



INFLUENCE OF VARIOUS ENDODONTIC IRRIGATING SOLUTIONS ON THE BOND STRENGTH OF BIODENTINE TO RADICULAR DENTIN

Dalia Abd Elhamid Sherif

ABSTRACT

Objective: To evaluate the effect of EDTA, Q-Mix, Chitosan, and NaOCl on Biodentine bond strength. **Materials and Methods:** Forty extracted human premolars with type I root canal system were used in this study. After crown removal and working length determination, all root canals were prepared using a ProTaper NEXT Nickel Titanium rotary system up to X4 apical size corresponding to X4. The specimens were then randomly divided based on the final irrigation regimen into 4 groups (n = 10): Group 1: 17% EDTA, Group 2: Q-Mix, Group 3: 0.2% Chitosan, and Group 4: NaOCl. All samples were obturated using Biodentine as a filling material. Each sample was sectioned horizontally to produce a 2 mm thick disc per specimen. A universal testing machine was used to evaluate the push-out bond strength. **Statistical Analysis:** Data were analyzed using one-way ANOVA followed by pairwise comparison using the Tukey method. The statistical level of significance was set at 0.05. **Results:** 17% EDTA showed the highest push-out bond strength followed by Q-Mix > 0.2% Chitosan > NaOCl. **Conclusion:** Different irrigants used during the Final irrigation of the root canal impact biodentine bond strength.

KEYWORDS: Chitosan, EDTA, NaOCl, Q-Mix, Push-out.

INTRODUCTION

Endodontic treatment's success revolves around eliminating micro-organisms harbored in the entire root canal system and preventing repeated infections⁽¹⁾. Adequate cleaning and shaping is the most critical step in endodontic procedure⁽²⁾.

The root canal system is anatomically complex, with a multitude of areas that are unreachable by mechanical means. Therefore, the purpose of canal cleaning is attained through a combination of chemical and mechanical techniques, including the physicochemical action of endodontic irrigants and preparation⁽³⁾.

Endodontic therapy involves the removal of necrotic, loose, and contaminated tissues using a

combination of instruments, irrigation, and intra-canal medicaments to create a leak-proof apical seal⁽⁴⁾.

It is unfeasible to achieve bacterial reduction and the removal of tissue organic remains without the use of root canal irrigants. It also provides lubrication, gross debridement, elimination of microbes, and tissue dissolution⁽⁴⁾. The smear layer, an iatrogenic layer formed on dentinal surfaces, should also be removed with the help of irrigation treatment. As a result, it is crucial to first remove this layer, as its presence hinders root canal filling materials from penetrating into root canal surfaces⁽⁵⁾.

The smear layer contains both inorganic and organic substances, making it resistant to removal

1. Endodontic Department, Faculty of Dentistry, Tanta University

• **Corresponding author:** dalia.sherif@dent.tanta.edu.eg

by a single irrigating solution. Therefore, a combination of organic and inorganic irrigating agents is suggested. Many chemical irrigants, like chelating agents and organic acids, are used to remove the smear layer⁽⁶⁾.

Sodium hypochlorite (NaOCl) has been used for more than 40 years and is the most used endodontic irrigant. It's a great tissue solvent and antibacterial agent⁽⁷⁾. As a result of its efficacy against pathogenic organisms, its ability to remove debris and pulp tissue, and its fulfillment of many of the appositional qualities that have been described, it is generally acknowledged as the irrigant of choice in endodontics⁽⁸⁾. However, at high concentrations, it is known to be extremely irritating to the periapical tissues⁽⁷⁾. In addition, the smear layer cannot be dissolved without the use of an acid or chelating agent that can remove inorganic elements. Root canals can only be properly cleaned if both organic and inorganic tissue-dissolving chemicals are used⁽⁹⁾.

Complete removal of the smear layer of root canal dentine is usually aided by ethylenediaminetetraacetic acid (EDTA) as a final irrigation solution⁽¹⁰⁾. The effectiveness of the EDTA chelating agent in removing the smear layer makes it a popular choice for root canal irrigation. Because of this, it is typically employed in studies that assess the efficacy of several irrigation solutions to see which one is most effective at removing the smear layer⁽¹¹⁾.

Q-Mix was developed for the dual purpose of removing the smear layer and facilitating the integration of root canal filling materials into the dentinal tubules⁽¹⁰⁾. Q-Mix consists of EDTA as a decalcifying agent, surfactant, and a CHX analog. Q-Mix also appeared as a dependable antimicrobial irrigant, when compared to CHX, it was found to be more effective against *Enterococcus faecalis*, and it removed the smear layer with close efficiency to EDTA⁽¹²⁾.

