Original article *International Journal of Dental Materials 2023;5(2)* ISSN:2582-2209

The effect of irrigating solutions on the hydration of tricalcium silicate cements: an *in vitro* study

K. Shivani Shreya [1,*], G. Deepthi[2], Md Khwaja Moinuddin[2], R. Pallavi [1], Asha Reddy[2], Amulya Reddy[2]

[1]Postgraduate Student, Department of Conservative Dentistry and Endodontics, Sri Balaji Dental College, Yenkepally, Moinabad, Ranga Reddy District, Telangana, India.

[2]Associate Professor, Department of Conservative Dentistry and Endodontics, Sri Balaji Dental College, Yenkepally, Moinabad, Ranga Reddy District, Telangana, India.

Article History

Received *25th March 2023*

Accepted *24th May 2023*

Available online *3rd July 2023*

*Correspondence

K. Shivani Shreya

Postgraduate Student,

Department of Conservative Dentistry and Endodontics,

Sri Balaji Dental College, Yenkepally,

Moinabad, Ranga Reddy District,

Telangana, India.

E-mail: kshivanishreya@gmail.com

DOI: *http://dx.doi.org/10.37983/IJDM.2023.5202*

Abstract

Background: Calcium silicate cements are hydraulic cements, routinely used for perforation repairs. During such repairs, these cements are invariably exposed to irrigating solutions.

Aim: This study aimed to understand the effect of irrigating solutions on the hydration of calcium silicate cements.

Materials and Methods: Sixty extracted teeth were taken and horizontal sections of 2mm were obtained. These samples were randomly divided into two groups viz. Biodentine and BioMTA Plus groups later these cements were condensed into the canal spaces and allowed to set until their setting time. These samples were further subdivided and allowed to encounter three irrigating solutions viz. Normal saline, 17% EDTA, and 2% Chlorhexidine for 5 minutes. These were allowed to mature in an incubator for seven days and subjected to Scanning Electron Microscopy and Energy Dispersive X-ray analysis.

Results: The SEM analysis of the Biodentine/control group displayed a petal-like appearance, with a Ca/Si ratio of 2. Whereas, the Biodentine/Normal saline, Biodentine/17% EDTA and Biodentine/2% Chlorhexidine group displayed crumbled paper-like appearance. The Ca/Si ratios for the Biodentine/Normal saline, Biodentine/17% EDTA and Biodentine/2% Chlorhexidine were 2.72, 1.6, and 4.21, respectively. In the BioMTA Plus group, all the SEM analyses displayed round crystalline structures in all groups. The Ca/Si ratio of BioMTA Plus/Control, BioMTA Plus/17% EDTA and BioMTA Plus/2% Chlorhexidine were 25.5, 17.42, 24.1, and 39.4, respectively.

Conclusion: The study concluded that the irrigating solutions did not affect the hydration mechanism of Biodentine and BioMTA Plus despite the variations in the Ca/Si ratios and surface morphology.

Keywords: Calcium silicate cement, Perforation repairs, Hydration, BioMTA Plus, Scanning Electron Microscopy, Energy Dispersive X-ray Analysis, Irrigating solutions, and Ca/Si ratios.

1. Introduction

Torabinejad pioneered tricalcium silicate cement, mineral trioxide aggregate (MTA), as a root-end filling material in the 1900s [1]. The original formulation consisted of 50-75% (wt.) calcium oxide and 15-25% silicon dioxide [2]. Upon trituration, these oxides formed tricalcium silicate, dicalcium silicate, tricalcium aluminate, tetra calcium aluminoferrite, calcium sulfate, and bismuth oxide [3]. In addition to root-end filling, MTA is also used for pulp capping, perforation repairs, resorptions, apexification and apexogenesis. Despite its clinical success, drawbacks like increased setting time, high solubility, difficult handling characteristics, and discolouration potential [4] prompted the development of modified mineral trioxide aggregate, BioMTA Plus. It is dispensed as powder and liquid and has a shorter setting time than its parent material. It chemically comprises calcium oxide, hydroxyapatite, oxides of silicon, iron, aluminium, sodium, potassium, bismuth, magnesium, zirconium, and calcium phosphate. The silicon and calcium compounds support tissue regeneration after wall perforation and intracanal resorption. It also contains hydroxyapatite, which integrates the compound into the bone structure [5].

