Radiopacity of Experimental Composite Resins Containing Radiopaque Materials

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This study aimed to investigate the relationship between the radiopacity of an experimental light-cured composite resin and the amount and type of its radiopaque material: Ti, SrCO₃, ZrO₂, BaSO₄, or Bi₂O₃. Radiopacity of each material was evaluated in terms of aluminum equivalent thickness using an aluminum step wedge. Then, the half-value layer thickness of the experimental composite resin, which had an aluminium equivalent thickness of 1 mm, was evaluated. It was found that the effects of elemental composition were not identical when evaluated in terms of aluminum equivalent thickness and by half-value layer. Nevertheless, both measurement methods indicated that radiopacity increased with increase in radiopaque material content as well as increase in atomic number of the element.

Key words: Aluminum equivalent thickness, Half-value layer, Composite resin

INTRODUCTION

Of late, it has become increasingly difficult to differentiate composite resins from actual teeth because of the vast esthetic improvements of composite resins. Therefore, many dental composite resins have a radiopacity characteristic to be distinguished from teeth for diagnosis^{1–2}. Alternatively, fluorescence observation has been suggested as another method to distinguish composite resin³. Several studies have evaluated the radiopacity of composite resins^{4–8}. However, these studies merely estimated the radiopacity of commercial products, and then compared these products with enamel and dentin^{1,2,4–8}. Few studies^{9,10} have evaluated the elemental compositions of these products to explain radiopacity; and even so, these studies did not clearly discuss the correlation between the amount of radiopaque materials and the intensity of radiopacity.

Radiopacity of dental composite resins is usually evaluated according to ISO standard 4049:2000¹¹, which measures aluminum equivalent thickness using a dental X-ray unit. The intensity of an X-ray beam is affected and altered by three components of an X-ray unit: tube voltage, filter, and bulb structure. As a result, radiographic images vary according to the X-ray sensor and X-ray unit used – even though the tube voltage is the same. This means that aluminum equivalent thickness constitutes only a relative value of each condition. Against this background, we believe that it is necessary to obtain absolute values that are based on fundamental radiological principles.

In radiology, a half-value layer is defined as “the thickness of an absorber, such as aluminum, required

to reduce the number of X-ray photons passing through it by one half”¹², and it is usually used to determine the radiopacity of a material. The half-value layer is needed to calculate the effective energy of an X-ray unit, the equivalent value, and the linear absorption coefficient of a material. In radiological terms, dental X-ray units are evaluated by their differences in effective energy – which is calculated using linear absorption coefficient based on half-value layer. The intensity of an X-ray beam is determined by the linear absorption coefficient (μ) and thickness (x) of a material. The initial X-ray intensity (I_0) is calculated by the following formula¹³:

$$I = I_0 e^{-\mu x}$$

When transmitted through the half-value layer of a material, the X-ray intensity is half of the initial X-ray intensity, as expressed below:

$$I = 0.5 I_0$$

Therefore, the relationship between the half-value layer (HVL, cm) and the linear absorption coefficient (μ) of a material is given as:

$$\mu(HVL) = \log_e 2$$

Barium (Ba), strontium (Sr), zirconium (Zr), and titanium (Ti) have been used lately as radiopaque materials for dental composite resins. Ti is used for metal copings or prosthetic frameworks, Zr for dental ceramic restorations, bismuth (Bi) is applied as a radiopaque sealer in endodontic treatments, and Ba is the most commonly used radiopaque material in medicine and dentistry. However, it is yet to be fully clarified which is the most

effective radiopaque material for composite resins. Therefore, the present study aimed to examine the relationship between the radiopacity of an experimental light-cured composite resin and the amount and type of the radiopaque material that it contained — namely titanium, strontium carbonate, zirconium oxide, barium sulfate, or bismuth oxide.

MATERIALS AND METHODS

Experimental radiopaque light-cured composite resins
Table 1 lists the radiopaque materials and resin components used in the present study.

Titanium (Ti), strontium carbonate (SrCO_3), zirconium dioxide (ZrO_2), barium sulfate (BaSO_4), and di-bismuth trioxide (Bi_2O_3), all used as powders without any purification or sieving, were selected as radiopaque materials for this study.

The matrix resin of all experimental composites was 70/30 mass% of urethane dimethacrylate (UDMA) and triethyleneglycol dimethacrylate (TEGDMA), while 0.50 mass% of camphorquinone (CQ) and 1.00 mass% of dimethylaminoethylmethacrylate (DMAEMA) were added as the photoinitiator and reducing agent, respectively.

