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Does MTA and Calcium Hydroxide Additives Effect on the Bond Strength of AH Plus Sealer?

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Research Article

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ABSTRACT

The aim of the this study was to evaluate the bond strength of the canal sealers formed experimentally by adding different levels of MTA and $Ca(OH)_2$ into AH Plus, and comparing with unaltered AH Plus, Total Fill and MTA Fillapex. 91 single-rooted human maxillary central teeth were prepared, with the following sealer type evaluated for bond strength (n=70): AH Plus; MTA Fillapex; TotalFill; and four experimental sealers (AH plus+ 5% $Ca(OH)_2$; AH plus+ 5% MTA; AH plus+ 10% $Ca(OH)_2$; AH plus + 10% MTA) (n=10). Three slices of 1 mm thickness were obtained from each root sample, and the bond strength of the test materials was measured using a push-out test. The data were analysed using one-way ANOVA and post hoc Tukey tests (p=0.05). The highest and lowest mean push-out values were obtained for AH plus+10% MTA and MTA fillapex groups, respectively ($p<0.05$). No statistically significant difference was found between AH-plus and the experimental sealers ($p>0.05$). It was observed that adding 5% or 10% of $Ca(OH)_2$ or MTA to AH-plus had no negative effect on the bond strength of AH plus.

INTRODUCTION

Innovations in the field of endodontics are now focused on how to achieve greater success in root canal treatment. So, there have been many studies conducted on root canal sealers used in endodontic treatment.

Biocompatibility, sealability, antibacterial property and radiopaque quality are among the required characteristics of an ideal root canal sealer [1-3]; although that ideal sealer does not yet exist [4], AH Plus is regarded by researchers to be at the gold standard level, with a high preferability rate [5-7]. Furthermore, some researchers have added different substances to AH Plus to improve its biocompatibility, toxicity and antimicrobial characteristics [8-10]. Duarte et al. [8] evaluated the amount of alkaline pH and Ca released by adding calcium hydroxide ($Ca(OH)_2$) into AH Plus at rates of 5% and 10%; their results showed that the addition of $Ca(OH)_2$ caused an increase in sealer alkalinity and Ca ion- release. Another study investigated whether or not $Ca(OH)_2$ added at rates of 5% and 10% caused any change in the physical properties of AH Plus [9]. It found that there was no significant change in the radiopacity, flowability, setting time, solubility or dimensional changes in AH Plus with the addition of 5% $Ca(OH)_2$, while a 10% $Ca(OH)_2$ addition affected the other physical properties of AH Plus, except for radiopacity and setting time [9]. Arias-Moliz et al. [10] researched the effect of benzalkonium chloride on the physical properties of AH Plus by adding different amounts of ben-

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zalkonium chloride into AH Plus in order to enhance its antibacterial efficiency. They reported that adding benzalkonium chloride altered the sealer's physical properties by increasing its antibacterial effect prominently [10].

In recent years, the use of new-generation root canal sealers with Mineral Trioxide Aggregate (MTA) and ceramic content has become quite popular, due to positive characteristics such as high biocompatibility and bioactivity [5,11]. According to manufacturer, MTA Fillapex canal sealer contains salicylate resin, natural resin, bismuth trioxide, nanoparticle silica, and MTA. It is a biocompatible canal sealer with antibacterial and hydrophilic characteristics, with high flow ability and low film thickness [12]. Kuga et al. [13], in a study evaluating pH and Ca ion-release and flow ability by adding 5% and 10% of $Ca(OH)_2$ into MTA Fillapex with a high level of flow ability, found that adding 5% $Ca(OH)_2$ increased the flow ability of the sealer to a level acceptable under ISO 6876:2001 specifications.

Total Fill root canal sealer with calcium silicate content is the equivalent of Iroot SP and Endosequence BC sealers; it has similar characteristics to white MTA in terms of content [14], in addition to which it contains calcium silicates, calcium phosphate monobasic, zirconium oxide, tantalum oxide and thickening agents [15]. It is a biocompatible root canal sealer with a high level of antimicrobial properties and osteoinductive quality that is never affected by the humidity of the dentin [15].

The changes that occur within the physical properties of AH Plus and MTA Fillapex root canal sealers with the addition of $Ca(OH)_2$ have been analysed in several studies. However, to the best our knowledge, there is available data in the literature that evaluates the effect of adding MTA or $Ca(OH)_2$ to AH Plus on bonding sealers to dentin. Therefore, this study evaluated the bond strength of canal sealers formed experimentally by adding different levels of MTA and $Ca(OH)_2$ to AH Plus and comparing them with unaltered AH Plus, Total Fill, and MTA Fillapex canal sealers. The null hypothesis of the study is that there is no difference between the bond strengths of the sealers.

