

# Effect of Maleic Acid and Ethylenediaminetetraacetic Acid on the Shear Bond Strength of RealSeal SE Sealer to Root Canal Dentin

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**Abstract** - Aim of this study was to evaluate the effect of 7% maleic acid and 17% ethylenediaminetetraacetic acid (EDTA) on the shear bond strength of RealSeal SE sealer to root canal dentin. Twenty incisors were split into coronal, middle and apical third and were treated in the following manner: Group 1: 5ml of saline (1 minute). Group 2: 2.5% sodium hypochlorite (5ml/min) followed by 7% maleic acid (5ml/min). Group 3: 2.5% sodium hypochlorite (5ml/min) followed by 17% EDTA (5ml/min). Sealer was placed on the root dentin and samples were subjected to bond strength measurement. There was no significant difference in bond strength between maleic acid and 17% EDTA in coronal & middle third. However, in apical third, 7% maleic acid showed higher bond strength. Least bond strength was observed with saline. Bond strength was maximum in apical third for both 7% maleic acid and 17% EDTA.

KEY WORDS: Bonding, EDTA, irrigation, maleic acid, resin sealer

## INTRODUCTION

The goal of root canal therapy is to completely debride and three dimensionally seal the root canal system. Gutta percha in combination with various sealers is one of the most commonly used obturating systems<sup>1</sup>. However, studies have demonstrated on the weak adhesion between gutta percha and the sealers and thereby not providing the bacterial tight seal of the root canal system<sup>2,3</sup>. Recently new type of obturating system known as the Resilon obturation system has been introduced with the claim of providing an effective bond to the root canal dentin. Resilon is used with dual cure resin adhesive sealer known as Epiphany<sup>4</sup>. The first generation of the Epiphany obturation system consisted of a core material (Resilon), dual curable resin-based sealer (Epiphany), and a self-etching primer. Epiphany bonds both to the obturation material as well as the canal walls thus creating a monoblock<sup>5</sup>, which provides a greater resistance to microbial leakage<sup>6</sup> and reinforces the teeth against root fracture<sup>7</sup>. The second generation of new Epiphany self-etch (SE) obturation system, known as RealSeal self-etch (SE), uses a polymerizable methacrylate carboxylic acid/anhydride as the acidic resin monomer has been now marketed. The use of self etch system has an advantage of reducing errors that might occur during bonding steps as well as the application time<sup>8</sup>.

Mechanical instrumentation of the root canal produces an amorphous irregular layer known as smear layer which covers the dentinal tubules<sup>9</sup>. It has been shown that smear

layer hinders the penetration of intracanal medicaments and sealers into the dentinal tubules<sup>10,11</sup>. Removal of the smear layer has been shown to allow greater sealer penetration into the exposed tubules, which may increase the adhesion and seal within the canal space<sup>12</sup>. The smear layer can be removed using various chemical agents like ethylenediaminetetraacetic acid (EDTA), citric acid, phosphoric acid and MTAD which is a mixture of tetracycline isomer (doxycycline), an acid (citric acid), and a detergent (tween 80)<sup>13-15</sup>. Combined application of EDTA and sodium hypochlorite (NaOCl) is commonly used for the effective removal of smear layer from the root canal system<sup>13</sup>. Maleic acid (MA) is a mild organic acid used as an acid conditioner in adhesive dentistry<sup>16</sup>. It has been demonstrated that, final irrigation with 7% MA followed by 2.5% NaOCl is more efficient than 17% EDTA followed by 2.5% NaOCl in the removal of smear layer from the apical third of the root canal system<sup>17</sup>. Studies have demonstrated that various endodontic irrigants may have adverse effect on the bond strength of resinous material to the root canal dentin<sup>18,19</sup>. To date there are no studies which have evaluated the effect of 7% MA irrigation on the bond strength of RealSeal SE sealer to the root canal dentin. Therefore, the aim of this study was to evaluate and compare the effect of 7% MA and 17% EDTA irrigation on the shear bond strength of RealSeal SE sealer to the root canal dentin.

