



## **Push-out bond strength and apical microleakage of (MTA Plus, Biodentine, and Bioceramic) as apical third filling (An in vitro study)**

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### **Abstract**

**Background:** This study was conducted to evaluate the sealing ability of (MTA Plus, Biodentine Bioceramic root repair material) as an apical third filling by using (Push-out bond strength test, apical microleakage test).

**Materials and methods:** Sixty straight palatal roots of the maxillary first molars teeth were used in this study, the roots were instrumented by using crown down technique with Protaper universal rotary system, the roots were randomly divided into three groups according to the materials used for filling apical third (n=20). Group (1): MTA Plus . Group (2): Biodentine. Group (3): Bioceramic repair material. For all groups complete obturation with easy & quick obturation system was used. After incubation period of three days ten roots for each group were embedded in clear acrylic resin and each root sectioned in apical to provide slice 2mm in thickness. The bond strength was measured using computerized universal testing machine. Ten roots remain from each group used for apical microleakage study. The roots submerged in 2% methylene blue for three days. The roots were cleared and the degree of linear dye penetration was measured in millimeter by stereomicroscope under 40 X magnification with calibrated scale ocular grid. The data were analyzed statistically using ANOVA and LSD test.

**Results:** In push-out bond strength showed the Biodentine has the highest mean values (19.687) in comparison with other groups followed by MTA Plus group which the mean value was (19.395), while the BC material group has the lowest mean value (10.977). In microleakage the BC material group has the high mean values (0.477) of apical dye penetration in comparison with other groups. Biodentine group has lowest mean values (0.359) of apical dye penetration.

**Conclusions:** The Biodentine higher push-out bond strength and less apical microleakage then other test materials.

**Keywords:** MTA Plus, Biodentine, Bioceramic, push-out bond strength, apical microleakage.

### **Introduction**

Beside proper cleaning and shaping of the root canal, the complete and hermetic obturation of the root canal system is a major objective in root-

canal treatment <sup>(1)</sup>. Sealing generally includes the use of semi-solid material (gutta percha) and sealing cement; the gutta percha serves as the core

material, whereas the root canal sealer is required to adhere to dentin and fill the discrepancies between the core-filling material and the dentinal wall. When teeth with incomplete root formation undergo pulp necrosis, dentin formation is interrupted and root development ceases. Consequently, the root canal is large, with thin and fragile walls, and the apex remains open. These features impair root canal instrumentation and prevent the achievement of an adequate apical stop. In such cases, in order to allow condensation of the filling material and promote apical sealing, it is imperative to create an artificial apical barrier or induce closure of the apical foramen with calcified tissue (apexification) <sup>(2)</sup>.

Mineral trioxide aggregate (MTA) was developed by Torabinejad in the early 1990's. The clinical efficacy of ProRoot MTA is well established. However, its drawbacks include difficult handling characteristics, long setting time <sup>(3)</sup>, **low** strength up-on initial set <sup>(4)</sup>. The MTA plus introduced to improve the mechanical properties of MTA like short setting time, increase sealing ability and strength <sup>(5)</sup>.

Biodentine in addition to the chemical composition based on the calcium silicate and water chemistry which brings the high biocompatibility of already known endodontic repair cements like MTA, it has increased physico-chemical properties like short setting time, high mechanical strength which make it clinically easy to handle and compatible, not only with classical endodontic procedures, but also for restorative clinical cases of dentine replacement <sup>(6, 7)</sup>.

Bioceramic material is hydraulic calcium silicate cement need water inherent in the dentinal tubules to drive the hydration reaction of material, Bioceramic material has non shrinkage upon setting and remains non

resorbable inside the root canal and retro-preparation <sup>(8)</sup>.

