Shear bond strengths of different adhesive systems to white mineral trioxide aggregate

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This study was conducted to compare the shear bond strengths of different adhesive systems to White Mineral Trioxide Aggregate (WMTA). To this end, 40 cylindrical acrylic blocks with a hole in the middle measuring 4 mm diameter × 2 mm height were prepared. The hole was filled with WMTA, and the specimens were allocated into four groups: Group 1 − Prime & Bond NT; Group 2 − AdheSE; Group 3 − Xeno III; and Group 4 − Adper Prompt L-Pop. In each group, a different adhesive system and a compomer (Dyract AP) were applied over WMTA. Shear bond strengths were measured using a universal testing machine, and then the data were subjected to one-way ANOVA and Scheffé's *post hoc* test (p<0.05). Significantly lowest shear bond strength value was obtained with Adper Prompt L-Pop, whereas Prime & Bond NT revealed higher shear bond strength than the others (p<0.05). In conclusion, an etch-and-rinse adhesive system — which exhibited significantly higher shear bond strength than self-etch adhesive systems — could be a preferred choice when placing compomer materials upon WMTA.

Key words: WMTA, Shear bond strength, Bonding

INTRODUCTION

Mineral trioxide aggregate (MTA) is a mineral powder. It consists of fine hydrophilic particles that set in the presence of moisture[1]. MTA was first described in dental scientific literature in 1993[2] and was given approval for endodontic use by the U.S. Food and Drug Administration in 1998[3]. Up to 2002, only one MTA material consisting of gray-colored powder was available, and in the selfsame year white mineral trioxide aggregate (WMTA) was introduced as ProRoot MTA (Dentsply Tulsa Dental, Tulsa, OK, USA) to address esthetic concerns[4]. Both formulae are 75% Portland cement, 20% bismuth oxide, and 5% gypsum by weight[5,6].

MTA is designed to seal pathways of communication between the root canal system and the periodontium. It has been recommended as a material for use in perforation repair and retrograde filling[2]. Subsequently, it was recommended for pulp capping[7], pulpotomy[8], and as an apical barrier in treatment of immature teeth with nonvital pulps and open apices[9].

In pediatric dentistry, no area of treatment has been more controversial than pulp therapy. In particular, the vital pulpotomy procedure has been a topic of debate for decades[10]. Recently, MTA has been introduced as a potential alternative dressing material for pulpotomy of primary molars. This is chiefly because of its array of beneficial features: (1) able to stimulate cytokine release from bone cells, thereby inducing hard tissue formation; (2) have a dentinogenic effect on the pulp; (3) possess antimicrobial properties; and (4) able to maintain pulp integrity after pulp capping and pulpotomy without cytotoxic effects[1,8,11-17]. However, more studies are required to establish the effectiveness of restorations placed over MTA.

In a previous study, we studied the bond strengths of compomer and composite materials to MTA. It was shown that the total-etch adhesive system (Single Bond) bonded to MTA more strongly — with statistically significant difference — than one-step self-etch adhesive system (Prompt L-Pop) for both composite and compomer materials[18]. The main difference between the tested adhesive systems lies in their etching properties, whereby it has been speculated that two-step self-etch adhesives might have enhanced bond strength to MTA than one-step self-etch adhesives, as was shown in dentin-adhesive bonding tests[19-22]. In light of this speculative suggestion, the aim of the present study was to compare the shear bond strengths of different adhesive systems (one etch-and-rinse adhesive, one two-step self-etch adhesive, and two one-step self-etch adhesive systems) to White MTA (WMTA).

MATERIALS AND METHODS

Materials
Four commercial adhesive systems — Prime & Bond NT (PB), AdheSE (AS), Xeno III (XE), and Adper

Prompt L-Pop (LP) — were tested in this study and applied as recommended by the manufacturers. The materials used are listed in Table 1.

Specimen fabrication
Forty specimens of WMTA were prepared using cylindrical acrylic blocks. Each block had a hole in the middle measuring 4 mm in diameter and 2 mm in height. The acrylic blocks were filled with WMTA and covered with a wet cotton pellet and a temporary filling material (Cavit, ESPE America Inc., Norristown, PA, USA). Then, the specimens were stored at 37ºC with 100% humidity for 48 hours to encourage setting. After the removal of the temporary material, the WMTA surface was neither rinsed nor polished.