## MATERIALS AND METHODS

### Preparation of specimens

Twenty extracted human maxillary central incisors free of cracks and caries were selected. Ethical review was obtained from the Institutional Review Board, Manipal University. Superficial soft tissues on the root surface were removed with a brush and the teeth were stored in 0.9% saline containing 0.2% sodium azide at 4°C. The teeth were decoronated at the cemento-enamel junction using

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low speed diamond disc. The roots were then randomly divided into two test groups and a control group. In the test groups, each root was divided into two halves with a low speed diamond disc and one half of each root was used for treatment with 7% MA and the other half was used for 17% EDTA. The roots in the control group were also longitudinally split and were used for the treatment with saline (Figure 1). Pulp remnants were removed from the root canal surface using a soft brush. Then, each half of the root in both the test and control groups was further split into coronal, middle and apical third. Each third of the root was then embedded in die stone blocks so that the root canal dentin surface was exposed (Figure 2). A smear layer was created on each specimen by abrading the root canal dentin surface using 400 grit silicon carbide paper on an grinder for 20 seconds.

Specimens were then treated in the following manner:

**Group 1 (Control):** The coronal (n=10), middle (n=10) and apical third (n=10) was irrigated with 5ml of 0.9% saline for 1 min. Then all the specimens were irrigated with 5 ml of distilled water for 1 minute.

**Group 2 (MA group):** The coronal (n=15), middle (n=15) and apical third (n=15) was irrigated with 2.5% NaOCl (5ml/min) followed by 7% MA (5ml/min). Then all the specimens were irrigated with 5 ml of distilled water for 1 minute.

**Group 3 (EDTA group):** The coronal (n=15), middle (n=15) and apical third (n=15) was irrigated with 2.5% NaOCl (5ml/min) followed by 17% EDTA (5ml/min). Then all the specimens were irrigated with 5 ml of distilled water for 1 minute.

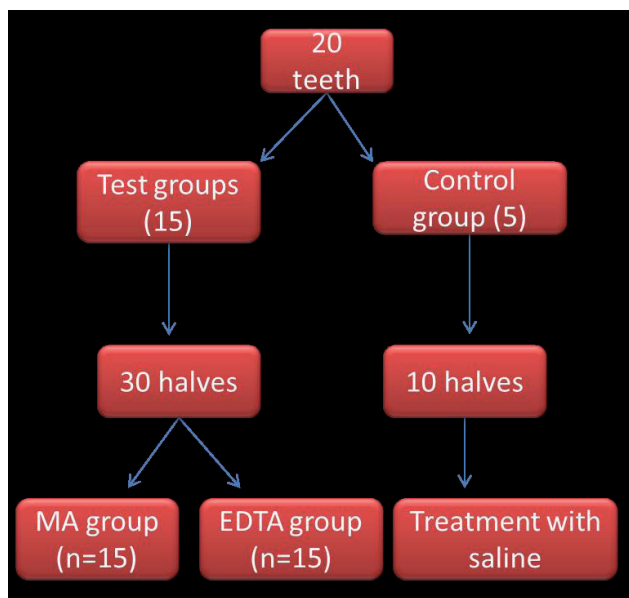
Irrigation in all the groups was performed using 27 gauge stainless steel bevelled needle.

