Current Science International
Volume: 13 | Issue: 03| July – Sept.| 2024

EISSN:2706-7920 ISSN: 2077-4435
DOI: 10.36632/csi/2024.13.3.28
Journal homepage: www.curresweb.com
Pages: 332-339

Push Out Bond Strength of a Bioceramic Sealer Modified by Magnesium Hydroxide Nanoparticles with Different Concentrations - An *In vitro* Study

Hagar M. Talaat[1], Dalia A.A. Moukarab[2] and Asmaa A. Metwally[3]

[1]Demonstrator in Biomaterials Department, Faculty of Dentistry, Assiut University, Assiut, Egypt.
[2]Associate Professor of Endodontics, Head of Endodontic Department, Faculty of Dentistry, Minia University, Minia, Egypt.
[3]Lecturer of Dental Materials, Biomaterials Department, Faculty of Dentistry, Minia University, Minia, Egypt.

Received: 15 July 2024 **Accepted:** 08 August 2024 **Published:** 15 August 2024

ABSTRACT

Objective: This study evaluated the push out bond strength of a bioceramic sealer modified by nano magnesium hydroxide with different concentrations (NMH). **Methods:** NMH was characterized using transmission electron microscope and X-ray diffraction analysis. Then, added to Ceramoseal HBC by 0.025%, 0.05% and 0.1% in weight. For push out bond strength test; 40 extracted upper central incisors were collected and prepared for endodontic treatment. The teeth were decronated, their root canals were mechanically cleaned and shaped then, grouped randomly into four groups according to the percentages of added nanoparticles. Group I: Bioceramic sealer without added nanoparticles. Group II: Bioceramic sealer + 0.025% NMH. Group III: Bioceramic sealer + 0.05% NMH. Group IV: Bioceramic sealer + 0.1% NMH. Obturation was performed using gutta percha and the tested materials. Specimens were placed in 100% relative humidity for 7 days for sealer setting. After that, 2mm coronal slices were cut from each root to be tested for push out bond strength by applying a deponding force through a computer controlled testing machine till bond failure and bond strength was calculated in MPa. The data were statistically analyzed using One way ANOVA test and Student t-test. **Results:** The results showed that highest mean bond strength value was recorded for group IV (9.72 ± 1.66), while the lowest value was recorded for group I (5.97 ± 1.28). **Conclusion:** Addition of NMH to bioceramic sealers enhanced their bond strength.

Keywords: Push out bond strength, Bioceramic sealers, Nano magnesium hydroxide.

1. Introduction

Endodontic treatment is aiming to provide a hermetic sealed system inside the root canal and prevent any future bacterial infection. It includes canal preparation, disinfection then obturation of the canals using a core filling material and endodontic sealers (Rathi *et al*., 2020)

Endodontic sealers have a very important role as obturating materials. They act as a binding paste between the rigid core and dentinal walls, fill the spaces, voids, lateral and accessory canals thus create a monoblock that is necessary for success of endodontic treatment (Komabayashi *et al*., 2020)

Different types of sealers are available according to their composition: zinc oxide eugenol, glass ionomer, calcium hydroxide, silicone, resin based and bioceramics. Bioceramic sealers have become widely used in endodontics over the last years. The main compositions of these sealers are calcium silicate and/or calcium phosphate. These materials make the sealers have antimicrobial, biocompatible and bioactive properties stimulating new hard tissue deposition (Donnermeyer *et al*., 2019).

Recently, nanomaterials have been used as additives to improve the properties of the endodontic sealers. The nanoscale size of these materials and their increased surface area to volume ratio have improved the efficacy and clinical service of many existing endodontic materials (Zafar *et al*., 2019).

Corresponding Author: Hagar M. Talaat, Demonstrator in Biomaterials Department, Faculty of Dentistry, Assiut University, Assiut, Egypt.
Email: Hager_mt@dent.aun.edu.eg

EISSN: 2706-7920 ISSN: 2077-4435 **DOI: 10.36632/csi/2024.13.3.28**

Nano magnesium hydroxide (NMH) are metallic nanomaterials with various biomedical applications due to their biocompatibility and low cost. These materials were introduced in dentistry owing to their bioactive properties and their antimicrobial activity against oral bacteria (Meng *et al*., 2020).

Bond strength of endodontic sealers is very important in maintaining the adherence of the obturation system to dentin. Therefore, higher bond strength obturation system will achieve a monoblock with the remaining tooth structure, prevent dislodgement of the filling under normal functions and ensure the hermetic seal inside the root canal (Scelza *et al*., 2015).

