



**PUSH OUT BOND STRENGTH OF GLASS IONOMER-
IMPREGNATED GUTTA PERCHA/GLASS IONOMER SEALER
SYSTEM TO ROOT CANAL DENTIN CONDITIONED WITH
DIFFERENT ENDODONTIC IRRIGANTS**

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ABSTRACT

The present study evaluated the push out bond strength of Active GP system versus Gutta-percha/AH Plus sealer using different irrigants. Fifty six human single rooted teeth were instrumented with a crown-down technique using Endosequence rotary Ni-Ti file system. The teeth were equally divided into two main groups and eight subgroups according to final irrigant: NaOCl, EDTA, Citric acid, and MTAD. Obturation was done by single cone technique in Active GP system, and with lateral compaction in GP/AH Plus group. Each obturated tooth was embedded in Epoxy cylinder, where three sections of 2 mm were done using the Isomet saw. The push out bond strength was done using universal testing machine working at a speed of 0.5 mm/min. Data were analyzed using one way analysis of variance followed by Newman-Keuls post-hoc test. Stereomicroscopic examination determined the type of bond failure. Results showed that in Active GP group, NaOCl dentin-treated subgroup had the highest bond strength mean value (6.98 ± 1.9 MPa) followed by citric acid subgroup (5.40 ± 1.1 MPa), then MTAD subgroup (4.71 ± 0.7 MPa), while EDTA subgroup recorded the lowest value (4.14 ± 1.4 MPa), however they were statistically nonsignificant ($P > 0.05$). In the GP/AH Plus group, EDTA dentin-treated subgroup showed statistically significant higher mean bond strength (5.9 ± 0.7 MPa) followed by NaOCl subgroup (5.40 ± 1.1 MPa), then citric acid subgroup (4.6 ± 0.6 MPa), while MTAD subgroup recorded the lowest value (3.5 ± 0.1 MPa). Failure in Active GP group was mainly cohesive in the Gutta-percha, while GP/AH Plus group showed mainly adhesive failure of AH Plus sealer with the gutta-percha.

KEY WORDS: Push out bond strength, Endosequence, Active GP system, AH Plus sealer

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INTRODUCTION

The ultimate goal of endodontic treatment is to protect the peri-radicular tissue from the effect of microorganisms and their byproducts⁽¹⁾. This is accomplished by the chemo-mechanical preparation of the root canal system.

Traditional Gutta-percha, which has been used for many years as an obturation material, does not bond to root dentin and must be used with sealer to achieve adaptation between the core Gutta-percha and the dentinal walls. AH Plus, an epoxy based sealer, has satisfactory physicochemical properties and good mechanical bond strength⁽²⁾.

Recently, manufacturers have introduced adhesive dentistry to the field of endodontics with a specific focus on obtaining a "monoblock" in which the core material, sealing agent, and the root canal dentin form a single cohesive unit. Active GP (Braseler USA, Savannah, GA) is a new glass-ionomer root canal-filling system that has been marketed as to create a single-cone monoblock obturation. The system comprises glass ionomer impregnated and coated gutta-percha cones that are bondable to a sealing agent composed of barium aluminosilicate glass powder and polyacrylic acid⁽³⁾. This was claimed to offer adhesive bonding of core material to intraradicular dentin via the glass-ionomer sealer.

Many materials are introduced to irrigate the root canals; Sodium hypochlorite (NaOCl) has the capacity to dissolve organic tissues and has antimicrobial action^(4,5). However it does not remove the smear layer⁽⁶⁾ and has to be combined with chelating solutions as Ethylenediaminetetraacetic acid (EDTA) or de-calcifying agents as citric acid. Citric acid is used alone or as a component of MTAD, (Mixture Tetracycline Citric Acid and Detergent)⁽⁷⁾. The usage of irrigants might affect the

dentin structure with possible effect on subsequent bonding with the obturation material.

Adhesion properties of root canal sealers to dentin are determined by several mechanical tests. Bond strength measurements are among the methods used to evaluate the effectiveness of adhesive systems, hence predicting their performance in oral environment⁽⁸⁾. The bond strength refers to the force per unit area required to break the bond between an adhesive material and tooth substrates such as enamel or dentin. The push out test has been described as the test fracture occurring parallel to the dentin-bonding interface, which makes it true shear test and a good evaluation of the bond strength⁽⁹⁾.

Thus the present study investigated whether the pretreatment of the root canal dentin with different irrigants would affect the push out bond strength of the Active GP/Glass ionomer system to root canal dentin. The null hypothesis tested was that there was no difference in the push-out bond strength between the two types of obturation systems.

