



ORIGINAL ARTICLE

Chemico-physical and mechanical evaluation of three calcium silicate-based pulp capping materials



Inas M. Al-Sherbiny^a, Mona H. Farid^b, Ashraf M. Abu-Seida^{c,*},
Inas T. Motawea^a, Hagar A. Bastawy^d

^a Dental Biomaterials Department, Faculty of Dental Medicine for Girls, Al-Azhar University, Cairo, Egypt

^b Oral Biology Department, Faculty of Dental Medicine for Girls, Al-Azhar University, Cairo, Egypt

^c Department of Surgery, Anesthesiology & Radiology, Faculty of Veterinary Medicine, Cairo University, Giza, Egypt

^d Department of Endodontics, Faculty of Dentistry, King Abdulaziz University (KAU), Jeddah, Saudi Arabia

Received 2 November 2019; revised 4 February 2020; accepted 10 February 2020

Available online 17 February 2020

KEYWORDS

Apatite forming;
Biodentine;
Biosealer;
MTA;
Lap shear strength;
Porosity

Abstract *Aim:* This study compared biointeractivity (pH of soaking water and calcium ions), porosity, water sorption, solubility, compressive strength, lap shear strength, as well as the apatite forming ability of three calcium silicate-based capping materials: Mineral trioxide aggregate (MTA), Biodentine (BD) and Tech Biosealer capping (BS).

Methods: One hundred and five discs of the tested materials were prepared in compliance with the manufacturer's instructions. The materials' pastes were placed in Teflon molds and allowed to set before testing. The pH and Ca⁺² ions were measured by a potentiometric method. Porosity, water sorption, and solubility were calculated through the measurement of initial mass, mass, saturated mass and dry mass. Apatite forming ability was measured by an Environmental Scanning Electron Microscope that was connected to a secondary electron detector for energy dispersive X-ray analysis. Meanwhile compressive strength was measured by a computer controlled universal testing machine. Lap shear strength was measured by computer software on the testing machine. All data were statistically analyzed.

Results: The tested materials showed Ca ions release and alkalization, which decreased with soaking time. The BD exhibited a very high Ca release at both short (3 h) and long times (28 days). Significant high values of open and apparent porosities, water sorption, and solubility were

* Corresponding author at: Department of Surgery, Anesthesiology & Radiology, Faculty of Veterinary Medicine, Cairo University, Giza – Giza Square 12211, Egypt.

E-mail address: ashrafseida@cu.edu.eg (A.M. Abu-Seida).

Peer review under responsibility of King Saud University.



measured for BS, which was followed by the MTA then BD ($P < .05$). The BD had significant higher compressive and lap shear bond strength than the MTA and BS ($P < .05$).

Conclusion: MTA, BD and BS are biointeractive bioactive materials that possess the ability to release ions and form calcium phosphate deposits. Unlike BD, BS is incapable of alternating MTA in pulp capping due to its high porosities, water sorption and solubility, as well as poor compressive and lap shear bond strength.

© 2020 The Authors. Production and hosting by Elsevier B.V. on behalf of King Saud University. This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>).

1. Introduction

Several biomaterials have been utilized in the past for pulp capping with various degrees of success. This success is predicated on the material's biocompatibility and ability for prevention of bacterial microleakage (Camps et al., 2000).

For several decades, calcium hydroxide ($\text{Ca}(\text{OH})_2$) had been used as a gold standard for pulp capping (Cox and Suzuki, 1994). However, $\text{Ca}(\text{OH})_2$ produces weak sealing to dentin, dissolves with time and produces tunnel defects in the reparative dentin bridge (El-Ashry et al., 2013).

Since two decades, ProRoot Mineral Trioxide Aggregates (MTA) has been used as a pulp capping material with rapid and thick reparative dentine formation. However, MTA also suffers from certain shortcomings, such as prolonged setting time, poor handling properties, and high price (Accorinte et al., 2008). The biggest predicament associated with MTA is its aesthetic impact attributed to tooth discoloration (Vallés et al., 2013; Ramos et al., 2016). Therefore, several agents are continuously developed to overcome the disadvantages of $\text{Ca}(\text{OH})_2$ and MTA (El Ashry, 2016; Negm et al., 2016; Saleh et al., 2016; Negm et al., 2017).

Biodentine is a calcium silicate-based material developed in 2011 with improved handling characteristics and shorter setting time than MTA (Richard and Marie, 2011).

