Brazilian Dental Science

unesp
UNIVERSIDADE ESTADUAL PAULISTA
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Instituto de Ciência e Tecnologia
Campus de São José dos Campos

ORIGINAL ARTICLE

DOI: https://doi.org/10.4322/bds.2022.e3368

Synthesis, physical and mechanical properties of an experimental nano calcium aluminate/tri calcium silicate root repair material compared to mineral trioxide aggregate and Biodentine. (Part one)

Síntese, propriedades físicas e mecânicas de um material experimental de reparo raiz de nano cálcio aluminato/tri-silicato de cálcio comparado com agregado trióxido mineral e Biodentina. (Parte um)

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ABSTRACT

Objective: Endodontic perforation is a challenging mishap that should be repaired with a biocompatible material, Mineral trioxide aggregate (MTA) and Biodentine are the most commonly used repair materials. However, these materials are expensive, (MTA) has prolonged setting time and difficult manipulation. The purpose of this study is to prepare the experimental nano calcium-aluminate/tri-calcium-silicate (CA/C_3S) material and comparing its physical properties with biodentine and MTA, to evaluate the experimental material eligibility to compete the commercial repair materials. And to perform part two (animal study) that will evaluate the cytotoxicity, the biocompatibility and the efficacy of (CA/C_3S) in furcal perforation repair compared to diode laser.
Material and Methods: A mixture of calcium carbonate and aluminum oxide was used to formulate calcium aluminate phase (CA), tri-calcium-Silicate phase (C_3S) was formulated by firing of calcium carbonate and quartz. The produced powders were investigated by X-ray diffraction, then (CA) and (C_3S) mixed with water.(CA/ C_3S) compared with MTA and biodentine for setting-time, micro-hardness, dimensional-stability and solubility.
Results: Mean setting time of (CA/C_3S) was (32.70±0.75min) which is significantly higher than MTA and Biodentine. The Mean microhardness of (CA/C_3S) was (56.50±7.41VHN) which has no statical difference with MTA and Biodentine. Solubility results showed weight increase for (CA/C_3S) as following (6.29±3.05)and loss of weight for MTA and Biodentine. The percentage of change in dimensions for(CA/C_3S) increased as following (0.64±0.78) while decreased for MTA and Biodentine.
Conclusion: The experimental (CA/C_3S) material showed good microhardness, dimensional stability and acceptable setting time that could be improved in further work.

KEYWORDS

Biodentine; MTA; Nano calcium aluminate; Tri-calcium-silicate, Root repair.

RESUMO

Objetivo: A perfuração endodôntica é um percalço desafiador que deve ser reparado com um material biocompatível, Agregado de trióxido mineral (MTA) e Biodentina são os materiais de reparo mais comumente usados. No entanto, esses materiais são caros, (MTA) tem tempo de presa prolongado e difícil manipulação. O objetivo deste estudo é preparar o material experimental de nano aluminato de cálcio/silicato tricálcico (CA/C3S) e comparar suas propriedades físicas com biodentina e MTA, para avaliar a elegibilidade do material experimental para competir com os materiais de reparo comerciais. E realizar a segunda parte (estudo animal) que avaliará a citotoxicidade, a biocompatibilidade e a eficácia do (CA/C3S) no reparo de perfuração de furca em comparação ao laser de diodo.

Material e Métodos: Uma mistura de carbonato de cálcio e óxido de alumínio foi usada para formular a fase de aluminato de cálcio (CA), a fase tri-cálcio-silicato (C3S) foi formulada por queima de carbonato de cálcio e quartzo. Os pós produzidos foram investigados por difração de raios X, em seguida (CA) e (C3S) misturados com água. (CA/ C3S) comparados com MTA e biodentina para tempo de presa, microdureza, estabilidade dimensional e solubilidade.

Resultados: O tempo médio de presa de (CA/C3S) foi (32,70±0,75min) que é significativamente maior que MTA e Biodentine. A microdureza média de (CA/C3S) foi (56,50±7,41VHN) que não tem diferença estática com MTA e Biodentine. Os resultados de solubilidade mostraram aumento de peso para (CA/C3S) conforme a seguir (6,29±3,05) e perda de peso para MTA e Biodentine. A porcentagem de mudança nas dimensões para (CA/C3S) aumentou como segue (0,64±0,78), enquanto diminuiu para MTA e Biodentine.

Conclusão: O material experimental (CA/C3S) apresentou boa microdureza, estabilidade dimensional e aceitável tempo de presa, que pode ser melhorado em trabalhos futuros.

PALAVRAS-CHAVE

Biodentina; MTA; Nano aluminato de cálcio; Tri-cálcio-silicato, Materiais de reparo.

INTRODUCTION

Endodontic perforation is the communication of the root canal system with the periodontium caused by a procedural error during endodontic treatment. Although extensive caries lesions or resorptive processes can also induce perforations, the majority of root perforations occur iatrogenically [1]. Root perforations that result in endodontic treatment failure represent approximately 10% of all unsuccessful cases [2].

The requirement of ideal perforation repair material is to provide adequate seal, biocompatible with surrounding tissues, induce osteogenesis and cementogenesis, radiopaque and relatively inexpensive [3]. Some historical attempts by many researchers using Calcium hydroxide, Gutta percha, zinc oxide Eugenol, glass Ionomer cements, amalgam and composites as perforation repair material but none of them meet the criteria for an optimal material for perforation repair [4].

