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ORIGINAL ARTICLE

Examining the Physicochemical Composition of Three Bioceramic Putties for Endodontic Use

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ABSTRACT

Objective: This study aimed to address the lack of comparative analyses of newly developed bioceramic materials by examining the chemical composition, thermodynamic profile, and microscopic surface features of three bioceramic putties: EndoSequence BC Root Repair Material Fast Set Putty (ESRRM-FS), BIO-C Repair (BCR), and Cera Putty (CP).

Methods: Samples of each of the three bioceramic putty obtained directly from manufacturers were prepared for analysis of physicochemical composition and microscopic features by differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), scanning electron microscopy (SEM) imagery, and energy-dispersive X-ray spectroscopy (EDS). The data obtained was qualitatively and statistically analysed. Statistical significance was determined at $p \leq 0.05$.

Results: DSC analysis indicated a standard polymeric vehicle for BCR and CP, coinciding with the polyethene glycol (PEG) thermal profile; the polymeric vehicle in ESRRM-FS remains to be identified. The material with the highest heat capacity was CP ($p<0.05$), followed by ESRRM-FS and BCR. TGA revealed an inflexion point at 394.12 ºC for ESRRM-FS, which may correspond to the mass loss of dihydroxylation of calcium hydroxide. A more homogenous structure was observed in scanning electron microscopy (SEM) images for ESRRM-FS. EDS analysis indicated BCR had minimal amounts of aluminium (2.06±0.44%) and a lower percentage of calcium than ESRRM-FS (9.11±1.38% vs. 11.3±0.87%). CP was composed of aluminium (49.35±7.01%), carbon (30.65±5.62%), and oxygen (16.75±2.44%); no silicon was identified. ESRRM-FS had no aluminium present and the highest calcium percentage (11.3±0.87%) ($p<0.05$).

Conclusion: BCR is a Portland cement-derived material with a lower percentage of calcium than ESRRM-FS and minimal amounts of aluminium. CP is a monocalcium aluminate cement, mainly composed of aluminium, carbon, and oxygen. ESRRM-FS is a biphasic material with the highest calcium percentage among all materials studied and no aluminium.

Keywords: Bioceramic putties, differential scanning calorimetry, energy-dispersive X-ray spectroscopy, scanning electron microscopy, thermogravimetric analysis

HIGHLIGHTS

- The chemical composition of three putties (EndoSequence BC Root Repair Material Fast Set Putty (ESRRM-FS), BIO-C Repair (BCR), and Cera Putty (CP)) were very different.
- ESRRM-FS showed a higher calcium content and did not show the presence of aluminium, whereas a higher amount of aluminium was identified in CP.
- ESRRM-FS and BCR showed endothermic heat capacity close to the body temperature (37°C), but all three putties showed mass stability below 100°C.
- ESRRM-FS showed an increased microscopical homogeneity with fewer crystalline forms and a smoother surface.

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INTRODUCTION

Tricalcium silicate types of cement are multipurpose bioceramic materials used for sealing root canals, apexification, perforation repair, resorption, and retrograde obturation in microsurgical treatments (1). Mineral trioxide aggregate (MTA) is a Portland cement-derived bioceramic material developed by Torabinejad and White in the 1990s, and it has shown excellent biocompatibility, antimicrobial properties, and osteoinductive action (2). In the last three decades, the MTA formula has been improved by reducing the iron content, substituting a radiopacifying agent to decrease discolouration potential, adding setting accelerators and removing calcium sulphate from the powder to reduce the long setting time (3). A significant milestone in developing bioceramic materials was the introduction of putty-like materials, such as ready-to-use pre-mixed bioceramic materials, which will not be set during storage (4). Because changing the water-to-cement ratio increases the porosity, permeability, and solubility of hydraulic cement (5, 6), adjusting the presentation to a pre-mixed syringe eliminates the risk of affecting its physical properties and potentially altering the water and powder ratio (7, 8). The impact of thermal heat on the composition of the following three pre-mixed, more readily available bioceramic putties has not been thoroughly examined.

EndoSequence Root Repair Material Putty (ESRRM, Brasseler, Savannah GA, USA) consists of calcium phosphate monobasic and calcium silicates, available in ready-to-use, pre-mixed homogeneous presentations (9). EndoSequence BC Root Repair Material Fast Set Putty (ESRRM-FS) exhibits excellent physical and biological properties (8, 10), high calcium release (11), and low discolouration potential (12). Moreover, this putty presents a fast-setting time (less than an hour), which offers a clinical advantage over similar biomaterials (8).

Bio-C Repair (Angelus, Londrina, PR, Brazil) (BCR) is another pre-mixed, ready-to-use, silicate-based putty used in endodontic surgery, perforation repair, and apexification (13). BCR comprises Portland cement-derived materials and zirconium oxide; it has shown optimal calcium release and alkalinising properties (14, 15) with minor tooth colour alteration (15). BCR also induces biomineralisation *in vivo* and *in vitro* (16, 17) and has excellent biocompatibility (13, 14, 17). Compared to MTA, BCR has adequate dimensional stability, filling capacity (18), and better bond strength (19).

