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# Push-out Bond Strength of Endoseal MTA and AH Plus Sealers After Using Apple Vinegar as Final Irrigating Solution

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## *ABSTRACT*

**Purpose:** The present study was performed to evaluate the bond strength of EndoSeal MTA and AH Plus after using EDTA or apple vinegar as final irrigant. **Materials and Methods:** Sixty extracted human single rooted teeth were decapitated, instrumented using Universal ProTaper rotary system up to size F4 and divided randomly into two main groups (n=30) according to the solution used as final irrigation; **Group I**: irrigated with 17% EDTA and **Group II** : 5 % Apple vinegar, then distributed according to sealer used in obturation with gutta-percha into four subgroups (n=15); **subgroup IA** (EDTA + AH Plus), **subgroup I B** (EDTA +Endoseal MTA), **subgroup IIA** (apple vinegar + AH Plus), and **subgroup IIB** (apple vinegar + Endoseal MTA). After obturation and incubation in 100% humidity at 37°C for one week. All roots were embedded in self cure acrylic resin and sectioned horizontally into slices of 2 mm thickness, the push- out test was applied using universal testing machine, bond strength (MPa) calculated and the data were tabulated and statistically analyzed. **Results:** AH Plus represented the highest bond strength after using EDTA or apple vinegar compared with Endoseal MTA, EDTA showed superior results with both types of sealer compared to apple vinegar, and the middle part of the root canal showed the highest bond strength ($P<0.05$) **Conclusion:** EDTA final irrigating solution reported higher values of the bond strength compared to apple vinegar with both AH Plus and Endoseal MTA, and AH Plus reported highest push-out bond strength with both irrigating solutions.

***KEYWORDS***

*Endoseal MTA, AH Plus, EDTA, Apple Vinegar, Push-Out*

- Paper extracted from Masters thesis titled " Evaluation of Bond Strength of EndoSeal MTA Obturation System After Using Apple Vinegar as Final Irrigant"

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## INTRODUCTION

Obturation phase in the root canal therapy is performed aiming to provide the root canal system with fluid tight seal filling that prohibits the microorganisms and their products from reaching the periapical tissue and preventing periapical inflammation [1]. Gutta-percha with its biological and physical properties is a gold standard core material used in combination with endodontic sealers to obturate the space of the root canal. Due to lack of gutta-percha (GP) adhesiveness to dentin surface, the endodontic sealers are utilized to seal disparity between the core material and dentinal wall and fill the root canal irregularities thus ideal endodontic sealer should adhere to both core material and root canal wall [2].

Adhesion of sealers to dentin surface is a fundamental characteristic, it can provide hermetic sealing against microbial leakage and resist material detachment during mechanical forces which is the primary factor associated with the success of root canal treatment[3]. The nature of root dentin, smear layer, and interlinkage between sealer and dentin are factors affecting adhesion of the seal to dentin, thus using irrigating solution as a routine step in endodontic preparation with various chemical solutions leads to structural changes as evidenced by the reduction of dentine microhardness and architecture structural changes that subsequently affecting sealers adhesion to great extent [4, 5].

Sodium hypochlorite (NaOCl), with its concentrations range 0.5% up to 5.25% is the popular irrigant solution in endodontic , with its distinctive dissolving property of organic components of smear layer however it failed to get rid of its inorganic part, thus Ethylenediaminetetraacetic acid (EDTA) has chelation ability utilized to complete eradication of smear layer inorganic components through its reaction with calcium ions present in dentin forming calcium chelate which easily removed from the root canal[6]. However; The use of natural alternatives as root canal irrigat might be advantageous considering several unfavorable properties of traditional irrigating solutions that affect the physical and chemical characteristics of root canal dentine ultrastructure [7, 8].

Recently, Apple vinegar deserves attention and has been investigated to use as assisting solution during root canal chemo-mechanical preparation. Apple vinegar showed promising results on endodontic microbiota eradication as it has bactericidal effect against *Staphylococcus aureus* and *Enterococcus faecalis* the most frequent microorganisms persistent in endodontic infection [9]. With regarding to its effect on smear layer, it showed a neat ability to clean the smear layer due to presence of malic acid, acetic acid, and citric acid as main ingredient that has chelating effect that can provide smear layer free dentinal wall with benefits of using natural biological agents that reduces hazard of using chemical chelators as tissue reaction and minify dentin mechanical properties [10-12].

