Radiopacity measurements of direct and indirect resin composites at different thicknesses using digital image analysis

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INTRODUCTION

Direct resin composite restorative materials (here in after referred to as “composites”) are widely used in dentistry for their optical and mechanical properties that closely resemble the natural teeth and also for their excellent adhesion to enamel and dentin1,2. Indirect composites considerably differ from direct composite systems in their monomer compositions and polymerization processes. Direct composites are polymerized using light sources which typically are quartz-tungsten-halogen lamps or light-emitting diode curing lights. Indirect composites, on the other hand, are polymerized using laboratory polymerization units. Indirect composites are cured outside the mouth, hence they allow high-energy ultraviolet (UV) light to be used to perform this extraoral polymerization procedure3).

Recently, nanofilled composites were developed because of increasing demand for a universal restorative material indicated for all types of direct restorations4,5). New microhybrid composites have a high filler content and considerably differ in form, size, and composition from the earlier generation of indirect composites1,2). Nanocomposites have increased filler loadings, reduced organic matrices and consequently, improved mechanical, physical, and optical characteristics6).

In principle, restorative materials should be radiopaque enough to be detected against enamel and dentin. In addition, radiopacity of composites serves to support the detection of secondary caries, marginal defects, restoration contours, missing contact points with adjacent teeth, cement overhangs, or interfacial gaps8-11). On the other hand, excessive radiopacity may reduce the ability to diagnose recurrent caries and other defects10,12,13). The radiopacity level of composites is affected by several factors, one of which is the type of filler particles (namely, glass and ceramic particles containing heavy metals such as aluminum, barium, strontium, and zirconium)9). Excessive incorporation of radiopaque fillers in the resin matrix results in reduced translucency of composites, but at the same time affects mechanical properties8,14). Apart from causing opacity, radiopaque particles also largely increase thermal expansion and hydrolyze silane bonding agents15).

Radiopacity of dental materials is usually determined by comparing with the radiopacity values of enamel, dentin, and aluminum16). While some authors stated that the radiopacity of any dental material should be equal to or higher than the radiopacity of dentin10,17), others suggested that restorative materials should have radiopacity values equivalent to or greater than that of enamel17,18). It was shown that the radiopacity of dentin was approximately equivalent to that of aluminum (Al) of the same thickness, and that enamel had approximately twice the radiopacity of Al at the same thickness19). According to the International Organization for Standardization (ISO), the radiopacity of a dental material is expressed as an optical density value or in terms of equivalent aluminium (Al) thickness (in millimeters) using a reference calibration curve under controlled radiographic conditions20). Accordingly, resin-based materials should have radiopacity equal to or greater than that of aluminium Al18).

Several studies in published literature have evaluated the radiopacity of composites7-19). To the best of our knowledge, however, none has compared the
radiopacity of direct and indirect composites. Composites differ in several respects of their composition: resin matrix, dispersed fillers, filler content, pigments, modifiers, and radiopaque oxides. These differences may influence the radiopacity level of direct and indirect composites.

The objective of this study was to evaluate the radiopacity levels of direct and indirect composites as a function of thickness, using digital radiography and pixel gray-scale measurement. The hypothesis tested was that the radiopacity of direct and indirect composites would not be affected by different filler loadings and resin matrices and at different thicknesses.

MATERIALS AND METHODS

Preparation of composite specimens

Seven direct composites (Aelite LS Posterior, Aelite All-Purpose Body, Quadrant Universal LC, Clearfil Majesty Posterior, Clearfil Majesty Esthetic, Filtek Ultimate Dentin, IPS Empress Direct Dentin) and six indirect composites (Ceromega, Epricord, Estenia C&B, Tescera, Signum Ceramis, Solidex) were selected for this study. Their chemical compositions, manufacturers, and batch numbers are listed in Table 1. Disk-shaped specimens (N=260, n=10 per group) of shade A2 and 6 mm diameter were fabricated in two thicknesses: 1 and 2 mm.

Specimens were prepared using a stainless steel mold in one increment. To prevent void formation, a glass plate was gently pressed over the mold before the latter was removed. Then, specimens were photo-polymerized using an LED light source (Elipar Freelight 2, 3M ESPE, St. Paul, USA) with an intensity of 1,000 mW/cm² and a wavelength of 430–450 nm for 40 s from each direction. Post-polymerization of indirect composites was performed in a laboratory polymerization unit (Blue Thunder, Toei Electric Co., Ltd., Kanagawa, Japan). Operating conditions were 250 W with three conventional halogen lamps at a wavelength of 350–550 nm for 3 min.