MATERIALS AND METHODS

Tooth Selection and Specimen Preparation

In this study, a total of 91 human single-rooted and single-canal maxillary central teeth without decay, of similar root lengths, and recently extracted for periodontal reasons, were used. The teeth were washed under water, with the debris that remained on the surface of the root cleansed with the help of a periodontal curette without harming the teeth's root surfaces. The teeth were then kept in thymol at 25°C until use in the study. Whether the apical foramens of the roots were open or not was controlled through the use of a 10 K-type file (Dentsply Maillefer, Ballaigues, Switzerland). Following this process, the crown of the teeth were cut from the cementoenamel junction so that the roots would remain 15 ± 0.5 mm. The root canals were prepared with Resiproc 40 (VDW, Munich, Germany) by using the Resiproc All program on VDW Silver (VDW, Munich, Germany). After the root canals were prepared, 5 ml of a 5.25% NaOCl solution were applied to the canals. In order to remove the organic and inorganic structure of the smear layer, the canals were washed for one minute with 5 ml of 17% EDTA and 5 ml of 5.25% NaOCl solutions. For the final irrigation process, 5 ml of distilled water was used to eliminate debris within the root canals and the effect of the irrigation solutions. The root canals were dried with paper points (Spident, NamDongKongDon, Incheon, Korea).

After preparation, the teeth were randomly divided into seven groups (n=10) according to the canal sealer to be used for obturation: Group 1: AH Plus (Maillefer, Dentply, Konstanz, Germany); Group 2: MTA Fillapex (Angelus, Londrina, PR, Brazil); Group 3: Total Fill (Brasseler USA, Savanah, GA, US); Group 4: AH Plus+5% $Ca(OH)_2$ (Kalsin; Spot Dental, Izmir, Turkey); Group 5: AH Plus+ 5% MTA (Angelus, Londrina, PR, Brazil); Group 6: AH Plus+10% $Ca(OH)_2$; and Group 7: AH Plus+10% MTA.

AH Plus, MTA Fillapex and Total Fill canal sealers were prepared according to the manufacturers' instructions. After the amount of AH Plus to be used in Groups 4 through 7 was measured, $Ca(OH)_2$ or MTA amounting to either 5% or 10% of the sealer weight was added, after which those experimental sealer samples were prepared. The sealers were placed into the root canals using a size #40 Lentulo spiral. The main cones Resiproc 40 were covered with the sealer and placed into the canal. The gutta-percha that remained outside the canal was cut away with the help of a heated instrument. The access cavities were closed with a temporary filling material (Cavit G, 3M ESPE, US), and the teeth were kept in a 100% humidity environment at 25°C for one week. As for the samples embedded within the cold acryl, three pieces of horizontal sections of 1 mm thickness were taken from the middle of each sample towards the coronal (6 mm from the apex) with a microcut device (Metkon, Microcut Precision Cutter, Bursa, Turkey) at low speed by means of water cooling. 30 samples were obtained for each experimental group (n=30).

Push-out Examination

The push-out test was applied using a universal testing machine (Instron, Norwood, MA, USA). A cylindrical plugger was used with a continuous load to each specimen. A 1 mm min^{-1} crosshead loading speed was applied in the apico-coronal direction until bond failure occurred. After dislodgement, the maximum load applied was recorded in newtons and converted to megapascals (MPa) by using the formulation described by El-Ma'aita AM et al. [16].

After the push-out bonding test was applied, the samples were examined under a stereomicroscope (Nikon, Tokyo, Japan) with 25X magnification to determine failure types, which were classified in the way that was described by Huffman et al. [17]. Adhesive failure: in the surface of the sealer-dentin; Cohesive failure: within the sealer; Mixed failure: involves both detachment from the wall of the dentin and detachments found within the sealer itself.

The data were analysed using IBM SPSS 20 with one-way variance analysis (ANOVA) and post hoc Tukey tests. The statistical significance level was determined as 0.05.

