Effect of 0.2% chitosan high molecule nanoparticle on the push-out bond strength of resin cement in post-restoration (An in-vitro study)

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ABSTRACT Debonding between post and resin cement often occurs due to inadequate smear layer removal. Currently, the wide use of Chitosan as an alternative irrigating solution can remove the smear layer. This study aimed to determine the effect of 0.2% chitosan high molecule nanoparticle (C_{HMN}) on the push-out bond strength of resin cement in post-restoration and types of adhesion failure using the different irrigating solutions. Endodontic treatment was performed on 24 mandibular premolars, which were divided into four groups, namely group 1: 0.2% C_{HMN}, group 2: 2.5% sodium hypochlorite (NaOCl) and C_{HMN}, group 3: 5.25% NaOCl and C_{HMN}, group 4: 2.5% NaOCl and EDTA 17%. The final restoration was done using prefabricated glass fiber posts, and each sample was sliced into three segments transversally with a thickness of 2 mm. A push-out test was performed with a universal testing machine, and failure modes were observed under a stereomicroscope. Data were analyzed with a one-way analysis of variance (ANOVA), LSD, and Kruskal-Wallis tests. The results showed a significant difference in push-out bond strength of resin cement in post-restoration between all groups. The Kruskal-Wallis test showed no significant difference between types of adhesion failure. In conclusion, 0.2% C_{HMN} affects the push-out bond strength of resin cement in post-restoration regardless of or without a combination of NaOCl. However, there were no effects on the types of adhesion failure.

Keywords: bond strength, 0.2% chitosan solution, endodontic irrigation, fiber post

INTRODUCTION

Endodontically treated teeth with extensive loss of tooth structure generally require the placement of a post to ensure adequate retention of a core foundation.1,2 In recent years, the increasing demands for esthetic posts has led to the development of metal-free post system, significantly fiber-reinforced posts.3 Fiber-reinforced posts have a modulus elasticity similar to dentin, reducing the risk of root fracture and failure. When they occur, they tend not to be severe.3,4

Most tooth failures encountered with the post are debonding and usually occur along with the post-space-dentin adhesive interface.1 These interfaces could be affected by several factors, such as orientations of dentinal tubules, presence of residues, endodontic sealer, type of adhesive system, and cementation strategies.2 Dual polymerizing resin cement associated with previous dentin conditioning on etching and rinse adhesive systems have demonstrated good results.2 The acidic hydrophilic monomers penetrate the demineralized dentin forming a hybrid layer that includes eliminating the smear layer.1,2,3

The preparation of post-space produces a new smear layer rich in sealer and gutta-percha remnants.3,5 The complete removal of the smear layer, which contains organic and inorganic materials, microorganisms, and canal sealer remnants, is essential to enhance post bonding to dentin with resin.2,5 Irrigation is the best method for

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removing the smear layer by flushing away debris and contaminated materials and improving the penetration of sealing cement through the dentinal tubules. Sodium hypochlorite (NaOCl) is the primary endodontic irrigant due to its ability to dissolve organic tissue and antimicrobial effect and decrease the bond strength. So, in addition, the use of a chelating agent like EDTA is needed for inorganic smear layer removal. These irritants may alter the chemical composition of dentin or the extent of collagen degradation, thus altering the interaction of dentin with the resin cement. 

Chitosan is a natural polysaccharide prepared by the deacetylation of chitin obtained from the shells of crabs and shrimps. This polysaccharide has biocompatibility properties to human cells, biodegradability, and bio-adhesion. Chitosan efficiently removes the smear layer, causing slight erosion on peritubular dentin. Silva et al. showed that 0.2% Chitosan is efficient in removing smear layer compared to 15% EDTA and 10% citric acid. This study aimed to determine the effect and difference of 0.2% chitosan high molecule nanoparticle and sodium hypochlorite at different concentrations on push-out bond strength and adhesion failure of cemented fiber post.

**MATERIALS AND METHODS**

**Endodontic procedures**

Twenty-four human mandibular premolars with single roots, extracted for orthodontic reasons, were selected. The roots were free of cracks, caries, resorption, and fully developed apices. The soft tissue and calculus were cleaned off by soaking teeth in 5.25% sodium hypochlorite for 20 mins and then stored in saline for further use. The samples were randomly divided into four groups of 6 teeth each. The teeth were decoronated at the cementoenamel junction using a low-speed diamond disc. The canal working length was established by inserting K file #10 (Mani, Japan) 1 mm short of the apex. The root canals were prepared with Protaper Universal rotary instruments (Dentsply-Maillefer, Switzerland) using the crown down technique. The root canals were enlarged to size #F3, 9% taper at the working length. The root canals were irrigated according to the treatment group preceding the use of each instrument. 

