Effect of Acidic Environment on Dislodgment Resistance of Different Silicate-based Root Repair Materials

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Research Article
Received: March 06, 2018; Published: April 10, 2018

Volume 2 Issue 5 May 2018

Abstract

Aim of the Study: The aim of the present study was to compare the dislodgement resistance of Biodentine, EndoSequence Root Repair Material putty and Tech Biosealer Apex as root repair materials in the presence or absence of an acidic environment could affect a compromised bond to dentin.

Material and Methods: One hundred eighty root sections were instrumented. The specimens were randomly divided into 3 groups (n = 60) and repair materials were fitted incrementally into the canal spaces. The specimens were placed in an incubator for allowed to set and then divided randomly into 4 subgroups (n = 15). The specimens were wrapped in pieces of gauze soaked in phosphate buffer saline solution (pH = 7.4) and butyric acid buffered at pH values of 6.4, 5.4, and 4.4. Push-out bond strength values were measured using a universal testing machine. The slices was examined under a stereomicroscope for evaluating to failure modes.

Results: The push-out bond values of pH 7.4 was higher than pH 4.4 in all the test materials. While the push-out bond strength values of Biodentine was significantly higher than Tech Biosealer in all pH values, there was no statistically significant difference between Biodentine and Endosequence Putty groups (P > 0.05). Inspection of the samples showed adhesive failure mode was the most frequently observed type in all groups.

Conclusion: Physical and chemical properties of the root repair materials, based calcium silicate, may affect in acidic pH. A low pH value facilitates leakage and dislocation of repair materials under mechanical loads.

Keywords: Acidic Environment; Bond Strength; Biodentine; Endosequence Root Repair Material Putty; Tech Biosealer Apex; Root Repair Material

Introduction

Repair materials have a wide application area in endodontics. These materials have been commonly used for apical plugs, root-end fillings and repairs of root and furcation perforations [1]. An ideal repair material should: be biocompatible; dimensionally stable; be radiopaque; be antibacterial; be insoluble in tissue fluids; be easy to manipulate; provide adequate seal; be able to adhere to the root-end cavity walls; remain in place under dislocating forces; and be unaffected by the presence of tissue fluid that may, in an infected area, be acidic [2,3].

Various repair materials have been used in endodontics, such as amalgam, bonding system, zinc oxide eugenol cements, glass ionomer cements and calcium silicate cements [4]. Calcium silicate-containing materials such as Mineral Trioxide Aggregate (MTA), Bioaggregate, Biodentine, EndoSequence Root Repair Material and Tech Biosealer have shown good sealing and are more biocompatible than conventional repair materials [5]. Biodentine (BD) (Septodont, Saint Maur des Fosses Cedex, France), a bioceramic-based dental material with dentin-like mechanical properties was developed towards MTA. BD contains tricalcium silicate, dicalcium silicate, calcium carbonate, zirconium oxide,
iron oxide and a water-based liquid containing calcium chloride as the setting accelerator [6,7]. Biodentine is recommended for use as an endodontic repair material because of its good sealing ability, biocompatibility, bioactivity, biomineralization properties, high compressive strengths, and short setting time [8].

EndoSequence Root Repair Material Putty (ESRRM Putty) (Brasseler, Savannah, GA, USA), another novel root repair bioceramic material, has been introduced for use as a root-end filling and perforation repair material. According to the manufacturer, ESRRM is composed of tricalcium silicate, dicalcium silicate, zirconium oxide, tantalum oxide, monobasic calcium phosphate and fillers. It is manufactured as a premixed product in both mouldable putty and pre-loaded syringe paste to provide the clinician with a homogeneous and consistent material that sets in the presence of moisture. This new material is hydrophilic, insoluble, radiopaque, aluminium-free, of high pH and easy to work with [9,10]. ESRRM has been demonstrated to be biocompatible, antibacterial and able to seal root-end cavities [11-14].

Tech Biosealer Apex (TB Apex) (Isasan, Como, Italy) is another calcium silicate-based endodontic material. Its powder is a mixture of tricalcium silicate, beta dicalcium silicate (β-Ca2SiO4), anhydrous calcium sulfate, calcium carbonate and bismuth oxide. Its liquid is composed of Dulbecco’s phosphate buffered saline (PBS) [4]. According to the manufacturer, Tech Biosealer apex has a high release of calcium hydroxide and excellent biocompatibility and is antibacterial, a superior apical sealing, dimensionally stable and hardens in the presence of organic fluids as a root repair material (www.isasan.com).

