# Radiopacity evaluation of contemporary resin composites by digitization of images

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## ABSTRACT

**Objective:** The aim of this study was to evaluate the radiopacity of different composite resins and compare the values to those of human enamel and dentine. **Materials and Methods:** Five specimens of each material with thicknesses of 2 mm were prepared and radiographed alongside aluminum step wedge and human enamel and dentin. Three occlusal radiographs for each material were taken and digitized using a desktop scanner. Mean gray values of the test materials were measured using Image J software. Then a conversion was performed according to establish the radiopacity of the test materials, in millimeters of equivalent Al. Data were analyzed using one-way analysis of variance and Duncan multiple range tests ($P < 0.05$). **Results:** The radiopacity values varied among the restorative materials ($P < 0.05$). The radiopacity values of the materials tested were, in decreasing order: Enamel Plus HRI > Z250 > Filtek Ultimate ≥ Z550 > Nexcomp ≥ Nanoceram Bright > enamel ≥ Estelite Sigma Quick > Clearfil Majesty Esthetic ≥ Reflexions XLS ≥ Aelite LS Posterior ≥ dentin ≥ 2 mm Al. **Conclusion:** All resin composite materials investigated in this study presented different radiopacity values. However, all materials had radiopacity values greater than dentin and had sufficient radiopacity to meet International Organization for Standardization 4049 standard.

**Key words:** Dental, radiography, radiopacity, resin composite

## INTRODUCTION

Recent advances and developments in resin composite restorative materials brought reduced particle size and increased filler loading, which significantly improved light-cured composite resins for universal use in anterior and posterior teeth.[1-3] One of the important characteristics that should be considered is the radiopacity of the resin composites used in anterior and posterior restorations.[1,3] Modern resin composites use glass particles with high atomic numbers, such as barium, strontium, and zirconium to produce a radiopaque material.[3,4] As quartz, lithium-aluminum glasses, and silica are not radiopaque, they incorporated with other filler particles into the inorganic filler phase of resin composites.[4]

Adequate radiopacity is required in order to distinguish a restorative material from the surrounding tissue.[5] It is very difficult to locate enamel-composite margins radiographically because of the relatively low radiopacity of composites.[4] Adequate radiopacity is required to evaluate restorations for marginal defects, marginal overhangs, interproximal contour, help differentiate the restorative material from dental caries, and detect microleakage.[5] In case of accidental aspiration or traumatic impaction, the location and the removal of fragments of the radiopaque restorative materials may be extremely important.[5,6]

Requirements for the radiopacity of dental restorative resin established by the International Organization for Standardization (ISO) standard 4049 specifies that the radiopacity of a 2 mm thick specimen of

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the material should be equal to that of a 2 mm or larger thickness of aluminum.[7] The American Dental Association also requires commercial dental restorative resins to have a radiopacity at least equal to that of aluminum.[5] One of the commonly used techniques to determine the radiopacity of dental materials is the digitization of conventional images obtained under standard radiographic conditions.[8-12] The radiopacity of a dental material specimen is usually expressed in terms of equivalent aluminum thickness (in millimeters) by comparing specific thickness of material to aluminum step wedges under typical radiographic conditions.[13-15]

Because the increasing demand for anterior and posterior esthetic restorations, it is important to evaluate their physical and chemical properties, including radiopacity. The aim of this study, therefore, was to evaluate the radiopacity of different universal resin composites by digitization of images, and compare the values to those of human enamel and dentine. The null hypothesis tested was that the material type would not affect the radiopacity of resin composites.

## MATERIALS AND METHODS

### Specimen preparation

Ten different commercial brands of light-cured resin composites were used: Filtek Ultimate (A2B shade, 3M ESPE, St. Paul, MN, USA), Filtek Z550 (A2 shade, 3M ESPE, St. Paul, MN, USA), Z250 (A2 shade, 3M ESPE, St. Paul, MN, USA), Enamel Plus HRI (UE2 shade, Micerium, Avegno, Italy), Aelite LS Posterior (A2 shade, Bisco, Schaumburg, USA), Reflexions XLS (MD shade, Bisco, Schaumburg, USA), Nanoceram Bright (A2 shade, DMP, Markopoulo, Greece), Nexcomp (A2 shade, Meta Biomed, Chungbuk, Korea), Clearfil Majesty Esthetic (A2 shade, Kuraray Medical, Okayama, Japan), and Estelite Sigma Quick (A2 shade, Tokuyama Dental, Tokyo, Japan). Information provided by the manufacturers is summarized in Table 1.

