

Radiopacity of Various Dental Biomaterials

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Abstract: This study aims to assess the radiopacity of various dental biomaterials such as pulp-cappings (Life/Dycal), ZOE bases (Zonalin KemDent/Kimia), root canal sealers (AH26/ZOE), gutta-perchas (Gapadent/Spident/Meta Biomed/Suisse) and MTAs (ProRoot/Root/Angelus) along with Calcium-Enriched Mixture (CEM) cement. Five circular specimens (1 mm in thickness/5 mm in diameter) were prepared from each material; five teeth were sliced horizontally with 1 mm thickness as well. Radiographic images of specimens were taken and recorded using digital radiography along with the graduated aluminum step-wedge. The x-ray unit was set at 65 kVp, 8 mA, 18.5 pulse sec⁻¹ and 0.12 sec exposure time and focus-film distance of 32 cm. Radiographs were imported to Digora software to compare radiographic density of the materials, dentin and aluminum step-wedge thicknesses. Data were exported to SPSS to be analyzed using ANOVA and Tukey test. The radiopacity of dentin was measured 1.027 mmAl. Life and Dycal showed the radiopacity of 2.906 and 2.254 mmAl, respectively (p<0.05). KemDent demonstrated higher radiopacity than Kimia (7.009 vs. 8.030 mmAl) (p<0.05). ZOE and AH26 as sealers exhibited the radiopacity of 5.891 and 8.612 mmAl, respectively (p<0.05). Gutta-perchas (Gapadent<Spident<Meta Biomed<Suisse) showed a radiopacity from 7.051-8.051 mmAl. Radiopacity of MTAs could be ranked as follows: [Angelus = 5.589 mmAl, Root = 5.519 mmAl (p>0.05)] and ProRoot = 5.009 mmAl (p<0.05). CEM cement demonstrated the radiopacity of 2.227 mmAl. All biomaterials tested had radiopacity value of at least twice of the dentin. All brands of gutta-percha/sealers met and even exceeded the minimum radiopacity of ISO No. 9877/9876.

Key words: Calcium enriched mixture, endodontic biomaterial, mineral trioxide aggregate, radiology, radiopacity, step-wedge

INTRODUCTION

Dental/endodontic materials such as root fillings, root-end fillings, root canal sealers, pulp cappings and liner/base materials should have adequate radiopacity to be easily distinguished by ISO 4049 (1988), ISO 6876 (2001), ISO 6877 (2006) and Gu and Rasimick (2006). In 1979, presented a standard measure to compare the radiopacity of different materials called aluminum step-wedge. In this system, the visual radiographic density of the material is measured and mentioned as the equivalent thickness of aluminum (mmAl) (Eliasson and Haasken, 1979). Assessment of dental materials in a clinical situation may become challenging due to both the thickness of adjacent teeth and osseous structures as well as mass of the material (Baksi Akdeniz and Eyuboglu, 2007).

Gutta-percha is the most commonly used material for root canal obturation (Aminzadeh *et al.*, 2006). It contains zinc oxide and barium sulfate which contribute to its radiopacity (Katz and Kaffe, 1990). According to the

International Organization for Standardization (ISO) radiopacity of obturation cones should be >3 mmAl (ISO 6877, 2006). Reported the average radiopacity of gutta-percha points around 7.4 mmAl (Katz and Kaffe, 1990).

As stated in the ISO 6876 (2001), radiopacity of the endodontic sealers should also be ≥3 mmAl. According to a more radiopaque sealer than gutta-percha cone seems better in order to increase the overall radiopacity of the root canal filling (Baksi Akdeniz and Eyuboglu, 2007). However, an excessively opaque sealer may mask defects in the gutta-percha filling (Gambarini and Testarelli, 2006). Calcium Hydroxide (CH) is the traditional pulp capping agent. By producing alkaline pH, CH affects remineralization of surrounding dentin when placed very close to the dental pulp. Ideally, pulp-capping materials should present adequate radiopacity to be differentiated from:

$$\text{mmAl equivalent} = \frac{\text{Radiopacity of sample} \times \text{Closest Al thickness of step-wedge}}{\text{Radiopacity of Al step-wedge}}$$

the adjacent dentin (Tam and Pulver, 1989). However, CH is used in a small quantity which makes the radiographic differentiation somehow difficult.

