

***In vitro* comparison of the immediate adhesive strength in dentin of three universal adhesive systems.**

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Abstract: Objective: To compare the adhesive strength in dentin of three universal adhesive systems *in vitro* by means of the shear test. Materials and methods: Seventy-five bovine teeth were selected and cut. Dentin was exposed from the buccal surface of the crowns with 220 grit sandpaper, and samples were then inserted in transparent acrylic bases (15x10mm). The samples were randomly divided in 3 groups (n=25): G1-Universal adhesive system Scotchbond™ Universal (3M ESPE-USA); G2- Universal adhesive system Peak Universal Bond (Ultradent); G3-Universal adhesive system Tetric N-Bond (Ivoclar Vivadent). The adhesive procedures were carried out according to the instructions of each manufacturer and the restorative procedures were carried out with micro-cylinders (made of Tygon type tubing 0.79x1.5mm) of the composite resin Filtek™ Z350XT-A2 (3M ESPE-USA). The samples were incubated at 37°C (+/-5°C) for 24 hours. Adhesive strength was evaluated in a universal test machine by means of the shear test (0.5mm/min, 500N) and the resulting fracture type was evaluated with a Dinolite digital microscope (x200). The results were analyzed by descriptive statistics (Mean±SD), and inferential statistics by a one-way ANOVA. Results: No statistically significant differences were found between the universal adhesive systems evaluated G1 (14.91±4.76), G2 (16.90±4.11) and G3 (17.34±4.04)/(p=0.114). Conclusions: The shear test resulted in similar values of immediate adhesive strength of the three universal adhesive systems used.

Keywords: shear strength; adhesives; dentin.

INTRODUCTION.

Adhesion to dentin continues to be a challenge for clinicians and researchers. It is not yet completely effective and there are still many flaws in the adhesive interface. This is due to the complexity of the substrate (collagen fibrils, peri- and intra-tubular demineralization), type of substrate (sclerotic or affected dentin) and other conditions related to the technique such as the number of clinical steps, humidity control and operator training.^{1,2}

In the last few years the dental industry has developed the so-called universal adhesive systems. These are usually presented in a single bottle and can be applied to different dental substrates (enamel, dentin, cement) with different acid etching techniques: total etching, selective etching and self-etching.³ They also allow for the creation of stable adhesive chemical bonds with direct or indirect restorative materials and to be used as adhesive primers to zirconia, precious and non-precious metals, composite resins and on silica-based ceramics.^{2,3} The use of universal adhesives aims to reduce the number of clinical steps, since universal

adhesives contain in a single bottle components such as hydrophobic and hydrophilic monomers Bis GMA, UDMA, HEMA, BPDMA, META) together with charged particles, solvents (ethanol, ketones) and water.^{1,2} Furthermore some also contain further components such as 10-MDP particles (10-methacryloyloxydecyl dihydrogen phosphate), polyalkenoic acid, chlorhexidine and organic solvents.^{4,5}

Recent scientific studies¹⁻⁵ have assessed the mechanical and physical properties of the new universal adhesive systems to different dental substrates or restorative materials, since the longevity of an adhesive restoration is directly associated with the effectiveness of the adhesive systems. This is why it is still important to study the adhesive strength of these new materials in the dentin/composite resin interface. As such, the objective of this study was to compare the immediate adhesive resistance in dentin *in vitro* of three universal adhesive systems. The hypothesis was that there are no significant differences in the immediate adhesive strength to dentin between the following adhesive systems: Scotchbond™ Universal (3M ESPE), Peak Universal Bond (Ultradent, USA) and Tetric N-Bond (Ivoclar Vivadent, Liechtenstein).

MATERIALS AND METHODS.

The present study was *in vitro* experimental study, whose unit of analysis consisted of a specimen of bovine tooth restored with three different universal adhesive systems (Table 1) and micro cylinders of composite resin, and shear tests. The sample size consisted of 75 teeth (test power 80%, confidence level 95%, alpha or error 0.05/Stata® version 13.0). The selection criteria were caries-free bovine teeth without enamel defects, fracture or wear. The distribution of the groups was: Group 1, SU (n=25), Scotchbond™ Universal (3M ESPE, USA); Group 2, PUB (n=25), Peak Universal Bond (Ultradent, USA); and Group 3, TNB (n=25), Tetric N-Bond (Ivoclar Vivadent, Liechtenstein).