Chitin, the primary structural protein in the exoskeletons of crustaceans, can be used to obtain

Chitosan via alkaline deacetylation. Because of Chitosan's continuous drug-releasing characteristic, biodegradability, bioadhesion, biocompatibility, and lack of toxicity, have garnered attention in dentistry research. Moreover, in acidic conditions, its chelating ability is high for various metal ions^(13,14). Chitosan's exceptional chelating capacity has made it a popular choice as a last-stage irrigant for smear layer eradication. Moreover, it exhibits potent antibacterial activity against both gram-negative and gram-positive bacteria, as well as various pathogenic fungi⁽¹⁵⁾. Unlike EDTA, Chitosan doesn't do as much damage and erosions to the underlying dentin while removing the smear layer⁽¹⁶⁾.

AS MTA has some drawbacks, such as manipulation difficulty and prolonged setting time, Biodentine (Septodont, Saint Maur des Fosses, France) a calcium silicate-based material with similar clinical applications to MTA, has been introduced to overcome those issues. Tricalcium silicate, calcium carbonate, zirconium oxide, a water-based liquid, and a water-reducing agent are the main components of Biodentine⁽¹⁰⁾. Biodentine has a shorter setting time of around 12 minutes compared to MTA, which takes much longer time averaging between 3 and 4 hours. It also shines with superior mechanical properties, biocompatibility and bioactivity. Therefore, it is widely known as a dentin substitute⁽¹⁷⁾.

To measure how well obturation materials adhere to root canal dentin, a push-out bond strength test is performed. It examines the intensity of the tested substance binding to the tooth structure, providing information about the material's adhesive capability and facilitating our understanding of the tested material's resistance to dislodgement^(18,19). It's a reliable and practical test of the material adaptation with the prepared dentinal root canal walls⁽¹⁹⁾.

For a successful endodontic treatment, it is essential to achieve complete obturation of the complicated root canal system with dimensionally inert, stable, and biologically compatible filling materials⁽²⁰⁾.

Several researchers tried to assess the effect of multiple endodontic irrigants in combination with multiple root canal sealers on filling material bond strength. Q-Mix irrigation solution's impact on Biodentine bond strength is under-studied. Therefore, the target of this study is to compare the push-out bond strength of Biodentine to root canal walls using several irrigants commonly used in endodontics (Q-Mix, 17% EDTA, Chitosan, and 2.5% NaOCl).

MATERIALS AND METHODS

Specimens' selection and preparation

The Research Ethics Committee (REC) at Tanta University's Faculty of Dentistry approved this study (R-END-11-22-4). Patients were given information about the study's goals and asked for permission to participate by signing an informed consent form, as required by the REC of the Faculty of Dentistry, Tanta University.

Forty freshly extracted straight single-rooted human mandibular premolars with almost identical dimensions and morphology were gathered for this study. Teeth were scaled to remove calculus and any remaining periodontal tissues. At 37 degrees Celsius, all samples were kept in distilled water until use. Using a high-speed diamond disc and a water-based cooling system, the samples were transverse-ly decoronated to yield about 16 mm \pm 1 of root⁽²¹⁾.

Then, the working length of each root canal was calculated by placing a #15 K file (Mani, Tochigi-Ken, Japan) into the canal until the file was just visible at the apical foramen, and then deducting 1 mm from this measurement. The root canals were prepared using the ProTaper Next (Dentsply, Sirona) system, with files increasing till file X4 (#40/06) to 1 mm shorter than the root apical foramen. Root canals were irrigated with 2.5% NaOCl during instrumentation⁽²¹⁾.

After the completion of instrumentation, the specimens were then randomly divided into 4 groups (n =10) based on the final irrigation solution:

Group 1: 5 mL of 17% EDTA (Pulpdent Corp., Watertown, MA).

Group 2: 1: 5mL Q-Mix (Dentsply Tulsa Dental Specialties Johnson City, TN).

Group 3: 0.2% Chitosan (NanoTech Company, 6 of October, Egypt).

Group 4: 5mL of 2.5% NaOCl solution (Clorox Co., 10 of Ramadan, Egypt).

The root canals were irrigated by inserting a stainless steel 29-gauge needle (NaviTip, Ultradent Products) to within 1 mm of the working length. The final irrigation solutions were applied for 1 minute. Finally, 5 cc of distilled water was used to irrigate the root canals and wash off any remaining precipitate. Sterile paper points (Dentsply-Maillefer, Ballaigues, Switzerland) were then used to dry the canals⁽¹⁰⁾.

Final irrigation was performed, and then Biodentine's mixture was done as the manufacturer's recommendations described. A capsule containing powder was given 30 seconds of mixing time in amalgamator (Softly; de Götzen, Italy) at 4,000 to 4,200 rpm after the liquid was poured from its single-dose container. It was inserted within the canal using an amalgam carrier increment by increment and condensed apically by gentle packing with a hand plugger with size 4 and covered by a moistened cotton pellet for 12 min to allow its initial setting⁽²²⁾.

