Original Article

Comparison of Radiopacity of Dentin Replacement Materials

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Abstract

Introduction: There are numerous commercially available dentin replacement materials but radiopacity level of these materials is unknown. The aim of this study was to evaluate radiopacity of seven dentin replacement materials in Class I cavities using a digital analysis system. **Methods:** TheraCal LC, Biodentine, Calcimol LC, Ultra-Blend Plus, Equia Forte, Ionoseal, and ApaCal ART were used as dentin replacement materials. Seventy molar teeth were prepared with Class I cavities and then were divided into seven groups. Each material tested was placed on floor of the cavity and then filled by Filtek Z250 composite (3M ESPE). Radiographic images were taken using an indirect digital system. Also, one disc-shaped specimen from each material was examined by energy-assisted X-ray spectroscopy for composition analysis. **Results:** Radiopacity values were significantly different among materials ($p < 0.0001$). Ultra-Blend Plus had the lowest radiopacity values. Calcimol LC, Equia Forte, and Ionoseal had significantly higher radiopacity levels compared to other materials and enamel. All materials demonstrated significantly higher radiopacity than dentin. **Conclusions:** Materials tested had different types and amounts of radiopacifier elements. Dentin replacement materials with lower radiopacity levels can create clinical challenges for diagnostic observations on margins.

Keywords: Dentin replacement materials, Radiopacity, Digital radiography, Pulp capping, X-ray spectroscopy

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Introduction

Protection of dentin-pulp tissues in deep cavities is essential to preserve pulp vitality. Pulp-capping procedure include use of protective material over deep pulp floor or open pulp tissue after caries removal (1). Base or liner is been employed for dentin replacement and pulp capping. These materials supply both an outward dentinal tube seal and a therapeutic effect for pulp against microorganisms, irritants and thermomechanical stimulus(2). The weak bonding of capping materials to dentin, adaptation failures, and polymerization shrinkage of such products may lead to micro-gaps at the dentin bonding interface. This would increase risk of secondary caries as a common cause of restoration failures (3, 4).

Various materials have been used for pulp capping, some of which contain calcium hydroxide–based materials, tricalcium silica–based materials, and glass-ionomer cements. These materials may release calcium, which is useful for pulp structure and supplies a pioneer for apatite construction and supports dentin remineralization. Glass ionomer cement may promote interfacial conservation as it release fluoride and have a buffering capacity (5). Pulp capping materials must have enough radiopacity for accurate diagnosis of secondary caries (6, 7). In addition, sufficient radiopacity allows clinicians to determine defects in margins, gaps between the material and tooth structure and contact with adjacent dental substrates (8). The application of visually non-radiopaque materials can easily go unnoticed, which may result in incorrect diagnosis and treatment planning. Radiopaque components are generally added to dental materials (e.g., composite resins, capping materials, and cements) to supply adequate radiopacity. These added components are composed of elements with higher atomic numbers, such as ytterbium, zirconium, barium, and strontium. An ideal radiopacifier should be nonhazardous and inert to structures, thus providing an adequate visualization of radiographic images without any adverse effects (9, 10).

However, because of commercial protectionism, adequate data concerning chemical components of a dental material are usually either missing or not completely explained by manufacturers.

International organizations have suggested methods for determining radiopacity of composite resins using an aluminum step wedge as a reference. The ISO standard (4049) for radiopacity test requires preparation of cylindrical material samples with a 1-mm thickness. Samples and a highly-purified aluminum wedge with thicknesses from 0.5 mm to 5 mm in increments of 0.5 mm are exposed together to standardized X-ray beam at a determined distance. This standard calls for material to be at least as radiopaque as an equal thickness of aluminum (11). Dentin has shown to have similar radiopacity to an equal thickness of pure aluminum (4, 12). A reasonable alternative method to detect radiolucency along restorations is to prepare a standardized cavity using an extracted tooth. It would allow for a radiopacity assessment of dental materials in clinically relevant areas (13, 14).