Cera Putty (Meta Biomed Co., Cheongju, Korea) (CP) is a similar pre-mixed endodontic cement. Unlike BCR and ESRRM FS, CP does not contain calcium silicates and is mainly composed of calcium aluminate, zirconium dioxide, lithium carbonate, and polyethene glycol (PEG) 400 (in an email from VD, Meta Biomed Inc. in June 2022). PEG contains one hydrophobic region and one hydrogen bonding site; it acts as an anhydrous, water-miscible carrier for CP and BCR. PEG also acts as a viscosity-enhancing agent by reducing the separation rate and increasing homogeneity in Portland cement-derived heterogeneous materials (20–22). During clinical use, water diffuses from the tissues into the putty to displace the vehicle, which in turn causes hydration and setting (22). Due to PEG's importance in the composition of these putties, PEG samples were also included in the thermal analyses of this investigation.

New, ready-to-use materials continue to enter the market, yet availability can vary in Europe and globally. Examining the range of obturation biomaterials, including bioceramic putty properties, is crucial in helping clinicians select a physical and commercial presentation based on a specific clinical application. For example, a recent study reported the microscopic surface features, chemical composition, and thermodynamic profile of endodontic sealers after heat exposure, showing essential changes in the physicochemical composition that can be affected by the obturation techniques (23). Various studies have assessed the composition and ultrastructural morphology of ESRRM-FS and BCR (14, 17, 22); however, no information regarding CP exists.

This study aimed to compare the microscopic surface features, thermodynamic profile, and chemical composition of three pre-mixed bioceramic putties, ESRRM-FS, BCR, and CP, utilising differential scanning calorimetry (DSC), thermogravimetric (TGA), and scanning electron microscopy (SEM) with energy dispersive x-ray (EDS) analysis techniques.

MATERIALS AND METHODS

Three bioceramic putties were examined in this study. The brand names, manufacturers, lot numbers, and compositions of the materials are listed in Table 1. To accurately characterise the original presentation of each material (to avoid contamination or second-reaction changes), all the materials were analysed fresh, with samples taken directly from their reservoir. No moulding was needed for DSC and TGA analyses. Still, a cylindrical aluminium sample holder (10 mm diameter) was used for SEM and EDS analyses, and a uniform layer of 1 mm width was prepared to cover its surface. The sample size was adjusted in each of the experiments performed.

DSC Analysis

Six samples (4 mg each) of each bioceramic putty were placed in an aluminium DSC crucible, promptly sealed and positioned in a DSC unit Q200 (TA Instruments, New Castle, DE, USA). Representative samples each of polyethene glycol (PEG), PEG 400, PEG 1450, PEG 4000, and PEG 8000 (Sigma-Aldrich, St. Louis, MO, USA) were included to obtain thermograms of the polymer reported by the manufacturers. The samples were thermally scanned starting at 20°C, and heating increased at 10°C per minute up to 250°C. Six samples of each product and the PEG samples were analysed. Thermal analysis software (Universal Analysis 2000 for Windows 2000, XP, Vista version 4.5A, TA Instruments) was used to determine thermal peak areas. Maximum and endothermic signals were also determined, and each putty sample's heat capacity (J/g) was calculated.

TGA Analysis

Six mg of each putty was placed in a platinum pan and accurately weighed by the TGA unit (Q500, TA Instruments). For each bioceramic putty, the experiment was done in triplicate (n=3). After placing each sample in the TGA furnace, the weight of each sample was recorded by the unit analyser, and then nitrogen gas heat was applied. Also, 6 mg of single samples of PEG 400, PEG 1450, PEG 4000, and PEG 8000 (Sigma-Aldrich) were included for comparison. Heat application was increased at 20°C per minute from 20°C to a maximum of 800°C. The on-

TABLE 1. Chemical composition of endodontic bioceramic putty by manufacturer

Commercial name (abbreviation)	Company (city, country)	Lot number	Composition
EndoSequence BC Root Repair Material Fast Set Putty (ESRRM-FS)	Brasseler Savannah, GA USA	2201FSPS	Calcium silicates, calcium phosphate monobasic, calcium hydroxide, zirconium oxide, tantalum oxide, filler, and thickening agents
BIO-C Repair (BCR)	Angelus Londrina, PR Brazil	60844	Tricalcium silicate, dicalcium silicate, tricalcium aluminate, calcium oxide, zirconium oxide, silicon oxide, polyethene glycol, and iron oxide
Cera Putty (CP)	Meta Biomed Cheongju Korea	CPT2202171	Calcium aluminate compounds, zirconium dioxide, lithium carbonate, polyethene glycol 400, and excipients

set temperature (To), when the weight loss begins, and the inflexion point (Tp), the point showing the most significant rate of change on the weight loss curve, were recorded for each TGA curve. TGA software (Universal Analysis 2000 for Windows 2000, XP, Vista version 4.5, TA Instruments) was used to analyse the data and determine the calorimetric curves.

