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Radiopacity and Chemical Assessment of New Commercial Calcium Silicate-Based Cements

Radiopacidad y Evaluación Química de Cementos Comerciales en Base a Silicato de Calcio

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ABSTRACT: The aim of the study was to evaluate the chemical composition and radiopacity of new calcium-silicate-based cements. Discs of 10 mm x 1 ± 0.1mm were prepared of BiodentineTM, TheraCal, Dycal and GC Fuji IX (n=5). The samples were radiographed directly on an PSP occlusal plate adjacent to an aluminium step wedge. The radiopacity of each specimen was determined according to ISO 9917/2007. Statistical analyses were carried out using ANOVA and Tukey's test at a significance level of 5 %. The chemical constitution of materials was determined by scanning electron microscopy (SEM) and energy dispersive x-ray element mapping. The radiopacities of the materials in decreasing order were: GC Fuji IX (3.45 ± 0.16 mm), Dycal (3.18 ± 0.17), BiodentineTM (2.79 ± 0.22), and TheraCal (2.17 ± 0.17). TheraCal showed the lowest radiopacity compared to the other materials, followed by BiodentineTM. Dycal and GC Fuji IX radiopacity values did not present significant statistical differences. Scanning electron microscopy and energy dispersive X-ray analysis revealed the presence of zirconium in BiodentineTM; and strontium, barium and zirconium in TheraCal as radiopacifying elements. The new calcium silicate cements present distinctive chemical composition. BiodentineTM contains zirconium as a radiopacifying element and has higher radiopacity values than TheraCal, which contains barium and strontium as radiopacifiers.

KEY WORDS: dental materials, silicate cement, chemical analysis, physical properties.

INTRODUCTION

Direct pulp capping consists of covering the exposed vital pulp with a dental material (Hilton, 2009). The aim of direct pulp capping is to maintain pulpal health, allowing patients to retain teeth longer and at lower costs than alternative interventions (Schwendicke & Stolpe, 2014). The dental material used should promote the formation of new reparative dentin (Gandolfi *et al*., 2015) and present sufficient radiopacity to allow its identification in radiographic examinations.

Conventionally, calcium hydroxide-based materials have been used due to their ability to stimulate pulp repair (Gandolfi *et al*., 2015). However, these materials have some disadvantages, such as high solubility and low mechanical properties (Desai & Chandler, 2009). More recently, calcium-silicate-based cements have demonstrated promising clinical results (Parirokh & Torabinejad, 2010). Mineral Trioxide Aggregate (MTA) was the first of these cements, introduced in 1993. Nevertheless, MTA has some disadvantages that discourage its use for pulp capping, such as long setting time and discoloration (Parirokh & Torabinejad). New commercialized calcium-silicate-based cements overcome some of these drawbacks, such as BiodentineTM, which has a faster setting time (Setbon *et al*., 2014) and better color stability (Vallés *et al*., 2016). Also, TheraCal LC was developed, which is a light-cured, resin-modified material.

The changes in the composition of the new calcium-silicate-based cements include changes in the incorporated radiopacifier. Bismuth oxide is added to

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MTA in a 1:4 (wt.%) ratio (Islam *et al*., 2006). However, several studies have shown that bismuth oxide negatively affects its biocompatibility (Camilleri *et al*., 2004; Gomes Cornélio *et al*., 2011) and physical properties (Coomaraswamy *et al*., 2007). Consequently, alternative radiopacifiers have been used in BiodentineTM and TheraCal. BiodentineTM uses zirconium oxide as a radiopacifier (Septodont, 2014). However, some authors have reported a wide range of radiopacity values (ranging from 1.5 to 4.1 mm Al) (Camilleri *et al*., 2013; Grech *et al*., 2013; Tanalp *et al*., 2013; Kaup *et al*., 2015). According to TheraCal's patent, ytterbium fluoride, barium sulfate or bismuth oxide could be incorporated as radiopacifiers (Suh *et al*., 2008), and to our knowledge, only one study has evaluated its radiopacity (Gandolfi *et al*., 2012).

