Radiopacity Evaluation of Bis-GMA/TEGDMA/Opaque Mineral Filler Dental Composites

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Received 22 May 2006; accepted 8 November 2006
DOI 10.1002/app.25779
Published online in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: The purpose of this work was to determine radiopacity values of composites based on (Bis-GMA/TEGDMA) monomers, at varying monomer compositions: (25/75), (50/50), and (75/25), mixed with five different radiopacifying filler agents: BaO, $BaSO_4$, La_2O_3, ZrO_2, and SrO at varying loadings ranging from 0 to 80 wt %. The radiopacity of four commercial dental composites were also evaluated and used as a comparative data. Following standard radiographic technique, X-ray radiography of 2.5 mm thickness dental composite specimens along with pure aluminum step-wedge, a reference, were performed. The optical density of specimens was measured using a transmission densitometer and the radiopacity values of samples were expressed in term of equivalent thickness of aluminum per mm thickness of material. The lanthanum oxide filler containing composites, which is a new radiopacifying agent, exhibited the greatest radiopacity levels than all other composites, for all monomer and filler loadings studied. The radiopacity values of these composites were greater than that of human dental enamel. Strontium oxide containing composites were less radiopaque than other materials especially in the case of composites based on (Bis-GMA/TEGDMA): (25/75). The composites containing more than 10 wt % of BaO, $BaSO_4$, and ZrO_2 for monomer compositions of (75/25) and (50/50) had radiopacity values greater than that of enamel. J Appl Polym Sci 104: 1632–1639, 2007

Key words: radiopacity; radiopacifying agent; dental composite; optical density; X-ray radiography; mineral fillers

INTRODUCTION

Typically, a dental resin-composite contains an organic matrix and inorganic fillers. Usually the filler particles are coated with coupling agent to bond to the resin matrix, which contains initiators for polymerization. The matrix phase is composed of organic dimethacrylate monomers. Most commercially available resin-composites contain bisphenol A glycidyl dimethacrylate or urethane dimethacrylate.[1] Because of the their high viscosity, triethylene glycol dimethacrylate is added as a comonomer to dilute the mixture.[2]

Acrylic polymers contain elements such as carbon, hydrogen, and oxygen with low electronic density, which makes them essentially X-ray-transparent.[3,4] The conversion of these translucent systems into X-ray-opaque materials, visible under X-ray imaging, can be performed by incorporating radiopacifying agents containing elements of high atomic number into the inorganic filler phase.[2–5] These radiopacifying agents, such as barium, strontium, zirconium, lanthanum or bismuth oxides, sulfates or carbonates, should be biocompatible. They may greatly vary in their concentration and composition, and consequently the radiopacity of composites varies as well. Excessive incorporation of radiopaque fillers results in reduced translucency of these materials but altered mechanical properties.[2,6,7]

X-ray contrast or radiopaque polymeric materials are increasingly in demand in various applications because they provide a quick, reliable, and nondestructive alternative for detecting the presence of such polymeric materials.[4,8] The advantages of radiopaque restoration materials allow clinicians to diagnose secondary caries and to evaluate radiographically the contours, voids, and contacts with the adjacent teeth.[1,7,9–13]

According to the International Standards Organization (ISO), the radiopacity property in dental composite materials is formally recognized and usually determined in comparison with that of enamel, dentine, or aluminum.[10] Several studies have indicated that, for optimum contrast, dental composites materials should have a radiopacity higher than, or at

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Contract grant sponsor: National Agency of the University Research Promotion (ANDRU).

least similar to, that of dental tissues, and preferably to enamel, to be able to detect secondary caries in radiographs.[2,3,6,7,10,14–16] So it is necessary to follow the variation of filler content loading in the composite to obtain required radiopacity of the dental composite. The condition is, at least, obtaining a radiopacity enamel value.

Aluminum has been widely used as a radiographic standard.[17,18] The absorption coefficient of aluminum is comparable with that of human dentine of the same thickness.[2,4,7,16,19] According to the ISO, a dental composite material should have a radiopacity equivalent of an equal thickness of pure aluminum.

The filler content of composites plays an important role in the properties of dental materials.[20,21] The incorporation of heavy metal to provide radiopacity can induce an adverse effect on other essential physical and chemical properties such as hardness[22,23] and polymerization shrinkage.[20,24] Incorporating large percentages of radiopaque fillers can also cause loss of dimensional stability of the composite.[25] Polymerization shrinkage, which is a major drawback of these restorative materials, consists of the conversion of intermolecular Van Der Waals distances of resin-monomers to the covalent bond-lengths during light-curing.[26,27] This negative effect may lead to the stress on the cavity walls of teeth resulting in marginal gaps, secondary caries, and clinical failure of the restoration.[28,29]

In the present work, the radiopacity of experimental dental composites based on (Bis-GMA/TEGDMA) monomers at varying monomer compositions (generally used), (25/75), (50/50), (75/25), is evaluated and expressed in terms of equivalent aluminum thickness. Anterior works report the results for some salts.[6,25,30–33] We have extended the study to five different radiopacifying fillers: BaO, $BaSO_4$, La_2O_3, ZrO_2, SrO in a wider range of 0–80 wt %. For comparative purpose, with experimental composites, the radiopacity of four commercial dental composites has been also evaluated and compared with that of human enamel and dentine. In perspective, the effect of the filler loading on other physical properties such as the polymerization shrinkage and the hardness will be carry out in succeeding works.