### Bonding Procedure

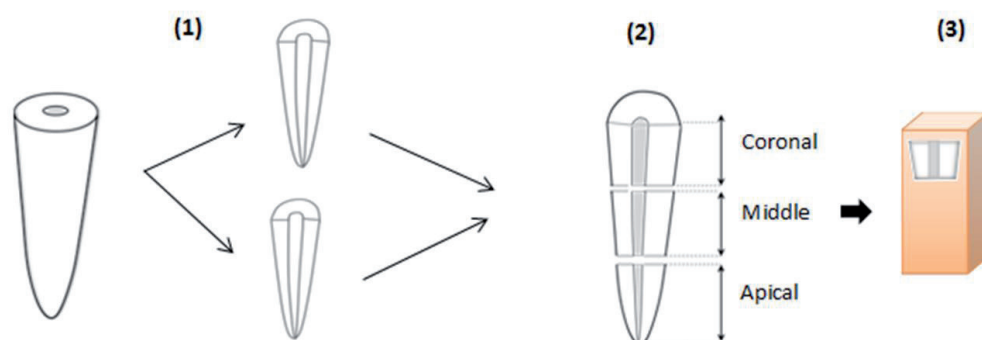
Root canals were dried using paper points. Polyethylene tubes of 1.8 mm internal diameter and 3 mm height were placed on the root canal dentin. RealSeal SE sealer was injected into the tubes with auto mixing syringe tip and light cured for 40seconds using LED lamp (3M ESPE, St. Paul, MN) (Figure 3). The samples were then stored at 37°C at 100% humidity for 7 days to ensure for the complete set of the sealer.

### Shear Bond Strength Evaluation

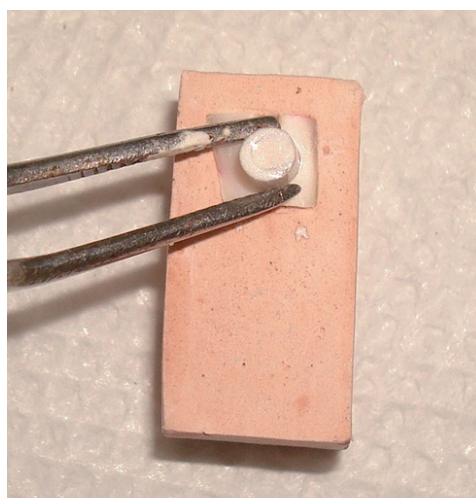
A shear bond test was used in this study because, it has been proven to be reproducible and effective method & provides homogenous results with considerably low variation of bond strength<sup>20</sup>. Also, in shear bond strength measurement, the specimens used will have flat surface which helps in the ease of specimen standardization<sup>21</sup>. Each specimen was attached to a testing jig on universal testing machine. Polyethylene tubes which were placed on the specimens were intact in order to prevent the distortion of the assembly. The assembly was then stressed to shear failure using a chisel blade which was oriented parallel to the dentin surface at a crosshead speed of 1.0 mm/min. Maximum load at debonding was noted and the shear bond strength was calculated in megapascals. Statistical analysis was performed using Kruskal -Wallis and Mann-Whitney U test.



**Figure 1.** Schematic representation of the division of the samples among the various groups.



**Figure 2.** Schematic representation of the specimen preparation for shear bond strength measurement. (1) Longitudinal sectioning of the specimens; (2) Each root half sectioned into coronal, middle and apical third; (3) Each third of the root canal dentin embedded in a die stone block.



**Figure 3.** Photograph demonstrating the placement of polyethylene tubes over root canal dentin.

**Table 1.** Shear bond strength of RealSeal SE in different thirds of root canal dentin after treatment with various irrigants.

	Test irrigants	n	Mean	Standard Deviation
Coronal third	Control	10	0.535	0.280
	EDTA	15	1.192	0.825
	MA	15	1.632	1.525
Middle third	Control	10	0.943	0.472
	EDTA	15	1.626	0.919
	MA	15	2.642	1.918
Apical third	Control	10	0.453	0.381
	EDTA	15	3.642	1.567
	MA	15	4.954	1.622

