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Shear Bond Strength of MTA with Different Types of Adhesive Systems: An *In vitro* Study

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Abstract: ***Aim**: To evaluate the shear bond strength of MTA with three different types of adhesive systems- self-adhering flowable composite, etch and rinse adhesive system and self etch adhesive system. **Material & Method**: MTA specimens (n= 60) were prepared using cylindrical acrylic blocks, having a central cavity with 4 mm diameter and 2 mm depth. MTA was mixed and placed in the prepared cavity, and was covered with a moist cotton pellet and temporary filling material. The specimens were divided into 3 groups which were further divided into 2 sub -groups (45 Minutes and 24 hours). After the application of bonding agents composite resin was placed over the MTA surface. The specimens were tested for shear bond strength and readings were statically analyzed. **Result**: After 24 hrs the mean value of etch and rinse group was significantly higher than self etch and the self adhering composite groups. Among the 45 minutes groups there were no significant difference. **Conclusion**: In single visit after 45 minutes self adhering flowable can be used successfully as a final restorative material in place of conventional flowable composite without using any alternative adhesive system over MTA.*

Keywords: Etch and rinse (ER) adhesive; mineral trioxide aggregate (MTA); self-adhering flowable composite; self-etch (SE) adhesive

1. Introduction

Mineral trioxide aggregate (MTA) was introduced in 1993 and since then it has become a gold standard for many endodontic procedures. MTA is a biocompatible material [1,2] and has excellent sealing ability. It is claimed to provide double seal due to its physical sealing ability as well as ability to induce cementogenesis that provides biological seal. MTA has ability to set in the presence of moisture. Despite several advantages, prolonged setting time, and compulsion to place wet cotton pellet over MTA in some procedures to complete the setting reaction is a major drawback that necessitates second visit and increased chair time.[3]

Conventional glass ionomer cement (GIC) can be layered over partially set MTA after 45 min to complete the procedure in a single visit without any deleterious effect on either of the material.[4,5] Atabek *et al*. concluded that restorative procedure might be postponed at least for 96 h after mixing MTA to allow the material to achieve its optimum physical properties.[6]

Some studies also suggested that composite resin with a bonding agent can be restored over MTA almost immediately after the placement of MTA. When hydration during MTA setting process is guaranteed, there are no particular problems related to composite resin restoration even if it is performed almost immediately after MTA filling.[7] However, acid etching before composite placement reduces the compressive strength and surface microhardness of MTA. Kayhan *et al.* observed that acid etching created surface changes that might have potential to enhance bonding of resinous materials.[8] The nature of the solvent (acetone, ethanol, or water) and the filler content of the adhesive may also influence the bond strength of MTA to resin.[9]

Dyad flow, a new generation flowable composite, is recently introduced in restorative dentistry; this self-adhering composite is designed to bond to tooth structure without the need for a separate adhesive or an etching step.[10] The reduction in the number of steps can lead to lesser chances of procedural errors and lesser chair side time and results in more patient comfort during placement and hence are less technique sensitive than total etch and self-etch (SE) adhesives.[11] The bonding mechanism of self-adhering composite relies on a monomer glycerol phosphate dimethacrylate (GPDM) adhesive. Dyad flow bonds in two ways as follows: Primarily, through the chemical bond between the phosphate functional groups of a GPDM monomer and calcium ions of the tooth and, secondarily, through a micromechanical bond as a result of an interpenetrating network formed between the polymerized monomers of dyad flow and collagen fibers of dentin.[10]

Studies evaluating the physical properties, bond strengths, and marginal leakage of self-adhering composite to tooth have been reported, but no study has compared the bond strength of self-adhering composite with SE and etch and rinse (ER) adhesive systems when placed over MTA. The aim of this study is to evaluate the shear bond strength of

MTA with three different types of adhesive systems, a self-adhering flowable composite, a SE adhesive system, and an ER adhesive system.

2. Materials and Methods

The materials used in this study were tested and applied as recommended by the manufacturers. The materials used are listed (in Table 1) 4 mm in diameter and 2 mm in depth. The MTA was mixed according to the manufacturer's instructions, placed into the central cavity in the acrylic blocks, and covered with a moist cotton pellet and temporary filling material (Cavit; 3M ESPE America, Norristown, PA). Half of the specimens were stored for 45 min, and the remaining 30 specimens were stored for 24 h at 37°C temperature and 100% humidity.

