The Radioopacity Evaluation for Various Nanocomposite Materials

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In accordance with ISO 4049/2000 Standard, the alleged radioopaque restorative materials must have a radioopacity value similar or higher with an aluminum layer of the same thickness. Depth polymerization of RDC represents half the depth of hardening under experimental conditions. European Standard requires a minimum of 0.5 mm for opaque diacrylic cements, 1 mm for opaque shades of restorative materials, respectively 1.5 mm for the other materials [11]. The aim of this study is to measure the radioopacity of different nanocomposite materials and coordinate this with the material's performance.

Keywords: radioopacity value, aluminium layer, diacrylic cements, restorative materials

Diacrylic composite resins are commonly used in restorative dentistry due to their physical and mechanical properties, ease of use, but mostly because of their aesthetic qualities.

Radioopacity of the composite materials is an optical indirect characteristic of the composite materials, being an important parameter in the detection of marginal secondary caries or relapses, also of some defects in proximal/marginal adaptation or interface, evaluation of marginal contour and of the material or cement in excess or of the proximal contact with adjacent teeth [1-3]. Radioopacity of composite resins must be at least equal to that of the dentin, in order to allow discrimination with the carious dentin, a number of authors suggesting that it would be preferable close values close to those of the enamel [1-3]. However, the radioopacity must not be excessive, because it could mask the marginal carious processes [4].

Differences in composition of the composite materials influences the intensity of radioopacity[11]. This results from the incorporation in the organic matrix of some various types of radiopaque fillers: glasses or ceramic particles with a content of heavy metals (barium, strontium, zirconium, lanthanides), barium sulfate, tungstanat of calcium, fluorides of lanthanides, aluminum or zirconium oxides [1,5]. Also, the thickness of the composite material, incidence of X-ray, the radiological film type and development technique can influence the radioopacity value [1,2,6]. The emergence of light-curing composite materials was considered a major step in the evolution of direct restoration techniques, thanks to its aesthetic and mechanical qualities, and the possibility to play back more realistic the variety of shades of the dental tissues. Their adequate light-curing is essential to the success of restorations [7]. Deficiencies in polymerization can lead to a decrease in the mechanical properties, water absorption and solubility increase, or in surface and marginal discoloration, an increase in the percentage of residual monomer with certain consequences upon biocompatibility [8-10].

Depth polymerization of RDC represents half the depth of hardening under experimental conditions. ISO 4049/2000 Standard requires a minimum of 0.5 mm for opaque diacrylic cements, 1 mm for opaque shades of restorative materials, respectively 1.5 mm for the other materials [11]. Along with the development of improved radiation sources, at least 2-3 mm of material can be polymerized in 20-30 s of irradiation. The irradiation decreases in the depth of material through absorption and dispersion by the organic matrix and filler particles [12]. The polymerization depth is influenced by the type and condition of the technical curing equipment (e.g. aging of lamp or filter, fracture or contamination of the optical guide), procedural factors (diameter of optical guide, distance and incidence of light toward the material) and considerations related to the cavity design or opacity and hue of composite material [13,14].

Trans illuminated light-curing is effective by a 3 mm enamel, respectively 2.5 mm conventional composite, on condition of increasing the polymerization time up to 60-120 s [13].

The aim of this study was to evaluate radioopacity for a range of commercial nanofiller composite materials (Premise ™/ Kerr Corp., Artiste ™/ Pentron Clinical Tetra EvoCeram® / Ivoclar Vivadent-) and for the experimental composites C13, C18 and C20 developed under ICCRR Cluj-Napoca. Also, there were tested the polymerization depth of the commercial and experimental nanocomposites mentioned above.

Experimental part

Material and method

Radioopacity evaluation was conducted for a specimen of each tested material (six specimens in total), using a Teflon mold of precision with diameter of 15 mm and a height of 1 mm. The composition of the studied materials is shown in table 1. The composite material was placed in the matrix slightly in excess, pressed down by a glass blade to allow a smooth surface without holes, then polymerized for 9x20 s.

It was used Translux Energy®/Heraeus-Kulzer halogen lamp, which has a light intensity of 900 mv/cm^2. The specimens were then removed from the mold and the thickness was checked with a micrometer. The specimens which were too thick were polished with fine abrasive paper (no. 1000) until a thickness of 1 ± 0.01 mm.

To perform the X-rays, there were used three films retro alveolar of E class (Kodak Insight®/Kodak), placed horizontally on a lead sheet of 2 mm thickness.

