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# Effect of Non-thermal Plasma Therapy on Pushout Bond Strength of Epoxy Resin and Tricalcium Silicate-**Based Endodontic Sealers**

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#### Abstract

Introduction: The current study aimed to assess the effect of non-thermal plasma (NTP) on the pushout bond strength (PBS) of epoxy resin and tricalcium silicate-based endodontic sealers. Methods: Forty single-canal extracted teeth were decoronated at the coronal region, underwent root canal preparation, and were assigned to four groups (n=10) for the application of AH26 sealer, NTP+AH26 (P-AH26), Endoseal TCS sealer, and NTP+Endoseal TCS sealer (P-TCS). The root canals were sectioned into 1 mm slices, and the PBS value was measured in a universal testing machine. Data were analyzed by the Friedman, Kruskal-Wallis, and Dunn tests (P < 0.05). **Results:** The PBS of TCS and P-TCS groups was not significantly different (P > 0.05). The PBS of the P-AH26 group was significantly lower than that of the AH26 group in the middle third (P < 0.05). The PBS of the AH26 group was higher than the other groups in all sections. The PBS in the apical third was lower than other sections in all groups (P < 0.05).

Conclusion: NTP had no significant effect on the PBS of Endoseal TCS. NTP significantly decreased the PBS of AH26 sealer in the middle third but had no significant effect on its bond strength in other sections.

Keywords: Epoxy resin AH26; Root canal sealants; Calcium silicate; Non-thermal atmospheric pressure plasma.

# Introduction

The primary objective of root canal therapy is to prevent reinfection by eliminating microorganisms and their metabolites from the root canal system.<sup>1</sup> Obturation, disinfection, and preparation of the root canal system are critical to the efficacy of root canal therapy.<sup>2</sup> However, root canal instrumentation along with the application of chemical agents such as irritants cannot eliminate the microorganisms from the root canal space due to the presence of complexities, isthmi, and accessory canals.<sup>3</sup> Considering the limitations of irrigating solutions and the development of novel techniques, such as plasmas, for root canal disinfection, non-thermal plasma (NTP) was recently proposed to ensure microbial eradication from the root canal system.4

Plasma is the fourth state of matter and a form of ionizing gas that contains high amounts of highly reactive particles such as free radicals, electrons, and ions.<sup>5,6</sup> NTP alters the surface energy of materials and increases the

bond strength to surfaces subjected to plasma therapy.<sup>7</sup> Previous studies showed the positive effects of NTP on dentin-adhesive bond strength through exposing the functional groups of superficial dentin collagens, eliminating the smear layer, and enhancing the surface properties.<sup>8,9</sup> Dentinal tubules have shown improved hydrophilicity and increased penetration depth of adhesives following plasma therapy.10

Root canals are obturated after instrumentation and disinfection. Root canal obturation and provision of a coronal and apical seal are important to prevent microleakage and reinfection, which can lead to treatment failure. Gutta-percha plus sealer is the most frequently used root filling material for the obturation of root canals.<sup>11</sup> An ideal sealer should entirely cover the root canal walls and bond to dentinal walls and guttapercha.<sup>12</sup> In general, the higher bond strength of a sealer to dentinal walls decreases the microleakage and increases the stability of root filling material.<sup>13</sup> Considering the fact

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that different root canal disinfection systems can alter the physicochemical properties of dentin and the penetration depth of a sealer, they may be able to affect the bonding property of endodontic sealers as well.<sup>14</sup>

At present, different sealers are used for root canal obturation. AH26 (Dentsply DeTrey, USA) is an epoxy resin-based endodontic sealer. It has favorable properties such as insignificant polymerization stress, low solubility, antimicrobial activity, biological properties, optimal flow, and high bond strength to dentin.<sup>15</sup> Endoseal TCS (Maruchi, South Korea) is a tricalcium silicate-based endodontic sealer with optimal properties such as suitable flow and enhancement of biomineralization.<sup>16</sup>

Considering the limited number of studies available on the bond strength of sealers to root dentin after plasma therapy and the gap of information regarding the properties of the Endoseal TCS sealer, the present study was conducted to find an efficient method for the preparation of the root canal system and achieve the highest bond strength of a sealer to dentin and favorable outcomes in endodontic treatment.