Mineral Trioxide Aggregate (MTA) and Biodentine are believed to be excellent perforation repair materials due to their biocompatibility, promotion of bone and tooth mineralization, periodontal ligament regeneration, and high sealing ability [5,6]. MTA is a powder composed of fine particles of tri calcium silicate, tricalcium aluminate, tricalcium oxide, silicate oxide and bismuth oxide [7]. MTA shows some drawbacks for example, difficult manipulation, slow setting time and high cost [8]. Biodentine is composed of powder (tricalcium silicate, oxide filler, dicalcium silicate, zirconium oxide, iron oxide shade, and calcium carbonate). liquid, contains calcium chloride that act as an accelerator [5].

Calcium aluminate shows excellent rheological characteristic, a high mechanical resistance, a good setting time, and a rapid hydration rate [9]. Calcium aluminate cement is classified as a hydraulic binder and it is also used for perforations repair and for bone defects filling [10]. It has high mechanical strength and the ability to form an intimate bond to the opposing tissues. The formation of such bond between the material and the tissue is a result of surface dissolution of the material followed by re-precipitation of a layer of poorly crystalline hydroxyapatite at the interface with the tissue [11]. Calcium aluminate cements also could act as a barrier preventing bacterial invasion through microleakages [12].

Tricalcium silicate-based hydraulic (water-setting) cements are currently the material of choice for regeneration and repair procedures in endodontics because of their bioactivity. These materials are used clinically for perforation repair and root-end filling [13]. Due to its similar composition and bioactivity, tricalcium silicate is an excellent alternative for the cement component in MTA, with the added benefit of reducing setting time when compared to MTA [14].

Because nanotechnology is the study of evaluating and developing materials in nano-dimensions by atom re-location and re-arrangement to create new materials with superior qualities. The presence of extremely fine particles results in enhanced material characteristics. These unique characteristics are the basis of quantum mechanics [15].

Therefore, the purpose of the current study is to prepare the experimental nano calcium aluminate /tri calcium silicate material and comparing its physical properties with Biodentine and MTA as perforation repair material. And in the aim to performing successive part two (animal study) that will evaluate ·the bone healing process and histological changes beneath furcal perforation site after using diode Laser, experimental (CA / C_3S) material and biodentine.

MATERIAL AND METHODS

Preparation of nano calcium aluminate (CA) powder

The calcium aluminate phase was synthesized in the laboratory using a 1:1 dry combination of ultra-pure limestone (99.8% $CaCO_3$) and Alumina (99.6% Al_2O_3), as described by Rivas Mercury et al. [16] and Iftekhar et al. [17]. The dry combination, which was calcined at 1000 °C for two hours before being ignited at 1500 °C for six hours, agreed with all existing studies in that the amounts of $C1_2A_7$ and CA_2 decreased while CA increased with increasing temperature and time, according the following chemical equation.

$$CaCO_3 + Al_2O_3 \xrightarrow{1500^\circ C} CaOAl_2O_3 + CO_2 \tag{1}$$

X-ray diffraction had been performed to verify the identity of the manufactured molecule (Figure 1), with a copper target and radiation; wavelength=0.154nm, X-ray generated at 40 kV with a current of 2-5mA. The produced material was refined in an agate mill for 15 hours to achieve the desired grain size of 50:100 nm [18].

Preparation of nano tri calcium silicate (C_3S) powder

C_3S phase (50:100 nanometer particles) was manufactured by firing formed cubes of 3:1 CaO: SiO_2 molar ratio, combining ultra-pure limestone and quartz (99.6% SiO_2), in the presence of % 0.5 boric acid at 1000 °C for two hours, according to Hewlett et al. [19] the product material was milled, completely remodeled using carbon tetrachloride, and then torched for two hours at 1450 °C, according the following chemical equation.

$$3CaCO_3 + SiO_2 \xrightarrow{1450^\circ C} 3CaOSiO_2 + 3CO_2 \tag{2}$$

The firing operation was repeated until the reaction was complete. The final product was tested for the presence of free lime. The size of the nanoparticles of the final phase powder was assessed by X-ray diffraction (XRD) (Figure 2) and Transmission Electron Microscopy (TEM) ((JEM-1230) at 100 kV).

Physical properties evaluation of the experimental nano calcium aluminate tri-calcium silicate CA/C_3S material, biodentine and MTA

The experimental material was mixed in a ratio of 1:1 powder of (CA) and (C_3S) using distilled water at room temperature until a homogenous plug was obtained with the liquid powder ration of (0.762gm.:0.1ml). Biodentine™

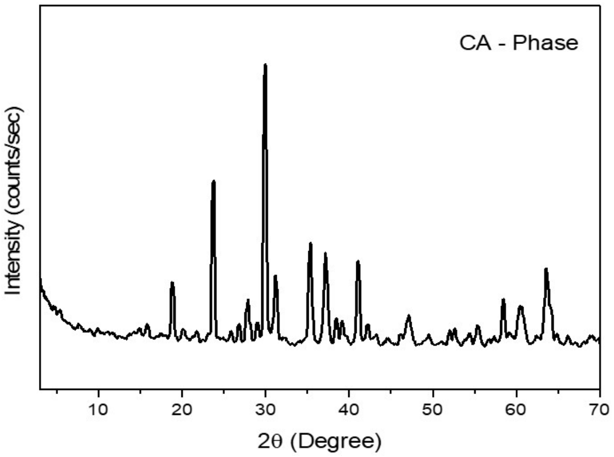

Figure 1 - Showing XRD of CA powder.

