

Evaluation of Radiopacity of Bulk-fill Flowable Composites Using Digital Radiography

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Clinical Relevance

The radiopacity values of flowable bulk-fill composite materials are greater than those of enamel, dentin, and most of the conventional flowable composites.

SUMMARY

New flowable composites that may be bulk-filled in layers up to 4 mm are indicated as a base beneath posterior composite restorations. Sufficient radiopacity is one of the several important requirements such materials should meet. The aim of this study was to evaluate the radiopacity of bulk-fill flowable composites and to provide a comparison with conventional flowable composites using digital imaging. Ten standard specimens (5 mm in diameter, 1

mm in thickness) were prepared from each of four different bulk-fill flowable composites and nine different conventional flowable composites. Radiographs of the specimens were taken together with 1-mm-thick tooth slices and an aluminum step wedge using a digital imaging system. For the radiographic exposures, a storage phosphor plate and a dental x-ray unit at 70 kVp and 8 mA were used. The object-to-focus distance was 30 cm, and the exposure time was 0.2 seconds. The gray values of the materials were measured using the histogram function of the software available with the system, and radiopacity was calculated as the equivalent thickness of aluminum. The data were analyzed statistically ($p < 0.05$). All of the tested bulk-fill flowable composites showed significantly higher radiopacity values in comparison with those of enamel, dentin, and most of the conventional flowable composites ($p < 0.05$). Venus Bulk Fill (Heraeus Kulzer) provided the highest radiopacity value, whereas Arabesk Flow (Voco) showed the lowest. The order of the radiopacity values for the bulk-fill flowable composites was as follows: Venus Bulk Fill (Heraeus Kulzer) \geq X-tra Base (Voco) $>$ SDR (Dentsply DeTrey) \geq Filtek Bulk Fill (3M ESPE). To conclude, the bulk-fill flowable

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restorative materials, which were tested in this study using digital radiography, met the minimum standard of radiopacity specified by the International Standards Organization.

INTRODUCTION

The use of “flowables”, which are low-viscosity composites obtained from formulations with a 20%-25% lower filler loading and a greater portion of diluent monomers than that of conventional composites, as a liner or base beneath posterior composite restorations certainly is not a new concept. However, the first-generation flowable composites were not suitable for full-depth posterior fillings, primarily because of their lower filler content, which leads to inferior mechanical properties and increased volumetric shrinkage compared to conventional paste-like composites. Basically, they could only be applied as a liner or sealer or used to restore very small cavities.¹⁻³ As the dental industry continued its search for materials with improved properties, the latest generations of flowable composite resins have higher filler content and increased mechanical properties and are recommended for use as a base beneath larger posterior composite resin restorations.⁴

To further simplify the restorative procedure and to save precious chair-side time, the latest trend in composite technology is the development of flowable restorative composites with enhanced curing, shrinkage, and physical properties that can be bulk-filled in layers up to 4 mm in thickness and replace dentin in a single increment.⁵⁻⁹ Although the concept sounds quite simple, there are several important requirements that a material must meet for this particular indication, such as depth of cure, specialized handling, low polymerization shrinkage stress, fracture toughness, and radiopacity.⁶

Radiopacity is one of the prerequisites of dental materials, especially restorative composite resins, because it can be used to evaluate the proximity of pulp and marginal adaptation, to detect secondary caries, and to assess the overall quality of a restoration, such as its adaptation, integrity, interproximal contours, contacts, overhangs, and voids.¹⁰⁻¹⁴ The radiopacity of a dental material is usually expressed as the equivalent aluminum (Al) thickness (in millimeters) because the radiopacity of Al has been reported to be similar to that of dentin.^{15,16} According to the International Standards Organization (ISO) 4049,¹⁷ in order to be deemed radiopaque, the radiopacity of a composite material should be equal to or greater than that of Al (with $\geq 98\%$ purity) with

the same thickness. On the other hand, some researchers recommend that the radiopacity of a composite material should be equal to or slightly higher than that of enamel, the Al equivalence of which is approximately twice the Al equivalence of dentin, particularly for posterior restorations, for an optimum contrast and the ideal detection of secondary caries in radiographs.^{10-13,16,18,19}

The obvious attraction of bulk-fill flowable composites, which enable the practitioner to save precious chair-side time as a result of quicker placement of restorations and improved material handling, has rapidly made them very popular; however, the number of *in vitro* and clinical studies that evaluate their properties is still limited. In this context, available studies are focused on polymerization shrinkage,⁷ depth of cure,⁹ marginal quality,⁸ flexural strength,⁹ bond strength,²⁰ microleakage,²¹ creep deformation,²² and surface morphology.²³ Although continuance of the radiopacity studies of materials is critical in order to evaluate new materials that are introduced in the market and to prevent any interpretation errors during radiographic diagnosis,¹³ to our knowledge there are no studies that address the radiopacity of bulk-fill flowable composites. Thus, the aim of this study was to investigate the radiopacity of four commercially available bulk-fill flowable composites and to compare it with those of conventional flowable composites using digital imaging.