Obtention of specimens

Seventy-five randomly selected bovine teeth were cleaned with curettes (Mc Call™ #13/14, #6-SM13/146, Hu-Friedy, USA) and with a Robinson brush (Dentalflux™, Spain) impregnated with pumice. The teeth were preserved in distilled water (50ml), which was replaced every 10 days. The storage time was a maximum of 15 days.

Root sectioning and dentin exposure

The separation of the crown-root was performed at 4 mm below the cemento-enamel junction in a digital cutting machine 1000 (Buehler, IsoMet™, USA). Once separated, the vestibular face was abraded with waterproof sandpaper 220 (ASA, Peru), until the dentin was exposed, followed by treatment with sandpaper 600 for 1min in order to create a smear layer. The dental crowns, randomly distributed in each group, were included individually in self-curing acrylic resin (Vitacryl, Peru) inside polyvinyl chloride tubes (25x15mm) (PVC, Peru) and stored in distilled water⁵ at a room temperature of 23°C for 12 hours.

Adhesive and restorative procedures

The three adhesive systems were applied according to the indications of the manufacturer of each product, using a total etching technique for 20s. For group 1 (G1), the universal adhesive system SU (3M ESPE, USA) was dispensed using a Microbrush® (Plus and Tube Series, USA), generating friction on the dentin (20 s), air was applied indirectly with the triple syringe for 5s. For group 2 (G2), the universal adhesive PUB (Ultradent, USA) was also dispensed in a Microbrush®, generating friction on the dentine for 10s, and air was applied indirectly for 5s. For group 3 (G3) the TNB universal adhesive (Ivoclar Vivadent, Liechtenstein) was likewise dispensed in a Microbrush®, generating friction on the dentine for 20s, and air was applied indirectly 5s. All the adhesives were polymerized with a Bluephase LED lamp (Ivoclar Vivadent, Liechtenstein), previously calibrated with a radiometer at 828 mW/cm² (Monitex, DigiRate LM-100, China): SU for 10s, PNB for 20s, and TNB for 10s. Finally, for the restorative procedures in the three groups, four Tygon tubes (0.79mmx1.5mm) were placed on the surface of the substrate and the composite resin Filtek™ Z350 XT-A2 (3M ESPE, USA) was inserted using an applicator instrument (Hu-Friedy TNCIGFT1, USA) in a single step, and was light-cured for 20s. Immediately, the micro-cylinder tubes were removed with a #15 scalpel, with transverse cuts along all the length of the tube. All the samples were stored in an incubator (Hotpack, Philadelphia, PA, USA) at 37°C (+/- 5°C) for 24 hours in distilled water.

Shear test

The shear test was performed on an Instron® universal testing system machine (3382 Series, USA - 0.5 mm/min,

Table 1. Universal adhesive systems and restorative materials used in this study.

Adhesive	Manufacturer	Main Components	ID
Scotchbond Universal Lot No. 616340	3M ESPE dental Products St. Paul, MN55144, USA	*10-MDP phosphate monomer, HEMA, dimethacrylate resins, Vitrebond™ copolymer, filler, ethanol, water, initiators, silane	SU
Peak Universal Bond Lot No. BDMCB	Ultradent dental Products, South Jordan UT, USA	Ethyl alcohol (<20%), 2-hydroxyethyl methacrylate(<16%), methacrylic acid (<6%), chlorhexidine diacetate (<0.3%).	PB
Tetric N Bond Universal Lot No. V20886	Ivoclar Vivadent dental products, Schaan, Liechtenstein	Methacrylates, ethanol, water, highly dispersed silicon dioxide, initiators and stabilizers	TB
Scotchbond Etchant Lot No. N748950	3M ESPE dental Products St. Paul, MN55144, USA	37% phosphoric acid Resin Composite	SE
Filtek Z350 XT A2 Lot No. N817424	3M ESPE dental Products St. Paul, MN 55144, USA	**Bis-GMA, UDMA, TEGDMA, Bis-EMA, silanated silica, silanated zirconia, photoinitiators	FZ