Therefore, this study evaluated the bond strength of Ceramoseal HBC bioceramic sealer modified by different concentrations of NMH.

2. Materials and Methods

This *in vitro* study was approved by Research Ethical Committee of Faculty of Dentistry, Minia University (RHDIRB2017122004) with protocol number (707/2023) at meeting number (94).

2.1. Preparation of NMH

Magnesium hydroxide nanoparticles were purchased from *Nano Gate company, Egypt*. They were sensitized by sol-gel technique as reported by Wahab *et al.* (2007). 0.2M of magnesium nitrate was prepared then dissolved in 100 ml of deionized water. While stirring, 0.5M of sodium hydroxide solution was added to the magnesium nitrate solution drop by drop. After few minutes, magnesium hydroxide precipitated in the bottom of the beaker. Stirring was continued for 30 minutes. Then, the formed precipitate filtered and washed using methanol to remove any ionic impurity. Centrifugal of precipitate was done at 5000 rpm/min for 5 minutes and dried at room temperature.

2.2. Characterization of nanoparticles

2.2.1. Transmission Electron Microscope (TEM)

Characterization was performed using TEM (HR-TEM, JEOL JEM-2100, Japan) at an accelerating voltage of 200 kV.

2.2.2. X-ray Diffraction (XRD)

X-ray pattern was done by Diffractometer system (XPERT-PRO Powder Diffractometer system, Netherlands), with 2 theta (20^{o} - 80^{o}), with Minimum step size 2Theta: 0.001, and at wavelength ($K\alpha$) = 1.54614^{o}.

2.3. Mixing of the bioceramic sealer with nanoparticles

NMH was mixed with the Ceramoseal HBC bioceramic sealer at concentrations of 0.025%, 0.05% and 0.1% in weight. These concentrations were determined following a pilot study evaluated the flow of the material at a decreasing concentration from 1%. These NMH concentrations didn’t affect the setting of the material. Mixing was done by manufacturer using ultrasonic homogenizer for five minutes to insure thorough homogenization and proper dispersion of nanoparticles inside the sealer. The chemical composition Ceramoseal HBC and NMH shown in table (1).

Table 1: Composition of main materials used in this study.

Brand Name	Description	Composition	Manufacturer	Lot number
CeramoSeal HBC	MTA based bioceramic sealer	Hydroxyapatite, MTA, silicone dioxide, calcium oxides and aluminum oxide plus calcium silicate, titanium dioxide, barium sulfate and excipient.	DM Trust, Egypt	#A291262
Nano Magnesium hydroxide (NMH)	White to light yellow powder, spherical shape, average size 80± 10 nm.	Nanoparticles of magnesium hydroxide with purity 99.9%.	Nano Gate company, Egypt	#MgH20221

2.4. Study design

Bioceramic sealer paste was divided into four groups (n=40) regarding the percentage of added nanoparticles. Group I: Bioceramic sealer without added nanoparticles. Group II: Bioceramic sealer + 0.025% NMH. Group III: Bioceramic sealer + 0.05% NMH. Group IV: Bioceramic sealer + 0.1% NMH. Each group was tested for bond strength properties using push out bond strength method.

2.5. Push out bond strength test

2.5.1. Teeth collection

Forty human extracted maxillary central incisors were collected. The inclusion criteria for the collected teeth were sound recently extracted single straight rooted teeth with single canals and fully formed apices. Any carious teeth, with restorations or any resorptive defects were excluded. Teeth were cleaned mechanically to remove soft tissue debris and stored in 0.1% Thymol solution at 4°c from extraction time till being used for a period not exceeding two months. After collection period, the teeth were removed from Thymol solution and washed under running water to remove thymol residues.

2.5.2. Teeth preparation

Teeth were decoronated at cementoenamel junction using a diamond disk mounted on a low-speed hand piece under water cooling. Working length was determined using #15 K file. The file was inserted into the canal till its tip to be visible from the root apex. Then, the file was withdrawn 1 mm away from apex and working length was determined. Root canals were prepared biomechanically at the determined working length using step-back technique. K files were used for canal preparation till size # 55 K file. Irrigation with 5% NaOCL was used between each instrument. H files were used in a filing action with step back sequence from size 60 to 80. The smear layer was removed using 1 ml of 17% ethylene diaminetetraacetic acid (EDTA). 10 ml saline was used as a final rinse. Excess saline was removed using paper points. Then roots were randomly divided into four groups according to the tested materials. Obturation procedure was performed using gutta percha by lateral condensation technique and materials tested. On completion of these procedures, the specimens were placed in 100% relative humidity for 7 days to insure complete sealer setting.