# **Materials and Methods**

# Sample Selection

This study was conducted with the ethical approval code IR.SBMU.DRC.REC.1400.047. A total of 40 extracted non-traumatized single-rooted and single-canal human maxillary central incisors with straight roots, no calcification, minimum root length of 15 mm, and closed apices were selected. Exclusion criteria included teeth exhibiting resorption defects, fractures, cracks, or extensive root caries. The root surface was cleaned with a periodontal curette, and the teeth were kept in room-temperature distilled water prior to use.

# **Root Canal Preparation**

The teeth underwent decoronation using a thin sectioning machine (Hamco Machines Inc., Rochester, NY, USA) under water spray to standardize the root length at 15 mm. The working length of each tooth was considered 1 mm shorter than the length of a #15 K-file introduced into the canal until its tip was visible at the apex. The instrumentation of all canals was conducted with a ProTaper rotary system with an endo-motor (E-CONNECT pro, Eighteeth, UAE) and SP1 Gold (China) rotary file to F3 by the crown-down technique. The canals were rinsed with 1 mL of 5.25% NaOCl (Hyponic NaOCL 5.25%, Nik Darman, IRAN) injected into the canal. To eliminate the smear layer, 3 mL of 17% EDTA (Canasol EDTA 17%, Nik Darman, IRAN) was injected into the canal and activated by a #10 K-file for 1 minute. To eliminate the EDTA from the root canal structure, 1 mL of distilled water was used to rinse the canals. Finally, 1 mL of NaOCl was injected into the canals and 5 mL of distilled water was used as final irrigation. Next, #40 gutta-percha with .04 taper was inserted into each canal as a master apical cone, and its 15 mm length and tugback were ensured.

# **Grouping of Specimens**

The teeth were randomly divided into the following 4 groups:

- AH26 group: The root canals in this group were obturated with gutta-percha and AH26 sealer.
- P-AH26 group: The root canals in this group underwent plasma therapy and were then obturated with gutta-percha and AH26 sealer.
- TCS group: The root canals in this group were obturated with gutta-percha and Endoseal TCS sealer.
- P-TCS group: The root canals in this group underwent plasma therapy and were then obturated with gutta-percha and Endoseal TCS sealer.

# Plasma Therapy

The argon plasma jet device with CiTO source (CiTO Corp., Stolberg, Germany) was used for plasma therapy with 2.5 bar pressure, 5 L/min flow rate, and 60 W power. The distance between the canal orifice and the plasma tip was 5 mm. Each tooth underwent plasma irradiation for 30 seconds.<sup>17</sup>

# **Root Canal Obturation**

The root canals were obturated with the single-cone technique. Endoseal TCS and AH26 sealers were injected into the respective root canals in 0.15 mL volume, and then a master apical cone was inserted to the working length into the canal. All canals then underwent periapical radiography to ensure optimal root canal obturation.

Gutta-percha was cut at the orifice by using a heat carrier, and Cavit temporary restorative material (Cavisol, Golchai, Iran) was applied at the canal orifice to create a coronal seal. The teeth were then incubated at 37 °C and 100% moisture for 2 days.

# Specimen Preparation for the Pushout Bond Strength Test of Sealers

The roots were mounted in a container containing acrylic resin. Next, sections were made in each root at 4, 8, and 12 mm from the apex with 1 mm slice thickness by using a cutting machine (Mecatome T210, Presi, France). The specimens were then incubated at 37 °C for 24 hours.

# **Pushout Bond Strength Test**

The pushout bond strength (PBS) was measured by a universal testing machine (Zwick Roell, Germany). Each slice was placed on the metal slab of the machine in such a way that its apical surface faced the piston tip. Next, the sealer-dentin interface was subjected to a vertical downward load with a crosshead velocity of 0.5 mm/

min until debonding happened. The graph of the applied load was drawn for each specimen by the software (Zwick Roell, Germany) of the universal testing machine. To calculate the PBS, the following formula was used:

Bond Strength (MPa) =  $\frac{\text{Debonding force (N)}}{\text{Bonded surfasce area (mm<sup>2</sup>)}}$ 

The bonded surface area was calculated using the formula below:

#### Bonded surface area: $\pi$ (R + r) h

where R is the coronal section radius, r is the apical section radius, and h is the thickness of each specimen.

