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# **Evaluation of the Radiopacity of Pulp Capping Materials**

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acceptable radiopacity and may allow radiological detection during vital pulp treatment.

#### **Pulpa Kuafaj Materyallerinin Radyopasitesinin Değerlendirilmesi**



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#### **INTRODUCTION**

The base, lining or restorative materials used in the restoration of teeth must have sufficient radiopacity. Materials with sufficient radiopacity allow the detection of overflowing fillings and caries and the control of the contour of the restored tooth. With radiopaque restorative materials, it is easy to differentiate dental tissues.<sup>1,2</sup> Restorative materials such as pulp capping materials, cements and composite resins can be made radiopaque by adding elements such as ytterbium, zirconium, barium and strontium. An ideal radiopacitor should be durable and safe. Radiopacitors should provide clear radiographic images without side effects.<sup>3</sup> Unfortunately, in order to protect commercial rights, incomplete or limited information is provided about the chemical content of dental materials.

Many pulp capping materials have been used from past to present. Calcium hydroxide is the most well-known and frequently used pulp capping material.<sup>4</sup> In addition, tricalcium silicate based cements (Mineral Trioxide Aggregate -MTA) and resin modified calcium silicate based cements are currently popular pulp capping materials.<sup>5</sup> These materials should have sufficient radiopacity to be easily distinguished from anatomical structures.<sup>6</sup> Pulp capping materials are tried to increase their radiopacity by adding filler or radiopaque components at various ratios by the manufacturers.<sup>7</sup> The degree of radiopacity required for optimal clinical performance varies according to the type of material.<sup>8</sup>

The American National Standards Institute/American Dental Association standards and the International Organisation for Standardisation (ISO 6876:2012 and ISO 13116:2014) set the radiopacity standard for materials used in endodontic treatments, such as pulp capping materials. This requires more than 3 mm Al for a 1 mm thick material. $9-11$  It has been reported that aluminium penetrometer and occlusal film should be used to evaluate the radiopacity of the materials. In addition, the voltage of the X-ray device should be  $65\pm5~\text{kVp}$ and the X-ray device-object distance should be  $30 \text{ cm}$ .<sup>11,12</sup> When determining the radiopacity of a material, a disc-shaped sample of a specific thickness and height is obtained from the material and radiographed. The radiographic image obtained is compared with the radiographic image of a wedge made of aluminium.<sup>13</sup>

There are many pulp capping materials on the market with different chemical contents and different radiopacity values. Although most of these materials are stated to be radiopaque by the manufacturer, it is not known that they have sufficient radiopacity.

In the literature analysis, there is no study evaluating the radiopacities of MTA-containing pulp capping materials and calcium hydroxidecontaining pulp capping materials. Therefore, the aim of this study was to investigate the radiopacities of ten different pulp capping materials and to compare them with the radiopacities of enamel, dentin and pure aluminium penetrometer. The null hypothesis tested was as follows: The pulp capping materials analysed have acceptable radiopacities and show higher radiopacity values than enamel and dentin.

## **MATERIAL AND METHODS**

Ten different pulp capping materials were used in this study. The manufacturers and contents of the materials are given in Table 1. This study was approved by Afyonkarahisar Health Sciences University Clinical Research Ethics Committee with the report dated







#### **Preparation of Sample**

In order to create samples from the pulp capping materials, 3 mica plates with 5 slots of 5 mm inner surface diameter and 1 mm depth were prepared. In addition, a 1 mm thick rectangular mica plate was prepared with a 1

mm thick molar crown section and a 1 mm thick rectangular mica plate where the aluminium penetrometer would be placed. After all pulp capping materials were placed in the moulds, they were covered with a microscope slide for proper hardening of the surfaces. Then, the microscope slide was removed and the top of the specimen was irradiated for 20 s using an LED light curing unit (Ellipar S10; 3 M ESPE, St. Paul, MN, USA) with an output irradiance of 1200 mW/cm<sup>2</sup> and standard curing mode. A radiometer (Demetron LED Radiometer; Kerr Corp., Orange, CA, USA) was used to control the output irradiance of the LED unit. The diameter of the light tip was 9 mm and the wavelength range of the device was 430-480 nm. There was no distance between the light tip of the LED unit and the top surface of the sample. Three standardised samples were obtained for each material. These samples were

incubated at 37 °C in a humid environment until completely set.