2.5.3. Test procedure

Test was done following Sarangi *et al.* (2017). A 2mm thick horizontal slices were sectioned from the coronal root part of tested root specimens using a diamond disk perpendicular to the long axis of the root under copious water coolant. After sectioning, each slice was embedded in acrylic resin (Acrostone#224012068) using a custom made teflon mould with two mm thickness and 15 mm diameter as shown in figure (1) and (2). Specimens then mounted to a loading fixture for testing as shown in figure (3). Through a computer controlled testing machine (Universal testing machine Instron 2710-115 max load 5Kn, Germany), all the slices were exposed to compressive force at a 1 mm/min crosshead speed. The load was applied using a 1mm diameter plunger. The plunger tip was positioned so that it only contacts the filling and doesn't cause any load on dentin around it. The load was established apicocoronally. The failure load was measured and bond strength was calculated for each slice as following:

$$\textit{Push out bond strength(mpa)} = \textit{Maximum load (N) / Adhesion area (mm}^2\textit{)}$$

Bonding area for each slice calculated using the formula of:

$$A = 2\,[3.14 \times r \times h],$$

Where r is the radius, and h is the thickness of slices in mm.

EISSN: 2706-7920 ISSN: 2077-4435 **DOI: 10.36632/csi/2024.13.3.28**

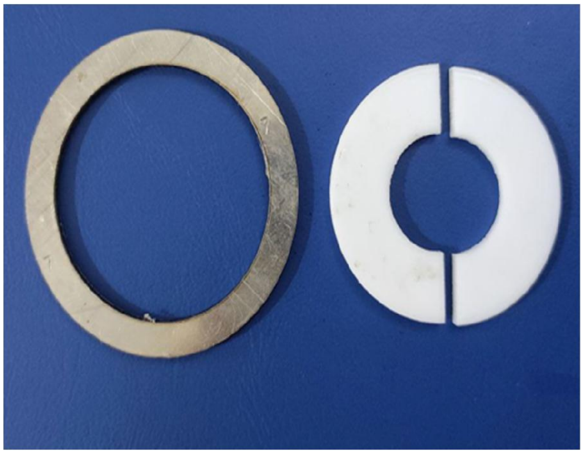

Fig. 1: Showing push out bond strength mould.

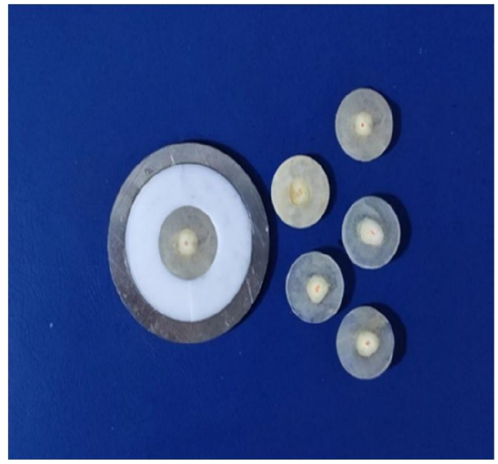

Fig. 2: Single transverse sections were made with 2mm thickness.

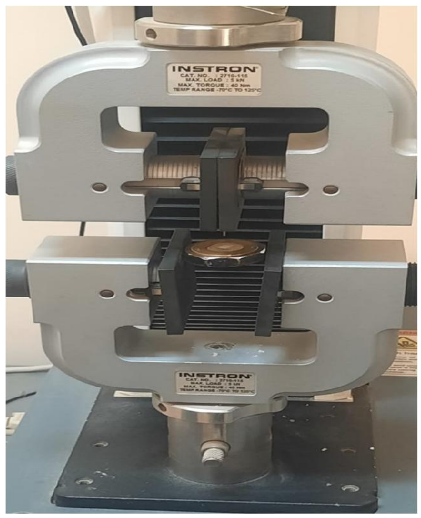

Fig. 3: Slices mounted in a loading fixture and test applied using a universal testing machine

2.6. Statistical analysis

Data analysis was performed using IBM SPSS software package version 20.0. (Armonk, NY: IBM Corp). Normality of distribution was tested by Shapiro-Wilk test. Data was described by mean and standard deviation. Significance of the results was judged at the 5% level.