Little research has addressed the radiopacity of BiodentineTM, and few studies have investigated TheraCal´s radiopacity and composition. Therefore, this work aims to evaluate the chemical composition and radiopacity of new commercial calcium-silicate-based cements.

MATERIAL AND METHOD

The calcium-silicate-based cements used in this study were BiodentineTM (Septodont, Saint- Maur-des-Fossés, France) and TheraCal (Bisco Inc., Illinois, USA). Dycal (Dentsply, Connecticut, USA) and GC Fuji IX Capsule (GC America Inc., Illinois, USA) were used as a reference.

Radiopacity evaluation. The radiopacity test was performed according to the methods described by ISO 9917:1 and 9917:2 for water-based cements. The dental materials were mixed following the manufacturer's instructions and placed into molds measuring 1 mm in thickness and 10 mm in diameter. The specimens were covered with glass coverslips and assembled with a clamp to ensure the correct thickness. TheraCal LC was supplied by the manufacturer in pre-mixed syringes; it was dispensed into the mold, then covered with a glass coverslip, assembled with a clamp and polymerized with a light-curing unit for 20 s (EliparTM LED, 3M ESPE, Seefeld, Germany), through upper and lower coverslips. Specimens with notorious clefts, voids, discontinuities or air bubbles were discarded. Thickness was checked with a digital calliper and only specimens whose thickness fell in the range of 1.0 +/- 0.1 mm were used.

Five specimens of each material were placed directly on a photostimulable phosphor plate (PSP, 48 × 54 mm, FireCR Dental, 3DISC Corp., Daejeon, Korea) adjacent to an aluminium (99 % pure) step wedge with step height ranging from 1–10 mm (Odeme, Santa Catarina, Brazil) (Fig. 1a). Radiographs were taken with an x-ray appliance model Myray RXAC (Imolia, Italia) at a tube voltage of 70 Kv, current of 8 mA, exposure time of 0.4 s, and target-film distance of 40 cm. A custom 3-dimensional (3D) printed device was used to ensure standardization of focal distance and angulation of the central ray.

The radiographs were processed (FireCR Dental Reader, 3DISC Imaging. Virginia, USA) and a digital image was obtained. The digital image file was exported to a greyscale analysis software Adobe Photoshop CS6 (Adobe, California, USA). The average grey value (between 0 and 255, with 0 representing pure black and 255 pure white) for each material sample and each step of the wedge were measured (Fig. 1b). A graph of aluminum thickness (in mm) vs. grey value of each aluminum step was plotted, and the logarithmic trend line was drawn. The radiopacity of each specimen, expressed in mm Al, was then determined using the equation of the trend line.

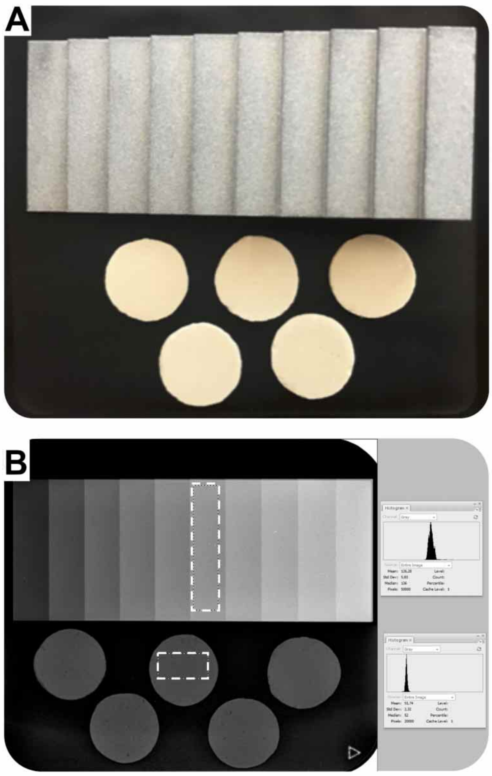

Fig. 1. Cement samples with aluminum step wedge placed on a PSP occlusal plate (a). Digital image with average grey value reading (b).

