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Original article

Radiopacity of resin-based CAD/CAM blocks assessed by areal grayscale pixel value measurement

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Abstract

Purpose: This study assessed radiopacity of resin-based computer-aided-design/computer-aided-manufacturing (CAD/CAM) materials by areal grayscale pixel value measurement.
Methods: Radiopacities of six resin-based CAD/CAM block materials and resin composite were evaluated and compared to that of enamel and dentin. Specimens of 1-mm thickness were placed on photostimulable phosphor plate and irradiated with digital x-ray unit. On the radiographic image, regions of interests were determined for each specimen and areal grayscale pixel values were measured. Elemental analysis was performed with energy-dispersive x-ray spectroscopy (EDS) on field emission scanning electron microscope (FESEM) images of the specimens. Data were analyzed statistically ($\alpha = 0.05$).
Results: Radiopacity values of the restorative materials were significantly different from each other ($P < 0.05$). Radiopacity values of two resin-based CAD/CAM materials were significantly lower than that of dentin ($P < 0.05$). All tested restorative materials contain zirconium, three materials contain barium, and only resin composite contains lanthanum.
Conclusion: Four CAD/CAM materials with higher amounts of zirconia or barium (>18%) had radiopacity values significantly higher than the dentin; while two materials with lower amounts of zirconia (<4%) and/or no-barium, had radiopacity values significantly lower than the dentin. EDS analysis suggests materials containing elements with higher atom numbers such as zirconia and barium could exhibit higher radiopacity.

Keywords; CAD/CAM, elemental analysis, energy-dispersive x-ray spectroscopy, radiopacity, resin-based materials

Introduction

Computer-aided design/computer-assisted manufacturing (CAD/CAM)-derived indirect restorations are becoming widely used due to advantages over conventionally prepared ones [1]. Several materials such as glass ceramics, zirconia, resin nano-ceramics and composite-ceramic hybrids are available for this purpose [2-4]. In recent years resin-based CAD/CAM blocks have become widely used due to ease of machinability [5], because they resist chipping and cracking during milling, and are less brittle than glass ceramics [6,7]. Additional advantages of hybrid ceramics are that they do not need post-firing, are easily polished, and allow easier completion of restorations [8]. They can be used for inlay/onlay restorations, endocrowns and long-term provisional restorations.

Resin-based CAD/CAM restorations have comparable survival rates with porcelain CAD/CAM restorations after 3 years of clinical service [9], with survival rates of above 85% after 2 years [10,11]. Within this period, clinicians perform routine evaluations of these restorations at least once a year. Evaluation of a dental restoration can be performed in two ways; clinical or radiographic. Clinical evaluation alone is not sufficient and radiographic evaluation is required in almost every follow-up session [12]. Thus, the radiopacity of the restoration should allow easy detection of recurrent/secondary caries during radiographic evaluation [13]. Restorative material should have adequate radiopacity to facilitate detection of secondary/recurrent caries, to evaluate adaptation of the restorative material, and help distinguish between the material and tooth tissues [14-16]. International Standards Organization (ISO) requires the restorative material's radiopacity be equal to or greater than the same thickness of aluminum (Al) (98% purity or more) [17]. Visibility of dentin in an x-ray is considered to be similar to that of Al. Therefore, it is important that a restorative material has radiopacity of at least 1 mm Al. A 1 mm thick restorative material having equal visibility to that of 1 mm thick Al is deemed to have equal visibility to 1 mm dentin in x-ray [17].

Several elements such as barium, zirconium, aluminum and strontium help to increase the radiopacity of resin composites. Similar elements are present in resin-based CAD/CAM materials for this purpose. These blocks contain varying amounts of different fillers, which could also have an impact on their radiopacity. In the literature, information regarding the radiopacity of resin-based CAD/CAM blocks is lacking. Therefore, this study evaluated the radiopacity of different hybrid blocks according to ISO 4049 [17] assessed by regional grayscale pixel value measurement. Additional elemental analysis was performed to determine the presence and rate of various elements, which might have an impact on radiopacity of the material. The first null hypothesis tested was that resin-based CAD/CAM materials would not have different radiopacity values; the second null hypothesis tested was that radiopacity of the resin-based CAD/CAM materials would not be greater than or equal to the radiopacity of dentin.