SEM and EDS Analyses

Three samples of each material were selected for analysis and prepared as described in the methodology above. A gold palladium powder alloy was used to sputter-coat samples, which were then secured on aluminium stubs with carbon-adhesive tape. A SEM (ASEM Microscopy JEOL JSM-6390LV, Peabody, MA, USA), operating at 10 kV, captured images of each sealer specimen's upper surfaces at 500×, 2,000×, and 5000× magnifications. An EDS (Oxford INCA X-Sight 7582 M, Oxford, UK) was used for the elemental analysis of all bioceramic putty samples. Finally, six areas from each material were randomly selected and analysed.

Statistical Analysis

DSC and EDS quantitative results were analysed. The results section presents means with their corresponding standard deviations to measure the standard uncertainty. A Shapiro-Wilk test was used to determine normality within the results, and a Kruskal-Wallis test was used to identify a statistical difference within the groups. A Wilcoxon test was used to analyse differences between specific groups.

SPSS (version 27.0, IBM SPSS Inc, Chicago, IL, USA) was used to process and analyse the data. The level of significance was set at $\alpha=0.05$.

RESULTS

DSC analysis demonstrates that temperature changes did not influence these materials' polymerisation or crystallisation process. The body temperature of 37°C may not affect the material's physical properties as no changes were detected below 40°C. None of the biomaterials showed exothermic enthalpies on the calorimetric curve. An increase in heat led to an endothermic change, indicating an increase in plastic deformation. Endothermic mean enthalpy was obtained at a lower temperature with ESRRM-FS (93.8±5.41°C), followed by CP (110.17±10.6°C) and then by BCR (126.96±19.1°C). These results were statistically different between ESRRM-FS and BCR ($p<0.05$) (Fig. 1). The mean endothermic enthalpy of PEG 400 was 124.06°C, for PEG 1450 was 55.80°C, for PEG 4000 was 62.46°C, and for PEG 8000

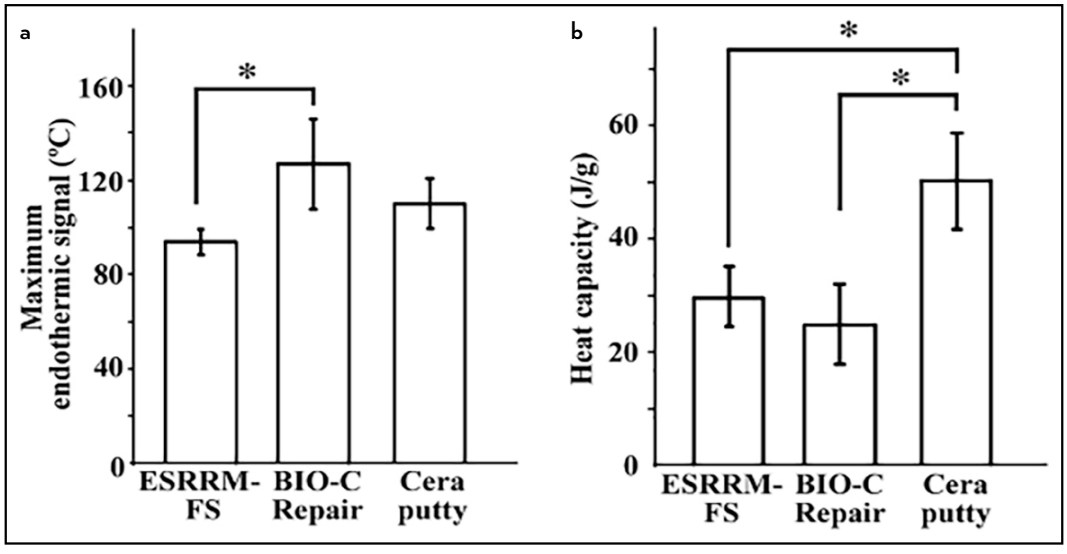

Figure 1. Differential scanning calorimetry (DSC) results (n=6 per material). (a) Maximum endothermic signal (°C). (b) Heat capacity (J/g)

*: $p<0.05$. ESRRM-FS: EndoSequence BC Root Repair Material Fast Set Putty, BIO-C Repair, and Cera Putty

65.90°C. The material with the highest mean heat capacity was CP (50.09±8.6 J/g), followed by ESRRM-FS (29.66±5.24 J/g) and BCR (24.78±7.12 J/g). These mean values were only statistically different for CP ($p<0.05$) (Fig. 1).

TGA showed that ESRRM-FS had the lowest onset temperature (To) (138.54°C), followed by CP (264.35°C) and BCR (321.45°C). Multiple inflexion changes were observed, indicating different heat sensitivity related to the various compositions of each material (Fig. 2, Table 2). The inflection point (Tp) of PEG 400 was 337.85°C, for PEG 1450 was 403.91°C, for PEG 4000 was 405.78°C, and for PEG 8000 was 404.45°C. BCR showed the most significant mass loss at 800°C (26.41%), followed by CP (20.11%) and ESRRM-FS (13.35%) (Fig. 2, Table 2).