A teflon ring mold with an internal diameter of 5 mm and a depth of 2 mm was used to prepare the specimens. The mold was placed on a glass slab and resin composites were packed into the mold until it was overfilled and then covered with another glass slab. The specimens were then light-cured for 40 s using the exit window of a quartz-tungsten-halogen light polymerization unit (Demetron LC, Kerr, Orange, CA, USA) that was placed against the glass slab. Before preparation of the specimens of each group, the light output was checked (600 mW/cm$^2$) by a radiometer (Demetron, Danbury, CT, USA). Five specimens were made of each resin composite material. The specimens were stored at 37°C for 24 h. The specimens with porosities were excluded from

**Table 1: Materials used in this study**

| Brand (manufacturer, lot no.) | Type (shade) | Composition | Filler loading |
|---|---|---|---|
| Filtek Ultimate (3M ESPE, N175893) | Nanofilled resin composite, light-cured, universal (A2B) | Filler type: Zirconia/silica, zirconia, silica<br>Resin matrix: BISGMA, BISEMA, UDMA, TEGDMA, PEGDMA | 78.5% by weight<br>63.3% by volume |
| Z550 (3M ESPE, N286648) | Nanohybrid resin composite, light-cured, universal (A2) | Filler type: Zirconia/silica, silica<br>Resin matrix: BISGMA, BISEMA, UDMA, TEGDMA, PEGDMA | 82% by weight<br>68% by volume |
| Z250 (3M ESPE, N270396) | Microhybrid resin composite, light-cured, universal (A2) | Filler type: Zirconia/silica<br>Resin matrix: BISGMA, BISEMA, UDMA, TEGDMA | 82% by weight<br>60% by volume |
| Enamel Plus HRI (Micerium, 2011004518) | Nanofilled resin composite, light-cured, universal (UE2) | Filler type: Glass filler, nano zirconium oxide<br>Resin matrix: BISGMA, UDMA, butanediol dimethacrylate | 80% by weight<br>63% by volume |
| Aelite LS Posterior (Bisco, 1100008554) | Nanohybrid resin composite, light-cured, posterior (A2) | Filler type: Glass filler, amorphous silica<br>Resin matrix: Ethoxylated BISGMA | 88% by weight<br>74% by volume |
| Reflexions XLS (Bisco, 1100001753) | Nanohybrid resin composite, light-cured, universal (MD) | Filler type: Glass filler, amorphous silica<br>Resin matrix: Ethoxylated BISGMA | 88% weight<br>76% volume |
| Nanoceram bright (DMP, 630233) | Nanohybrid resin composite, light-cured, universal (A2) | Filler type: Barium glass<br>Resin matrix: Methacrylate polymers | 80% by weight |
| Nexcomp (Meta Biomed, MX11062202) | Nanohybrid resin composite, light-cured, universal (A2) | Filler type: Barium aluminum boro silicate<br>Resin matrix: Ethoxylated BISGMA, BISGMA, UDMA | 75% weight |
| Clearfil Majesty Esthetic (Kuraray, 00043A) | Nanohybrid resin composite, light-cured, universal (A2) | Filler type: Barium glass<br>Resin matrix: BISGMA, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic methacrylate | 78% by weight<br>66% by volume |
| Estelite Sigma Quick (Tokuyama Dental, E492M) | Submicron filled resin composite, light-cured, universal (A2) | Filler type: Silica-zirconia filler, composite filler<br>Resin matrix: BIS-GMA, TEGDMA | 82% by weight<br>71% volume |

BISGMA: Bisphenol A diglycidyl ether dimethacrylate, BISEMA: Bisphenol A polyethylene glycol diether dimethacrylate, UDMA: Diurethane dimethacrylate, TEGDMA: Triethylene glycol dimethacrylate, PEGDMA: Polyethylene glycol dimethacrylate the study and replaced to provide five homogeneous specimens of each material.

Three freshly extracted noncarious human third molars were also used in this study. The roots were removed two mm beneath the cementoenamel junction and the remaining tooth portion was mounted in gypsum blocks. The teeth were then sectioned mesiodistally by using a low-speed diamond saw (Microcut125, Metkon, Bursa, Türkiye). The tooth slices involving each enamel and dentin substrate were ground flat with carbide paper and the specimens 2.0 mm in thickness were obtained. The tooth slices were kept in distilled water until use.

An aluminum stepwedge (6063 alloy, 98% purity) ranging from 2.0 to 12.0 mm in thickness was used. The aluminum step wedge was used as an internal standard for each radiographic exposure, which allowed the radiopacity of each material to be calculated in terms of aluminum thickness.

