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The Bond Strength of Universal Adhesives with Different Acidities to Calcium Silicate-Based Materials

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ABSTRACT

Objective: To compare the micro-shear bond strengths of 3 different universal adhesives to 2 different calcium silicate-based materials.

Methods: A hole was prepared in the center of the top surfaces of 60 cylindrical acrylic blocks, and the blocks were randomly divided into 2 groups (n=30) according to biomaterial filling; NeoMTA Plus (Avalon Biomed Inc. Bradenton, FL, USA) and ProRoot MTA (Dentsply Tulsa, Tulsa, OK, USA). Biomaterials were prepared according to the manufacturers' instructions. The sample surfaces were flattened after the initial setting, and the samples were incubated for 24h at 37°C. After placing the biomaterials in the prepared holes, the specimens were randomly divided into 3 subgroups (n=10) according to adhesive; G-Premio Bond (GC Corp., Tokyo, Japan), All-Bond Universal (Bisco, Inc., Schaumburg, IL, USA) and Single Bond Universal (3M ESPE, St Paul, MN, USA). Adhesives were applied to the specimens and polymerized. A micro-hybrid composite resin (Filtek Z250, 3M ESPE, MN, USA) was then placed on the specimen surfaces and polymerized. Micro-shear bond strengths were tested using a universal testing device (LRX, Lloyd Instruments, Farnham, UK). A Shapiro-Wilk test confirmed normal distribution, and 2-way ANOVA was used for statistical analysis.

Results: No significant differences were found in the shear bond strengths of any of the tested adhesives to either of the calcium silicate-based materials ($p>0.05$).

Conclusion: The results indicate that the acidity of a universal adhesive does not affect the bond strength of composite resin to calcium silicate-based materials.

Keywords: Calcium Silicate, NeoMTA, ProRoot MTA, Shear Strength, Universal Adhesive

1. INTRODUCTION

Vital pulp treatment (VPT) aims to maintain the vitality and health of dental pulp in cases where pulp exposure has occurred due to caries or traumatic injury (1). Several procedures have been developed to protect pulp vitality in permanent teeth. Indirect pulp capping involves the controlled excavation of deep caries to prevent pulpal exposure followed by the application of a biomaterial as a protective layer to maintain pulp vitality. With direct pulp capping, a protective layer of biomaterial is applied directly on exposed pulp tissue, and with pulpotomy, the same procedure is performed after partial amputation of the pulp (2). The biomaterial used in VPT must not only be biocompatible and capable of maintaining pulp vitality, it also needs to adhere to both dentin and restorative material and resist the forces generated during restoration placement and function (3). Calcium hydroxide (CH) has long been accepted as the gold standard; however, the materials has some limitations, such as degradation after acid-etching, insufficient adhesion, high solubility in oral fluids, tunnel defects inside the dentin bridge, and pulp-chamber obliteration caused by excessive dentin formation (4).

Several materials have been proposed as alternatives to CH, including calcium hydroxide liners, dentin bonding agents, mineral trioxide aggregate, glass ionomer cement, zinc oxide/eugenol, calcium silicate, and medical Portland cement (5). Mineral Trioxide Aggregate (MTA), comprised of bismuth oxide and modified Portland cement, was developed as a durable, biocompatible alternative for use in various endodontic applications (6). Over the years, studies have shown that MTA can be used successfully in pulpotomy procedures, pulp capping, apexification and root-canal obturation as well as for treating perforations and internal root resorption (6,7). In addition to biocompatibility, MTA possesses low solubility, is able to set in wet conditions and in the presence of blood, and can prevent bacterial leakage;

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however, the material is difficult to manipulate and has a long setting time (8).

The bond strength between the pulp-capping and restorative materials plays a very important role in restoration quality (9). Some studies have suggested that restoration with a resin composite and a bonding agent can be performed immediately following MTA placement. Moreover, acid-etching prior to composite application has been shown to produce surface changes that increase the bond strength of resin-based materials; however, it has also been shown to reduce the compressive strength and surface microhardness of MTA (10,11). Recently, universal adhesives (UAs) have been introduced that can be used in either etch&rinse, self-etching, or selective-etching modes, depending upon clinical conditions and the clinician's preferences. These new, single-bottle adhesives have become popular due to their simple application procedures and short application times (12). These adhesive systems differ from etch-and-rinse adhesives in several aspects, such as the initial pH, type of acidic monomer, the concentration of water and solvents, and the hydrophilicity of the bonding layer and they can be classified as mild, moderate and acidic systems depending on their initial pH (13). Previous studies have stated that the bond strength of MTA to resin can be affected by adhesive solvent type (acetone, ethanol, or water) and filler content (10); however, the effect of UA acidity on the bond strength of MTA to resin composite has not been investigated. Therefore, this study aimed to compare the micro-shear bond strength of three diferrent UAs with different acidity levels (G-Premio Bond-1,5 (14), Single Bond Universal-2,7 (15) and All-Bond Universal-3,2 (16) to ProRoot MTA and NeoMTA Plus. The null hypothesis was that there would be no difference in the micro-shear bond strength of UAs with different acidity levels to the calcium silicate-based biomaterials tested.