EXPERIMENTAL

The experimental composites were prepared as following:

1. First step: the two monomers, bisphenol A glycidyl dimethacrylate (Bis-GMA) (Aldrich, France) and triethylene glycol dimethacrylate (TEGDMA) (Aldrich: 98%), are mixed and homogenized in adequate proportions (50/50), (75/25), (25/75).
2. Second step: 2 wt % of Camphoroquinone (CQ) (Fluka: 98%), the light-visible photo-initiator, and 2 wt % of triethylamine (TEA) (Aldrich: 99.5%) used as an accelerator, were incorporated to the monomer mixture and mixed until total dissolution.
3. Third step: the radiopacifying filler powders were then added, in various proportions, into the mixture to provide loadings ranging from 0 to 80 wt %. Five different radiopacifying agents were studied: BaO, $BaSO_4$, La_2O_3, ZrO_2, and SrO. Each experimental composite was well blended to obtain a homogenous mixture.

Four commercial light curing micro-hybrid composites based on (Bis-GMA/TEGDMA/UDMA: urethane dimethacrylate) are given below:

1. Bright Light (HSR Primo Orthontics, England) contains 80 wt % inorganic fillers
2. Composan LCM (Promedica, Germany): 76.5 wt % of inorganic fillers
3. Arabesk Top (Voco, Cuxhaven, Gemany): 56 wt % of inorganic fillers
4. Te-Econom (Ivoclar Vivadent, Liechtenstein, Germany): 81 wt % of inorganic fillers.

Each of experimental and commercial composite pastes was packed into polypropylene molds, 8 mm in diameter and 2.5 mm thick, for producing cylindrical samples. Molds were placed between two clamped glass slides. The samples were then light cured for 60 s on the top and the bottom of the specimen using a visible light source (LA 500 Blue light curing light, 500 mW/cm^2, 450–490 nm, Aposa Enterprise).

The thickness of cylindrical specimens was measured using a digital micrometer reading to $\pm$ 1 μm.

To measure radiopacity, specimens were placed on medical X-ray film together with a high purity aluminum step-wedge (Biomaterials Science Research Group, School of Dentistry, University of Manchester, UK) with thickness varying from 1 to 10 mm with increments of 1 mm, on the same radiographic film. The X-ray film was positioned on a sheet of lead, 2.5 mm thick, to minimize back-scatter.

A standard dental X-ray source was positioned perpendicularly to the radiographic film. The unit was operated with an accelerating voltage of 60 kV, intensity of 7 mA, with a focus to film distance of 35 cm and a time of 0.1 s. Following standard techniques, the films were developed in an automatic X-ray processor. After development, the optical density of each material image on the films was measured by means of a transmission densitometer (model

331S/N 046396, Rothband East). Five readings were taken from each image of each material and for each step of the step-wedge. The average of these five readings was calculated. The optical density data obtained from the step wedge images were used to draw a curve of optical density of aluminum as function of its thickness.

The radiopacity of specimens was expressed in terms of the equivalent thickness of aluminum (mm Al) per unit thickness of material, deducted in reference to the calibration curve of the radiographic optical density of the aluminum step-wedge.

THEORETICAL BACKGROUND

After their discovery by Roentgen, X-rays were quickly accepted as a diagnostic tool in different fields such as medical and biomedical applications. X-rays, produced by oscillation or acceleration of an electric charge, are considered as a part of the electromagnetic radiation spectrum with wavelength range between 0.01 and 0.04 nm.[5,34]

The absorption of X-rays by an object is determined by the density of the material, its composition, and by the nature of the atoms. The X ray absorption coefficient, μ, of an element is related to its atomic number Z and is expressed as:[4,34]

$$\mu = k\lambda^3 Z^4 + b \tag{1}$$

where λ is the wavelength of the beam and k and b are the constants.