SEM images of ESRRM-FS demonstrated a more homogenous structure than those of BCR and CP. At 2,000× and 5,000× magnifications, BCR and CP showed crystalline structures with a heterogeneous distribution. The particle size in BCR and CP mostly ranged from 1 to 5 micrometres. The particle shape in BCR and CP was irregular (Fig. 3).

EDS revealed different elemental compositions within the materials by mean atomic percentage (Figs. 4, 5). For example, copper was only identified in CP (1.28±0.5%), and ESRRM-FS was the only material that showed the presence of sodium (1.2±0.227%). Silicon was not identified in CP but in ESRRM-FS (2.12±0.25%) and BCR (2.16±0.43%). Aluminium was absent in ESRMM-FS but was found in BCR (2.06±0.44%) and, most importantly, in CP (49.35±7.01%) with a significant difference ($p<0.05$). The mean atomic percentage of carbon was not statistically different among ESRRM-FS (30.48±1.46%), BCR (37.93±3.65%), and CP (30.65±5.62%). However, all the putties showed the presence of oxygen, with statistically different percentages ($p<0.05$). All the biomaterials also revealed the presence of calcium, with significant differences among all groups. ESRRM-FS showed a higher mean level of calcium (11.3±0.87%), followed by BCR (9.11±1.38%) and CP (0.59±0.19%). Finally, zirconium was also present in all the putties at significantly different levels. BCR showed a higher mean level of zirconium (2.8±0.43%), followed by ESRRM-FS (1.68±0.18%) and CP (1.17±0.17%) (Figs. 4, 5).

DISCUSSION

Different calorimetric properties were evaluated for the three bioceramic putties tested in this research. Although it is not common for clinicians to consider these aspects when choosing different biomaterials, they can offer important information to assess clinically. DSC analyses the endothermic

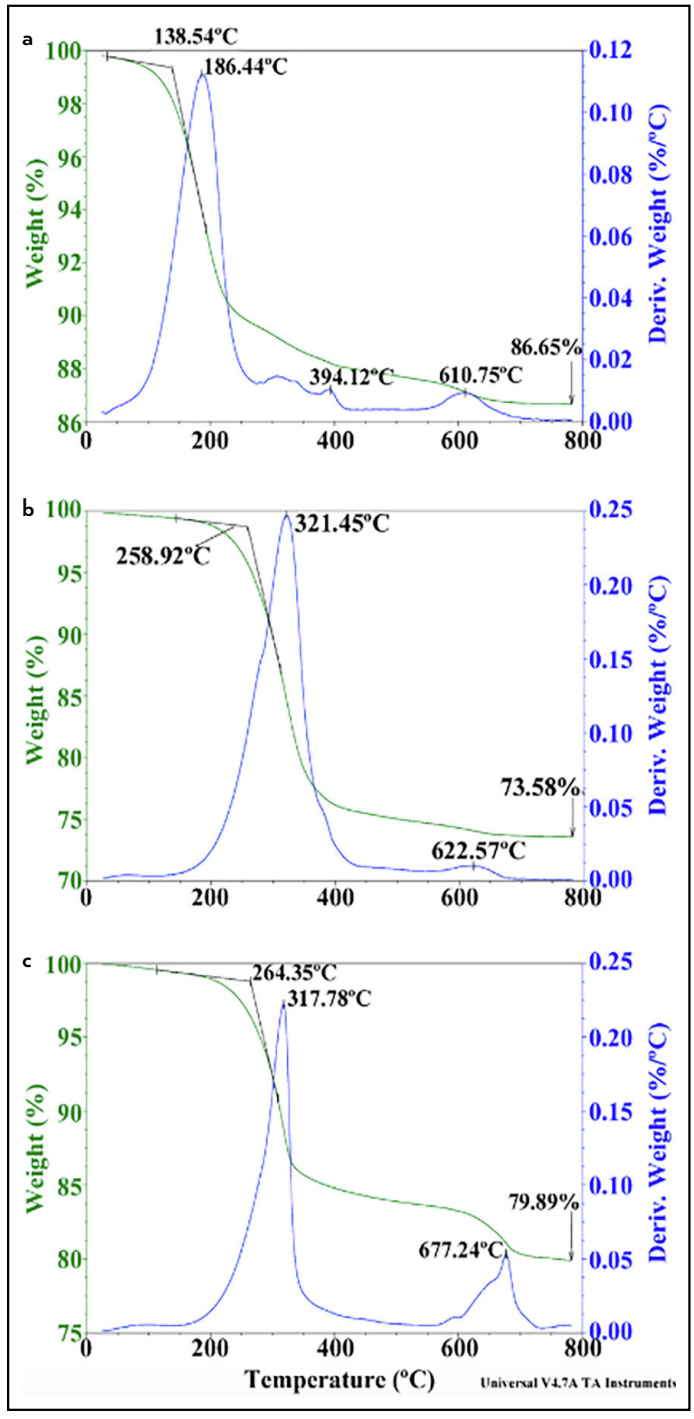

Figure 2. Thermogravimetric (TGA) analysis of bioceramic putties (n=3 per material). (a) EndoSequence BC Root Repair Material Fast Set Putty, (b) BIO-C Repair, (c) Cera Putty