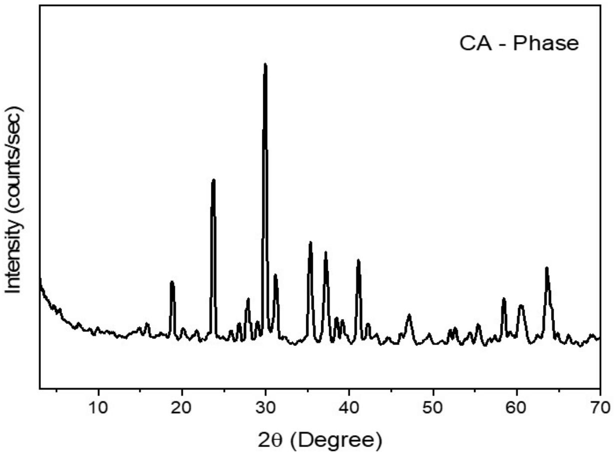

Figure 2 - Showing XRD of C_3S powder.

(Septodont, Saint Maur des Fosse's, France) capsule prepared, according to manufacturer manual. MTA (Angelus Industria de Produtos Odontologicos, Londrina, Brazil) powder was hand mixed with sterile water according to the manufacturer instructions till formation of a homogenous plug.

Setting time evaluation

According to ISO 6876:2012 [20], five samples of the three tested materials were formed as discs (n = 5 per material) by placing each material into stainless steel ring-shaped forms with dimensions of 10 mm in diameter and 1 mm in thickness. The indentation technique using a Gilmore needle with a diameter of 2±0.1 mm and a weight of 100±0.5 g was used to determine the initial setting time of the materials. The samples were kept in an oven at 37 °C and 95% humidity for the experiments. The average setting time of each sample was calculated by manipulating each material until the Gilmore needle created no more indentations on the surface of the samples.

Microhardness evaluation

Three samples were prepared for each material, the three tested material were mixed and condensed with an amalgam carrier and hand condensers into 15 acrylic cylinders (n = 3 per material) with internal diameter of 6 mm and height of 3 mm. Condensation was done against a glass slab. The surface facing glass slab was marked. To ensure complete setting, specimens were kept in 100% humidity at 37 °C for 24-hours. The unmarked surface of each sample was wet-polished at room temperature using sandpaper discs (600-grit and 1,200-grit, 3M, St Paul, MN) and minimum hand pressure [21].

Vickers microhardness tester (Nexsus 4000/60, INNOVATEST, Netherlands, Europe) in Vickers Hardness Units (VHN) was used with a load of 300g and dwell time of 10-sec. The microhardness of the unmarked surface was examined for each sample. The resulting indentation was measured under a microscope and VHN was calculated. Three separate indentations were made for each surface at least 1mm apart. The mean value of the three readings was taken as the microhardness of that sample.

Solubility evaluation

Using on ANSI/ADA No. 57 test [22], the three materials were prepared and filled in stainless steel ring forms (n = 3 per material) with a diameter of 20 mm and a thickness of 1.5 mm. To ensure stability during the test, a 5-cm nylon thread was added into the middle of the samples while the material was placed into the mould. The samples were then taken from the stainless-steel ring forms and weighed (initial Mass M_0) using a precision scale (Adam Equipment Inc., Oxford, USA) before being suspended by the nylon threads attached to plastic flasks containing 7.5 mL of deionized water for seven days. The plastic flasks were stored at 37 °C. The samples were then taken out, washed, and put in a silica dehumidifier chamber. The samples were reweighted (final mass M_1), and the mass loss of each sample was calculated and recorded as the sample's solubility as follows:

$$\%Mass\ Loss = \frac{M_0 - M_1}{M_0} \times 100 \tag{3}$$

Dimensional stability evaluation

According to ISO 6876:2012 [20], three teflon cylindrical split mould, 6 mm internal diameter and 12 mm height, were positioned on the slide glass for each tested material, and the materials were loaded into the mould. (n = 3 per material) were preserved in an incubator at 37 °C with a humidity of 95% for a duration that tripled the final setting time of each tested material. With 600 grit sandpaper, both sides of the mould were polished.

Teflon mould was removed and their initial length (Lset) was measured using a digital micrometer precision Caliper, each sample was then stored in 30-ml distilled water at 37±1 °C, for thirty days and then re-measured (L_{30}). The percent dimensional change was calculated as follows:

$$\frac{L_{30} - L_{set}}{L_{set}} \times 100 \tag{4}$$

Statistical analysis

For each test, the mean and standard deviation values were calculated for each group. The data was examined for normalcy using the Kolmogorov-Smirnov and Shapiro-Wilk tests, and the data revealed a parametric (normal) distribution. To compare two groups in related samples, the paired sample t-test was performed.

To compare more than two groups in unrelated samples, a one-way ANOVA followed by a Tukey post-hoc test was performed. The level of significance was fixed at P 0.05. IBM® SPSS® Statistics Version 20 for Windows was used for statistical analysis.

RESULTS

Setting time evaluation

There was a statistically significant difference between all groups, where the highest mean setting time value showed by (CA/C_3S) with (32.70±0.75 min), followed by MTA with (15.39±0.39 min), then Biodentine with (12.50±0.30 min). where ($p<0.001$). As shown in Figure 3.

Microhardness evaluation

The Mean microhardness of Biodentine was (61.73±5.15VHN), while MTA was (56.77±6.77 VHN) and (CA/C_3S) was (56.50±7.41VHN). there was no statistical difference regarding microhardness between the three tested materials, where (p=0.572), as shown in Figure 4.

