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Radiopacity evaluations of the novel calcium-silicate and glass-ionomer-based materials

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ABSTRACT

Aims: Radiopacity is a crucial property for a liner or base material, and these materials should provide an optimal contrast for detecting secondary caries in radiographic examinations. The purpose of this study was to assess the radiopacity characteristics of four calcium-silicate-based and two glass-ionomer-based materials used as a liner or base in direct or indirect vital pulp therapy.

Methods: A total of 60 cylindrical-shaped and 1 mm thick specimens were prepared from a calcium-silicate (Biodentine, Septodont), a calcium-silicate (MTA, Angelus), a light-cured resin-modified calcium silicate (TheraCal LC, Bisco), a dual-cured resin-modified calcium silicate (TheraCal PT, Bisco), a glass hybrid glass-ionomer (Equia Forte HT, GC), and a resin-modified glass ionomer (Glass Liner, Wp Dental) material (n=10). Digital radiographic images of the specimens, a molar tooth section with 1 mm thickness, and an aluminum step wedge were obtained by a digital radiography system (Heliodent Plus, Dentsply Sirona) with 60 kV voltage, 7 mA current, and 0.25 seconds exposure time. The mean gray values (MGV) of digital images were determined using the ImageJ software program (National Institute of Health, Bethesda, MD, USA). Kruskal-Wallis and Mann-Whitney tests ($p<0.05$) were used to analyze the data.

Results: Among the tested materials, the highest radiopacity value was found in MTA, and the lowest radiopacity value was obtained in Glass Liner. The radiopacity levels of the materials studied were MTA>Biodentine>Equia Forte HT>Theracal PT>Theracal LC>Glass Liner, respectively. All the tested liner or base materials exhibited significantly greater radiopacity values when compared to those of dentin ($p<0.05$). MTA has statistically significantly higher, Biodentine, Theracal PT, Theracal LC, and Glass Liner have statistically significantly lower radiopacity values than enamel ($p<0.05$).

Conclusion: All the restorative materials tested exhibited higher radiopacity than dentin, with ThereCal LC and Glass Liner displaying lower radiopacity than enamel, ThereCal PT, Biodentine, and Equia showing equivalent radiopacity to enamel, and MTA demonstrating higher radiopacity than enamel.

Keywords: Radiopacity, dental materials, biocompatible materials, calcium silicate cement, dental radiography

INTRODUCTION

In operative dentistry, the main goal with deep caries teeth is to preserve the pulp vitality. Pulp-capping treatments involve the covering of the pulp with a biocompatible material after the removal of carious tissue, aiming to preserve the pulp vitality in order to prevent bacterial leakage and promote dentin bridge formation.[1,2] Liners and bases have been employed as one of the approaches for pulp capping.

Currently, a diverse selection of pulp capping agents possessing distinct characteristics, benefits, and limitations are accessible in the field of dentistry.[3] Recently, novel biomaterials known as calcium silicate-based materials have been introduced, serving both as sealers and cements. Bioceramics, commonly known as calcium silicate-based cements, have emerged as a substitute for the traditional application of calcium hydroxide.[4,5] In modern dentistry, there is a broad range of calcium silicate-based cements available, which are used for a variety of indications, including direct and indirect pulp capping, regenerative endodontic treatments. These cements are favored for their notable biocompatibility, bioactivity, and biomineralization properties.[6]

Mineral trioxide aggregate (MTA), primarily consisting of calcium oxide, bismuth oxide, and silica, is a hydrophilic cement that offers numerous advantages,

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including biocompatibility, low solubility, prevention of bacterial leakage, and the capacity to release calcium hydroxide. Nevertheless, there are some disadvantages such as difficulty in handling and extended setting time, requiring layering with other restorative materials before the final restoration is completed, which may cause marginal loss of adaptation and leakage.[2,7]