Biodentine is a dentin replacement material and contains tricalcium silicate, calcium carbonate, zirconium oxide, calcium oxide, and calcium chloride [6]. The properties of Biodentine have been improved by incorporating an accelerator and softener and substituting bismuth oxide with zirconium oxide [6-8]. Compared to MTA, Biodentine is superior due to its excellent biocompatibility and bioactivity, sealing ability, and compressive strength [6,9]. It has decreased discolouration potential [10-12] and increased aesthetics [13]. These cements are widely used in pulp capping, perforation repairs, intracanal resorption, root-end filling material, pulp amputation, apexification, and apexogenesis [6,14].

In the clinical case of perforations, the attempt to repair the defect prior to cleaning and shaping prevents iatrogenic damage to the peri radicular tissues caused due to the extrusion of irrigants, gutta-percha, root canal sealer, and contaminants [15].

The commonly used repair materials, calcium silicate cements invariably encounter different irrigating solutions during cleaning and shaping. Lee et al. stated that 17% EDTA retards the hydration of mineral trioxide aggregate [16]. Nandini et al. suggested that 2% CHX should be avoided for 24 hrs after placement of WMTA [17]. Guneser et al. concluded that 3.5% NaOCl, 2% CHX, and normal saline altered the surface topography of Biodentine but was more resistant to dislodging forces [18]. However, there are no studies evaluating the effect of irrigating solutions on the hydration mechanism of Bio MTA Plus. Thus, this study aimed to evaluate the effect of irrigating solutions on the hydration mechanism of Biodentine and novel Bio MTA Plus.

2. Materials and methods

BioMTA Plus (Cerkamed Company, Poland) and Biodentine (Septodont, France) materials were used in the study. Sixty extracted teeth were collected and were decoronated at the cemento-dentinal junction. The mid-root dentin was sliced horizontally to obtain a thickness of 2 mm. In each slice, the canal space was enlarged using gates glidden drill (MANI, INC) to size 6. These cut sections were divided randomly into two groups, Group 1: BioMTA Plus and Group 2: Biodentine, with 32 in each.

Both BioMTA Plus and Biodentine materials were manipulated according to the manufacturer's recommendations and condensed into the root canals of their respective group cut sections and were allowed to set. Then, the samples from each group were divided into four subgroups (A, B, C, and D) with 8 (n=8) in each based on the type of irrigating solutions. The samples were soaked for five minutes in normal saline, 2% Chlorohexidine (CHX), and 17% EDTA solutions (subgroups B, C, and D, respectively). Subgroup A is the control group that was not exposed to any irrigating solution. The samples of both groups were soaked in phosphate-buffered saline solution for seven days in an incubator at 37 degrees centigrade until testing.

The samples were platinum sputtered and subjected to Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray (EDX) analysis at 6k magnification.

The statistical analysis has been performed using SPSS (Statistical Package for Social Science) 26.0, USA. The one-way ANOVA test has been used to statistically analyze the Ca/Si ratios obtained from the control and the other subgroups to understand the amount of hydration that has taken place. The confidence intervals were set at 95% with a P-value <0.05 being statistically significant.

3. Results

The SEM and EDX analysis of Biodentine and BioMTA Plus specimens are given in Figures 1 to 4. The scanning electron microscopy for the Biodentine control group showed uniformly arranged petal-like structures. The Biodentine/Normal saline group exhibited a crumbled paper-like appearance. The Biodentine/2% CHX group had a similar appearance to the control group, but two samples displayed needle-like crystals interspersed with granular amorphous structures. The uniformly distributed granular structures were observed in the Biodentine/17% EDTA group (Figure 1).

The scanning electron microscopy for Bio MTA Plus/control group displayed small round granular structures. Bio MTA Plus/Normal saline, Bio MTA Plus/CHX, and Bio MTA Plus/17%EDTA showed similar appearances to that of the control groups (Figure 3).

The EDAX analysis of both the groups of Biodentine and BioMTA Plus groups displayed peaks of Ca, Si, O, and P (Figures 2 and 4). However, the Ca/Si ratios of the subgroups showed variations. In the present study, the Ca/Si ratios of the BioMTA Plus group were shown to be higher than the Biodentine group.

