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Laboratory Research

Longitudinal Evaluation of Radiopacity of Resin Composites: Influence of Photoactivation and Accelerated Photoaging

AD Cruz • IAM Costa • FS Calazans • MF Aguiar • MO Barceleiro

Clinical Relevance

Radiopacity has been considered an unchangeable property with great significance for assessing the quality of a restoration by radiographic images. Any changes in this property over time, under the influence of photoactivation and photoaging, might affect radiographic diagnosis.

SUMMARY

This study aimed to assess longitudinally the radiopacity of resin composites under the influence of photoactivation and photoaging processes. Ten specimens (1 mm thick and 4 mm in diameter) of three different microhybrid resin composites, Filtek Z250 XT (R1), TPH 3 Spectrum (R2), and Opallis (R3), were prepared for this study. For longitudinal assessment of radiopacity, radiographic images were obtained five times. The first time (T1), the specimens were not photoactivated; the second time (T2), the specimens were photoactivated; and the next three times, photoaging was carried out, with images obtained at 24 hours (T3), 48 hours (T4), and 72 hours (T5) after this process. The photoaging was conducted using LED light (700 lumens, 9 W, 6400 k) under controlled environmental conditions at 37°C (±1°C) and 65% (±5%) relative humidity. The digital system DIGORA Optime was used. The digital images were measured using the histogram function, and then the pixel intensity values were converted into mmAl (the standard unit of radiopacity) using a linear regression function, with minimal adjustment of $R^2 \geq 0.9$. Data in mmAl were statistically analyzed using an analysis of variance (α=0.05). R2 resin composite showed higher values of radiopacity, R1 resin composite showed intermediate values, and R3 resin composite showed lower values. Only at T1 did

*Adriana Dibo da Cruz, DDS, MsC, PhD, Dental School, Fluminense Federal University, Nova Friburgo, RJ, Brazil

Ingrid Assis Mota Costa, DDS, Dental School, Fluminense Federal University, Nova Friburgo, RJ, Brazil

Fernanda Signorelli Calazans, DDS, MSD, PhD, Dental School, Fluminense Federal University, Nova Friburgo, RJ, Brazil

Marcelo Freitas Aguiar, DDS, MsC, PhD, Dental School, Fluminense Federal University, Nova Friburgo, RJ, Brazil

Marcos Oliveira Barceleiro, DDS, MSD, PhD, Dental School, Fluminense Federal University, Nova Friburgo, RJ, Brazil

*Corresponding author: Prof. Adriana Dibo da Cruz Universidade Federal Fluminense, Rua Dr Silvio Henrique Braune, 22, Centro, Nova Friburgo, Rio de Janeiro, Brazil. 28625-650; e-mail: adrianadibo@id.uff.br

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Table 1: *List of Materials Tested in This Study*

Material	Abbreviation	Manufacturer	Shade
Filtek Z250 XT	R1	3M ESPE, St Paul, MN, USA	A3
TPH 3 Spectrum	R2	Dentsply International Inc, Petropolis, RJ, Brazil	A3
Opallis	R3	FGM Produtos Odontológicos, Joinville, SC, Brazil	A3

the higher radiopacity of R2 composite differ significantly from other groups ($p = 0.0000$). After application of treatments (photoactivation and photoaging), all radiopacity values were similar (p-values to T2=0.0507, T3=0.0536, T4=0.0502, T5=0.0501) due to consecutive increase of radiopacity of R1 and R3 composites from T2. Photoactivation and photoaging processes influenced the radiopacity, but changes occurring in the degree of radiopacity were dependent on the composition and chemical characteristics of each composite used.

INTRODUCTION

Conventional two-dimensional radiographic images have been routinely used as the method of choice for oral diagnoses in general dentistry. However, despite the current revolution in radiology that has been driven by the advent of digital systems, some limitations remain unchanged because radiographic diagnosis, a "visual specialty," continues to depend on the observer's ability to interpret radiographic images.[1]

Radiographic images are maps of x-ray attenuation coefficients, which largely depend on the physical and chemical properties of the radiographed objects[2] in addition to their three-dimensional characteristics. Radiographic diagnosis is very challenging because there is a wide range of attenuation coefficients that cause dissimilar contrasts in images formed by different complex objects from facial areas, including restorative materials, lesions, and material prosthetics. Any change in x-ray attenuation coefficients can cause a variation in the resulting image and, consequently, could affect the observer's performance in a specific diagnosis. In the study of Cruz and others,[3] it was observed that radiopacity from materials with attenuation was statistically similar, ranging from dentin to enamel, which can promote a subjective influence on the diagnosis of secondary caries–like lesions, with the highest radiopacity, closer to the enamel, causing a negative influence.