As an alternative to MTA, Biodentine (Septodont, Saint-Maur-des-Fossés, France), a tri-calcium silicate material consisting of zirconium oxide, tricalcium silicate, calcium chloride, calcium carbonate and water, has been introduced to the market. Biodentine is highly recommended as a dentin substitute due to its favorable characteristics, including excellent sealing properties, high compressive strength, a rapid hardening rate (typically within 9-12 minutes), as well as its biocompatibility, bioactivity, and remineralization properties.[8]

TheraCal LC is a novel calcium silicate cement which serves as a liner and base beneath composite restorations. It is light-cured and MTA filled. Its composition consists of around 45% Portland cement, around 10% radiopaque components like bismuth oxide, about 5% hydrophilic thickening agent (fumed silica), and nearly 40% resin content.[8] TheraCal LC, in comparison to MTA, demonstrates a quicker setting time, reduced solubility, and enhanced flowability.[9]

The new resin-based chemical formulation, TheraCal PT (Bisco Inc., Schaumburg, IL, USA), releases calcium because of hydrophilic structure of the matrix. The manufacturer recommends its use as a liner and for indirect/direct pulp capping purposes.[10] It offers advantages such as dual-cure polymerization, with a maximum setting time of 5 minutes. This enables the application of a permanent restorative material in a single session. Additionally, TheraCal PT exhibits properties similar to Angelus MTA and Biodentine on human dental pulp stem cells, setting it apart from TheraCal LC.[11]

Glass ionomers and resin-modified glass ionomers (RMGIs) represents examples of lining materials that can release fluoride and form an ionic bond with the tooth structure.[1] Nowadays, resin-modified glass ionomers are commonly preferred due to their lower solubility, fluoride-releasing capability, excellent bonding properties, and higher resistance compared to traditional glass ionomers.[7]

Radiopacity property is accepted as a crucial characteristic for restorative materials. It refers to the relative resistance of a material to the passage of electromagnetic radiation, such as radio waves and X-ray photons, resulting in a white appearance on a radiograph. This feature enables a clear contrast between the surrounding structures and the restorative material, ensuring proper visualization on X-ray images. Thanks to radiopacity, secondary caries beneath restorations, overhangs in proximal restorations, and open margins, as well as gaps within the restoration, which are some of the primary causes of restoration failure, can be assessed. Furthermore, radiopacity also enables the assessment of restoration integrity at recall appointments, proximity to the pulp chamber, and proximal contacts. There are several factors that influence the radiopacity of a material, including its thickness, composition, type of filler (fillers with lower atomic numbers are more radiolucent compared to those with higher atomic numbers), and weight percentage. Additionally, the presence of opacifying agents such as barium, zirconium, lanthanum, strontium, bismuth oxide, carbonates, and sulfates, also contributes to the radiopacity of the material.[12]

To the best of our knowledge, there is no published radiopacity study comparing Therecal PT to other liners. The purpose of this study was to compare the radiopacity of six different liner materials (four calcium silicate-based cements and two glass ionomer materials). The objective of this research was to evaluate the radiopacity of MTA, Biodentine, ThereCal LC, ThereCal PT, Equia Forte HT and Glass Liner using digital radiography. The null hypothesis under examination was that there would be no statistically significant differences among the radiopacity values of the six tested liner materials.

METHODS

Preperation of Study Samples

The protocol for the study was approved by Bahçeşehir University Clinical Researches Ethics Committee (Decision No: 2023-16/01). All procedures were carried out in accordance with the ethical rules and the principles. The study tested a range of materials, including calcium-silicate (Biodentine, Septodont, Saint-Maur-des-Fossés, France), a calcium-silicate (MTA, Angelus), light-cured resin-modified calcium silicate (TheraCal LC, Bisco), dual-cured resin-modified calcium silicate (TheraCal PT, Bisco), glass hybrid glass-ionomer (Equia Forte HT, GC), and resin-modified glass ionomer (Glass Liner, Wp Dental). Table 1 provides information on the materials examined in the study, their corresponding study codes, and their characteristics. The sample size of the study was determined using G*Power Ver. 3.1 software (Franz Faul, Universität Kiel, Kiel, Germany). The calculations were conducted using an error probability (alpha) of 0.05, an effect size of 0.55, and a power level of 95%.

