argolo Souza Amaral, ³Ian Matos Vieira and ⁴Saryta Argolo Souza Amaral

¹Graduate in Dentistry; Faculdade Independente do Nordeste (FAINOR), Vitória da Conquista, Bahia, Brazil

²Master in Healthcare Sciences from FACOP; Teacher at Northeast Faculdade Independente do Nordeste (FAINOR), Vitória da Conquista, Bahia, Brazil

³Master and PhD in Dental Materials from Unicamp; Assistant teacher of Dentistry at Bahia South East State University (UESB)

⁴Master in Dental Clinics from Bahiana School of Medicine and Public Health (EBMSP); Teacher at Faculdade Independente do Nordeste (FAINOR), Vitória da Conquista, Bahia, Brazil

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ABSTRACT

Objective: To assess bond strength of different conventional and universal adhesive systems to dentine substrate. **Methods:** 40 bovine teeth had their roots severed and vestibular surface planed until dentine exposure, and were randomly divided into five groups (n=16), according to the adhesive system used: Group SB2- *Adper Single Bond 2*; Group A- *Ambar*; Group APS- *Ambar APS*; Group SBU- *Single Bond Universal*; Group AU- *Ambar Universal*. Two transparent cylindrical matrices were fixated over the hybridized dentine and the compound resin Z250 XT was applied. **Results:** Group SB2 (5.44 MPa) showed the lowest bond strength, followed by Group APS (6.74 MPa), which showed no significant difference from each other, but were significantly different from Groups A, SBU and AU (p<0.05). Group AU (9.38 MPa) showed the highest bond strength, followed by Group A (8.55 MPa) and Group SBU (8.09 MPa); these three groups were statistically similar. **Conclusion:** The five adhesive systems tested showed acceptable bond to dentine substrate, although Groups AU, A and SBU have shown the largest strength among them.

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INTRODUCTION

Dentistry has faced several changes following the development of restorative materials with adhesive properties to dental tissues, leading to a better preservation of the dental structure through more conservative cavity preparations. Therefore, dental materials companies have anticipated the need for new restorative composites and, along with them, the development of adhesive systems (Gönülol et al., 2015). Dental adhesives are combinations of resin monomers (hydrophilic and hydrophobic) with different molecular weight and viscosity. The material's fluidity is obtained by adding resinous diluents, organic solvents (acetone, alcohol, among others), and water (AlShaafi, 2017; Gomes et al., 2010; Bacchi et al., 2010). These systems provide a micromechanical bond and, in some

cases, a chemical bond between the restorative composite and the dentine substrate through the formation of a hybrid layer with the use of different adhesion techniques named conventional technique, or etch and rinse, and self-etch (Dominguette et al., 2012). In the etch and rinse technique, phosphoric acid 30% to 40% alone leads to an increase in the surface energy, removal of smear layer, and exposure of collagen fibers. Thus, the primer/adhesive penetrates the microporosities created and produces the so-called resinous tags, components of the hybrid layer - the main responsible for the resin and dentine structure bond (Braz, Ribeiro et al., 2011; Rodrigues et al., 2015).

On the other hand, the self-etch mechanism does not require the use of acid alone because it remains incorporated to the adhesive compound through a complex mixture. Thus, demineralization is simultaneous with hybridization where the smear layer is incorporated (Muñoz et al., 2009).

*Corresponding author: Thiago Santos Dantas Araújo

Graduate in Dentistry; Faculdade Independente do Nordeste (FAINOR), Vitória da Conquista, Bahia, Brazil