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Composite materials	The organic phase	(% mass)	Inorganic fillers
Premise™ (Kerr Corp.)	Bis-fenol-A-dimethacrylate ethoxylate, TEGDMA, initiators, stabilizers	84	Prepolymerised particles, 30-50 µm, barium glass 0,4 µm, silica 0,02 µm
Artiste™/ Pentron Clinical	PCBis-GMA, BisGMA, UDMA, HDDMA, photoinitiators, accelerators, stabilizers, silanes, pigments	75	Barium borosilicate glass, silica nanoparticles, zirconium silicate,
Tetric EvoCeram®(Ivoclar-Vivadent)	dimethacrylates	82-83	Barium glass, ytterbium trifluoride, mixed oxide
C13 (ICCRR)	Bis-GMA, TEGDMA	80	Barium glass, quartz, colloidal silica, zinc glass with zinc and calcium oxides
C18 (ICCRR)	Bis-GMA, TEGDMA	78	Zinc glass, quartz, colloidal silica
C20 (ICCRR)	Bis-GMA, TEGDMA	77.8	Glass with strontium and zirconium oxides, quartz, colloidal silica, hydroxyapatite with zirconium oxide

Table 1
THE COMPOSITION OF COMPOSITE MATERIALS USED IN THE STUDY

On the surface of each film were placed, according to the position in the scheme below (fig. 1.), a standard of aluminum (purity 99%) with 9 gradations of thickness each of 0.5 mm, from 0.5 to 4.5 mm, and below of standard there were placed two composite specimens.

The exposure lasted 0.08 s, from a focal length of 20 cm, being carried out with the device Oralix AC®(Gendex Dental Systems, USA) at 65 KV. The films were then developed automatically with the device Periomat Plus® (PGI UK Ltd.) The optical density of the three obtained X-rays was measured by photodensitometer Vitoreen 7-424®(Vitoreen, USA). There were performed three measurements for each stage of the aluminum standard, respectively for each specimen of composite, being then performed an average for each one of them.

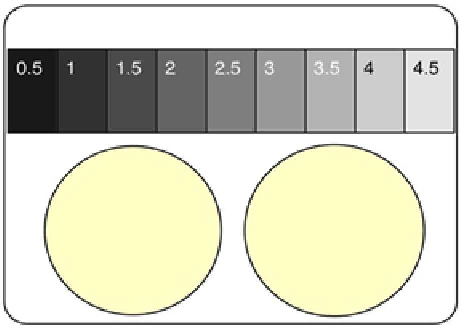


Fig. 1. Layout of the aluminium standard (top) and of the 2 composite specimens (down) on the radiological film surface

Radioopacity of the material was calculated in relation to the optical density using the following formula:

$$R = DO(comp)x100/DO(al)$$

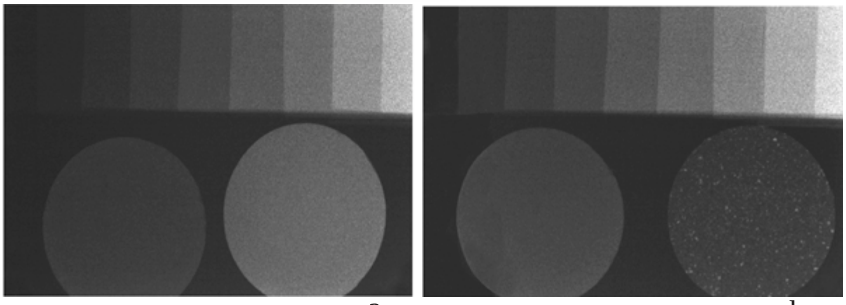
where:

R represents radioopacity of the composite specimen with 1 mm thickness;

DO(comp) represents the optical density of the specimen; and *DO(al)* represents the optical density of the 1 mm-thick step from the aluminum standard.