Recently, a new calcium silicate material referred to as Tech Biosealer capping (TBC) was introduced to the market with high biocompatibility and good biological action (Gandolfi et al., 2013a). According to the manufacturer's instructions (Isasan srl, Rovello Porro, Co, Italy), TBC is capable of being used as a pulp capping material with a setting time of 55 h. TBC is formed by powder: mixture of white Calcium Enriched Mixture (CEM), calcium sulfate, calcium chloride, montmorillonite, and liquid: DPBS (Dulbecco's Phosphate Buffered Saline).

This study compared white MTA, Biodentine and TBC in terms of biointeractivity, porosity, water sorption, solubility, compressive strength, lap shear strength and apatite forming ability.

2. Materials and methods

2.1. Materials and samples' preparation

This research was carried out at Faculty of Dental Medicine for Girls, Al-Azhar University, Cairo, Egypt. Biodentine, Tech-Biosealer capping material and ProRoot MTA were used in the present study. A total of 105 discs of the three tested materials was prepared according to Vallés et al. (2013) and

Ramos et al. (2016). The materials' cements were placed in Teflon molds (8.0 ± 0.1 mm diameter and 1.6 ± 0.1 mm thickness), allowed to set at 37°C and 99% relative humidity for 24 h, then removed from the molds (Fig. 1) and tested as follows:

2.2. Biointeractivity

The alkalizing activity (pH of soaking water) and calcium ion release were measured. Five discs of each material were individually immersed vertically in 10 mL of deionized water (pH 6.6) in closed tubes and stored at 37°C . The soaking water was collected and replaced at 3 h and 24 h, as well as 3, 7, 14, and 28 days. The collected water was individually analyzed for pH and Ca^{+2} ions by a potentiometric method (Gandolfi et al., 2013a). The pH was measured using a selected temperature-compensated electrode (Sen Tix Sur WTW, Weilheim, Germany) that was connected to a multi-parameter laboratory meter (inoLab 750 WTW, Weilheim, Germany) previously calibrated with standard solutions. Calcium ions were measured as p.p.m. using a calcium probe (Calcium ion electrode, Eutech instruments Pte Ltd, Singapore, Singapore) after addition of 0.2 mL (2%) of the ionic strength adjuster (ISA, 4 moL/L KCl, WTW, Weilheim, Germany).

2.3. Physical properties

Porosity, water sorption and solubility were measured. A total of 5 discs of each tested material was individually weighted to calculate the initial mass (I) by an analytical balance (Bel Engineering series M, Monza, Italy), then immersed vertically in 20 mL of distilled water and the mass (S) was determined while suspended in water. After 24 h of immersion, the discs were removed from the water and any excess water was removed by a moistened filter paper and the saturated mass (M) was weighted. Finally, the discs were dried at 37°C . Each disc was weighted 3 times for confirming of the complete dryness. The final dry mass (D) was recorded. Porosity, water sorption and solubility were calculated according to Gandolfi et al. (2013a) as follows:

$\text{VOP} = \text{M} - \text{D}$ (Where : VOP = volume of open pores, M = saturated mass, D = dry mass).

$\text{VIP} = \text{D} - \text{S}$ (Where : VIP = volume of the impervious portion, D = dry mass, S = suspended mass)

$\text{V} = \text{M} - \text{S}$ (Where : V = exterior volume, M = saturated mass, S = suspended mass).



Fig. 1 The prepared discs samples of mineral trioxide aggregates (MTA), Biodentine (BD) and Biosealer (BS).

The apparent porosity (P) was calculated in percentage, following Archimedes' principle.

$$P = [(M - D)/V] \times 100 \text{ (Where : } M = \text{ saturated mass, } D = \text{ dry mass, } V = \text{ exterior volume).}$$

The water sorption (A) was calculated in percentage as follows:

$$A = [(M - D)/D] \times 100 \text{ (Where : } M = \text{ saturated mass, } D = \text{ dry mass).}$$

The solubility (S) was calculated in percentage as follows:

$$S = [(I - D)/D] \times 100 \text{ (Where : } I = \text{ initial mass, } D = \text{ dry mass).}$$

2.4. Apatite forming ability

This test was performed for the evaluation of formation of calcium phosphate deposits on the materials' surface according to Witherspoon et al. (2006).

After immersion of discs in 20 mL of HBSS (Hank's Balanced Salt Solution, Lonza Walkersville, Inc., Walkersville, MD, USA) at 37 °C for 1, 7, and 28 days according to Gandolfi et al. (2011), the surface of each disc was examined in dried status by an Environmental Scanning Electron Microscope (ESEM, Zeiss EVO 50; Carl Zeiss, Oberkochen, Germany) that was connected to a secondary electron detector for energy dispersive X-ray analysis (EDX; Oxford Instruments, Abingdon, UK) with computer controlled software (Inca Energy Version 18, Oxford Instruments, Abingdon, UK). EDX determined the micro-chemical spectra, element mapping, and semi-quantitative compositional tables. Ca/P ratio was calculated from the obtained data.