# Mode of Failure

A stereomicroscope (SMZ 800; Nikon, Japan) was utilized to examine both sides of each specimen at x20 magnification to ascertain the mode of failure. According to the location of failure, each section was allocated to one of the three categories listed below:

(I) Adhesive failure: when there was no sealer remaining on the root canal walls.

(II) Cohesive failure: when the walls of the root canal were entirely covered with sealer.

(III) Mixed failure which was the occurrence of adhesive and cohesive failures in combination: when the sealer was left as separate spots in areas on the root canal wall.

# **Statistical Analysis**

SPSS version 26 (Chicago, IL, USA) was utilized to analyze the data. The data were assessed for normal distribution using the Kolmogorov-Smirnov test. The non-parametric Kruskal-Wallis test was employed to compare the PBS of the four groups due to the non-normal distribution of the data. The Friedman test was applied to compare the PBS of different sections of the canal within each group, while the Dunn test was used for pairwise comparisons of the groups regarding the PBS. A descriptive percentage report on the mode of failure was provided.

#### Results

The Kruskal-Wallis test showed a significant difference in PBS between the groups in each of the apical, middle, and coronal thirds (P<0.05).

In the coronal section, the difference in PBS was not significant between the AH26 and P-AH26 groups. AH26 and P-AH26 showed significantly higher PBS values than the TCS and P-TCS groups. The difference between the TCS and P-TCS groups was not significant (P>0.05).

In the middle third, the AH26 group showed significantly higher PBS than the other groups (P < 0.05). The difference in PBS was not significant between the remaining three groups (P > 0.05).

In the apical third, no significant difference was found

in the PBS of AH26 and P-AH26. The difference between the TCS, P-TCS, and P-AH26 groups was not significant either.

A significant difference was found in PBS in the apical, middle and coronal thirds within each of the AH26, P-AH26, and TCS groups (P < 0.05). In the AH26 group, the PBS in the apical third was significantly lower than that in the coronal and middle thirds (P < 0.05). In the AH26, P-AH26, and TCS groups, the difference in the PBS of coronal and middle thirds was not significant (P > 0.05). Table 1 presents the median PBS of the groups in different sections.

Figure 1 shows the frequency distribution of different modes of failure in the study groups. In the AH26 and P-AH26 groups, the cohesive mode of failure was the dominant mode in all sections. In the TCS and P-TCS groups, the dominant mode of failure was cohesive and mixed.

#### Discussion

The application of NTP is a novel topic in endodontics. The efficacy of this technology as an adjunct to root canal preparation (due to its antimicrobial properties) has been previously studied; however, studies regarding its effect on the bond strength of endodontic sealers are scarce.<sup>18</sup> This study assessed the effect of NTP (argon) on the PBS of two endodontic sealers.

The PBS test was used in the present study since it enables equal stress distribution parallel to the dentinsealer interface in dentin discs, and allows the assessment of bond strength in different segments of the root canal system.<sup>17</sup>

In the current study, dentin discs with 1 mm thickness were used to decrease the risk of fracture and wear of specimens. According to Chen et al,<sup>19</sup> the ratio of pin diameter to root canal diameter should be between 0.6 to 0.85. Thus, the pin diameter was selected to be 0.75 mm for coronal and middle sections, and 0.35 mm for apical sections in the present study.

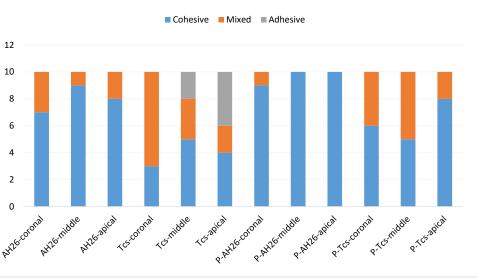
 $\label{eq:comparison} \begin{array}{l} \textbf{Table 1.} \ \text{Comparison of the PBS of the Three Groups in Different Sections} \\ (\text{in MPa}) \end{array}$ 

Group	Section		
	Coronal	Middle	Apical
AH26	8.62 <sup>aA</sup>	8.92ªA	3.09 <sup>aB</sup>
P-AH26	6.35 <sup>aA</sup>	$2.28^{bdAC}$	1.37 <sup>acBC</sup>
TCS	1.17 <sup>bAC</sup>	2.97 <sup>cdA</sup>	$0.41^{\text{bcBC}}$
P-TCS	0.79 <sup>bA</sup>	0.75 <sup>cdA</sup>	$0.52^{bcA}$

Values indicate the mean PBS in each group.