All specimens were radiographed to assess the quality of the obturation. For Biodentine complete setting, the specimens were stored at 100% humidity for 7 days.

The samples were marked at 7 and 9 mm from the apex to obtain the mid-middle portion of the root and then placed in transparent acrylic blocks. Using a water-cooled diamond disc, each block was sectioned perpendicular to its root longitudinal axis to obtain a 2.00 \pm 0.05 mm thick slice from the mid-middle portion of it. The thickness of each section was accurately measured with a digital caliper. Loading was done on a universal testing

machine at a speed of 0.5 mm/min in an apico-coronal direction. The maximum load applied to the filling material before dislodgement was recorded in newtons which expressed the bond strength in megapascals⁽²³⁾.

By dividing this force by the surface area of the material-dentine interface ($N/\pi DH$), where π is the constant ≈ 3.14 , D is the average root canal diameter in millimeters, and H is the height of the root dentin slice in millimeters, we were able to determine the push-out bond strength in megapascals (MPa)⁽¹⁸⁾.

Statistical analysis

Statistical analysis was used to analyze and interpret the differences and significance of this study's data. Statistical tests were performed with

One-way Analysis of Variance (ANOVA) with a standard significance level ($P = 0.05$) to assess the significance between the tested irrigants. Furthermore, intragroup significance level was checked using pairwise comparisons between the tested irrigants using the post hoc Tukey method.

RESULTS

As shown in Table 1 and Figure 1, the EDTA group displayed the highest resistance to displacement with a mean of 7.3106 MPa, while the least push-out bond strength value was observed in the NaOCl group with a mean of 5.1686 MPa, the Q-Mix and Chitosan groups pushout bond strength values were close with a slight edge for Q-Mix, with mean values of 5.7144 and 5.5302 MPa respectively.

TABLE (1) Mean values of push-out bond strength for all test groups and their standard deviations [in Mpa]. And P-values for Tukey pairwise comparisons for significant subgroups at each level.

Group	N	Mean	Conf. (±)	Std.Error	Std.Dev.	Significance and P Value by Tukey Method			
						EDTA	Q-Mix	Chitosan	NaOCl
EDTA	10	7.3106	1.17377	0.518872	1.640817	 	No	Yes	Yes
Q-Mix	10	5.7144	1.248213	0.55178	1.744882	0.06258	 	No	No
Chitosan	10	5.5302	0.434009	0.191856	0.606702	0.03123	0.99052	 	No
NaOCl	10	5.1686	0.866821	0.383184	1.211733	0.00695	0.81160	0.93518	

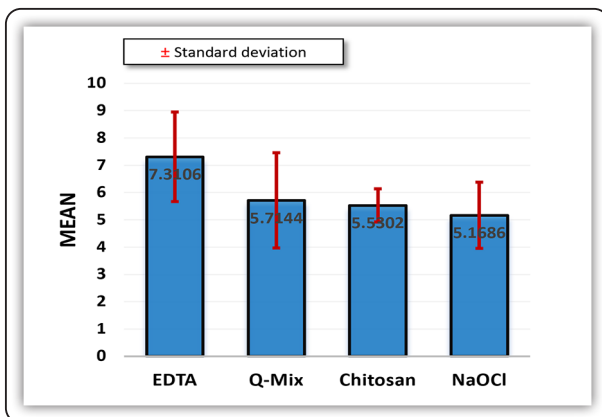


FIG (1) Mean values of push-out bond strength for all test groups and their standard deviations in Mpa.

On analyzing statistically using One-way Analysis of Variance (ANOVA), the groups were significant (p -value = 0.007). As shown in Table 1 Tukey method revealed significance between EDTA and NaOCl groups, it also revealed significance between EDTA and Chitosan groups. There was no significance between any other group pairs.

DISCUSSION

In this study, several procedures have been made to provide the highest possible level of standardization between the test groups. Approximately similar dimensions of a single root canal system extracted human mandibular premolars were used. Furthermore, the length standardization for all samples to 16 ± 1 mm, and root canal instrumentation was performed with a master apical file up to size X4 (size# 40/06) in all groups.

Irrigation is an essential step during and after instrumentation for lubrication of the canal and effective smear layer removal. Root canal sealers are more effectively adapted to the canal walls when they are able to penetrate the dentinal tubules, in a properly prepared root canal.

A variety of endodontic irrigants, including NaOCl, HEDP, CHX, and EDTA, are now in use. However, novel irrigants like MTAD, Q-MIX, SMEAR CLEAR, and TETRA CLEAN have been introduced for use in endodontics⁽¹⁸⁾.