A previous study has recommended routine analyses of radiopacity of recent dental materials to confirm they meet minimum requirements (6). There are numerous commercially available pulp-capping materials with various ingredients that provide different levels of radiolucency. Although most manufacturers have stated their materials to be radiopaque, no studies have examined the degree of radiopacity to facilitating caries diagnosis and making changes adjacent to restorations. The aim of this in vitro study was to assess radiopacity of various pulp-capping materials applied over the pulp floor in a standardized cavity.

Materials and Methods

Specimen preparation

Seventy non-carious and unrestored human third molars were collected. The crown dimensions of the teeth measured by a digital caliber were within similar range (0.5 ± 0.01 mm). The roots were embedded on a silicon mold filled with a self-curing acrylic resin up to 2 mm under the cemento-enamel junction. Class I cavities were prepared (4-mm mesio-distal width, 5-mm bucco-lingual width, and 4-mm depth) using a cylindrical diamond bur (836 G 014; Wilofa Diamant, Germany) in a high-speed turbine cooled by air/water. Each diamond bur was changed with a new bur after five teeth were prepped. A digital micrometer (Tchibo Caliper, Hamburg, Germany) was used to verify preparation dimensions. The specimens were divided into seven groups consistent with test materials: TheraCal LC, Biodentine, Calcimol LC, Ultra-Blend Plus, Equia Forte, Ionoseal, and ApaCal ART. The properties of materials used are shown in Table I. Each material was applied over the cavity floor up to a 2-mm thickness, under manufacturer's recommendations. Single Bond Universal adhesive (3M ESPE, Deutschland GmbH, Seefeld, Germany) was applied to all remaining cavity surfaces according to manufacturer instructions and then Filtek Z250 (3M ESPE, St Paul, MN, USA) was used as the restorative material to fill the cavity.

Table I: Compositions and manufacturers of materials used in this study

Materials	Manufacturers	Compositions
Filtek Z250	3M ESPE, St Paul, MN, USA	Bisphenol a polyethylene glycol diether dimethacrylate, bisphenol a diglycıdyl ether dimethacrylate , diurethane dimethacrylate, trıethylene glycol dimethacrylate, zirconia/silica filler
Theracal LC	Bisco, Schaumburg, U.S.A	Bisphenol a diglycidylmethacrylate, portland cement, barium zirconate powder
ApaCal ART	Prevest DenPro, Digiana, Jammu, İndia	Tricalcium phosphate, calcium hydroxide, hydroxyapatite, uretane dimethacrylate, triethylene glycol dimethacrylate, barium zirconate, stabilizers, photoinitiators
Calcimol LC	Voco, Cuxhaven, Germany	Uretanedimethacrylate, calcium hydroxide, 2-dimethylaminoethyl methacrylate
Ultra-Blend plus	Ultradent Products, Cologne, Germany	Calcium hydroxyapatite, calcium hydroxide, triethylene glycol dimethacrylate, diuretane dimethacrylate
Ionoseal	Voco, Cuxhaven, Germany	Bisphenol a diglycidylmethacrylate, fluoro-aluminasilicate glass, 1,6-hexanediylbismethacrylate, uretanedimethacrylate, triethylene glycol dimethacrylate
Biodentine	Septodent, Saint-Maur-des-Fosses Cedex, France	Aqueus calcium chloride solutions, excipients, and tricalcium silicate powder
Equia Forte	GC Corporation, Tokyo, Japan	Polybasic carboxylic acid (liquid), iron oxide (powder)