#### **Digital radiography**

An 11-step 99.6% pure aluminium penetrometer with each step 1 mm thick was used for radiopacity comparison (Figure 1). The mica plate with the discs on which the materials were placed, the aluminium penetrometer and the section of enamel and dentine was mounted on a phosphor plate (Dürr-Dental, Bietigheim, Germany).



**Figure 1:** Digital radiographic image of pulp capping materials, molar crown section and aluminium block

This study started immediately after the annual calibration of the dental X-ray machine. After the dental X-ray machine (ORIX 70; Ardet Dental & Medical Devices, Milan, Italy) was set at 65 kVp, 7 mA and 0.2 s irradiation parameters with an object-beam distance of 30 cm, the X-ray subject was adjusted at a 90°

angle to the surface of the mica plate and irradiation was performed. The phosphor plate was scanned with a scanner (ScanX®, Air Techniques, Hicksville, NY, USA) and the images were digitised (Mediadent V8, ImageLevel, Kruibeke, Belgium).

#### **Evaluation of digital images**

In the digital images obtained after irradiation, the radiopacity of each step of the aluminium penetrometer, pulp capping materials, the tooth crown cross-section were determined using ImageJ 2.14.0/1.54f software (National Institutes of Health, Maryland, USA). The examination areas were selected to centre the materials and the average radiopacity of the selected areas was determined using the average intensity determination feature of the programme (Figure 1). Five measurements from each sample (15 measurements for each material) were made with the programme and the average of the values was calculated. The obtaining of radiographic images and radiopacity measurements of the samples were performed by a single researcher to ensure standardisation. Based on the average pixel values obtained by imageJ of the radiopacity, the equation was used to determine which aluminium step radiopacity of the materials was equivalent in millimetre aluminium  $(mmAl).^{14,15}$ 

X − Y  $\frac{x-1}{z-y}$  × Sample Thickness + mmAl below material MGVC

MGV= Mean Grey Values

 $X = MGV$  of the specimen – MGV of the step of step wedge immediately below the specimen's MGV.

Y= MGV of the step of step wedge immediately above the specimen's MGV – MGV of the step of step wedge immediately below the specimen's MGV.

Z=MGV of the aluminum step wedge increment immediately above the material's MGV.

#### **Statistical Analysis**

In order to eliminate the differences that may occur during the mixing of the pastes and to evaluate the consistency of the radiopacities of the pastes, 5 different X-rays were taken from the samples prepared with the same paste. For each sample and each step of the penetrometer, 5 colour assessments were made on the digital images and the average of these 5 assessments was taken. One-way ANOVA and post-hoc Tamhane test were applied for statistical analysis of the data obtained. The significance level was set as  $p < 0.05$ .

#### **RESULTS**

For each material, 15 images were evaluated. The OGD values of the aluminium block in a randomly selected image are given in Table 2. The radiopacity values of the material groups in mmAl are given in Table 3.

**Table 2** Aluminum block in a randomly selected image OGD values according to steps

	Mean +Sd
1 mm	$33.21 \pm 6.75$
$2 \text{ mm}$	$47.76 \pm 6.7$
$3 \text{ mm}$	$60.76 \pm 6.47$
4 mm	$72.48 \pm 6.42$
$5 \text{ mm}$	$82.52 \pm 6.17$
6 mm	$91.40\pm 6.25$
7 mm	$99.62 \pm 6.17$
$8 \text{ mm}$	$107.21 \pm 6.14$
9 mm	$115.16 \pm 6.12$
$10 \text{ mm}$	$121.31 \pm 6.03$
$11 \text{ mm}$	$127.31 \pm 5.97$

**Table 3** Radiopacity values of the Materials (Mean and standard deviation)



Different top symbol letters indicate statistically significant difference. ( $p < 0.05$ )