2. METHODS

Ethics committee approval was not taken due to in vitro design of the study. This study does not include human participants. Thus, no consent form was required. Sample size was calculated a priori using the effect size of a previous study (17) with analysis of variance (fixed effects, omnibus, 1-way) test from F test family and an alpha-type error of 0.05 and a power beta of 0.95 (G*Power 3.1 for Mac.; Heinrich Heine, Universitat Dusseldorf, Dusseldorf, Germany). Ten specimens per group were indicated as the minimum sample size to observe the same effect.

Material composition and manufacturer details are given in Table 1. A 5-mm dia. x 2-mm h. hole was prepared in the centre of the top surfaces of 60 cylindrical acrylic blocks, which were then randomly divided into 2 groups (n=30) according to biomaterial filling. NeoMTA Plus (Avalon Biomed Inc. Bradenton, FL, USA) and ProRoot MTA (Dentsply Tulsa, Tulsa, OK, USA) were prepared according to the manufacturers' instructions by mixing the ProRoot MTA powder with the liquid provided and the NeoMTA Plus powder with the anti-washout gel provided. The materials were transferred into the holes and compacted using a spatula. The samples were covered with wet cotton pellets, stored at 37°C and 100% humidity for 24 hours to allow the materials to set completely, and then polished with 600-grit SiC paper (#600, Tigre; Pinceis Tigre SA, Castro, Brazil) for 60 seconds to obtain uniform, flat surfaces.

Table 1. *The chemical compositions and manufacturer details of the tested materials*

Material	Main components	Manufacturer
ProRoot® Mineral Trioxide Aggregates	Tricalcium silicate, bismuth oxide, dicalcium silicate, tricalcium aluminate, calcium sulfate dehydrate or gypsum	Dentsply Tulsa Dental, OK, USA
NeoMTA Plus	**Powder:** Tricalcium silicate (Ca_3SiO_5), Dicalcium silicate (Ca_2SiO_4), and Tantalum oxide (Ta_2O_5). **Liquid:** Water (H_2O) and proprietary polymers.	Avalon Biomed, Bradenton, Florida
G-Premio Bond	MDP, 4-MET, MEPS, methacrylate monomer, acetone, water, initiator, silica filler,pH:1,5	GC, Tokyo, Japan
Single Bond Universal	MDP, Bis-GMA, HEMA, DMA, methacrylate functional copolymer, filler, ethanol, water, initiators, silane pH:2,7	3M ESPE, St Paul, MN, USA
All-Bond Universal	MDP, Bis-GMA, HEMA, ethanol, water, initiators pH:3,2	Bisco Inc, Schaumburg, IL, USA
Filtek Z250 Universal Restorative System	Zirconia/silica filler, UDMA, Bis-GMA and Bis-EMA resins.	3M ESPE,MN, USA

Bis-GMA: bisphenol-A-glycidyldimethacrylate; Bis-EMA: ethoxylated bisphenol-A-dimethacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate; 4-MET: 4-methacryloyloxyethyl trimellitate; MEPS: methacryloyloxyalkyl thiophosphate methylmethacrylate; UDMA: urethane dimethacrylate; DMA: N, N-dimethylacrylamide.

Specimens were then randomly divided into 3 subgroups according to universal adhesive (n=10). G-Premio Bond (GC Corp., Tokyo, Japan), All-Bond Universal (Bisco, Inc., Schaumburg, IL, USA) and Single Bond Universal (3M ESPE, St Paul, MN, USA) were applied according to the manufacturers' instructions and polymerized with a LED light-curing unit (Elipar S100, 3M ESPE, MN, USA) for 10 s. A silicone tube (0.8 mm internal dia. x 2 mm h.) was positioned on the centre of each sample, and composite resin (Filtek Z250, 3M ESPE,MN, USA) was placed inside the tubes and polymerized for 20 seconds. Specimens were stored at 37°C and 100% humidity for 24 hours.