TABLE 2. Thermogravimetric (TGA) analysis of the examined bioceramic putties

Product	Onset point (To) (°C)	Inflection points (Tp) (°C)	Maximum mass loss at 800°C (mass%)
EndoSequence BC Root Repair Material Fast Set Putty (ESRRM-FS)	138.54	186.44 / 394.12 / 610.75	13.50
BIO-C Repair (BCR)	321.45	321.45 / 622.57	26.42
Cera Putty (CP)	264.35	317.78 / 677.24	20.11

To: Onset point; Tp: Inflection point

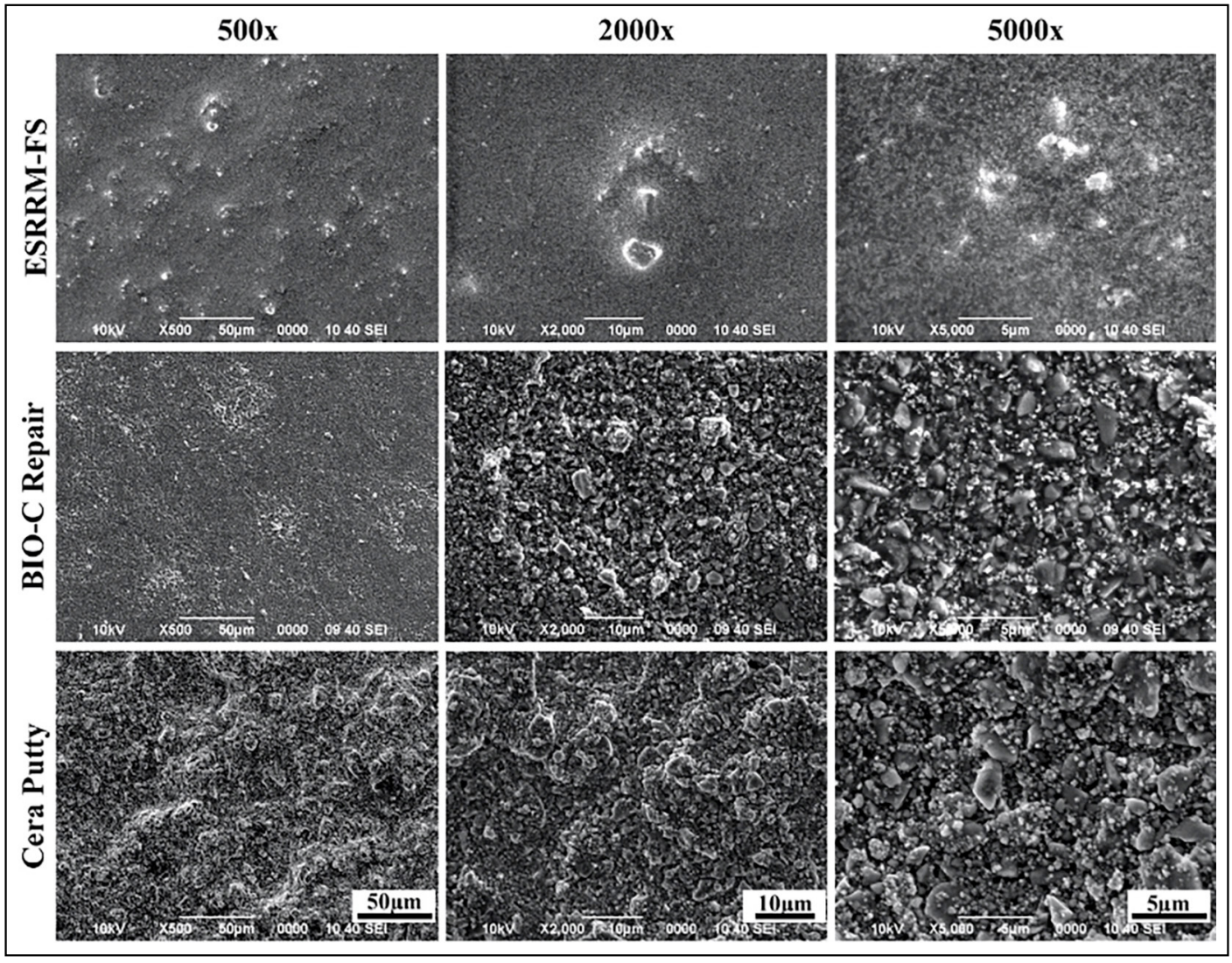

Figure 3. Scanning electron microscopy (SEM) representative images of bioceramic putties at 500×, 2,000×, and 5000× magnifications (n=3 per material)