Lowercase letters indicate the comparison of PBS of different groups in the same section (column), while uppercase letters indicate the comparison of PBS of different sections within the same group (row).

The presence of at least one similar letter indicates the absence of a significant difference between two groups, and dissimilar letters indicate the presence of a significant difference between two groups.  $P \le 0.05$  was considered statistically significant.



# Mode of failure

Figure 1. Frequency Distribution of Different Modes of Failure in the Study Groups

Of the gases used for NTP therapy, helium and argon are more commonly used for hydrophilic modification of surfaces because the plasma jets containing these gases are more stable. Wang et al<sup>20</sup> in 2016 reported that helium and argon plasma jets had comparable efficacy for hydrophilic modification of polymethyl methacrylate surfaces in the short term. In the present study, the argon gas plasma jet was used for plasma therapy. Ritts et al in 2010 reported that plasma therapy for 30 seconds increased the bond strength of dental composite to dentin, while longer irradiation times decreased the bond strength. The long duration of plasma therapy can degrade the collagen fibers and weaken the structure of the dentin surface.<sup>21,22</sup> Despite the fact that plasma has non-thermal properties and does not traumatize the periapical and periodontal tissues, the shorter duration of plasma irradiation is clinically more favorable.18 Thus, plasma was irradiated for 30 seconds in the present study.

According to previous studies, the elimination of the smear layer can increase the bond strength of endodontic sealers by exposing a strong collagen network and more dentinal tubules, which increases the adaptation of the sealer to dentin.<sup>23-25</sup> In the present study, NaOCl and EDTA were used to eliminate the smear layer.

According to the present results, the application of NTP had no significant effect on the PBS of Endoseal TCS in any section. In addition, plasma therapy significantly decreased the PBS of AH26 in the middle third. In the apical and coronal thirds, plasma therapy decreased the PBS of AH26, but the difference between the AH26 and P-AH26 groups was not significant in these sections.

In plasma therapy, high-energy plasma particles serve as a type of molecular sandblasting and cause the separation of particles from the structure of canal dentin. These particles serve as a smear layer and deposit at the site, causing eventual obstruction of dentinal tubules and decreasing the penetration depth and bond strength of the sealer.<sup>26</sup> According to the results of Kuçi et al,<sup>27</sup> the presence of the smear layer has no adverse effect on the penetration depth of tricalcium silicate-based sealers into dentinal tubules.

Tricalcium silicate-based sealers have higher flowability than resin-based sealers due to the smaller size of particles, and can therefore bond to the smear layer and penetrate into dentinal tubules.<sup>28</sup> This finding can explain no significant reduction in the PBS of Endoseal TCS following plasma therapy in the present study.

Dong et al<sup>29</sup> in 2015 showed that the wetting of root canals with water spray after plasma therapy resulted in the formation of lower amounts of the smear layer and a higher number of open dentinal tubules in the canals compared with no use of water spray. Additional research is needed to determine the impact of canal wetting after plasma therapy on the PBS of endodontic sealers.

Ritts et al<sup>21</sup> discussed that despite the positive effect of plasma therapy on the bond strength of composite to peripheral dentin, its effect on internal dentin was not significant. In addition to the variation in dentinal tubule size, their organization and orientation, and collagen fiber content of these two layers, higher water content of internal dentin can cause the separation of adhesive from dentin and result in eventually lower bond strength to internal dentin, compared with peripheral dentin.8 In endodontic treatment, sealers are penetrated and bonded to internal dentin; this fact can explain the reduction in the bond strength of resin sealer following plasma therapy. Menezes et al18 demonstrated that plasma therapy reduced the bond strength of MTA-Fillapex sealer but had no significant impact on the bond strength of AH Plus sealer. The current results were, to some

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degree, consistent with those findings. The present results were in contrast to those of Garlapati et al<sup>30</sup> who stated that NTP increased the PBS of both AH Plus and BioRoot RCS sealers. Differences in the results may be due to the use of two different types of sealers in the two studies.