Table 1. Types, manufacturers, composition and application procedures of the materials used in the study

Material	Manufacturer	Type	Composition	Application step
Angelus MTA	Angelus, Londrina, Brazil	CSC (Calcium silicate cement)	Powder: tricalcium silicate, dicalcium silicate, tricalcium aluminate, silicon oxide, potassium oxide, aluminum oxide, sodium oxide, iron oxide, calcium oxide, bismuth oxide, magnesium oxide, insoluble residues of crystalline silica Liquid: water	Powder + liquid (mixed manually)
Biodentine	Septodont. France	CSC	Powder: tricalcium silicate, dicalcium silicate, calcium oxide, calcium carbonate, zirconium oxide, iron oxide Liquid: calcium chloride, water-soluble polymer, water	0.7 g capsule of powder + 5 drops of liquid (30 s; 4000–4200 rpm) Pour five drops liquid from into the capsule. Place the capsule on a mixing device and mix 30 s.
TheraCal LC	Bisco, Schaumburg, IL, USA	CSC	Light-curing single paste: resin bis-phenyl glycidyl methacrylate (BisGMA) & polyethylene glycol dimethacrylate (PEGD) modified calcium silicate filled with CaO, calcium silicate particles (type III Portland cement), Sr glass, fumed silica, barium sulphate, barium zirconate	Dispensed directly from a flowable syringe (no mixing) Inject the material into the mold in 1 mm increments Light cure each increment for 20 s.
TheraCal PT	Bisco, Schaumburg, IL, USA	CSC	Silicate glass-mix cement (50–75 wt%, Polyethylene Glycol Dimethacrylate (10–30 wt%) Bis-GMA (5–10 wt%), Barium zirconate (1–5 wt%), Ytterbium fluoride (CAS no. 13760-80-0), Initiator.	Dispensed directly from a flowable syringe (mixing) Inject the material into the mold in 1 mm increments Light cure each increment for 20 s.
Glass Liner	WP Dental		Qualitative composition: Glass ceramic, glass ionomer powder, silica, camphorquinone, hexanediol dimethacrylate, Bis-GMA, BHT, DMTBA Quantitative composition: Fillers 65%; activators, accelerators and stabilisers 1%, dimethacrylates 34%	The material, at a thickness of 1 mm, was polymerized using an LED light curing device.
Equia HT	GC Tokyo Japan	Glass Hybrid	Surface-treated FAS glass, highly 1901091 reactive surface-treated fine FAS glass, high-molecular-weight polyacrylic acid, polyacrylic acid	The capsule material was mixed in a capsule mixer for 10 s. Then capsule was placed into the mold with applier as 1mm.

Ten disk-shaped samples (total of 60 samples) were prepared using a circular metal mold with a 10 mm diameter and 1 mm thickness, ensuring a precise thickness of 1±0.1 mm for each sample. A freshly extracted third molar tooth intended for orthodontic use, was used to acquire samples of enamel and dentin. The extracted tooth was thoroughly cleansed, and the roots were excised below the cementoenamel junction. Subsequently, the crown of the tooth was carefully sectioned longitudinally using a slow-speed diamond saw (Isomet, Buehler Ltd., Lake Bluf, IL, USA) with water cooling, resulting in enamel and dentin samples with 1 mm thickness. These enamel and dentin samples were then placed in light-proof containers filled with distilled water and maintained at a temperature of 37°C for a period of 24 hours before the radiographic examination.

Digital Radiography and Radiopacity Calculation

A 12-step aluminum (Al) step wedge, with increments of 0.5-mm thickness, was crafted from a high-purity Al alloy (1050, 99.5% purity) for the purpose of standardizing and calibrating the radiographic images. This step wedge also served to gauge the radiographic density of the samples in terms of millimeters equivalent of aluminum (mm Al).