All specimens were finished using 600- and 1,000-grit silicon carbide papers (Struers, Willich, Germany) and cleaned ultrasonically in 70% ethyl alcohol. Their thicknesses were measured using a digital micrometer (Mitutoyo America Corporation, Aurora, IL, USA) to verify that the thickness remained within a critical tolerance of 0.01 mm. All composite specimens were stored dry in darkness at room temperature for 24 h until performance of radiopacity measurement.

Preparation of tooth specimens

Premolar extracted not longer than 6 months ago for orthodontic reasons was used for enamel/dentin specimens. Enamel and dentin (1 and 2 mm each) slices were prepared by sectioning the tooth longitudinally using a slow-speed diamond saw (Isomet, Buehler Ltd., Lake Bluff, IL, USA) under water cooling. Tooth specimens were stored in water.

Radiopacity measurement

Composite specimens were positioned at the center of a phosphor storage plate (PSP; Digora, Soredex, Orion, Helsinki, Finland) alongside the tooth slice and Al stepwedge. The Al stepwedge was made of 1100 alloy, with thicknesses varying from 1 to 10 mm in uniform steps of 1 mm each. A custom-made acrylic device was used to standardize the focus-to-film distance (30 cm) and the positions of both the PSP and head of X-ray machine, so that central X-ray beam was directed at a 90° angle to the PSP surface. For radiographic exposure, a dental X-ray unit (Trophy, Vincennes, France) operating at 70 kV and 8 mA was used. Exposure time was 0.2 s.

Digital images were obtained by scanning PSPs using a Digora scanner. The same PSP was used for all X-ray exposures to preclude differences among the plates. On each radiographic image, regions of interest (ROIs) of 50×50 pixels were selected at the center of each specimen and on each step of the Al stepwedge. Mean gray values (MGVs) of each ROI were measured by using the histogram function of an image analysis program (Image-Pro Plus 4.0, Media Cybernetics Inc., Silver Spring, MD, USA). The MGV of each pixel was represented in a scale ranging from 0 (black) to 255 (white). Radiopacity measurement was repeated three times, and the mean MGV thereby calculated. One operator who was blinded to the material type made the measurements.

A calibration curve was generated from the MGVs of Al stepwedge steps by using a software (Curve Expert 1.3). MGVs of specimens were then converted into equivalent thickness of Al using a formula derived from that curve.

Statistical analysis

Statistical analysis was performed using SPSS for Windows, Version 11.0 (SPSS Inc., Chicago, IL, USA). One-way and two-way analyses of variance (ANOVA) were used to detect significant differences between the materials. Tukey’s test was performed for interactions and post hoc multiple comparisons. Logarithmic regression model equations were calculated for MGVs and Al thicknesses. P values less than 0.05 were considered to be statistically significant in all tests.

RESULTS

Table 2 lists the mean and standard deviation values of the radiopacity of investigated materials in two different thicknesses, 1 and 2 mm. Radiopacity was significantly affected by the factors of composite type (p<0.05) and thickness (p<0.001). Interaction between these two factors was also significant (p<0.001). For both thicknesses, there were statistically significant differences (p<0.05) between direct composite group (1 mm: 1.69–5.58 mmAl; 2 mm: 2.71–9.38 mmAl) and indirect composite group (1 mm: 1.23–5.5 mmAl; 2 mm: 1.84–9.16 mmAl). The radiopacity values of all 2-mm-thick specimens were higher than those of 1 mm.