Elemental analysis of the cements. For each material, one of the specimens was dehydrated, mounted on aluminium stubs and gold coated. Specimens were examined using a scanning electron microscope (SEM, Jeol JSM-IT300LV, JEOL USA Inc., USA) coupled to an energy dispersive x-ray (EDX) detector for elemental analysis with computer-controlled software (Aztec EDS system, Oxford Instruments, Abingdon, UK). Micrographs of the material surface at 1000× magnifications with element EDX mapping were captured, and EDX quantitative chemical analysis was carried out.

Statistical analysis. The radiopacity test data was evaluated using SPSS software (SPSS Inc., Chicago, IL, USA). The results obtained for all materials were submitted to the Shapiro-Wilk normality test. After proving the normality of the sample data distribution, the data were submitted to ANOVA and post hoc Tukey test at a 5 % level of significance.

RESULTS

Radiopacity measurements. The radiopacity evaluation results are shown in Table I. TheraCal showed the lowest radiopacity values, followed by BiodentineTM. Dycal and GC Fuji IX Capsule showed the highest radiopacity value and presented statistically similar radiopacity values ($p > 0.05$).

Table I. Radiopacity values of dental cements in equivalent mm of aluminium.

Materials	Means (± standard deviation)
TheraCal LC	2.17±0.17[a]
Biodentine	2.79±0.22[b]
Dycal	3.18±0.17[c]
GC Fuji IX GP	3.45±0.16[c]

Different letters indicate statistically significant differences (analysis of variance and post hoc Tukey, $p < 0.05$)

Compositional analysis. The major elements (< 10 wt.%) of BiodentineTM are oxygen, carbon and calcium; its minor element components (1–10 wt.%) are silicon, zirconium and chlorine. The constituent elements display a homogeneous distribution, except zirconium, which is observed as accumulations (Fig. 2 a-c). TheraCal is composed mainly of carbon and oxygen, with silicon, calcium, strontium, barium and aluminium as minor element components. The constituent elements display a homogeneous distribution (Fig. 2 d-f). Dycal and GC Fuji IX EDX analysis are shown in Figure 3.

DISCUSSION

In the present study, the radiopacity and chemical composition of new commercial calcium silicate-based cements were investigated. Pulp capping treatment involves the direct cement application on the pulp. This material should promote dental pulpal complex reparation (Gandolfi *et al*., 2015) and have sufficient radiopacity to allow its identification. However, since neat calcium-silicate cements have low intrinsic radiopacity, it is necessary to incorporate different radiopacifier to increase this (Camilleri & Gandolfi, 2010).

In the present study, BiodentineTM had an equivalent radiopacity of 2.79 +/- 0.22 mm Al. Other studies have reported a wide range of radiopacity values for this cement. Camilleri *et al*. (2013) reported a radiopacity between 4 and 5 of mm Al, Grech *et al*. (2013), reported 4.1 mm Al, Tanalp *et al*. (2013) reported 2.8 mm Al and Kaup *et al*. reported a radiopacity value of 1.5 mm Al. This variation between studies could be due to a poor standardization of the manufacturing of the material, as has previously been suggested (Kaup *et al*.), or due to methodological variations with other studies, such as film-to-focus distance (Kaup *et al*.) and different conditions to store samples (Camilleri *et al*., 2013; Grech *et al*.). In the present study, a 3D printed device was used to standardize the film-to-focus distance and to ensure that the central ray is perpendicular to the film. In contrast with other studies (Kaup *et al*.), digital radiography was used rather than conventional radiographs, avoiding the use of an optical densitometer and possible errors due to film processing (Cutajar *et al*., 2011). Also, the aluminum step wedge used in these tests had 1-mm increment, as suggested by ISO standards.