## Materials and methods

Sixty freshly extracted maxillary first molars teeth with straight palatal root were selected from different health centers for this study according to specific criteria. Using diamond disc mounted on straight hand-piece and under water coolant the palatal root of teeth was sectioned perpendicular to the long axis of the root at the furcation area to facilitate straight line access for canal instrumentation and filling procedure. The length of the root was determined by digital caliper and marker to (10) mm from apex to cemento-enamel junction. The root canals were prepared with Crown-Down technique by Protaper universal rotary system (Dentsply, Maillefer, Switzerland) to master apical file F5 at 250 rpm and 2 N/c torque. Five millimeter of 2.5% NaOCL with 27-G syringe was used for irrigation between each file size with a final rinse of 5 ml, 17% EDTA (PD, Swiss endo) for 1min. Followed by 10 ml of distilled water to remove any remnant of the irrigation solutions <sup>(9)</sup>. The roots were randomly divided into three groups (n=20) according to apical filling materials used:

**Group (1):**In this group, the MTA Plus (Cerkamed medical company, Polska) mixed according to the manufacturer's instructions. The MTA insertion into the canal by using micro apical placement (Endo map system), then condensation of material by finger plugger toward apical part of root until obtained 4mm thickness of MTA from working length by adjusting the stopper on finger plugger. After each 2mm of material checking radiographically to show any voids on filling

material, then moisture cotton was put inside canal over material to complete setting of material.

**Group (2):** In this group, Biodentine (Septodont, France) was used according to manufacturer instruction, Biodentine material available as powder in capsule and liquid in pipette. Six drops of liquid add to powder in capsule then the capsule mixing in amalgamator for 30 seconds. The canal of root was dried with paper point then material remove from capsule by spatula to plastic container. Biodentine insertion to canal by using micro apical placement (Endo map system), then condensation by finger plugger to apical part of root until obtained 4mm thickness of material from working length by adjustment of stopper on finger plugger. After each 2mm of material checking radiographically to show any voids in material.

**Group (3):**In this group Bioceramic (Total Fill, FKG, Swiss endo) was used according to manufacture, BC repair material available in premixed syringe with special tips for delivery of material, the syringe cap from BC root repair material syringe was removed and securely an intra-canal tip was attached with a clockwise twist to the hub of the syringe. After root canal was dried with paper point. BC root repair material was dispensed into root canal by compressing the plunger of the syringe. Then continuous until (4mm) thickness of material obtained from working length. After that material checking radiographically. Then moisture cotton was put inside canal over material to complete setting of material.

The samples of all groups obturation with E &Q plus system,

according to manufacturer instruction after 24 hours from put of apical third filling. The back-filling of canal was achieved by injection of thermoplasticized gutta percha by using E&Q gun, each time injection 2mm segment and compaction of increment with a prefitted plugger<sup>(10)</sup>. Then the access opening sealed with temporary filling material serve as a barrier to ingress fluid then all samples put incubator for 3day<sup>(11, 12)</sup>

Ten roots for each group were embedded in clear orthodontic resin<sup>(13)</sup>. The sectioning of root was made by using Diamond Cut-off Saw<sup>(11, 14)</sup>, cut was made horizontally to obtain one sections apical at 2 mm and 4mm from anatomical apex in 2mm in thickness. The thickness of each section was measured with a digital caliper, thus, study groups provided (10) specimens from each group.

### Push-out bond strength test

Push-out test was performed by applying a compressive load to the apical aspect of each slice via a cylindrical plunger mounted on Tinius-Olsen Universal Testing Machine managed by computer software The diameter of both apical and coronal side of section was measured from which the radius was calculated<sup>(15)</sup>. The sizes of punch pins was used 0.50 mm diameter for the apical slices. The punch pins should provide almost complete coverage over the filling material without touching the canal wall<sup>(11,16)</sup>. The root filling in each section subjected to loading using a universal testing machine<sup>(11, 17)</sup>. Loading was performed on microcomputer electrical control universal testing machine (WDW50) at a speed of 0.5mm / min in an apical-coronal direction until the first dislodgment of filling material and a sudden drop along the load deflection. The punch was positioned so that only

contacts the filling material and avoided contact with root canal walls<sup>(18)</sup>. The maximum failure load was recorded in Newton (N) and was used to calculate the push-out bond strength in megapascals (MPa) according to the following formula<sup>(19)</sup>:

$$\text{Push-out bond strength (MPa)} = \frac{\text{maximum load } N}{\text{Adhesion area of root canal filling material}}$$