Specimens were divided into four groups of 10 specimens each. Group 1: PB (two-step etch-and-rinse adhesive); Group 2: AS (two-step self-etch adhesive); Group 3: XE (one-step self-etch adhesive); and Group 4: LP (one-step self-etch adhesive). In each group, the corresponding adhesive system was applied over WMTA according to the manufacturer's instructions (Table 1). Following which, a compomer material (Dyract AP, Dentsply DeTrey, Konstanz, Germany) was applied into a cylindrical-shaped plastic matrix with an internal diameter of 2 mm and a height of 2 mm. Light curing was administered

Table 1   Materials used in the study

| Material (manufacturer)	Composition	Mode/Steps of Application
Mineral Trioxide Aggregate (White MTA (Dentsply Tulsa Dental, USA))	Tricalcium silicate, bismuth oxide, dicalcium silicate, tricalcium aluminate, calcium sulfate dehydrate or gypsum	1. Mix powder/liquid ratio: 1/3.
Compomer (Dyract AP (Dentsply DeTrey, Konstanz, Germany))	UDMA resin/TCB resin, strontium and aluminum-fluorosilicate glass, strontium fluoride, photoinitiators/stabilizers	1. Light-polymerize 20 s.
Prime & Bond NT (Caulk/Dentsply International Inc., Milford, DE, USA)	Di-trimethacrylate resin, PENTA, functionalized amorphous silica, photoinitiators, stabilizers, cetylamine, hydrofluoride, acetone	1. Apply 35% phosphoric acid etchant 15 s. 2. Rinse and blot-dry. 3. Apply adhesive. 4. Gentle air stream. 5. Light-polymerize 10 s.
AdheSE (Ivoclar Vivadent, Schaan, Liechtenstein)	Primer Phosphoric acid acrylate, Bis-acrylamide, water, initiators and stabilizers Adhesive Dimethacrylate, hydroxyethyl methacrylate, highly dispersed silicon dioxide, initiators and stabilizers	1. Dry surface. 2. Apply primer. 3. Gentle air stream 4. Apply adhesive. 5. Gentle air stream. 6. Light-polymerize 10 s.
Xeno III (Dentsply DeTrey, Konstanz, Germany)	Liquid A 2-Hydroxyethyl methacrylate (HEMA), purified water, ethanol, butylated hydroxytoluene (BHT), highly dispersed silicon dioxide Liquid B Phosphoric acid modified polymethacrylate resin, mono fluoro phosphazene modified methacrylate resin, urethane dimethacrylate, butylated hydroxy toluene (BHT), camphorquinone, ethyl-4-dimethylaminobenzoate	1. Dry surface. 2. Mix liquids A and B. 3. Apply adhesive. 4. Gentle air stream. 5. Light-polymerize 10 s.
Adper Prompt L-Pop (3M ESPE, St. Paul, USA)	Red cushion: Methacrylic phosphates, BISGMA, photoinitiator Yellow cushion: water, HEMA, polyalkenoic acid polymer	1. Dry surface. 2. Apply adhesive. 3. Gentle air stream. 4. Light-polymerize 10 s.
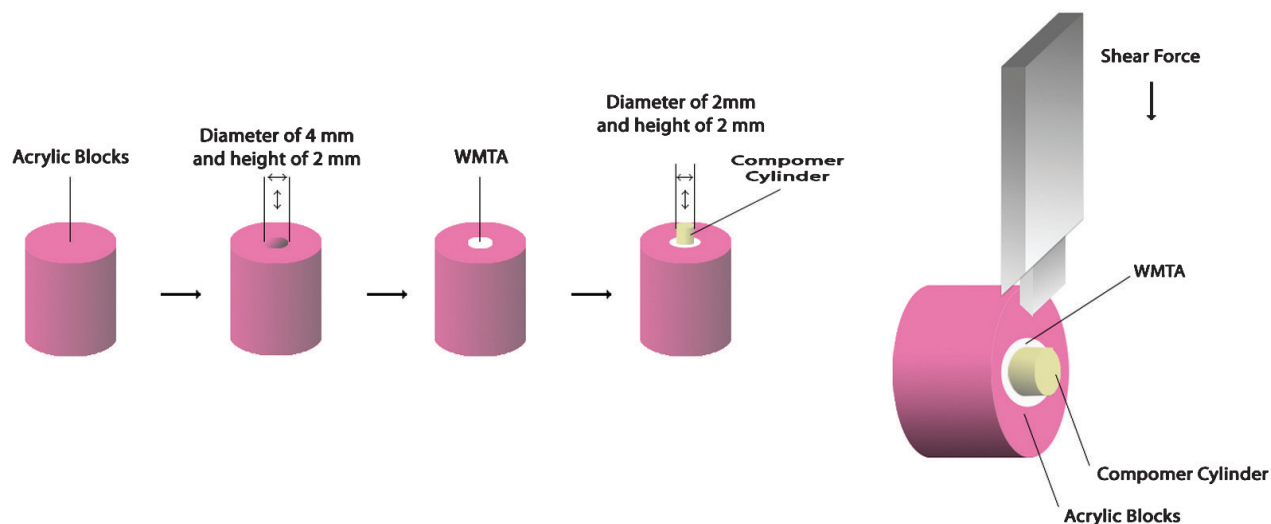
Fig. 1　Schematic illustrating the study design.