There are numerous commercially available resin composites with different compositions that generate dissimilar radiopacities.[3-5] According to draft technical regulations,[6] the radiopacity of a composite material needs to be equal to or greater than that produced by a reference of aluminum (Al) of the same thickness (mmAl is the standard unit of radiopacity) and cannot vary 0.5 mmAl below any value. The Al used to produce the image reference shall have a minimum technical purity of 98% (less than 0.1% copper and less than 1% iron). Nevertheless, in spite of these draft technical regulations, the degree of radiopacity of resin composites clinically identified as the best for radiographic diagnosis has not yet been established.

Thus, considering that the radiographic diagnosis can be affected by changes in radiopacity, the aim of this study was to assess longitudinally the radiopacity of resin composites under the influence of photoactivation and photoaging processes. The radiopacity was initially considered as an unchangeable property; thus, the null hypothesis was that neither photoactivation nor photoaging would likely influence radiopacity in any way.

METHODS AND MATERIALS

The materials evaluated in this study (listed in Table 1) consisted of three different dental microhybrid resin composites, as follows: Filtek Z250 XT (R1; 3M ESPE, St Paul, MN, USA), TPH 3 Spectrum (R2; Dentsply International Inc, Petropolis, RJ, Brazil), and Opallis (R3; FGM Produtos Odontológicos, Joinville, SC, Brazil). The information about the components is listed in Table 2.

For preparation of 10 specimens of each material, resin composites were inserted into 1-mm-thick stainless steel ring molds with an internal 4-mm-diameter hole. In order to achieve uniformly smooth surfaces, the molds were placed between two glass slides covered with Mylar strips and then submitted to 1 kg/cm^2 pressure for 1 minute to remove the excess material. The target was to obtain the first digital radiographs of these resin composites without the action of polymerization process for the longitudinal assessment. Therefore, at the first time (T1), the resin composites were not photoactivated as described in Table 3. At the next step, the second

Table 2: *Composition/Information on Ingredients of the Studied Materials Cited in Material Safety Data Sheet Available*

Material's Abbreviation	Ingredient	CAS No.	Percentage[a]
R1	Silane-treated ceramic	444758-98-9	65–90
	Bis-GMA	1565-94-2	1–10
	Silane-treated silica	248596-91-0	1–10
	Bis-EMA	41637-38-1	1–10
	UDMA	72869-86-4	1–10
R2	Fiberglass wool	65997-17-3	<50
	Lead bisilicate	65997-18-4	<30
	TEGDMA	109-16-0	<10
	Bis-EMA	24448-20-2	<10
	Urethane modified bis-GMA dimethacrylate	—	<10
	Siloxanes and silicones, di-Me, reaction products with silica	67762-90-7	<3
	Silane, dichlorodimethyl-, reaction products with silica	68611-44-9	<3
	titanium dioxide	13463-67-7	<1
	Colorants	—	—
	Inorganic iron oxides	—	—
R3	Silane-treated ceramic	444758-98-9	65 – 75
	Bis-GMA	1565-94-2	6 – 8
	Bis-EMA	24448-20-2	5 – 10
	Silane-treated silica	248596-91-0	5 – 10
	Bis(2-methacryloxyethyl)-N,N′-1,9-nonylene biscarbamate	41137-60-4	5 – 10
	TEGDMA	109-16-0	<5
	Ethyl 4-dimethylaminobenzoate	10287-53-3	<1
	DL-camphorquinone	465-29-2	<1