The results of the one-way ANOVA test showed that there was a significant difference between the groups ( $p < 0.05$ ). Neoputty (5.19) mmAl) group showed the highest radiopacity values, while Dycal (1.23 mmAl) group showed the lowest radiopacity value ( $p < 0.05$ ). Among the MTA groups, the highest radiopacity value was in the Neoputty group, while the lowest radiopacity value belonged to the Biofactor MTA (2.71 mmAl) group. There was no statistical difference between Therecal LC (1.52 mmAl) and Therecal PT (1.24 mmAl) groups (p = 0.993). In addition, Dycal, Calcimol LC, TheraCal groups and BIOfactor MTA groups had radioactivity values lower than 3 mmAl, which is the minimum radioactivity value determined by ISO (6876/2001) and ANSI/ADA (57/2000). While the radiopacity values of Dycal, Calcimol LC, Theracal LC, Therecal PT were lower than enamel, there was no statistically significant difference with dentin ( $p < 0.05$ ).

## **DISCUSSION**

This study aimed to investigate the radiopacities of ten different pulp capping materials and compare them with the radiopacities of enamel, dentin and pure aluminum penetrometer. The present in vitro study partially rejects the null hypothesis that the tested pulp capping materials exhibit acceptable radiopacity and have high radiopacity values compared to enamel and dentin.

Although radiology has been used in medicine for many years, intraoral digital radiographs have been used in dentistry since 1980. <sup>16</sup> In dentistry, radiographs are frequently used to control restorative treatments and to monitor restorations. Therefore, restorative materials must have radiopaque properties. Radiopaque materials are more easily distinguished from surrounding tissues. In addition, it is easier to evaluate the restorative

treatment borders, condensation and quality of the material on radiographs. The use of radiopaque materials in restorations enables the detection of secondary caries, restoration adaptations, defective proximal contacts and marginal defects.<sup>17</sup> In case of accidental ingestion of radiopaque restorative materials or if they enter the respiratory tract, it is easier to detect them as foreign material.<sup>18</sup>

To evaluate the radiopacities of materials used in dentistry, the digitised images of conventional radiographs or the direct method with digital radiography can be preferred.<sup>19,20</sup> In the radiographic image of a material obtained with digital radiography, the radiopacity of the material may have a different value compared to conventional radiography.<sup>21</sup> Digital radiography reduces the amount of radiation exposure and eliminates the need for film developing chemicals used in conventional radiography. Thus, less environmental pollution occurs. Clearer radiographic images can be obtained with digital radiography.<sup>22</sup> In addition, digital radiography has become more advantageous than conventional radiographs because it is faster and easier to obtain images.<sup>23</sup> Due to these features, digital radiographic imaging is frequently used to evaluate the radiopacities of dental materials in in vitro studies. Two different systems can be used in digital radiography: direct sensor systems (CCD) and phosphor plates (PSP) semi-direct image plate systems. Phosphor plates (PSP) are used more frequently because they are wireless and flexible.<sup>24</sup> In digital radiography, the image is formed by combining the x and y coordinates of the image elements (pixels) and the degrees of grey values. The grey values at the coordinates defined by the pixels are measured and the radiopacity of the material is determined by assigning a value between 0 and 255 to this measurement with a software program.<sup>25</sup> The radiopacity values of the materials are then converted to mmAl. $^{26}$  In the study, digital

images of the samples were analysed using Image J software to determine the radiopacity of pulp capping materials.<sup>15</sup>

To assess the radiopacity of dental materials, materials of a given thickness under controlled radiographic conditions (65 kVp, 7 mA, exposure, 0.2 seconds) are compared with an aluminium step block.<sup>27</sup> In this study, an object-focus distance of 30 cm, the same exposure parameters (65 kVp, 7 mA, exposure 0.2 seconds) were maintained for each sampling, and an aluminium block with 11 mm steps in 1 mm increments was used.