Equation (1) shows that the absorption coefficient increases with the atomic number. Therefore radiopacity of a material can be obtained by the incorporation of high atomic mass element in the restorative material. This property is applied to dental composites allowing X-ray imaging of filled teeth. The additives mixed with the monomers are inorganic compounds of heavy metal salts, high absorbers of X-rays in the normal medical dose-range, such as barium, zirconium, strontium, vanadium, and lanthanum. These attenuate the X-ray beam as it passes through the composite, reducing the intensity by absorption or scattering. Because of a high attenuation coefficient, the radiographed material appears lighter in the radiographic film image. This visible image provides the required contrast to the radiopacity calculation method.[5]

X-ray radiation has been found to follow the Beer-Lambert formula:[35]

$$I = I_0 e^{-\mu x} \tag{2}$$

where I_0 is the intensity of the incident radiation. I_0 decreases as a function of thickness (x) of the material; μ is the absorption or attenuation coefficient, characteristic of the substance and the wavelength of the radiation (cm^{-1}).

Radiopacity of materials is calculated from optical density determination of a radiographic film image. Optical density is a logarithmic measure of the ratio (transmitted light I_0/incident light I) through the film image:[35]

$$\mathrm{OD} = -\log_{10}(T) = \log_{10}(I_0/I) \tag{3}$$

where OD, optical density, measured by an optical densitometer, quantifies the contrast of the developed film; T is the transmittance.

Optical density thus depends on the X-ray absorption of the radiopacifying agents, the film characteristics, exposure parameters, and experimental conditions. As a comparative reference close to the radiopacity of dentine, aluminum has been chosen as a standard.[21,25] The absorption coefficient of aluminum is approximately equivalent to that of human den-

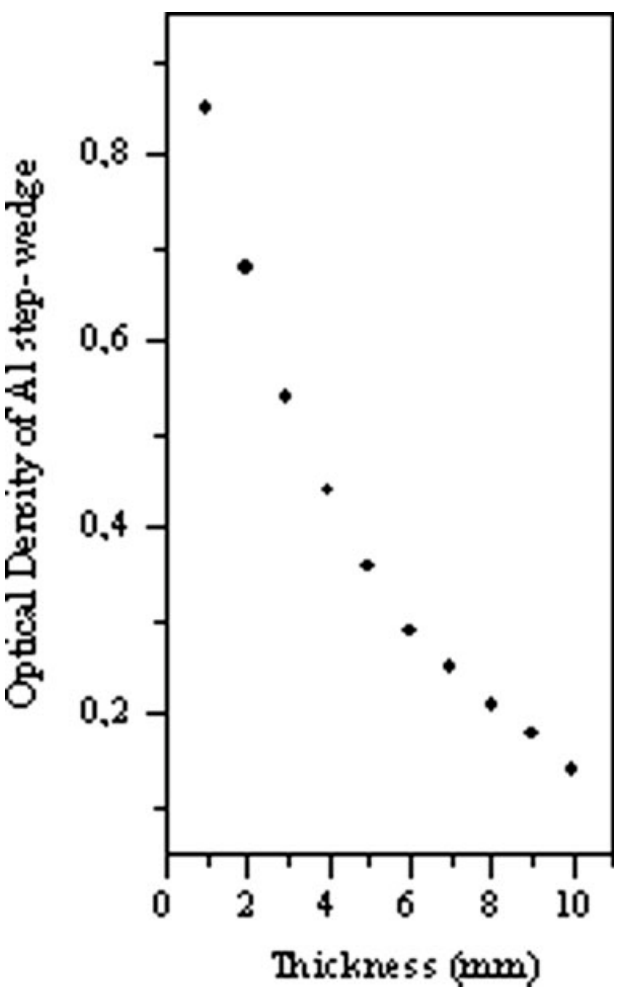

Figure 1 Optical density of Al step-wedge versus thickness (mm).