Chart 1. Materials, compositions, and bonding procedures

Materials/Manufacturer	Composition	Bonding procedures
Adper Single Bond 2 (3M ESPE, St. Paul, MN, USA)	BisGMA, HEMA, dimethacrylate, ethanol, water, polyacrylic acids and polyalenoic acid methacrylate copolymer, photoinitiator	Etch with phosphoric acid 37% - 15 sec, rinse for 10sec, dry with cotton ball. Adhesive - Apply two layers (rubbing the first product drop for 10 seconds), air-blast for 10 sec, apply the second drop passively for 10 sec, photoactivate for 10 sec
Ambar (FGM Produtos Odontológicos, Joinville - SC - Brazil)	Active Ingredients: MDP (10-Methacryloyloxy decil dihydrogen phosphate) Methacrylic monomers, Photoinitiator, Coinitiators, and Stabilizer. Inactive Ingredients: Inert Load (Silica nanoparticles) and Vehicle (ethanol).	Etch with phosphoric acid 37% - 15 sec, rinse for 10 sec, dry with cotton ball. Adhesive - Apply two layers (rubbing the first product drop for 10 seconds), air-blast for 10 sec, apply the second drop passively for 10 sec, photoactivate for 10 sec
Ambar APS (FGM Produtos Odontológicos, Joinville - SC - Brazil)	Active Ingredients: Methacrylic monomers, Photoinitiator, Coinitiators, and Stabilizer. Inactive Ingredients: Inert Load (Silica nanoparticles) and Vehicle (ethanol).	Etch with phosphoric acid 37% - 15 sec, rinse for 10 sec, dry with cotton ball. Adhesive - Apply two layers (rubbing the first product drop for 10 seconds), air-blast for 10 sec, apply the second drop passively for 10 sec, photoactivate for 10 sec
Single Bond Universal - (3M ESPE, St. Paul, MN, USA)	Acid phosphate monomers (MDP), silane, water, ethanol, HEMA, dimethacrylate resins, polyacrylic acid and polyalenoic acid methacrylate copolymer, initiators, load	Apply two layers (rubbing the first product drop for 10 seconds), air-blast for 10 sec, apply the second drop passively for 10 sec, photoactivate for 10 sec
Ambar Universal (FGM Produtos Odontológicos, Joinville - SC - Brazil)	Active Ingredients: MDP (10-Methacryloyloxy decil dihydrogen phosphate) Methacrylic monomers, Photoinitiator, Coinitiators, and stabilizer. Inactive Ingredients: Inert Load (Silica nanoparticles) and Vehicle (ethanol).	Apply two layers (rubbing the first product drop for 10 seconds), air-blast for 10 sec, apply the second drop passively for 10 sec, photoactivate for 10 sec

The objective of this study is to assess the bond strength of different adhesive systems, both conventional and universal to the dentine substrate through micro shearing.

MATERIALS AND METHODS

In this study, the samples consisted of 40 recently extracted bovine lower anterior teeth. The teeth were kept under cooling until the manufacturing of the specimens. The roots were sliced using a double-sided flexible diamond disc (Ref. 7016, KG Sorensen, Barueri, SP, Brazil), and each teeth's vestibular surface was planed using a series of silicon carbide sandpapers (grit sizes 200, 400 and 500) mounted on a water-cooled rotating horizontal electric polishing machine (Model APL-4, Arotec, Cotia, SP, Brazil). Planing took place until deep exposure of dentine and achievement of a plane surface. In order to use the dentine mud formed, the adhesive procedures were conducted immediately after surface abrasion with silicon carbide and according to the manufacturer's recommendations (Chart I). Groups (n=16) were then randomly divided according to bonding agent used: Group SB2- Adper Single Bond 2; Group A- Ambar; Group APS- Ambar APS; Group SBU- Single Bond Universal; Group AU- Ambar Universal. A LED photopolymerizer device (Optilight Max 440 – Gnatus, Ribeirão Preto, SP, Brazil) was used in photoactivation of all procedures. Specimens were prepared according to the methodology developed by McDonough *et al.* (2002) and Shimada *et al.* (2002) for the micro shearing assay. Two transparent cylindrical matrices (Tygontubing, TYG-030, Saint-Gobain Performance Plastic, MaimeLakes, FL, USA – 0.75 mm of inner diameter and 0.5 mm of height – 0.44 mm² of area by πR^2) were placed over each hybridized dentine sample. Compound resin (Z250 XT - 3M ESPE, St. Paul, MN, USA) was applied with the aid of an exploratory probe#5 (SSWhite/Duflex, Rio de Janeiro, RJ, Brazil), to fill the matrices inner volume. After 20 seconds of photopolymerization, the matrices were removed using a disposable carbon steel scalpel #15 (Solidor/LAMEDID, Barueri, SP, Brazil) and, once the cylinders were exposed, another 20-seconds photopolymerization was sustained, totaling 40 seconds of photopolymerization. Specimens were stored in distilled water at 37°C, for 24 hours, after which, the