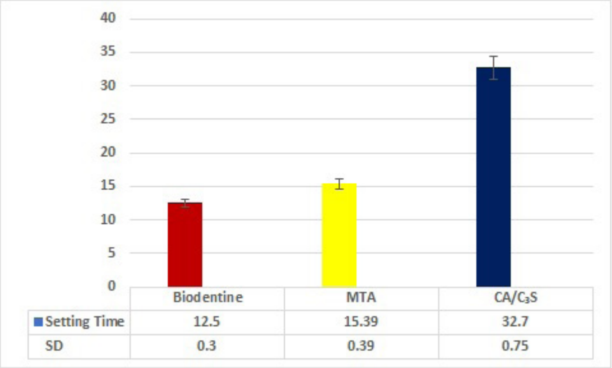

Figure 3 - Bar chart representing setting time for different groups.

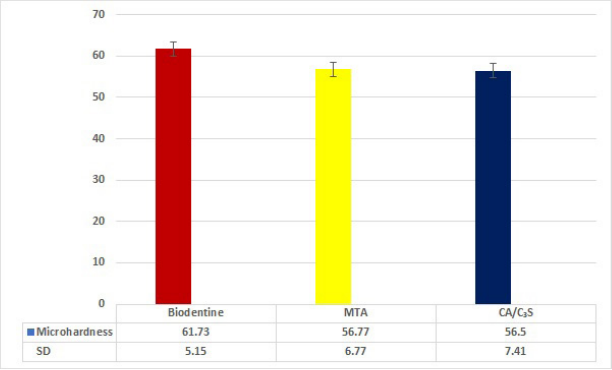

Figure 4 - Bar chart representing microhardness for different groups.

Solubility evaluation

The effect of time on the tested materials, illustrated in Table I

a) Biodentine: The difference between (Baseline) and (After one week) groups was statically significant where ($p=0.002$); **b) MTA:** The difference between (Baseline) and (After one week) groups was statically not significant, where ($p=0.611$); **c) CA/C_3S.** The difference between (Baseline) and (After one week) groups was statically not significant, where ($p=0.061$).

Solubility percentage of change of the tested materials

The difference between the three groups was statistically significant, (Biodentine) showed decrease percentage of change (-6.89±0.52), (MTA) had percentage decrease of change (-0.19±0.28) and (CA/C_3S) showed increase of percentage of change with (6.29±3.05) where ($p<0.001$).

Dimensional stability evaluation

The effect of time on the tested materials, illustrated in Table II

Biodentine, MTA and CA/C3S showed no statical significant difference concerning the effect of time on dimensional stability where P value (p=0.053).

Dimensional stability percentage of change of the tested materials

The difference between the three groups was statistically significant, (Biodentine) showed decease of length by (-0.77±0.52), (MTA) showed decease of length by (-0.98±0.39)

Table I - The solubility means and standard deviation (SD) values for the different groups

	Solubility				
	Baseline		After one week		p-value
	Mean	SD	Mean	SD	
Biodentine	0.36^{aA*}	0.00	0.34^{aB*}	0.00	0.002*
MTA	0.26^{bA*}	0.03	0.26^{bA*}	0.03	0.611ns
CA/C_3S	0.33^{aA*}	0.04	0.35^{aA*}	0.04	0.061ns
p-value	***0.010****		***0.013****		

*Significant (p<0.05) ns; non-significant (p>0.05). Small different letters in the same column indicates significant difference. Capital different letters in the same row indicates significant difference.

Table II - The dimensional stability means and standard deviation (SD) values for the different groups

	Dimensional stability				
Variables	Baseline		After one month		p-value
	Mean	SD	Mean	SD	
Biodentine	12.26^{aA}	0.22	12.17^{aA}	0.27	0.126ns
MTA	11.94^{aA}	0.07	11.82^{aA}	0.08	0.050ns
CA/C$_3$S	12.23^{aA}	0.28	12.31^{aA}	0.19	0.289ns
p-value	***0.198ns***		***0.053ns***		

ns: non-significant (p>0.05). Small different letters in the same column indicates significant difference.
Capital different letters in the same row indicates significant difference.

and (CA/C$_3$S) showed increase of length by (0.64±0.78) where (p=0.029).

DISCUSSION

In this study the experimental calcium aluminate/tri calcium silicate cement was formulated to serve as a perforation repair material in an attempt to overcome some of the disadvantages of MTA, poor handling properties and delayed setting time [8] producing more affordable material than biodentine and MTA would have a good clinical impact.

Selecting the combination of calcium aluminate and tricalcium silicate is based on the high biological, mechanical and physical properties of both materials which could produce eventually a competitor product. Calcium aluminate a high mechanical resistance [9], a good setting time, and a rapid hydration rate classified as a hydraulic binder, act as a barrier preventing bacterial microleakage, has the ability to induce in situ hydroxyapatite generation since its chemical composition and thermal expansion coefficient are similar to those of teeth and human bones [10,23,24]. While silicate-based hydraulic cements are bioactivity materials. Ideal for perforation repair and. Tricalcium silicate has the ability to replace the cement component in the MTA due to similar composition and bioactivity [13,25].

The physical properties of a dental material are important because they determine whether or not the material is acceptable for clinical usage [26]. The physical properties of CA/C$_3$S, Biodentine, and MTA are compared in this research. The results reveal variation in physical properties.

Setting time was the first test adopted in this research since it is affected by the composition, grain size, and percent humidity of the air, along with the surrounding temperature [27]. Setting time is regarded as the most significant physicochemical characteristic since most of other material characteristics are dependent on it. The methodology for the present study was based on ANSI/ADA No. 57 and ISO 6876 standards [20,22].