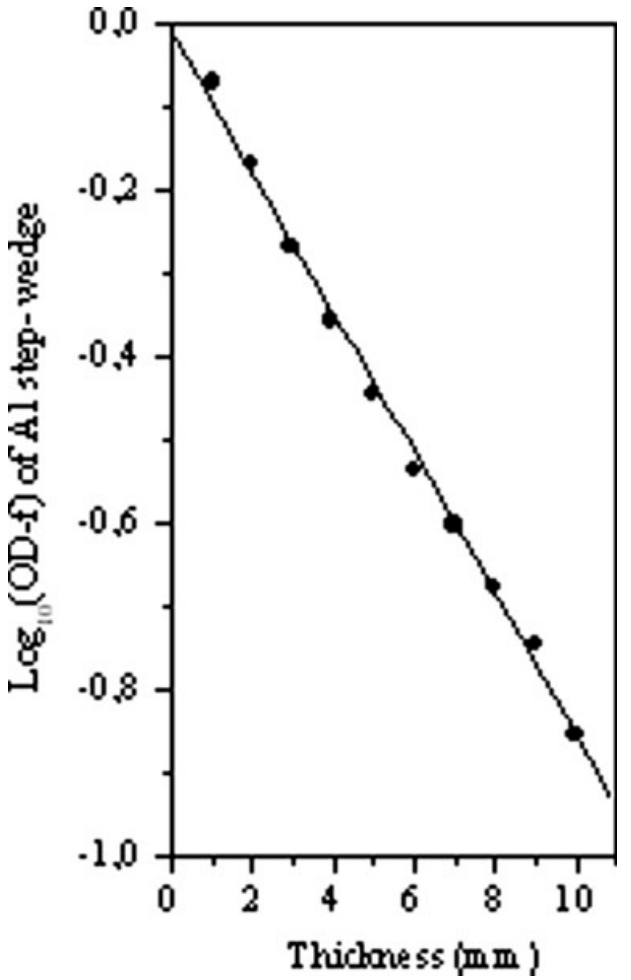

Figure 2 Logarithm of optical density of Al step-wedge versus thickness (mm).

tine of the same thickness.[1] To facilitate the detection of secondary caries, dental composite materials should have a radiopacity similar or higher than to that of human dentine of equivalent thickness.[25] Thus, the aluminum step-wedges have been radiographed along with the specimens and a calibration curve has been plotted for each film to transpose the measured optical densities into an equivalent thickness of aluminum standard.[36]

According to the ISO, for dental composite materials, pure aluminum (at least 99.5%) should be used as a radiographic standard. However, aluminum with this purity is not easy to manufacture into a step-wedge shape. So it is allowable to add, minute quantities of other elements such as copper, in alloys used to produce step-wedges.[36]

RESULTS AND DISCUSSION

Figure 1 illustrates calibration graph showing the optical density (OD) of an aluminum step-wedge as a function of aluminum thickness (mm). The obtained curve in Figure 1, which is not linear, can be re-plotted with $\log_{10}$ (OD $- f$) versus aluminum thickness (mm). The obtained plot is shown in Figure 2. Linear regression gave a correlation coefficient: $r^2 = 0.998$. The log (f), which corresponds to the optical density of the X-ray film due to the background radiation from different environment sources, must be subtracted from the optical density values of all studied specimens on the same X-ray film.

The following linear relationship can be used to describe the linearity dependence of the $\log_{10}$ (OD $- f$) on the Al thickness:[36]

$$\log_{10}(\mathrm{OD} - f) = C + m\ (\mathrm{Al\ thickness}) \qquad (4)$$

Here, m is the slope of the curve (Fig. 2) and C the intercept on the $\log_{10}$ (OD $- f$) axis.

After rearranging eq. (4):

$$\mathrm{Al\ thickness} = \mathrm{Rp} = \frac{C - \log_{10}(\mathrm{OD} - f)}{-m} \qquad (5)$$

Rp is considered as the radiopacity (mm Al) of a given thickness of specimen.

To obtain the radiopacity in aluminum equivalent of 1 mm specimen thickness, the Rp value must be divided by the measured specimen thickness:[36]

$$\mathrm{Rp}' = \frac{\mathrm{Rp}}{\mathrm{thickness\ of\ specimen\ (mm)}} \qquad (6)$$

The value of Rp′ corresponds to the radiopacity aluminum equivalent of 1 mm specimen thickness.

Thus, the specimens' radiopacity values were expressed as aluminum equivalent thicknesses for experimental composites based on (BisGMA/TEGDMA) monomers at varying monomer compositions: (25/75), (50/50), and (75/25) mixed with five different radiopacifying fillers: BaO, $BaSO_4$, La_2O_3, ZrO_2, and SrO at

TABLE I
Radiopacity (mm Al) of Samples Based on (Bis-GMA/TEGDMA, 25/75) Blended with Five Radiopacifying Agents (Salts): BaO, $BaSO_4$, La_2O_3, ZrO_2, and SrO from 0 to 80 wt %

Salt, wt %	SrO	ZrO_2	BaO	$BaSO_4$	La_2O_3
0	0.99	0.99	0.99	0.99	0.99
5	1.55	1.50	1.44	1.31	1.59
10	1.54	1.56	1.47	1.40	1.69
20	1.60	1.61	1.56	1.50	1.87
30	1.60	1.66	1.61	1.62	2.11
40	1.62	1.67	1.70	1.60	2.12
50	1.69	–	1.75	1.75	2.28
60	1.67	1.72	1.88	1.78	2.32
80	1.77	1.91	1.97	2.06	2.42

TABLE II
Radiopacity (mm Al) of Samples Based on (Bis-GMA/TEGDMA, 50/50) Blended with Five Radiopacifying Agents (Salts): BaO, $BaSO_4$, La_2O_3, ZrO_2, and SrO from 0 to 80 wt %