2.5. Compressive strength

The discs were stored in 100% humidity at 37 °C for 1, 7 and 28 days and tested according to Natale et al. (2014). Data were collected using computer software (Nexygen-MT; Lloyd Instruments, Beijing, China). Compressive load was applied along the long axis of the specimen at cross-head speed of

0.5 mm/min until failure. The maximum failure load was recorded in Newton however; the compressive strength was calculated in MPa.

2.6. Lap shear bond strength

Lap shear test evaluated the bond strength of the tested materials with Resin-modified glass ionomer according to Estrela et al. (2000). After the setting time (15 min, 55 h, 96 h and 10 min for setting of BD, BS, MTA and Resin-modified glass ionomer, respectively), all samples were stored in molds at 100% humidity and 37 °C for 24 h. Double split Teflon molds were used for preparation of samples. Shear force was done by tensile mode of loading with the force parallel to Resin-modified glass ionomer/tested material interface until failure occurred at a cross-head speed of 0.5 mm/min. Data were recorded using computer software on the testing machine (Nexygen-MT; Lloyd Instruments, Beijing, China).

2.7. Statistical analysis

Descriptive statistics were performed for results of each material. Two way ANOVA test of significance was done for comparing variables affecting mean values. One way ANOVA and pairwise Tukey's post-hoc tests were performed. Statistical analysis was performed using Aasistat 7.6 statistics software for Windows (Campina Grande, Paraiba state, Brazil). *P* values ≤ 0.05 were considered significant.

3. Results

3.1. Biointeractivity

Table 1 shows the measured pH values of the tested materials at different times. The tested material, time and the interaction between the two variables had a significant effect on pH values ($P < .05$).

Regardless of time intervals, there was no significant difference between BD and BS ($P > .05$); both showed the significant highest mean pH values. MTA showed the significant lowest mean pH value.

Table 1 Mean and standard deviation values of pH of the tested materials at predetermined time intervals.

Materials	3 h	1 day	3 days	7 days	14 days	28 days	<i>P</i> value
BD	11.68 ^{aA} ± 0.4	11.6 ^{aA} ± 0.5	11.06 ^{aA} ± 0.7	9.238 ^{bB} ± 0.3	9.42 ^{aB} ± 0.2	9.54 ^{aB} ± 0.7	<0.0001*
MTA	10.94 ^{bA} ± 0.4	9.96 ^{aA} ± 2.0	9.24 ^{bA} ± 0.2	8.64 ^{cA} ± 0.4	7.82 ^{bB} ± 0.2	7.1 ^{bC} ± 0.2	<0.0001*
BS	11.34 ^{aA} ± 0.1	11.68 ^{aA} ± 0.2	10.9 ^{aA} ± 0.5	10.4 ^{aA} ± 0.3	7.9 ^{bB} ± 0.4	7.66 ^{bB} ± 0.	<0.0001*
<i>P</i> value	0.0149*	0.0718 NS	0.0002*	<0.0001*	<0.0001*	<0.0001*	

Different capital letters in same row indicating a significant difference between the time intervals ($P < .05$). Different small letters in same column indicating a significant difference between the tested materials ($P < .05$), *: significant ($P < .05$).

Regardless of the capping material; there was no significant change in pH values from 3 h to day 1 and from day 1 to day 3 ($P > .05$). From day 3 to day 7 and from day 7 to day 14, there was a significant decrease in pH ($P < .05$). From day 14 to day 28, there was no significant change in pH values ($P > .05$).

3.2. Calcium ion release

Table 2 shows the values of Ca^{+2} ions that were released by the tested materials at different times. The tested material, time and interaction between the two variables had a significant effect ($P < .05$).

Regardless of time intervals, generally, BS had a significant higher mean calcium content than the BD and MTA ($P < .05$). Regardless of the capping material, 3 h interval had the significant highest mean calcium content ($P < .05$). There was a significant decrease in calcium content at day 1 ($P < .05$). From day 1 to day 3 and day 14 to day 28, there was no significant change ($P > .05$). From day 3 to day 7, there was a significant increase in calcium content that was followed by a significant decrease from day 7 to day 14 ($P < .05$).

3.3. Physical properties

BS recorded the significant highest mean percent of porosity, water sorption and solubility measurements. BS was followed by the MTA and the lowest significant mean values were recorded for BD. No significant difference was reported between MTA and BD ($P > .05$, Table 3).

3.4. Apatite forming ability

Ca/P deposits of various amounts and densities were detected on the surface of all materials after 7 days. Ca^{+2} ions, Si, Cl, C and P reflexes with absence of Al were recorded at all times.