MATERIALS AND METHODS

1- Teeth selection and preparation

Fifty six freshly extracted straight, single-rooted human teeth with single canal and fully formed apices were collected. After thoroughly washing under running water, the teeth were immersed in 5.25% Sodium Hypochlorite* for 30 minutes to remove soft tissues on the root surface. The teeth were then gently cleaned with ultrasonic scaler to remove any remaining soft tissues or calculus, and stored in 0.2% sodium azide** until time of use.

The teeth were randomly distributed into two main experimental groups according to the obturation material used:

* Clorox, Egypt company for households products.

** El-Gomhoreya company, Egypt.

- Group I (n=28) teeth obturated with Active GP system.
- Group II (n=28) teeth obturated with Gutta-percha and AH Plus sealer.

Furthermore each main group was subdivided into 4 subgroups according to the final irrigant type:

- Subgroup A (n=7): Final irrigation with 5.25% NaOCl (5 ml, 5 min.).
- Subgroup B (n=7): Final irrigation with 17% EDTA (5 ml, 5 min.).
- Subgroup C (n=7): Final irrigation with 25% Citric acid (3 min.).
- Subgroup D (n=7): Final irrigation with 1.3% NaOCl followed by MTAD (5 min.).

Access cavities through the crowns were performed. Canal patency and uniform apical diameter were confirmed by passing file #15* through the apical foramen. Working length for each root canal was determined by passing #15 K-file till it flushed with the apex and then subtracting 1mm from this length. Manual apical preparation was done by K-flex-O** #20, 25, 30 in watch winding motion. All samples were further instrumented using Endosequence System***. A crown-down technique was used with successive files starting from size # 35 taper 6% till size # 50 taper 6%. Endo Torque America Plus motor**** adjusted at 600 rpm with torque 2N/cm was used. After each rotary file, irrigation was done using 5ml of 2.6 % NaOCl. Final irrigation step with different irrigants was done followed by flushing by 10 ml of distilled water. For both groups, irrigation was performed by

a disposable plastic syringe with 30 gauges needle placed passively into the canal as far as 2 mm from the apical foramen.

Access cavity was closed by a cotton and temporary filling. Each tooth was then embedded in Clear polyester resin in a custom-made splitting copper mold. After setting of the epoxy resin, the temporary filling was removed from the access cavity. Each canal was dried with paper points**** #50. The canal patency was reconfirmed by passing #15 file just out of the apical foramen.

2- Root canal obturation

Group I (Active GP /Active GP sealer) (n=28), Single cone technique was used for the Active GP system following the manufacturer's recommendations. The apical two thirds of the master cone (# 50/0.06 taper) was coated with the Active GP/glass ionomer sealer and inserted slowly in the canal to the full working length.

Group II (Gutta-percha / AH Plus sealer) (n=28), lateral compaction technique was used using conventional Gutta-percha points and AH Plus sealer. For each canal, an ISO standardized 2% master cone # 50 was fitted at the working length. A # 30 finger spreader***** which could be placed to within 1 to 2 mm of the working length alongside the master cone was used. Afterwards, auxiliary cones size 25 were used for the lateral compaction.

For both groups, the excess core material was severed with a hot instrument at the canal orifice. Radiographs were taken from buccal and mesial views for each tooth to ensure the adequacy of the obturation.

* Dentsply maillefer, Ballaigues, Switzerland.

** Dentsply maillefer, Ballaigues, Switzerland.

*** Rotary Ni-Ti file system with 0.06 taper and 25mm long (Brasseler, Savannah, GA, USA).

**** Medidenta International Inc. Woodside, NY, USA.

***** Meta Biomed Co. LTD,1115-6 Namchon-Ri, MyeonCheongwon-Gun, Chungbuk, Korea.

***** Mani Inc, Japan.

3- Specimen preparation and Push out bond strength test (Figure 1)

Slicing was done using precision diamond Isomet saw* by 0.3mm thickness disc. Approximately 7 slices were obtained from each tooth with total of 392 slices. Slices which presented oval canal or visible inadequate filling or voids were excluded. Finally, three slices from each tooth were selected to perform the push out test, giving a total of 168 slices for all groups; 21 slice per subgroup.

Each section was photographed from its apical aspect using digital microscope with direct show video capture software**. A 10 mm ruler was set beside each section to aid in magnification calibration. Proper determination of the canal diameter allowed proper selection of the size of the plunger size which should be less than the canal diameter by 0.1mm.