The International Organisation for Standardisation (ISO:4049) has recommended that an Al step wedge of 99.6% purity be used as a reference when assessing radiopacity. It also recommends that the radiopacities of the materials tested should be equal to or greater than aluminium of the same thickness and that any value claimed by the manufacturer should not be less than 0.5 mm.<sup>28</sup> Aluminium is used as a reference in radiopacity evaluations because it is easy to produce and shows similar radiopacity to dentin.<sup>29</sup> The purity of the aluminium step is very important. The presence of 4% copper content in the aluminium alloy leads to lower radiopacity measurements and errors compared to 99.5% aluminium alloy.<sup>30</sup> In this study, a special stepped wedge made of 99.6% purity aluminium blocks was used. ANSI/ADA<sup>9</sup> recommends that aluminium step block should be manufactured in thicknesses ranging from 1 to 10 mm, with steps of 1 mm each. In radiopacity studies, an absolute white image (grey scale value 255) can be found when the aluminium step block is manufactured up to 10 mm thick. Gu et al. $25$  found a grey scale value of 255 using aluminium step wedges with thicknesses ranging from 1 to 15 mm. In this study, the steps of the 11-step aluminium block were represented on a scale of approximately 33.21-127.31 instead of 0-255. In this study,

Teflon moulds with a diameter of 5 mm were used to prepare test samples according to ANSI/ADA Specification No. 57 for the evaluation of the radiopacities of the materials.<sup>9</sup> Thus, the volume of the samples was reduced and more samples were placed on the phosphor plate.<sup>31</sup>

The use of tooth sections (enamel, dentin) has served as a reliable control in studies investigating the radiopacity of materials used in dentistry.<sup>32</sup> The inorganic content of enamel is structurally higher than the inorganic content of dentin. Enamel prisms have a different structure than dentin canals. For these reasons, the radiopacity of enamel is higher than the radiopacity of dentin.<sup>33</sup> In a study, the radiopacities of enamel and dentin were determined as 2.19 and 1.25 mmAl, respectively.<sup>15</sup> In accordance with the literature, the radiopacity value of enamel and dentin was 1.96 mmAl and 1.21 mm Al, respectively.

In this study, the radiopacity of pulp capping materials used to maintain the vitality and function of the dental pulp was investigated. Neoputty (5.19 mmAl) group showed the highest radiopacity values, while Dycal (1.23 mmAl) group showed the lowest radiopacity value ( $p < 0.05$ ). Pulp capping materials should be distinguishable from dentin.<sup>34</sup> In addition, a threshold of 3 mmAl, the minimum radiopacity value specified by ISO (6876:2012) and ANSI/ADA (57/2000), was used when interpreting these materials.<sup>11,35</sup> While MTA Angelus was found to have a radiopacity value higher than 6 mmAl in radiopacity studies, it showed a radiopacity value of 3.66 mmAl in another study. $36,37$  Bismuth oxide was added to MTA Angelus to give it radiopaque properties. In the studies carried out, it was stated by the researchers that bismuth oxide negatively affects the hardening reaction of the material.<sup>38</sup> Bismuth oxide has also been found to be toxic to human dental pulp cells. $39$  It has been reported that the addition of bismuth oxide to

the content of Portland cement creates defects in the physical structure of the material and causes a porous structure. This may increase the solubility and degradation of the material.<sup>40</sup> Powder: liquid mixing ratios of materials can affect radiopacity values. Although a 3:1 ratio is recommended by the manufacturer, changing this mixing ratio leads to a change in the radiopacity value. For example, it has been reported that the preparation of White MTA in the form of 4:1 powder/liquid ratio can obtain high radiopacity.<sup>41</sup> Although the high radiopacity of the material provides a benefit, the extent to which this affects the physical and mechanical structure of the material is a matter of debate. In order not to reduce the quality of the treatment applied, the powder / liquid ratio recommended by the manufacturer should be followed.

The radiopacity value of BIOfactor MTA, one of the materials tested in this study, was below 3 mmAl according to the standard set by ISO<sup>10</sup> and ANSI/ADA.<sup>9</sup> However, no statistical difference was found when compared with Angelus MTA, MTA Cem and NeoMTA2 groups. Mutlu and  $Akbulut^{37}$  found the radiopacity value of BIOfactor MTA higher than 3 mmAl, which is different from the results of our study. Unlike other calcium silicate based materials, BIOfactor MTA contains yttrium oxide as radiopaque agent. Costa et al.<sup>42</sup> reported that ytterbium oxide showed higher cell viability and was more biocompatible than bismuth oxide. Ytterbium oxide does not affect the physicochemical and biological properties of calcium silicate based materials as well as maintaining their bioactive potential.<sup>42</sup> Further research should be conducted to evaluate the biocompatibility, bioactivity and physicochemical properties of BIOfactor MTA. NeoPUTTY (5.19 mmAl ), which showed the highest radiopacity value in our study, is a commercially available bioceramic material that is premixed and contains bioactive