### Analysis of failure modes

After the push-out bond strength test, each sample was inspected with a Stereomicroscope, at 40x magnification to determine the failure mode. Each sample was evaluated and placed into one of 3 failure modes<sup>(11,15,20)</sup>.

**Type I:** adhesive failure,

**Type II:** cohesive failure, within the filling material.

**Type III:** mixed failure, which contains both adhesive and cohesive failures

### Microleakage study

Ten roots which remained from each group were cover with one layer of nail varnish and two layer of sticky wax by dipping technique except 1mm apical. The methylene blue was used as leakage indicator for all group<sup>(12)</sup>. A puncher was used to made hole in the center of rubber cap to create space into which the coronal third of each root passed and fixed to rubber cap. The apical 4mm of each root was immersed in plastic container containing methylene blue and deposited in an incubator at 37C for 72 hours. At end of this period, the roots were removed from the methylene blue and wash under running water in position opposite to apical foramen for 15 minutes, the sticky wax was scraped from the root surface with lacron carver and wash again under running water 30 minutes<sup>(21)</sup>. The roots were

decalcified with 5% nitric acid for a period of 5days, the roots were then washed under running tap water for 30 minutes and dehydrated by 99% ethyl alcohol for 3 days with daily change of alcohol, then all roots became transparent by immersion in methyl salicylate for 24 hours<sup>(22)</sup>. Linear dye penetration was measured from apical foramen to the maximum extension of dye using light stereomicroscope under light (40x) magnification with calibrated scale ocular to establish the degree of apical dye penetration in millimeters<sup>(21, 23)</sup>. ANOVA test and LSD test was performed as statistical analysis for push-out bond strength and microleakage. Person coefficient (r) used to test correlation between push-out bond strength and apical dye penetration in microleakage for each group of material.

### Results

Mean values and standard deviation of push-out bond strength for all groups presented in (Table-1). The Biodentine group has the highest mean values in comparison with other groups followed by MTA Plus group, while Bioceramic group has the lowest mean value. Analysis of variance (ANOVA) test was performed and showed that there were very high significant differences among materials in (Table-2). There were very highly significant differences ( $p \leq 0.001$ ) between Biodentine and Bioceramic group, also there were very highly significant differences between MTA Plus and Bioceramic ( $p \leq 0.001$ ) in (Table-3), there was no significant difference between Biodentine and MTA Plus at ( $P \geq 0.05$ ) in push-out bond strength.

The analysis of failure mode for push out test is presented in (Table -4). It was showed that the predominant mode of failure in MTA Plus group and Biodentine group was cohesive

failure within filling material. In BC material group the failure mode was predominantly adhesive (between filling material and dentine) and mixed (adhesive and cohesive).

Mean values and standard deviation of microleakage for all groups presented in (Table-5). The Biodentine group has the lowest mean values in comparison with other groups followed by MTA Plus, while the Bioceramic group has highest mean value in dye penetration. Analysis of variance (ANOVA) test was performed and showed that there were non significant differences among test materials ( $P \geq 0.05$ ) in (Table-6)

The Pearson's correlation coefficient in (Table-7), the correlation between push-out and apical dye penetration was positive linear correlation (r-value between 0 and +1).

## Discussion

Successful root canal treatment depends on the thorough debridement of the root canal system, the elimination of pathogenic organisms and finally the complete sealing of the canal space to prevent ingress of bacteria from the oral environment and spread to the periapical tissue<sup>(24)</sup>.