On day 1, all materials showed low Si and appearance of Na, Mg, and P. The surface was covered by globular precipitates and P reflexes increased in intensity with time.

BD displayed Ca, Si, Na and Cl reflexes (Fig. 2Aa). A layer of globular Ca/P precipitates was noticed on the surface at days 1 and 7.

At day 7, BD showed uniform surface containing interspersed granules (width was approximately 5 μm) and displayed Ca, Si, Na and Cl reflexes, decreased Si and visible Na, Mg, and P (Fig. 2Ab). P reflexes increased with the soaking time and consequently the Ca/P atomic ratio decreased.

At day 28, BD showed the same surface at day 7 with Ca, Zr, and Cl reflexes, no Si and visible Na, Mg, and P (Fig. 2Ac). P reflexes increased with the soaking time and consequently the Ca/P atomic ratio decreased to 2.5.

MTA showed an irregular surface with evident granules and displayed Ca, Si, and Bi reflexes with no S at all evaluation periods.

At day 1, the surface of MTA was covered by aggregating spherulites forming irregular Ca/P deposits that increased with time and showed high Ca, Si and Mg as well as traces of Bi and P.

At day 7, high Ca and Mg peaks, noticeable amounts of P, Si and Bi were reported.

At day 28, MTA showed an irregular Ca/P coating at the surface, increased P peak, weakened Mg reflex, undetectable Bi and Al elements. The Ca/P atomic ratio was 50–60 at 24 h and decreased from 10 to 15 at 7 days to 1.8 after 28 days.

After the elemental X-ray Microanalysis and the element mapping were performed on day 1, MTA revealed Ca, Si, Zr, Na, and P (Fig. 2Ba). At day 7, Ca, Si, Mg, Na, and P were reported and after soaking, the reflexes of Ca and Si decreased while P and Na increased (Fig. 2Bb). At day 28, the surface showed irregular distributed globular Ca/P precipitates and visible Bi (Fig. 2Bc).

Table 2 Mean and standard deviation values of Ca^{+2} ions (measured as p.p.m.) of the tested materials at predetermined time intervals.

Materials	3 h	1 day	3 days	7 days	14 days	28 days	<i>P</i> value
BD	95.68 ± 7.5 ^{bA}	21.1 ^{aD} ± 6.5	19.06 ^{aD} ± 3.3	43.2 ^{bB} ± 2.5	28.9 ^{aC} ± 7.3	32.8 ^{aC} ± 6.6	<0.0001*
MTA	26.9 ^{cA} ± 3.4	24.32 ^{aA} ± 7.7	15.36 ^{bB} ± 1.8	18.7 ^{cB} ± 3.0	27.2 ^{aB} ± 2.6	23.12 ^{bB} ± 1.7	<0.0001*
BS	169.54 ^{aA} ± 24.4	31.1 ^{aC} ± 6.2	21.6 ^{aC} ± 4.0	130.7 ^{aB} ± 22.1	25.46 ^{aC} ± 6.0	24.1 ^{bC} ± 6.7	<0.0003*
<i>P</i> value	<0.0001*	0.0991 NS	0.029*	<0.0001*	0.641 NS	0.0308*	

Different capital letters in same row indicating a significant difference between the time intervals ($P < .05$). Different small letters in same column indicating a significant difference between the tested materials ($P < .05$), *: significant ($P < .05$).

Table 3 Mean and standard deviation values of percent of porosity, water sorption and solubility of the tested materials.

Tested materials	Porosity		Water sorption		Solubility	
	Mean \pm SD	<i>P</i> value	Mean \pm SD	<i>P</i> value	Mean \pm SD	<i>P</i> value
BD	6.19 \pm 0.76 ^B	0.0208*	6.85 \pm 0.92 ^B	0.0220*	2.94 \pm 0.09 ^B	0.0006*
MTA	17.39 \pm 4.16 ^B		9.61 \pm 4.03 ^B		3.27 \pm 0.85 ^B	
BS	26.04 \pm 4.26 ^A		15.12 \pm 4.22 ^A		4.84 \pm 0.73 ^A	

Different letters indicating a significant difference ($P < .05$), *: significant at $P < .05$.

BS showed a granular surface with Ca, Si, Bi, Cl, S, and Al elements. The surface was covered partially (at day 1) then completely (at days 7 and 28) by spherulitic Ca/P deposits. The P peak and Mg reflex were absent in fresh BS, but appeared and progressively increased with time. Si, Al, S, and Bi reflexes declined then disappeared. The Bi peak became undetectable after 7 days. The Ca/P atomic ratio was 10–20 at days 1 and 7 and 3.06 at day 28.