Experimental radiopaque light-cured composite resins were prepared as follows. Spherical-shaped silica powder and the powder of one of the radiopaque materials were mixed as filler for the composite resin at radiopaque material contents of 0, 20, 40, 60, 80, and 100 vol%. Radiopaque material contents of 50 and 10 vol% were also mixed for SrCO_3 and Bi_2O_3 , respectively. Filler silanization was not performed. The filler was then mixed with the matrix resin at a filler content of 12.5 vol%. In this manner, 27 kinds of experimental radiopaque light-cured composite resin were prepared.

Specimens preparation

For aluminum equivalent thickness measurement, bar-shaped specimens ($2.0 \times 2.0 \times 25.0$ mm) were prepared. For half-value layer measurement, sheet-shaped specimens ($35.0 \times 35.0 \times 0.5$ mm and 35.0×35.0

$\times 0.1$ mm) were prepared.

Each bar-shaped specimen was fabricated using a polypropylene mold as follows. One experimental radiopaque light-cured composite resin was packed into the mold, covered with plastic strips and a glass slide, and irradiated for five minutes from both sides in a laboratory light-polymerizing unit (Labolight II, GC, Tokyo, Japan). Then, the bar-shaped specimen was polished to a thickness of 1.00 mm using a precise polishing machine (Dialap, Malto, Tokyo, Japan) with SiC paper (#600) under dry conditions. Three specimens were prepared for each composite resin at radiopaque material contents of 0, 20, 40, 60, 80, and 100 vol%.

Sheet-shaped specimens were fabricated using two $50 \times 50 \times 2$ mm polypropylene plates. Brass strip of 0.5 mm thickness or plastic strip of 0.1 mm thickness was placed on the polypropylene plate to create a 35×35 mm space. The experimental radiopaque light-cured composite resin was filled into this space, covered with the other polypropylene plate, and irradiated in the laboratory light-polymerizing unit for five minutes from both sides. Five sheets of each thickness were prepared with an experimental radiopaque light-cured composite resin at radiopaque material contents of 60 vol% Ti, 50 vol% SrCO_3 , 40 vol% ZrO_2 , 40 vol% BaSO_4 , and 10 vol% Bi_2O_3 . The aluminum equivalent thickness of these experimental radiopaque light-cured composite resins was approximately 1.0 mm.

The thickness of each specimen was measured using a micrometer (CPM15-25DM, Mitsutomo, Tokyo, Japan; minimum reading is 0.001 mm).

Aluminum equivalent thickness measurement

An aluminum step wedge (99.5% purity; step depth: 0.5 mm; thickness: 0.5 mm to 7.0 mm; width: 3.0 mm) was prepared as the reference material. A bar-shaped specimen and the aluminum step wedge were simultaneously irradiated by a dental X-ray unit (CCX AD 33, Trophy, France; tube voltage: 70 kV_p; tube current: 8 mA; exposure time: 0.5 seconds; focus-subject distance (FSD): 38 cm). The X-ray

Table 1 Materials used in the present study

Material	Product Name	Company
Barium sulfate	Barium sulfate (Grade 1)	Wako Pure Chemical Industries
Titanium powder	TMP-350 (purity: 99%)	Sumitomo Sitix Co.
Strontium carbonate	Strontium carbonate (purity: 95.0%)	Wako Pure Chemical Industries
Zirconium (IV) oxide	FZCA-4C (purity: 95.28%)	Tateho Chemical Industries Co.
Bismuth (III) oxide	Bismuth (III) oxide (Grade 1)	Wako Pure Chemical Industries
Spherical-shaped silica filler	PLV-6	Tatsumori Ltd.
UDMA	Art resin SH-500B	Negami Chemical
TEGDMA	3G	Shin Nakamura
Camphorquinone	Camphorquinone	Aldrich Chemical
Dimethylaminoethylmethacrylate	(2-dimethylamino) ethylmethacrylate	Wako Pure Chemical Industries