### 1-Push-out bond strength

The Biodentine group showed the highest mean of push-out bond strength, the highest bond strength of Biodentine could be explained by the Biodentine is calcium silicate based material with improved in physio-chemical properties through control the purity of calcium silicate by the active biosilicate technology which consist in eliminating aluminate and other impurities. The Biodentine has polycarboxylate-based hydrosoluble polymer system described as (water reducing agent)

or super plasticizer, help in maintaining the balance low water content (decrease porosity) and consistency of the mixture the cement, the water reducing agent lead to proper wetting of the dentin, reducing the needed water for the reaction, increases the compressive strength and microhardness of the cement<sup>(7)</sup>. Furthermore Biodentine has short setting time (12 minutes) which achieved by combination different effect like particle size, higher specific surface, and adding calcium chloride to liquid component as accelerator. In addition the hardening process of Biodentine results from of the formation of crystals that are deposited in a supersaturated solution, calcium silicate hydrate gel, considered as the matrix of the cement, and the crystals of  $\text{CaCO}_3$  are filling the spaces between grains of cement, which improves the mechanical properties of the cement<sup>(25)</sup>. The adhesion of Biodentine cement to dental surfaces may result from a physical process of crystal growth within dentine tubules leading to a micromechanical anchor, the possible ions exchanges between the cement and dental tissues constitute formed a tag-like structure composed of the material itself or calcium or phosphate rich crystalline which higher in Biodentine than other materials<sup>(26)</sup>.

When comparing the Biodentine group with MTA Plus no significant difference was found. This could be related to that MTA Plus has fine (Nano) particles size, which improved it is handling characteristics, homogenous mixing, increase speed of hydration process, improve strength and mechanical properties. Expansion

of MTA during setting, and particles size of MTA Plus allow material to penetrate deeply in dentinal tubules. In addition short setting time (15 minutes) of MTA Plus<sup>(5)</sup>.

Very highly significant difference was showed between Biodentine and Bioceramic material group, and there was very highly significant difference between MTA Plus group and Bioceramic material group. This could be related to the combination effect of the chemical and mechanical bonding of Bioceramic material to dentin walls. Because of the apical dentine less patent tubules than coronal dentine and more complex structure of tubular dentine apparently yields itself better to infiltration compared to sclerotic apical. In some apical area the dentine is irregular and devoid of tubules<sup>(27)</sup>. However, this does not necessarily mean that sclerotic dentine cannot be sealed as well as tubular dentine<sup>(28, 29)</sup>. In addition diffusion of ions through dentine result from chemical bonding between material and dentin was less pronounced and of shorter duration effect for EndoSequence Bioceramic repair material than MTA<sup>(30)</sup>. Long setting time (minimum 4 hours) of Bioceramic root repair material. This result disagree with<sup>(18)</sup> they found push bond strength of Bioceramic repair material was significantly higher than MTA, their result may be related to the slice used for push-out test taken from middle portion of teeth, als.

In the present study the predominant failure mode of Biodentine is cohesive, according to<sup>(31)</sup> the Biodentine is time-dependent at least two weeks are necessary to reach final setting

stage after initial setting, this material continues to improve in terms of internal structure toward more dense material with decrease porosities. MTA Plus group showed predominate failure mode was cohesive this explained by the MTA Plus expansion during setting, in addition (Nano) particles of MTA Plus allow the material to deeper penetration in dentinal tubules<sup>(32)</sup>. while in Bioceramic material group showed predominate failure mode was adhesive and mixed this related to the apical dentine less patent tubules than coronal dentine, in some apical area the dentine is irregular and devoid of tubules<sup>(27)</sup>, the Bioceramic material depends on mechanical-chemical bonding with dentinal tubules, because complex structure of tubular dentine apparently yields itself better to infiltration compared to sclerotic apical.