### Fractographic Analysis

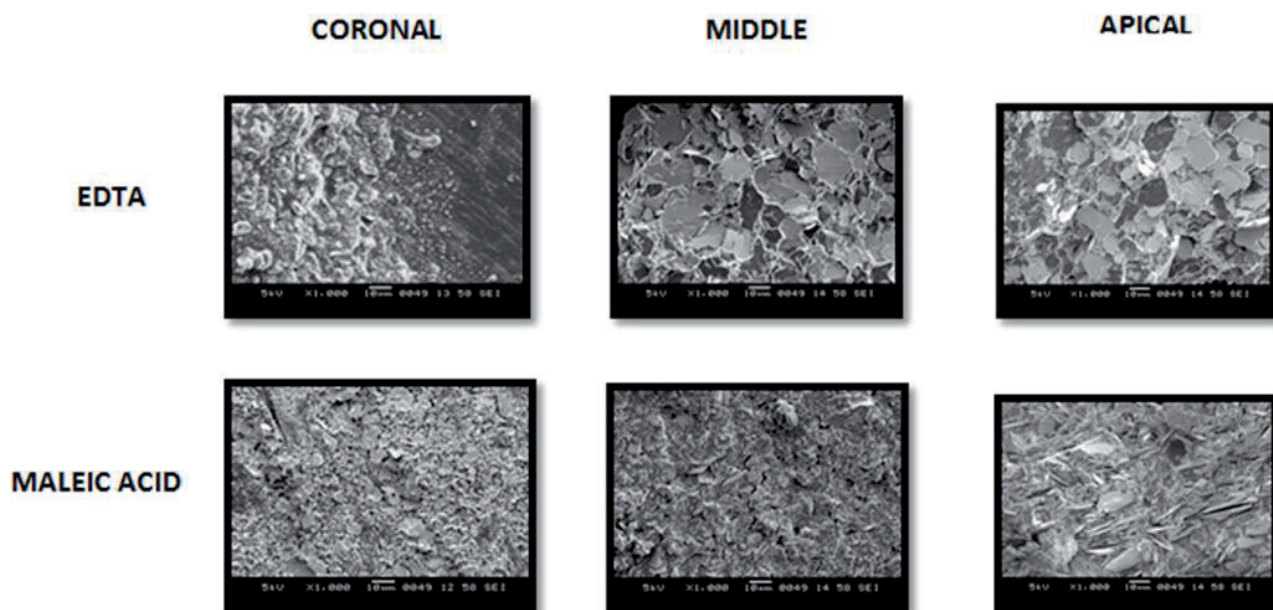
The fracture mode was analysed under a stereomicroscope at a magnification of 10X to identify the mode of failure between dentin- sealer interfaces. The failure modes were classified as (1) adhesive between sealer material and dentin interface; (2) cohesive within the sealer material; (3) mixed failure. Later, two representative fractured specimens from each group were mounted on metallic stubs, gold sputtered using an ion sputter and was examined under scanning electron microscope at 1000X magnification.

### RESULTS

The mean and the standard deviation values of the shear bond strengths for the different groups are presented in Table 1. The results demonstrated that, there was no significant difference in the shear bond strength between 7% MA and 17% EDTA in the coronal ( $p=0.56$ ) and middle third ( $p=0.35$ ). However, in the apical third there was a statistical significant difference between 7% MA and 17% EDTA ( $p=0.04$ ), in which 7% MA irrigation resulted in better bond strength than irrigation with 17% EDTA. Saline which was used as a control demonstrated the least bond strength of all the groups. Large standard deviation seen in the between the groups may be attributed to the smaller sample size used in this study.

While comparing the different thirds of the root canal system for the bond strength measurement, the apical third showed higher shear bond strength compared to the coronal third for both 7% MA and 17% EDTA groups.

The analysis of the specimens under scanning electron microscope for the failure mode demonstrated the bond failure in the coronal, middle and apical thirds of the root canal to be predominantly cohesive for all the groups (Figure 4).



**Figure 4.** Scanning electron microscopic images showing cohesive mode of failure in MA and EDTA treated groups wherein all the dentinal tubules are covered with the sealer.