The present study showed that the CA/C$_3$S mean setting time is (32.70 ± 0.75) which was relatively longer than MTA and Biodentine. The increased setting time of the experimental material (CA/C$_3$S) could be attribute to the different chemical compositions similar to Biodentine and MTA, as it is composed by weight of (50%) Calcium Aluminate phase and (50%) Tri Calcium Silicate phase. A study was done by Acuña-Gutiérrez et al. [28] examining the setting time of Calcium aluminate cement alone and found that setting time without adding an accelerator was 429 mins, however on adding an accelerator setting time was reduced up to 25 mins .With the increase of the alumina content in the cement which has initial setting time of two hours according to Sunnegårdh-Grönberg et al. [29] the hydration process has been retarded. Radwan et al. [30] also found that when C$_3$S paste was prepared with distilled water it has long setting time up to time (180 min) .On the other hand according to Liu et al. [31], the addition of 15% Tri Calcium Aluminates to C$_3$S accelerated the hydration process and reduced the setting time to (from 110 min to 43 min) and improved the compressive strength .The setting time mean value of Biodentine in this study was (12.50±0.30min) which was near the value mentioned in the product sheet [32] (9-12 min) and supported by the study performed by Dawood et al. [33] with a mean value of (13.1min), while the mean value of MTA in this study was (15.39±0.39min) and this was supported by the study of Santos et al. [34] that found that Angelus MTA mean setting time value was (15 min).

The setting time of the experimental material CA/C$_3$S is relatively higher than Angelus MTA and Biodentine in the current study but still less than other commercially FDA approved types of MTA for example ProRoot [8], that exhibit longer initial setting time up to 70 minutes as mentioned in various literatures [5,35]. Which make the experimental material clinically acceptable to be use.

The surface microhardness of materials reflects the strength of the materials in general and shows their degree of setting in different environments [36]. The Vickers' hardness test, which is routinely used to test such physical properties, can provide a solid indication of resistance to localised plastic deformation [37]. This feature is important in dental treatment because surface behavior is associated to usury and scratch, and therefore resistance is related to clinical long-term efficiency [38]. The three materials showed statistically similar mean values of microhardness, the Mean microhardness of (CA/C_3S) was (56.50±7.41VHN). while Biodentine was (61.73±5.15VHN) and MTA was (56.77±6.77VHN) similar to the average values mentioned in similar studies on the same subject made by many authors [33,39,40].

The experimental material's C_3S phase is responsible for the development of material setting and strength via the synthesis of calcium silicate hydrate (C-S-H). The hydration of the C_3S phase starts immediately after mixing with distilled water, and the paste solidifies to form a rigid structure [19,41]. At temperatures above 20 °C, the produced cement hydrates CAH_{10} and C_2AH_8 are metastable. They are eventually transformed into C_3AH_6. The current cement hydrates CAH10 and C_2AH_8 release water during the conversion process, resulting in larger voids in the cement material that are filled with water. As a result, the porosity and permeability of cement material are significantly enhanced. As illustrated in these two equations, when C_3AH_6 is converted, new reaction products are generated and water is liberated [42].

$$CA \quad + \quad 10H_2O \longrightarrow CAH_{10} \qquad (5)$$

$$3CA + 12H_2O \longrightarrow C_3AH_6 + 2AH_3 \qquad (6)$$

Solubility of the materials was evaluated by the ISO standards after 24 hours, however longer study periods may be utilized because the most commonly used time span is seven days [43]. An endodontic repair material's sealing ability and dimensional changes are intimately linked to its solubility, which leaves voids that may facilitate bacterial colonization and passage into periapical tissues [44]. In the current study Biodentine showed statistically highest solubility levels followed by MTA which was seen in similar studies [40,45]. Alazrag et al. [46] found that after seven days, Biodentine was shown to be more soluble than Angelus-MTA similar to the findings in our research, Biodentine has a greater solubility than MTA. Biodentine has a high solubility, which might be attributed to the presence of calcium carbonate and water soluble polycarboxylate in its chemical composition, which help in reducing the water-powder ratio and produce a more workable cement. MTA-Angelus has lesser solubility than Biodentine, which might be explained by the absence of Calcium carbonate and the presence of a setting accelerator in Biodentine's chemical composition, which results in a shorter setting time [47]. While the experimental CA/C_3S showed an increase in weight which could be attributed to the continuous hydration process. Since the material has a short setting time and strong cohesiveness, CA phase often exhibits little leakage. At the start of the hydration process, the gel phase (AH_3), as well as the two major hydrated molecules CAH_{10} and C_2AH_8, are developed. The hydration process begins with the dissolving of the CA phase in the aqueous curing medium, which results in the formation of Ca^{2+}, $Al(OH)^{4-}$, and OH- ions, followed by the precipitation of the hydrated phases [42].