Micro-shear bond strength was tested using a knife-edge blade mounted in a Universal Testing Device (LRX, Lloyd Instruments, Farnham, UK) (Figure 1). A load was applied with a crosshead speed of 1mm/min., and the load at failure was recorded in Newtons and converted into MPa. Failure modes were evaluated by a single operator under a stereomicroscope (Nikon SMZ 745T; Tokyo, Japan) at x40 magnification and categorized as either mainly adhesive, mainly cohesive within the resin cement, or mixed (Figure 2).

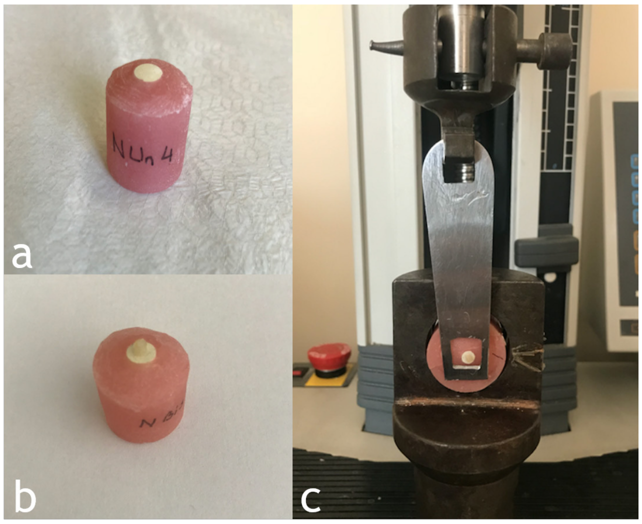

Figure 1. *a) Preparation of the samples, b) sample bonded with composite material, c)The universal testing machine*

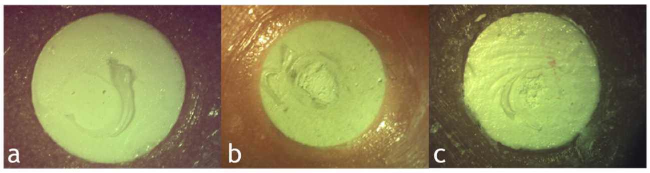

Figure 2. *Images of fractured samples showing: a) Adhesive failure in resin composite bonded to NeoMTA Plus b) Cohesive failure of resin composite bonded to ProRoot MTA c) Mixed failure in ProRoot MTA.*

2.1. Statistically analysis

A Shapiro-Wilk test confirmed a normal distribution of data. Micro-shear bond-strength data were statistically analyzed using two-way analysis of variance, and a chi-square test was used to analyse the distribution of failure modes. Statistical analysis was performed with the software SPSS v.21.0 (IBM, SPSS Inc., Chicago, IL, U.S.A.) with the level of significance set at 5%.

3. RESULTS

Mean micro-shear bond strength values and standard deviations of the groups are given in Table 2. No significant differences were observed in the micro-shear bond strengths of any of the adhesives tested to either NeoMTA Plus or ProRoot MTA ($p>0.05$). Moreover, all the tested adhesives showed similar bond-strengths to both of the calcium silicate-based biomaterials tested ($p>0.05$). Failure modes are given in Table 3. The majority of failures in the ProRoot MTA subgroups were cohesive failures, whereas both adhesive and cohesive failures were observed in the NeoMTA Plus subgroups.

Table 2. *Mean Shear Bond Strength Values (MPa) and Standard Deviations of Each Groups (n=10). Different superscript lower case letters in each row and capital letters in each column indicate indicate statistically significant differences (P<0,05)*

	NeoMTA Plus	ProRoot MTA	N
G-Premio	3.58 ± 0.64^{aA}	3.35 ± 0.53^{aA}	10
All-Bond Universal	2.71 ± 1.15^{aA}	3.09 ± 0.81^{aA}	10
Single Bond Universal	3.75 ± 1.12^{aA}	3.11 ± 0.41^{aA}	10

Table 3. *Distribution of Failure Modes within Groups (n=10)*

		Adhesive	Cohesive	Mixed
NeoMTA Plus	G-Premio	4	4	2
	All-Bond Universal	6	1	3
	Single Bond Universal	2	2	6
ProRoot MTA	G-Premio	-	6	4
	All-Bond Universal	2	2	6
	Single Bond Universal	2	7	1

4. DISCUSSION

The hypothesis of this study was accepted, because there was no difference in the micro-shear bond strength of UAs with different acidity levels to the calcium silicate-based biomaterials tested.