- Group 1: 3 ml of 0.2% chitosan high molecule nanoparticle
- Group 2: 3 ml of 2.5% NaOCl + 0.2% Chitosan high molecule nanoparticle
- Group 3: 3 ml of 5.25% NaOCl + 0.2% Chitosan high molecule nano particle
- Group 4: 3 ml of 2.5% NaOCl+EDTA 17%

All canals were dried using #F3 Protaper paper points (Dentsply, Switzerland). The canals were obturated with gutta-percha master cone #F3 (Dentsply, Switzerland) coated with AH 26 sealer (Dentsply, Switzerland) using the thermoplasticized technique. The roots were placed at 100% humidity for 48 hours at 37°C, to allow the sealer to set.

**Post space preparation**

To preserve apical seal, gutta-percha was removed with a Peaso reamer #2 (Mani, Japan), leaving 4 mm gutta-percha in the apices. The post spaces were prepared to a depth of 10 mm. The post space was then rinsed according to the final irrigation regimen. 

- Group 1: 5 ml of 0.2% chitosan high molecule nanoparticle
- Group 2: 5 ml of 0.2% chitosan high molecule nanoparticle
- Group 3: 5 ml of 0.2% chitosan high molecule nanoparticle
- Group 4: 5 ml of EDTA 17%

**Bonding of fiber posts**

The canals were etched (Scothbond 3M) for 15 seconds and rinsed with distilled water. Excess water was removed from post spaces with paper points. The bonding agent (Adper Single Bond Plus, 3M) was applied with a micro brush and was polymerized for 10 seconds, with the tip of the light unit directly in contact with the canal orifice. For cementation of fiber posts (Parkell, Switzerland), equal amounts of luting agent paste and catalyst (Rely X Ultimate Clicker, 3M) were thoroughly mixed and applied to the post space walls. Light curing was performed for 20 seconds so that the tip of light was directly in contact with the coronal end of the posts. All specimens were stored in saline for 24 hours before the thermocycling process.

**Push-out test**

Each root was sectioned into three horizontal sections (coronal, middle, apical) of 2 mm thickness, each using a diamond disc at low speed. Due to the fiber posts' tapered design, post diameter was
measured on each surface of the post sections using digital calipers. The push-out test was performed by using the universal testing machine (Torsee’s Universal Testing Machine, Japan) at a crosshead speed of 0.5 mm/min. A 1 mm diameter plunger for coronal sections, 0.8 mm for middle sections, and 0.5 mm for apical sections was centered on the post segment without stressing the surrounding dentin surface. The load was applied in an apical-coronal direction until the post dislodged. The bond strength (MPa) was calculated using the following formula:

\[ A = \pi (r_1 + r_2) \sqrt{(r_1 - r_2)^2 + h^2} \]

Where \( \pi \) was the constant 3.14, \( r_1 \) was the coronal post radius \( r_2 \) (the apical post radius), and \( h \) (thickness of the slice in millimeters).

Analysis of failure mode

After the push-out bond strength evaluation, the failure mode of debonded specimens was analyzed using a stereomicroscope. The failure mode was classified according to the following criteria:
1. Adhesive failure between post and dentin
2. Adhesive failure between post and resin cement
3. Cohesive failure within resin cement
4. Mixed failure

Statistical analysis

The data were analyzed using (SPSS Version 17; IBM Corp) using one-way analysis of variance (ANOVA) and Post-Hoc Tukey LSD tests with the significant level at \( \alpha = 0.05 \). Kruskal-Wallis tests were also used to analyze the failure mode of debonded specimens.

RESULTS

Table 1. Push-out bond strength mean values (MPa) and the respective standard deviation

<table>
<thead>
<tr>
<th>Group</th>
<th>Mean (MPa)</th>
<th>SD</th>
<th>Minimum</th>
<th>Maximum</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>29.05</td>
<td>1.76</td>
<td>25.95</td>
<td>30.96</td>
</tr>
<tr>
<td>2</td>
<td>31.50</td>
<td>2.03</td>
<td>27.55</td>
<td>33.22</td>
</tr>
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<td>3</td>
<td>24.53</td>
<td>2.72</td>
<td>19.54</td>
<td>27.73</td>
</tr>
<tr>
<td>4</td>
<td>13.55</td>
<td>1.47</td>
<td>11.27</td>
<td>15.39</td>
</tr>
</tbody>
</table>

* Statistically significant P-value (p<0.05)

The mean push-out bond strength values (MPa) of the group tested are shown in Table 1. The results of one-way ANOVA showed a highly significant difference (p = 0.000), among the root canal irrigants used. Group 2 showed the highest mean push-out bond strength (31.50 ± 2.03 MPa) among all the tested groups, while the lowest values were recorded in group 4 (13.55 ± 1.47 MPa), with statistically significant differences between these two groups.