ESRRM-FS: EndoSequence BC Root Repair Material Fast Set Putty, BIO-C Repair, and Cera Putty

and exothermic changes that can occur after temperature changes so that this analysis can measure the quantity of heat radiated or absorbed excessively by the sample based on a temperature difference between the sample and the reference material (24). By analysing the heat capacity (or the temperature difference between the sample and reference pans), one can examine how the material may behave under different biological and thermal conditions, extreme external changes, or if body temperature can favour physical changes within the sample. TGA analysis is used to analyse the mass changes through the desired temperature range that allows specific determinations (25) to observe the stability of the mass of different materials and the presence or absence of specific components. By calculating the first-order derivative for the temperature, the inflexion point (Tp) - the maximum mass loss rate at a specific temperature - can be determined (26). The inflexion point is crucial to understanding the vulnerability of certain materials under thermal changes or whether the polymers reported in their composition behave as expected in the biological temperature range.

DSC analysis can detect amorphous and crystalline phases of set MTA and Portland cement (27). In the DSC analysis, the endothermic enthalpy of ESRRM-FS, BCR, CP, and PEG 400 were 93.8±5.41°C, 126.96±19.1°C, 110.17±10.6°C, 124.06°C, respectively. These temperatures were above human body temperature, approximately 37°C; thus, human body temperature did not affect the physical properties of our study's tested putties. Our findings indicated that the endothermic enthalpy of BCR resembled that of PEG 400 and that BCR showed no statistical significance from CP. This result implies that BCR and CP used similar polymeric vehicles such as PEG 400. However, the endothermic enthalpy of ESRRM-FS showed statistical significance from BCR. While the specific polymeric vehicle used in ESRRM-FS is unknown, our finding agrees with Camilleri et al., which suggested that ESRRM-FS used a different polymeric vehicle (28).

TGA analysis provided a better understanding of the polymeric vehicle in the analysed putties than DSC analysis. TGA results showed an inflexion point at 337.85°C for PEG 400, which closely corresponded to BCR (321.4°C) and CP (317.78°C). This

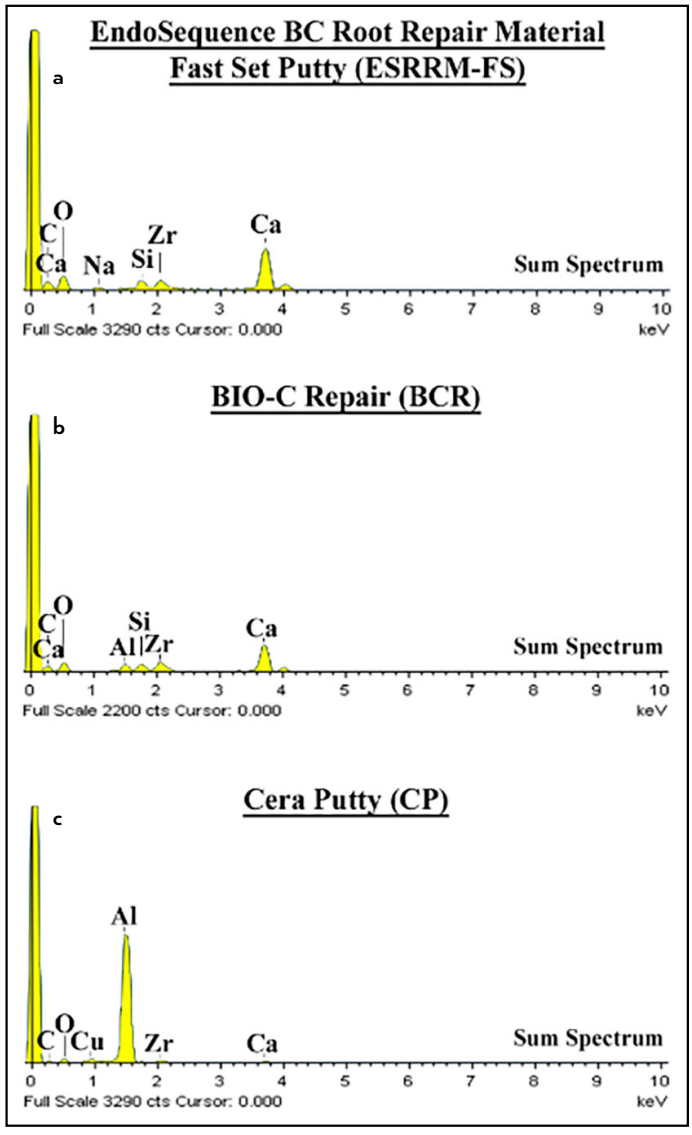

Figure 4. Energy-dispersive spectrometer (EDS) representative histograms(n=6 per material). (a) EndoSequence BC Root Repair Material Fast Set Putty, (b) BIO-C Repair, (c) Cera Putty