Materials and Methods

This study was conducted in accordance with all the provisions of the World Medical Association Declaration of Helsinki and Ankara University Faculty of Dentistry's local human subjects oversight committee guidelines and policies of the ethics committee for the study of human and animal (36290600/46).

Specimen preparation

In this study, radiopacities of six different hybrid CAD/CAM block materials and one posterior resin composite were evaluated according to ISO 4049 standard [17]. The materials used in this study are listed in Table 1. For each CAD/CAM material, 5 slices of 1 mm thickness were obtained by cutting the CAD/CAM blocks with a low speed diamond cutting disc (Microcut 201, Metcon, Bursa, Turkey) under water-cooling. Similarly, 5 samples of resin composite discs were prepared by inserting the material in a custom-made teflon mold (4 mm diameter and 1mm height) placed on a mylar strip. After composite insertion, another mylar strip was placed on the top surface of the mold and a microscopic glass slide was used to remove excess composite material by gentle pressure. Composite samples were then cured over the glass slide for 20 s with a light emitting diode curing unit (SDI Radii Plus, SDI, Melbourne, Australia). Irradiance was assured to be equal or higher than 1,000 mW/cm^2 which was measured using a radiometer (Hilux Ledmax curing lightmeter, Benlioğlu Dental,

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Table 1 Restorative materials used in the study

Type	Material	Shade	Organic composition	Filler composition	Filler weight %	Manufacturer
Resin-based CAD/CAM	CeraSmart (CRS)	A2 LT	Bis-MEPP, UDMA, DMA	silica and barium glass nanoparticles	71	GC, Tokyo, Japan
	Grandio Blocks (GRB)	A2 LT	UDMA, DMA	nanohybrid fillers	86	Voco, Cuxhaven, Germany
	Lava Ultimate (LVU)	A2 LT	BisGMA, UDMA, BisEMA, TEGDMA	silica and zirconia nanoparticles and zirconia/silica nanoclusters	80	3M ESPE, St. Paul, MN, USA
	Shofu Block HC (SHB)	A2 LT	UDMA, TEDGMA	silica powder, micro fumed silica, zirconium silicate	61	Shofu, Kyoto, Japan
	Tetric CAD (TTC)	A2 MT	Bis-GMA,Bis-EMA, TEGDMA, UDMA	barium aluminum silicate glass, silicon dioxide	71	Ivoclar Vivadent, Schaan, Liechtenstein
	Vita Enamic (VTE)	A2 LT	UDMA, TEGDMA	ceramic particles: SiO_2, Al_2O_3, Na_2O, K_2O, B_2O_3, ZrO_2, CaO	86	Vita Zahnfabrik, Bad Sackingen, Germany
Resin composite	Clearfil Majesty Posterior (CMP)	A2	Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate	silanated glass ceramics, surface treated alumina micro filler, silanated silica filler	92	Kuraray, Osaka, Japan

Bis-MEPP, bisphenol A ethoxylate dimethacrylate; UDMA, urethane dimethacrylate; DMA, *N*,*N*-Dimethylacrylamide; BisGMA, bisphenol A-glycidyl methacrylate; BisEMA, ethoxylated bisphenol A glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate

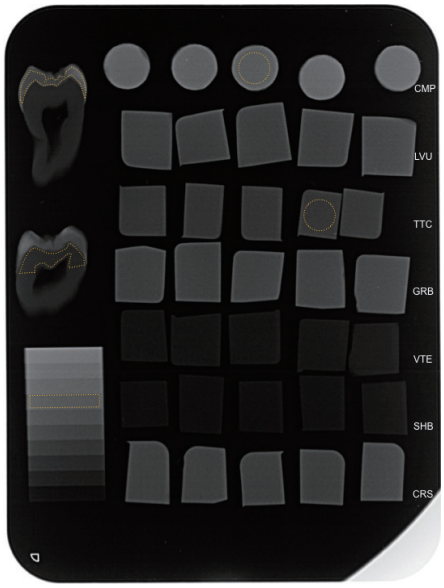


Fig. 1 X-ray images of restorative materials, enamel and dentin slices, and aluminum step-wedge. Yellow dotted areas show representative regions of interest used for the areal grayscale pixel value measurements. Circular specimens are resin composites; rectangular specimens are resin-based CAD/CAM materials. CRS, CeraSmart; GRB, Grandio Blocks; LVU, Lava Ultimate; SHB, Shofu Block HC; TTC, Tetric CAD; VTE, Vita Enamic; CMP, Clearfil Majesty Posterior

Ankara, Turkey).