Zinc Oxide Eugenol (ZOE) is a common constituent of the cavity base, temporary fillings, liners and sealers. ZOE consists of grains of zinc oxide embedded in a zinc eugenolate matrix. These components are held together by van der Waals forces and particle interlocking so the material is mechanically weak. On the other hand, a large number of studies emphasize the advantageous biological properties of ZOE (Markowitz *et al.*, 1992). In order to distinguish caries adjacent to the restorations and the dental structures, this material also should be sufficiently radiopaque. Mineral Trioxide Aggregate (MTA) is a water-based endodontic material which due to excellent biological properties, low solubility and marginal adaptation has been widely used by clinicians for the past decade (Parirokh and Torabinejad, 2010a, b; Torabinejad and Parirokh, 2010). MTA is a type I Portland cement radiopacified by bismuth oxide (Asgary *et al.*, 2009a).

It is produced as either a white or grey prototype which are chemically different (Asgary and Parirokh, 2005). Studies report that PC and MTA have similar constituent elements except for the bismuth oxide in MTA (Estrela and Bammann, 2000; Funteas and Wallace, 2003; Asgary and Parirokh, 2004). Grey MTA is reported to have a radiopacity of 5.34 mmAl (Danesh and Dammaschke, 2006). The prototype used by

Torabinejad *et al.* (1995) has a radiopacity of 7.17 mmAl. MTA has a lower radiopacity compared to Super-EBA, IRM, gutta-percha and Amalgam (Danesh and Dammaschke, 2006).

A novel endodontic biomaterial, Calcium Enriched Mixture (CEM) cement has recently been introduced with satisfactory clinical properties; the predominant elements in the CEM are calcium, sulfur, phosphorus and silicon (Asgary *et al.*, 2008). Decreased setting time, higher flow ability, higher antimicrobial effect, less film thickness compared to MTA and ability to form hydroxyl apatite crystals with endogenous ionic origin are the advantages of this cement (Asgary *et al.*, 2008, 2009b). Different studies have compared the radiopacity of various endodontic materials.

However to the best of the knowledge, CEM radiopacity has not been considered in previous studies. Thus, this *in vitro* study was designed to compare the radiopacity of different endodontic (bio) materials including the novel CEM cement.

MATERIALS AND METHODS

Preparation of moulds: Flat Perspex blocks, approximately 33 cm in length, 15 cm in width and 1 mm thick were used to form the moulds. Five holes, 5 mm in diameter each were drilled in each block as shown in Fig. 1.

Preparation of specimens: Dental/endodontic materials as shown in Table 1 were evaluated in terms of radiopacity

Table 1: Tested materials and their composition

Products	Manufacture	Components
Gutta-percha	Spident Co., Ltd, Korea Meta Biomed Co., Ltd, Korea Gapadent Co., Ltd, China Dentaires S.A Vevey (Suisse)	Gutta-percha Zinc oxide Barium sulfate Coloring agent
AH 26	Dentsply, DeTray, GmbH, Konstanz, Germany	Powder: 43% bismuth oxide, 37% calcium hydroxide, 14% hexamethyleneteramine and 5% Titanium dioxide Paste B: Bisphenol epoxy resin
ZOE sealer Life	BP, Kemdent Works, Wiltshire, UK Kerr Corporation, Italy	Powder: zinc oxide 99.99%, Liquid: eugenol Base: Calcium hydroxide, zinc oxide, butyl benzene sulfonamide catalyst: barium sulfate, titanium dioxide, methyl salicylate
Dycal	Dentsply, Espana y Portugal Avda de Burgos, Madrid, Spain	Base paste: 1,3 Butylene glycol disalicylate, zinc oxide, calcium phosphate, calcium tungstate, iron oxide pigment. Catalyst paste: calcium hydroxide, N-ethyl- o/p_toluene sulfonamide, zinc oxide, titanium dioxide, zinc stearate, iron oxide pigments (dentine shade only)
Zonalin Kimia 28 g 10 mL ⁻¹	KimiaDent, Tehran, Iran	Zinc oxide: 99.35%, Zinc acetate: 0.65%, eugenol: 99%
Zonalin Kem-dent 25 g 12 mL ⁻¹	Associated Dental Products Ltd, purton, swindon	Zinc acetate 0.2% ww Zinc oxide BP 99.8% ww
ProRoot MTA Root MTA	Dentsply, Tulsa Dental, OK, USA Lotfi and Saghiri Research group, Iran	Portland cement (75%), bismuth oxide (20%) and gypsum (5%) Calcium silicate, calcium aluminate, calcium aluminoferrite, Bismuth Oxide, calcium sulfate
MTA Angelus	Angelus Industria De Produtos Odontologicos, Ltda., Londrina, PR, Brazil	Tricalcium silicate, dicalcium silicate, tetracalcium aluminoferrite, tricalcium aluminate, bismuth oxide (80% portland cement, 20% bismuth oxide)
CEM cement	BoniquDent, Tehran, Iran	Calcium silicate, calcium phosphate, calcium oxide, calcium salts, barium sulfate and zirconia