with a light-emitting diode light curing unit (Elipar FreeLight 2, 3M ESPE, St. Paul, MN, USA) with an intensity of 1200 mV/cm² for 20 seconds.

Shear bond strength test
The polymerized specimens were stored in 100% relative humidity at 37°C for 24 hours. For shear bond strength testing, the specimens were secured in a holder placed on the platen of the testing machine and then sheared with a knife-edge blade on a universal testing machine (Lloyd LRX, Lloyd Instruments, Fareham, Hants, UK) at a crosshead speed of 1.0 mm/min. Shear bond strength in MPa was calculated by dividing the peak load at failure with the specimen surface area (Fig. 1).

Fracture analysis
Fractured test specimens were examined under a stereomicroscope at a magnification of ×25 (Stemi 2000C, Carl Zeiss, Gottingen, Germany). Specimen fractures were classified as follows: CWMTA − cohesive failure exclusively within WMTA; CC − cohesive failure exclusively within compomer; A − adhesive failure that occurred at the WMTA-compomer interface; or M − mixed failure when two modes of failure happened simultaneously. Fracture analysis was performed by a single observer who was completely uninformed about the experimental groups.

Statistical analysis
One-way analysis of variance (ANOVA) was used to detect significant differences (p≤0.05) in bond strength among the experimental groups. *Post hoc* comparisons were made using the Scheffé's test.

Table 2　Mean shear bond strength values of adhesives (MPa) to WMTA

Groups	n	Mean±SD
Group 1 (PB)	10	23.44±1.81[a]
Group 2 (AS)	10	13.67±1.96[b]
Group 3 (XE)	10	16.47±4.68[b]
Group 4 (LP)	10	5.44±0.86[c]

Different superscript letters indicate significant differences by one-way ANOVA and *post hoc* Scheffé's test (p<0.05).

RESULTS

Table 2 shows the descriptive statistics of shear bond strength for each group. Analysis of variance that compared the experimental groups revealed the presence of significant differences among the groups (p<0.05). Scheffé's *post hoc* test indicated that Group 1 (PB) showed significantly higher bond strength to WMTA (p<0.05) than all the other groups. Group 2 (AS) and Group 3 (XE) were ranked in the intermediary subset, while Group 4 (LP) exhibited the lowest shear bond strength (p<0.05).

Table 3 shows the fracture modes of the experimental groups. Most of the observed modes of failure were adhesive except for specimens bonded with PB. For the latter specimens, it was predominantly cohesive failure. It is noteworthy that majority of adhesive failures were associated with lower bond strengths.