One-way ANOVA test was used to compare between more than two groups, and Post Hoc test (Tukey) for pairwise comparisons. Student t-test was used to compare between two studied groups.

3. Results

3.1. Characterization of nanoparticles

3.1.1. Transmission Electron Microscope (TEM)

The TEM image of Magnesium hydroxide revealed that the prepared Mg(OH)2 in nanoscale with average size 80± 10nm and spherical like shape as shown in figure (4).

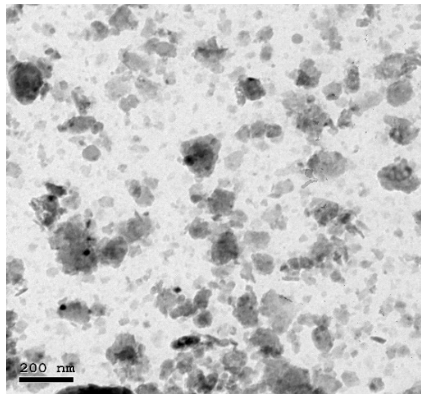

Fig. 4: Shows the TEM image of NMH

3.1.2. X-ray Diffraction (XRD)

The XRD pattern is shown in figure (5). The patterns are consistent with the pattern of crystalline magnesium hydroxide Nanoparticles. The XRD of NMH which was verified using the JCPDS card no. 00-007-0239, shows broad peaks with high intensities of NMH crystallites with a maximum peak from (101) crystal plane at 2 Theta of 38.017 degrees.

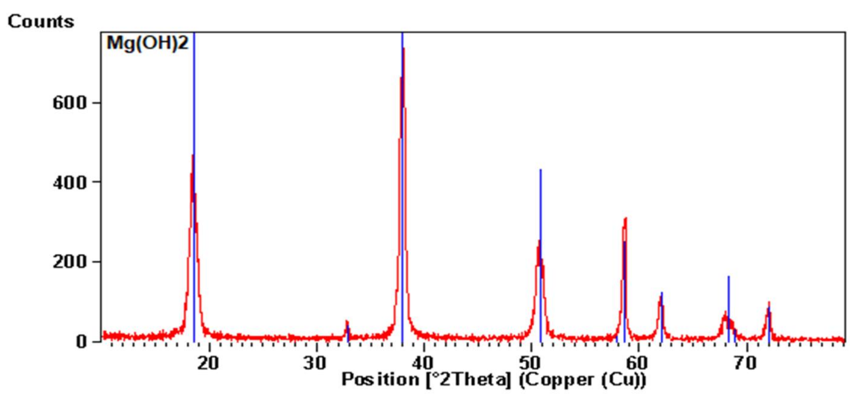

Fig. 5: Shows XRD analysis.

3.2. Push out bond strength

The mean bond strength (MPa) of the tested groups was ranged between 5.97 and 9.72. The highest mean value was recorded for group Ⅳ (9.72 ± 1.66); while the lowest value was recorded for group I (5.97 ± 1.28). The results were statistically significant as presented in table (2) and figure (6).

Table 2: Mean and SD value (MPa) for push out bond strength test.

Groups	Push out bond strength test (MPa)		P value
	Mean mm	±SD	
Group I (Control)	5.97^{b}	±1.28	0.002^{*}
Group II	6.06^{b}	±1.26	
Group III	8.10^{ab}	±1.44	
Group IV	9.72^{a}	±1.66	

Significance level p≤0.05, *significant
Means with the same superscript letter are not significantly different.