along with dentin. Five samples of each material were prepared using the mould described before. All materials were handled according to the manufacturers' instruction. The materials were mixed and used to fill to a slight excess, the mould which was resting on a Mylar strip supported by a glass plate (a glass microscope slide). Another Mylar strip was placed on top of the mould, followed by another glass plate which facilitated manual compression thus ensuring that excess material was displaced and the specimen had perfectly flat surface. Gutta-percha cones were warmed in 60°C water and were adapted into the holes. Excess gutta-percha was removed from the opening of the holes with a hot scalpel. Specimens with voids, bubbles or cracks were discarded and replaced. Each mould was incubated at 37°C. Moreover, five freshly extracted human molar teeth were cut horizontally into 5 disks (1 mm thickness) using a microtome (Jung, Heidelberg, Germany). The disks were kept in distilled water during the study period.

The images of the specimens along with the aluminum step-wedge (made out of 99.5% pure aluminum from 1-8 mm in uniform steps of 1 mm each) were taken using Suni digital radiographic system sensors (Suni Medical Imaging Inc, San Jose, CA, USA) and a dental x-ray unit (Castellini, Bologna, Italy) operating at 65 kVp, 8 mA and 0.12 sec exposure time and focal length of 32 cm.

Digitized images were imported to Digora for Windows software version 2.7 (Orion Corporation Soredex, Helsinki, Finland) where measure density tool was applied to identify equal-density areas in the radiographic images as shown in Fig. 2. This process allowed comparison between the radiographic density of the materials and the radiopacity of the different aluminum step-wedge thicknesses. Each specimen and each step measured for 10 times. The final density value of each material was analyzed by calculating the mean of fifty measurements (ten measurements x five specimen). The following equation was used to convert the values into mmAl:

$$\text{mmAl equivalent} = \frac{\text{Radiopacity of sample} \times \text{Closest Al thickness of step - wedge}}{\text{Radiopacity of Al step - wedge}}$$