Confocal Laser Microscope (CFLM) Analysis

The remaining teeth (n=21) were separated into 7 groups (n=3) according to the canal sealers to be used for viewing the relationship between the canal sealers and the dentin. In order to provide a fluorescent characteristic for the sealers, a few grains of Rhodamine B dye (Sigma-Aldrich, St Louis, MO) were added to a level of ~0.1% [18]. The canal fillings were completed as described above. Following the setting process of the sealers, a section of 1 mm thickness was taken from the middle third of each sample. Later, the sections were polished with silicon carbide abrasive papers.

The samples were examined through a CFLM (Zeiss LSM 510, Carl Zeiss, Göttingen, Germany). An argon laser was used as the light source with a wave length of 543 nm. The images were recorded at 512X512 resolution through a 10X magnification. The recorded images were examined after being transferred into the Zeiss LSM Image Browser v.4.2.0 program (Carl Zeiss Micro Imaging GmbH, Jena, Germany).

RESULTS

The push-out bond strength values are presented in **Figure 1**. The highest and lowest mean push-out values were obtained for the group AH Plus+10% MTA and MTA Fillapex (P<0.05). No statistically significant difference was found between AH Plus and the experimental sealers (P>0.05). When the experimental sealers were compared with one another, no statistically significant difference was found (P>0.05); when they were compared with Total Fill and MTA Fillapex, the experimental sealers showed statistically significant higher bond strength values (P<0.05). The bond strength of AH Plus was higher than Total Fill and MTA Fillapex (P<0.05). There was no difference between Total Fill and MTA Fillapex statistically (P>0.05). **Figure 2** shows the distribution of failure types according to the groups. Cohesive failure types were seen most commonly in the AH Plus and MTA Fillapex groups, while adhesive failure types were most often seen in the Total Fill group; mixed failure types were observed most commonly in the experimental sealers. The relationship of the sealers with the dentin structure in the CFLM images is shown in **Figures 3 and 4**.

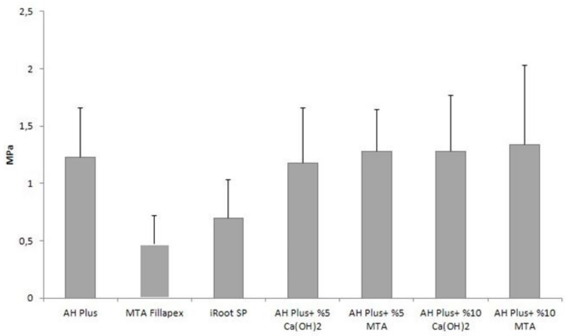

Figure 1. The values (MPa) indicating the bond strength of the sealers with the walls of the root canal dentin along with the standard deviation.

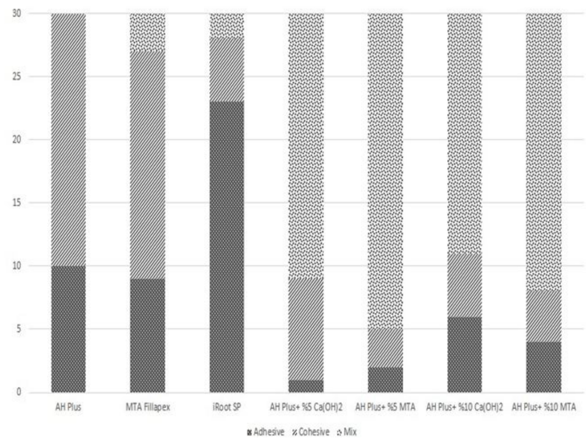

Figure 2. The distribution of adhesive, cohesive and mixed failure types according to the sealers.