Salt, wt %	SrO	ZrO_2	BaO	$BaSO_4$	La_2O_3
0	1.10	1.10	1.10	1.10	1.10
5	1.46	1.83	1.72	1.54	1.88
10	1.52	1.79	1.78	1.60	2.00
15	1.52	1.92	–	1.69	2.12
20	1.67	1.93	1.96	1.79	2.17
25	1.60	1.97	2.04	1.82	2.32
30	1.73	1.99	2.02	1.97	2.31
35	2.12	2.01	2.08	2.00	2.37
40	2.09	2.08	2.18	2.13	2.51
50	2.18	1.99	2.23	2.16	2.82
60	2.40	1.95	2.26	2.35	2.92
70	2.38	1.95	2.36	2.56	3.07
80	2.47	2.12	2.46	2.62	3.22

varying loadings ranging from 0 to 80 wt %. These data are given in Tables I–III. The radiopacity values of dentine and enamel, according to the aggregate values obtained in many studies already cited, are respectively, 1 and 2 mm. These levels may be used for comparison with the radiopacities of composite materials.

As shown in Tables I–III, composites that contain lanthanum oxide filler have radiopacity values significantly greater than that of all other experimental composites and that exceeded dentine at all studied compositions. This result can be explained by the fact that the lanthanum exhibits a high atomic number compared with that of the other metals (Table IV) and consequently, it presents a high radiopacity level. It can be noticed that the greater atomic number leads to higher the radiopacity value of the composites at all different monomers and all filler loadings studied, which is in agreement with the eq. (1).

The data reported in Tables I–III are graphically represented by plotting the radiopacity (mm Al) of

TABLE III
Radiopacity (mm Al) of Samples Based on (Bis-GMA/TEGDMA, 75/25) Blended with Five Radiopacifying Agents: BaO, $BaSO_4$, La_2O_3, ZrO_2, and SrO from 0 to 80 wt %

Salt, wt %	SrO	ZrO_2	BaO	$BaSO_4$	La_2O_3
0	1.06	1.06	1.06	1.06	1.06
5	1.38	1.70	1.53	1.34	1.75
10	1.37	1.80	1.58	1.50	1.94
20	1.48	1.92	1.77	1.66	2.27
30	1.57	2.05	1.86	1.88	2.35
40	1.59	2.12	2.03	1.94	2.61
60	1.85	2.13	2.18	2.00	3.07
80	1.97	2.11	2.43	2.42	–

TABLE IV
Atomic Number of the Studied Elements

	Strontium, Sr	Zirconium, Zr	Barium, Ba	Lanthanum, La
Z	38	40	56	57

composites as the function of the filler loading percent:

- At different monomer compositions (Figs. 3–5).
- For each studied radiopacifying agent (Figs. 6–10).

These Figures show that the increase of the salt content (wt %) leads to the gradual increase of the radiopacity. The Figures show that the radiopacity of samples without radiopacifying agent (0 wt % filler composites) at all different monomer compositions, even they are close to that of dentine, stay nevertheless smaller than that of enamel, which is more difficult for radiographic detection of caries and defects adjacent to restorations.

It may not be possible to evaluate small defects and the extent of the restorations if the radiopacity is smaller than dentine. However, all studied materials have radiopacity values higher than that of the dentine. So it has been recommended that the radiopacity of resin composites should be equal to or greater than human tissues. Some authors have claimed that materials with a radiopacity less than that of enamel are not suitable for use.[37]

As shown in Figures 3 and 4, in the range of filler loading (30–50 wt %), barium, strontium, and zirconium containing composites have radiopacity values

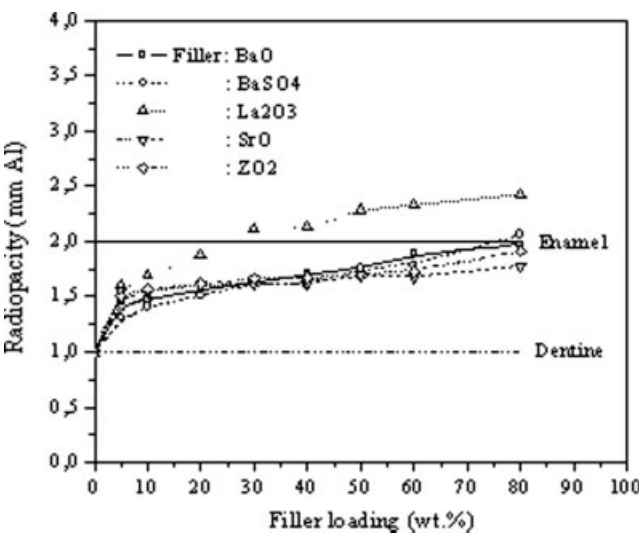

Figure 3 Radiopacity (mm Al) of (Bis-GMA/TEGDMA, 25/75) versus filler loadings (wt %).