METHODS AND MATERIALS

Specimen Preparation

Table 1 presents the brand names, manufacturers, lot numbers, filler types, and filler loadings of the bulk-fill flowable and conventional flowable composites evaluated in the current study.

Ten standard specimens (5 mm in diameter, 1 mm in thickness) were prepared from each of four different bulk-fill flowable and nine different conventional flowable composites using Plexiglas plates containing wells. The specimens were prepared in one increment and in accordance with the instructions provided by the manufacturers. In order to prevent void formation, the Plexiglas plate was placed on a glass microscope slide, the materials were placed in the mold until it was overfilled, and then another glass microscope slide was gently pressed over the mold of each material to flatten the surface. The specimens were then light-cured using a LED light source (D-Lux, DiaDent, South Korea) with an intensity of 1100 mW/cm² and a

Table 1: *The Brand Names, Manufacturers, Lot Numbers, Filler Types, and Filler Loadings of the Tested Bulk-fill Flowable and Conventional Flowable Composites*

Brand Name	Type of Material	Manufacturer	Lot No. and Shade	Filler Type and Loading, %wt/%vol
SDR	Bulk-fill flowable	Dentsply DeTrey, Konstanz, Germany	1105141 U	Barium-alumino-fluoro-borosilicate glass, strontium alumino-fluorosilicate glass 68/45
Filtek Flowable Bulkfill	Bulk-fill flowable	3M ESPE, St Paul, MN, USA	N402927 A2	Zirconia/silica, ytterbium trifluoride 64.5/42.5
X-tra Base	Bulk-fill flowable	Voco, Cuxhaven, Germany	1246488 A2	Barium glass, ytterbium trifluoride, fumed silica 75/58
Venus Bulk Fill	Bulk-fill flowable	Heraus Kulzer GmbH, Hanau, Germany	010100 U	Barium-alumino-fluorosilicate glass, ytterbium trifluoride, silica 65/38
Vertise Flow	Self-adhering flowable composite	Kerr, Orange, CA, USA	2970580 B1	Barium glass, colloidal silica, ytterbium fluoride 70/48
Filtek Ultimate Flow	Flowable composite	3M ESPE, St Paul, MN, USA	N431040 A2	Ytterbium trifluoride, silica, zirconia/silica 65/46
Charisma Opal Flow	Flowable composite	Heraus Kulzer GmbH, Hanau, Germany	010105 A2	Barium-alumino-fluorosilicate glass, ytterbium trifluoride, silica 65/41
X-Flow	Flowable composite	Dentsply DeTrey, Konstanz, Germany	1202000075 B1	Strontium-alumino-sodium-fluoro-phosphorsilicate glass, silica, iron pigments, titanium dioxide 60/38
Arabesk Flow	Flowable composite	Voco, Cuxhaven, Germany	1247249 A3	Barium-strontium-borosilicate, silica 64/50.3
Grandio Flow	Flowable composite	Voco, Cuxhaven, Germany	1247340 A2	Barium-alumina borosilicate, silica 80/65.6
GrandioSo Flow	Flowable composite	Voco, Cuxhaven, Germany	1249058 A2	Glass ceramic fillers, silica 81/65
GrandioSo Heavy Flow	Flowable composite	Voco, Cuxhaven, Germany	1247548 A2	Glass ceramic fillers, silica 83/68
Esthet-X Flow	Flowable composite	Dentsply DeTrey, Konstanz, Germany	110124 A2	Barium-fluoro-alumino-borosilicate glass, silica 61/53

wavelength of 420-490 nm for 20 seconds (in accordance with the manufacturer directions). Prior to the preparation of the specimens, the light power density was verified as 1100 mW/cm² with a radiometer (Demetron/Kerr, Danbury, CT, USA). All specimens were finished using 600-grit silicon carbide paper (Struers, Willich, Germany) and cleansed with 70% ethyl alcohol. A micrometer (293 MDC-MX Lite, Mitutoyo Corp, Tokyo, Japan) was used to measure their thickness and to verify that the thickness remained within a critical tolerance of 1 ± 0.01 mm. All composite specimens were stored in distilled water at room temperature for 24 hours until the radiopacity measurement was performed.