The sectioned CAD/CAM slices and resin composite discs were wet-ground using 600, 800, and 1,200 grit silicon carbide abrasive papers on a polishing machine (Gripo 2N Polisher Grinder, Metcon). Two extracted non-carious permanent third molar teeth were longitudinally sectioned to obtain 1 mm-thick slice per tooth, involving the enamel and dentin. Teeth were extracted 1 week before the experiment and were stored in distilled water at 37°C until specimen preparation. Similarly, obtained tooth slices were stored in distilled water at 37°C until radiopacity measurement. The thicknesses of all specimens were checked and verified by a digital micrometer (Mitutoyo America Corp., Aurora, IL, USA) with a resolution of 0.001 mm.

Digital imaging and gray value measurement

A photostimulable phosphor (PSP) occlusal plate measuring 57 × 76 mm (VistaScan Image Plate, Size 4, Dürr Dental SE, Bietigheim-Bissingen, Germany) was used for radiographic imaging. Tooth slices, resin composite and CAD/CAM samples along with an aluminum step-wedge were positioned on the PSP plate. Step-wedge was 99% pure aluminum, 50 mm long and 10 mm wide, graded from 0.5 to 5 mm in 0.5 mm increments. PSP plate was then irradiated with a dental x-ray unit (Expert DC, Gendex Dental Systems, Des Plaines, IL, USA) at 70 kVp and 7 mA with 0.4 s exposure time. Focal spot distance was 30 cm and 1.5 mm Al equivalent filtration was used. Immediately after irradiation, PSP plate was scanned with a scanner (VistaScan, Dürr Dental SE) and radiographic image was obtained. Imaging software (DBSWIN Imaging Software, Dürr Dental SE) was used to export the radiographic image to 8-bit TIFF image file format (4,542 × 6,026 pixels). The digital image was imported to an image analysis software (Image J, version 1.52, U.S. National Institutes of Health, Bethesda, MA, USA) for grayscale analysis of the restorative materials, tooth and aluminum step-wedge.

The software could measure the mean grayscale pixel value of the user-defined area. In the software, a region of interest (ROI) was determined for this measurement for each specimen and the aluminum step-wedge. For resin composites and hybrid blocks the same exact circular ROI was determined (Fig. 1). Care was taken to determine the area of interest at maximum, but was not too close to the edge of the specimens and the aluminum step-wedge to exclude the shining artifacts, thus removing any risk of affecting the results [18]. For enamel and dentin, ROI was determined with the freehand selection tool of the software. Since coronal enamel and dentin surface areas of the tooth were smaller than those of the restorative materials, ROI had to be chosen as smaller for enamel and dentin. After defining the ROIs for the aluminum step-wedge, enamel, dentin and restorative materials, mean (±SD) grayscale pixel values of the selected ROIs were measured using image analysis software.

Statistical analysis

The curve of the measured grayscale pixel values versus the thickness of each step of the aluminum step-wedge was plotted with curve fitting software (Curve Expert Pro, version 2.6.5, Hyams Development, curveexpert.net, Chattanooga, TN, USA). Mean (±SD) radiopacity values expressed as aluminum thickness (mm Al/1.0 mm specimen) for enamel, dentin and the restorative materials were then interpolated using this calibration curve. After interpolation, weighted mean and pooled standard deviations were calculated for each group. Kolmogorov-Smirnov test revealed a normal distribution of radiopacity data ($P > 0.05$) and Levene's test of homogeneity of variances revealed that variances were equal ($P > 0.05$). Then the data was analyzed with one-way ANOVA. Tukey's test was used for comparison of the radiopacity values of the restorative materials. Commercially available software (Prism 6.0, GraphPad Software, La Jolla, CA, USA) was used for all statistical analyses ($\alpha = 0.05$).