The present results revealed that the PBS of AH26 sealer was higher than that of Endoseal TCS in all sections. Covalent bonds between the epoxy resin and amine groups of dentin collagen fibers in the use of epoxy resin-based sealers may create a stronger bond compared with the micromechanical interactions of the root canal wall and calcium silicate through plaque-like structures.<sup>31</sup> This finding was in agreement with the results of previous studies that reported higher bond strength of epoxy resin-based sealers.<sup>18,28,32-34</sup>

In the present study, the PBS in the coronal and middle thirds was higher than that in the apical third in all groups. Dentinal tubules are cleaner, larger, denser, and vertically more organized in the coronal and middle thirds, leading to increased dentinal tubule penetration of endodontic sealants and their bonding to root canal walls in these areas. This finding was in agreement with the available literature.<sup>26</sup>

In general, variations exist in the reported bond strength values of sealers. It should be noted that despite the common use of the PBS test, this test does not always have a standardized design, and thus, its results may be conflicting in different studies. Furthermore, geometrical parameters and materials used for this test can have significant effects on the results.<sup>19</sup>

Regarding the mode of failure, the present results indicated that the cohesive mode of failure was dominant in all sections in the AH26 group. The dominant modes of failure were cohesive and mixed failures in the Endoseal TCS group. This finding is in line with the outcomes reported by Donnermeyer et al<sup>34</sup> in 2018.

Obviously, the higher PBS of the sealer is correlated with the cohesive mode of failure, while the lower values of PBS are correlated with mixed and adhesive failure modes. The dominance of the cohesive mode of failure in the AH26 group may indicate lower bond strength of resin sealers to gutta-percha compared with their bond strength to root dentinal walls. Moreover, the dominance of mixed and cohesive failures and the presence of adhesive failure in the Endoseal TCS group may indicate that the bond strength of this sealer to gutta-percha is almost comparable to its bond strength to root dentinal walls.<sup>34,35</sup>

Further studies are required to cast a final judgment regarding the effects of NTP on human tissues. Moreover, due to the differences in tooth preparation protocols and application of plasma in previous studies, it is challenging to conduct precise comparisons of studies and interpret their findings. Complementary in vitro and clinical studies are necessary to comprehend the cellular and molecular pathways involved in plasma therapy and enhance the knowledge of researchers and dental clinicians regarding different applications of NTP and its benefits.

# Conclusion

Plasma therapy had no significant effect on the PBS of Endoseal TCS in any section. In addition, plasma therapy decreased the PBS of AH26 only in the middle third, and it had no such effect on the coronal and apical thirds. The comparison of PBS in different sections revealed higher PBS in the coronal and middle thirds than the apical third, but the difference in PBS between the coronal and middle thirds was not significant in any group. In the AH26 group, cohesive failure was the dominant mode of failure, whereas cohesive and mixed failure modes were dominant in the Endoseal TCS group.

#### **Authors' Contribution**

Conceptualization: Soolmaz Heidari, Babak Shokri. Data curation: Saeede Zadsirjan. Formal analysis: Atoosa Yazdani, Tina Estarami. Funding acquisition: Atoosa Yazdani. Investigation: Atoosa Yazdani, Tina Estarami. Methodology: Saeede Zadsirjan, Atoosa Yazdani. Project administration: Saeede Zadsirjan. Resources: Atoosa Yazdani. Software: Atoosa Yazdani. Supervision: Saeede Zadsirjan, Mohammad Asnaashari. Validation: Mohammad Asnaashari. Visualization: Soolmaz Heidari, Babak Shokri. Writing-original draft: Atoosa Yazdani. Supervision: Saeede Zadsirjan.

#### Competing Interests

None.

### **Ethical Approval**

The study was approved by the ethics committee of the School of Dentistry of Shahid Beheshti University of Medical Sciences (IR. SBMU.DRC.REC.1400.047).

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