Radiopacity measurements

The specimens were placed over a phosphor plate on the lingual surface where the lingual surface was on the plate and the buccal side was in the tube side position. Later, radiographic exposure was applied using dental X-ray unit (Acteon Group, X-mind DC, Rome, Italy) set at 70 kV and 8.0 mA. The exposure time was 0.25 seconds and the focal spot to the object distance was 50 cm. Three exposures were implemented for each specimen. Immediately after the exposure, all phosphor plates were scanned using a Vistascan device (Durr Dental, Bietigheim-Bissingen, Germany) and the radiographs were transferred to a personal computer. The radiopacity (in pixels) of specimens were measured by resident software (RadiAnt DICOM Viewer) supplied by the manufacturer. To standardize opacity measurements, three hypothetical vertical lines were described: One line 1 mm apart from inner side of distal and one line 1 mm apart from mesial axial walls of the cavity, and third line is in the center of mesial-distal distance (Figure 1). All measurements were made in 1-mm increments apart from interface on the vertical plane of restorative surfaces and teeth. The mouse cursor was placed under this measurement line on dentin, capping material, or on Filtek Z250 to determine the radiopacity values for each specimen. Radiographic measurements for enamel were taken from the tip of each specimen cusp. Mean values of each group was calculated, and each material group was compared with dentin, enamel, and composite values.

Scanning electron microscope/energy-assisted X-ray spectroscopy system analysis

One disc-shaped specimen (2-mm thickness and 5-mm diameter) from each material was fabricated by a Teflon mold. To ensure conductivity of all samples, specimen surface (Quorum brand, SC-7620 model) was coated with gold thickness of 200 angstroms. Later, a composition analysis and elemental mapping of each sample was performed by an energy-assisted X-ray spectroscopy (EDS) system based on scanning electron microscopy (SEM; Oxford Instruments Brand, Inca X-act model).

Statistical analysis

The radiopacity data confirmed assumptions of parametric analyses. Therefore, statistical analyses were performed using one-way ANOVA and post-hoc Tukey HSD test. All data were evaluated using SPSS 23.0 software (IBM Corp, Chicago, IL). The statistical significance level was set at $p < 0.05$.

Results

The radiopacity means of different materials, dentin, Filtek Z250, and enamel are presented in Table II. The radiopacity values revealed significant differences among tested materials ($p < 0.0001$). Ultra-Blend Plus had the lowest radiopacity values. Calcimol LC, Equia Forte, and Ionoseal had significantly higher radiopacity values compared to other materials, but were not significantly different from each other. Biodentine had a statistically similar radiopacity value to both TheraCal LC ($p = 0.153$) and ApaCal ART ($p = 0.638$). All materials had significantly higher radiopacity levels than dentin ($p < 0.05$). Ionoseal, Calcimol LC, and Equia Forte had higher radiopacity than enamel, while Ultra-Blend Plus had a significantly lower radiopacity than enamel ($p = 0.001$). ApaCal ART, Biodentine, and TheraCal LC had statistically similar radiopacity values to enamel. Filtek Z250 had significantly greater radiopacity than all other materials except Ionoseal. Filtek Z250 opacity was even higher than dentin and enamel. Figure 2 shows radiographic images of cavities filled with tested materials.

Table II: Radiopacity of replacement materials, dentin, composite resin, and enamel Mean (SD)

Groups	Materials	Dentin	Composite resin	Enamel
ApaCal ART	182.86 (5.16)Bb	162.06 (6.07)Aa	200.26 (3.78)Ca	186.20 (5.18)Ba
Calcimol LC	193.66 (3.94)Cd	159.26 (5.44)Aa	199.86 (2.29)Da	184.33 (2.49)Ba
Equia Forte	195.00 (3.33)Cd	161.80 (5.33)Aa	200.53 (3.70)Da	185.33 (5.80)Ba
Ionoseal	196.60 (2.92)Cd	157.13 (5.18)Aa	197.40 (2.77)Ca	184.60 (2.38)Ba
Biodentine	185.26 (3.12)Bbc	160.60 (5.16)Aa	197.33 (3.45)Ca	185.80 (3.50)Ba
TheraCal LC	188.93 (3.36)Bc	160.26 (5.70)Aa	198.60 (2.89)Ca	186.06 (2.89)Ba
Ultra-Blend Plus	177.93 (5.04)Ba	158.86 (6.33)Aa	199.60 (5.22)Da	185.61 (4.33)Ca

Different uppercase letters represent significant difference in horizontal line. Different lowercase letters represent significant difference in vertical line (One-way ANOVA and Tukey HSD tests; $P < 0.05$).