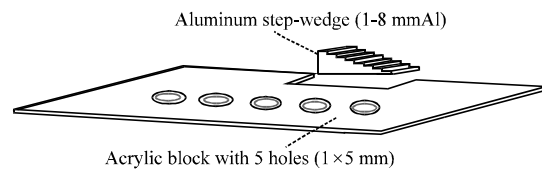


Fig. 1: Aluminum step-wedge and mould with five holes for the filling materials

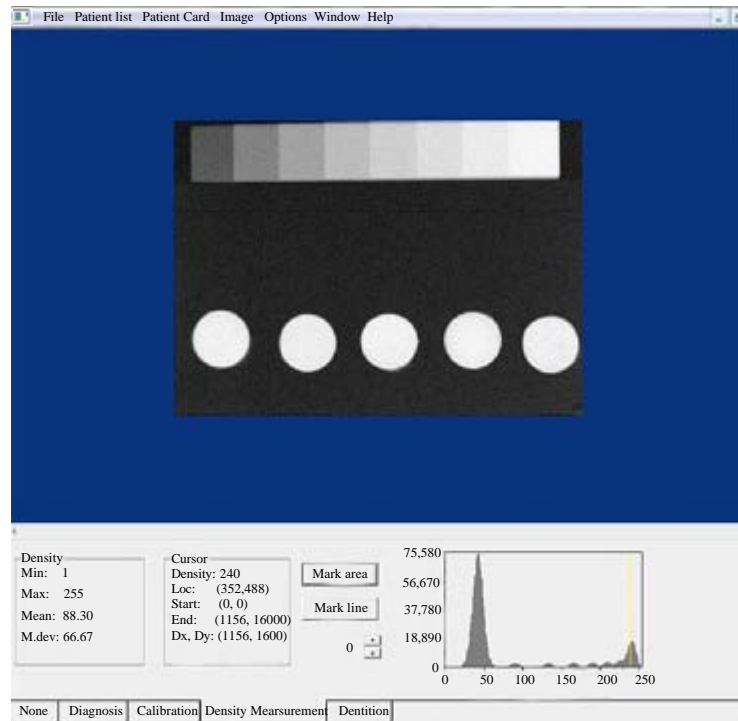


Fig. 2: Computer screen shows the radiopacity measurement by Digora software

This equation established the equivalence of each material's radiopacity to a particular thickness of the aluminum step-wedge, expressed in mm.

Statistical analysis was performed using SPSS (the Statistical Package for Social Sciences). Shapiro-Wilk test was used to assess normality of the distribution of the continuous data. The differences among materials were evaluated using one-way ANOVA. For multiple comparisons Tukey HSD method was used. $p < 0.05$ were considered as statistically significant.

RESULTS AND DISCUSSION

The mean radiographic density values of all endodontic materials used in this study along with dentin, in mmAl are shown in Table 2. Dentin showed the lowest mean value of radiopacity (1.027 mmAl). All gutta-perchas and sealers showed the radiopacity of >3 mmAl which is the minimum recommended by ISO standards 9877/9876 ($p < 0.05$). Pulp-capping materials, ZOE bases, MTAs and CEM cement all demonstrated significantly more radiopacity than dentin ($p < 0.05$) (Fig. 3). According to Tukey test, there was statistically significant difference among specific experimental groups showed in Table 2 with English alphabet letters. Graph of radiopacity density versus step-height in step-wedge is plotted in Fig. 4.

In this study, digital radiography in conjunction with Digora software were used to evaluate the radiopacity of various dental materials. This method was different in comparison with some previous studies (Beyer-Olsen and Orstavik, 1981; Gorduysus and Avcu, 2009) which used conventional radiography. Total degree of darkening of an exposed film is referred to as radiographic density. However in conventional radiography an unexposed film also shows some density owing to the base and added tint as well as development of unexposed silver halide

crystals. This is called gross fog which has an optical density of 0.2-0.3 (White and Pharoah, 2004). It clearly shows a significant impact on the overall radiopacity of the materials shown on the film. Furthermore, conventional methods for analyzing the radiopacity of the materials in relation to the optical density needs more

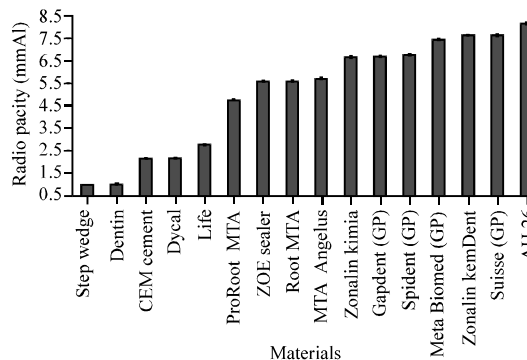


Fig. 3: Radiopacity of materials in comparison with dentin (1 mmAl)