Because of the smear layer, irrigating solutions, medication, and sealers may not be able to reach the dentinal tubules or may take much longer to do so. In order to increase adhesion between the filling material and the root canal wall, it is now commonly recommended that the smear layer should be removed prior to root canal obturation.

When used alternately, EDTA and NaOCl can effectively remove the smear layer without triggering any inflammatory reactions in the preapical region. Long-term EDTA use, as noted by Calt et al., leads to significant tubular and intertubular dentin erosion⁽²⁴⁾. As a natural polysaccharide, Chitosan is more biocompatible and nontoxic, yet it also has chelating properties. Hence, the use of biocompatible smear layer removing agents is preferred⁽¹³⁾.

Also, the Q-Mix irrigating solution was used in this study, It's a mixture of chlorhexidine gluconate and EDTA. In addition to the saline and

surfactant, it also contains the calcium-chelating agent polyaminocarboxylic acid, the antibacterial agent bisbiguanide, and the saline. Previous studies showed that Q-Mix effectively removed the smear layer from the root canals and increased the sealer's ability to integrate into the dentinal tubules⁽²⁵⁾.

The binding strength of materials to dentin can be evaluated in a number of ways, including shear, tensile, and push-out tests. A push-out bond strength test was employed for this study as it is thought to be quick, easy, accurate, repeatable, and easy to interpret. It is more sensitive since it can judge the efficacy of sealers with weak bond strengths⁽²⁶⁾.

Due to the fact that the radicular dentin is not uniform, and its tubular density decreases from coronal to apical region, in this study, only 2 mm thick segments were selected from the middle portion of the roots to be used in the push-out bond strength test, in order to prevent premature debonding. Furthermore, the root canal's prepared wall surface can vary greatly depending on the method of chemo-mechanical preparation used⁽²⁶⁾.

Three different chelating chemicals were utilized to eliminate the smear layer in this study. The best irrigant for removing the smear layer has been 17% EDTA. Q-Mix and Chitosan at 0.2% concentration have lately been proposed as alternates to EDTA. Therefore, these were used as the test irrigants. As one minute is the ideal period suggested for best chelating effects, that's how long the final irrigation with the chelating chemicals lasted, in order to simulate the clinical situation⁽²⁷⁾.

Current findings suggest that root-filling materials' binding strength can also be affected by the chelating agent and irrigation solution used. It could be due to variations in the ability of the final irrigation solutions to remove the smear layer.

Results for bond strength were greater for samples irrigated with EDTA than with any of the other test irrigants. One possible explanation for this finding is that EDTA promotes sealer penetration

and the creation of a highly qualified hybrid layer bonding by producing collapse in the dentin matrix structure. AS 17% EDTA has a pH of 7.4, this effect could be justified by its acidity according to Garcia-Godoy et al.,⁽²⁸⁾.

Q-Mix contains EDTA, CHX, and a detergent, which may explain why Biodentine was more easily dislodged after using it than after using EDTA. Surface hardness, sealing ability, setting time, and resistance to dislodgement forces may all diminish after exposure to 2% CHX, even though it is not an acid⁽²⁹⁾.

However, there was no statistically significant difference between the Q-mix and EDTA groups in terms of the mean value of push-out bond strength. These results agree with Leal et al.; and Keerthana et al.; studies^(30,31). This finding is at odds with the findings of Bayram et al., who discovered that the bond strength of bioceramic root canal sealer was similarly affected by Q-mix and 17% EDTA. The lack of gutta-percha and the methods of irrigation employed in their investigation could account for the discrepancy⁽²⁶⁾.

After being exposed to chitosan solutions, Biodentine showed a similar decrease in resistance to displacement. Exposure to chitosan weakens push-out bonds because of the chelation property's interference with the setting reaction⁽³²⁾. However, Darrag et al. found that a 0.2% chitosan concentration is effective for removing the smear layer without the need for the decalcifying impact of 17% EDTA. Another study by Silva et al. confirmed this by showing that chitosan's modest decalcifying activity was effective at removing the smear layer with minimal erosion of intra-radicular dentin^(33,34).

The NaOCl group had the weakest average push-out bond strength, and the difference between the EDTA and NaOCl groups was statistically significant. This may be because NaOCl root canal irrigant is only capable of dissolving organic molecules, leaving the inorganic smear layer intact⁽³⁵⁾.

CONCLUSION

Within the limitations of this in-vitro study, it can be concluded that:

- None of the used irrigating solutions was capable of completely removing the smear layer within root canals.
- Irrigation with Q-Mix and EDTA irrigation solutions as a final irrigant produces a higher push-out bond strength of Biodentine to radicular dentin relative to Chitosan and NaOCl.

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