### Radiographic procedures

Ten groups with five specimens of each material were placed directly on a 57 × 76 mm Ultra-speed occlusal radiographic film (Eastman Kodak Co, Rochester, NY, USA), together with an Al step wedge and three tooth slices of both enamel and dentin, which were used for comparison [Figure 1]. A 2 mm thick lead sheet was placed under the film in order to prevent back-scattered radiation. All specimens were placed at a 40 cm focus-film distance for 0.32 s in a dental X-ray unit (PlanmecaIntra, Helsinki, Finland) with 2 mm Al equivalent total filtration at 63 Kv, 8 mA. This procedure was repeated in order to obtain three different radiographic sets of the same specimens. The X-ray unit was kept in the same position throughout the experiment. All the radiographs were processed at once in an automatic processor (Dürr XR 24 Bietigheim, Germany) at 28°C for 4.3 min with fresh solutions.

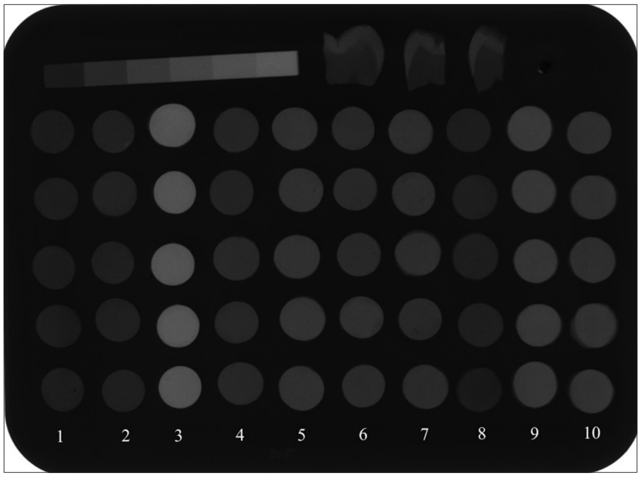


**Figure 1:** A digitized occlusal radiographic film obtained with the graduated aluminum step wedge, dentin and enamel, human molar tooth slices (top row), and five specimens of each test material. (1) Aelite LS Posterior, (2) Clearfil Majesty Esthetic, (3) Enamel Plus HRI, (4) Estelite Sigma Quick, (5) Filtek Ultimate, (6) Nanoceram Bright, (7) Nexcomp, (8) Reflexions XLS, (9) Z250, (10) Z550

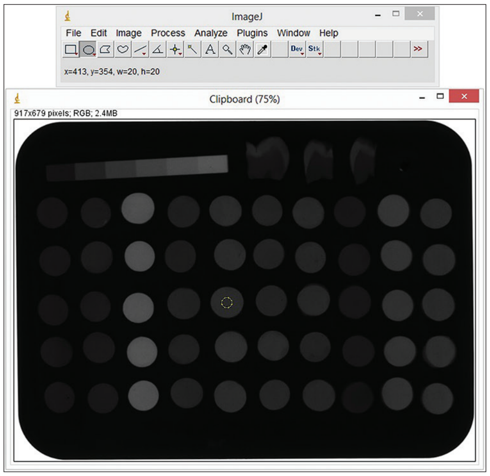
The radiographs were digitized using a desktop scanner with a transparent adapter (Epson Perfection V700, Japan) at 16-bit gray value and 300 dpi resolution and saved in tag image file format. On each radiographic image, a 20 × 20 pixel region of interest was selected on the center of each test material, on dentin and enamel of each tooth specimen and on each step of the step wedge [Figure 2]. The image was enlarged in order to accurately define the enamel and dentin layers. Mean gray values (MGV) of the each test material, step wedge and enamel and dentin on three digitized radiographs were measured using ImageJ 1.46r software (National Institutes of Health, USA). The mean of three MGVs was accepted as the MGV of test materials. The radiopacity value was determined according to the radiographic density and converted into millimeters of Al (mm Al). Conversion was performed using the following conversion equation: [9]

$$\frac{A \times 2}{B} + \text{mm Al immediately below RDM}$$

Where:

A = Radiographic density of the material (RDM) – radiographic density of the aluminum step wedge increment immediately below RDM.

B = Radiographic density of the aluminum step wedge increment immediately above RDM – radiographic



x=413, y=354, w=20, h=20

917x679 pixels; RGB; 2.4MB

**Figure 2:** The circle on the center of a composite sample demonstrates the selected region of interest

density of the aluminum step wedge increment immediately below RDM.
2 = 2 mm increments of the aluminum step wedge.