The Ca/Si ratio for Biodentine/control group was found to be 2 (Figure 2A). The Ca/Si ratio for Biodentine/Normal saline, Biodentine/EDTA, and Biodentine/CHX was 2.72, 1.6, and 4.21, respectively (Figures 2B, 2C and 2D). The Ca/Si ratio for Biodentine/CHX group was greater than the Biodentine/Normal saline group followed by the Biodentine control group and Biodentine/17% EDTA (Figure 5).

The BioMTA Plus/control group was found to have a Ca/Si ratio of 25.5 (Figure 2A). The BioMTA Plus/Normal saline, BioMTA Plus/EDTA, and BioMTA Plus/CHX have a Ca/Si ratio of 17.42, 24.1, and 39.4, respectively (Figures. 4B, 4C and 4D). Whereas the Ca/Si ratios for BioMTA Plus/CHX were more than the BioMTA Plus/control group, BioMTA Plus/EDTA group, and Bio MTA Plus/normal saline (Figure 6).

Though the subgroups have shown variations in terms of Ca/Si ratios, they were proven to be statistically insignificant (P-value >0.05). This means that the difference in the ratios of Ca/Si shown has no significant effect on hydration.

4. Discussion

The tricalcium silicate cements are also known as hydraulic cements as they mature in the presence of moisture. In the hydration reaction of MTA, tricalcium silicate and dicalcium silicate dissolves to form calcium silicate hydrate. The calcium silicate hydrate consists of calcium and silicon from the powder and hydroxyl ions from the mixing liquid. Upon completion of the hydration, a porous calcium silicate gel is formed. The calcium hydroxide is formed from the excess calcium ions leached from the powder particles [19].

The hydration of Biodentine begins with the reaction between tricalcium silicate cement and water. It leads to the formation of calcium silicate hydrate gel on the cement particles and calcium hydroxide. Upon further hydration, the calcium silicate hydrate coats the unreacted powder particles and retard the reaction. The calcium carbonate acts as a nucleation site for calcium silicate hydrate that reduces the induction period [20].