*10-MDP:10-Methacryloyloxydecyl dihydrogen phosphate. **Bis-GMA: bisphenol A diglycidyl methacrylate. Bis-EMA: (Bisphenol A polyethylene glycol diether dimethacrylate). TEGDMA: triethyleneglycol-dimethacrylate; UDMA (urethane dimethacrylate).

Table 2. Universal adhesive systems and restorative materials used in this study.

Universal Adhesive System	Mean (Mpa)	S.D.	p-value*
Adhesivo Scotchbond™ Universal (3M ESPE)	14.91	4.76	
Peak® Universal Bond (Ultradent).	16.90	4.11	0.114
Adhesivo Tetric® NBond Universal (Ivoclar-Vivadent).	17.34	4.04	

*One-way ANOVA ($p \leq 0.05$)

cell 500N). Shear device 000401 (ODEME Dental OD07, Brazil) was positioned at 2mm perpendicular to the resin micro-cylinder. The test culminates with the separation of the cylinder from the dentin surface, with maximum force measured in megapascals (MPa).

The univariate statistical analysis was performed by obtaining the Mean (\pm Standard Deviation) of the adhesive strength of each group and comparing between adhesive strength and universal adhesive system used. Also, a normal distribution was determined by the Shapiro Wilk test. For the bivariate analysis we used a one-way ANOVA test to compare the adhesive strength of the different universal adhesives ($p \leq 0.05$, Stata® version 13.0).

The resulting fractured surfaces were analyzed with a Dino-Lite Edge digital microscope (AM5216 Series, USA-200x). The surface pattern was classified as: fracture produced in the adhesive interface (adhesive type A); fracture presents fragments of adhesive system and composite resin (mixed type M); fracture produced in dentine (Cohesive type in dentin CD); and fracture

produced in the restoration material (Cohesive type in composite resin CR).^{4,6} A inferential statistical analysis was performed using the Chi-square test ($p \leq 0.05$, Stata® version 13.0).

RESULTS.

No significant differences were found between the three universal adhesive systems evaluated: SU (14.91 ± 4.76), PUB (16.90 ± 4.11) and TNB (17.34 ± 4.04); $p = 0.114$. (Table 2)

The microscopic evaluation of the surfaces showed a greater percentage of mixed type and adhesive fractures resulting from the shear test. The most common fracture type was that of mixed type, at 50.67% (152/300 micro-cylinders), followed by 39.33% adhesive type fractures (118/300 micro-cylinders), 4% cohesive-resin type (12/300 micro-cylinders) and 6% cohesive-dentine type 6% (18/300 micro-cylinders). A significant difference ($p = 0.03$) was observed in favor of mixed and adhesive type fractures.

DISCUSSION.

The purpose of this study was to compare the immediate adhesive resistance in dentine of three universal adhesive systems *in vitro*, and no statistically significant differences were found between them, similar to previous reported results.^{1,6}

Several authors have carried out similar studies using the shear test to measure the adhesive strength between restorative material (composite resins, ceromer, vitreous ceramics) and substrates (enamel, dentin, cement). Also this test allows the evaluation of samples of smaller diameter and height, since it increases the sensitivity of the measurements, resulting in more precise values.^{4,6}

Mixed type of fractures were the most prevalent in this study. According to the scientific literature, the most frequent types of failures are of adhesive and mixed type; this indicates that the measurement of adhesive resistance employed was valid. Also, bovine teeth were used in this study, which are ideal substitutes for human teeth due to their morphological and physiological similarities.^{1-3,6}

The substrate is one of the most important factors affecting the stability and longevity of adhesion in dentistry. Several authors have evaluated the adhesive strength on dentin of different adhesive systems over time and concluded that the impregnation of synthetic resin monomers in demineralized dentin is a disadvantage, due to moisture, permeability and pulpal hydrostatic pressure related to the composition of the substrate.⁷⁻⁹