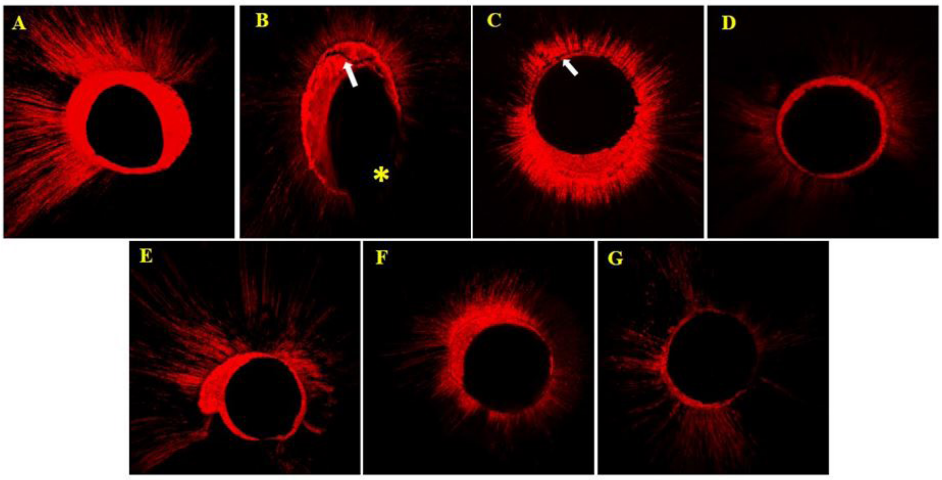

Figure 3. The CFLM images of the penetration of the sealers, (A) AH Plus, (B) MTA Fillapex, (C) Total Fill, (D) AH Plus+5% $Ca(OH)_2$, (E) AH Plus+5% MTA, (F) AH Plus+10% $Ca(OH)_2$, and (G) AH Plus+10% MTA, into the dentin (X10). The white arrow given in (B) indicates to the gap area seen within MTA Fillapex sealer, while *, however, shows the weak adaptation area, and the white arrow in (C) indicates to the gap area seen between TotalFill sealer and the dentin.

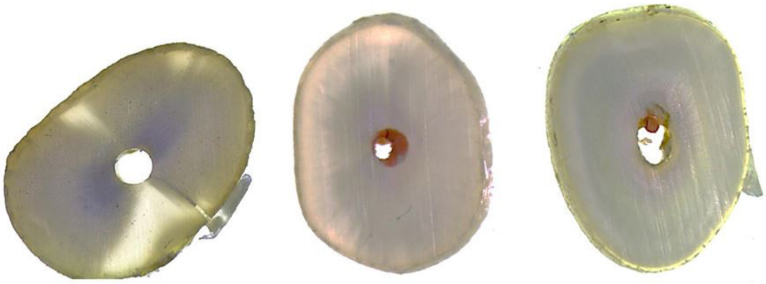

Figure 4. Failure modes for the experimental groups.

DISCUSSION

The adhesion of root canal sealers to the dentin structure is a required characteristic for filling materials [19]. Such adhesion is necessary to eliminate leakage and provide resistance of the material to displacement forces that occur while undergoing condensation of permanent restorative materials [19,20]. A number of factors, such as the intermolecular surface energy of the dentin structure, surface tension of the sealers and wetting capability, may affect the properties of adhesion [21].

The penetration of the canal sealers into the dentin tubules, accessory canals and isthmi is of importance in terms of eliminating the risk of re-infection caused by bacteria remaining within these anatomic structures [22,23]. The mechanical interlocking caused by the extensions of the sealer within the dentin tubules may increase the sealing ability and retention of the material [23-25]. However, contrary to this common belief, no positive correlation could be established between seal ability and the penetration of the sealers into the dentin tubules [26].

The smear layer formed in the course of the root canal preparation, which may become infected later; negatively affect the penetration of irrigation agents and sealers into the dentin tubules [21]. It was reported that the use of NaOCl and EDTA had effectively removed both the organic and inorganic content of the smear layer and the pulpal tissue residues [27,28]. Additionally, EDTA reduces the surface energy of the dentin texture, which has two different substrates—hydroxyapatite with high surface energy and collagen with low surface energy—and increases its wetting capacity [29-32]. For this reason, the combination of 5.25% NaOCl and 17% EDTA was used in the present study to remove the smear layer.

Due to the fact that the dentin canals showed differences in number and size in different parts of the root and that removing the smear layer from the apical parts of the root was more difficult than with other parts, the adhesion in question was evaluated by choosing a section only from the middle third of the root, in order to eliminate the anatomical differences in this study [33].

According to the results of the present study, the addition of $Ca(OH)_2$ or MTA into AH Plus did not change the bond strengths in a statistically significant when compared with unaltered AH Plus sealer. The bond strength of AH Plus and the experimental sealers proved to be greater in a statistically significant way than Total Fill and MTA Fillapex sealers. In line with these findings, the null hypothesis of the study was rejected.

As reported by Sousa-Neto [34], the fact that AH Plus demonstrated highly chemically-connective behaviour towards the dentin molecules may have been the result of the covalent bonding of epoxy links to the organic phase of the dentin (the collagen amine groups released in the dentine). Previous studies have emphasized that the high bond strength levels of AH Plus could be due to the low polimerization stress of the sealer and long-term dimensional stabilization [35,36]. In contrast to the sealers with alkaline characteristics and bioceramic content [37], the mildly acidic structure of AH Plus may cause self-etching during the contact between the sealer and the dentin [32]; if so, the present study's results reinforce the connection and adaptation of AH Plus with the dentin [38]. Hence, a convenient medium is achieved for the adhesion of hydrophobic materials like AH Plus. The hydrophobic structure of AH Plus may have provided effective micro-bonding and made it easy for the resin to penetrate into the dentin tubules [39].