Table III shows elemental analyses on materials surface. Figure 3 shows EDS layered images of each material in backscatter mode. All materials had different compositions with various sized particles and radiopacifiers within the matrix. ApaCal ART, Calcimol LC, Ionoseal, and Ultra-Blend Plus included barium, while Filtek Z250 and Biodentine contained zirconium as a radiopacifier. Equia Forte contained aluminum, silicon, flour, and strontium as a radiopacifier. Cracks were observed on the surface of glass ionomer (Equia Forte), which probably were due to drying effect of SEM machine vacuum.

Table III: Mean (SD) of atomic percentage (wt. %) of elements on material surfaces

	ApaCal ART	Calcimol LC	Equia Forte	Ionoseal	Biodentine	TheraCal LC	Ultra-Blend Plus	Filtek Z250
C	42.1 (0.7)	53.5 (0.7)	37.3 (0.9)	41.0 (0.6)	13.9 (0.6)	31.0 (0.8)	53.1 (0.6)	33.2 (0.6)
O	33.5 (0.6)	27.0 (0.5)	29.7 (0.1)	28.2 (0.5)	45.5 (0.8)	39.5 (0.7)	29.3 (0.6)	34.6 (0.8)
Ca	3.6 (0.2)	0.8 (0.0)	0.3 (0.0)	2.6 (0.1)	31.4 (0.5)	13.4 (0.2)	6.4 (0.1)	0.5 (0.0)
P	1.2 (0.1)	_	1.2 (0.1)	0.5 (0.1)	_	_	2.8 (0.1)	_
F	_	_	6.8 (0.3)	_	_	_	_	_
CI	_	_		_	1.3 (0.1)	_	_	_
Al	1.6 (0.2)	0.9 (0.0)	7.5 (0.1)	3.9 (0.1)	_	2.3 (0.1)	_	0.2 (0.0)
Ba	6.3 (0.2)	0.8 (0.0)	_	12.2(0.2)	_	2.6 (0.2)	6.2 (0.2)	_
Si	6.9 (0.1)	7.1 (0.1)	6.3 (0.1)	6.1 (0.1)	6.0 (0.1)	10.9 (0.2)	_	19.6 (0.3)
Zr	_	_	_	_	1.4 (0.2)	_	_	11.6 (0.3)
Sr	_	_	6.0 (0.2)		_	_	_	_

Discussion

The relative radiopacity of a restorative material is essential for identifying secondary caries and defects, and distinguishing the restorative margin from tooth structure. Insufficient radiopacity of a restorative material may lead to visual challenges in providing a satisfactory restoration in clinics. The techniques used for radiopacity analyses may involve using an aluminum step wedge and a tooth specimen along with a test material located over a radiographic sensor. In the current study, tooth selection and cavity preparation were standardized considering that recognition of radiopacity around restorations also relies on thickness and density of remaining tooth structure. A crucial issue in radiopacity evaluation technique used in this study was systematic analysis of the material placed inside prepared tooth, which allowed clinicians to investigate material radiopacity that was clinically vital for restorations closer to pulp (13).

In the present study, radiopacity values showed significant differences among materials. Radiopacity of resin-based dental materials depends on percentage and type of fillers (15). Filtek Z250 showed significantly higher radiopacity than other materials, except Ionoseal. This can be explained by higher weight of silica (silicon dioxide) and zirconia fillers. Inorganic fillers in the resin matrix caused a higher attenuation of X-rays. High atomic number elements such as zirconium and barium,