Statistical processing

Optical density values for the aluminum standard on each of the three X-rays were statistically analyzed with the Student's test at a significance level of 95%.



a b

Fig. 2. Radiographic images for two of the obtained films (a- specimens of Premise and Tetric EvoCeram; b- specimens of Artiste and C13)

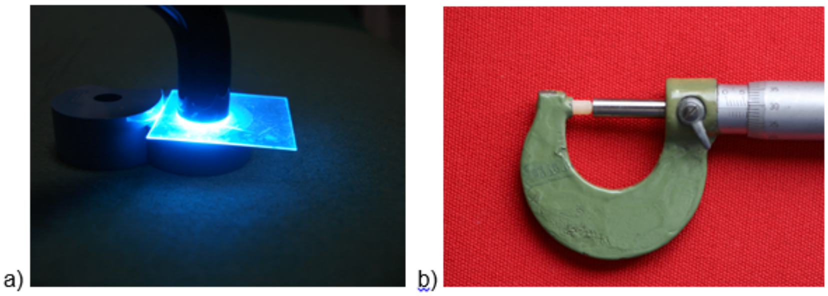


Fig. 3 Irradiation of the specimen in the mold (a), followed by measuring the curing depth with micrometer (b)

Depth curing of the studied composites materials was determined according to their specifications of ISO 4049/ 2000 [27]. For each of the 6 studied composite materials there were achieved 10 specimens. There were used A2 enamel shades for commercial materials, respectively the unique shades A1 (for C13), A2 (for C20) and A3,5 (for C18) provided for experimental composites.

The composite was placed up to the top in a cylindrical mold of opaque plastic, with a diameter of 4 mm and a depth of 8 mm. The mold was then covered with a transparent foil/film and the excess material was pressed with a glass blade. After removing the glass blade, on the surface of the transparent foil there was placed the optical guide of the polymerization halogen lamp Translux Energy®/Heraeus-Kulzer, which has a light intensity of 900 mv/cm^2 , and an irradiation was carried out for 20 s in continuous mode. After polymerization of each of the 5 specimens, the radiation intensity of the lamp was verified with radiometer (Optilux®, KerrHawe, Bioggio, Switzerland). Immediately after curing, the specimens were removed from the mold and uncured composite material was scraped with a spatula palstic.

The height of the strengthened specimen was measured with an accuracy of ± 0.1 mm using a micrometer, and the value obtained was divided by the two resulting the polymerization depth.

Statistical analysis

The average values and standard deviation were calculated for each group of specimens [176]. The variables in the study were the depth of polymerization of the 6 groups of specimens, for which was used the test AVOVA at a significance level of 0.05 and PostHoc Scheffe test. For the analysis of the differences in polymerization depth according to the irradiation time, there was used T-test. For this, it was used PASW Statistics 18.0 software.

Results and discussions

Radioopacity

Quality control of the performance and development of radiographic images was confirmed in figure 4., which represents a standardization of the method. The statistical analysis of the optical density values obtained for each gradation of aluminum standard of three X-rays did not show statistically significant differences.

The Student's test identified significant differences between radioopacity of the tested specimens. The highest radioopacity was presented by the Tetric EvoCeram nanocomposite material. Premise was more radiopaque than Artiste and both are significantly more radiopaque than the three experimental composites C13, C18 and C20. The radiological image corresponding to composite specimen C13 shows visible dispersed filler particles more radiopaque than the rest of material. The composite material C20 has an intermediate radioopacity between the one of dentin and enamel, while the composite C13 a radioopacity very close to that of the enamel (table 2).

All the composites have a higher radioopacity than of the dentin (1mm aluminum), and three commercial materials are more radiopaque than enamel.

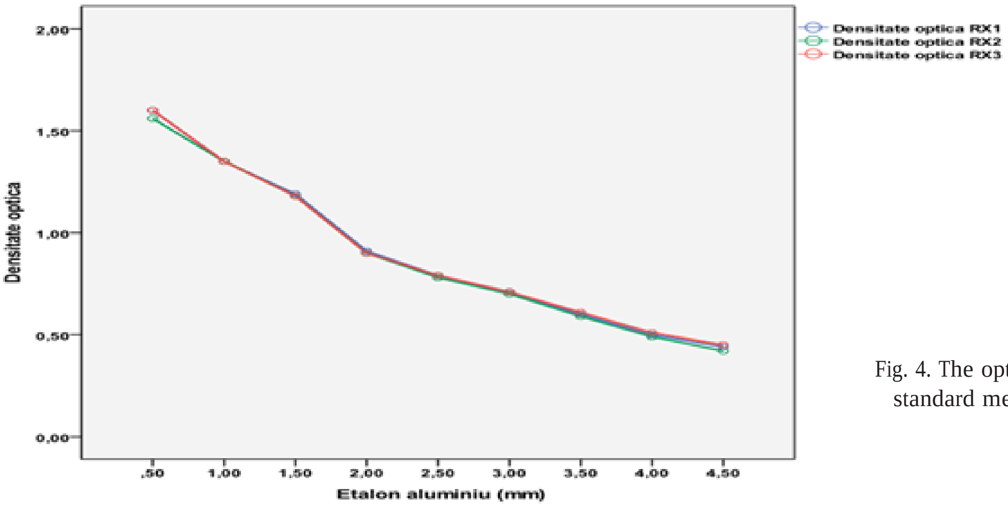


Fig. 4. The optical density of the aluminum standard measured on the three X-rays