In this study, although no statistically significant difference was found between the bond strengths of Total Fill and MTA Fillapex canal sealers, they were found to have lower levels than AH Plus. This result accords aggrement with Oliveira et al. [40] and Amin et al. [41] Some researchers have suggested the fact that the reason that bioceramic-based sealers with hydrophilic structures, the setting process and adhesive characteristics of which change in the presence of humidity within the canal, exhibit low adhesion to the dentin may be due weakening of the wetting capacity of the dentin surface [42,43]. However, there are also some studies in which the bond strength values of bioceramic-based sealers show differences. Ersahan et al. [2] and Sagsen et al. [44] found that the bond strength of Iroot SP sealer showed similarity to AH Plus. On the other hand, Nagas et al. [15], in a study evaluating the bond strengths of several sealers in different humid environments, determined that Iroot SP possessed more bond strength than AH Plus and MTA Fillapex. This discrepancy in study results may be due to differences in experimental design, such as sealer brands, filling procedures and examination periods.

MTA Fillapex canal sealer showed lower bond strength than all the experimental groups. The fact that the bond strength of MTA Fillapex proved to be lower in comparison to the other sealers is similar to results in studies [5,44]. The Ca and OH ions released due to MTA content in the course of the setting process of MTA Fillapex create an apatite formation [45], which reduces the connection of the sealer by functioning as an interface that hinders bonding between the dentin surface and the tag-like structure of the sealer [44]. In light of this information, the fact that MTA Fillapex exhibited low bonding characteristics in this study may be due to the resin structure of MTA Fillapex and the high level of MTA content [46]. On the other hand, the fact that the bond strength of the experimental sealers formed by adding 5% and 10% MTA to AH Plus was higher than MTA Fillapex may be due to the fact that the amount of MTA added to AH Plus was notably lower than the MTA amount within present in MTA Fillapex.

Duarte et al. [9] showed that 5% $Ca(OH)_2$ addition did not affect the physical properties of AH Plus. Based on this fact, depending on the unaffected physical properties of AH Plus in this study, the bond strength between AH Plus and the experimental groups into which 5% $Ca(OH)_2$ or 5% MTA was added led to similar results. A previous study, however, suggested that 10% of $Ca(OH)_2$ addition did increase the film thickness of AH Plus while reducing its fluidity [9]. While a low level of film thickness is generally preferred for the long-term sealing ability of root canal sealers [47], this is not so for AH Plus canal sealer [48]; it has been reported that when the film thickness of AH Plus increases, its antibacterial activity also increases, and there is reduction in its volumetric shrinkage. In a study conducted by Rahimi et al. [49], that evaluated the effect of different canal sealers' film thicknesses on the bond strength, it was shown that increases in the film thickness of sealers with epoxy resin-content, such as AH Plus, also enhanced the bonding of the sealer to the dentin. In this study, the reason why the highest bonding was seen within the group in which AH Plus + 10% MTA was added could be due to the increase in the film thickness.

In our study, three samples from each group were examined on CFLM to explore the relationship of the sealers with the dentin structure. In these images, the gap-containing areas seen within MTA Fillapex sealer clarify why the cohesive failure type is the most common failure within this group. In the Total fill group, however, the gap areas seen between the sealer and the dentin may be the cause of adhesive bonding failure. According to researchers, these gaps between dentin and sealer are formed as the result of the detachments that occur when the sealer penetrating into the dentin canal shrinks in the course of the setting process [50,51]. Al-Haddad et al. [43] showed that sealers with bioceramic content had formed more gap-containing areas than AH Plus in their confocal study. These gaps, which were also seen in confocal samples in the present study, may negatively affect the bond strength of the samples. The CFLM analysis of the samples provided an insight into the adaptation of sealers to the dentin wall.

CONCLUSION

It was observed that adding 5% or 10% $Ca(OH)_2$ or MTA with superior biological properties into AH Plus had no negative effect on the bond strength of the sealer. Future studies can be conducted to analyse antibacterial properties and biocompatibility quality for the clinical use of the experimentally prepared sealers employed this study.

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