micro shearing assay was conducted. After this time, samples containing the specimens were bonded to the micro shearing universal testing machine test device (Oswaldo Fizola AME-2Kn, São Paulo, SP, Brazil). Force was applied through a shear loading at the composite cylinders base with a steel wire (0.20 mm of diameter), at a velocity of 0.5 mm/min, until bonding rupture. Values at the time of rupture were recorded in Newton and converted to Mega Pascal (Mpa) according to:

$$\text{Mpa} = \frac{\text{Newton}}{\text{area (mm}^2\text{)}}$$

Bond strength was calculated and expressed in Mpa, and the average reading was determined for each specimen. Results were statistically analyzed with variance analysis (ANOVA) and Tukey's *post hoc* test with 5% of significance. The analyses were conducted with the aid of Microsoft Excel 2016 (Microsoft Office system for Mac 2011) and SPSS 21 (SPSS Inc., Chicago, IL, USA).

RESULTS

Data were subjected to Kolmogorov-Smirnov normality test. Once established normal distribution for all data, the possible variations of micro shearing bond strength were analyzed with one-way ANOVA comparing the different adhesive systems (Group SB2 - Adper Single Bond 2; Group A - Ambar; Group APS - Ambar APS; Group SBU - Single Bond Universal; Group AU - Ambar Universal). To identify significantly different means, a detailed variance analysis (ANOVA) with Tukey's *post hoc* test was necessary. Values of $p \leq 0.05$ were considered significant, i.e., minimum significance level of 5%. ANOVA showed significant difference between the groups regarding bond strength ($p < 0.05$). Variance homogeneity and Tukey's *post hoc* test were conducted. Group SB2 (5.44 MPa) showed the lowest bond strength, followed by Group APS (6.74 MPa), deemed statistically equivalent, but different from Groups A, SBU and AU ($p < 0.05$). Group AU showed the largest bond strength (9.38 MPa), followed by Group A (8.55 MPa) and Group SBU (8.09 MPa); however, there was no statistical difference between these three (Table 1).

Table 1. Description and comparison of bond strength values to micro shearing (MPa) of studied groups

Groups	min	max	Mean (SD)	p-value
SB2	2.98	8.44	5.44 (1.51) a	0.00
A	4.94	14.17	8.55 (2.51) b,c	
APS	3.85	10.29	6.74 (1.93) a,b	
SU	4.48	15.64	8.09 (2.99) c	
AU	6.56	16.89	9.38 (2.71) c	

* Different lower case letters in the same column indicate statistically different averages (Tukey $p < 0.05$).