image was immediately recorded using an image sensor of an X-ray image capturing unit (Compuray, Yoshida, Tokyo, Japan) in tagged image file (TIFF) format. Images were analyzed using an image analyzing software (NIH Image ver. 1.62, National Institutes of Health, USA) on a personal computer (Power Mac G4, Apple, USA). Minimum and maximum gray levels were linearly scored in a range between 0 (white; X-ray was completely absorbed) and 255 (black; X-ray was completely passed). An area size – 20×20 pixels – was selected on each step of the aluminum step wedge and its mean gray level calculated. The same procedure was repeated for each specimen at three randomly selected areas. After which, a scatter diagram was plotted using the results of the thickness of the aluminum step wedge and its gray level. The aluminum equivalent thickness of each specimen was then calculated based on gray level using regression analysis of the aluminum step wedge. Effective atomic number (Z_e) was calculated using the atomic numbers ($Z_1, Z_2, \dots, Z_n$) of each element in the material and electron ratios ($a_1, a_2, \dots, a_n$) of the elements in the material by the following formula¹³⁾:

$$Z_e = (a_1 Z_1^{2.94} + a_2 Z_2^{2.94} + \dots + a_n Z_n^{2.94})^{1/2.94}$$

Half-value layer measurement

Half-value layer measurements were done using two types of dental X-ray unit (CCX AD 33, irradiation condition identical to that given above, and HI-EMIX D-RV-90, Tokyo Emix, Japan; tube voltage: 60 kV_p; tube current: 8 mA; exposure time: 0.5 seconds). The experimental schema is illustrated in Fig. 1.

Two lead plates were prepared. One was a 90×80×1.5 mm plate with a 1.5-cm diameter hole for an aperture, and the other was a 40×40×10 mm plate with a 2.0-cm diameter for a sample stage. An ioni-

zation chamber (660-4A, Victoreen, Moedling, Austria) with a scale meter (Victoreen 660, Victoreen, Moedling, Austria) was set at 30 cm above the ground. The sample stage was located at a distance of 65 cm from the ionization chamber. A specimen of selected thickness was then placed on the sample stage and covered by the other lead plate with the aperture. Distance between the specimen and the focus of the X-ray source was 35 cm.

Radiation exposure with and without the specimen was measured by the ionization chamber. When irradiation dose was half of that without specimen, the thickness of the radiopaque material was measured and recorded as the half-value layer of the composite resin. If the half-value layer was not determined using the prepared sheet, it was calculated using interpolation method. An aluminum half-value layer attenuator set (Al 99.9% purity; RMI 115A & RMI 115H, Gammex RMI, WI, USA) was used to determine the half-value layer of aluminum. Linear absorption coefficients of the experimental composite resins were thereby calculated.

In addition, half-value layers were subjected to statistical analysis using two-way ANOVA and Tukey's test ($\alpha=0.05$).

RESULTS

Aluminum equivalent thickness measurement

Fig. 2 shows the relationship between the thickness of the aluminum step wedge and the gray level. As shown, gray level of the aluminum step wedge increased proportionally with increasing aluminum thickness in the density profile.

Fig. 3 shows the relationship between the radiopaque material content of filler and aluminum equivalent thickness. As shown, the aluminum equivalent thickness of radiopaque materials in-

Fig. 1 Schematic illustration of half-value layer (HVL).

Fig. 2 Relationship between gray level and aluminum step wedge.

Fig. 3 Relationship between radiopaque material content and aluminum equivalent thickness.

creased as the radiopaque material content increased. Especially for Ti, the relation between radiopaque material content and aluminum equivalent thickness showed a sigmoid curve.

Radiopacity increased with increase in radiopaque material content. Table 2 shows the amount of radiopaque material calculated for aluminum equivalent thickness of 1.0 mm. When ranked in terms of the volumetric amount of radiopaque material calcu-

lated for 1.0-mm aluminum equivalent thickness, the materials were ranked as follows: $\text{Bi}_2\text{O}_3 < \text{BaSO}_4 = \text{ZrO}_2 < \text{SrCO}_3 < \text{Ti}$. When ranked in terms of effective atomic number, the same trend was observed. When ranked in terms of the mass amount of radiopaque material calculated for 1.0-mm aluminum equivalent thickness, the materials were ranked as follows: $\text{Bi}_2\text{O}_3 < \text{BaSO}_4 < \text{SrCO}_3 < \text{ZrO}_2 < \text{Ti}$. When ranked in terms of elemental mass%, the same trend was observed. When ranked in terms of linear absorption coefficient, the materials were ranked as follows: $\text{Ti} < \text{BaSO}_4 < \text{Bi}_2\text{O}_3 < \text{SrCO}_3 < \text{ZrO}_2$.