To conduct the radiographic assessments, the Al step wedge, enamel and dentin samples, and one sample of every restorative material were positioned onto a phosphor plate sized 2+(PSP VistaScan® Sytem, Dürr Dental, Bietigheim-Bissingen, Germany). The plate was then exposed to X-rays using a wall-mounted X-ray device with 2.5 mm aluminum equivalent filtration (Heliodent Plus, Dentsply Sirona, Bensheim, Germany) at a source-to-object distance of 30 cm. The exposure parameters were set at 60 kVp, 7 mA, and an exposure time of 0.25-second in accordance with the manufacturer's instructions. This procedure was repeated twice for every sample within all the groups. Subsequently, the phosphor plates (PSPs) were promptly scanned using a theoretical spatial resolution of 25 line pairs per millimeter (lp/mm) with the VistaScan Mini View system (Dürr Dental, Bietigheim-Bissingen, Germany). All the resulting images were converted in 8-bit TIFF format using the DBSWIN 5.2.0 software (Dürr Dental, Germany).

We employed ImageJ v1.5e, a freely available image analysis software (National Institute of Health, Bethesda, MD, USA) to determine the mean gray values (MGVs) of both the specimens and the stepwedge. ImageJ is a recognized and open-access tool that has been commonly chosen in previous studies to evaluate the radiopacity levels of restorative materials. To perform the measurements, a 10×10-pixel region of interest (ROI) was randomly chosen in five distinct areas on each specimen and on each aluminum step. MGV values were recorded for each ROI, and the average of these values from the five sections was calculated for every specimen in every radiograph (Figure 1).

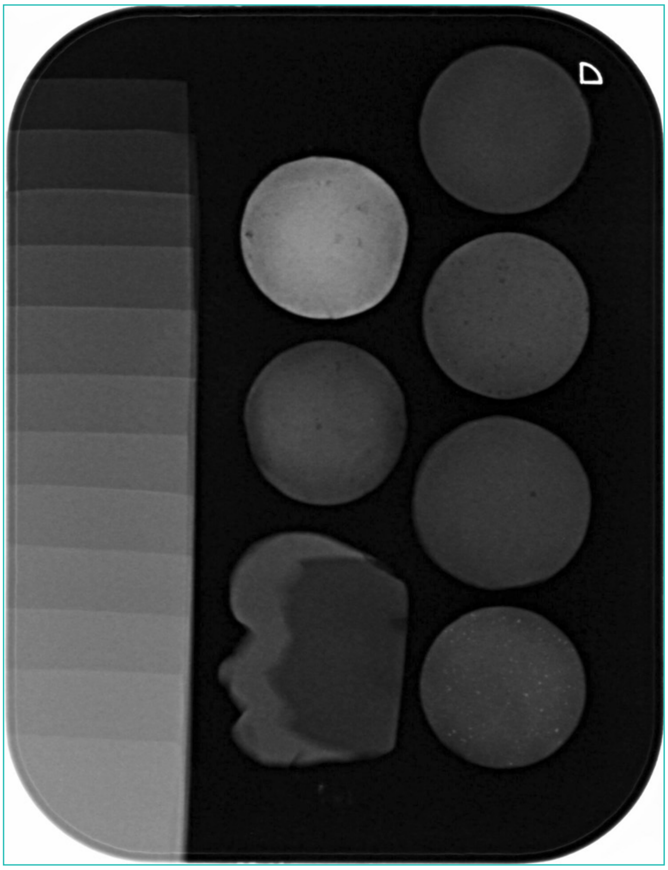

Figure 1. Digital radiographic image of using calcium silicate cement samples, glass ionomer cements, enamel, dentin and aluminum step-wedge

A calibration protocol was established for each radiographic image using the mean gray values (MGVs) derived from the aluminum (Al) stepwedge present in each image. This involved performing linear interpolation on the MGV values associated with aluminum and the corresponding stepwedge thickness. Subsequently, the resultant interpolation function was employed to compute the radiopacity of individual specimens, quantified in millimeters equivalent of aluminum (mm Al).