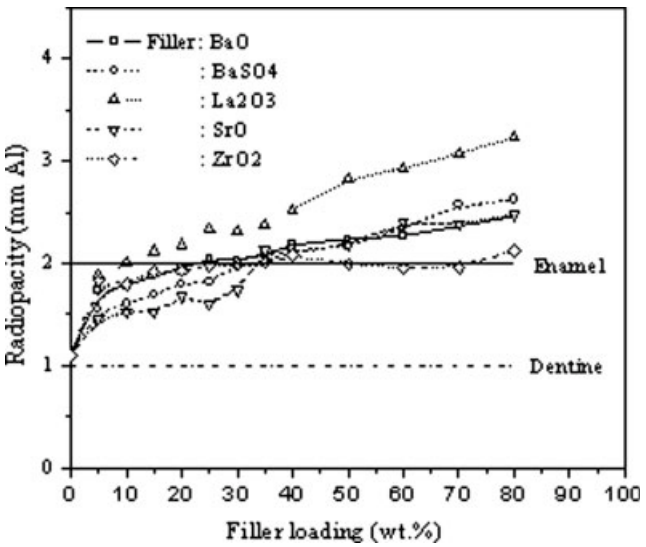

Figure 4 Radiopacity (mm Al) of (Bis-GMA/TEGDMA, 50/50) versus filler loadings (wt %).

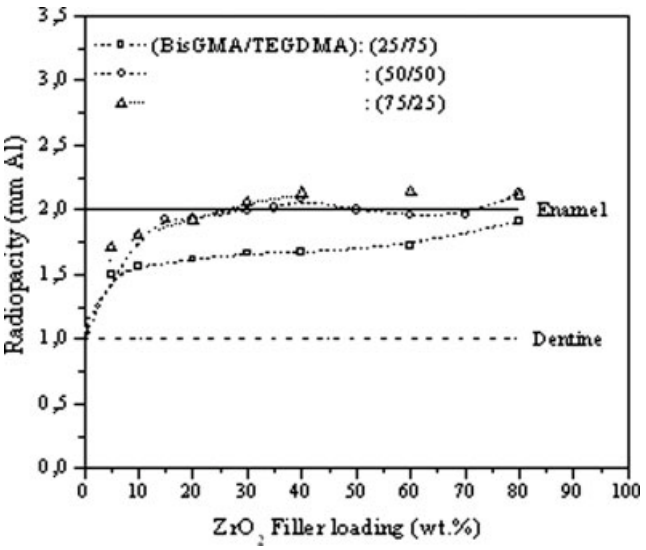

Figure 6 Radiopacity (mm Al) of (Bis-GMA/TEGDMA) versus SrO filler loadings (wt %) at varying monomer loadings.

close to each other. The lanthanum filler exhibit superior radiopacity values in the same range.

Figure 3 indicates that the radiopacity values of (Bis-GMA/TEGDMA: 25–75/filler) composites (BaO, $BaSO_4$, ZrO_2, and SrO) are lower than that of enamel but greater than that of dentine in all the range of filler loading studied (0–80 wt %).

The lanthanum filler composite exhibited the highest radiopacity values, which are greater than that of enamel from $\sim$ 30 wt %.

For (Bis-GMA/TEGDMA: 50–50/filler), Figure 4 shows that the radiopacity values of composites, except those containing lanthanum from 5 wt %, in the range (5–30 wt %), are similar to or lower than that of enamel, but stay greater than that of dentine. In the range (35–40 wt %) these composites exhibit radiopacity values close to that of enamel, and from filler loading of 40 wt %, the radiopacity values of strontium, barium, and lanthanum composites are greater than that of enamel, whereas those of zirconium composites stay close to that of enamel.

In Figure 5, (Bis-GMA/TEGDMA: 75–25/filler), the radiopacity values of SrO composite are lower than that of enamel but greater than dentine across the range of filler loading studied (0–80 wt %). This result is explained by the lower atomic number of

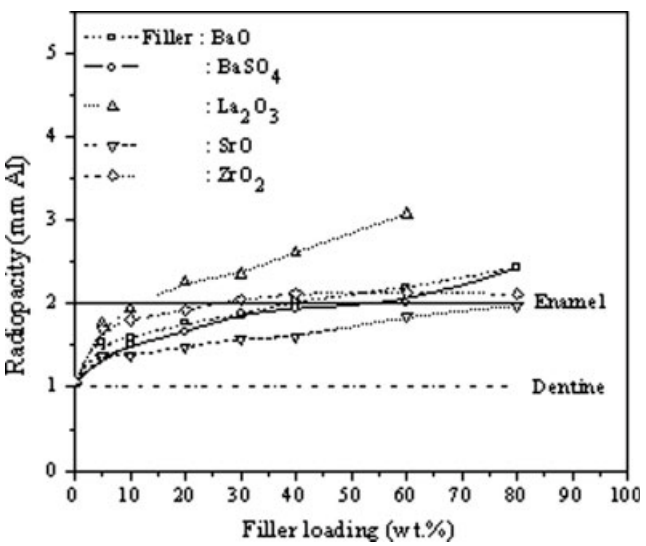

Figure 5 Radiopacity (mm Al) of (Bis-GMA/TEGDMA, 75/25) versus filler loadings (wt %).