Half-value layer measurement

As shown in Table 3, half-value layers ranged from 1.32 to 2.89 mm. Two-way ANOVA revealed that X-ray unit and radiopaque material were significant. The half-value layer of CCX AD 33 was significantly higher than that of HI-EMIX D-RV-90. The half-value layers of Ti and BaSO₄ were significantly higher than that of the other radiopaque materials. On the other hand, the half-value layer of ZrO₂ was significantly smaller than that of the other radiopaque materials.

DISCUSSION

Ti, Sr, Zr, Ba, and Bi were selected as the radiopaque materials in the present study because they were already applied in clinically available dental materials

Table 2 The amount of the radiopaque material calculated for composite resin having 1.0-mm aluminum equivalent thickness

	Ti	SrCO ₃	ZrO ₂	BaSO ₄	Bi ₂ O ₃	Al*
Compound vol% of filler	60	50	40	40	10	
Compound mass% in composite resin	22.9	17.0	19.1	15.4	8.3	
Element mass% in composite resin	22.9	10.1	14.1	9.1	7.5	
Density of composite resin (d)	1.39	1.32	1.39	1.33	1.32	2.70**
Effective atomic number of composite	13.4	16.8	19.8	23.1	31.4	13
$Z^3 \times d$	3344	6259	10790	16394	40866	5932

* reference

**density of metal

Table 3 Half-value layer (HVL) of the radiopaque composite resins and the aluminum equivalent value of HVL ($\text{HVL}/\text{HVL}_{\text{Al}}$)

	Ti 60vol%	SrCO ₃ 50vol%	ZrO ₂ 40vol%	BaSO ₄ 40vol%	Bi ₂ O ₃ 10vol%	Al*
CCX AD 33						
HVL	2.89	1.91	1.36	2.80	2.02	2.10
$\text{HVL}/\text{HVL}_{\text{Al}}$	1.38	0.91	0.65	1.33	0.96	
μ	2.40	3.63	5.10	2.48	3.43	3.30
HI-EMIX D-RV-90						
HVL	2.64	1.71	1.32	2.49	1.77	1.90
$\text{HVL}/\text{HVL}_{\text{Al}}$	1.39	0.90	0.69	1.31	0.93	
μ	2.63	4.05	5.25	2.78	3.92	3.65

*reference

to provide radiopacity. Ti, SrCO₃, ZrO₂, BaSO₄, and Bi₂O₃ were chosen because these compounds are chemically stable, non-toxic, and composed of other elements that have a small radiopaque effect.

Silanization is well known to improve the interfacial properties of fillers and to increase the maximum amount of filler content. While there have been several reports on silanization of silica particles^{14,15}, silanization of the compounds used in the present study has not been fully investigated. Moreover, the effect of silanization on radiopacity has not been clearly explained. On these grounds, the radiopaque materials used in the present study were not silanized. As a result, filler content with the experimental radiopaque light-curing composite resin was controlled at a relatively small amount of 12.5 vol% — because if filler content were to exceed 12.5 vol% of BaSO₄, the consistency would not be uniform.

In the present study, volumetric filler content of radiopaque material was used versus the mass% generally used for the filler content of dental composite resins^{16–18}. The radiopacity of a material is evaluated by the change in intensity of the radiation transmitted, which is affected by three material-related factors: density, thickness, and the atomic number of the elements which constitute the material. On these grounds, it is very important to evaluate the radiopacity of a material in terms of elemental density in unit volume.

Two X-ray units of different tube voltages were used. In the USA and European countries, X-ray units with 70 kV tube voltage are popular, while in Japan X-ray units with 60 kV tube voltage are popular¹⁰. The linear absorption coefficients of aluminum obtained by CCX AD 33 and HI-EMIX D-RV-90 were calculated to be 3.30 and 3.65 cm⁻¹, respectively. The density (ρ) of aluminum is 2.698 g/cm³ at 25°C. According to the diagram of effective energy and mass attenuation coefficient (μ/ρ)¹⁹, the mass attenuation coefficient of aluminum is 3.441 cm²/g at 20 keV and 1.128 cm²/g at 30 keV. Therefore, the effective energy of CCX AD 33 and HI-EMIX D-RV-90 were computed as 29.64 keV and 29.04 keV, respectively. Thus, the difference in effective energy between the two dental X-ray units was actually small in spite of different tube voltages. This could be explained by the different rectifying system of the two X-ray units: CCX AD 33 used a full-wave rectifier, while HI-EMIX D-RV-90 used an inverter. Therefore, HI-EMIX D-RV-90 showed a relatively high effective energy in spite of lower tube voltage.