In various endodontic procedures, root repair materials are often applied in contact with tissue fluids or inflamed tissues. This environment may have a normal pH or might have lower pH levels because of inflammation, abscess or periapical pathosis. After the endodontic treatment, the pH will return to slightly alkaline (pH = 7.4) within 7 days or less [15-17]. Therefore, during the setting process the surface of materials may be subjected to acidic or slightly alkaline pH levels. An acidic pH value causes leakage and dislocation of repair materials under mechanical loads of occlusion or condensation of materials [18].

The study aimed to compare the dislodgment resistance of Biodentine, EndoSequence Root Repair Material putty and Tech Biosealer Apex as root repair materials in the presence or absence of an acidic environment that could cause a compromised bond to dentin.

Material and Methods

Sixty extracted human mandibular premolar teeth with straight roots, mature apices, no caries or restorations, and no cracks were used in the present study. Buccolingual and mesiodistal radiographs of all teeth were taken to confirm that the canal anatomy was composed of a single-rooted canal without calcification, resorptions, or previous root canal treatments. The teeth were cleaned and stored in 0.5% chloramine-T solution until use. Crowns of the selected teeth were sectioned at the cementoenamel junction (CEJ) with safe-sided diamond disk (NTI diamond disc, Axis Dental, USA), and then, mid-root dentin was sectioned horizontally into slices with a thickness of 1.00 ± 0.05 mm. One hundred eighty root dentin slices were obtained by using a water-cooled diamond saw microtome (Isomet, Buehler; Lake Bluff, IL, USA). The lumens of the specimen disks were instrumented with sizes 2 to 5 Gates Glidden burs (Dentsply Maillefer, Ballaigues, Switzerland) to achieve a standardized diameter of 1.3 mm. All samples were rinsed with distilled water to remove debris produced during the procedure. The root sections were randomly divided into 3 groups (n = 60), and the following test materials were used: The repair materials were prepared according to the manufacturer’s recommendations. Then, repair materials were fitted incrementally without pressure on the canal spaces of the dentin slices. Excess material, on the surface of the samples, was trimmed with a scalpel.

Subsequently, the samples were wrapped in wet gauze, placed in an incubator and allowed to set for 10 minutes at 37ºC with 100% humidity. Immediately after incubation, the specimens were then divided randomly into 4 subgroups (n = 15) and wrapped in pieces of gauze soaked in phosphate buffer saline solution (pH = 7.4) and butyric acid buffered at pH values of 6.4, 5.4, and 4.4 and then incubated for 4 days at 37ºC.

Push-out Test

For the push-out test, the samples were tested in a universal test machine (Instron Corp, Norwood, MA, USA). The compressive load was applied by exerting a download pressure on the surface of the test materials at a speed of 1 mm/min using a 1.00-mm diameter cylindrical stainless steel plunger while avoiding contact with dentine during testing. The maximum load applied to materials was recorded in newtons (N) prior to debonding. The force needed to dislodge the test material (F; in kN) was transformed into megapascal (MPa) by dividing the force by the adhesion area of the filling material (N/2prh), where p is the constant 3.14, r is...
the root canal radius, and \( h \) is the thickness of the root dentin slice in millimeters. The slices were examined under a stereomicroscope (Olympus SZX-ILLB100; Olympus Optical, Tokyo, Japan) at 40x magnification for evaluating failure modes, which were named: adhesive failure that occurred at test material and dentin interface, cohesive failure within test material and mixed failure.

**Scanning Electron Microscopy Analysis**

Specimen surfaces were coated with gold and analyzed under a scanning electron microscope.

**Statistical Analysis**

The data was statistically analyzed using one-way analysis of variance and the Tukey’s post hoc tests. All statistical analyses were performed using IBM SPSS, Ver. 20.0 software (IBM SPSS, Inc. Chicago, IL). The significance level was set at \( P = 0.05 \).