Table 2
RADIOOPACITY VALUES FOR THE SIX COMPOSITE MATERIALS (mm ALUMINUM)

Composite materials					
Premise	**Tetric EvoCeram**	**Artiste**	**C13**	**C18**	**C20**
2.6	3.7	2.2	1.9	1.3	1.6

Table 3
THE AVERAGE AND STANDARD DEVIATION FOR THE POLYMERIZATION DEPTH (mm) OF THE TESTED COMPOSITE MATERIALS. N1=N2=N3=N4=N5=N6=10

	Polymerization depth (20 s)		Polymerization depth (40 s)	
	Average	Standard deviation	Average	Standard deviation
Premise	2.65	0.084		
Tetric EvoCeram	2.87	0.067		
Artiste	2.91	0.087		
C13	1.49	0.056	1.85	0.084
C18	1.30	0.066	1.50	0.047
C20	1.25	0.052	1.44	0.051

Table 4
THE RESULTS OF THE ANOVA TEST IN TERMS OF THE DIFFERENCES BETWEEN THE POLYMERIZATION DEPTH (MM) OF THE TESTED COMPOSITES

Group 1	Group 2	Difference between average values (G1-G2)	p
Premise	Tetric EvoCeram	-0.22	**0.0001**
	Artiste	-0.26	**0.0001**
	C13	1.16	**0.0001**
	C18	1.35	**0.0001**
	C20	1.40	**0.0001**
Tetric EvoCeram	Artiste	-0.04	0.898
	C13	1.38	**0.0001**
	C18	1.57	**0.0001**
	C20	1.62	**0.0001**
Artiste	C13	1.42	**0.0001**
	C18	1.61	**0.0001**
	C20	1.66	**0.0001**
C13	C18	0.19	**0.0001**
	C20	0.24	**0.0001**
C18	C20	0.05	0.773

The polymerization depth

The average and standard deviation for the polymerization depth of the composite materials at 20 s, respectively 40 s ares shown in table 3.

The highest value was for the composite Artiste (2.91±0.087 mm) and the lowest for composite C20 (1.25±0.052 mm).

The results of the ANOVA test reveal the fact that between the average values of the six composites there are statistically significant differences ($p<0.0001$) with respect to the polymerization depth for a irradiation time of 20 s. The PostHoc Scheffe test shows which are the pairs of composites and the differences between their average values are statistically significant (table 4). There were statistically significant differences between all the pairs of tested materials, excepting Artiste -Tetric EvoCeram, respectively C18 - C20. The results of the T-test reveal the fact that between the polymerization depth values for a duration of 20 s and reference values of 1 mm for opaque shades, respectively 1.5 mm for the rest of the shades of ISO 4049/2000 Standard, there are statistically significant differences, excepting for the composite C13 and reference value of 1.5 mm (table 5).

All the six tested materials had medium polymerization depths, significantly higher than the reference value of 1 mm, and the three commercial composites were significantly higher compared to the reference value of 1.5 mm. The composites C18 and C20 showed a polymerization depth significantly lower than the reference

Table 5
REPORTING POLYMERIZATION DEPTH (mm) OF THE COMPOSITE AT THE REFERENCE VALUES OF 1 mm, RESPECTIVELY 1.5 mm. N1=N2=N3=N4=N5=N6=10

Composite	Reference value = 1 mm		Reference value = 1,5 mm	
	p	Difference	p	Difference
Premise	**0.0001**	1.65	**0.0001**	1.15
Tetric EvoCeram	**0.0001**	1.87	**0.0001**	1.37
Artiste	**0.0001**	1.91	**0.0001**	1.41
C13	**0.0001**	0.49	0.591	-0.01
C18	**0.0001**	0.30	**0.0001**	-0.20
C20	**0.0001**	0.25	**0.0001**	-0.25

Table 6
COMPARISON OF THE POLYMERIZATION DEPTH (mm) FOR IRRADIATION TIMES OF 20 SECONDS, RESPECTIVELY 40 s. N1=N2=N3=10

Composite	Irradiation time	Average	Standard deviation	p
C13	20 s	1.49	0.056	**0.0001**
	40 s	1.85	0.084	
C18	20 s	1.30	0.066	**0.0001**
	40 s	1.50	0.047	
C20	20 s	1.25	0.052	**0.0001**
	40 s	1.44	0.051	

value 1.5 mm and C13 had a polymerization depth of 1.49 (± 0.056) mm.