To obtain enamel and dentin specimens a non-carious intact freshly extracted third molar was used in this study. One-millimeter-thick regular enamel and dentin slices were prepared by sectioning the tooth transversally using a low-speed diamond saw (Isomet 1000, Buehler Ltd, Lake Bluff, IL, USA) under water cooling. For thickness adjustment, the tooth slices, which contained both enamel and dentin, were ground flat with 600-grit silicon carbide paper

and checked using a micrometer to verify that the thickness remained within the 1 ± 0.01-mm limits. The tooth slices were kept in distilled water until use.

During each radiographic exposure, an Al step wedge (99% purity) with fifteen 1-mm incremental steps, which allowed the calculation of the radiopacity of each material in terms of Al thickness (mmAl), was used as an internal standard.

Radiographic Procedures

One specimen of each material was placed directly on a phosphor storage plate (PSP) (VistaScan, Dürr Dental, Bietigheim-Bissingen, Germany; size 4; 5.7×7.6 cm) alongside the Al step wedge and tooth slice of both enamel and dentin, which were used for comparison. A custom-made acrylic device was used to standardize the object-to-focus distance (30 cm) and the positions of both the PSP and the head of x-ray machine to ensure that the central x-ray beam was directed at a 90° angle to the PSP surface. For radiographic exposure, a dental x-ray unit (Evostyle NG, New Life Radiology, Torino, Italy) with a total

Table 2: The Means and Standard Deviations (SDs) of Equivalent Aluminum (Al) Thickness (Radiopacity) Values of the Test Materials and Comparison of These Values with Enamel, with Dentin, and with Each Other

Test Material	Radiopacity Declared by Manufacturer	Equivalent Al Thickness Mean \pm SD	Statistical Differences*
Enamel		2.09 \pm 0.48	A
Dentin		1.07 \pm 0.30	B
Bulk-fill flowable materials			
Venus Bulk Fill	3	3.67 \pm 0.47	c, a
X-tra Base	3.5	3.39 \pm 0.37	d, a,b
SDR	2.2	2.81 \pm 0.38	e, b,c
Filtek Flowable Bulk Fill	2.4	2.46 \pm 0.35	f, c,d
Conventional flowable materials			
Charisma Opal Flow	Not available	3.65 \pm 0.45	g, a
Vertise Flow	3.2	3.57 \pm 0.46	h, a
Esthet-X Flow	Not available	2.57 \pm 0.46	j, c,d
GrandioSo Heavy Flow	Not available	2.47 \pm 0.49	k, c,d
GrandioSo Flow	Not available	2.35 \pm 0.53	A, c,d,e
X-Flow	2	2.22 \pm 0.53	A, c,d,e
Grandio Flow	2	2.05 \pm 0.52	A, d,e
Filtek Ultimate Flow	1.7	1.89 \pm 0.41	A, d,e
Arabesk Flow	Not available	1.69 \pm 0.51	L, e

* Different capital letters in the column indicate statistically significant differences in equivalent Al thickness values in comparison to enamel and dentin (one-sample *t*-test, $p < 0.05$). Different lowercase letters in the column indicate statistically significant differences in equivalent Al thickness values among the tested materials (Bonferroni test, $p < 0.05$).

filtration equivalent to 2 mm Al operating at 70 kVp and 8 mA was used. The exposure time was 0.2 seconds. Following exposure, the PSPs were scanned immediately with a Dürr VistaScan Mini scanner (Dürr Dental), and digital images were obtained. This procedure was repeated for 10 times, and the same PSP was used for all exposures to preclude any potential differences related to the plates. The images were viewed using the software provided with the system (DBSWIN 5.2.0, Dürr Dental).

Measurement of Mean Gray Values

Mean gray values of the materials, each step of the Al step wedge, and dentin and enamel were measured using the histogram analysis function of DBSWIN 5.2.0 software with no enhancement or further manipulation of contrast. Five measurements were obtained from five different areas of each specimen, each step of the Al step wedge, and dentin and enamel in each digital image. All of the measurements were performed by an operator who was blinded to the identities of the materials in order to eliminate interobserver inconsistency. The means of these readings were calculated and converted into equivalent Al thickness using the step-wedge values in Curve Expert 1.4 software (www.curveexpert.net).

Statistical Analysis

Statistical analysis was performed using Statistical Package for Social Sciences (SPSS) for the Windows 15.0 operating system (SPSS Inc, Chicago, IL, USA), with the significance level set at 5%. The means and respective standard deviations of the equivalent Al thickness values of the tested materials were calculated. The Kolmogorov-Smirnov test was performed to verify the normal distribution in the sample. One-way analysis of variance and Bonferroni tests were used to compare the equivalent Al thickness values of the tested materials. The mean radiopacity values for each material were compared with those of enamel and dentin using the one-sample *t*-test.