Except for Epricord (1.22, 1.84 mmAl), the
<table>
<thead>
<tr>
<th>No.</th>
<th>Brand</th>
<th>Manufacturer (Batch number)</th>
<th>Type (Direct/Indirect)</th>
<th>Organic Matrix</th>
<th>Filler</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Aelite All-Purpose Body</td>
<td>Bisco Inc., Schaumburg, IL, USA (120015017)</td>
<td>Microhybrid (Direct)</td>
<td>Ethoxylated Bis-DMA, TEGDMA</td>
<td>Glass filler, amorphous silica (76 wt%)</td>
</tr>
<tr>
<td>2</td>
<td>Aelite LS Posterior</td>
<td>Bisco Inc. (120013252)</td>
<td>Hybrid (Direct)</td>
<td>Ethoxylated Bis-GMA</td>
<td>Glass filler, amorphous silica (88 wt%)</td>
</tr>
<tr>
<td>3</td>
<td>Clearfil Majesty Esthetic</td>
<td>Kuraray Medical Inc., Tokyo, Japan (0061B)</td>
<td>Microhybrid (Direct)</td>
<td>Bis-GMA, TEGDMA</td>
<td>Barium glass, silica (85.5 wt%)</td>
</tr>
<tr>
<td>4</td>
<td>Clearfil Majesty Posterior</td>
<td>Kuraray Medical Inc. (00115C)</td>
<td>Nanohybrid (Direct)</td>
<td>Bis-GMA, TEGDMA, ArDMA</td>
<td>Alumina, glass-ceramics (92 wt%)</td>
</tr>
<tr>
<td>5</td>
<td>Filtek Ultimate Dentin</td>
<td>3M ESPE, St. Paul, MN, USA (N251021)</td>
<td>Nano (Direct)</td>
<td>Bis-GMA, UDMA, Bis-EMA, TEGMA, PEGDMA</td>
<td>Nonaggregated silica, nonaggregated zirconia, aggregated silica/zirconia (78.5 wt%)</td>
</tr>
<tr>
<td>6</td>
<td>IPS Empress Direct Dentin</td>
<td>Ivoclar Vivadent, Schaan, Liechtenstein (P38261)</td>
<td>Nanohybrid (Direct)</td>
<td>Dimethacrylate</td>
<td>Barium, alumina, fluoroalumina, zirconia, mixed oxide, ytterbium trifluoride (81.2 wt%)</td>
</tr>
<tr>
<td>7</td>
<td>Quadrant Universal LC</td>
<td>Cavex, Haarlem, The Netherlands (S010401C)</td>
<td>Microfilled (Direct)</td>
<td>Bis-GMA, TEGDMA</td>
<td>Barium, alumina, silica, fluoride fillers (72 wt%)</td>
</tr>
<tr>
<td>8</td>
<td>Ceromega</td>
<td>Shofu Inc., Kyoto, Japan (051274)</td>
<td>Microhybrid (Indirect)</td>
<td>UDMA, Urethane diacrylate</td>
<td>Zirconium silicate (73 wt%)</td>
</tr>
<tr>
<td>9</td>
<td>Epricord</td>
<td>Kuraray Medical Inc. (00115A)</td>
<td>Hybrid (Indirect)</td>
<td>UTMA, TEGDMA,</td>
<td>Organic filler, glass, silica (85.4 wt%)</td>
</tr>
<tr>
<td>10</td>
<td>Estania C&amp;B</td>
<td>Kuraray Medical Inc. (000433)</td>
<td>Hybrid (Indirect)</td>
<td>UTMA, Methacrylate</td>
<td>Surface treated alumina micro filler, silanated glass ceramic filler (92 wt%)</td>
</tr>
<tr>
<td>11</td>
<td>Tescrea</td>
<td>Bisco Inc. (009098)</td>
<td>Hybrid (Indirect)</td>
<td>Ethoxylated bis-GMA, UDMA</td>
<td>Glass, amorphous silica (81 wt%)</td>
</tr>
<tr>
<td>12</td>
<td>Signum Ceramis</td>
<td>Heraeus-Kulzer, Hanau, Germany (010143)</td>
<td>Nanohybrid (Indirect)</td>
<td>Resin based on multifunctional methacrylic esters</td>
<td>Glass-ceramic filler, nanoparticles, silica inorganic filler (73 wt%)</td>
</tr>
<tr>
<td>13</td>
<td>Solidex</td>
<td>Shofu Inc. (031248)</td>
<td>Microhybrid (Indirect)</td>
<td>Bis-GMA, UDMA, TEGMA</td>
<td>Silicon dioxide, aluminum dioxide (53 wt%)</td>
</tr>
</tbody>
</table>

Bis-DMA: Bisphenol A dimethacrylate; TEGDMA: Triethyleneglycol dimethacrylate; Bis-GMA: Bisphenol A diglycidyl methacrylate; ArDMA: Aromatic dimethacrylates; UDMA: Urethane dimethacrylate; Bis-EMA: Bisphenol A diethoxymethacrylate; TEGMA: Triethylene glycol dimethacrylate; PEGDMA: Polyethylene glycol dimethacrylates; UTMA: Urethane tetramethacrylate.