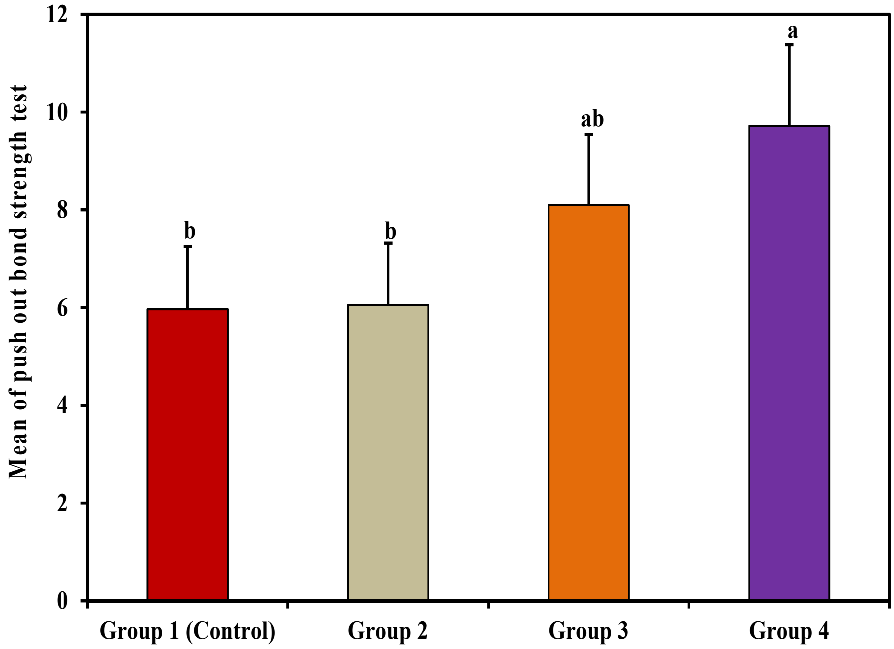

Fig. 6: Bar chart illustrating mean value of push out bond strength test (MPa)of tested groups.

4. Discussion

Endodontic therapy is important in achieving a proper clean sealed root canal system preventing any microbial leakage, recurrent infection and creating a proper environment for the repair of periapical tissues (Rathi *et al.,* 2020).

Bioceramic root canal sealers have been lately used in endodontics due to their biocompatible properties and chemical composition which encourage forming a crystalline structure like natural tooth composition enhancing creation of a strong chemical dentin sealer bond (Al-Haddad *et al*., 2016).

Ceramoseal HBC is a premixed MTA based sealer containing nano hydroxyapatites in its composition in addition to calcium silicate and calcium oxides. This unique composition makes this sealer have an alkaline effect which encourages its antimicrobial effect. Moreover, this sealer has hydrophilic properties and utilizes dentin moisture forming a chemical interstitial bond with dentin which increases sealing and adaptation inside the root canal preventing future bacterial invasion (Emam *et al.,* 2024).

Recently, nanoparticles have become increasingly used in medicine and dentistry. Nanomaterials are those materials with components less than 100 nm in at least one dimension. The nano size of these materials allows them to exhibit properties like reactivity, strength, electrical and optical characteristics which are not present in their larger scale counterparts. The main causes for this change in behavior are their increased surface area and the prevalence of quantum effect (Song *et al*., 2019).

NMH are metallic nanoparticles known for their high antibacterial effects, biocompatible properties and their low cost. Inspired by the positive properties of these NPs, this study was conducted to test the effect of adding them to Ceramoseal HBC bioceramic endodontic sealer with different concentrations (Nakamura *et al*., 2021).

Bond strength is a crucial property of endodontic materials as it describes how firmly the material is bonded to the dentin. Bond strength tests include shear, tensile, and push out bond strength. Push out test is the most widely used owing to its reliability as the test conditions are similar to the clinical situation that the tested materials are placed in prepared canals with a natural canal shape and tubular arrangement. Moreover, the teeth used for push out testing are natural human teeth with their complex structure of dentin, enamel and cementum that mirrors the real life conditions of how closely the tested material will interact with human tissue (Bichile *et al*., 2020).

Regarding to the present study, group Ⅳ presented the highest bond strength when compared to other tested groups that means there is increase in bond strength values with increasing concentrations of NMH. The increase in bond strength of modified groups may be due to the bioactive properties of NMH as mentioned by sun *et al*. (2020) who demonstrated that there is increase in the bioactive properties and osteoblast differentiation in sealer modified by 3% NMH compared to control group.

Regarding this reaction [$Mg(OH)2 \rightarrow Mg^{+2} + 2OH^{-}$], released Mg^{+2} has a significant role that is similar to Ca^{+2}. It is primarily stored in the dentin and plays a crucial role in stabilizing hydroxyapatite crystals forming a strong bonding interface between dentin and sealer. Furthermore, OH^{-} increases the alkaline media which prefers deposition of more hard tissue as demonstrated by Lee *et al*. (2019)**.** Park *et al*. (2019) had also shown that magnesium has a more potent promotion of osteoblast differentiation than calcium.

Based on Farzaneh *et al*. (2018), the nanoparticle size of the released ions compared with their conventional counterparts has a more penetration depth in all regions of the root and the dentinal tubules. Therefore, it is possible to achieve better bonding properties and promote more hard tissue formation.

5. Conclusions

Addition of NMH to bioceramic sealer has a promising enhancement in their push out bond strength.

