

Original Article

The comparative evaluation of shear bond strength of a bioactive material to different universal bonding agents – An *in vitro* study

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Abstract

Background: An ideal dental repair material should possess certain important properties such as adequate adhesive ability, insolubility, dimensional stability, biocompatibility, and bioactivity. Newer materials claiming better performance are continuously being introduced in the market to optimize the care of dental patients.

Aim: The aim of this study was to evaluate the shear bond strength of three different universal adhesives to OrthoMTA.

Materials and Methods: Sixty-four specimens of OrthoMTA measuring 4 mm internal diameter and 2 mm height were prepared and divided into two main groups. After 12 min, 32 samples were randomly selected and divided into four subgroups of eight samples each. Subgroup-I: Single Bond Universal, Subgroup-II: Prime-and-Bond NT, Subgroup-III: Palfique Universal bond, Subgroup-IV: Control. After the application of adhesives, the composite resin was applied using a cylindrical plastic matrix of 2 mm internal diameter and 2 mm height over OrthoMTA. This procedure was repeated 24 h after mixing an additional 32 samples, respectively. Shear bond strengths were measured using Universal testing machine and fractured specimen were examined under stereomicroscope. Data were statistically analyzed using a two-way ANOVA test and Tukey's multiple post hoc test.

Results: Statistical analysis revealed that subgroup-III exhibited higher bond strength at both 12 min and 24 h time intervals. It was also observed that most of the failures occurred cohesively within OrthoMTA.

Conclusion: Shear bond strength was higher at 24 h than compared to 12 min. Subgroup-III exhibited higher bond strength than other subgroups.

Keywords: Fracture analysis; OrthoMTA; shear bond strength; stereomicroscope; universal bonding agents; universal testing machine

INTRODUCTION

Mineral trioxide aggregate (MTA), an ideal material for perforation repair, pulp capping, apexification, obturation, and root-end filling material, was introduced

in 1993 by Torabinejad.^[1] Despite various advantages, MTA exhibits some limitations such as extended setting time, difficult handling properties, and discoloration of hard tissues.^[2,3] To overcome these drawbacks, recently, a new tricalcium silicate-based restorative material was introduced by BioMTA in Seoul, South Korea. OrthoMTA is composed of 76.3% of tricalcium silicate, 11.8% of dicalcium silicate, 8% of tricalcium aluminate, 0.8% of tetracalcium aluminoferrite, 0.7% of free calcium oxide. Manufacturers claim that OrthoMTA is the first

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orthograde root canal grafting material. The main advantage of OrthoMTA over MTA is reduced setting time and better sealing ability.

OrthoMTA has gained popularity as the Portland cement present in MTA and is replaced by a new generation of nanomaterials that eliminate the toxic compounds and heavy metals from the composition of MTA.^[4] OrthoMTA is a good bioactive material. Its bioactive nature is brought about by the dissolution of calcium, which then forms a complex with phosphate to form hydroxyapatite crystals that grow and fill the space between MTA and dentin, which induces entombing effect. A study by Kum *et al.* showed that both ProRoot MTA and OrthoMTA had equally favorable biocompatibility.^[5]

OrthoMTA is recommended as an alternative pulp capping agent for vital pulp therapy. While the use of OrthoMTA in vital pulp therapy has gained popularity, what to place over MTA as a permanent restorative material has become a crucial issue. However, the adhesion of restorative materials to MTA has not been studied extensively, and thus, it is not very well-known. The purpose of the study was to evaluate the shear bond strength of three different universal adhesives to OrthoMTA and also to evaluate the fracture modes of the specimen after the shear bond strength test. This study was begun by considering the null hypothesis stating that there is no significant difference between any groups and subgroups.

MATERIALS AND METHODS

Three different universal adhesive systems such as Single Bond Universal (3M), Prime and Bond NT (Dentsply) and Palfique Universal Bond (Tokuyama) were tested in the study and were applied according to the manufacturer's instructions. The materials used are listed in Table 1.

Specimen fabrication

A total of 64 holes were prepared on polycarbonate sheet with an internal diameter of 4 mm and a height of 2 mm.

Table 1: List of materials and their compositions

Materials	Composition
Single bond Universal (3M ESPE, USA)	MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylate modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane.
Prime and Bond NT (Dentsply Sirona, USA)	Di-and trimethacrylate resin, PENTA, functionalized amorphous silica, photoinitiators, stabilizers, cetylamine, hydrofluoride, and acetone.
Palfique Universal bond (Tokuyama, Japan)	Bond A - Phosphoric acid monomer (New 3D-SR monomer), MTU-6, HEMA, Bis-GMA, TEGDMA, acetone Bond B - γ-MPTES, borate, peroxide, acetone, isopropyl alcohol, water
Estelite Posterior (Tokuyama, Japan)	Silica-zirconia filler (84%), Bis-GMA (1%-10%), Triethylene glycol dimethacrylate (1%-10%), Bisphenol A polyethoxy methacrylate (1%-10%), camphorquinone (<1%)