The surface of BS displayed Ca, Si, P and Zr elements (Fig. 2Ca). At day 7, BS had Ca, Si, Bi, Cl, S, and Al elements (Fig. 2Cb). At day 28, BS showed Ca, Na, Cl, Mg, P elements (Fig. 2Cc). BS recorded the highest mean of Ca/P ratio value (2.48) that was followed by BD (2.19) then MTA (1.77) with no significant difference ($P > .05$).

3.5. Compressive strength

Table 4 shows the values of compressive strength of tested materials at predetermined time intervals.

After one, 7 and 28 days, BD recorded the significant highest mean compressive strength value ($P < .05$) that was followed by the MTA then BS. There was no significant difference between the MTA and BS at day 7 as well as BD and MTA at day 28 ($P > .05$). There was no significant difference between different storage times ($P > .05$).

3.6. Lap shear bond strength

The values of lap shear bond strength were 6.12 ± 0.41 , 4.54 ± 0.99 and 4.45 ± 0.76 for the BD, MTA and BS, respectively. BD showed the significant highest mean lap shear bond strength value that was followed by the MTA then BS ($P < .05$). There was no significant difference between the MTA and BS ($P > .05$).

4. Discussion

Biocompatibility, biointeractivity, bioactivity, mechanical and physical properties of pulp capping materials are essential for the reparative dentin formation (Abo El-Mal et al., 2019).

The capability of releasing calcium and hydroxide ions has a crucial role in the successful pulp capping through enhancement of mineralization (Gandolfi et al., 2013a). In this study calcium release and pH values were measured in distilled water to standardize the test (Gandolfi et al., 2013a). The three tested materials released Ca and OH ions due to the hydration of calcium silicate particles that results in the formation of calcium silicate hydrate gel, $\text{Ca}(\text{OH})_2$ and release of Ca and OH ions.

The BD exhibited the highest release of calcium ions and alkalinizing ability at day 28 due to its large particles, the

calcium silicate and chloride contents and the rapid hydration. These findings were supported by the results of Gandolfi et al. (2013a,b).

Porosity determines the amount of leakage, outcome of therapy, adsorption, permeability, strength and density of the capping material (Kaur et al., 2017). In our study, the lowest values of open porosity, water sorption and solubility were recorded in BD and MTA. These results are consistent with a previous study (Camilleri et al., 2013). Low solubility of BD could be correlated to the restricted amount of the mixing fluid (a nonwater-based liquid). This mixing fluid contains water-reducer superplasticizer such as polycarboxylate (based on polycarboxylic ether) that is commonly used to reduce the amount of water required to disperse the particles, to enhance the fluidity and to make the cement self-consolidating (Gandolfi et al., 2013b). In contrast, Fridland and Rosado (2003) reported significant higher solubility and porosity of the ProRoot MTA than those of BD. This was in disagreement with the results of the current study that showed no significant difference in solubility between the BD and MTA. They explained their results by the increase in water to powder ratio of the MTA which increases the release of calcium from the MTA and accelerates its solubility.

The highest recorded values of calcium release, solubility and apparent porosity for BS may be due to the presence of CaCl_2 in the composition of BS that decreases the setting time, enhances the calcium release and initiates the signals on pulp cells (Rashid et al., 2003). Both open and apparent porosities correlate with the high ion release.

The tested materials had high bioactivity and exhibited a rapid modification of their surface morphology and chemical composition after immersion in phosphate solution. Apatite formation was started at the early time of immersion and a uniform apatite layer was seen after day 7.

The MTA exhibited evenly distributed deposits on the surface after the day 1 due to the rapid release of calcium ions after immersion and production of an alkaline pH on the surface with nucleation and crystallization of HA. The apatite layer increased with the soaking time that indicates the maturation of a carbonated-apatite phase. Similar findings were reported by Ding et al. (2009). ESEM-EDX analyses of the MTA showed the formation of regular round agglomerates, evident carbonation and no S due to absence of the calcium sulphate component.

Regarding the BD, the surface was strongly different from that of the MTA and BS. Calcium and Si were displayed due to the tricalcium silicate component and sulphur (S) as well as Al elements were not detected. Calcium ions release is directly proportional to Ca/P deposits. Cl originates from calcium chloride and decreases the setting time (Abdullah et al., 2002). Moreover, C originates from calcium carbonate and