Field emission scanning electron microscope (FESEM) and energy-dispersive x-ray spectroscopy (EDS) analysis

The microstructure and composition of tested materials were analyzed using FESEM and EDS. For the analysis of filler morphology and constitution, three samples from each group were dried for five days in a desiccator. Then, samples were mounted on stubs with carbon plaster and were sputter coated with Au (60%) /Pd (40%) approximately 7 nm in thickness to reduce electrostatic charging with ion sputter coater (Emitech K550X, Quorum Technologies Ltd., Ashford, UK). Following sputter coating, samples were mounted on FESEM (Inspect F50, FEI Company, Hillsboro, OR, USA) equipped with EDS (Octane Plus, Ametek, Berwyn, PA, USA) to be examined under vacuum conditions. For analysis, the system was operated

at 20 kV, with a constant working distance, and images with magnifications ranging from ×5,000 to ×30,000 were taken. For elemental analysis, EDS mapping was performed with Octane Plus EDS system (EDAX Inc. Mahwah, NJ, USA) in ×5,000 magnification samples (565,248 pixels for each sample).

Results

Radiopacity analysis results

ROI for each restorative material was 89,198 pixels; whereas mean ROIs for enamel and dentin were 71,706 and 112,547 pixels, respectively. ANOVA results showed that differences among the means were significant ($P < 0.05$). Multiple comparison results of radiopacity values (mm Al/1.0 mm specimen) of tested materials are given in Table 2. Mean radiopacity values of the restorative materials were significantly different from each other ($P < 0.05$). The resin composite, Clearfil Majesty Posterior (CMP) showed the highest radiopacity values ($P < 0.05$). Radiopacity values of Vita Enamic (VTE) and Shofu Block HC (SHB) blocks were significantly lower than that of dentin ($P < 0.05$).

FESEM/EDS analysis results

Qualitative SEM analysis at various magnifications (×5,000, ×10,000, and ×30,000) showed each material had a unique microstructure, in particular, different particle sizes (Fig. 2). Elemental compositions (%w/w) of restorative materials are given in Table 3 and Fig. 2. EDS analysis revealed the presence of 4 common elements, silicon, carbon, oxygen and zirconium among the tested restorative materials. Additionally, barium aluminum, sodium, potassium and lanthanum were found in various amounts in some of the restorative materials. All tested restorative materials contain zirconium, while five materials (CeraSmart [CRS], Grandio Blocks [GRB], Tetric CAD [TTC], VTE, CMP) contain aluminum; three materials (CRS, GRB, TTC) contain barium and only CMP contains lanthanum.

Discussion

Regional grayscale pixel value measurement for assessment of radiopacity of CAD/CAM materials showed that each material used in this study had different radiopacity values, therefore, the first null hypothesis was rejected. The second null hypothesis was partially rejected, as some of the materials showed radiopacity values higher than that of the dentin.

Numerous studies investigating the radiopacity of restorative materials employed random pixel measurement analysis, in which grayscale values of randomly selected limited number of points/locations (3-5 points) were measured and the average of these measurements was considered as the radiopacity of that sample [19-21]. In contrast to the above-mentioned arbitrary measurement approach, in this study, ROI was determined to include the maximum surface area of each sample to be measured. ROI of the restorative materials contained 89,197 pixels per sample. With this approach, the radiopacity of the largest area possible for each material was measured and thus significantly higher accuracy was achieved. Another study employed a similar ROI based approach to ours, but did not provide further details regarding the pixel size of the ROI [22].

Despite advances in caries detection methods [16], radiographic examination remains the primary method for the detection of secondary caries in the posterior teeth [23]. However, several factors may influence this procedure, such as the quality of the radiographic image, the clinician's experience and radiopacity of restorative materials. It is important for the restorative material to exhibit sufficient radiopacity, so that it is easily distinguishable from the surrounding enamel and dentin. Contrasting difference simplifies the detection of secondary caries, if any, and reduces the incidence of false-positive results. The restorative material needs to be more radiopaque than dentin [23] or even closer to enamel [13,24]. In this study radiopacity of dentin was 1.34 mm Al/1.0 mm. Other studies found that radiopacity of dentin varies between 1.006 and 2.37 mm

Table 2 Multiple comparison results of radiopacity values (mm Al/1.0 mm specimen) of restorative materials, enamel and dentin. Different letters indicate significant differences

		Weighted mean	±	Pooled SD	
Resin-based CAD/CAM	CRS	2.29	±	0.08	a
	GRB	2.39	±	0.08	b
	LVU	2.34	±	0.08	c
	SHB	0.55	±	0.06	d
	TTC	1.90	±	0.08	e
	VTE	0.71	±	0.06	f
Resin composite	CMP	4.00	±	0.09	g
Tooth	D	1.34	±	0.11	h
	E	2.76	±	0.33	i