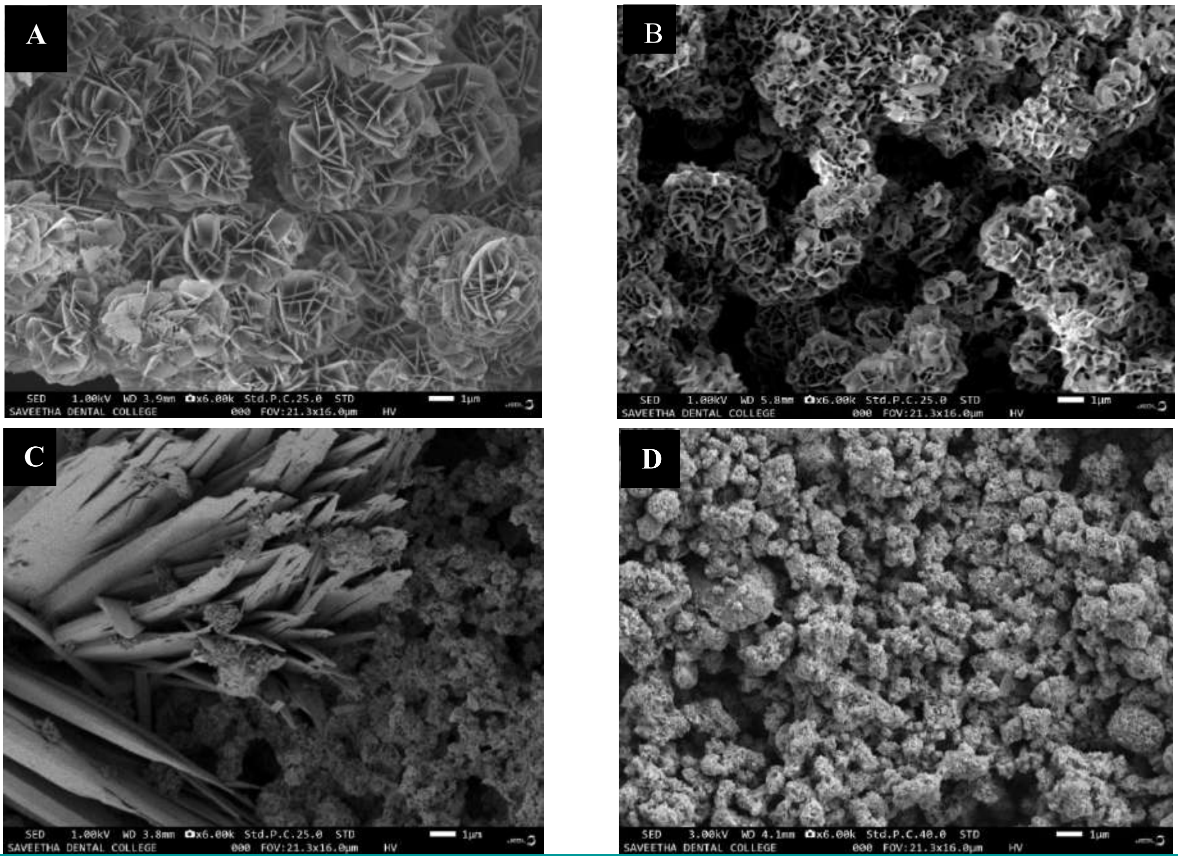

FIGURE 1. SEM images of Biodentine A) Control group, B) Biodentine/ Normal saline, C) Biodentine/ 2% CHX, D) Biodentine/ 17% EDTA.

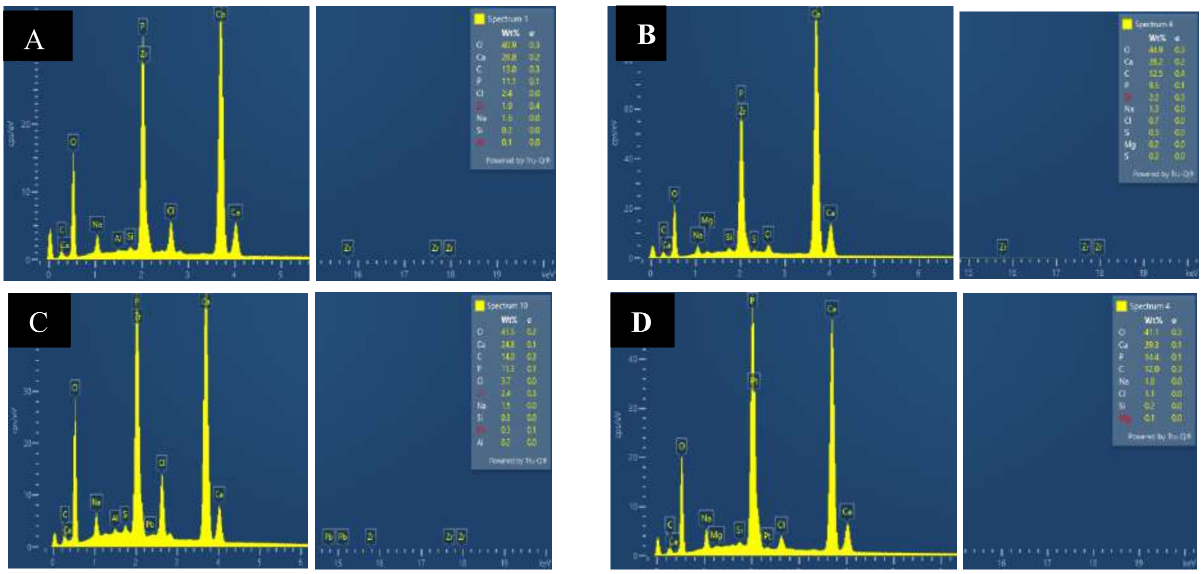

Figure 2. EDX images of Biodentine A) Control group, B) Biodentine/ Normal saline, C) Biodentine/ 2% CHX, D) Biodentine/ 17% EDTA.