and their high concentration result in more radiopacity in images (10). On the other hand, Ionoseal showed similar radiopacity values to Filtek Z250; however, Ionoseal contains a lower proportion of fillers. This might be attributed to have higher proportion of barium (wt% = 12.2). Although Ionoseal, a resin-modified glass cement, has fluoroaluminosilicate glass particles fluorine element could not be detected in its composition by EDS analysis. This event could be interpreted because of lower amount of fluorine. Ionoseal, Calcimol LC, and Equia Forte had statistically similar radiopacity values despite their different compositions. This may be associated with different proportions and types of radiopacifying agents and fillers (16). The radiopacity values of Apacal ART, Biodentine, and TheraCal LC were significantly higher than that of Ultra-Blend Plus. Ultra-Blend Plus has barium in similar proportion (wt% = ±6.2) to that of Apacal ART, but higher than that of TheraCal LC (wt% = 2.6); however, it does not contain silica particles (silicon dioxide). This may explain why it had the lowest radiopacity value. Moreover, it can be difficult to distinguish Ultra-Blend Plus from dentin in certain areas (Figure 2). Biodentine contains tricalcium silicate powders and zirconium oxide as radiopacifiers. TheraCal LC is also a methacrylate-based resin incorporated with tricalcium silicate and barium zirconate as a radiopacifier (17). Farrugia et al. (18) reported that Biodentine has a significantly higher radiopacity than TheraCal LC, which conflicts our findings. This difference could be explained by different methodologies used in evaluations. Thickness of restorative material, remaining tooth structure, and X-ray angulation can affect radiopacity (19).

The optimal radiopacity of a dental material is still debatable. Some authors have reported that an ideal radiopacity of a resin composite should be close to that of dentin for secondary caries detection and that the material with a higher radiopacity presents the worst accuracy for caries diagnoses (4). Others have determined that radiopacity of resin composites should be higher than that of enamel to accurately discern the margin between restoration and tooth (12), or equal to that of enamel to detect recurrent caries (20). In the present study, all materials had higher radiopacity values compared to dentin. However, three (Ionoseal, Equia Forte, and Calcimol LC) of seven pulp-capping materials evaluated presented significantly higher radiopacity levels than enamel. Materials with lower radiopacity than enamel are not suitable in deep areas of cavities when these materials are used as an initial increment because of difficulty to detect marginal integrity or overhang between tooth structure and restorative material (21). Also, a previous study has suggested using materials with similar radiopacity to make radiographic examinations easier when cavities are restored with different materials (22). In the current study, pulp-capping materials, except for Ionoseal, were less radiopaque than conventional resin composite. Consequently, they attributed to a diagnostic challenge on radiographs when used under restorative materials with a minimal thickness of 0.5 to 1 mm.

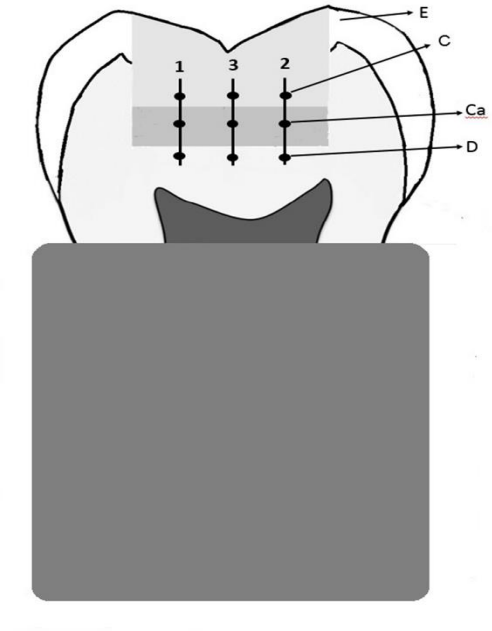

Figure 1: Location of the radiographic measurements (1, 2, 3), radiodensity of capping material (Ca), radiodensity of dentin (D), radiodensity of composite resin (C), radiodensity of enamel (E).