The plunger tip attached to a universal testing machine (Lloyd LRXplus; Llyod Instruments Ltd, Fareham, UK) was positioned so that it only contacted the filling material in each section (Figure 1). The machine delivered compressive loading at a crosshead speed of 1 mm/min using a 1, 0.7, 0.5 mm diameter stainless steel cylindrical plungers. The push-out force was applied in an apico-coronal direction until bond failure occurred, which was manifested by extrusion of the obturation material and a sudden drop along the load deflection curve. The force was recorded by using Nexygen data analysis software (Llyod Instruments Ltd). The maximum failure load was recorded in Newtons and was used to calculate the bond strength in mega Pascals (MPa) according to the following formula: Push out bond strength (MPa) = Load (N) / Area (mm²) (10). The area was calculated from the formula: Area = ($\pi r_1 + \pi r_2$) x L. where, π is the

constant 3.14, r_1 is the radius of smaller apical canal diameter of each slice, r_2 is the radius of the larger coronal canal diameter of each slice, h is the height of the sample in millimeters and L is calculated as

$$\sqrt{(r_2 - r_1)^2} + \sqrt{h_2}$$

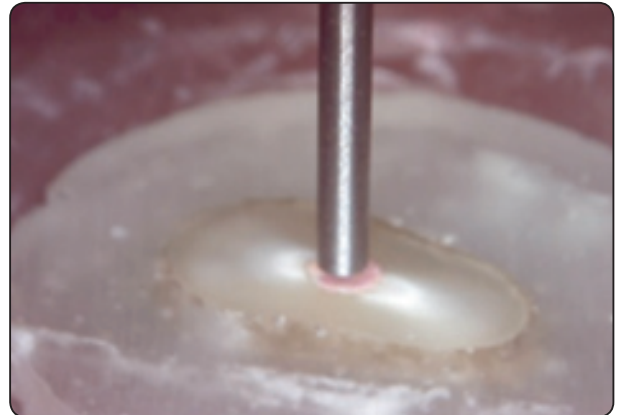


Fig. (1) Suitable plunger size just on top of the filling material during push out test.

4- Fractographic analysis

Following the push out testing, dentin surfaces were inspected with a stereomicroscope (Sharper image corporation, Embarcadero, San Fransisco, CA, USA) at X 25 magnification to determine the failure mode: i) adhesive failure either at the sealer-dentin interface or between the sealer-core material. Or ii) cohesive failure within the filling material (sealer or core material). Or iii) mixed failure which contains both adhesive and cohesive failures.

5- Statistical Analysis

Parametric analysis was performed with one way analysis ANOVA followed by Newman Keuls post-hoc tests between irrigations within each group. Student t-test was used to evaluate significance between the two main groups. Statistical analysis was performed using Graphpad Prism-4 statistics software for Windows. P value ≤ 0.05 was considered to be statistically significant in all tests.

* Model 800-900, Leco Corporation, Lake view Avenue, St. Joseph, Michigan, USA in the British University, Material department.

** Sharper image corporation, Embarcadero, San Fransisco, CA, USA.

RESULTS

I- Comparison of the effect of irrigants within each sealer group (Table 1)

Active GP (Figure 2)

NaOCl irrigation recorded the highest mean bond strength (6.98±1.9 MPa), followed by Citric acid (5.40±1.1 MPa), then MTAD (4.71±0.7 MPa). While EDTA recorded the least mean bond strength (4.14±1.4 MPa). Statistically, there was no significant difference (P=0.09).

AH-Plus (Figure 2)

EDTA irrigation recorded the highest mean bond strength in the AH-Plus group (5.9±0.7 MPa), followed by NaOCl (5.2±0.4MPa), then Citric acid (4.6±0.6MPa). While MTAD recorded the least mean bond strength (3.5±0.1MPa). Statistically, there was significant difference between the irrigants (P=0.0001).

II- Comparison of the effect of each irrigant between the two sealer groups

NaOCl recorded higher mean bond strength in the Active GP group than in the AH Plus group.

Statistically, there was a significant difference (P=0.0451).

EDTA recorded higher mean bond strength in the AH Plus group than in the Active GP group. Statistically there was significant difference (P=0.0142).

Citric acid showed higher mean bond strength in the Active GP group than in the AH Plus group. Statistically there was no significant difference (P=0.1186).

MTAD recorded higher bond strength in the Active GP group than in the AH Plus group. Statistically there was a significant difference (P=0.0015).

III- Fractographic analysis of the failure mode (Table 2)

It was shown that mainly the failure mode in the Active GP group was cohesive failure mainly within the Active GP cone with some adhesive failure between the Active GP sealer and Active GP cone. While in the AH-Plus group failure mode was absolute adhesive failure of the AH Plus sealer mainly with the Gutta-percha and partly with dentin.