Radiopacity values of all composites were greater than dentin (1.23, 2.24 mmAl). No statistically significant differences were identified between Aelite All-Purpose Body (1.68, 2.70 mmAl), Epricord and dentin (1.23, 2.24 mmAl). Among the composites, IPS Empress Direct Dentin (5.58, 9.38 mmAl)
and Estenia C&B (5.49, 9.16 mmAl) showed significantly higher radiopacity ($p<0.05$), followed by Clearfil Majesty Posterior (4.24, 8.19 mmAl), Quadrant Universal LC (3.76, 7.67 mmAl), Filtek Ultimate Dentin (3.74, 6.96 mmAl), Signum Ceramis (3.25, 6.09 mmAl), Ceromega (2.74, 4.68 mmAl), Tescera (2.59, 4.44 mmAl), and Aelite LS Posterior (2.53, 5.22 mmAl) in a descending order.

Radiopacity of 1-mm-thick Clearfil Majesty Esthetic (1.97 mmAl) was slightly lower than that of enamel (2.20 mmAl), but 2-mm-thick Clearfil Majesty Esthetic (4.47 mmAl) was slightly higher than that of enamel (4.05 mmAl) with no significant differences ($p>0.05$).

**DISCUSSION**

This study evaluated the radiopacity of direct and indirect composites with different chemical compositions as a function of thickness, using digital radiography and pixel gray-scale measurement. Since the results were significantly affected by composite type and thickness, the null hypothesis was rejected.

Radiopacity of composites generally depends on the content percentage and type of fillers$^{12,23,24}$. Composites composed of fillers with low atomic numbers (such as silicone and alumina) appear radiolucent, whereas materials having fillers with high atomic numbers (such as zinc, strontium, zirconia, barium glass, barium sulfate, lanthanum, and ytterbium) appear radiopaque$^{12,24,25}$. For both direct and indirect composites, when filler volume reaches or exceeds 70% and the amount of opacifiers in filler particles is raised above 20%, their

<table>
<thead>
<tr>
<th>No.</th>
<th>Materials</th>
<th>Mean mmAl (SD) 1 mm</th>
<th>Mean mmAl (SD) 2 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>IPS Empress Direct</td>
<td>5.58±0.57$^a$</td>
<td>9.38±0.33$^a$</td>
</tr>
<tr>
<td>2</td>
<td>Estania</td>
<td>5.49±0.82$^a$</td>
<td>9.16±0.52$^a$</td>
</tr>
<tr>
<td>3</td>
<td>Clearfil Majesty Posterior</td>
<td>4.24±0.38$^b$</td>
<td>8.19±0.36$^b$</td>
</tr>
<tr>
<td>4</td>
<td>Quadrant Universal LC</td>
<td>3.76±0.52$^{bc}$</td>
<td>7.67±0.47$^{bc}$</td>
</tr>
<tr>
<td>5</td>
<td>Filtek Ultimate Dentin</td>
<td>3.74±0.66$^{bd}$</td>
<td>6.96±0.47$^c$</td>
</tr>
<tr>
<td>6</td>
<td>Signum Ceramis</td>
<td>3.25±0.80$^{c,d,a}$</td>
<td>6.09±0.98$^{c}$</td>
</tr>
<tr>
<td>7</td>
<td>Ceromega</td>
<td>2.74±0.48$^{f}$</td>
<td>4.68±0.60$^{d}$</td>
</tr>
<tr>
<td>8</td>
<td>Tescera</td>
<td>2.59±0.52$^{e,i}$</td>
<td>4.44±0.63$^{d}$</td>
</tr>
<tr>
<td>9</td>
<td>Aelite LS Posterior</td>
<td>2.53±0.49$^{h}$</td>
<td>5.22±0.41$^{d}$</td>
</tr>
<tr>
<td>10</td>
<td>Enamel</td>
<td>2.20±0.17$^{g,k}$</td>
<td>4.05±0.18$^{d}$</td>
</tr>
<tr>
<td>11</td>
<td>Clearfil Majesty Esthetic</td>
<td>1.97±0.44$^{k,l,i}$</td>
<td>4.47±0.22$^{d}$</td>
</tr>
<tr>
<td>12</td>
<td>Solidex</td>
<td>1.86±0.48$^{k,l}$</td>
<td>2.86±0.38$^{e}$</td>
</tr>
<tr>
<td>13</td>
<td>Aelite All-Purpose</td>
<td>1.68±0.78$^{k,l,k}$</td>
<td>2.70±0.52$^{e}$</td>
</tr>
<tr>
<td>14</td>
<td>Dentin</td>
<td>1.23±0.13$^{i,k}$</td>
<td>2.24±0.14$^{f}$</td>
</tr>
<tr>
<td>15</td>
<td>Epricord</td>
<td>1.22±0.60$^{i,k}$</td>
<td>1.84±0.40$^{f}$</td>
</tr>
</tbody>
</table>
radiopacity levels might exceed that of human enamel or other conventional direct composites with a lower amount of fillers.