Different types of sealers have been introduced in the endodontic field, AH Plus an epoxy resin-based endodontic sealer used with gutta percha because of acceptable physical properties and biocompatible. It has advantages of its potential for better wettability of dentin and gutta percha, low solubility and setting time, good adhesion to dentine with long-term sealing ability, and more radiopaque. It has a massive market history and considerably used as a standard and reference sealer in many investigations [13, 14]. Lately, calcium-silicate based sealers were introduced in attempt to enhance the efficiency of endodontic sealers . Endoseal MTA is a new calcium silicate MTA-based sealer, the sealer is based on pozzolan cement and premixed paste-type which has remarkable biological and physical monarchy of MTA. It comprises calcium silicates and luminates, calcium sulfates, and calcium aluminoferrite [15]. Endoseal MTA reported to have different endodontic employments as obturation of the root canal system and perforation repair, and also has advantages of being biocompatibile and induce hard tissue formation, has antibacterial

impact, acceptable physic-chemical characteristics as short setting time, appropriate flow, premium film thickness, and direct easy application into the root canal system [16-18].

Adhesion of root canal sealer to dentin wall can be evaluated using the push-out test, it determines level of bond strength through measuring shear bond strength at dentin-sealer interface. It is favored method for determining the force needed to dislodge the root canal filling material out of the dentine cylinder using suitable drill, this force represent the filling bond strength. Push-out test has advances of being simple with no need for sophisticated paraphernalia, minimal variability, homogeneous stress allocation, and records repetitive digital readings of the bond strengths, thus this test represents preferable assessment method of the bond strength than the regular shear tests [19, 20].Therefore, this in-vitro experiment was desinged in order to assess the bond strength of both AH Plus and Endoseal MTA sealers to dentin wall of the intraradicular after using EDTA and apple vinegar as final rinse using push-out test.

The null hypothesis was that there is no difference of bond strength between AH Plus and Endoseal MTA to root canal wall treated either by EDTA or apple vinegar as final irrigant .

## MATERIAL AND METHODS

### Study design

The current study was planned and conducted in Endodontic Department, Faculty of Dental Medicine for Girls, Al-Azhar University. The experiment was designed, analyzed and interpreted according to the Consolidated Standards of Reporting Trials (CONSORT) [21]. Ethical approval for the use of extracted human teeth was obtained in accordance with guidelines from Research Ethic Committee (REC) Code (REC-EN-22-01), Faculty of Dental Medicine for Girls, AL-Azhar University.

### Sample size

Sample size was calculated according to the following formula**:**

$$\text{Sample size (N)} = (Z\alpha/2+1-\beta)^2 * 2*SD^2/ \Delta^2,$$

Where SD= Standard deviation of push- out bond strength value of apical section estimated from previous study [22], and $\Delta^2$ = effect size estimated to be 0.35 between the samples, $\alpha$ = Level of significance, and $1-\beta$ = Power.

$$N= (1.96+0.84)^2*2*(0.34)^2/ (0.35)^2=14.79$$

Accordingly, 15 teeth were used in each group with total sample 60 teeth.

### Teeth selection:

Sixty human, single, straight rooted, freshly extracted teeth with root canal system type I, of nearly equal length, free from root caries, crack, resorption, or previous root canal treatment were selected for this experimental study. Periapical radiograph of the mesio-distal and bucco-lingual directions were performed to confirm selection criteria. Soft tissue and deposits were cleaned using ultrasonic scaler (Woodpecker UDS-P LED Ultrasonic Scaler Ende, China), then were preserved in a jar filled with 10% natural buffered formalin at the room temperature.

### Sample preparation:

All teeth crowns were removed at cementoenamel junction using diamond disk mounted in low -speed hand piece under large amount of water coolant aiming to standardize sample length 16mm±1. The working length was adjusted using #10 K- File (Dentsply / Sirona, Ballaigues, Switzerland) which inserted to the canal until its tip was observed extruded at apical foramen and deducting 1 mm from this length, then the apical foramen was closed using wax.

The root canal system was prepared using ProTaper Universal rotary system (Maillefer, Ballaigues, Switzerland) up to the # F4 file, speed and torque for each file was adjusted according

to manufacturer instructions. After each file application; 3ml of 2.6% sodium hypo- chlorite (NaOCl) (Alex. Deteregents and Chemical Co., Egypt) was used to irrigate the root canal system dispensed slowly using a 31gauge Navitip needle (Navi-Tip, Ultradent product, South Jourdan, UT).