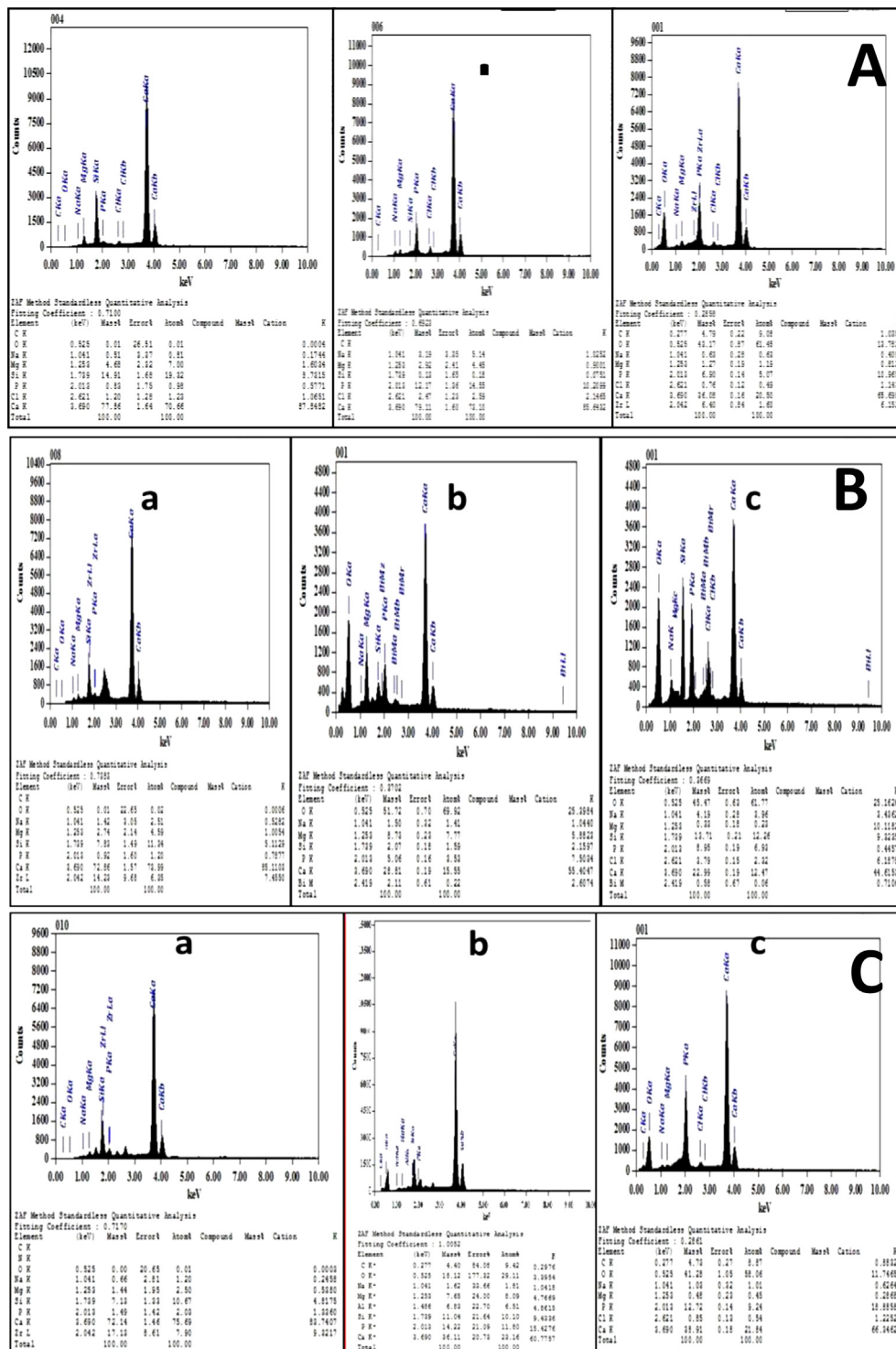


Fig. 2 Energy dispersive x-ray microanalysis: Mineral profile of Biodentine cement (A), MTA cement (B) and Biosealer cement (C) after 1 (a), 7 (b) and 28 days (c).

enhances the dispersion and water reduction. Additionally, calcium carbonate activates mineralization. The large amount of calcium release favors the formation of Ca/P deposits. However, the Ca/P atomic ratio (approx. 2.5) obtained at day 28, together with the detection of Si component may be responsible for the thin or uneven Ca/P coating and the presence of calcium carbonate. These findings are supported by the results of [Gandolfi et al. \(2013b\)](#).

The BS exhibited a thick coating with clear globular Ca/P deposits after 28 days due to the presence of Ca-rich calcium phosphates and carbonated phases.

Apatite formation has a crucial role during the capping procedure because it enhances the sealing ability, fills the marginal porosities and provides a suitable environment for the reparative dentin formation ([Weller et al., 2008](#)). Calcium-silicate cements produce a good apatite layer due to high ion

Table 4 Mean and standard deviation values of compressive strength (MPa) of tested materials at predetermined time intervals.