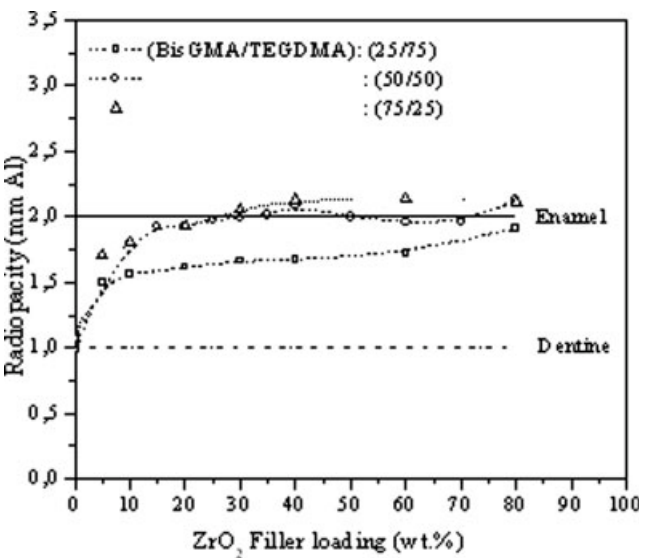

Figure 7 Radiopacity (mm Al) of (Bis-GMA/TEGDMA) versus ZrO_2 filler loadings (wt %) at varying monomer loadings.

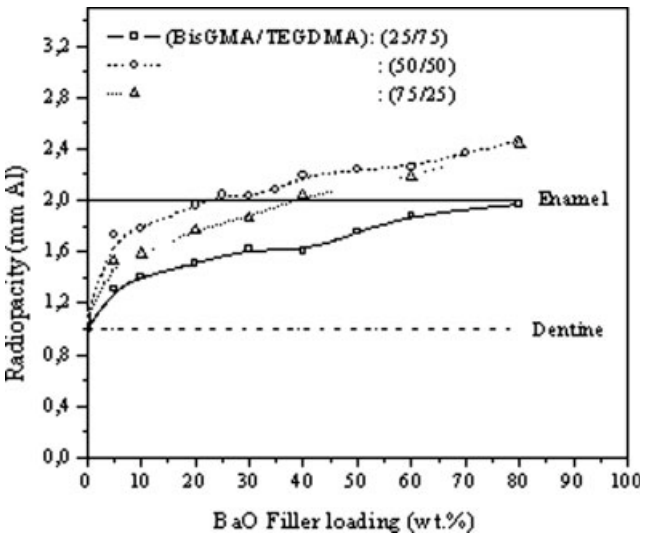

Figure 8 Radiopacity (mm Al) of (Bis-GMA/TEGDMA) versus BaO filler loadings (wt %) at varying monomer loadings.

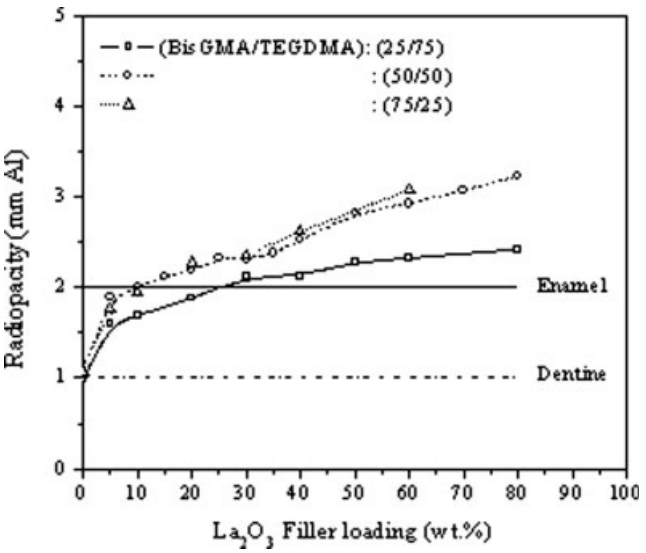

Figure 10 Radiopacity (mm Al) of (Bis-GMA/TEGDMA) versus La_2O_3 filler loadings (wt %) at varying monomer loadings.

strontium compared to the other elements. This Figure shows also that from ~ 10 wt %, the lanthanum composite exhibit radiopacity values greater than that of enamel. For barium and zirconium composites, in the range (5–40 wt %) the radiopacity values were close to or lower than that of enamel but were greater than that of dentine, and from 40 wt %, these composites exhibited radiopacity values greater than that of enamel.