**Sample grouping:**

The sixty sample were divided randomly using closed opaque uncoded envelops before application of final irrigation to two main groups (**I &II**) 30 sample each according to final rinse; **Group I** (n=30) the final irrigation was applied using 17% EDTA solution (Amrit Chem& Min. Ag, Mohali, India) 3ml for 1 minute, while **Group II** (n=30) the final rinse used was 5% Apple vinegar (Kemal Kükrer Apple Vinegar- Turkey) 3ml for 1 minute. Each main group was then randomly divided into two subgroups (**A&B**) according to type of root canal sealer used for obturation either using AH Plus (**subgroups IA &IIA**) or Endoseal MTA (**subgroups IB &IIB**).

**Samples obturation:**

***Subgroups IA &IIA:***

After rinsing the root canal with 5 mL of distilled water and dried with sterile #30 paper points, the obturation procedures was performed using lateral condensation technique. After selection and verification of master cone #F4 GP (Meta Biomed, Chungcheong Buk-do, Korea), AH Plus sealer (Dentsply, Konstanz, Germany) was mixed according to manufacturer instructions by squeezing appropriate and equal amount of base and catalyst then mixed until homogeneous color and creamy consistency was observed. Master cone was coated with the AH Plus sealer and placed in the root canal up to the working length then accessory cone (# 15 taper 0.02) were used to complete the canal filling that condensed using preselected spreader of length 2 mm of the working length. After verifying the obturation by radiograph, the extruded part of GP was then seared off at the orifice level using heated plugger and condensed using cold plugger, then the orifices of the canal were sealed using temporary filling then incubated at 37˚C and 100% humidity for 7 days.

**Subgroups IB &IIB:**

Samples in those subgroups IB and IIB were obturated following the same steps in subgroups IA &IIA, however the sealer used was Endoseal MTA (Endo- seal; Maruchi, Wonju, Korea) which applied into the root canal directly using the premixed syringe and tip and the canal obturation procedures were completed. The orifices sealed and incubated at 37˚C and 100% humidity for 7 days.

**Push-out bond strength test:**

The root samples were positioned vertically and at the center in cold cured clear acrylic resin blocks (Acrostone Co., industrial zone, 15km northwest of Cairo, Egypt), after setting the roots were horizontally sectioned perpendicular to the long axis of the root using Isomet 4000 micro saw (Buehler, USA) cutting machine. Three sections of 2 mm thickness were obtained at 3, 7 and 11mm from the apex to represent apical, middle and coronal third respectively, slice were confirmed using a digital caliper then all segments were coded.

Each specimen was then carefully positioned on a custom made metallic block with circular cavity at the middle and push-out test was performed using the universal testing machine (Instron, Norwood, MA, USA). The load was applied at a cross-head speed of 0.5 mm/min by 3 plungers of different sizes (1 mm, 0.7 mm and 0.5 mm) for coronal, middle and apical sections respectively in an apical-coronal direction. All samples were subjected to compressive loading via a computer-controlled materials testing machine and data were recorded using computer software. The maximum failure load was recorded in newtons (N) and converted into megapascals (MPa). The bond strength was calculated from the recorded peak load divided by the computed surface area as calculated by the following formula: $[A = (3.14x\ r^1 X\ 3.14x\ r^2)\ L]$,

Where $r^1$ apical radius, $r^2$ coronal one, L= [(r1-r2)2+$h^2$]$^{0.5}$ & h is the thickness of the sample in millimeters].

## STATISTICAL ANALYSIS

Analysis of Data was done by SPSS program (statistical package of social science; SPSS Inc., Chicago, IL, USA) version 16 for Microsoft Windows. Data were presented as mean and standard deviation (SD) values. ANOVA test was done to compare means of the groups and post hoc Tukey test was used to detect which group is responsible for the significance. Results displayed table (1).

## RESULTS

The mean value of push-out bond strength recorded in (MPa) and standard deviations (SD) of samples of all tested segment of the four subgroups were given in table (1).

**Table (1)** *The means value, standard deviation (SD) for the comparison between push- out bond strength of the tested materials.*

| Push out Strength [MPa] | Coronal | Middle | Apical | P value |
|---|---|---|---|---|
| **Subgroup IA** | | | | |
| Mean | 5.12 | 5.35 | 4.97 | |
| SD | 2.00 | 0.98 | 2.39 | 0.856 |
| Min.- Max | 1.94-8.28 | 3.80-6.97 | 1.31-8.98 | |
| **Subgroup IB** | | | | |
| Mean | 2.00 | 4.003 | 3.13 | 0.000* |
| SD | 0.96 | 0.85 | 0.65 | |
| Min.- MAx | 0.27-3.24 | 2.83-5.89 | 1.87-4.13 | |
| **Subgroup IIA** | | | | |
| Mean | 3.47 | 4.18 | 3.97 | 0.400 |
| SD | 1.36 | 1.60 | 1.39 | |
| Min.- MAx | 1064-5.92 | 1.67-7.17 | 1.99-7.41 | |
| **Subgroup IIB** | | | | |
| Mean | 0.95 | 1.31 | 1.20 | 0.325 |
| SD | 0.68 | 0.82 | 0.46 | |
| Min.- MAx | 0.06-2.34 | 0.50-3.34 | 0.33-1.76 | |
| **P value** | 0.000* | 0.000* | 0.000* | |