The T-test results show that the value of the polymerization depth T of for a period of 40 s is significantly higher than the polymerization depth at 20 s for all the three experimental materials (table 6).

Radioopacity of the composite materials allow the diagnosis of relapsed or marginal tooth decays, gaps, dehiscence of interface or of material in excess from cervical areas, as well as the assessment of marginal and proximal contour, also the contacts with adjacent teeth. For these reasons, it is advisable that the restorative material to be radiopaque; the radioopacity value preferably to be moderate, slightly over the one of enamel in order not to hide any adjacent tooth restoration [1,5,8].

ISO 4049/2000 and ADA specifications No.27 [15,16] provide for the alleged radiopaque restorative materials a minimum of radioopacity equivalent to that of a pure aluminum layer of the same thickness. Radioopacity of enamel is about 2 mm aluminum and the one of dentin is slightly higher than 1 mm aluminum [17,18].

In the present study, the determination of radioopacity was carried out according to the ISO 4049/2000 procedure, method A. Both commercial and experimental nanocomposites have met the requirements of this standard, having radioopacities higher than 1 mm aluminum, but only the commercial composite materials showed a higher radioopacity than of the enamel (over 2 mm aluminum).

Radioopacity is influenced by composition differences in the composite materials, being determined by the type and percentage of the inorganic filler with radiopaque effect: glasses based on heavy metals (barium, strontium, zirconium, etc.), barium-containing ceramic particles, fluorides of lanthanides, aluminum or zirconium oxides [196,201,206]. Inorganic filler of quartz is radiolucent [19].

The most radiopaque composite was Tetric EvoCeram, with a value of 3.7 mm aluminum, likely due to the specific radiopaque filler of ytterbium trifluoride and the high proportion of inorganic fillers (82-83% of weight) [21].

Premises and Artiste nanocomposites had similar values of radioopacities, explained by their common content of barium glass, respectively of zirconium silicate for Artiste. The increased value of radiopacity for Premises could be explained by a significant higher percentage of inorganic filler (84% of mass) [20]. Of the experimental nanocomposites, the most radiopaque was C13, a composite with high percentage of filler (80%) and a content of barium glass. The radiopaque particles which can be detected on the radiological image of the specimen could be due to a higher percentage of inorganic macro-filler, along with micro and nanofiller. The composite C20 shows a homogeneous radioopacity, and as an intermediate value between the enamel and dentin, determined by a filler of glass with strontium and zirconium oxides and hydroxyapatite with zirconium oxide. The most radiotransparent experimental material was C18, possibly due to a higher percentage of quartz in the filler composition.

When used as a first restorative layer of a fluid composite, numerous studies emphasize the need for an increased radioopacity for a better detection of adaptation to the dental substrate [22-24].

The polymerization depth was measured in this study by an indirect method by scraping the soft remaining material and of measuring the thickness of the hardened material, as specified by the ISO4049/2000. Direct methods of measuring the conversion degree, as well as infrared spectroscopy and Raman laser spectroscopy are more complex, more expensive and time-consuming, at the same time they being are more qualitative rather than quantitative [25,26].

The working procedure in this study uses an opaque mold for testing the polymerization depth, although in clinical conditions, the specific optical properties of dental structures allow different transmission of light and thus obtaining other results; for this reason, for a better simulation of the clinical conditions, some authors have tested the polymerization depth in cavities prepared inside of extracted teeth [27,28].

The polymerization depth is an important physical parameter of the composite materials, with a special relevance in choosing the clinical techniques of layering and of the polymerization method [29]. Ideally, the degree of conversion should be the same throughout the mass of filling.