**Results**

The mean push-out bond strength (MPa) values ± standard deviation of all experimental groups are shown in table 1. The statistical analyses revealed an interaction between the decrease of the pH value and push-out bond strength for all tested materials; in other words, mean bond strength values decrease from pH 7.4 up to pH 4.4. While the greatest mean bond strength 9.79 ± 1.95 MPa was observed after exposure to a pH value of 7.4 for Biodentine, the lowest mean bond strength 2.71 ± 1.10 MPa was observed after exposure to a pH value of 4.4 for Tech Biosealer. The push-out bond values of pH 7.4 was higher than pH 4.4 in all of the test materials (\( P < 0.05 \)). However, no significant difference was found between pH levels of 7.4 to 6.4 and pH levels of 5.4 to 4.4 in each group (\( P > 0.05 \)). While the push-out bond strength values of Biodentine were significantly higher than Tech Biosealer in all pH values (\( P < 0.05 \)), there was no statistically significant difference between Biodentine and EndoSequence Putty groups (\( P > 0.05 \)).

**Discussion**

During the periapical infections, the pH of periapical tissues decreased. This change affects dentin and root end filling material. Moreover, not only is pH changing periapical surgery but it also affects some clinical scenarios, such as open apex non-vital teeth with periapical lesions or lateral or furcal perforations with radiolucent lesions. Recently developed, calcium silicate based materials are often preferred by the clinicians because of their chemical, mechanical and biological properties as a hydraulic cement, stimulating bone formation and sealing. None of the proposed materials, such as amalgam, IRM, calcium hydroxide and glass-ionomer cements, contain ideal material requirements to repair an endodontic root perforation. Chosen “old” materials do not have osteogenic, cementogenic or antibacterial properties and are, therefore, unable to ensure sealing [19].

The pH value of calcium silicate based materials is higher than other different based materials. When these materials are placed as an apical plug or sealing material directly in contact with the lesions, the materials might be exposed to an acidic environment for a longer time. Therefore, this acidic pH value of the host tis-

### Table 1: Mean and Standard Deviation of push-out (MPa) of Different Calcium Silicate Based Materials.

<table>
<thead>
<tr>
<th>Material</th>
<th>pH</th>
<th>7.4</th>
<th>6.4</th>
<th>5.4</th>
<th>4.4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Biodentine</td>
<td></td>
<td>9.79 ±</td>
<td>8.84 ±</td>
<td>6.32 ±</td>
<td>6.27 ±</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.95</td>
<td>1.96</td>
<td>2.68</td>
<td>2.24</td>
</tr>
<tr>
<td>EndoSequence Putty</td>
<td></td>
<td>7.70 ±</td>
<td>6.49 ±</td>
<td>5.06 ±</td>
<td>4.15 ±</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.87</td>
<td>1.64</td>
<td>2.06</td>
<td>1.83</td>
</tr>
<tr>
<td>Tech Biosealer Apex</td>
<td></td>
<td>5.08 ±</td>
<td>4.34 ±</td>
<td>2.89 ±</td>
<td>2.71 ±</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.66</td>
<td>1.86</td>
<td>1.17</td>
<td>1.10</td>
</tr>
</tbody>
</table>

On the other hand, except of pH 7.4, there was no significant difference in push-out bond strength between Endosequence Putty and Tech Biosealer groups (\( P > 0.05 \)). Adhesive failure for all groups was predominantly observed regardless of the type of materials used.

**SEM analysis**

Representative surfaces of different calcium silicate-based materials, including Biodentine, EndoSequence Putty and Tech Biosealer, are shown in figure 1.
sues, because of preexisting pathologic conditions due to bacterial-induced local metabolic acidosis or tissue inflammation at the time of placement, might affect the physical and chemical properties of calcium silicate-based material used while repairing root and furcation perforations, root-end fillings, and apical plugs [1,20].

Root repair material should be able to adhere to the root-end cavity walls and seal the connection between the root canal system and periapical tissues. Therefore, the bond strength of this material to dentin is an important factor [1,16].

Goracci, et al. reported that the push-out test has been shown to be efficient and reliable for bond strength. [21]. In the present study, the push-out test method was used to evaluate the bond strength between BD/ESRRM Putty/TB Apex and dentin while exposed to different pH environments.

In biomimetic apatite coating, the morphology and composition of the hydroxyapatite crystals relate to different factors, such as environmental pH [22]. pH 7.00 is ideal for hydroxyapatite formation [23]. In some clinical situations, endodontic repair materials may be exposed to an inflamed environment with a low pH [3,15]. In the present study, butyric acid was selected to simulate the clinical conditions associated with periradicular infections [17].

The findings in this study showed that push-out bond strength of all the test materials decreased significantly after exposure to pH levels of 4.4, as compared with exposure to a pH level of 7.4. These results could be attributed to a lack of formation of hydroxyapatite crystals and an ensuing hybrid layer between the calcium silicate-based materials and dentin in an acidic environment [16-18].