RESULTS

Table 2 presents the means and standard deviations of equivalent Al thickness (radiopacity) values of the test materials and comparison of these values with enamel, with dentin, and with each other as well as the available radiopacity values declared by the manufacturers.

Venus Bulk Fill provided the highest radiopacity value (3.67 \pm 0.47), whereas Arabesk Flow showed the lowest (1.69 \pm 0.51). The dentin and enamel reference radiopacity values were 1.07 \pm 0.30 mm

and 2.09 ± 0.48 mm equivalent Al, respectively. All of the tested bulk-fill flowable composites showed significantly higher radiopacity values in comparison to enamel and dentin ($p < 0.01$). In addition, significantly higher radiopacity values were obtained with the tested bulk-fill flowable composites in comparison to most of the conventional flowable composites ($p < 0.05$). The analysis of the results obtained with the conventional flowable composites revealed that all of them showed significantly higher radiopacity values in comparison to dentin ($p < 0.01$). Charisma Opal Flow, Vertise Flow, Esthet-X Flow, and GrandioSo Heavy Flow provided significantly higher radiopacity values in comparison to enamel ($p < 0.01$ and $p < 0.05$); GrandioSo Flow, X-Flow, Grandio Flow, and Filtek Ultimate Flow showed similar radiopacity values to enamel ($p > 0.05$); and Arabesk Flow showed significantly lower radiopacity values in comparison to enamel ($p < 0.05$).

All of the materials met the ISO minimum standard of radiopacity, which is equal to or greater than that of the same Al thickness.

DISCUSSION

The flowable composite resin materials assessed in this study presented variable radiopacity values. All of the 13 flowable composite materials provided radiopacity values that are higher than those of dentin and 1-mm-thick Al and thus met the ISO 4049 standard.¹⁷ As mentioned previously, some authors^{11,24} have suggested that for improved clinical detection, the minimum radiopacity values for flowable composite materials should be higher than that of dentin or similar to or slightly higher than that of enamel. Unfortunately, the radiopacity value measured for one of the conventional flowable composite materials, Arabesk Flow, was significantly lower than the reference radiopacity value of enamel ($p < 0.05$).

The radiopacity level of resin composites generally varies depending on several factors. Among these factors, the type and amount of radiopaque filler particles incorporated to confer this property are the most important.^{12,25-28} Several authors^{16,29-31} evaluated the filler content and its relation to the radiopacity of resin composite materials, and they revealed that introduction of fillers with high atomic numbers, such as ytterbium (atomic number: 70), lanthanum (atomic number: 57), barium (atomic number: 56), zirconium (atomic number: 40), yttrium (atomic number: 39), strontium (atomic number: 38), and zinc (atomic number: 30), led to higher radiopacity values. The higher the atomic number of the

element added to the radiopaque filler of the material composition, the higher the radiopacity of the materials due to increased x-ray absorption capacity. In order to achieve a radiopacity value for dental composites that is higher than that of human enamel, the filler volume should be more than 70%, and the mass percentage of opacifiers in filler particles should exceed approximately 20%.²⁹ A higher amount of fillers with high atomic numbers may lead to higher radiopacity, even if they make up a lower percentage of the weight or volume. In this study, the wide range of radiopacity occurs principally as a result of different kinds and volumetric proportions of radiopacifying agents, given the diversity of manufacturers. The information provided in Table 1 demonstrates that barium, ytterbium, zirconium, and strontium are present in the fillers of the majority of the tested flowable composites. The flowable composites with the highest radiopacity values include both barium and ytterbium, explaining their high radiopacity.

Some of the flowable composite resin materials tested in this study are available in different shades. Even though ISO 4049¹⁷ suggests that a certain shade should be used for testing radiopacity, Furtos and others,²⁷ Marouf and Sidhu,³² and Pekkan and Ozcan,³³ who investigated the effects of shade on radiopacity, revealed that there was no statistically significant difference between different shades of materials and concluded that radiopacity is material specific. These studies show that shade designation has no effect on the radiopacity of a material. Since the composition of the composites and the filler content remain the same, no significant difference could be expected between the radiopacity of different shades of the same material.^{27,32,33} Therefore, the shades that were available to the researchers were used in this study.