## 2-Microlakage

Leakage studies constitute a major of contemporary endodontic research. The most common method used remains measuring of liner penetration of dye, but the nature and amount of leakage observed with this technique cannot be extrapolated to an in vivo situation. Measurement of dye penetration were made after decalcifying and clearing the root which it renders the root transparent, enable three dimensional observation of the dye penetration, Dye penetration technique has been used in this study because of its ease of performance as compared to other available techniques<sup>(33)</sup>. In this study, all materials had apical dye penetration in microleakage and non-significant difference among

materials groups, this related to (the MTA Plus, Biodentine, Bioceramic) are calcium based material which is the most biocompatible and bioactivity material<sup>(34, 35)</sup>. With high PH of materials, antibacterial, and regeneration properties of materials<sup>(36,37, 38)</sup>, in addition mechanical-chemical adhesion of materials to dentine<sup>(39)</sup>.

### 3-Corelation between push-out bond strength and microleakage.

Many obturation systems were proposed the endodontics as to approach the good sealing ability and adhesion to dentin. Adhesion of the root filling to the dentinal walls has two main advantages; in a static situation, it should eliminate any spaces that allow percolation of fluids between the filling and the wall. In a dynamic situation, it is needed to resist dislodgment of the filling during subsequent manipulation<sup>(40)</sup>. In this study showed the correlation between push-out bond strength and apical dye penetration in microleakage for each group of material was positive liner correlation, this related to (MTA Plus, Biodentine & Bioceramic) materials penetration in dentinal tubules when contact with dentine, ions loading in dentinal tubules during setting, the crystallization growth occurs within the tubules, participating in this way, in the micro-mechanical anchor. In addition, ions exchanges between material and dentine lead to formed a tag-like structure composed of the material itself or calcium or phosphate rich crystalline deposits, this improving push-out bond strength and sealing ability of material result from that less microleakage<sup>(31, 39)</sup>.

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Table (1): Descriptive statistics of the push- out bond strength (MPa) of different materials

Max.	Min.	S.D.	Mean	N	Materials
26.249	14.583	3.896	19.395	10	MTA
17.234	3.446	4.117	10.977	10	Bioceramic
22.007	15.908	1.767	19.687	10	Biodentine

Table 2: ANOVA test for mean the push- out bond strength (MPa) among different materials

p- value	F-test	Mean Square	d.f.	Sum of Squares	ANOVA
0.000 (HS)	20.824	244.689	2	489.378	Between Groups
		11.750	27	317.258	Within Groups
			29	806.636	Total

Table 3-: LSD test for mean the push- out bond strength (MPa) between each two material

p-value	Mean Difference	Materials	
0.000 (V.HS)	8.418	Bioceramic	MTA Plus
0.851 (NS)	-0.291	Biodentine	
0.000 (V.HS)	-8.710	Biodentine	Bioceramic

Table (4): Frequency distribution and percentages of the failure mode of different materials

Total	Mode of failure			No.	Materials
	Mixed	Cohesive	Adhesive		
10	3	7	0	No.	MTA Plus
100	30	70	0	%	
10	4	2	4	No.	Bioceramic
100	40	20	40	%	
10	3	6	1	No.	Biodentine
100	30	60	10	%	

Table (5): Descriptive statistics of the microleakage (mm) of different materials

Max.	Min.	S.D.	Mean	N	Materials
0.500	0.220	0.088	0.364	10	MTA Plus
0.790	0.270	0.186	0.477	10	Bioceramic
0.450	0.250	0.059	0.359	10	Biodentine

Table (6): ANOVA test for mean apical dye penetration for all groups

p-value	F-test	Mean Square	d.f.	Sum of Squares	ANOVA
0.071 (NS)	2.927	0.045	2	0.089	Between Groups
		0.015	27	0.411	Within Groups
			29	0.500	Total

Table (7): correlation between the push-out bond strength and apical dye penetration in microleakage in each group of material

Biodentine	Bioceramic	MTA Plus	Relation
0.213	0.338	0.011	r
0.554	0.340	0.977	p-value