Statistical analysis

Statistical analyses were performed using SPSS version 25.0 (IBM SPSS Statistics, Chicago, IL, USA). The Shapiro-Wilk test was used to assess the normality of data included in the study. Comparison tests were performed at a significance level (p) of 0.05. Given that the variables did not exhibit normal distribution ($p>0.05$), the analytical approach proceeded with non-parametric test methodologies. Group comparisons were conducted employing the Kruskal-Wallis test. The assessments of intra-group disparities were carried out utilizing the Mann-Whitney test and decisions regarding the outcomes were determined in relation to the Bonferroni-corrected p-value.

RESULTS

The Shapiro-Wilks test showed that radiopacity (in mm Al) values did not follow a normal distribution ($p<0.05$). According to the Kruskal-Wallis test, there were statistically significant differences among the tested materials ($p<0.05$). The means and standard deviations for the mean MGV and radiopacity values of the studied materials, enamel, and dentin are shown in **Figure 2** and **Table 2**. The enamel and dentin radiopacity values were 2.13±0.07 and 1.13±0.06 mm Al, respectively. The radiopacity values of all materials tested were higher than the radiopacity of dentin. Among the tested materials, glass liner had the lowest radiopacity value (1.49±0.23). MTA (5.15±0.69) was significantly more radiopaque than all other calcium silicate-based or glass ionomer materials, as well as enamel ($P<0.05$). Biodentine, Equia Forte HT, and TheraCal PT exhibited radiopacity values similar to that of enamel ($P>0.05$). Biodentine, Theracal PT, and ThereCal LC have a statistically higher radiopacity than Glass Liner ($P<0.05$), but similar to Equia Forte HT ($P>0.05$). Radiopacity of calcium silicate materials ranged from 1.66 to 5.15 mm Al, while glass ionomer materials ranged from 1.49 to 2.28.

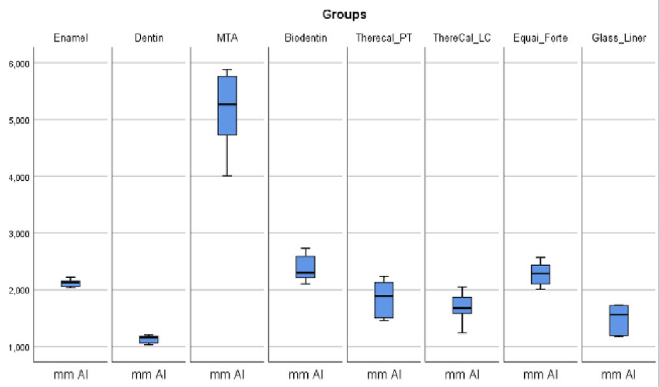

Figure 2. Graphical representation of mmAl radiopacity values of the experimental groups

Table 2. Radiopacity and mean gray value (MGV) of the studied materials, enamel, and dentin (mean±SD)

Material	Mean Radiopacity Value (mm Al)	Mean MGV
MTA	5.15±0.69^{a}	124.84±13.42
Biodentin	2.38±0.24^{bx}	69.99±4.73
Equia_Forte	2.28±0.2 ^{bx}	67.84±2.99
Enamel	2.13±0.07 ^{bcx}	63.87±1.48
Therecal_PT	1.83±0.33^{cd}	56.09±7.92
ThereCal_LC	1.66±0.26 ^{d}	51.58±5.81
Glass liner	1.49±0.23 ^{e}	47.04±4.44
Dentin	1.13±0.06 ^{f}	37.35±1.55

* Means in a column followed by the same letter are not significantly different by the Kruskal-Wallis test at α=0.05.