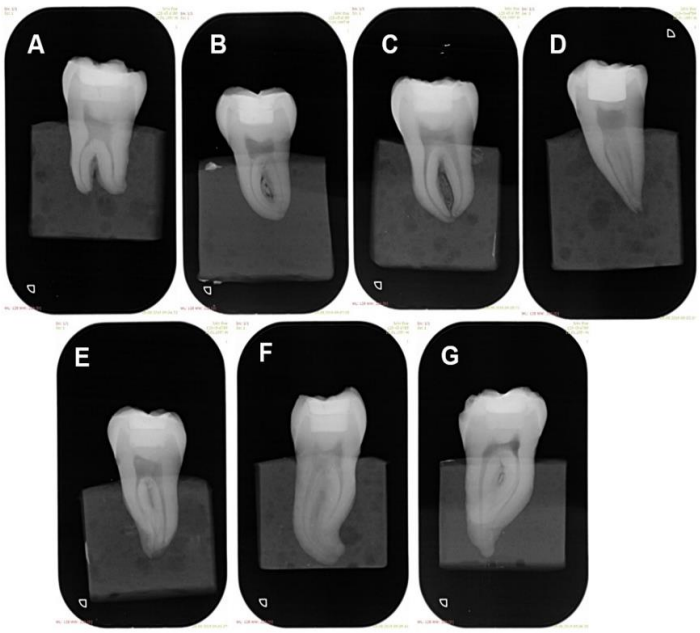

Figure 2: Radiograph images of teeth. A) ApaCal ART, B) Calcimol LC, C) Equia Forte, D) Ionoseal, E) Biodentine, F) TheraCal LC, G) Ultra-Blend Plus

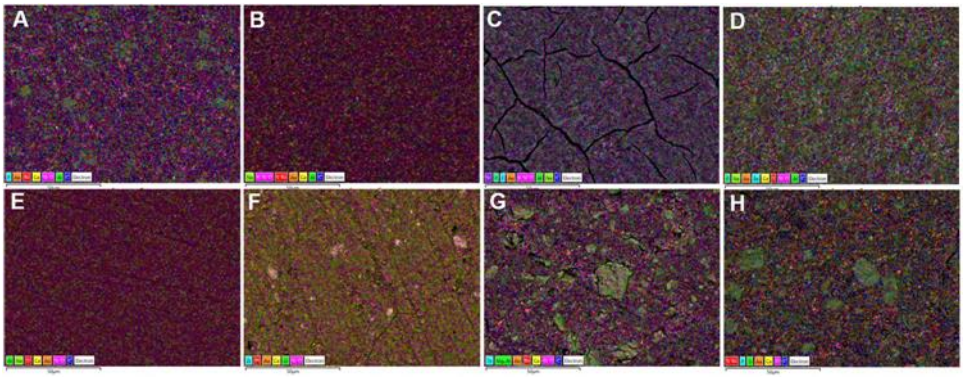

Figure 3: EDS layered images of materials in backscatter mode. A) ApaCal ART, B) Calcimol LC, C) Equia Forte, D) Ionoseal, E) Filtek Z250, F) Biodentine, G) TheraCal LC, H) Ultra-Blend Plus

Conclusions

The radiopacity of materials used in this study were considerably different. All materials showed significantly higher radiopacity than dentin. Ionoseal, Calcimol LC, and Equia Forte had higher radiopacity values than did enamel. We also determined that materials that were used have different types and amounts of radiopacifier elements (barium, zirconium, and strontium).

Conflict of interest

The authors declare that there are no conflict of interest.

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References

1. Schwendicke F, Brouwer F, Schwendicke A, Paris S. Different materials for direct pulp capping: systematic review and meta-analysis and trial sequential analysis. Clin Oral Investig. 2016;20(6):1121-1132.

2. Arandi NZ. Calcium hydroxide liners: a literature review. Clin Cosmet Investig Dent. 2017;9:67-72.

3. Peliz MI, Duarte S, Jr., Dinelli W. Scanning electron microscope analysis of internal adaptation of materials used for pulp protection under composite resin restorations. J Esthet Restor Dent. 2005;17(2):118-128.

4. Cruz AD, Esteves RG, Poiate IA, Portero PP, Almeida SM. Influence of radiopacity of dental composites on the diagnosis of secondary caries: the correlation between objective and subjective analyses. Oper Dent. 2014;39(1):90-97.