similarity may be related to the presence of PEG in the polymeric matrix, as reported in the chemical composition of BCR and CP. A low inflexion point of ESRRM-FS at 186.44°C, which did not correlate with any PEGs inflexion points, may be explained by the incorporation of a different type of polymeric-anhydrous vehicle as a manufacturing trade secret based on Camilleri's theory to mix super-plasticisers with endodontic cement (29). Only ESRRM-FS includes calcium hydroxide in the chemical composition among the three putties tested. However, since PEG can interact with calcium hydroxide and calcium silicates by changing the hydration reactions, it is speculated that PEG was not found in the chemical composition of ESRRM-FS. It is known that the alkalinity of calcium hydroxide can decompose PEG by interacting with calcium ions (22) and intercalating with the calcium silicate hydrate complex in the interlayer space (30, 31). In the present study, the high TGA inflexion points of ESRRM-FS, BCR, and CP were 610.75°C, 622.57°C, and 677.24°C, respectively (Table 2 and Fig. 2). This temperature range (610°C to 677°C) coincides with calcium carbonate decomposition to calcium oxide and carbon dioxide, as it has been extensively confirmed in the cement industry (32, 33). The presence of calcium carbonate is not reported in the chemical composition of any of the tested putties; however, Grech et al. (34) reported that another hydraulic bioactive material, Biodentine (Septodont, Saint Maur des Fossés, France), had calcium carbonate added to the clinker as a nucleation site for calcium silicate hydrate. Its presence could also be explained by the surface carbonation of calcium hydroxide (27). In future research, it would be essential to examine if calcium carbonate is present in the original composition of the putties or if the signal identified in TGA results from a subproduct or chemical change after a temperature increase.

SEM coupled with EDS (SEM and EDS) is a well-established method for calcium silicate-based sealers to evaluate surface characterisation (35). Therefore, to better understand these materials' composition beyond their physical behaviour, SEM and EDS analysis was conducted for calcium silicate-based putties in the present study and summarised in Figures 4 and 5. The percentages of calcium, silicon, and sodium varied among putties. A higher percentage of calcium in ESRRM-FS than in BCR (11.3% vs 9.11%) could be explained by the fact that calcium phosphate monobasic was added only to ESRRM-FS while both ESRRM-FS and BCR contained calcium silicates. The percentage of calcium and silicon in ESRRM-FS closely matches the one reported in a previous study for ESRRM, a similar material with a more extended setting time (9). The silicon content was similar between ESRRM-FS and BCR. The percentage of silicon and carbon obtained in the present study for BCR is comparable to the reported by Ghilotti et al. (13); however, our study found less aluminium and calcium and more zirconium in BCR. While sample sizes were small, sodium was only detected in ESRRM-FS among the three putties tested. The addition of sodium activators or setting-accelerator chemicals such as sodium hydroxide, sodium sulphate, or sodium carbonate could explain the presence of this element in ESRRM-FS, but further chemical analysis is necessary to confirm this observation. It is worth noting that a previous investigation by Zamparini et al. (11) also detected sodium in ESRRM putty and paste, but only after 28 days of immersion in Hanks' Balanced Salt Solution. In contrast, our present study used fresh samples.

The metallic elements in putty composition impact radiopacity and cytotoxicity in the patient care setting, and the percentages of these elements also varied among putties. BCR mainly comprised carbon and oxygen; this putty showed the highest rate of zirconium among the three putties in our study. Future research could examine how this statistical difference among ESRRM-FS, BCR, and CP might correlate with the radiopacity in the clinical setting. The presence of aluminium in BCR and its absence in ESRRM-FS suggest an innovative manufacturing process of ESRRM-FS. For example, the sol-gel synthesis technique allows for the creation of a bioactive cement composed mainly of tricalcium silicate and free of tricalcium aluminate, which is cytotoxic (36). An innovative manufacturing process could also be indicated by the more homogeneous structure due to a finer particle size of ESRRM-FS in all SEM images (Fig. 3) in our present study (37).