properties. It contains radiopaque tantalum oxide (tantalite). It has also been reported that its hardness, non-stickness, resistance to washing, bioactivity and ability to be used without waste make this material more preferable in the clinical environment.<sup>43</sup> NeoMTA2 (3.20 mmAl) has the same content as NeoPUTTY but showed a lower radiopacity value. The manufacturer did not provide information on the amount of radiopaque tantalum oxide in these two materials.<sup>44</sup> We think that the fact that NeoPUTTY was previously mixed and ready to use and NeoMTA2 was in powder-liquid form may cause differences in radioactivity. Since the materials in powder-liquid form are mixed at different ratios, differences in radiopacity may occur.

The radiopacity values of Calcimol LC, TheraCal LC and TheraCal PT groups, which are light-cured pulp capping materials, were below 3 mmAl and no statistical difference was found between these groups.  $(p>0.05)$  This may be related to the different ratios and types of radiopacifying agents and fillers.<sup>45</sup> TheraCal LC is a methacrylate-based resin combined with tricalcium silicate and barium zirconate as radiopacifiers. It was introduced as a lightcuring material for vital pulp treatments. TheraCal PT, on the other hand, is a material with a dual cure curing reaction and is especially recommended for pulpotomy treatments, but it is also stated by the manufacturer that it can be used in direct and indirect pulp capping. This material contains barium zirconate and ytterbium fluoride as radiopacifiers.<sup>5</sup> No study evaluating the radiopacity of ThereCal PT, which came to the market in 2019, was found in the literature. Gandolfi et al.<sup>46</sup> found the radiopacity of TheraCal LC and Dycal to be below 3 mmAl, which is consistent with our study. In another study evaluating the radiopacity of Dycal, MTA was found to have a lower radiopacity than Angelus.47

BioMTA+  $(3.75 \pm 0.38)$  contains calcium zirconium complex as radiopacifier and its radiopacity was found higher than 3 mmAl in our study. In a study evaluating the radiopacity of MTAs, the radiopacities of BioMTA+ and MTA Angelus were found to be higher than 3 mmAl in accordance with our study. However, in this study, the radioactivity of MTA Cem was lower than 3 mmAl, while the radioactivity of MTA Cem was 3.71 mmAl in our study.<sup>48</sup> The difference in the radioactivity of MTA Cem may be due to the fact that the material is in powder-liquid form and the preparation conditions are different. Radiopacity studies performed in vitro cannot completely simulate intraoral conditions. The limitation of this study is that the effects of humidity, temperature and ageing in the oral environment could not be analysed. In addition, the release of ions such as barium, silicon and strontium from the material can reduce the radiopacity. The amount of dust and liquids in radiopacity studies is not quantitatively measured, which causes differences in the results of the study. Methodological differences such as x-ray source, current, voltage, irradiation time and film-focus distance affect the results of radiopacity studies.<sup>49,50</sup> Further studies in oral conditions and simulating the ageing procedure are needed.

# **CONCLUSIONS**

Within the limitations of this in vitro study, Neoputty (5.19 mmAl) had the highest radiopacity value, while BioMTA+, MTA Angelus, MTA Cem and NeoMTA2 showed radiopacity values higher than enamel and dentin and above 3 mmAl. Dycal, Calcimol LC, Theracal LC, Therecal PT had radiopacity values close to the radiopacity of dentin. Studies evaluating the radiopacity of pulp capping materials should be carried out periodically, and manufacturers should regularly renew the formulation of their products to obtain better properties.

# **Ethical Approval**

The necessary ethical approval for this study was received by the Afyonkarahisar Health Sciences University Non-Pharmaceutical and Medical Device Ethics Committee (Decision no:2023/464).

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The authors have no relevant financial or nonfinancial interests to disclose.

## **Conflict of Interest**

The authors deny any conflicts of interest related to this study.

## **Author Contributions**

Design: LAU, ES, Data collection and processing: ES, Analysis and interpretation: LAU, ES, Literature review: LAU, ES, Writing: LAU.

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