6. Recommendations

Bioceramic sealers modified by 0.1% NMH are recommended to be used in endodontics for their increased bond strength. Further studies are required to evaluate the biological and bioactive properties of NMH modified bioceramic sealers and other physico-chemical properties of these sealers.

7. Acknowledgments:

Thanks to DM Trust company, Egypt for their support and help.

References

Al-Haddad, A., and Z.A. Che Ab Aziz, 2016. Bioceramic-based root canal sealers: A review. Int. J. Biomater. 1:9753210-20.

Bichile, M.L., R. Mahaparale, S. Mattigatti, K.D. Wahane, and S.V. Raut, 2022. Push-out bond strength of mineral trioxide aggregate with addition of titanium dioxide, silver, and silicon dioxide nanoparticles: An: *in vitro*: comparative study. JCDE. 25(5):541-6.

Donnermeyer, D., S. Bürklein, T. Dammaschke, and E. Schäfer, 2019. Endodontic sealers based on calcium silicates: a systematic review. Odontology,107(4):421-36.

Emam, S.A., A.H. Mahran, and M.M. Elshafei, 2024. Evaluation of cytotoxicity and adaptability of a novel bioceramic root canal sealer: An *in vitro* and scanning electron microscope study. JCDE., 27(3):326-30.

Farzaneh, B., S. Azadnia, and R. Fekrazad, 2018. Comparison of the permeability rate of nanoparticle calcium hydroxide and conventional calcium hydroxide using a fluorescence microscope. J. Dent. Res., 15(6):385-90.

Komabayashi, T., D. Colmenar, N. Cvach, A. Bhat, C. Primus, and Y. Imai, 2020. Comprehensive review of current endodontic sealers. Dent Mater J., 39(5):703-20.

Lee, S.K., C.-M. Han, W. Park, I.H. Kim, Y.K. Joung, and D.K. Han, 2019. Synergistically enhanced osteoconductivity and anti-inflammation of PLGA/β-TCP/Mg (OH) 2 composite for orthopedic applications. Mater. Sci. Eng., C.,94:65-75.

Meng, Y., D. Zhang, X. Jia, K. Xiao, X. Lin, Y. Yang, et al., 2020. Antimicrobial activity of nano-magnesium hydroxide against oral bacteria and application in root canal sealer. MSM, 26:1-10.

Nakamura, Y., K. Okita, D, Kudo, D.N.D, Phuong, Y. Iwamoto, Y. Yoshioka, et al., 2021. Magnesium hydroxide nanoparticles kill exponentially growing and persister Escherichia coli cells by causing physical damage. J. Nanomater.,11(6):1584-95.

Park, J.-W., T. Hanawa, and J.-H. Chung, 2019. The relative effects of Ca and Mg ions on MSC osteogenesis in the surface modification of microrough Ti implants. Int J Nanomedicine, 5697-711.

Rathi, C.H., M. Chandak, P. Nikhade, N. Mankar, M. Chandak, S. Khatod, et al., 2020. Functions of root canal sealers-a review. J Evolution Med Dent Sci., 9(17):1454-58.

Sarangi, P., R. Mallick, S.K. Satapathy, G. Sharma, F. Kouser, and S. Mohapatra, 2017. An *In vitro* Comparison of pushout bond strength of resilon with MetaSEAL and AH plus sealers. Contemp. Clin. Dent., 8(4):613-6.

Scelza, M., D. Da Silva, P. Scelza, F. De Noronha, I. Barbosa, E. Souza, *et al*., 2015. Influence of a new push-out test method on the bond strength of three resin-based sealers. Int. Endod. J., 48(8):801-6.

Song, W., and S. Ge, 2019. Application of antimicrobial nanoparticles in dentistry. Molecules., 24(6):1033-48.

Sun, X., A. Sun, X. Jia, S. Jin, D. Zhang, K. Xiao, *et al*., 2020. *In vitro* bioactivity of AH plus with the addition of nano-magnesium hydroxide. Ann. Transl. Med., 8(6):313-26.

Wahab, R., S. Ansari, M.A. Dar, Y.S. Kim, H.S. Shin, editors, 2007. Synthesis of magnesium oxide nanoparticles by sol-gel process. Mater. Sci. Forum, 558:983-86.

Zafar, M.S., A.A. Alnazzawi, M. Alrahabi, M.A. Fareed, S. Najeeb, and Z. Khurshid, 2019. Nanotechnology and nanomaterials in dentistry. J. Dent. Biomater: Elsevier, 477-505.