OrthoMTA was mixed according to the manufacturer's instructions. The polycarbonate blocks were fully filled with OrthoMTA [Figure 1]. Then, the specimens were divided into two groups of 32 samples each. One group was stored at 37°C with 100% humidity for 12 min and the other group for 24 h to encourage setting. After 12 min, 32 samples were randomly selected and divided into four subgroups of eight samples each:

- Subgroup 1: Single bond Universal (3M)
- Subgroup 2: Prime and Bond NT (Dentsply)
- Subgroup 3: Palfique Universal bond (Tokuyama)
- Subgroup 4: Control (no adhesive)

In subgroups 1, 2, and 3, the corresponding adhesive systems were applied over OrthoMTA according to the manufacturer's instructions [Figure 2] and were light-cured according to manufacturer's instructions, whereas in subgroup 4, no adhesive system was applied.

A composite material (Estelite posterior, Tokuyama) was applied into a cylindrical shaped plastic matrix with an internal diameter of 2 mm and a height of 2 mm and were light-cured according to the manufacturer's instructions. The same procedure was repeated at 24 h after mixing an additional 32 samples, respectively.

Shear bond strength test

The polymerized specimens were stored in 100% relative humidity at 37°C for 24 h. 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 the junction of OrthoMTA and composite resin 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.



Figure 1: Polycarbonate blocks filled with OrthoMTA

Fracture analysis

Fractured test specimens were examined under a stereomicroscope at a magnification of $\times 25$ (Stemi 2000C: CarlZeiss, Gottingen, Germany). Specimen fractures were classified as follows: Cohesive failure exclusively within OrthoMTA, cohesive failure exclusively within restorative material, the adhesive failure that occurred at the OrthoMTA restorative material interface or mixed failure when two modes of failure happened simultaneously.

Statistical analysis

The mean shear bond strength of specimens was statistically analyzed using a two-way ANOVA test and Tukey’s multiple *post hoc* test.

RESULTS

The mean values and standard deviations of shear bond strengths are given in Table 2 and show that among the two-time intervals, bond strength after 24 h was significantly >12 min time interval. Furthermore, the bond strength of subgroup 3 was significantly higher than other groups at both time intervals. Table 3 shows that when shear bond strength of the adhesive systems were compared between the groups and subgroups using a two-way ANOVA test, it was found that there were significant differences. Table 4 shows that there were significant differences between the groups and subgroups when Tukey’s multiple *post hoc* test was applied between the groups and subgroups. Table 5 depicts that most of the observed modes of failure in the test groups were cohesive in OrthoMTA and adhesive

failure. None of the specimens failed cohesively within the composite resin.

DISCUSSION

Since OrthoMTA is recommended for use as a dentine substitute under restorations, the bond strength between restorative materials and OrthoMTA is important for the quality of restoration. In this study, the bond strength of a resin composite when bonded to OrthoMTA with 3 different universal adhesive systems was evaluated at 2-time intervals (12 min and 24 h). We found that the mean bond strength values ranged from 4.70 MPa to 23.79 MPa. The lowest value was obtained for subgroup 2 at 12 min, and the highest value was obtained to subgroup 3 at 24 h period. Failure analysis showed adhesive, cohesive, and/or mixed fractures, depending on the different adhesives tested. In this study, a general trend was observed; specimens that presented with lower bond strength failed more at composite resin and OrthoMTA interface (adhesive). On the other hand, specimens with higher bond strength failed more cohesively in OrthoMTA.

The highest bond strength value obtained in subgroup 3 might be because of the new three dimensional self-reinforcing (3D-SR) monomers and MTU-6 components present in Bond A and also y-MPTES present in Bond B,

Table 2: Summary of shear bond strength in two main groups (24 h and 12 min) and four sub Groups (1, 2, 3, 4)

Groups	n	Mean	SD	SE	CV
24 h in sub Group 1	8	17.31	5.75	2.03	33.21
24 h in sub Group 2	8	14.16	3.64	1.29	25.71
24 h in sub Group 3	8	23.79	2.99	1.06	12.57
24 h in sub Group 4	8	2.24	0.75	0.27	33.42
12 min in sub Group 1	8	15.69	2.93	1.03	18.66
12 min in sub Group 2	8	4.70	1.40	0.49	29.67
12 min in sub Group 3	8	22.31	2.18	0.77	9.76
12 min in sub Group 4	8	1.86	0.48	0.17	25.88

SD: Standard deviation, SE: Standard error, CV: Coefficient of variation

Table 3: Comparison of two main groups (24 h and 12 min) and four sub Groups (1, 2, 3, 4) with shear bond strength by two way analysis of variance

Sources of variation	Degrees of freedom	Sum of squares	Mean sum of squares	F	P
Main effects					
Main groups	1	167.73	167.73	18.8234	0.0001*
Sub groups	3	3929.46	1309.82	146.9898	0.0001*
2-way interaction effects					
Main groups \times sub groups	3	209.90	69.97	7.8517	0.0002*
Error	56	499.01	8.91		
Total	63	4806.11			