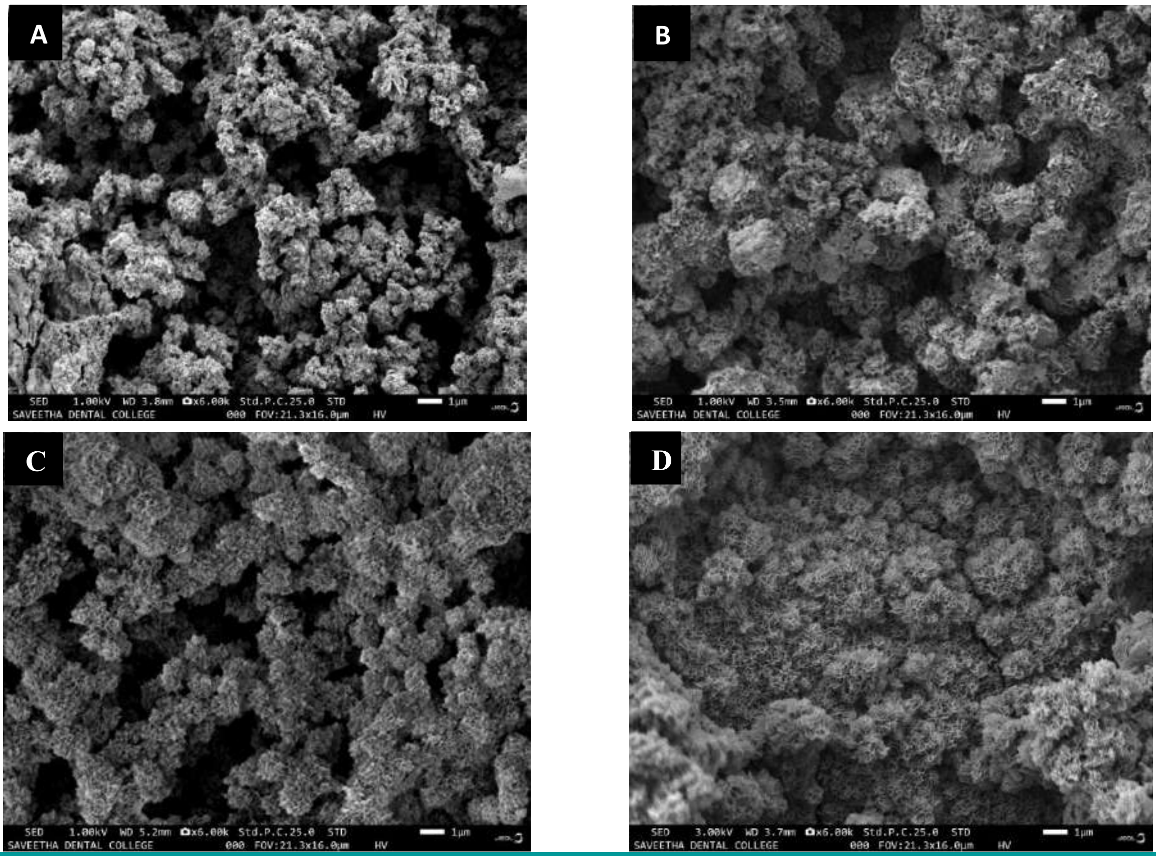

FIGURE 3: SEM images of BioMTA Plus. A) Control group, B) BioMTA Plus/Normal saline , C) BioMTA Plus/2% CHX, D) BioMTA Plus/17% EDTA.