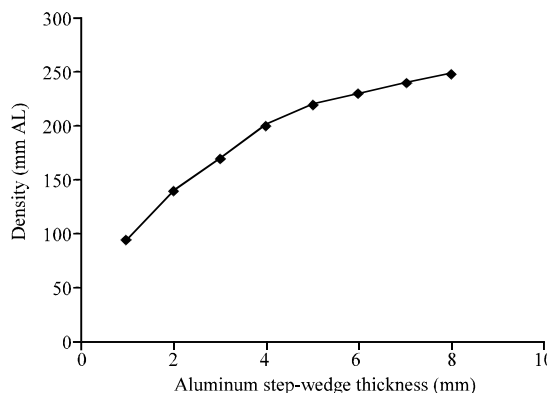


Fig. 4: Calibration of aluminum step-wedge

Table 2: One way ANOVA test results

Materials	Types	Mean±SE	95% Confidence Interval			
			Lower bound	Upper bound	Min.	Max.
Dentin		1.020±0.002 ^a	1.020	1.034	1.023	1.037
Gutta-perchas	Gapadent	7.051±0.021 ^b	6.991	7.110	7.000	7.130
	Spident	7.095±0.023 ^b	7.028	7.161	7.002	7.137
	Meta Biomed	7.844±0.032 ^c	7.753	7.935	7.726	7.916
	Suisse	8.051±0.050 ^d	7.911	8.191	7.856	8.127
Root canal sealers	ZOE	5.891±0.013 ^d	5.854	5.927	5.857	5.928
	AH26	8.612±0.027 ^f	8.536	8.688	8.537	8.691
Pulp cappings	Dycal	2.254±0.009 ^e	2.229	2.280	2.234	2.284
	Life	2.906±0.020 ^b	2.849	2.964	2.838	2.951
ZOE bases	Kimia	7.009±0.048 ^e	6.873	7.145	6.896	7.169
	Kemdent	8.030±0.021 ^d	7.970	8.090	7.964	8.100
Mineral	ProRoot MTA	5.009±0.006 ^f	4.991	5.028	4.993	5.026
Trioxide	Root MTA	5.910±0.033 ^f	5.822	6.007	5.849	6.023
Aggregates	Angelus MTA	5.987±0.027 ^e	5.912	6.063	5.899	6.064
Calcium enriched mixture		2.227±0.023 ^e	2.163	2.292	2.189	2.300

Different letters indicate a statistically significant difference (Tukey-Kramer test; $p < 0.05$)

expensive equipment compared to digital radiography. Considering this and also some other disadvantages of conventional method in point of variability in the quality of the radiographs, loss of information or incorrect differentiation of shades by naked eye this study used digital radiography with Digora software. This software gives the opportunity of direct reading of digital x-ray and comparing the material and step-wedge together. In this study 10 measurements were collected from each specimen in this study instead of 3 or 5 measurements (Bortoluzzi and Guerreiro-Tanomaru, 2009). In addition, the distance from focal point to the object was fixed at 32 cm. It is worth noting that the best results were obtained when a long-cone paralleling technique and a focal length of at least 30 cm were used (Aken, 1969).

Katz and Kaffe (1990) compared the radiopacity of different brands of gutta-percha by using step-wedge. In the same year, Curtis and Von Fraunhofer (1990) applied step-wedge in his study for evaluating the radiodensity of composite resins. In later studies, this method was used by many others with different standards for various goals (Parissis and Iakovidis, 1994; Tanomaru-Filho and Jorge, 2007). Meanwhile, it has been shown that using a step-wedge with an appropriate composition is an important milestone. ISO 6876 (2001) require aluminum of at least 98.0% purity with no >1% iron and 0.1% copper. However, findings showed that alloys with >0.05% copper should be excluded (Watts and McCabe, 1999). This study has followed the findings of Watts and McCabe (1999).

Radiographic density is influenced by thickness and density of the subject. As the thickness of the aluminum step-wedge increases, less photons pass through it to expose the film and the radiopacity increases (Fig. 4). As the step-wedge thickness increases pass a certain point (6 mm here), density shows diminishing returns and the density curve vs. step-wedge aluminum thickness starts to saturate. It means that after the 6th step the observed differentiation in radiopacity may become less noticeable.

Dentin radiopacity used as a control was uniform in all five specimens demonstrated almost 1 mmAl radiopacity confirming previous studies (Beyer-Olsen and Orstavik, 1981; Devito and Ortega, 2004).

Ideal root canal sealers should present some certain physical and chemical properties. Also, one of the most important characteristic should be an acceptable degree of radiopacity to make them clearly visible on radiographs. Sealer's radiopacity has been an important factor in assessing the endodontic obturation (Baksi Akdeniz and Eyuboglu, 2007) but there is a controversy when comparing its radiopacity with accompanies gutta-percha. According to this study and

other findings almost all commercial gutta-perchas met and even exceed the ISO 6877 (2006). Gutta-percha has been considered as the major root-canal filling material. However, sealers should just produce a hermetic seal in the filling. It has been reported that the radiopacity of sealers should exceed the radiopacity of gutta-percha in order to increase the overall radiopacity of the obturation (Baksi Akdeniz and Eyuboglu, 2007). However, sealers with the radiopacity more than that required by the ISO standards may cover the filling defects and lead to misdiagnosis.