After removing the temporary material and moist cotton, the MTA surface was not rinsed or polished. Sixty samples were divided into three groups according to the type of adhesive system used and these groups were further divided into two subgroups (N = 10) according to the different time intervals. The divisions of groups are listed in Table 2.

In the SE and ER groups, the corresponding adhesive system was applied over MTA according to the manufacturer's instruction following which a conventional flowable composite Brilliant Flow (Coltene Whaledent, Switzerland) was placed over MTA surface with the help of cylindrical plastic tube having an internal diameter of 2 mm and a height of 3 mm. Light curing was done with a quartz-tungsten-halogen (QTH) light curing unit (Spectrum 800, DENTSPLY Caulk Milford, DE, USA) at an intensity of 600 mW/cm2 for 20 s.

In the DF Group, the self-adhering composite was placed over MTA in increments. A layer of 1.5 mm was first placed inside the cylindrical plastic tube and agitated over the MTA surface with the help of an application brush. Light curing was done using a QTH light curing unit (Spectrum 800, DENTSPLY Caulk Milford, DE, USA) at an intensity of 600 mW/cm2 for 20 s and then the remaining height of the plastic tube was filled in the second pour and light curing was done.

Preparation of MTA specimens

Table 1: Materials used, composition and method of application

Product name (manufacturer)	Composition	Application
ProRoot MTA (Dentsply Tulsa	Tricalcium silicate, bismuth oxide, dicalcium silicate, tricalcium	Mix MTA with distilled water according to
Dental, USA)	aluminate, calcium sulfate dehydrate or gypsum	manufacturer's instruction
Dyad Flow (DF) (Kerr, Orange, CA, USA)	GPDM, HEMA, prepolymerized filler (20 µm),	Brush a thin layer (<1.5 mm) of dyad flow for
	Barium glass filler (0.7-1 µm), nano-sized colloidal silica (10-40 nm), nano-sized Ytterbium fluoride (40 nm), zinc oxide (pH 1.9)	15-20 s and light cured for 20 s and subsequent layer added
Brilliant flow (Coltene	Methacrylates, barium glass, silanized amorphous silica,	Apply the material using applicator tip and
Whaledent, Switzerland)	hydrophobic	light cured
Prime and bond NT (Dentsply,	Di-trimethacrylate resin, PENTA, functionalized amorphous silica,	Apply 35% phosphoric acid etchant for 15 s.
Konstanz, Germany)	photoinitiator, stabilizers, cetylamine hydrofluoride, acetone	Rinse and blot dry. Apply adhesive. Gentle air stream. Light polymerize for 20 s.
All bond 7 (Coltene	Primer: MDP, HEMA, hydrophilic dimethacrylate, DL-	Apply, wait for 20 s, light cure the bond for
Whaledent, Switzerland)	CAMPHORQUINONE, water Bond: MDP, HEMA, BIS-GMA	10 s

Sixty MTA specimens were prepared by using cylindrical acrylic blocks. Each block had a central cavity measuring

The plastic tubes were removed carefully and the specimens were stored at 37°C temperature and 100% humidity for 24 h to encourage setting.

Shear bond strength measurement

The specimens were mounted in a universal testing machine (Instron Corp, Canton, MA). A crosshead speed of 0.5 mm/ min was applied to each specimen by using a knife-edge blade until the bond between the MTA and composite failed. The values were calculated in newtons and converted into megapascals (MPa) by using formula 4 × *F*/□d2. Here, "*F*" is the force in newton and "*d*" is the diameter of composite block. The means and standard deviations were calculated. The mean bond strengths of the groups were compared by using one-way analysis of variance (ANOVA) and Tukey's *post hoc* test (significance level, $P < .05$).

Table 2: Different groups at different time intervals for bond strength test

Group 1: Dyad flow	Group 2: Self-etch	Group 3: Etch and rinse
45 min	45 min	45 min
24 h	24 h	24 h

3. Results

The means and standard deviations of the shear bond strengths are given in Table 3. There was no significant difference between dyad flow and SE groups at both the time intervals. However, there is a significant difference between the mean bond strengths at 45 min and 24 h for the ER group, when compared with SE and dyad flow groups.