Tested materials	1 day	7 days	28 days	P value
BD	54.27 ^{Ab} ± 6.1	60.22 ^{Ab} ± 1.4	72.97 ^{Aa} ± 7.6	<0.0001*
MTA	20.15 ^{Bb} ± 0.6	24.23 ^{Bb} ± 2.2	60.57 ^{Aa} ± 5.5	<0.0001*
BS	11.95 ^{Ca} ± 1	22.22 ^{Ba} ± 1.5	29.59 ^{Ba} ± 2.6	<0.1637
P value	<0.0001*	<0.0001*	<0.0001*	

Different capital letters in same row indicating a significant difference between the time intervals ($P < .05$). Different small letters in same column indicating a significant difference between the tested materials ($P < .05$), *: significant ($P < .05$).

release and consequently improve the clinical outcomes (Parirokh et al., 2010).

Good compressive strength of the capping material is essential for withstanding the masticatory forces. The BD showed better compressive strength than that of the MTA and BS due to diminish of porosity by time as mentioned before (Kaur et al., 2017). This result confirms the results of solubility and porosity tests.

Unlike conventional shear tests, lap shear test was selected in this study because the load is applied to the whole bonded interface area and relevant adhesive performance data are obtained (Palma et al., 2018). The conventional shear tests apply load to a point using chisel edge. Several interacting variables such as; type of test, specimens geometry, size of the bonded surface area, the loading condition, operation variability and more, make the direct comparison of the obtained shear bond strength values between different studies a very difficult task (Valandro et al., 2007).

The tested specimens remained untouched for 15 min, 96 h and 55 h for setting of the BD, MTA and BS, respectively and then the resin modified glass-ionomer was applied. The BD exhibited the highest shear bond strength that was followed by the MTA then BS. This difference could be attributed to the difference in bonding mechanism of the tested cements.

5. Conclusion

MTA, BD and BS are biointeractive bioactive materials that possess the ability to release ions and form calcium phosphate deposits. Unlike BD, BS is incapable of alternating MTA in pulp capping due to its high porosities, water sorption and solubility, as well as poor compressive and lap shear bond strength.

Ethical statement

All international and institutional ethical measures were followed during this study. All author approved the writing and submission of this article and none of them has any conflict of interests. This article did not published elsewhere before in any form.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

References

- Abdullah, D., Ford, T.R., Papaioannou, S., Nicholson, J., McDonald, F., 2002. An evaluation of accelerated Portland cement as a restorative material. *Biomater. J.* 23, 4001–4009.
- Abo El-Mal, E.O., Abu-Seida, A.M., El Ashry, S.H., 2019. A comparative study of the physicochemical properties of hesperidin, MTA-Angelus and calcium hydroxide as pulp capping materials. *Saudi Dent. J.* 31, 219–227.
- Accorinte, M., Loguercio, A., Reis, A., 2008. Response of human dental pulp capping with MTA and calcium hydroxide powder. *Oper. Dent.* 33, 488–495.
- Camilleri, J., Grech, L., Galea, K., Keir, D., Fenech, M., Formosa, L., et al., 2013. Porosity and root dentine to material interface assessment of calcium silicate-based root-end filling materials. *J. Clin. Oral Investig.* 14, 256–264.
- Camps, J., Dejou, J., Remusat, M., Abut, I., 2000. Factors influencing pulpal response to cavity restorations. *Dent. Mater.* 16, 432–440.
- Cox, C., Suzuki, S., 1994. Re-evaluating pulp protection calcium hydroxide linear vs. cohesive hybridization. *J. Am. Dent. Assoc.* 125, 823–831.
- Ding, S.J., Shie, M.Y., Wang, C.Y., 2009. Novel fast-setting calcium silicate bone cements with high bioactivity and enhanced osteogenesis in vitro. *J. Mat. Chem.* 19, 1183–1190.
- El-Ashry, S., Abu-Seida, A.M., Al-Boghdady, H., El-Batouty, K., Abdel-Fattah, M., 2013. The effect of different formulations of calcium hydroxide on healing of intentionally induced periapical lesions in dogs. *Pak. Vet. J.* 33, 48–52.
- El Ashry, S.H., Abu-Seida, A.M., Emara, R.A., 2016. The influence of addition of osteogenic supplements to mineral trioxide aggregate on the gene expression level of odontoblastic markers following pulp capping in dogs. *Vet. Arhiv.* 86, 685–697.
- Estrela, C., Bammann, L.L., Estrela, C.R., Silva, R.S., Pecora, J.D., 2000. Antimicrobial and chemical study of MTA, Portland cement, calcium hydroxide paste, Seal apex and Dycal. *Braz. Dent. J.* 11, 3–9.
- Fridland, M., Rosado, R., 2003. Mineral trioxide aggregate (MTA) solubility and porosity with different water-to-powder ratios. *J. Endod.* 29, 814–817.
- Gandolfi, M.G., Taddei, P., Siboni, F., Modena, E., Ciapetti, G., Prati, C., 2011. Development of the foremost light-curable calcium-silicate MTA cement as root-end in oral surgery. Chemical-physical properties, bioactivity and biological behavior. *Dent. Mater.* 27, 123–134.
- Gandolfi, M.G., Taddei, P., Modena, E., Siboni, F., Prati, C., 2013a. Biointeractivity-related versus chemi/physiosorption related apatite precursor-forming ability of current root end filling materials. *J. Biomed. Mater. Res. B Appl. Biomater.* 101, 1107–1116.
- Gandolfi, M.G., Siboni, F., Polimeni, A., Bossù, M., Riccitiello, F., Rengo, S., et al., 2013b. In vitro screening of the apatite-forming ability, biointeractivity and physical properties of a tricalcium silicate material for endodontics and restorative dentistry. *Dent. J.* 1, 41–50.