TABLE (1) Mean total bond strength (Mean±SD) recorded in mega Pascal for both sealer groups with different dentin surface treatments.

Group /Irrigant	Active GP	AH Plus	P value
NaOCl	6.98±1.9	5.2±0.4	0.0451*
EDTA	4.14±1.4	5.9±0.7	0.0142*
Citric acid	5.40±1.1	4.6±0.6	0.1186ns
MTAD	4.71±0.7	3.5±0.1	0.0015*
P value	0.090 ns	0.0001*	

ns; non-significant (p>0.05)

*; significant (p<0.05)

TABLE (2) Different failure modes for both groups (Active GP group and AH Plus group) with different dentin surface treatment.

Group/Irrigant	Active GP			AH-Plus		
	Adhesive	Cohesive	Mixed	Adhesive	Cohesive	Mixed
NaOCl	0%	100%	0%	100%	0%	0%
EDTA	20%	67%	13.30%	100%	0%	0%
Citric acid	25.60%	57%	16.70%	100%	0%	0%
MTAD	5.60%	89%	5.60%	100%	0%	0%

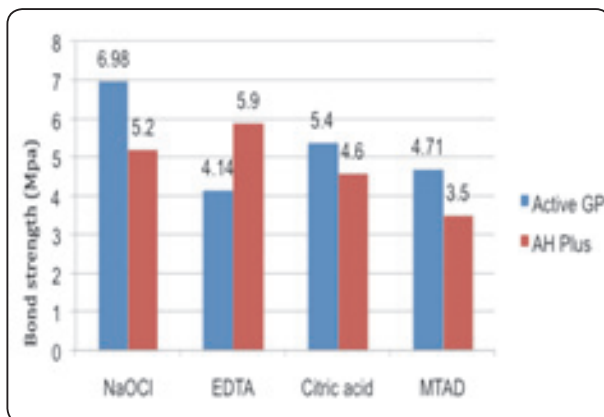


Fig. (2) Bar chart of mean bond strength values recorded in mega Pascal for Active GP group and AH-Plus group with different irrigants.

DISCUSSION

Many obturation systems were proposed to the endodontist 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 ⁽¹¹⁾.

Active GP is a recently introduced obturation system by Brassler USA, which is based on glass-ionomer with very few reports on its properties. In this system, the bond strength of the glass-ionomer

based sealer is expected to be satisfactory and might be attributed to its enhanced chemomechanical interaction to the newly glass-ionomer coated Gutta-percha cone. Because of its multiple ionized carboxylate groups, the polyacrylic acid within the sealer had been shown to have a chelating reaction with the calcium ions of the dentin matrix ⁽¹²⁾.

NaOCl was selected in the present study as it is the standard irrigant commonly used during root canal treatment. EDTA is a chelating agent used in the present study alternatively with NaOCl as it is the standard for smear layer removal ^(13,14). Citric acid irrigation was also tested in this present study for its advantages as a disinfectant and a demineralizing agent for smear layer removal in combination with NaOCl, however, it should be cautiously used as its prolonged exposure may cause dentinal tubules collapse and denaturation ^(13,14). The effect of MTAD as an irrigant was also worth testing in the present study. It is an acid (pH = 2.15) which contains antibiotic (doxycycline), detergent (tween 80) and citric acid ⁽⁷⁾. The doxycycline and citric acid are expected to remove inorganic portion of the smear layer and demineralize the underlying dentin. According to *Tay et al* ⁽¹⁵⁾, MTAD produced a 10 to 12 mm thick zone of demineralized dentin compared with only a 4 to 6 mm thick zone produced by EDTA. *Torabinejad et al* ⁽⁷⁾ reported that it could aid in producing smear free dentin with opened

dentinal tubules. Furthermore, the detergent permits increased dentin surface energy and wettability, hence increasing intertubular dentin permeability as well as exposing collagen matrix and intertubular fluid.

In the present study, the push out bond strength test was used in which an axial load is applied to push a punch of known diameter through a specimen of known thickness⁽¹⁶⁾. The advantage of this test is that it is less sensitive to small variations amongst specimens and to variations in stress distribution during load application⁽¹⁷⁾.

Results of the present study showed that in the Active GP group, when NaOCl was used alone, Active GP sealer recorded a non-statistically significant the highest mean bond strength value, compared with other irrigants. This may be because NaOCl is not totally effective in smear layer removal. The partial presence of smear layer being a reservoir for calcium ions exchange is needed for good adhesion with the glass-ionomer sealer^(14, 18).