DISCUSSION

In this study, the radiopacity properties of MTA, Biodentine, TheraCal LC, TheraCal PT, Glass Liner, and Equia HT were compared. The null hypothesis stating that there would be no statistically significant difference in radiopacity value among the six tested materials was evaluated and ultimately rejected.

Despite recent advancements in radiological diagnostic tools and the increasing integration of Cone Beam Computed Tomography (CBCT) into clinical practice, two-dimensional periapical radiographs continue to be the standard method for assessing the condition of teeth and the quality of applied restorations.[13] Suitable radiopacity is crucial for the detectability of restorative materials on a radiograph, allowing them to be readily differentiated from the adjacent anatomical structures.[14] Poorterman et al.[15] emphasized the importance of radiographic examination by noting that less than 15% of insufficient restorations can only be detected through clinical examination.

The presence of secondary caries serves as a primary reason for the replacement of restorations. It is imperative for base and liner materials to possess optimal radiopacity to create contrast with recurring caries, thus facilitating accurate diagnosis. Furthermore, it is expected that the interface between the tooth and the restoration should be radiopaque enough to be distinguished from the tooth structure, enhancing the clarity of diagnostic imaging. It has been reported that materials with moderate radiopacity are more suitable in this regard, and that the optimal radiopacity should be slightly higher than that of enamel.[16,17] In the present study, Biodentine and Equia Forte HT fitted this description. Additionally, MTA showed greater radiopacity compared to enamel, however, there might be another concern with high radiopacity levels. In radiographic evaluations, a high contrast between radiopaque region adjacent to a less radiopaque area may lead an visual illusion called the Mach Band phenomenon and may obstruct the detection of caries on surfaces neighboring the restoration.[16]

In the current study, only TheraCal LC and Glass Liner exhibited radiopacity, which was significantly lower than that of enamel. Having lower radiopacity than enamel may lead to misdiagnosis by clinicians, resulting in the mistaken identification of these materials as dentin, pulp, caries, or gaps.[17] The radiopacity value of TheraCal LC, as found in the current study, was 1.66±0.26, which is lower compared to the radiopacity value reported by Corral et al.[18] (2018) (2.17±0.17 mm Al) and higher compared to those reported by Gandolfi et al.[9] (2012) (1.07±0.17 mm Al). These discrepancies among studies highlight the need for further investigation. In addition, there is a lack of available literature findings on the radiopacity of TheraCal PT. Although these materials were not compared with others in various studies, the fact that they are more radiopaque than dentin but less so than enamel in our study suggests the need for further investigation. This also indicates that radiographic evaluations should be conducted with caution.

In their study, Yaylacı et al.[19] compared the radiopacity of 17 different restorative materials at thicknesses of 1mm, 2 mm, and 4 mm and identified the radiopacity of 1mm Equia Forte HT as 2.10±0.16, which was statistically similar to that of enamel. In accordance with this study, the present study found the radiopacity of the Equia Forte HT group to be 2.28±0.2, which was statistically akin to that of enamel. Furthermore, the presence of fluoroaluminosilicate glass and iron oxide in Equia Forte HT's composition may have contributed to this result.

ISO 4049 establishes pure aluminum as the benchmark for assessing the radiopacity of dental restorative materials, with enamel and dentin recommended as secondary standards.[20] This study, aligning with several in vitro research on the radiopacity of restorative materials, utilized both a pure aluminum step wedge as well as the hard tissues of human teeth as standards for comparison. Williams and Billington have reported that the radiopacity of enamel at 1 mm thickness matches that of aluminum at 2.1 mm thickness, and that dentin has the same radiopacity as aluminum of equivalent thickness.[21] The results of the current research also indicate that the radiopacity of dentin (1.13±0.06) is similar to that of aluminum of the same thickness. In contrast the radiopacity of enamel (2.13±0.07) is approximately twice that of aluminum of the same thickness.