- Kaur, M., Singh, H., Dhillon, J.S., Batra, M., Saini, M., 2017. MTA versus biodentine: review of literature with a comparative analysis. *J. Clin. Diag. Res.* 11, 1–5.
- Natale, L.C., Rodrigues, M.C., Xavier, T.A., Simoes, A., de Souza, D. N., Braga, R.R., 2014. Ion release and mechanical properties of calcium silicate and calcium hydroxide materials used for pulp capping. *J. Endod.* 48, 85–89.
- Negm, A., Hassanien, E., Abu-Seida, A.M., Nagy, M., 2016. Physical evaluation of a new pulp capping material developed from Portland cement. *J. Clin. Exper. Dent.* 8, e278–e283.
- Negm, A.M., Hassanien, E.E., Abu-Seida, A.M., Nagy, M.M., 2017. Biological evaluation of a new pulp capping material developed from Portland cement. *Exp. Toxicol. Pathol.* 69, 115–122.
- Palma, P.J., Marques, J.A., Falacho, R.I., Vinagre, A., Santos, J.M., Ramos, J.C., 2018. Does delayed restoration improve shear bond strength of different restorative protocols to calcium silicate-cased cements?. *Materials (Basel)*. 11 (11), 2216–2225.
- Parirokh, M., Torabinejad, M., 2010. Mineral trioxide aggregate: a comprehensive literature review - Part III: Clinical applications, drawbacks, and mechanism of action. *J. Endod.* 36, 400–413.
- Ramos, J.C., Palma, P.J., Nascimento, R., Caramelo, F., Messias, A., et al, 2016. 1-year in vitro evaluation of tooth discoloration induced by 2 calcium silicate-based cements. *J. Endod.* 42 (9), 1403–1407.
- Rashid, F., Shiba, H., Mizuno, N., Mouri, Y., Fujita, T., Shinohara, H., et al, 2003. The effect of extracellular calcium ion on gene expression of bone-related proteins in human pulp cells. *J. Endod.* 29, 104–107.
- Richard, G., Marie, O., 2011. Dental composition. Patent WO 2011/124841, US2013/0025498.
- Saleh, R., Nagi, S.M., Khallaf, M.E., Abd El-Alim, S.H., Zaazou, M. H., Abu-Seida, A.M., et al, 2016. In-Vivo assessment of dentin bridge formation after using MTA and experimental propolis paste as direct pulp capping material. *Res. J. Pharmaceut. Biol. Chem. Sci.* 7, 1244–1250.
- Valandro, L.F., Ozcan, M., Amaral, R., Leite, F.P., Bottino, M.A., 2007. Microtensile bond strength of a resin cement to silica-coated and silanized In-Ceram Zirconia before and after aging. *Int. J. prothodont.* 20, 70–72.
- Vallés, M., Mercadé, M., Duran-Sindreu, F., Bourdelande, J.L., Roig, M., 2013. Color stability of white mineral trioxide aggregate. *Clin. Oral Investig.* 17 (4), 1155–1159.
- Weller, R.N., Tay, K.Y., Garrett, L.V., 2008. Microscopic appearance and apical seal of root canals filled with gutta-percha and ProRoot Endo Sealer after immersion in a phosphate-containing fluid. *Int. Endod. J.* 41, 977–986.
- Witherspoon, D.E., Small, J.C., Harris, G.Z., 2006. Mineral trioxide aggregate pulpotomies: a case series outcomes assessment. *J. Am. Dent. Assoc.* 137, 610–618.