According to ISO-6876:2002, entitled “Dental root canal sealing materials”, the radiopacity should be equivalent to not less than 3 mm Al, and according to ISO 9917:2007, “Water-based cements” should be at least 1 mm Al. Therefore, according to the results of this study, BiodentineTM does not comply with ISO-

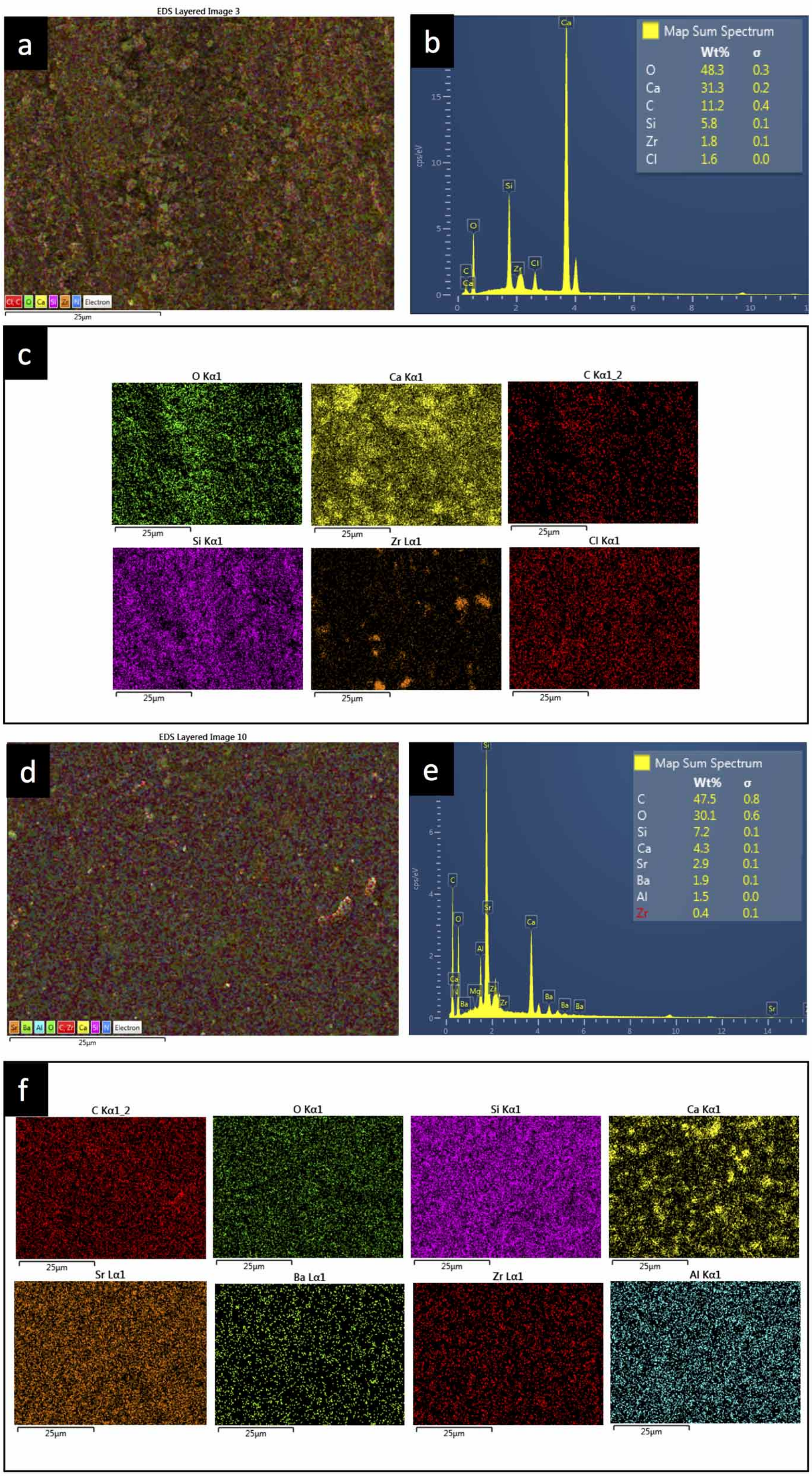

Fig. 2. Representative SEM elemental distribution maps at 1000 x magnification (a, d) with EDX bulk analysis (b, e) and elemental distribution maps of radiopaque elements (c, f) of BiodentineTM (a–c) and TheraCal (d–f).