## DISCUSSION

Achieving predictable bonding to the root canal dentin has long been a goal and challenge in endodontics. Among the resin based sealers, both the 2<sup>nd</sup> generation Endo Rez system and 4<sup>th</sup> generation RealSeal SE sealers may not bond well to radicular dentin if the smear layer is not removed<sup>8</sup>. Several studies have reported that, the removal of the smear layer improves the penetration of the sealers into the dentinal tubules and thus enhances the bond strength to root canal dentin<sup>22, 20</sup>. The results of the present study demonstrated that, 7% MA group had a better bond strength than 17% EDTA in the apical third. This could be attributed to the superior smear layer removal quality of 7% MA in the apical third of the root canal system<sup>17</sup>. It can also be attributed to the increased surface roughness produced by MA compared to 17% EDTA<sup>23</sup>, since micromechanical bonding of resin sealers requires the presence of surface irregularities. In the present study, the shear bond strength values were found to be higher in the apical third in both the test groups. This could be related to the increased area of solid intertubular dentin present in the apical third than the middle and coronal part of the root canal system<sup>24</sup>. 7% MA was used in this study because higher concentrations of MA have shown to have an erosive effect on root canal dentin<sup>25</sup>. In this study, irrigation time was standardized for one minute in all the groups since it has been reported that, irrigation with 17% EDTA for more than 1 min causes excessive peritubular and intertubular dentinal erosion<sup>26</sup>. NaOCl was not used as a final irrigant because studies have reported that, NaOCl is an oxidizing agent and thus it produces an oxygen rich layer on the dentin surfaces<sup>18, 27</sup>. This layer can inhibit the polymerization of the resin and interfere with the penetration of the resin into the dentinal tubules and thus may affect the sealer-dentin bond strength<sup>28</sup>.

In this study, all the teeth used were of similar nature and morphology (maxillary central incisors) so that, their degree of mineralization is similar, which in turn may affect the bond strength of the resin sealer. It has been reported that, the degree of residual moisture present in the root canal could decrease the conversion of the monomer, thus leading to incomplete polymerization of the resin and significant reduction in the bond strength<sup>29</sup>. Hence, in the present study, the canals were blot dried using paper points until the last paper point was observed completely dry. Air-water syringe was not used for drying the canal because it has been demonstrated that, desiccation of the root canal by air water spray may inhibit the penetration of the hydrophilic sealers and may compromise the bond strength<sup>30</sup>. As the resistance to the dislocation of the sealer increases, the disruption of the sealer dentin interphase becomes less and the failure is more likely to occur within the sealer itself. In the present study, the mode of failure observed with MA and EDTA was found to be predominantly cohesive in all the thirds of the root canal system. This could be attributed to the complete bond formation at the resin/dentin interphase.

## CONCLUSION

Based on the results obtained and experimental conditions of the present study, it can be concluded that irrigation with 7% MA can increase the shear bond strength of RealSeal SE

sealer to the apical third of the root canal dentin compared to 17% EDTA. Shear bond strength of RealSeal SE sealer was observed to be maximum in the apical third of the root canal system for both 7% MA and 17% EDTA irrigation.

## MANUFACTURER DETAILS

- Resilon Research LLC, Madison, CT
- RealSeal SE, Sybron Endo, Glendora, CA, USA
- Sodium azide, Sigma Chemical Co, St. Louis, MO
- Diamond disc, Horico, Germany
- Grinder, Buehler Ltd, Lake Bluff, IL
- Paper points, Dentsply-Maillefer, Ballaigues, Switzerland
- LED lamp, Fast cure, DP Corp, Florida, USA
- Universal testing machine, Instron 3366, UK
- Stereomicroscope, Zeiss, Thornwood, NY, USA
- Scanning electron microscope, LEO 440i, Tokyo, Japan
- Endo Rez system, Ultradent, South Jordan, UT

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