*Significance difference (p value< 0.05).*

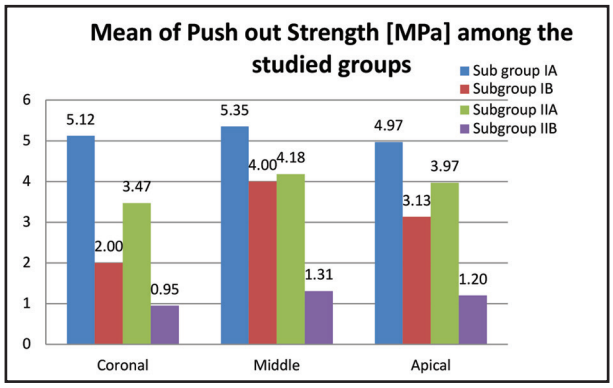


Figure (1) : A bar chart comparing the mean value and SD of push out bond strength between the four subgroups at the apical, middle and coronal root canal levels.

The comparison between the four subgroups, the highest push-out bond strength mean values were observed in **subgroup IA** (EDTA + AH Plus) at different root canal levels coronal, middle, and apical (5.12±2.00, 5.35±0.98 , and4.97±2.39) respectively, followed by **subgroup II A** (apple vinegar + AH plus) which recorded mean value for bond strength at coronal, middle, and apical (3.47±1.36, 4.18±1.60, and 3.13±0.65) respectively, thus AH plus sealer exhibited the highest bond strength values between the tested material. However; the results concerning Endoseal MTA sealer the highest mean value for push-out bond strength was observed in **subgroup IB** ( EDTA + Endoseal MTA) with value at the root canal levels coronal, middle, and apical ( 2±0.96, 4.003±0.85, 3.13±0.65) respectively, while the lowest bond strength values were observed in **subgroup IIB** (apple vinegar + Endoseal MTA) which recorded mean value as (0.95±0.68, 1.31±0.82, and 1.20±0.46) for coronal, middle, and apical thirds of the root canal levels (Fig. 1).

Comparing the results regarding the type of the final irrigating solution the root canals rinsed with EDTA in **subgroups IA** and **IB** showed highest mean values for bond strength either with AH Plus or Endoseal MTA. However; the samples rinsed with apple vinegar **subgroups IIA** and **IIB** showed the lowest mean values particularly with Endoseal MTA sealer which showed the lowest bond strength level between the four subgroups.

With regarding to bond strength mean values related to different root canal levels, the results showed that the middle part of the root canal reported the highest mean values of bond strength with significant difference compared to coronal and apical levels among the all-tested subgroup regardless the obturation materials or the final rinsing solution.

## DISCUSSION

The use of endodontic sealer with a thermoplastic GP for root canal obturation is avital step for root canal treatment success. The adhesion of sealer represented as a joint created amidst to the radicular dentin will provide good sealing and resistance to dislodgment force during function. The dislocation resistance of the sealer popularly evaluated using push-out bond strength test which was reported to be simple to reproduce, recorded even low values of the bond strength and easy to interpret [20].

Final irrigation that applied following the root canal shaping aiming to remove the smear layer and decrease microbial burden prior to obturation, was found to affect the radicular dentin properties and subsequently sealer bond strength[23]. Apple cider vinegar is naturally sourced, highly biocompatible irrigant recently studied to be used as contributory solution during chemo-mechanical preparation. It has attracted attention due to its promising results when compared to traditional irrigation protocol of using NaOCl and EDTA. It was reported that apple vinegar can remove the smear layer, and have antimicrobial effect against microorganisms that related to root canal infection [24]. In this study apple vinegar was selected as final rinse to evaluate its effect on the tested sealers.