**Statistical analysis**
Radiopacity values (in mm Al) for each material and enamel and dentin were compared using one-way analysis of variance (ANOVA), and pairwise comparisons were made by Duncan's multiple range tests with SPSS for Windows (Version 18.0, Chicago, USA). For all tests, the probability level for statistical significance was set at $\alpha$ =0.05.

## RESULTS

There were statistically significant differences among the restorative materials when the results were compared using the one-way ANOVA [$P < 0.05$, Table 2]. The means and standard deviations for the MGV and radiopacity values expressed as Al equivalent millimeters of the restorative materials tested and enamel and dentin are presented in [Table 3]. The mean radiopacity values of the resin composites ranged from 2.21 ± 0.11 to 11.56 ± 0.98 mm Al.

Dentin had the lowest radiopacity value [2.09 ± 0.15, Table 3]. All the resin composites tested had radiopacity values greater than the radiopacity of dentine and 2 mm Al, except for Reflexion XLS and Aelite LS Posterior. These two resin composites showed radiopacity similar to the dentin substrate ($P > 0.05$).

Enamel showed a radiopacity equivalent to 3.42 ± 0.27 mm Al. Enamel Plus HRI had the highest radiopacity value (11.56 ± 0.98), which was significantly higher than those of human enamel and the other materials tested [$P < 0.05$, Table 3]. Z250, Filtek Ultimate, Z550, Nexcomp, and Nanoceram Bright were also more radiopaque than the enamel specimen and statistically different to enamel ($P < 0.05$). One material (Estelite Sigma Quick, 3.29 ± 0.37 mm Al) showed radiopacity similar to enamel ($P > 0.05$). Radiopacity values of Clearfil Majesty Esthetic, Reflexions XLS, and Aelite LS Posterior were lower than the radiopacity of enamel ($P < 0.05$), which presented no statistically significant difference among them [$P > 0.05$, Table 3].

## DISCUSSION

It is desirable for the clinician to radiographic differentiation between restorative composites and dentin and many authors suggest that the materials should present a radiopacity not less than that of the dentin that is being replaced, in order not to be misinterpreted as decalcified dentin.[16,17] In this study, all the resin composites evaluated provided similar or higher radiopacity than the same thickness of aluminum and the dentin specimen that fulfilled the requirements of ISO 4049 in terms of radiopacity.[7]

Radiopacity greater than, or equal to, enamel is considered a prerequisite for especially posterior use to improve the radiographic diagnosis of secondary caries.[18-21] In addition to that, variations in thickness of materials may influence the resultant radiopacity although it is less important than molecular structure of a material.[22] Some authors mentioned that small enamel-restricted cavities filled with less radiopaque materials than enamel can hardly be detected by radiographic examination due to the superposition of healthy enamel.[22] Moreover, enamel shade is often used as a thin layer in stratified polychromatic anterior

**Table 2: One-way analysis of variance test results**

| Source | df | Sum of squares | Mean square | *F* value | *P* |
|---|---|---|---|---|---|
| Between groups | 12 | 1081.224 | 90.102 | 472.079 | 0.000* |
| Within groups | 158 | 30.156 | 0.191 | | |
| Total | 170 | 1111.380 | | | |

*Statistically significant difference. SD: Standard deviation

**Table 3: Radiopacity of 10 ligth-cured resin composite materials with human enamel and dentin radiopacity values (mean±SD) as reference equivalent thickness of aluminum for 2 mm specimen thickness, and statistical differences between the groups**

| Material | Mean±SD | |
|---|---|---|
| | Mean gray value | Radiopacity value (mm Al equivalent) |
| Enamel Plus HRI | 76.37±2.70 | 11.56±0.98$^{A}$ |
| Z250 | 46.64±2.69 | 5.67±0.39$^{B}$ |
| Filtek Ultimate | 40.15±2.64 | 4.75±0.38$^{C}$ |
| Z550 | 39.92±2.65 | 4.72±0.32$^{C}$ |
| Nexcomp | 35.10±2.82 | 4.06±0.41$^{D}$ |
| Nanoceram Bright | 34.00±3.30 | 3.97±0.56$^{D}$ |
| Enamel | 30.38±2.14 | 3.42±0.27$^{E}$ |
| Estelite Sigma Quick | 29.45±2.76 | 3.29±0.37$^{E}$ |
| Clearfil Majesty Esthetic | 24.34±2.08 | 2.61±0.25$^{F}$ |
| Reflexions XLS | 21.36±2.00 | 2.25±0.23$^{FG}$ |
| Aelite LS Posterior | 21.31±1.44 | 2.21±0.11$^{FG}$ |
| Dentin | 19.86±1.59 | 2.09±0.15$^{G}$ |
| Step wedge (2 mm) | 19.84±1.85 | 2.00±0.00$^{G}$ |

Means in a column followed by the same capital letter are not significantly different by Duncan's multiple range test at $\alpha$=0.05, $n$=5 for resin composites, $n$=3 for enamel/dentin specimens, SD: Standard deviation and posterior composite restorations to build up the contact areas and anatomical proximal contours.[23] Thus, a material with higher radiopacity than enamel should be used in these situations, since the optical density of materials applied with a lower thickness is critical.