ProRoot MTA is the most commonly used and most studied brand of endodontic repair material and is used in surgical as well as non-surgical procedures. Composed mainly of tricalcium silicate (53.1%), dicalcium silicate (22.5%), bismuth oxide (21.6%), with small amounts of tricalcium aluminate and calcium sulfate (18), its thin, hydrophilic particles cure in a humid environment at 12.5 pH over a period of 3-4 hours (19). NeoMTA Plus is a new material composed of finely powdered tricalcium silicate that incorporates tantalum oxide (Ta_2O_5) rather than bismuth oxide as a radiopacifying agent to prevent discoloration and is mixed with a water-based gel to impart good processing properties (20). By changing the powder-gel ratio, NeoMTA Plus can be used in a variety of applications, with a thin mixture used in orthograde filling and a thicker mixture used in retrograde filling. According to the manufacturer, NeoMTA Plus can be used in vital pulp treatment (for pulp capping, pulpotomy, and as a cavity liner/base), root apexification, root repair (resorption and perforation), root-end filling, and root-canal sealing (21).

The adhesive properties of restorative materials are most commonly evaluated according to bond-strength, assessment of which has become a well-recognized method of analyzing material performance *in vitro*. In this study, the adhesive properties of tricalcium silicate-based materials and universal adhesives were evaluated by measuring shear bond strengths (10).

Due to its hydrophilic properties, ProRoot MTA requires moisture to initiate setting (22). For this reason, the application of a damp cotton pellet for 3-4 hours is recommended in order to supplement tissue fluids and provide two-sided hydration during perforation repair and pulp capping (6). A previous study reported this method to provide optimal results with a 24 h application, whereas double-hydration of ProRoot MTA for 72 hours resulted in a significant reduction in bond strength that the authors attributed to the potential negative impact of excessive humidity and possible solubility of the material (23). Therefore, in the present study, MTA was stored in 100% humidity for 24 h before bonding.

In order to simplify application procedures and reduce technical errors, one-step self-etch adhesives have been developed. Studies have noted that the acidic monomers used in self-etch adhesives play a key role in their enamel and dentin bonding performance (24). Generally, self-etch adhesives are classified as either “strong” (pH<1), “intermediately strong” (pH≈1.5), “mild” (pH≈2) and “ultra-mild” (pH≥2.5) (25), with the pH value known to strongly affect the solubility of the smear layer and the depth of demineralization of underlying dentin (26,27). Considering the differences in pH of the adhesives used in this study (G-Premio Bond: 1.5; Single Bond Universal:2.7; All-Bond Universal:3.2), the degree of biomaterial dissolution was expected to vary. Previous studies have stated that micromechanical retention and therefore bond strength increases with increases in surface porosity of tricalcium silicate based cements (28,29). Phosphoric acid etching has been suggested to provide greater microretention and potentially increases the bonding effectiveness of resinous materials by significantly enhancing the surface energy of substrate material (10). Moreover, Yelamali et al. have reported two-step etch-and-rinse and two-step self-etching primer systems to perform significantly better than an all-in-one adhesive system in terms of bond strength when bonding composite resin to white MTA (30). In contrast to these findings, the present study indicated differences in acidity among universal adhesives to have no significant effect on shear bond strentgh values, suggesting that microretention is not noticeably improved by lowering the acidity of self-etch adhesives.

Although the adhesives tested vary according to pH, they all contain the monomer 10-MDP, whose binding mechanism to dentin has been explored in a previous study. According to the authors, when an adhesive containing MDP is rubbed onto dentin, the surface is partially demineralized up to a submicron depth, and the MDP reacts with the released Ca ions to create nano-layers of MDP-Ca salts within the hybrid layer (31). In light of this earlier finding, it may be suggested that the bonds between the adhesives tested in the present study and both ProRoot MTA and NeoMTA are strengthened by chemical chelation of the Ca ions contained in these tricalcium silicate-based biomaterials (1).

However, despite the interaction between the MDP monomer and the calcium ions, the bond strengths of the adhesives to the tested cements were considerably lower than the threshold values of 17-20 MPa that are reportedly required to sufficiently resist contraction forces and produce gap-free restoration margins (32,33). It is likely that the bond between the cement and adhesives could be increased by using the same UAs in etch&rinse mode to increase porosity of the resin cement, thereby improving microretention.

With regard to fracture modes, according to Tate et al. (34) a bond is considered to be acceptable when fracture occurs within a material rather than at the bonded interface, i.e. when the fracture is cohesive rather than adhesive. In the present study, cohesive fracture predominated in the ProRoot MTA group.

5. CONCLUSION

The findings of the present study showed that the acidity of universal adhesives has no effect on the bond strength of tricalcium silicate-based biomaterials to restorative materials.

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