At irradiating a layer of composite material, the light passes through it and looses its intensity due to dispersion and absorption of the filler particles and organic matrix, resulting in decreased efficiency of polymerization of the deeper areas in the material [30,31]. For a given composite, the conversion rate depends on: the light irradiation at the surface of material, exposure time and thermal energy in the system [31,32]. Numerous studies have shown that all the RDCs, after the polymerization, will have a rate of 25-45% residual monomers, so the conversion degree being therefore between 55-75% [32-35]. The polymerization depth is lower in the case of composites with smaller filler particles, these diffusing the light more intensely. Also, increasing the proportion of inorganic fillers or the more opaque and darker shades of the composite materials will adversely affect the polymerization depth [10,11]. For this reason, in this study we chose the same color and opacity (A2 enamel) for all the tested commercial nano-composites.

All these data confirm the recommendation of the most authors that the composite layers should not exceed 2 mm in thickness in order to obtain a satisfactory polymerization [12,13].

The nanocomposites, in the presence of inorganic filler particles smaller than the wavelength of visible radiation allow obtaining shades with a high translucency, which will allow more efficient passage of light through the composite layer [37].

In the present study we tested the polymerization depth for the same hue A2 enamel in the case of the commercial nanocomposites, respectively for the unique hues A1 (for C13), A2 (for C20) and A3,5 (for C18) provided for the experimental composites. Due to the high level of visual opacity, the experimental composites were considered opaque shades/dentin and their polymerization depth was evaluated both for irradiation of 20 s as well as 40 s, in order to determine any significant differences.

The highest values for the polymerization depth were shown in the composites Artiste and Tetric EvoCeram (see table 3), in correlation with their high visual translucency, despite the very high percentage of inorganic fillers (82-83 wt% for Tetric EvoCeram). The polymerization depth values for a irradiation of 20 s with the lamp mentioned above, are between 2.65 (±0.084) and 2.91 mm (±0.087) mm for hue A2 enamel of the commercial materials and they are significantly higher ($p<0.05$) than the reference value (1.5 mm) required by ISO 4049/2000 [27].

In the case of experimental composites, their increased visual opacity advocates for placing them in the group or opaque shades or dentin. This opacity could be due both to a higher percentage of inorganic filler as well as an increased weight of the micron particle which diffuse and absorb the light radiation more pronounced than the nanoparticles. Also, the presence of the nano-aggregated particles causes a lower translucency of the composite than the isolated nanomice particles [11].

At an irradiation of 20 s, the experimental composites had three average values of the polymerization depth from 1.25 (±0.052) mm and 1.49 (±0.056) mm, value significantly higher than the reference value of 1 mm, for which they can be considered and used in the class of opaque shades.

The extension time at 40 s of irradiation significantly increased the polymerization depth in all three experimental materials; composite C13 showed a polymerization depth of 1.85 mm (± 0.084), and composite C18 an average of 1.50 mm (± 0.047), which, according ISO 4049/2000 and ADA specification No.27, enables them to be used as shades of dentin or enamel.

The average value of 1.4 mm for polymerization depth of the composite C20 is placed just below the threshold of 1.5 mm of the above-mentioned standard, which places it in the category of opaque shades [27].

Conclusions

The obtained results in this study showed the link between the composition of materials and their radioopacity degree, and revealed the differences between the polymerization depth of various nanocomposite materials. It has been observed that:

- the tested nanocomposites show values above 1 mm aluminum of radioopacity, meeting the ISO 4049/2000 requirements for radioopacity of direct restorative materials;
- all the commercial compositions present radioopacity values of more than 2 mm, higher than of the dental enamel, which allows a very good radiological assessment;
- the composite Tetric EvoCeram is the most radiopaque, which is explainable by the specific content of ytterbium trifluoride;
- the studied experimental composites are radiopaque at the intermediate values between those of the dentin and enamel, which allows their radiological detection and evaluation;
- the most radiopaque experimental composite is C13 with an increased content of inorganic filler (80% in mass) and barium glass in its composition;
- depth polymerization at an irradiation for 20 s with the used halogen lamp is in a range between 2.65mm (Premise) and 2.98mm (Artiste) for the enamel shades of the commercial composites, significantly greater than the depth of 1.5 mm recommended by ISO 4049/2000 Standard;
- in a 20-second irradiation, the experimental composites have a polymerization depth between 1.25 mm (C20) and 1.49 mm (C13), just enough for the only classified opaque shades according to ISO 4049/2000 Standard;
- increasing the duration of irradiation to 40 s leads to a significant increase of the polymerization depth at an accepted level for enamel or dentin shades in the case of C18 and C13, but not in the case of C20;
- in direct restorations by layering, the placement thickness of composite materials must not exceed 2 mm.

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