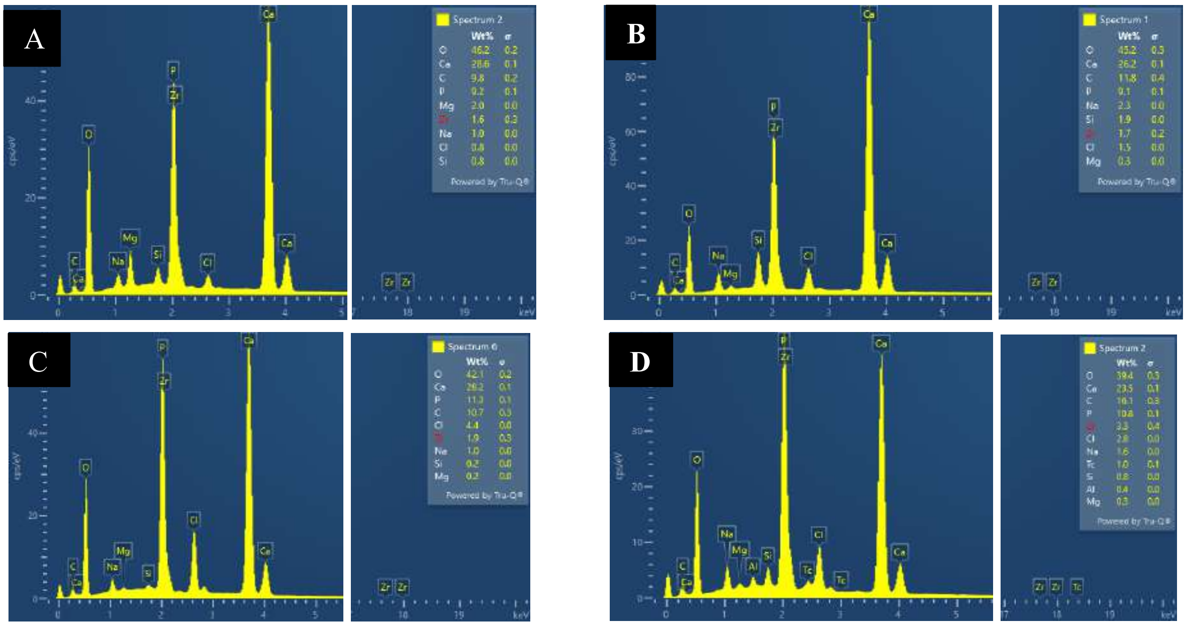

FIGURE 4. EDAX images of BioMTA Plus. A) Control group, B) BioMTA Plus/Normal saline , C) BioMTA Plus/2% CHX, D) BioMTA Plus/17% EDTA.

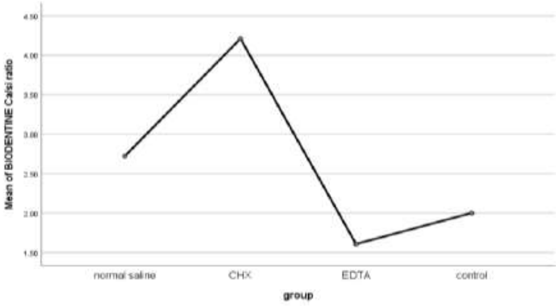

Figure 5. Ca/Si ratio mean plots of Biodentine

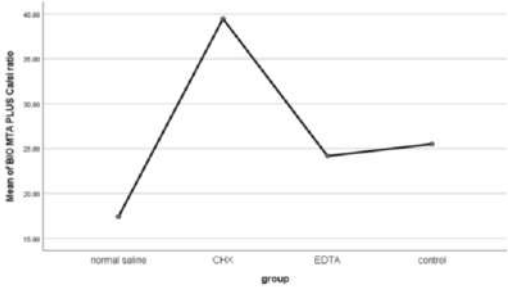

Figure 6. Ca/Si ratio mean plot of BIO MTAPLUS

The elements present in the hydration products attain their maximum concentration on the 7th day [21]. Hence, in the present study, the samples were allowed to hydrate for the same duration.

The SEM images of the Biodentine/control group showed a petal-like appearance, in contrast to other studies [22-24]. The SEM images of the Biodentine/Normal saline group displayed a crumble paper-like appearance. Two samples of Biodentine/2% CHX showed needle-like crystals interspersed with granular amorphous structures and Biodentine/17% EDTA group displayed granular, amorphous structures.

The SEM images of the BioMTA Plus/control group showed round granular crystalline structures. The other subgroups displayed similar morphology to the control group.