6876 requirements of radiopacity but does for ISO-9917:2007. However, regarding ISO-6876 for root canal sealing material, when BiodentineTM is used in pulp capping treatments, it is not used for permanent obturation of the root canal. Therefore, under strict consideration, the material for this application is outside the scope of the standard. Similarly, ISO-9917 applies only to cements that are set by an acid-base reaction, which is not the case for calcium-silicate-based cements, which are set by a hydration reaction.

Nevertheless, clinicians have reported difficulty detecting BiodentineTM when assessed by radiography, which has been noted as a disadvantage of this material (Bachoo *et al*., 2013). Since BiodentineTM is indicated for a wide range of indications, including pulp capping, pulpotomy, repair of root/ furcation perforation, and apexification (Septodont), the thickness of the applied material often varies. Nevertheless, for all of the above applications, it is important to distinguish the cement from anatomical structures on a radiograph. The need for application-specific standards for calcium silicate-based cements (conventional and resin-modified) has been proposed (Gandolfi *et al*., 2012).

The radiopacity is related to the atomic number of the elements that constitute the material and its physical density. Tooth structures are mainly made up of calcium and phosphorus, with atomic numbers of 20 and 15 respectively. Therefore, materials with a higher atomic number will be easier to detect in radiographs. In this study, the

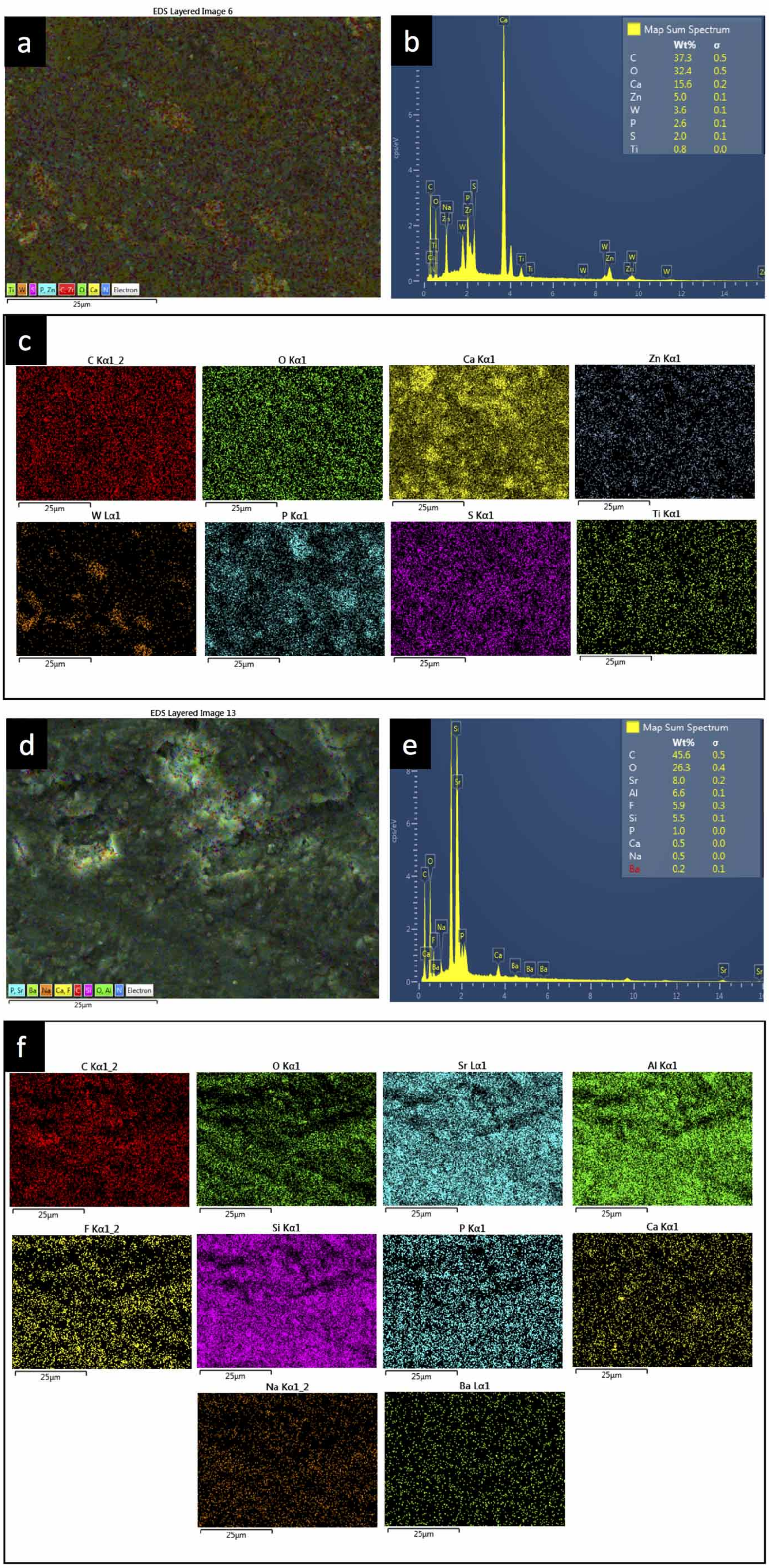

Fig. 3. Representative SEM elemental distribution maps at 1000 x magnification (a, d) with EDX bulk analysis (b, e) and elemental distribution maps of radiopaque elements (c, f) of Dycal (a–c) and GC Fuji IX (d–f).

presence of zirconium as a minor component (1.8 wt.%) of BiodentineTM was demonstrated, which has an atomic number of 40. Interestingly, in contrast with other elemental constituents of the cement, zirconium has an uneven distribution, which is probably the result of zirconium oxide particles present in the BiodentineTM powder. Zirconium oxide particles have been detected in set BiodentineTM, and it has been suggested that these particles do not take part in the setting reaction of the cement (Camilleri *et al*., 2013). Previous studies have shown that BiodentineTM powder contains 5.1 wt.