The differences in the mean radiopacity values of the same restorative materials tested in this study and those of previous studies may be due to the differences in testing conditions and imaging techniques.^{34,35}

In the available literature, various methods for radiopacity measurements of restorative materials have been explored, such as conventional radiography assessed by an optical densitometer or spectrophotometers; indirect digital radiography involving digitization of chemically processed radiographs using a CCD camera, a laser scanner, or a flatbed scanner; and the use of specific software to identify gray values, or direct digital radiography.^{12,13,18,30,36-38} However, ISO 4049¹⁷ is based on conventional

radiography (a monochromatic x-ray source performing within the range of 65 ± 5 kVp using a size 4 D-speed film at a target-film distance of 30-40 cm). In recent years, more attention has been given to the method used to measure radiopacity and to the comparison of digital and conventional radiography.^{37,39,40} Optical density is a logarithmic measure of the ratio of transmitted to incident light through the film image, while digital image analysis provides a direct record of radiographic density, as determined by pixel shade, which is automatically represented by computer software in a scale of gray shades ranging between 0 (black) and 255 (white) for an 8-bit system, or 0 (black) and 65,535 (white) for a 16-bit system. In the present study, a 16-bit digital system was utilized. Digital analysis can produce measurements equivalent to those obtained with film with reduced noise and can provide rapid, reproducible, precise, and consistent numerical values for comparative radiodensity studies. Therefore, it is considered to have the same or higher accuracy in comparison to transmission densitometry.^{19,36,37,39,41} Direct digital imaging also eliminates the loss of information that may occur with the use of indirect digital imaging.³⁸ For these reasons, direct digital image analysis is accepted as a fast and easy resource for the interpretation of radiographic density of the restorative materials in dental practice and might be a suitable alternative for the evaluation of the radiopacity of dental restorative materials.²⁷ Although ISO 4049¹⁷ requires the use of conventional radiography, the researchers preferred utilizing a PSP system to evaluate the radiopacity of flowable composite materials based on the affirmative results presented in the preceding studies.^{13,28,37,38,40} However, in order to avoid any distortion of the results in the radiopacity measurements, the images were acquired without automatic gain control or other postacquisition processing.^{28,39}

In addition to the use of a digital system instead of conventional films, another inconformity with ISO 4049¹⁷ was the use of an Al step wedge with 1-mm incremental steps instead of 0.5-mm steps. However, in the past, several researchers^{14,35,40,42} made use of step wedges with steps higher than 0.5 mm, contrary to what is recommended by the ISO protocols, and they were able to generate an accurate model of linear regression.

In this study, only 1-mm-thick specimens were tested, as recommended by ISO 4049.¹⁷ As reported in previous studies,^{42,43} the use of thicker specimens would have led to higher radiopacity values, in comparison to 1-mm increments, due to the fact that

bulk-fill flowable composites are bulk-filled in layers up to 4 mm.

ISO 4049 standard¹⁷ suggests that the minimum radiopacity of restorative materials should be equal to or greater than that of an equivalent Al thickness and that $\geq 98\%$ pure Al, the radiopacity of which is close to that of human dentin, should be used as a radiographic standard when testing the radiopacity of resin-based filling materials. Furthermore, several authors^{15,44} have proposed the use of dentin and enamel specimens as a secondary standard in addition to Al step wedge. In this context, the relative radiopacities of restorative materials, enamel, and dentin are expressed as Al equivalent values. The reference radiopacity values of dentin and enamel in this study were 1.07 ± 0.30 mm and 2.09 ± 0.48 mm equivalent Al, respectively. The values of dentin and enamel accord with the results of previous studies^{18,19,28} in which dentin radiopacity was close to 1 mm equivalent Al and enamel radiopacity was close to 2 mm equivalent Al.

According to ISO 4049,¹⁷ if the manufacturer claims that a product is radiopaque, its radiopacity should be equal to or greater than that of the same thickness of aluminum and should not be less than 0.5 mm below any value claimed by the manufacturer. When the results of this study were compared with the available radiopacity values declared by the manufacturers, it was observed that the majority of the values are close to the results of this study despite the existence of negligible differences. Thus, it is possible to conclude that the radiopacity data provided by the manufacturers can be relied upon. Since manufacturers constantly reformulate the composition of their products in order to achieve improved properties, studies that evaluate the radiopacity of base and liner materials should be repeated periodically.

CONCLUSION

According to the research methodology and within the limitations of this study, all of the commercially available bulk-fill flowable restorative materials tested in this study were found to be more radiopaque than enamel, dentin, and most of the conventional flowable composites, and they met the minimum standard of radiopacity specified by ISO.

Acknowledgments

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Regulatory Statement

The study design and protocol was approved by the Ethics Committee of Medipol University (protocol number: 108400987-315/290).

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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