The result of the present study showed that the greatest mean bond strength was observed after exposure to a pH value of 7.4 for Biodentine. This finding is in agreement with Elaghy, et al. [1], where they found the surface microhardness of Biodentine was significantly higher than other experimental groups at pH 7.4. It could be explained that the material could not harden as successfully in a low pH environment.

The study revealed that the bond strength of ESRRM Putty decreased significantly after exposure to pH 4.4 as opposed to pH 7.4. This finding is in contrast to Shokouhinejad., et al. [20], who reported that there was no significant difference between the dislodgement resistance of ESRRM Putty after exposure to both pH = 7.4 and pH = 4.4. It may explain the difference between the varying thicknesses of root slices in studies. In the present study, dislocation resistance of BD was significantly greater than that of TB Apex in all pH values. This result may be attributed to the delivery form of these materials, which was a premixed capsulated form for BD and a separate powder and liquid for TB Apex. Scanning electron microscopy evidence also suggests that acidic solutions affects the development of a porous surface and a lack of needle-like crystals.

The limitation of this study—the force needed for the displacement of root repair materials from root dentin to occur—was significantly lower in samples stored at lower pH values.

Conclusion

Physical and chemical properties of the root repair materials, based calcium silicate, may affect in acidic pH. A low pH value facilitates leakage and dislocation of repair materials under mechanical loads.

Bibliography


Volume 2 Issue 5 May 2018
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**Table 3:** Distribution of participant according to working experience and responses to knowledge and attitude
Discussion

Oral health care needed for hospitalized patients is associated with nurse’s knowledge and practices during hospitalization. In this study the percentages of nurses who were trained oral health care as part of their initial education was (87.4%) compared to non-trained nurses (12.6), this result contradict the England study [10] where most nurses had received training during initial nursing course (42.41%) or whilst on the job and minority had attended later training course in oral care (15,14.5%). The present study showed that majority of nurses (76%) were graduated with Diploma degree while only 2% have Masters. A study carried out in Riyadh concluded that Health professionals with diploma degree showed higher oral health knowledge compared with university graduates. The possible explanation for this could be that diploma holders were more involved in-patient care as compared with university graduates, who were more likely to be involved in patient administrative work [11].

The results of the present study regarding nurses knowledge about dental plaque and gingivitis, presented that 32.6% of nurses have idea about the correct meaning of dental plaque while, 28.6% knew the meaning of gingivitis these findings were similar to the study conducted by Mohamed Abdalbaseer 2012 [11], who concluded that Plaque, consisting of bacteria and their intercellular products, is generally considered as the primary etiological factor in both caries and gingivitis [12]. Less than 28% of health professionals identified the meaning of dental plaque. This suggests poor knowledge of dental plaque and its formation on the tooth and gums. For effective primary prevention of dental caries and periodontal disease, knowledge of plaque and its removal is essential. The finding that no significant correlation between nurse’s experience and their knowledge, attitude and practice towards mouth care of hospitalized patients, believed to be in contrast with European countries [13] where the more experienced the nurse, the more knowledge, attitude and practices of oral care. In Taiwan also, there was significant correlation between the age of the nurses and their total score on oral care practices. The potential bias in this study comes with study carried out in Sudan by Sara and Amel Mudadwi [14], that all nurses were worked at university or governmental institutions. However, the survey carried by Binkley in USA [15] found that private hospitals provided more oral health care.

Conclusion

Diploma was the highest degree of study among nurses in Abha hospitals. The oral hygiene measures although was found to be part of their study, fifth of the nurses have no any idea about gingival diseases and oral care measures needed for hospitalized patients. Experience alone without adoption of courses and formulation of oral health care protocol will not add any benefits to the hospitalized patients. The study highlights the need for setting oral health care protocol and adoption of advanced training for nurses in Abha hospitals. As this study was carried out in three hospitals, its finding couldn’t be generalized to all the hospitals in Abha city, yet the finding can possibly be taken as indicators.

Conflict of Interest

Non-declared.

Acknowledgement

We gratefully acknowledge the support by Dr. Ahmed Bakri, Dr. Betsy Josef and all participating nurses.


**Citation:** Amel Ibrahim Faragalla, *et al.* “Knowledge, Attitude and Practice of Nurses to Oral Health Care for Hospitalized Patients in Abha City-Saudi Arabia”. *Acta Scientific Dental Sciences* 2.5 (2018): 01-08.