However, fillers alone are not responsible for the radiopacity levels of composites. Radiopaque monomers also enhance radiopacity as they bond to the polymer matrix so that at the same time they improve the mechanical properties. According to the results of this study, IPS Empress Direct Dentin direct composite and Estenia C&B indirect composite showed the highest radiopacity. The high radiopacity level of IPS Empress Direct Dentin, which is a nanohybrid composite, could be attributed to having barium (Ba atomic number 56) and ytterbium (Yb atomic number 70) in its composition as fillers. As for Estenia C&B, it had a higher percentage of 2-μm nanoceramic microfillers at 92 wt%. Results of this study agreed with those of Sabbagh et al. Using a PSP-based digital system (Digora) to measure radiopacity, they reported a linear correlation between radiopacity and the percentage of fillers by weight.

Epricord, a hybrid composite which had no fillers of high atomic weight (according to information provided by manufacturer), showed the lowest radiopacity. Clearfil Majesty Esthetic, Solidex, and Aelite All-Purpose Body had radiopacity values ranging between dentin and enamel, and their fillers included barium, glass, silica, silicon dioxide, aluminum oxide, and amorphous silica. Interestingly, Clearfil Majesty Esthetic had a lower filler percentage (85.5 wt%) than Clearfil Majesty Posterior (92 wt%), but it showed higher radiopacity. In this case, the higher radiopacity could be attributed to Clearfil Majesty Esthetic having a higher quantity of barium glass-based radiopaque fillers. Both materials were produced by the same manufacturer, and results of this study agreed with the radiopacity values provided by the manufacturer.

In clinical situations, diagnostic challenges arise when radiographic images present barely discernible radiopacity differences between dental tissues and restorative materials. According to Cruz et al., an ideal radiopacity for dental composites is one that is closer to the dentin image and which ultimately produces a similar attenuation to that of sound dentin tissue. Excessively radiopaque restorations may hinder a clinician’s ability to spot marginal defects due to the Mach effect. The latter phenomenon is a visual illusion which enhances the contrast between two areas of different radiopacities, making the dark border area darker. This effect might be misinterpreted as pathosis in certain situations.

In the current study, IPS Empress Direct Dentin, Estenia C&B, Clearfil Majesty Posterior, Quadrant Universal LC, Filtek Ultimate Dentin, and Signum Ceramis showed higher radiopacity than enamel. According to ISO 4049, radiopacity of dental composites should exceed that of dentin but should present radiopacity values equal to or higher than that of the same thickness of aluminum. One limitation of this guideline is that no definitive upper limit for radiopacity has been established. This is one aspect which warrants further research.

Various methods for radiopacity measurements of restorative materials have been explored, such as conventional X-ray films assessed by an optical densitometer, digitized dental films, or digital radiographic films evaluated by digital image analysis programs. Digital radiography has produced reproducible and consistent results when compared among different radiopacity studies. This is probably because it does not involve film development, which may otherwise influence the final radiographic image and produce varied radiopacity results. Moreover, digital image analysis allows numerical values to be obtained. In the present study, the Digora system was chosen because it has been described as an effective method in similar radiopacity studies. As described in previous studies, the radiopacity values of resin composites obtained with the Digora system in this study were correlated with conventional X-ray films and pixel values were converted into equivalent thickness of aluminum in millimeters (mmAl).

When choosing resin composite materials for restorations, especially for patients with high caries risk, clinicians should take into consideration the radiopacity levels of the desired filler types and resin matrices. With an increase in minimally invasive applications that involve composites, the radiopacity findings of this study stemming not only from 2-mm-thick specimens, but also from 1-mm-thick specimens, bear considerable clinical significance.

CONCLUSIONS

Based on the results of this study, the following conclusions were drawn:

1. Radiopacity of direct and indirect resin composite materials varied according to material type, brand, and thickness, where 2-mm-thick composites showed more radiopacity.
2. All the tested composite materials, with the exception of Epricord, showed radiopacity that met the ISO 4049 standard and presented radiopacity values higher than dentin.
3. Among the direct composites, IPS Empress Direct Dentin (nanohybrid) showed the highest radiopacity and Aelite All-Purpose Body (hybrid) the lowest. Among the indirect composites, Estenia C&B (hybrid) showed the highest radiopacity and Epricord (hybrid) the lowest.

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2) Tay FR, Wei, SHY. Indirect posterior restorations using a new chairside microhybrid resin composite system. Adhesive Dent 2001; 3: 89-99
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