Therefore, the radiopacity of the sealers should not be too high and a moderate degree of radiopacity is preferred. In other words, a very high radiopacity, similar to that of AH 26 does not provide the best condition for detecting voids in the fillings. So it is suggested that a maximum limitation for sealer's radiopacity to be determined by ISO.

Numerous studies have been suggested that dental materials including pulp cappings and ZOE bases need to show a radiopacity not less than dentin/enamel (Prevost and Forest, 1990; Shah and Chong, 1996). In this study, the mean radiopacity of pulp-cappings and ZOE bases showed a statistically higher radiopacity than that of dentin. Radiopacity of Life (2.254 mmAl) and Dycal (2.906 mmAl) were significantly higher than that reported by Devito and Ortega (2004). It may be related to the different research method which conducted. Unfortunately, there is not any recommendation by ISO standards for the radiopacity of these materials.

Shah and Chong (1996) claimed that the root-end filling materials with <3 mmAl radiopacity are indistinguishable. However, there is a challenge to achieve this level of radiopacity mainly due to small amounts of the material used. Considering that there is no ISO standard for the radiopacity of retro-filler materials, it is necessary to determine the minimum radiopacity required. In this study, all three kinds of MTA cements, e.g., Root, ProRoot and Angelus showed an acceptable amount of radiopacity >3 mmAl.

Notable difference in the amount of Root MTA and MTA Angelus radiopacity was not detected. Moreover to the best of the knowledge, there is not any investigation about the radiopacity of Novel CEM cement which has been clinically used as root-end filling (Asgary *et al.*, 2010), perforation repair (Samiee *et al.*, 2010), pulpotomy agent (Malekafzali and Shekarchi, 2011; Asgary and Eghbal, 2012; Asgary *et al.*, 2012; Nosrat and Seifi, 2012) and in regenerative endodontics (Nosrat and Seifi, 2011). Thus, the present study was conducted to evaluate radiopacity of this novel cement. Although ISO for retro-fillings is unavailable, the results revealed that the CEM cement has higher radiopacity than dentin. It is

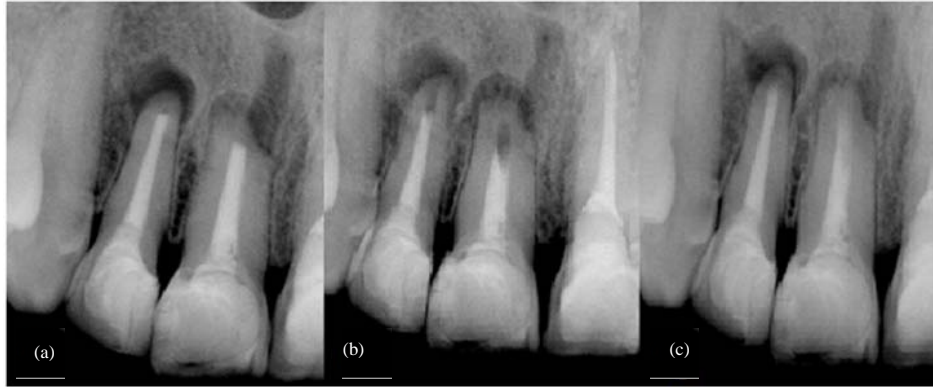


Fig. 5: a) Before periradicular surgery; b) After root-end cavity preparation; c) Immediate after root-end filling with CEM cement

worth noting that although radiopacity of CEM cement is much less than gutta-percha or root canal sealer but it seems that results may vary in clinical cases (Fig. 5) owing to the diameter of the dentin, overlying bone around teeth, the diameter of the cavity preparation (amount of the material) and other possible related issues.

CONCLUSION

Based on the results of this research, all of the sealers and gutta-perchas showed adequate radiopacity according to the ISO 6876/6877. However, it is suggested that this standard should be revised in order to recommend a maximum radiopacity. In addition, ISO for root-end filling materials, pulp-capping and ZOE bases are required. Provided that all of the materials had the radiopacity more than dentin (1 mmAl), their density was considered acceptable.

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