5. Nedeljkovic I, De Munck J, Slomka V, Van Meerbeek B, Teughels W, Van Landuyt KL. Lack of Buffering by Composites Promotes Shift to More Cariogenic Bacteria. J Dent Res. 2016;95(8):875-881.

6. Pedrosa RF, Brasileiro IV, dos Anjos Pontual ML, dos Anjos Pontual A, da Silveira MM. Influence of materials radiopacity in the radiographic diagnosis of secondary caries: evaluation in film and two digital systems. Dentomaxillofac Radiol. 2011;40(6):344-350.

7. Pekkan G, Ozcan M. Radiopacity of different resin-based and conventional luting cements compared to human and bovine teeth. Dent Mater J. 2012;31(1):68-75.

8. Fonseca RB, Branco CA, Soares PV, Correr-Sobrinho L, Haiter-Neto F, Fernandes-Neto AJ, et al. Radiodensity of base, liner and luting dental materials. Clin Oral Investig. 2006;10(2):114-118.

9. Valente LL, Peralta SL, Ogliari FA, Cavalcante LM, Moraes RR. Comparative evaluation of dental resin composites based on micron- and submicron-sized monomodal glass filler particles. Dent Mater. 2013;29(11):1182-1187.

10. Amirouche A, Mouzali M, Watts DC. Radiopacity evaluation of bis-GMA/TEGDMA/opaque mineral

filler dental composites. J Appl Polym Sci. 2007;104(3):1632-1639.

11. ISO-Standards. ISO 4049 Dentistry— Polymer-based Restorative Materials. 2009. Available from: https://www.iso.org/standard/67596.html

12. Hitij T, Fidler A. Radiopacity of dental restorative materials. Clin Oral Investig. 2013;17(4):1167-1177.

13. Soares CJ, Santana FR, Fonseca RB, Martins LR, Neto FH. In vitro analysis of the radiodensity of indirect composites and ceramic inlay systems and its influence on the detection of cement overhangs. Clin Oral Investig. 2007;11(4):331-336.

14. Soares CJ, Rosatto C, Carvalho VF, Bicalho AA, Henriques J, Faria ESAL. Radiopacity and Porosity of Bulk-fill and Conventional Composite Posterior Restorations-Digital X-ray Analysis. Oper Dent. 2017;42(6):616-625.

15. Attar N, Tam LE, McComb D. Flow, strength, stiffness and radiopacity of flowable resin composites. J Can Dent Assoc. 2003;69(8):516-521.

16. Candeiro GT, Correia FC, Duarte MA, Ribeiro-Siqueira DC, Gavini G. Evaluation of radiopacity, pH, release of calcium ions, and flow of a bioceramic root canal sealer. J Endod. 2012;38(6):842-845.

17. Camilleri J. Hydration characteristics of Biodentine and Theracal used as pulp capping materials. Dent Mater. 2014;30(7):709-715.

18. Farrugia C, Lung CYK, Schembri Wismayer P, Arias-Moliz MT, Camilleri J. The Relationship of Surface Characteristics and Antimicrobial Performance of Pulp Capping Materials. J Endod. 2018;44(7):1115-1120.

19. Fonseca RB, Haiter-Neto F, Fernandes-Neto AJ, Barbosa GA, Soares CJ. Radiodensity of enamel and dentin of human, bovine and swine teeth. Arch Oral Biol. 2004;49(11):919-922.

20. Goshima T, Goshima Y. Radiographic detection of recurrent carious lesions associated with composite restorations. Oral Surg Oral Med Oral Pathol. 1990;70(2):236-239.

21. Bouschlicher MR, Cobb DS, Boyer DB. Radiopacity of compomers, flowable and conventional resin composites for posterior restorations. Oper Dent. 1999;24(1):20-25.

22. Akerboom HB, Kreulen CM, van Amerongen WE, Mol A. Radiopacity of posterior composite resins, composite resin luting cements, and glass ionomer lining cements. J Prosthet Dent. 1993;70(4):351-355.

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