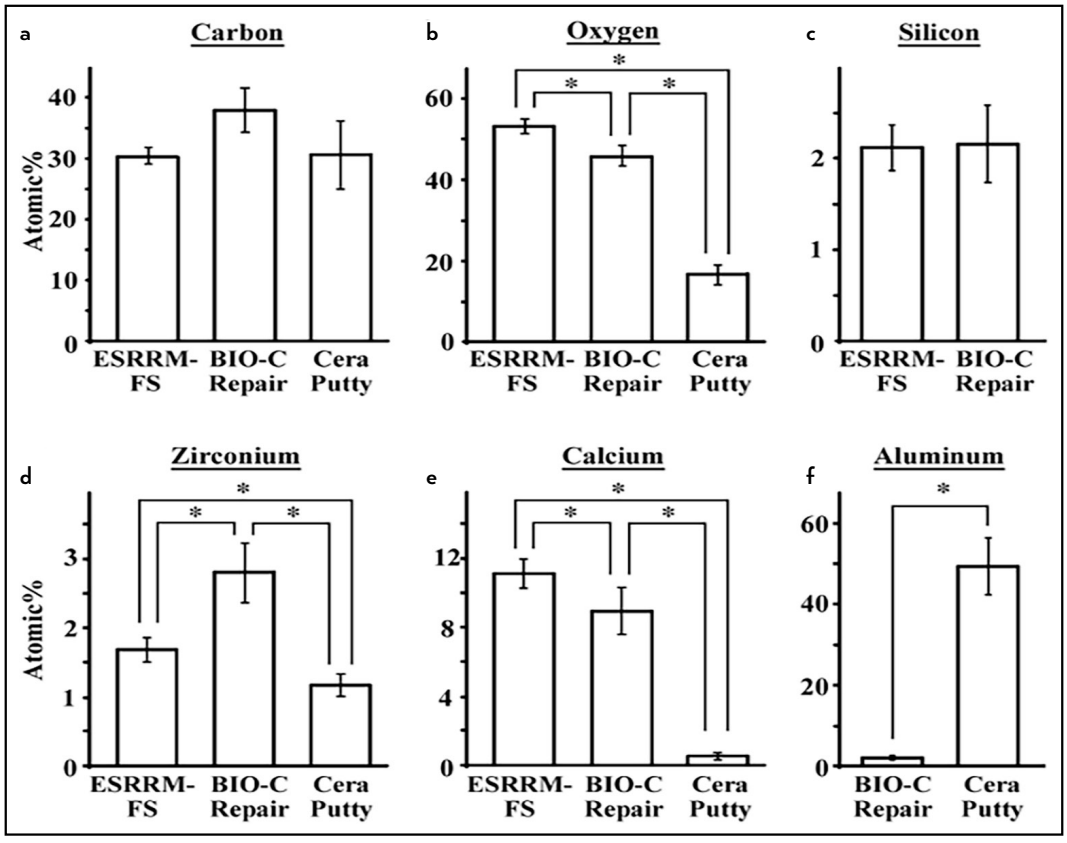

Figure 5. Energy-dispersive spectrometer (EDS) elements atomic percentage of bioceramic putties (n=6 per material). (a) carbon, (b) oxygen, (c) silicon, (d) zirconium, (e) calcium, (f) aluminium

*: p<0.05. ESRRM-FS: EndoSequence BC Root Repair Material Fast Set Putty

CP includes an alkali salt, such as lithium carbonate, as a setting accelerator. Lithium carbonate has also been shown to accelerate the crystallisation of alumina cement by substituting calcium ions with lithium ions in the transition phases (38); however, no lithium was identified in CP in our study, perhaps due to a low amount undetected by the EDS analysis. The absence of silicon, low calcium, and a large percentage of aluminium (49.35±7.01%) in CP suggests this putty is a monocalcium aluminate-based material. Monocalcium aluminate cements have different setting reactions than Portland cements and have a higher speed of strength development and greater resistance to acid resistance, which in theory could help in inflamed areas where the acidic pH might interfere with the setting reaction of tricalcium silicate putties (1). Because CP is a newer material, little is known about this product. How this large percentage of aluminium in CP affects the periapical tissues and cement formation in clinical use remains to be studied. It is of interest if CP can be biologically comparable to ESRRM-FS.

The materials' thermodynamic profiles showed stability in the body temperature range; the results also confirm a safe temperate range of 65–123°C, which was also previously reported in a recent study by Chavarria-Bolanos et al. (23). These biopolymeric matrixes based on PEG could be potential delivery systems for the bioactive molecules identified in our study, offering a new field of clinical research. Researchers could compare dimensional stability, colour stability, particle size, and setting time as new materials become available. Future studies also must analyse the stability of these biomaterials in different environments, such as pH changes or exposure to blood and biological fluids.

Although this in vitro study exhibits inherent limitations related to clinical conclusions, valuable observations can be derived for clinicians. Pre-mixed materials, such as putties, can improve clinical consistency and homogeneity while reducing variations arising from dental assistant skill and experience when mixing a powder and a liquid. The excessive presence of aluminium may concern the clinician as it could potentially impact the prognoses of endodontic treatments. Of the three putty materials studied, ESRRM-FS is preferred because it was the only one that did not show the presence of aluminium and a higher content of calcium, which may represent a biological advantage and increased microscopical homogeneity. BCR is the second preferred material because the presence of aluminium is less than that of CP.

CONCLUSION

Under the present experimental conditions, all three putties showed different physicochemical properties. BCR is a Portland cement-derived material with a lower percentage of calcium than ESRRM-FS and minimal amounts of aluminium. CP is a monocalcium aluminate cement, mainly composed of aluminium, carbon, and oxygen. ESRRM-FS is a biphasic material with the highest calcium percentage among all materials studied and no aluminium. The thermal profiles of BCR and CP indicate the presence of a common polymeric vehicle containing PEG, as reported by the manufacturers. In contrast, the polymeric vehicle in ESRRM-FS remains to be identified.

Disclosures

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