Ever since the introduction of MTA with its superior biological, physical and sealing properties met several modifications to be used as root canal sealer. Endoseal mineral trioxide aggregate was recently introduced as premixed and pre-loaded substance, with adequate working consistency and reduced setting time [25]. Different studies had shown higher bond strength recorded for AH Plus sealer than other types which make it selected as ideal model reference against it bond strength of other sealers can be compared [13,14, 37]. Consequently, the current study was aimed to evaluate the push-out bond strength of AH Plus and Endoseal MTA sealers after using two irrigation of the root canal either with EDTA or apple vinegar as final rinse.

In this study an effort was directed to ensure the clinical simulation and standardization of samples in each group. This was achieved through selection criteria of using freshly extracted human single rooted with nearly root length (16 ± 1 mm), and following the same protocol for root canal preparation, obturation and incubation. Additionally, canals were traditionally obturated using lateral condensation technique to allow direct contact between interradicular dentin walls and the sealer which become adapted to canal shape and penetrate deeply into the dentinal tubules, providing mechanical retention mimic that of clinical case. The samples were randomly distributed into the experimental groups and prepared by single operator to decrease chance of bias [26].

This study results indicated that using AH Plus/ GP showed the highest mean push-out bond strength either after rinsing with EDTA or apple vinegar, this can be attributed to covalent bond formation between bared amino groups in the collagen of dentin and epoxide ring of AH Plus sealer [27].Also the results can be related to good properties of epoxy resin based sealers of long-range dimensional stability, minimal shrinkage during setting, superior flow allowing filling the irregularities and deeper penetration into dentinal tubules. Furthermore, its PH is slightly acidic which may act as self- etching when come in direct contact with radicular dentin that improve adaptation and bonding [28, 29].

The effect of the finishing irrigating solution on the value of bond strength of AH Plus showed that the using of EDTA in **subgroup IA** improved the bond strength compared to apple vinegar in

**subgroup II A**. These results can interpreted on base that using EDTA produce higher demineralization impact which subsequently expose extra collage network that improve AH Plus adhesion, since the covalent bond formed between sealer epoxy rings and the exposed collagen amine groups [30]. EDTA also resulted in higher surface roughness compared to apple vinegar that provide micro-irregularities improving retention of resin tags sealers and increasing the bond strength [31]. The results of the current study were in accordance with result of other studies [31-33].

The results of the study revealed significantly low bond strength results of Endoseal MTA sealer in both **subgroups IB** and **IIB,** these lower records can be due to calcium and hydroxyl ions releasing during inherent setting property, this ions form appetite by MTA when coming in contact to phosphate containing moisture, then deposited in collagen network promoting mineral nucleation forming micro-tags that have low adhesion quality [34]. The study results were in accordance with other studies demonstrated that calcium silicate-based sealers represented weak bond strength to radicular dentin when compared to AH Plus which showed significantly higher bond strength, also recent systematic review and meta-analysis told that bioceramic sealers do not perform superior than epoxy resin sealers with dislodgement resistance test [23, 28,29, 34, 35].

The effect of the finishing irrigating solution on the bond strength of Endoseal MTA showed that the bond strength was significantly higher after using EDTA in **subgroup IB** than that reported in apple vinegar **subgroup IIB**, the results can also interpreted as the same as mentioned in the subgroups IA and IIA of AH Plus of more demineralization effect and higher surface roughness after application of EDTA. These results were in accordance with study reported that the final EDTA irrigation significantly improved the bond strength of Endoseal MTA, however the other study used 7% maleic acid which is one ingredient of apple vinegar [36].

The results regarding the bond strength at different root canal levels, the highest mean values were observed at the middle level compared to coronal and apical levels in all tested subgroups. This can be explained as the average number of the dentinal (on unit area 1mm$^2$) was reported to be lowest number in the middle third compared to both coronal and apical thirds, with subsequent greater intertubular dentin which increase the area of dentin surface available to demineralization effect of the irrigating solutions and bonding to the root canal sealers, it was reported that the dentin surface area might be responsible for enhanced bond strength [38,39].

The null hypothesis of this study was rejected, since AH Plus showed bond strength to the root canal dentin higher than that of Endoseal MTA, and using of EDTA and apple vinegar as final irrigating solution affect the bond strength of both sealers.

## CONCLUSION

Within the limitations of this study.

- The bond strength of epoxy resin AH Plus was higher than that of Endoseal MTA after using either EDTA or apple vinegar as final irrigant.
- Highest bond strength of both AH Plus and Endoseal MTA was observed in root canal rinsed with EDTA.

## RECOMMENDATION

Further studies can be performed to evaluate the tooth fracture resistance in teeth irrigated with apple vinegar and obturated with silicone based sealers.

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