The Ca/Si ratios indicate the amount of hydration that occurred. The Ca/Si ratios greater than 1.5 indicate the presence of the hydration products. The Ca/Si ratios of all the subgroups were greater than 1.5, indicating that hydration has taken place [25].
The Ca/Si ratio for BioMTA Plus/EDTA group and Biodentine/EDTA group was found to be lesser than the control group similar to the study by LEE et al. [16]. The reason is that the EDTA is a known chelator, which chelates the calcium found in Bio MTA Plus cement thus, reducing the Ca/Si ratio. A similar reason can be explained for the Biodentine/ 17% EDTA subgroup.
The Ca/Si ratio for Biodentine/Normal saline is greater than that of the control groups, as the higher concentrations of Na and Cl ions might act as a nucleation site for hydration [16]. Whereas the Ca/Si ratio for Bio MTA Plus/Normal saline group is lesser than the control group, further research is required to address this.
As stated by Nandini et al. [17], CHX irrigation should be avoided for 24hrs after the placement of WMTA. However, in the present study, no such caution was observed as the Ca/Si ratio was found to be higher than the controls in both groups, suggesting that the hydration might not have been affected. Further research is required to address this and understanding of the concept.
Despite the variations in the Ca/Si ratios and the surface morphology in the groups, there has been no statistically significant difference in the amount of hydration.

This study did not evaluate the effect of blood or saliva contamination on the hydration mechanism. Further studies are required to understand this aspect.

5. Clinical significance

The present study analysed the effects of irrigating solutions on the hydration of tricalcium silicate cements during furcation repairs. As these repairs were attempted prior to cleaning and shaping, they inevitably come in contact with the irrigating solutions that might interfere with the hydration thus affecting the physical characteristics.

6. Conclusion

The effect of irrigating solutions on the hydration mechanism of calcium silicate cement has been discussed and concluded that there is no statistically significant difference in irrigating with the solutions used in this study. However, these cements must be allowed to be set until the manufacturers mentioned setting time to allow the hydration to occur.

Conflicts of interest: Authors declared no conflicts of interest.

Financial support: None

References

1. Torabinejad M, Watson TF, Ford TR. Sealing ability of a mineral trioxide aggregatewhen used as a root end filling material. J Endod 1993;19:591–5. https://doi.org/10.1016/S0099-2399(06)80271-2
2. Torabinejad M, White DJ. Tooth filling material and use. *US Patent Number 5,769,638, 1995.*
3. Camilleri J. The chemical composition of mineral trioxide aggregate. J Conserv Dent. 2008 Oct;11(4):141-3. https://doi.org/10.4103/0972-0707.48834