% of zirconium oxide (Camilleri *et al*., 2013). However, higher incorporations of zirconium oxide (30 %) have been shown to increase the radiopacity values to more than 6 mm Al, maintaining adequate physical properties (Cutajar *et al*.).

In the present study, oxygen, carbon, calcium, silicon, zirconium and chlorine were detected as constituent elements of set BiodentineTM. This elemental composition correlates well with the components of the cement reported by the manufacturer, with powder composed of tricalcium and dicalcium silicate, calcium carbonate and oxide and zirconium oxide, and liquid composed of calcium chloride and hydrosoluble polymer (Septodont). Camilleri *et al*. (2013) have previously described the presence of these elements with SEM/ EDX analysis, except carbon and chlorine. However, the same study described the presence of calcium carbonate particles in the set cement, engulfed in the calcium silicate hydrate (Camilleri *et al*., 2013). Chlorine has been added in the form of calcium chloride to the liquid to accelerate the reaction (Septodont).

TheraCal presented an equivalent radiopacity of 2.17 +/- 0.17 mm of Al, which was lower than the radiopacity of BiodentineTM. To our knowledge, and probably due to the novelty of this cement, only Gandolfi *et al*., have previously evaluated its radiopacity, reporting an equivalent radiopacity of 1.07 mm Al (Gandolfi *et al*., 2012). Similar to BiodentineTM, TheraCal is not covered by the scope of ISO 9917, part 2 for resin-modified cements nor 6876:2002. TheraCal is indicated as a material for direct and indirect pulp capping. The manufacturer suggests the application of the material in layers of maximum 1 mm thickness, which for pulp capping procedures should be just sufficient to seal the exposure of the pulp (Bisco, 2014). Consequently, it is relevant that the material is sufficiently radiopaque to be distinguishable, even when the material is used as a thin layer.