The most popular X-ray detector in dentistry is the intraoral film, which is usually sensitive to X-ray beams at 40 keV²⁰. However, the sensitivity of the ionization chamber is almost independent of the energy used in clinical diagnoses. The sensitivity of the CCD sensor used was not clearly elucidated;

however, its sensitivity to X-ray energy might be similar to that of intraoral film. The gray scale of the CCD image was modified to increase in proportion with increase in aluminum thickness, according to the result of Fig. 3. The effective energy of dental X-ray units used in the present study was approximately 30 keV. The radiopacity results of aluminum equivalent thickness using the CCD sensor and those of half-value layer using the ionization chamber did not agree because the sensitivities of the X-ray detectors, CCD sensor, and ionization chamber were different. Radiopacity results might also change if intraoral film had been used for evaluation.

The density profile of aluminum showed a straight line. However, the aluminum equivalent thickness of experimental composites did not proportionally increase with increase in radiopaque material content. The edge effect of aluminum at 1.6 keV is not observed in the X-ray energy range used in clinical diagnoses. However, Ti, Sr, Zr, and Ba show the K-edge effect at 5.0, 16.1, 18.0, and 37.4 keV, respectively¹⁹. Further, the characteristics of CCD sensor were not disclosed. For these reasons, aluminum equivalent thickness did not increase proportionally with the amount of radiopaque material. In other words, the aluminum equivalent thickness of the experimental composite resins in Table 2 would not be 2 mm if the thickness of the experimental composite resins were 2 mm.

The aluminum equivalent thicknesses of 1-mm thick dentin and enamel were reported as 0.79 and 2.22 mm, respectively¹¹. Those of the 1-mm thick commercial composite resins reported in earlier papers were 1.65–2.52¹¹, 1.40–3.00⁸, and 0.73–3.72 mm¹⁰. The aluminum equivalent thicknesses of the experimental composite resins containing radiopaque materials, except those with higher Bi₂O₃ content, were within the range of previous reports. As shown in Fig. 3, Ti, SrCO₃, ZrO₂, BaSO₄, and Bi₂O₃ should constitute about 50, 30, 30, 30, and 8 vol%, respectively for the aluminum equivalent thickness of dentin, and 60, 40, 40, 40, and 10 vol%, respectively for the aluminum equivalent thickness of 1 mm.

Atomic numbers — which affect radiopacity — of Ti, Sr, Zr, Ba, and Bi are 22, 38, 40, 56, and 83, respectively. The measured aluminum equivalent thickness increased as element's atomic number increased. However, when comparing the half-value layers of ZrO₂ and BaSO₄, ZrO₂ showed greater radiopacity despite the same volumetric content, similar density, and smaller effective atomic number and density. This result agreed with a previous paper which measured the aluminum equivalent thickness of commercial products¹⁰. However, other elements contained in these commercial products might influence the reported result.

The effects of elemental composition on radiopacity evaluated in terms of aluminum equivalent

lent thickness and by half-value layer were not identical. Nevertheless, the common underlying trend was that radiopacity increased with a greater amount of radiopaque material content and with a greater atomic number of the constituent element. Photoelectric absorption is related to $Z^3 \times d^{13}$. Therefore, radiopacity of a material should be inversely proportional to $Z^3 \times d$. However, both aluminum equivalent thickness and half-value layer did not obey this theory. This was most probably due to the K-edge effect and the different sensitivities of detectors.

In the present study, bismuth was the most radiopaque material. However, it is not used for composite resin fillers probably because of its poor mechanical properties in glass. Barium glass is known to hydrate easily²¹, and the contents of Ti and Zr in glass are limited because of their high melting points. Therefore, the content of these radiopaque materials for glass filler of composite resins is somewhat limited.

Aluminum equivalent thickness measured using intraoral films might provide sufficient information for diagnoses in dental clinics. However, it should be borne in mind that aluminum equivalent thickness is easily influenced by the type of intraoral film used and by the irradiation conditions of the dental X-ray unit.

CONCLUSIONS

Considering the limitations of this study, the following conclusions were drawn:

- 1) Aluminum equivalent thickness of the experimental composite resins increased as the amount of radiopaque material content increased.
- 2) Aluminum equivalent thickness of the experimental composite resins depended on the atomic number of the constituent element.
- 3) Half-value layer of the five experimental composite resins varied according to the radiopaque material used.

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