The result of the Kruskal-Wallis test revealed no significant difference between the failure mode of debonded specimens. The failure analysis showed that group 2 has adhesive-dentin failure was predominant among the experimental groups.


DISCUSSION

The results of this study indicated that after the push-out bond strength, statistically showed a significant difference in all four groups, especially in group 2 (2.5% NaOCl and 0.2% chitosan high molecule nanoparticle) and group 4 (NaOCl 2.5% and EDTA 17%). This result indicated that Chitosan used as irrigation influenced the bond strength.

Chitosan is a natural polysaccharide obtained by the deacetylation of chitin has biocompatible properties, a high chelating capacity, and antimicrobial effects against a broad range of gram-positive and gram-negative bacteria as fungi. Chitosan can restore collagen changes because an increase in the numbers of inter and intramolecular crosslinks in collagen enhances the mechanical properties and increases dentin collagen's resistance to enzymatic degradation. Pimenta et al. stated that the Chitosan has a chelating property that causes erosion yet does not cause any alteration in intertubular dentin. The chelating effect of 0.2% chitosan compared to other irrigation, allied to its advantageous properties and low concentration, indicate that this chelating irrigation should be for dentin decalcification.

Despite not fully knowing its mechanism of action, it is believed that adsorption, ionic exchange, and chelation are responsible for the formation of complexes between chitosan and the metal ions. The type of interaction that occurs depends on the ion involved, the chemical structure of Chitosan and the pH of the solution. Currently, two theories explain the chelating mechanism of Chitosan. The first, known as the bridge model, state two or more amino groups of a chain of chitosan bind to the same metal ion. The second theory supports only one amino group of one Chitosan involved in the binding, the metal ion 'anchored' to the amino group.

Silva et al. observed that 0.2% chitosan for 3 min appeared to remove the smear layer, causing little dentin erosion. Silva et al. stated that 0.2% chitosan effectively removed the smear layer from the middle and apical thirds of the root canal. The ability of the solution to remove the smear layer, thus improving cement contact and penetration into dentinal tubulous.

A combination of 2.5% sodium hypochlorite (NaOCl) and 17% EDTA is generally used as root canal irrigation to get the effect of organic and inorganic smear removal. However, this combination is less effective in removing the smear layer on the apical third of the root canal. Silva et al. observed that a combination of 2.5% NaOCl and 17% EDTA is ineffective in removing the smear layer in the apical third. This is probably due to smaller apical canal diameter hindering the penetration of this irrigant and limited contact with...
the root canal, resulting in ineffective removing the smear layer on the apical third.\textsuperscript{2,13} This study revealed that 2.5% NaOCl has a higher bond strength than 5.25% NaOCl, combined with 0.2% chitosan high molecule nanoparticle. NaOCl forms a thin layer of oxygen along the dentin surface, which causes potent inhibition of the interfacial polymerization of resin bonding material.\textsuperscript{3,7} The generation of oxygen bubbles at the resin-dentin interface also interferes with resin infiltration into the tubular dentin.\textsuperscript{7} Furthermore, NaOCl removes the organic matrix of dentin which causes dissolution of collagen fibrils that impedes the formation of a consistent hybrid layer.\textsuperscript{14} Thus, this reduces the bonding between the adhesive resin and dentin.

For anatomical reasons, the coronal and middle thirds have greater tubules density and diameter than the root canal's apical region, which explains the higher bond strength.\textsuperscript{1,2} However, the thickness in the distribution of resin cement with the probable void formation or traces of remnant gutta-percha and sealer after post space preparation have been associated with lower bonding potential in the apical region.\textsuperscript{7}

The failure mode in G2 (combination of 2.5% NaOCl and 0.2% chitosan) was predominantly adhesive-dentine, implying that the weak link was the bond between the resin cement and dentin. It demonstrated the relative weakness of resin cement and dentin bond due to polymerization shrinkage or voids in the resin cement, resulting in the gap formation at the interface.\textsuperscript{15}

CONCLUSION

Based on the results presented in this study, it can be concluded that a combination of 2.5% sodium hypochlorite and 0.2% chitosan high molecule nanoparticle showed significantly can affect the push-out bond strength on resin cement in post-restoration. Further studies/research on push-out bond strength should be conducted using different types of cement and a transmission electron micrograph (TEM) assessment to observe the depth of cement adhesion to dentin.

REFERENCES