Dimensional change has a significant impact on cement stability because when endodontic cements contract, their sealing capability is diminished, allowing the deployment of infectious processes. On the other hand, cement expansion may result in adhesion failures between the cement and the dentin [48]. Thus, ANSI/ ADA verified that during the dimensional change test, the contraction of the examined material should not exceed 1% and the expansion should not be more than 0.1% [49]. MTA showed little change in dimension after 30 days, which might be attributed to its Portland cement-based composition, which contains an insoluble silica matrix that retains its integrity even when exposed to water [50]. The present study showed that the CA/C_3S exhibit 0.6% expansion after 30 days that may also be explained by the continuous hydration process that occurs in the CA phase. Research on the dimensional changes of calcium aluminate cement conducted by the cement manufacturer revealed that the dimensional change of the cement could be controlled by material alterations. Expansion was measured to be in the 0.1-0.3% or higher, and as high as 1-3% [51]. Generally, the formed hydrated compounds molecules of the two phases used in this work have a larger volume in addition to the liberation of Ca $(OH)_2$ (free lime) as a byproduct and the conversion process of CA phase are mainly responsible for the increasing values of the dimensional stability due the expected expansion of the formulated pastes.

CONCLUSION OF THE CURRENT STUDY

The experimental (CA/ C_3S) material within the limits of this study showed good microhardness, dimensional stability and acceptable setting time that could be improved in further work by adding setting time accelerator. Manufacturing and enhancing such experimental material could offer the clinicians more affordable substitute for MTA and Biodentine.

RECOMMENDATIONS

Performing the successive part two (animal study) to evaluate ·the bone healing process and histological changes beneath furcal perforation site after using diode laser, experimental (CA/ C_3S) material and biodentine, parallel with additional studies to ensure the ease-of-use antibacterial activity and biocompatibility of the experimental (CA/C_3S) material.

Author’s Contributions

MAMN: Designed and directed the project. LMA: Involved in planning and supervised the work. MEK: Developed the theory and performed the computations. DHER: Performed the analytic calculations and performed the numerical simulations. DS: Developed the theoretical framework. MMR: Performed the measurements.

Conflict of interest

No conflict of interest.

Funding

No conflict of interest.

Regulatory Statement

This University, The approval code for this study is (CU-IACUC CU I F 24 21). study was coducted in accordance with insitutional animal care and use ,cairo

REFERENCES

1. Al-Nahlawi T, Ala Rachi M, Abu Hasna A. Endodontic perforation closure by five mineral oxides silicate-based cement with/without collagen sponge matrix. Int J Dent. 2021;2021:4683689. http://dx.doi.org/10.1155/2021/4683689. PMid:34539787.
2. Dastorani M, Shourvarzi B, Nojoumi F, Ajami M. Comparison of bacterial microleakage of endoseal MTA sealer and pro-root MTA in root perforation. J Dent. 2021;22(2):96-101. http://dx.doi.org/10.30476/DENTJODS.2020.86042.1164. PMid:34150945.
3. Kakani AK, Veeramachaneni C, Majeti C, Tummala M, Khiyani L. A review on perforation repair materials. J Clin Diagn Res. 2015;9(9):ZE09-13. http://dx.doi.org/10.7860/JCDR/2015/13854.6501. PMid:26501031.
4. Hegde M, Varghese L, Malhotra S. Tooth root perforation repair: a review. Oral Health Dent Manag. 2017;16(2):1-4.
5. 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(8):ZG01-05. http://dx.doi.org/10.7860/JCDR/2017/25840.10374. PMid:28969295.
6. Alsulaimani RS. Immediate and delayed repair of 2 sizes of furcal perfoations in dogs' teeth using mineral trioxide aggregate cement. J Endod. 2018;44(6):1000-6. http://dx.doi.org/10.1016/j.joen.2018.02.026. PMid:29680721.
7. Cervino G, Laino L, D'Amico C, Russo D, Nucci L, Amoroso G, et al. Mineral trioxide aggregate applications in endodontics: a review. Eur J Dent. 2020;14(4):683-91. http://dx.doi.org/10.1055/s-0040-1713073. PMid:32726858.
8. Schmitt D, Lee J, Bogen G. Multifaceted use of ProRoot MTA root canal repair material. Pediatr Dent. 2001;23(4):326-30. PMid:11572491.
9. Jesus LS, Volpato CAM, Bortoluzzi EA, Teixeira CS, Rossetto HL, Pires-de-Souza FCP, et al. Tooth discoloration induced by the different phases of a calcium aluminate cement: one-year assessment. J Esthet Restor Dent. 2021;33(7):999-1009. http://dx.doi.org/10.1111/jerd.12739. PMid:33929073.
10. Barbosa WT, García-Carrodeguas R, Fook MV, Rodríguez MA. New cement based on calcium and strontium aluminates for endodontics. Ceram Int. 2019;45(16):19784-92. http://dx.doi.org/10.1016/j.ceramint.2019.06.233.
11. Sakai E, Sugiyama T, Saito T, Daimon M. Mechanical properties and micro-structures of calcium aluminate based ultra high strength cement. Cement Concr Res. 2010;40(6):966-70. http://dx.doi.org/10.1016/j.cemconres.2010.01.001.
12. Jacobovitz M, Vianna ME, Pandolfelli VC, Oliveira IR, Rossetto HL, Gomes BP. Root canal filling with cements based on mineral aggregates: an in vitro analysis of bacterial microleakage. Oral Surg Oral Med Oral Pathol Oral Radiol Endod. 2009;108(1):140-4. http://dx.doi.org/10.1016/j.tripleo.2009.03.013. PMid:19540451.
13. Parirokh M, Torabinejad M. Mineral trioxide aggregate: a comprehensive literature review. Part I: chemical, physical, and antibacterial properties. J Endod. 2010;36(1):16-27. http://dx.doi.org/10.1016/j.joen.2009.09.006. PMid:20003930.
14. Grech L, Mallia B, Camilleri J. Investigation of the physical properties of tricalcium silicate cement-based root-end

filling materials. Dent Mater. 2013;29(2):e20-8. http://dx.doi.org/10.1016/j.dental.2012.11.007. PMid:23199808.