Enamel Plus HRI is a polychromatic layered nanofilled resin composite system with zirconium oxide nanoparticles (mean particle size of 20 nm, filler content of 12% by weight) and high refractive index (the same as enamel) glass particles (mean particle size of 1 µm, filler content of 68% by weight) to provide an anatomical strafication, with equal thicknesses compared with dental tissues.[24] The material presented the highest radiopacity among the tested composites. Because the filler component of composite resins is not disclosed by chemical analysis in this study and some detailed information is not provided by the manufacturer, type of the glass filler used in Enamel Plus HRI is not known. However, the filler design for Enamel Plus HRI (Universal Enamel, UE2 shade) improved its radiopacity by the combination of high atomic number element zirconium and new type of filling glass particles.

Radiopacity is achieved by incorporating finely divided heavy-metal glass particles in some microhybrid composites.[4] Recently, there have been some studies of $SiO_2$-$ZrO_2$ mixed particles and $SiO_2/ZrO_2$ core-shell structure prepared by the sol-gel chemical processing in the literature.[25,26] One of these studies indicated that the composites filled with $SiO_2/ZrO_2$ core-shell filler met ISO 4049 criteria and presented suitable radiopacity for diagnostic purposes.[25] Toyooka *et al*.[15] evaluated the radiopacity of resin composite materials by the chemical analyses of fillers and found that zirconium dioxide was radiopacifier equal to or even stronger than bariumoxide.

Today, radiopacity is most commonly achieved by using nanomeric zirconia or by incorporating the zirconia in the nanoclusters along with silica in nanocomposites.[4] In addition to Enamel Plus HRI, the other composites used in this study which had the similar or higher degree of radiopacity values with enamel are Z250 (microhybrid), Filtek Ultimate (nanofilled), Z550 (nanohybrid), and Estelite Sigma Quick (submicron filled). These highly-filled composites in which the total content of the fillers of 60-71% by volume contain zirconia/silica cluster fillers, silica nanoparticles, and zirconia nanoparticles.[27,28]

In this study, nanohybrid resin composites (Aelite LS Posterior, Reflexions XLS, Nanoceram Bright, Nexcomp, Clearfil Majesty Esthetic) which contain glass particles as a radiopaque filler were also evaluated. Among these composites, only Nexcomp (4.06 ± 0.41) composed of 75% weight barium glass filler particles and Nanoceram Bright (3.97 ± 0.56) composed of 80% by weight barium aluminum boro silicate glass particles had significantly higher radiopacity than enamel (3.42 ± 0.27).

The radiopacity of a resin is higher if the composition of the resin includes larger amount of elements with high atomic numbers at higher filler content.[29,30] Although barium is considered to be strongest radiopacifier for the filler of composites, some authors stated that barium ions are not biocompatible when leached out into the oral fluid.[25] In contrast, zirconium has been stated as a chemically inert, biocompatible material that slightly reduces the chemical stability of $SiO_2$ fillers of resin composites in the oral environment.[25] Zirconia was introduced into dentistry in the end of the 1990s and accelerated use of that material in dentistry have been indicated as a result of its excellent strength, superior fracture resistance, and suitable optical properties.[31] In modern composites, radioactive compounds such as thorium and uranium have been used in order to mimic fluorescence of human dentine, opalescence of human enamel and to achieve the necessary X-ray opacity. Zirconia contains small amounts of radionuclides from the uranium-radium and thorium actinide series.[31,32] Because zirconia may contain a certain amount of radioactive isotopes, maximum acceptable concentration should also be considered according to the standard regulating radioactive compounds in dental materials.[33]

Within the limitations of this study, the following conclusions can be drawn: The hypothesis was rejected that the radiopacity of resin composites is dependent on the material type. The contemporary restorative resin composites assessed in this study presented different radiopacity values. However, all materials tested fulfilled with the requirement of ISO 4049 guidelines. Future studies that evaluate the correlation between type, percentage, proportional amount of the radiopaque element in filler and radiopacity of dental restorative materials should be undertaken in order to evaluate new restorative material compositions in the market.

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