Table 3 Fracture modes of the specimens after shear bond test

Groups	n	Adhesive (A)	Mixed (M)	Cohesive in WMTA (CWMTA)	Cohesive in compomer (CC)
Group 1 (PB)	10	4	−	6	−
Group 2 (AS)	10	7	1	2	−
Group 3 (XE)	10	7	−	3	−
Group 4 (LP)	10	9	−	1	−

DISCUSSION

The diverse uses of MTA in a variety of surgical and non-surgical endodontic applications have been widely reported[1,3,8,9,17,23]. A particularly useful clinical application is that resin-based restorative materials can be applied directly on set MTA[1]. This is especially important in pediatric dentistry because of reduced clinical steps and application time. However, information is scarce on the adhesion of resin-based restorative materials to MTA.

On the quality of fillings, the bond strength between two restorative materials is of paramount importance. It has been estimated that a bond strength ranging from 17 to 20 MPa may be required to resist contraction forces sufficiently to produce gap-free restoration margins[24,25]. In the context of the present study, only the shear bond strength result attained with PB exceeded this value range.

In other words, this study showed that the shear bond strengths of self-etch adhesives were lower than that of etch-and-rinse adhesive. This finding was in agreement with those of previous studies[19,22,26]. Several reasons have been advocated to account for the suboptimal performance of self-etch adhesives: (1) the combination of acidic hydrophilic and hydrophobic monomers into a single step may compromise the polymerization of the adhesive; (2) the inherently low strength of the adhesive polymer; and (3) the lower degree of polymerization of the resin monomer due to a major solvent/oxygen inhibition effect during light activation of these materials[27].

Amongst the self-etch adhesives, the results of the present study revealed that AS (a two-step self-etch adhesive) exhibited higher shear bond strength than LP (a one-step self-etch adhesive). Similarly, this result was in agreement with those of previous studies which found that the bond strengths of two-step self-etch adhesives were significantly higher than those of one-step self-etch adhesives[19-22]. However, XE was a one-step self-etch adhesive and its shear bond strength was higher than that of LP, but not significantly different from AS. The large disparity in bonding performance among the different self-etch adhesives can in part be ascribed to the influence of the pH value, the influence of the solvent, and the influence of filled/unfilled adhesives[28].

pH of self-etch adhesives: LP and XE (pH<1) are classified as strong self-etch adhesives, whereas AS (pH≈1.5) is considered to be a medium-strong self-etch adhesive[29,30]. Typically, low-pH self-etch adhesives have been documented with relatively lower bond strength values[22]. Within the limitations of the present study, no correlation was observed between the pH of the adhesives and their shear bond strength values. Therefore, it was thought that the nature of the solvent and the filler content of the adhesive might have a greater influence on shear bond strength values than the pH of the adhesive.

Nature of the solvent: The shear bond strength result obtained with XE could be explained by the nature of its solvent. XE contained water and ethanol, whereas the other self-etch adhesives contained water only. According to Jacobsen and Söderholm[31], bonding systems based on water may result in lower bond strength due to incomplete polymerization of the monomers. Furthermore, the water content of WMTA could have interfered with the polymerization of the self-etch adhesives, thereby resulting in reduced WMTA-adhesive bond strength values.

Influence of filled/unfilled adhesives: The filled adhesive systems (AS and XE)[30,32] tested in this study resulted in statistically higher bond strengths to WMTA than the unfilled adhesive system (LP)[30,33]. Filled, low-viscosity resins are thought to have sufficient strain capacity to relieve stresses between the shrinking resin-based restoration and the rigid substrate[20]. As for means to improve bonding between unfilled adhesives and the rigid tooth substrate, Pashley *et al.*[33] suggested applying a second layer of the unfilled adhesive after light-curing the first layer.

In the present study, the most common mode of fracture was adhesive failure at the WMTA-compomer interface. This result indicated that a strong chemical bond was not formed at the interface for many of the specimens in this study. In contrast,

where a stronger bond existed between the compomer material and WMTA, a higher percentage of cohesive failure was yielded within that experimental group.

Currently, little is known about the effects of acid-etching procedures on WMTA. Nonetheless, when WMTA becomes hard and strong enough to be used as a restoration material, etch-and-rinse adhesive systems should be the preferred choice. Furthermore, to overcome any drawbacks associated with acid etching, the new type of light-cure MTA may be used as pulpotomy material. For more insights and a better understanding of the adhesion mechanism of adhesive systems to WMTA, further investigations are certainly needed.

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