When EDTA was used as a final irrigant, it recorded a non-significant least mean bond strength value in the Active GP group. This could be attributed to the prolonged application time (5 min) which might have caused funneling out of the dentinal tubules, decreasing the available intertubular dentin for bonding, as well as decreasing the available amount of calcium ions necessary to achieve adequate bonding.

Whereas Citric acid and MTAD showed intermediate values of bond strength, evidently lower than that of NaOCl, although not statistically significant. Partial decalcification of dentin caused by acid component of both irrigants might have also affected the available amount of calcium needed for glass-ionomer bonding.

Results, in the Gutta-percha/AH Plus group showed that the usage of EDTA significantly revealed the highest mean bond strength values which came in agreement with the findings

reported by *Sly et al*⁽¹⁹⁾. Pretreatment with this chelating agent might have altered the surface energy of dentin which significantly increased the wetting ability of dentinal wall. Thereby providing a suitable dentin substrate for the adhesion of materials of a hydrophobic nature as the resinous AH Plus. Furthermore, the effective removal of the smear layer by EDTA allowed for the extension of the resin into the opened dentinal tubules, creating efficient micromechanical retention⁽⁸⁾.

Using MTAD, however, resulted in the least significantly mean bond strength values with the Gutta-percha/AH Plus group. The offered minor retention of MTAD-treated surface could be due to its insufficient dentin tubular area and inadequate depth of demineralization⁽¹³⁾. Although, MTAD already contains Tween 80, which was supposed to enhance the dentin surface energy and surface wettability, and in turn increase penetration and diffusion into the dentinal tubules, yet it seemed that its effect, according to the findings of this study, was insufficient.

NaOCl and citric acid showed intermediate 2nd and 3rd ranking for bonding with Gutta-percha/AH Plus. Although NaOCl does not totally remove the smear layer, surprisingly it reported 2nd rank for bond strength with Gutta-percha/AH Plus. Baumgartner and Cuenin⁽²⁰⁾ showed some exposed dentinal tubules in the smear layer in all instrumented surfaces regardless of the concentration of the NaOCl. The use of NaOCl alone or in combination with EDTA was shown by Villegas et al⁽²¹⁾ to enhance obturation material penetration into accessory canals. On the other hand 3rd ranking of citric acid might have been caused by the prolonged application time (3min), causing peritubular and intertubular dentin erosion and funneling of the dentinal tubules⁽¹⁵⁾.

When comparing Active GP group with Gutta-percha/AH Plus group, the different mechanisms of bonding of both sealers played a role whereas: Active GP system depends mainly on chemomechanical bonding and the AH Plus sealer depends

mainly on mechanical retention of the resin tags. NaOCl showed the significantly higher mean bond strength value in the Active GP group than AH Plus. The partial presence of smear layer being a reservoir for calcium ions exchange is needed to achieve good adhesion with the glass ionomer sealer. Seemingly NaOCl might be recommended as the irrigant of choice for the Active GP system. EDTA, showed significantly increased mean bond strength values in the Gutta-percha/AH Plus group than that of the Active GP group which was in agreement with Hashem et al⁽²²⁾. The removal of the smear layer, by EDTA favours micromechanical retention of AH Plus sealer but depleted calcium ions necessary for the Active GP bonding. EDTA as a final irrigation might be recommended as the irrigant of choice for the Gutta-percha/AH Plus obturation. Citric acid showed a non-significantly higher mean bond strength value in the Active GP group than AH Plus. Its combined action of partial decalcification as well as funneling of the dentinal tubules might have affected bonding to both sealers. MTAD resulted in 3rd ranking of bond strength values with the Active GP compared to a bottom rank in the Gutta-percha/AH Plus group. The use of MTAD as a final irrigant with Gutta-percha/AH Plus is the least recommended irrigant.

Regarding the mode of failure, in the Active GP group failure mode was mainly cohesive within the core material itself which might be due to weakening in the Gutta-percha when sialinated with glass-ionomer coating. Researches are thus advised to be directed to strengthening the cohesive strength of this core material.

In the Gutta-percha/AH Plus group the failure mode, was adhesive whether between the Gutta-percha and the AH Plus mainly or between the dentin and AH Plus partly. So further researches and improvements should be directed to the improvement of the bond strength of the sealer with either Gutta-percha and dentin.

CONCLUSIONS

Within the limitations of the present in-vitro study, the following could be concluded:

- 1- Using Active GP system, the type of irrigant generally did not adversely affect the push out bond strength, although 5.25% NaOCl might be the irrigant of choice.
- 2- Using AH Plus sealer, the type of irrigant affected the push out bond strength. EDTA is the most recommended irrigant of choice, while MTAD is the least recommended.

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