CRS, CeraSmart; GRB, Grandio Blocks; LVU, Lava Ultimate; SHB, Shofu Block HC; TTC, Tetric CAD; VTE,Vita Enamic; CMP, Clearfil Majesty Posterior; D, Dentin; E, Enamel

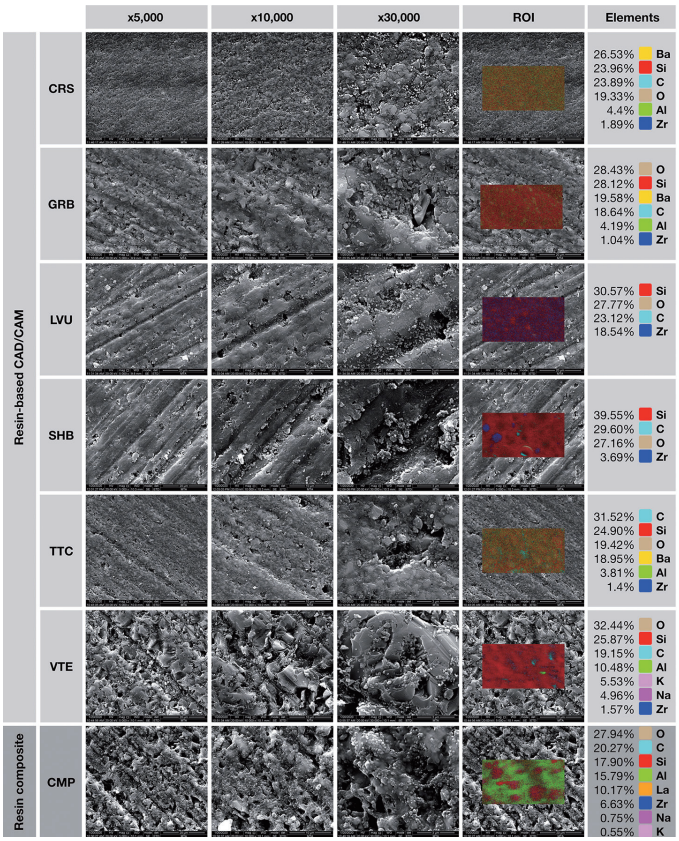


Fig. 2 Field Emission Scanning Electron Microscope (FESEM) images (at ×5,000, ×10,000, and ×30,000 magnifications) and energy-dispersive X-Ray Spectroscopy (EDS) results. EDS analysis was performed on FESEM images at ×5,000 magnification. Colored rectangular areas show the regions of interest for EDS analysis. Elemental analyses are given in %w/w. CRS, CeraSmart; GRB, Grandio Blocks; LVU, Lava Ultimate; SHB, Shofu Block HC; TTC, Tetric CAD; VTE, Vita Enamic; CMP, Clearfil Majesty Posterior

Table 3 Elemental compositions (%w/w) of restorative materials assessed by energy-dispersive x-ray spectroscopy (EDS)

		Si	O	C	Zr	Al	Ba	Na	K	La
Resin-based CAD/CAM	CRS	23.6	19.33	23.89	1.89	4.4	26.53	-	-	-
	GRB	28.12	28.43	18.64	1.04	4.19	19.58	-	-	-
	LVU	30.57	27.77	23.12	18.54	-	-	-	-	-
	SHB	39.55	27.16	29.6	3.69	-	-	-	-	-
	TTC	24.9	19.42	31.52	1.4	3.81	18.95	-	-	-
	VTE	25.87	32.44	19.15	1.57	10.48	-	4.96	5.53	-
Resin composite	CMP	17.9	27.94	20.27	6.63	15.79	-	0.75	0.55	10.17

CRS, CeraSmart; GRB, Grandio Blocks; LVU, Lava Ultimate; SHB, Shofu Block HC; TTC, Tetric CAD; VTE, Vita Enamic; CMP, Clearfil Majesty Posterior