It has been revealed that the minimum radiopacity value recommended for the identification and distinguishing of endodontic sealing materials is equal to 3 mm of aluminum, as required by the ISO 6876/2001 specifications. Additionally, it has been reported that materials with a radiopacity value lower than 3 mm of aluminum will be difficult to distinguish from dentin.[22] In the comparison of calcium silicate cements used in this study, the MTA group, with a radiopacity value of 5.15±0.69, was the only one found to meet the required minimum radiopacity threshold. Furthermore, in alignment with previous studies, the MTA group has demonstrated the highest radiopacity values.[23,24]

The radiopacity of a material is directly linked to the atomic numbers of the elements that constitute it; this represents the quantity of protons in the nucleus of the composing atoms, which defines the electrical charge and thus the force that attaches an electron to its orbit.[13] Given that bismuth has an atomic number of 83, it can be considered more radiopaque in comparison to zirconium and tantalum, with atomic numbers of 40 and 73, respectively.[25] Thus, Angelus MTA, which includes bismuth oxide as a radiopacifying agent, demonstrates enhanced radiopacity due to the high atomic number of bismuth.

In the current study, the radiopacity value of Biodentine was determined to be 2.38±0.24. While Angelus MTA incorporates bismuth as its radiopacifying agent,[24] Biodentine utilizes zirconium oxide, as per the information provided by its manufacturer.[23] This variance in radiopacity between Biodentine and ProRoot MTA can be attributed to their use of distinct radiopacifiers. Kaup et al.[23] reported the radiopacity value of Biodentine as equivalent to 1.5 mm of aluminum, Tanalp et al.[26] found it to be 2.8±0.48 mm of aluminum, and Grech et al.[27] measured Biodentine's radiopacity at 1 and 28 days post-preparation, finding values of 3.3 and 4.1 mm of aluminum, respectively. These findings from literature demonstrate variations from the manufacturer's claim that Biodentine possesses a radiopacity equal to 3.5 mm of aluminum.

The wide range of radiopacity values observed for Biodentine, from 1.5 to 4.1 mm aluminium, may be due to the lack of standardization in the production of the material as previously reported and methodological differences in other studies, such as the film-focusing distance.[23] The storage conditions of the samples may also have played a role.[18] Additionally, the variance could be attributed to the quantity of zirconium oxide in Biodentine, which is reported to be superior in biocompatibility compared to bismuth oxide.[28] Further research is needed to determine the reasons for these discrepancies in radiopacity measurements.

Limitations

One significant limitation of this study is the inability to fully replicate the conditions of the oral environment. The radiopacity of restorative materials may be influenced by various factors present in the oral environment, including oral fluids, soft tissues, and adjacent dental structures. Furthermore, the leaching of ions from radiopacifiers within the material into an aqueous environment could diminish its radiopacity. Further research is needed to evaluate restorative materials under conditions that more closely resemble the oral environment, including studies on the effects of aging.

CONCLUSION

All the restorative materials tested exhibited higher radiopacity than dentin, with ThereCal LC and Glass Liner displaying lower radiopacity than enamel, ThereCal PT, Biodentine, and Equia Forte HT showing equivalent radiopacity to enamel, and MTA demonstrating higher radiopacity than enamel. The radiopacity of materials can be influenced by their structural characteristics as well as their types.

Currently, there are also commercial materials used as lining for restorations that exhibit insufficient radiopacity. As manufacturers continuously reformulate their products to enhance characteristics and reduce costs, there is a growing need for studies to assess the radiopacity of these lining materials.

ETHICAL DECLARATIONS

Ethics Committee Approval

The study was carried out with the permission of Bahçeşehir University Clinical Researches Ethics Committee (Date: 20.09.2023, Decision No: 2023-16/01).

Informed Consent

Because the study was designed retrospectively, no written informed consent form was obtained from patients.

Referee Evaluation Process

Externally peer reviewed.

Conflict of Interest Statement

The authors have no conflicts of interest to declare.

Financial Disclosure

The authors declared that this study has received no financial support.

Author Contributions

All the authors declare that they have all participated in the design, execution, and analysis of the paper, and that they have approved the final version.

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