15. Javidi M, Zarei M, Naghavi N, Mortazavi M, Nejat AH. Zinc oxide nano-particles as sealer in endodontics and its sealing ability. Contemp Clin Dent. 2014;5(1):20-4. http://dx.doi.org/10.4103/0976-237X.128656. PMid:24808690.

16. Rivas Mercury JM, De Aza AH, Pena P. Synthesis of CaAl2O4 from powders: particle size effect. J Eur Ceram Soc. 2005;25(14):3269-79. http://dx.doi.org/10.1016/j.jeurceramsoc.2004.06.021.

17. Iftekhar S, Grins J, Svensson G, Lööf J, Jarmar T, Botton GA, et al. Phase formation of CaAl2O4 from CaCO3-Al2O3 powder mixtures. J Eur Ceram Soc. 2008;28(4):747-56. http://dx.doi.org/10.1016/j.jeurceramsoc.2007.08.012.

18. Radwan M, El-Hamid H, Abo-Almaged H, Salem H, Omar N. Evaluation of calcium aluminate/calcium phosphate based bio-cements as root-end filling material. Orient J Chem. 2017;33(6):2692-706. http://dx.doi.org/10.13005/ojc/330602.

19. Hewlett P, Liska M. Lea's chemistry of cement and concrete. Oxford: Butterworth-Heinemann; 2019.

20. International Organization for Standardization. ISO 6876: dental root canal sealing materials. Geneva: ISO; 2012.

21. Kucukyildiz EN, Dayi B, Altin S, Yigit O. In vitro comparison of physical, chemical, and mechanical properties of graphene nanoplatelet added Angelus mineral trioxide aggregate to pure Angelus mineral trioxide aggregate and calcium hydroxide. Microsc Res Tech. 2021;84(5):929-42. http://dx.doi.org/10.1002/jemt.23654. PMid:33410148.

22. American National Standards Institute – ANSI. American Dental Association – ADA. New American Dental Association specification no. 30 for dental zinc oxide-eugenol type restorative materials. Washington; 2013.

23. Engqvist H, Schultz-Walz JE, Loof J, Botton GA, Mayer D, Phaneuf MW, et al. Chemical and biological integration of a mouldable bioactive ceramic material capable of forming apatite in vivo in teeth. Biomaterials. 2004;25(14):2781-7. http://dx.doi.org/10.1016/j.biomaterials.2003.09.053. PMid:14962556.

24. Lööf J. Calcium-aluminate as biomaterial: synthesis, design and evaluation [thesis].Uppsala: Acta Universitatis Upsaliensis; 2008.

25. Chen CC, Ho CC, David Chen CH, Ding SJ. Physicochemical properties of calcium silicate cements for endodontic treatment. J Endod. 2009;35(9):1288-91. http://dx.doi.org/10.1016/j.joen.2009.05.036. PMid:19720233.

26. Islam I, Chng HK, Yap AU. Comparison of the physical and mechanical properties of MTA and portland cement. J Endod. 2006;32(3):193-7. http://dx.doi.org/10.1016/j.joen.2005.10.043. PMid:16500224.

27. Ørstavik D. Materials used for root canal obturation: technical, biological and clinical testing. Endod Topics. 2005;12(1):25-38. http://dx.doi.org/10.1111/j.1601-1546.2005.00197.x.

28. Acuña-Gutiérrez IO, Escobedo-Bocardo JC, Almanza-Robles JM, Cortes-Hernandez DA, Saldivar-Ramirez MM, Resendiz-Hernandez PJ, et al. Development of LiCl-containing calcium aluminate cement for bone repair and remodeling applications. Mater Sci Eng C Mater Biol Appl. 2017;70(Pt 1):357-63. http://dx.doi.org/10.1016/j.msec.2016.09.022. PMid:27770903.

29. Sunnegårdh-Grönberg K. Calcium aluminate cement as dental restorative: mechanical properties and clinical durability. Umeå: Institutionen för Odontologi, Umeå Universitet; 2004.

30. Radwan MM, Nagi SM, Abd El-Hamid HK. Physico-mechanical characteristics of tri-calcium silicate pastes as dentin substitute and interface analysis in class II cavities: effect of CaCl2 and SBF solutions. Heliyon. 2019;5(6):e01975. http://dx.doi.org/10.1016/j.heliyon.2019.e01975. PMid:31294120.

31. Liu WN, Chang J, Zhu YQ, Zhang M. Effect of tricalcium aluminate on the properties of tricalcium silicate-tricalcium aluminate mixtures: setting time, mechanical strength and biocompatibility. Int Endod J. 2011;44(1):41-50. http://dx.doi.org/10.1111/j.1365-2591.2010.01793.x. PMid:20812942.

32. Septodont. Biodentine active biosilicate technology: scientific file. France: Septodont Saint-Maur-des-Fossés; 2010.

33. Dawood AE, Manton DJ, Parashos P, Wong R, Palamara J, Stanton DP, et al. The physical properties and ion release of CPP-ACP-modified calcium silicate-based cements. Aust Dent J. 2015;60(4):434-44. http://dx.doi.org/10.1111/adj.12255. PMid:25424362.

34. Santos AD, Araujo EB, Yukimitu K, Barbosa JC, Moraes JC. Setting time and thermal expansion of two endodontic cements. Oral Surg Oral Med Oral Pathol Oral Radiol Endod. 2008;106(3):e77-9. http://dx.doi.org/10.1016/j.tripleo.2008.04.021. PMid:18602305.