Al [13,19-21,23,25,26]. Radiopacity of resin-based materials is related to the amount and composition of filler particles. In order to give radiopaque characteristics to polymeric materials, manufacturers add various radiopacifiers, such as barium, zirconium, ytterbium, aluminum, strontium. Therefore, in this study, additional EDS tests were performed to investigate the possible relation between the radiopacity of the materials and their radiopacifier contents. The SEM images were obtained at three different magnifications, ×5,000, ×10,000 and ×30,000. But, for EDS mapping, lower magnification (×5,000) was used to analyze a larger area, since the resin composite and resin-based CAD/CAM materials used in this study have quite heterogenous structures. This allowed more generalized content analysis. Radiopacity of the two resin-based CAD/CAM materials (SHB and VTB) was lower than that of the dentin and therefore they did not fulfill the requirement of ISO 4049 [17]. EDS analysis showed that, unlike other tested materials, these two materials do not contain any barium and have limited amounts of zirconium (1.57% for VTE and 3.69% for SHB) which might have caused this outcome. For VTE, the presence of 10.48% aluminum did not significantly contribute to radiopacity. Other tested resin-based CAD/CAM materials (CRS, GRB, Lava ultimate (LVU), TTC) showed higher radiopacity than that of the dentin and accordingly met the requirement of ISO 4049 [17]. They either contain more than 18% barium or zirconia, which led to radiopacity close to that of the enamel (2.76 mm Al/1.0 mm). Although the resin composite (CMP) contains no barium, it exhibited the highest radiopacity among the restorative materials tested. This could be attributed to the presence of 10.17% lanthanum and 6.63% zirconium. It is known that elements with higher atomic numbers give more radiopaque appearance to the materials they have been added to [Cabasso, I (2011) Radiopaque polymers. In: Mark HF (Ed) Encyclopedia of polymer science and technology. https://doi.org/10.1002/0471440264.pst456]. Atomic numbers of the detected elements in the restorative materials used in this study are; sodium 11, aluminum 13, potassium 19, zirconium 40, barium 56, lanthanum 57. It is possible that zirconium, barium and lanthanum significantly contributed to the radiopacity of the tested materials.

A recently published study [21] analyzed the radiopacity of several resin-based CAD/CAM materials. Similar to the results of the current study, SHB and VTE exhibited radiopacity lower than that of the dentin; whereas, CRS and LVU were higher. The authors additionally analyzed the inorganic content of the materials but did not provide the amount of the constituents. Although the common materials used in the studies were found to contain similar elements such as silicon, zirconium and barium, not all element identifications were similar. This could be due to the different analysis techniques used in the studies. The above-mentioned study employed x-ray fluorescence analysis, which is not particularly capable of detecting elements with lower atomic number (3 to 13). In contrast, EDS analysis, which is more suitable for detection of light elements such as aluminum was used in this study. Therefore, it is not possible to directly compare the elemental analysis between the studies. The resin-based CAD/CAM materials are luted on the tooth tissues with resin cements. Therefore, radiopacity characteristics of the cement could also have an impact on radiographic evaluation of the finished CAD/CAM restoration. If the clinician uses a resin-based CAD/CAM material with very low radiopacity, the use of resin cement with higher radiopacity than that of the dentin could be of benefit. Similarly, the opposite is also possible. However, radiopacity of restorative material/resin cement combinations could not be easily assessed due to different cavity configurations and restorative material thickness, and this could be the subject of another investigation. In summary, clinicians should be aware of the radiopacity characteristics of the CAD/CAM restorative material they use. Manufacturers should also seek to improve the radiopacity of their resin-based CAD/CAM materials and ensure they have radiopacity that is at least equivalent to that of the dentin.

One limitation of the current study is related to EDS analysis. In the EDS analysis, a smaller portion the total surface area of the specimen was scanned due to the inherent nature of the EDS analysis. In its current setup, EDS processing time for a single specimen was 8 h. A larger area could have been selected for evaluation, but obviously it would have needed an extremely long processing time for scanning the whole surface area of each specimen. Since a smaller portion for EDS analysis was chosen, it is possible that other areas which were not scanned could have different percent composition. Although resin-based CAD/CAM materials are monolithic, they are composite materials that are made up of organic and inorganic phases; thus, it is possible that constituents are not homogenously distributed in the bulk of the material. This could have had an impact on the outcomes of this study.

In this study, six resin-based CAD/CAM materials demonstrated varying degrees of radiopacity. Four of the resin-based CAD/CAM materials which contain higher amounts of zirconia or barium (>18%) had radiopacity values significantly higher than that of the dentin; while two of them which contain lower amounts of zirconia (<4%) and/or no barium, had radiopacity values significantly lower than that of the dentin. EDS analysis suggests that materials containing elements with higher atom numbers like zirconia and barium could exhibit higher radiopacity.

Conflict of interest

The authors of this article 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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