Figures 6–10 represent the variation of radiopacity of each monomer composition and for particular salt filler. For all studied salt fillers, the (Bis-GMA/TEGDMA, 25/75) monomer compositions present lower radiopacity than (50/50) and (75/25) compositions. The (25/75) composites, in the case of strontium, zirconium, and barium fillers, had radiopacity values lower than that of enamel across the range of filler loading studied (0–80 wt %); whereas the radiopacity of (Bis-GMA/TEGDMA: 25–75/La_2O_3) were greater than that of enamel from 25 wt %.

In Figures 6, 8, and 9, the (50/50) monomer composites containing, respectively, BaO, $BaSO_4$, and SrO fillers exhibited higher radiopacity values than for (75/25) and (25/75) monomers. This may result from a better distribution of the salt filler in the tridimensional network.

The results of the radiopacity values, expressed as aluminum equivalent thicknesses, obtained of commercial light curing micro- hybrid composites: Bright Light, Composan LCM, Arabesk Top and Te- Econom are reported in Table V.

As shown in Table V, the radiopacity values of all commercial composites studied are higher than that of enamel. The radiopacity value of the composite Te-econom is twice (5.06 mm Al) the required value. We notice that this excessive radiopacity level can

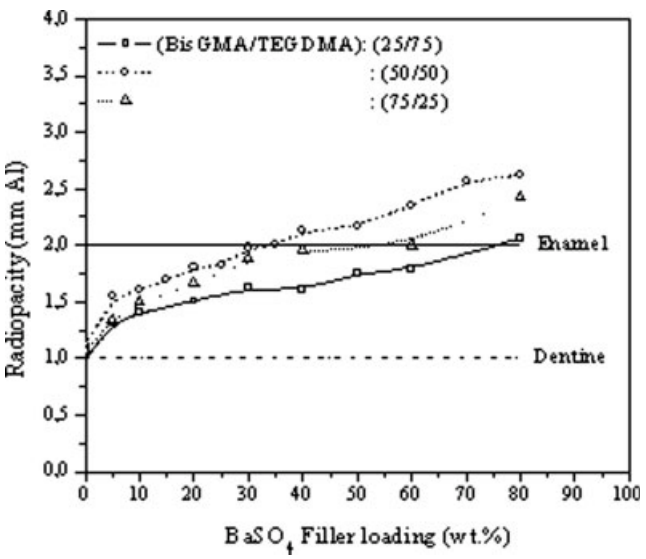

Figure 9 Radiopacity (mm Al) of (Bis-GMA/TEGDMA) versus $BaSO_4$ filler loadings (wt %) at varying monomer loadings.

TABLE V
Radiopacity (mm Al) of Commercial Composites

Commercial composites	Radiopacity (mm Al)
Bright light	2.92
LCM	2.32
Arabesk top	2.74
Te-econom	5.06
Dentine	1
Enamel	2

induce drawbacks on the mechanical properties. The other commercial composites exhibit adequate radiopacity values close to that of enamel. Thus, our results show that all evaluated commercial composites have clinically relevant radiopacity.

CONCLUSIONS

Measurements of the radiopacity, defined in terms of equivalent thickness of aluminum per unit thickness of material, of composite materials based on (Bis-GMA/TEGDMA), at varying monomer compositions: (25/75), (50/50), and (75/25), mixed with five different radiopacifying agents: BaO, $BaSO_4$, La_2O_3, ZrO_2, and SrO at varying loadings ranging from 0 to 80 wt %, have revealed a wide range of radiopacity values.

The radiopacity values of 0 wt % salt filler, at all different studied monomer compositions, were smaller than that of enamel but close to that of dentine, which is barely sufficient to distinguish dental fillings radiographically.

All the materials studied were more radiopaque than dentine. Lanthanum oxide conferred higher radiopacity values than those required by ISO 4049[38] standards.

Radiopacity values equivalent or superior to that of enamel can be achieved by the composite (Bis-GMA/TEGDMA/La_2O_3) for monomer compositions of (50/50) and (75/25), which contains at least 10 wt % of La_2O_3. Composites containing barium oxide and barium sulfate fillers for composition of (50/50) exhibit radiopacity values close to and greater than that of enamel from 30 wt %, whereas for strontium oxide, a radiopacity greater than that of enamel can be achieved with at least 35 wt % for composition (50/50). With zirconium oxide, radiopacity values slightly greater than that of enamel, can be obtained from 30 wt % for composition of (75/25).

The radiopacity evaluation of commercial composites shows that the ISO standard is respected.

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