35. Antunes Bortoluzzi E, Juárez Broon N, Antonio Hungaro Duarte M, de Oliveira Demarchi AC, Monteiro Bramante C. The use of a setting accelerator and its effect on pH and calcium ion release of mineral trioxide aggregate and white Portland cement. J Endod. 2006;32(12):1194-7. http://dx.doi.org/10.1016/j.joen.2006.07.018. PMid:17174682.

36. Namazikhah MS, Nekoofar MH, Sheykhrezae MS, Salariyeh S, Hayes SJ, Bryant ST, et al. The effect of pH on surface hardness and microstructure of mineral trioxide aggregate. Int Endod J. 2008;41(2):108-16. http://dx.doi.org/10.1111/j.1365-2591.2007.01325.x. PMid:18042226.

37. Deniz Arısu H, Eligüzeloglu Dalkilic E, Alkan F, Erol S, Uctasli MB, Cebi A. Use of artificial neural network in determination of shade, light curing unit, and composite parameters' effect on bottom/top vickers hardness ratio of composites. BioMed Res Int. 2018;2018:4856707. http://dx.doi.org/10.1155/2018/4856707. PMid:30539012.

38. Moraes RR, Marimon JL, Schneider LF, Sinhoreti MA, Correr-Sobrinho L, Bueno M. Effects of 6 months of aging in water on hardness and surface roughness of two microhybrid dental composites. J Prosthodont. 2008;17(4):323-6. http://dx.doi.org/10.1111/j.1532-849X.2007.00295.x. PMid:18266654.

39. Rahimi S, Shahi S, Torabi Z, Rezaie Y, Ghasemi N, Abolhasani S. The effect of a mineralized bone graft on the surface microhardness of mineral trioxide aggregate and biodentine. Iran Endod J. 2018;13(1):83-7. http://dx.doi.org/10.22037/iej.v13i1.14953. PMid:29692841.

40. Kaup M, Schafer E, Dammaschke T. An in vitro study of different material properties of Biodentine compared to ProRoot MTA. Head Face Med. 2015;11(1):16. http://dx.doi.org/10.1186/s13005-015-0074-9. PMid:25934270.

41. Camilleri J. Characterization of hydration products of mineral trioxide aggregate. Int Endod J. 2008;41(5):408-17. http://dx.doi.org/10.1111/j.1365-2591.2007.01370.x. PMid:18298574.

42. Taylor HF. Cement chemistry. London: Thomas Telford; 1997. http://dx.doi.org/10.1680/cc.25929.

43. Espir CG, Guerreiro-Tanomaru JM, Spin-Neto R, Chavez-Andrade GM, Berbert FL, Tanomaru-Filho M. Solubility and bacterial sealing ability of MTA and root-end filling materials. J Appl Oral Sci. 2016;24(2):121-5. http://dx.doi.org/10.1590/1678-775720150437. PMid:27119759.

44. Nguyen T. Obturation of the root canal system. Pathways of the Pulp. 1994;6:219-71.

45. Singh S, Podar R, Dadu S, Kulkarni G, Purba R. Solubility of a new calcium silicate-based root-end filling material. J Conserv Dent. 2015;18(2):149-53. http://dx.doi.org/10.4103/0972-0707.153053. PMid:25829696.

46. Alazrag MA, Abu-Seida AM, El-Batouty KM, El Ashry SH. Marginal adaptation, solubility and biocompatibility of TheraCal LC

compared with MTA-angelus and biodentine as a furcation perforation repair material. BMC Oral Health. 2020;20(1):298. http://dx.doi.org/10.1186/s12903-020-01289-y. PMid:33121465.

47. Bortoluzzi EA, Broon NJ, Bramante CM, Felippe WT, Tanomaru M Fo, Esberard RM. The influence of calcium chloride on the setting time, solubility, disintegration, and pH of mineral trioxide aggregate and white Portland cement with a radiopacifier. J Endod. 2009;35(4):550-4. http://dx.doi.org/10.1016/j.joen.2008.12.018. PMid:19345803.

48. Bodanezi A, Carvalho N, Silva D, Bernardineli N, Bramante CM, Garcia RB, et al. Immediate and delayed solubility of mineral trioxide aggregate and Portland cement. J Appl Oral Sci. 2008;16(2):127-31. http://dx.doi.org/10.1590/S1678-77572008000200009. PMid:19089204.

49. Acris de Carvalho FM, Silva-Sousa YTC, Saraiva Miranda CE, Miller Calderon PH, Barbosa AFS, Domingues De Macedo LM, et al. Influence of ultrasonic activation on the physicochemical properties of calcium silicate-based cements. Int J Dent. 2021;2021:6697988. http://dx.doi.org/10.1155/2021/6697988. PMid:33574844.

50. Fridland M, Rosado R. Mineral trioxide aggregate (MTA) solubility and porosity with different water-to-powder ratios. J Endod. 2003;29(12):814-7. http://dx.doi.org/10.1097/00004770-200312000-00007. PMid:14686812.

51. Kraft L, Hermansson L. Deformation characteristics in various calcium aluminate cement admixtures investigated with three different methods [Internet]. In: Int Symp on Ultra High Performance Concrete; 2004; Kassel, Germany. Uppsala: Uppsala University; 2004 [cited 2022 jan 2]. Available from: http://urn.kb.se/resolve?urn=urn:nbn:se:uu:diva-70208

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Date submitted: 2022 Jan 02
Accept submission: 2022 Feb 21