According to the TheraCal's patent, the radiopaque material incorporated in the cement could be ytterbium fluoride, barium sulfate or bismuth oxide (Suh *et al*.). In this study, strontium (3 wt.%), barium (1.7 wt.%) and zirconium (0.4 wt.%) were detected in the TheraCal, which have high atomic numbers (38 and 56 respectively). The addition of barium sulfate and strontium zirconate to calcium silicate cements, as radiopacifiers, has been tested previously (Camilleri & Gandolfi; Xuereb *et al*., 2016). Cement replaced by 25–30 % barium sulfate showed radiopacity values greater than 3 mm (Camilleri & Gandolfi). However, the leaching of barium and strontium in calcium silicate-based cements has been reported (Xuereb *et al*.). Therefore, it would be interesting to assess if the leaching also occurs in a resin-modified calcium silicate, such as TheraCal.

According to EDX analysis, in addition to the radiopaque elements, TheraCal presents carbon, oxygen, silicon, calcium and aluminium. Other studies have reported a similar composition (Camilleri, 2014). In agreement with other studies (Camilleri; Demirkaya *et al*., 2016), the presence of aluminum in the cement was also demonstrated. Aluminum has been associated with several adverse health effects, including neurotoxicity, genotoxicity, Alzheimer´s disease, dementia, hyperactivity, and learning disorders in children. However, no significant increase of Al plasma levels in the liver of rats was seen when TheraCal was assessed after being implanted into a tooth socket, which is in contrast with other calcium silicate cements, such as MTA, which showed increased plasma Al levels (Demirkaya *et al*.).

Dycal and GC Fuji IX were included in this study as reference materials; both presented radiopacity values higher than 3 mm Al. Dycal is a self-setting calcium hydroxide-based cement used for pulp capping treatments (Desai & Chandler). The presence of tungsten was detected, which possess a high atomic number of 74. GC Fuji IX is a glass ionomer cement used as dentine replacement; strontium and barium as radiopacifiers were detected in its composition.

CONCLUSIONS

BiodentineTM contains zirconium as a radiopacifying element and has higher radiopacity values than TheraCal, which contains barium and strontium as radiopacifiers.

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RESUMEN: El objetivo de este estudio fue evaluar la composición química y la radiopacidad de nuevos cementos en base a silicato de calcio. Discos de 10 mm x 1 ± 0,1 mm fueron preparados con BiodentineTM, TheraCal, Dycal y GC Fuji IX (n=5). Las muestras fueron radiografiadas directamente en una película PSP oclusal adyacente a una cuña escalonada de aluminio. La radiopacidad de cada espécimen fue determinada de acuerdo a la norma ISO 9917/2007. Se realizaron los análisis estadísticos con las pruebas ANOVA y test de Tukey con un nivel de significancia de 5 %. La constitución química de los materiales fue determinada con microscopía electrónica de barrido y con mapeo por análisis con dispersión de energía de rayos X. La radiopacidad de los materiales en orden decreciente fue: GC Fuji IX (3,45 ± 0,16 mm), Dycal (3,18 ± 0,7 mm), BiodentineTM (2,79 ± 0,22 mm), y TheraCal (2,17 ± 0,17 mm). TheraCal mostró la menor radiopacidad comparada con los otros materiales, seguido de BiodentineTM. Los valores de radiopacidad de Dycal y GC Fuji IX no presentaron diferencias estadísticas significativas. Los análisis de microscopía electrónica de barrido y mapeo por análisis con dispersión de energía de rayos X revelaron la presencia de zirconio en BiodentineTM; y de estroncio, bario y zirconio en TheraCal, como elementos radiopacos. Los nuevos cementos en base a silicato de calcio presentan una composición química distintiva. BiodentineTM contienen zirconio como elemento que provee radiopacidad y tiene mayor valor de radiopacidad que TheraCal, el cual contiene bario y estroncio como agente radiopaco

PALABRAS CLAVE: materiales dentales, cementos de silicato, análisis químico, propiedades físicas.

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