4. Discussion

MTA have been used successfully as a material for use in perforation repair and retrograde filling and eventually, it was recommended for pulp capping, pulpotomy, and as an apical barrier in the treatment of immature teeth with nonvital pulps and open apices as pulp capping agents in vital pulp therapy.[12,13] Following pulp capping, direct resin composites can be used for final restoration.[14] However, the use of SE adhesive or etching procedure over MTA surface has been reported to reduce microhardness, with the selective loss of matrix from around the crystalline structures on the MTA surface. Investigators have recommended that the application of ER and SE adhesive over MTA be postponed for 96 h[8] and that low condensation forces be used for coronal restoration over MTA[15] because of the material's low initial compressive strength. Flowable composite can be used without compression forces, and the new FC (dyad flow) does not require surface pretreatment before bonding because it acts as both an adhesive and a composite.[9]

The most common method to evaluate adhesive properties of restorative materials is bond strength assessment.

Table 3: Mean shear bond strength values in MPa

Time intervals	Dyad flow	Self-etch	Etch and rinse
45 min	3.4±1.17 MPa	3.8±1.25 MPa	5.2±1.54 MPa
24 h	4.2±1.32 MPa	4.8±0.98 MPa	7.3±1.49 MPa

This has become a well recognized method to analyze an important part of the *in vitro* performance of materials.[16] So the shear bond strength test has been used in this study to evaluate the adhesive properties of MTA with a self-adhering composite (dyad flow), a SE adhesive, and an ER adhesive. The mean bond strength of dyad flow, —SE, and ER groups is lesser at 45 min in comparison with 24 h interval and statistically there is no significant difference between dyad flow, SE and ER groups at 45 min (P = 0.875, 0.753, and 0.943, respectively). However, there is a significant difference between the mean bond strengths at 45 min and 24 h for the ER group, when compared with SE and dyad flow groups whereas there is no significant difference in the mean bond strengths between the dyad flow and SE groups at 24 h. (at 45 min P = 0.038 and at 24 h P = 0.001).

Recent studies evaluating the bond strength of adhesives to MTA, using various bonding systems, concluded that superior MTA-composite bond strength can be achieved with an ER adhesives at 24 h in comparison to SE system.[6,8]

SE adhesives are gaining popularity because of simplified bonding procedures and reduced technique sensitivity. The SE approach uses acidic adhesive monomers that simultaneously demineralize and infiltrate into the dentin. The intensity of the interaction of SE adhesive systems with dentin was mostly dependent on the acidity and aggressiveness of the primer used.[17] SE adhesives have been classified based on their ability to penetrate smear layers and depth of demineralization as ultra-mild (pH >2.5), mild (pH ~2), moderately strong (pH 1-2), and strong (pH ≤1). The manufacturer of dyad flow has declared that it has a pH of 1.9, and One Coat 7.0 (Coltene Whaledent, Switzerland), a mild SE adhesive, has a reported pH of 2.0. Thus, dyad flow can be expected to interact with the dental substrate in a manner similar to that of a mild SE adhesive.[18] The results of this study showed that there is no significant difference between the bond strength of dyad flow, SE adhesive, and ER adhesive after 45 min. However, the bond strength of the composite to MTA was significantly higher for the ER group after 24 h.

Previous studies have evaluated the bond strengths of adhesives to MTA using various bonding system. Although acid etching reduces the surface microhardness of MTA and weakens the structure of the material, the results of these studies have shown that superior MTA/composite bond strength can be achieved with ER adhesives in comparison with one-step SE systems.[6] Phosphoric acid etching significantly enhances the surface energy of the substrate, thereby provides significantly more microretention and potentially increases the bonding effectiveness of resinous materials.[8] However, according to this study, there is no significance difference between dyad flow, —SE, and ER group at 45 min. The mild etching efficacy of dyad flow (pH 1.9) in comparison to aggressive phosphoric acid in ER group might be a reason for lower bond strength value at 24 h. Dyad flow and SE adhesive have pH of 1.9 and 2.0, respectively, and due to similar etching effectiveness the bond strength values are almost same.

5. Conclusion

Within the limitation of this *in vitro* study, it can be concluded that there is no significant difference in the mean bond strength values for dyad flow, — SE, and ER groups at 45 min. However, there is a significant difference in the mean bond strength values for ER adhesives when compared to dyad flow and SE adhesives at 24 h. Thus, to complete the clinical procedure in a single visit and to reduce the clinical steps, dyad flow can be used as an alternative to SE or ER adhesives as a definitive restoration after 45 min.

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