DISCUSSION

Studies on the bond strength of bonding agents require recently extracted human teeth; however, due to the preventative dentistry ideology, the difficult standardization, and bioethical issues, studies using human teeth are difficult to attain. Some authors have investigated potential surrogates for *in vitro* studies and found similarities between human and bovine teeth both in histological and morphological aspects (Carvalho *et al.*, 2015). Here we used bovine teeth based on the claims of authors such as Neto *et al.* (2015) and Pimenta-Dutra *et al.* (2017) that there are no statistical differences in bond strength of human and bovine teeth, both for enamel and dentine, under scan electron microscopy. Results found for the universal adhesive systems using the self-etch technique show no difference between Groups SBU and AU. Hence, we have showed that the simplification of the self-etch technique is advantageous to adhesion. This simplification, with reduction in application time, represents one of the main and most desired characteristics of these new materials, which include the so-called single bottle and self-etching systems (Carvalho *et al.*, 2012). In this technique, the previous substrate acid etching is suppressed, and hybridization is done without the smear layer removal, which theoretically is incorporated to the hybrid layer (Reiset *et al.*, 2004; Ramos *et al.*, 2016; Bumrungruan *et al.*, 2016; Chiang *et al.*, 2016). The main advantage of using these adhesive systems is the enhanced control over substrate humidity since acid etching is simultaneous to the primer application. To this end, the concentration of acid monomers was raised from 6% to about 20%, increasing acidity sufficiently to demineralize and infiltrate the dental substrate at the same time, thus eliminating another downside - the potential discrepancy between the depth of the demineralized substrate and the actual monomer penetration. The hybrid layer is formed from the primer and adhesive penetration into the demineralized dentine and represents the depth of dentine demineralization (Verna *et al.*, 2018; Protásio *et al.*, 2016; Fróis *et al.*, 2012; Alqahtani, 2015).

When compared with the different systems classification performance, typically, the universal adhesive agents bond strength was higher than that showed by conventional adhesive systems. According to Giannini *et al.* (2015), the self-etch technique has the advantage of being a single-step procedure, where the surface is etched while the primer penetrates the tubules, incorporating all smear layer and dissolved hydroxyapatite present. Among the conventional adhesive systems assessed here, Group A showed higher bond strength to dentine than Group SB2 ($p < 0.05$). According to Arinelli *et al.* (2016), this behavior is explained by the bonding agent composition. According to the manufacturer's disclosed information and to El Sayed (2015), this difference is due to the lack of the monomer 10-methacryloyloxydecyl dihydrogen

phosphate - monomer responsible for chemical bonding - in *Adper Single Bond 2*, as well as to the water added to its composition as an integral part of the solvent, while Ambar has methacrylates dissolved in ethanol. Lobo *et al.* (2012) stress that, although water improves permeability and wet ability of the bonding agent, its low volatility prevents its complete evaporation, which can result in reduced resistance and incomplete polymerization of the material.

Our data show that Group AU presents the highest bond strength among all groups. According to Vinagre *et al.* (2014), the success of this technique is due to the suppression of the acid etching phase and rinsing. Arinelli *et al.* (2016) stress the importance of the bonding agent composition for the obtained result. Although Ambar Universal and Adper Single Bond Universal are universal systems, the better results showed by the first are due to the lack of water in its composition (El Sayed *et al.*, 2015). Also, Ambar Universal contains the monomer 10-methacryloyloxy decyl dihydrogen phosphate (10-MDP), a particle capable of binding to calcium ions originated from hydroxyapatite crystal dissolution, characterizing an additional adhesion to the 10-MDP particles, thus improving the agent's bond (Arinelli *et al.*, 2016; El Sayed *et al.*, 2015). In addition to that, as proven by El Sayed *et al.* (2015), the chemical bond provided by 10-MDP favors a better performance and, as a consequence, increases the bond strength of the bonding agent. This corroborates our results for Groups A, SBU and AU, which also showed similar results due to their composition. In this scenario, one can claim that the bonding agent composition and failures minimization during the procedure interfere directly with adhesion and resistance of the dentine substrate. Given the results presented here, it is possible to conclude that this work, within its limitations, brings a valuable contribution to scientific knowledge, addressing five bonding systems widely employed in compound resin restorations currently. However, further studies on this line of research are recommended, with larger sample sizes submitted to different times and types of test (Couto *et al.*, 2016)

Conclusion

The results obtained show that the five adhesive systems tested here present acceptable bond to the dentine substrate. Also, we showed a better performance of Groups A, SBU and AU, which can be explained by the technique, and mainly by the agents' composition. Thus, it is concluded that these factors can directly influence the adhesive strength and, as a consequence, the bonding longevity.

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