*P<0.05

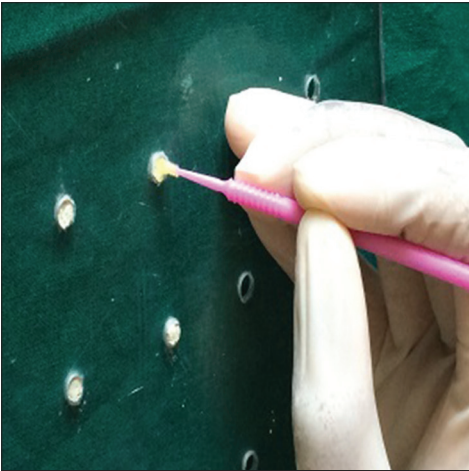


Figure 2: Adhesive system applied over OrthoMTA

Table 4: Comparison of four sub Groups (1, 2, 3, 4) with shear bond strength by Tukeys multiple *post hoc* procedures

Groups	Sub Group 1	Sub Group 2	Sub Group 3	Sub Group 4
Mean	16.50	9.43	23.05	2.05
SD	4.49	5.56	2.64	0.64
Sub Group 1	-			
Sub Group 2	$P=0.0002^*$	-		
Sub Group 3	$P=0.0002^*$	$P=0.0002^*$	-	
Sub Group 4	$P=0.0002^*$	$P=0.0002^*$	$P=0.0002^*$	-

* $P<0.05$. SD: Standard deviation**Table 5: Fracture modes of the specimens after shear bond strength test**

	Total	Single bond universal		Prime n bond NT		Universal bond		Control	
		12 min	24 h	12 min	24 h	12 min	24 h	12 min	24 h
Adhesive	25	1	1	2	2	2	1	8	8
Mixed	14	2	1	3	2	3	3	-	-
Cohesive in OrthoMTA	25	5	6	3	4	3	4	-	-
Cohesive in composite resin	0	-	-	-	-	-	-	-	-

which are unique in Palfique Universal Bond. Furthermore, both Single Bond Universal and Palfique Universal Bond contain HEMA as one of its components, whereas Prime and Bond NT contain PENTA.

The main constituent in MTA is tricalcium silicate, which is used as an endodontic material^[6] and bone cement.^[7] Tricalcium silicate cement has been found to have shorter setting time, good injectability, and bioactivity.^[8] One such formulation is OrthoMTA (BioMTA), which was recently developed as dentin replacement material. There are no studies evaluating the bond strength of restorative materials when bonded to OrthoMTA with adhesive systems for the purpose of outcome comparison. However, the main component of MTA is tricalcium silicate;^[9] the outcomes of this study could be compared with earlier studies about MTA.

In a study, the shear bond strength of universal adhesive systems to Biodentine was studied,^[10] and they found that mean bond strength was significantly less at 12 min time interval than compared to 24, 48, and 72 h.

One-step self-etch adhesive also called “all-in-one” adhesive, which contains an acid, primer, and adhesive components in one solution, allows one-step application only. Over the last few years, these adhesive systems have become increasingly popular.^[11-13] Furthermore, some studies exhibited less sensitivity with self-etch adhesives, as etch and rinse step is eliminated.^[14] Thus, a new type of single-step self-etch adhesive, categorized as “universal” or “multi-mode” has been recently introduced for patient care. These adhesive systems are recommended by dental manufacturers for use both with and without acid pretreatment of enamel surfaces.

The effect of acid etch on the surface morphology of angelus MTA and TheraCal LC was studied;^[15] it was found that there was the selective loss of surface matrix after

acid application, forming increased surface porosity and micropores with the removal of cement particles. They also found that one-step self-etch adhesives showed higher bond strength compared to the other two-step self-etch adhesives and etch and rinse bonding techniques. Shin *et al.*^[13] studied the effect of MTA surface treatments on the morphology and bond strength to composite resin. They found that acidic treatment of the MTA surface affected the micromorphology and the bond strength to composite. They also found that one-step self-etch adhesive system had stronger bond strength to White MTA.

Neelakantan *et al.*^[16] found that one-step self-etch adhesive demonstrated higher bond strength to white MTA than did the two-step self-etch adhesive and the etch-and-rinse adhesive systems immediately and 24 h after fabrication. Savadi Oskoei *et al.*^[17] studied Shear Bond Strength of Calcium Enriched Mixture Cement and MTA to Composite Resin with Two Different Adhesive Systems. They found that one-step self-etch adhesives showed higher bond strength to CEM and MTA than other adhesive systems.

CONCLUSION

Under the limitations of the study, the Shear bond strength was significantly higher at 24 h than compared to 12 min time interval. Subgroup 3 (i.e., Palfique Universal bond by Tokuyama) showed higher bond strength at both 12 min and 24-h time intervals. Hence null hypothesis is rejected. Thus, to complete the clinical procedure in a single visit, Universal Bond by Tokuyama can be applied under composite restorations.

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Conflicts of interest

There are no conflicts of interest.

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