Regarding universal adhesive systems, the success of the adhesive strength is related to the chemical bond that is initially formed between the functional monomers of the universal adhesive and the hydroxyapatite (HAP) of the dental substrate, where there is a balance between the hydrophilic monomers and hydrophobic adhesive.^{4,5}

The present study evaluated the adhesive strength of three different universal adhesive systems, SU, PUB and TNB, with dentin; with no statistically significant differences were found between them. This could be due to the similar composition shared by almost all universal adhesive systems (in different proportions depending on the manufacturer), such as the adhesive monomers Bis-GMA, UDMA, HEMA, BPDM, 4 META, together with charged particles, solvents (ethanol, ketones) and water.^{1,2} These provide a balance between hydrophilicity (HEMA) that allows for the moistening, infiltration and interaction with the dentin substrate, and

hydrophobicity (Bis-GMA), that once polymerized, avoids the sorption of water and prevents the breakdown of the adhesive interface throughout time.³

Among the universal adhesive systems evaluated, the SU universal adhesive contains 10-MDP particles, a molecule that can create stable bonds between calcium (Ca^{2+}) monomers present in the adhesive and hydroxyapatite (HAP), which forms a calcium salt. This salt has low solubility and high resistance to biodegradation. It also contains polyalkenoic acid, which allows for the formation of stable chemical bonds with dentin, similar to glass ionomer cement.⁴ On the other hand, the TNB adhesive presents in its composition a matrix based mainly on hydrophilic monomers (HEMA) that promote adhesion and improve the immediate adhesive resistance of the adhesive system by the diffusion of the monomers in the dentin, thus facilitating the formation of a hybrid layer. Finally, the PUB system is composed of solvent (ethanol <20%), methacrylate acid (<6%), 2-hydroxyethyl methacrylate (<16%) and 0.2% chlorhexidine, the presence of which would enhance preservation and longevity of the hybrid layer by inhibiting metalloproteinases (MMPs).⁴

The pH of the current universal adhesives varies between 2.2 and 3.2 depending on the manufacturer. Universal adhesives are generally considered to have a "mild" (pH 2.2) or "extra mild" (pH greater than 2.5) conditioning capacity.³ This pH range may be beneficial to adhesion to dentin.^{2,5,9,11}

These new materials can be applied with different techniques of acid etching (total etching, selective and self-etching) depending on the clinical situation and preference of the operator.^{3,8,10} The technique of total engraving (for 20s) was used in the present study. Previous studies have shown using transmission electron microscopy (TEM) that these adhesives applied using self-engraving can create hybrid layers 0.2 to 0.5nm thick due to the acidic monomers they possess, thus being classified as mild-etch adhesives.⁵ As such the application of the technique of total or selective engraving would be recommended to obtain a suitable impregnation of the surface of the substrate (enamel or dentin) and to create hybrid layers 1 to 1.5 nanometers thick.^{5,10}

On the other hand, the use of substances such as chlorhexidine (CHX), a disinfecting agent and inhibitor of MMPs, has also recently been assessed in studies on the adhesive strength of universal adhesive systems. A study by Bravo *et al.*, evaluated the adhesive resistance, by shear test,

of a selective etching adhesive system, a self-etching adhesive and a universal adhesive. The results showed similar adhesive strength values at 72 hours in the groups treated with and without 2% CHX; however the groups treated with CHX presented higher adhesive resistance at 3 and 6 months.¹¹

The emergence of new universal adhesive systems warrants *in vitro* or clinical studies to determine with greater precision the bonding strength of these materials over time. Also to assess the effect of disinfecting agents such as 2% CHX solutions

or specific components of the universal adhesive system that improve the adhesive strength of different substrates or restorative materials and guarantee the longevity of adhesive restorations.

CONCLUSION.

The immediate dentine adhesive strength of the three universal adhesives tested was similar when evaluated by shear test.

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