4. Parirokh M, Torabinejad M. Mineral trioxide aggregate: a comprehensive literature review--Part III: Clinical applications, drawbacks, and mechanism of action. J Endod. 2010 Mar;36(3):400-3. https://doi.org/10.1016/j.joen.2009.09.009
5. BioMTA+, CERKAMED, accessed 26 June 2022, < https://cerkamed.com/product/bio-mta-plus/>
6. Kaur M, Singh H, Dhillon JS, Batra M, Saini M. MTA versus Biodentine: Review of literature with a comparative analysis. J Clin Diagn Res. 2017;11:ZG01–ZG05. https://doi.org/10.7860/JCDR/2017/25840.10374
7. Yoldaş SE, Bani M, Atabek D, Bodur H. Comparison of the potential discoloration effect of bioaggregate, biodentine, and white mineral trioxide aggregate on bovine teeth: in vitro research. J Endod. 2016 Dec 1;42(12):1815-8. https://doi.org/10.1016/j.joen.2016.08.020
8. Kaup M, Schäfer E, Dammaschke T. An in vitro study of different material properties of Biodentine compared to ProRoot MTA. Head Face Med. 2015 Dec;11(1):1-8. https://doi.org/10.1186/s13005-015-0074-9
9. Berger T, Baratz AZ, Gutmann JL. In vitro investigations into the etiology of mineral trioxide tooth staining. J Conserv Dent. 2014 Nov;17(6):526–530. https://doi.org/10.4103/0972-0707.144584
10. Ramos JC, Palma PJ, Nascimento R, Caramelo F, Messias A, Vinagre A, Santos JM. 1-year in vitro evaluation of tooth discoloration induced by 2 calcium silicate–based cements. J Endod. 2016 Sep 1;42(9):1403-7. https://doi.org/10.1016/j.joen.2016.06.012
11. Shokouhinejad N, Nekoofar MH, Pirmoazen S, Shamshiri AR, Dummer PM. Evaluation and comparison of occurrence of tooth discoloration after the application of various calcium silicate–based cements: an ex vivo study. J Endod. 2016 Jan 1;42(1):140-4. https://doi.org/10.1016/j.joen.2015.08.034
12. Kohli MR, Yamaguchi M, Setzer FC, Karabucak B. Spectrophotometric analysis of coronal tooth discoloration induced by various bioceramic cements and other endodontic materials. J Endod. 2015 Nov 1;41(11):1862-6. https://doi.org/10.1016/j.joen.2015.07.003
13. Kahler B, Rossi-Fedele G. A Review of Tooth Discoloration after Regenerative Endodontic Therapy. J Endod. 2016 Apr;42(4):563-9. https://doi.org/10.1016/j.joen.2015.12.022
14. Duarte MAH, Marciano MA, Vivan RR, Tanomaru Filho M, Tanomaru JMG, Camilleri J. Tricalcium silicate-based cements: properties and modifications. Braz Oral Res. 2018 Oct 18;32(suppl 1):e70. https://doi.org/10.1590/1807-3107bor-2018.vol32.0070
15. Yan P, Peng B, Fan B, Fan M, Bian Z. The effects of sodium hypochlorite (5.25%), Chlorhexidine (2%), and Glyde File Prep on the bond strength of MTA-dentin. J Endod. 2006 Jan;32(1):58-60. https://doi.org/10.1016/j.joen.2005.10.016
16. Lee YL, Lin FH, Wang WH, Ritchie HH, Lan WH, Lin CP. Effects of EDTA on the hydration mechanism of mineral trioxide aggregate. J Dent Res. 2007 Jun;86(6):534-8. https://doi.org/10.1177/154405910708600609
17. Nandini S, Natanasabapathy V, Shivanna S. Effect of various chemicals as solvents on the dissolution of set white mineral trioxide aggregate: an in vitro study. J Endod2010;36:135–8. https://doi.org/10.1016/j.joen.2009.09.005
18. Guneser MB, Akbulut MB, Eldeniz AU. Effect of various endodontic irrigants on the push-out bond strength of Biodentine and conventional root perforation repair materials. J Endod. 2013 Mar;39(3):380-4. https://doi.org/10.1016/j.joen.2012.11.033
19. Camilleri J. The chemical composition of mineral trioxide aggregate. J Conserv Dent. 2008 Oct;11(4):141-3. https://doi.org/10.4103/0972-0707.48834
20. Camilleri J, Sorrentino F, Damidot D. Investigation of the hydration and bioactivity of radiopacified tricalcium silicate cement, Biodentine and MTA Angelus. Dent Mater. 2013 May;29(5):580-93. https://doi.org/10.1016/j.dental.2013.03.007
21. Shokouhinejad N, Nekoofar MH, Razmi H, Sajadi S, Davies TE, Saghiri MA, Gorjestani H, Dummer PM. Bioactivity of EndoSequence root repair material and bioaggregate. Int Endod J. 2012 Dec;45(12):1127-34. https://doi.org/10.1111/j.1365-2591.2012.02083.x
22. Ashofteh Yazdi K, Ghabraei S, Bolhari B, Kafili M, Meraji N, Nekoofar MH, Dummer PMH. Microstructure and chemical analysis of four calcium silicate-based cement in different environmental conditions. Clin Oral Investig. 2019 Jan;23(1):43-52. https://doi.org/10.1007/s00784-018-2394-1
23. Elbanna A, Atta D, Sherief DI. *In vitro* bioactivity of newly introduced dual-cured resin-modified calcium silicate cement. Dent Res J (Isfahan). 2022 Jan 28;19:1. https://doi.org/10.4103/1735-3327.336686
24. Han L, Okiji T. Bioactivity evaluation of three calcium silicate-based endodontic materials. Int Endod J. 2013 Sep;46(9):808-14. https://doi.org/10.1111/iej.12062
25. Cong X, Kirkpatrick RJ (1996). 29Si MAS NMR study of the structure of calcium silicate hydrate. Adv Cem Based Mater 3:144-156. https://doi.org/10.1016/S1065-7355(96)90046-2

How to cite this article: Shivani Shreya K, Deepthi G, Khwaja Moinuddin Md, Pallavi R, Asha R, Amulya R. The effect of irrigating solutions on the hydration of tricalcium silicate cements: an *in vitro* study. Int J Dent Mater. 2023;5(2):31-36. DOI:*http://dx.doi.org/10.37983/IJDM.2023.5202*