Abbreviations: Bis-EMA, bisphenol-A ethoxylate dimethacrylate; Bis-GMA, bisphenol-A glycidyl methacrylate; di-ME, Dimethylsilicone; R1, Filtek Z250 XT; R2, TPH 3 Spectrum; R3, Opallis; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.
[a] *Specific chemical identity and/or exact percentage (concentration) of all chemical components of the resin composites are not available due to trade secrets.*

time of treatment (T2), the photoactivation process occurred. Thus, these resin composites were photoactivated for 20 seconds, according to the manufacturer's instructions, using an Elipar S10 (1200 mW/cm^2) LED curing light unit (3M ESPE). The light intensity of the LED curing device was measured after every five uses by radiometer (SDI LED Radiometer/ SDI Victoria, Australia) at a distance of 0 mm, checking for any variation. Then, the Mylar strips were removed. Immediately after photoactivation, the second digital radiographs of the same resin composites were obtained. In the next three steps, involving the photoaging process and occurring over a period of 72 hours, the digital radiographs were obtained at 24 hours (T3), 48 hours (T4), and 72 hours (T5) after the photoaging process of the same resin composites. The photoaging was conducted using an LED light (Superled Ouro 100, Ourolux, Brazil) at luminous efficacy of 700 lumens, color temperature of 6400 k, power of 9 W, under controlled environmental conditions at 37°C (±1°C) and 65% (±5%) relative humidity.

The radiographic images (Figure 1) were obtained using a digital system using a receptor of the photostimulable phosphor plate system (DIGORA Optime; Soredex, Milwaukee, WI, USA) in a Helio-

Table 3: *Treatments Applied for Each Specimen*

Sequence of Treatment	Abbreviation	Photoactivation	Photoaging	Radiography
First time	T1	No	No	Yes
Second time	T2	Yes	No	Yes
Third time	T3	Yes	Yes for 24 h	Yes
Fourth time	T4	Yes	Yes for 48 h	Yes
Fifth time	T5	Yes	Yes for 72 h	Yes

Figure 1. *Illustration of radiographic technique. (A): Front view of set of objects, cylindrical locator device of the x-ray tube and acrylic plate, (B): a top view of set of aluminum (Al) wedge and stainless steel ring molds with resin specimens, (C): the Al wedge, (D): a card bite cover of the photostimulable phosphor plate, with (E): active side and (F): opposite side.*

dent 60B x-ray machine (Siemens, Erlangen, Germany) that operated at 60 kVp, 10 mA, 40-cm focus–receptor distance, and 0.12-second exposure time. A 1.2-cm-thick acrylic plate was placed between the objects and the cylindrical locator device of the x-ray tube to replicate the soft tissue.

For calculating the radiopacity of the resin specimens, in terms of their Al-equivalent thicknesses (mmAl), one Al wedge (99.8% purity) with 12 steps, each 1-mm thick, was used as the internal standard of radiopacity. Thus, all radiographic images were obtained from the set of a specimen of each material and the Al step wedge (Figure 2). Each set was radiographed three times.

The digital radiographs were measured using the histogram function of the ImageJ 1.43u software (Wayne Rasband, National Institutes of Health,

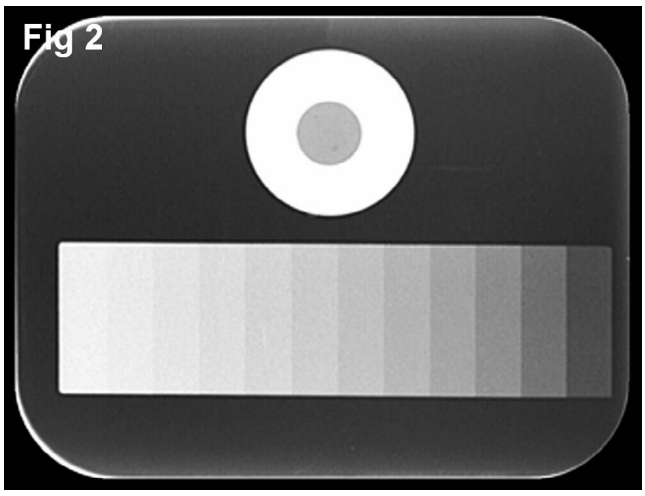

Figure 2. *Sample of image with set of specimen and aluminum step wedge used in this study.*

Table 4: *The Mean and Standard Deviation (SD) Values in Aluminum- (Al-) Equivalent Thickness (mmAl) of Each Material Tested in Accordance With Treatment Applied*[a]

Materials	Treatment				
	T1	T2	T3	T4	T5
R1	1.974 (0.548) B	2.574 (0.481) AB	3.032 (0.398) A	3.246 (0.685) A	3.441 (0.341) A
R2	3.731 (0.455) A	3.252 (0.588) A	3.322 (0.480) A	3.556 (0.602) A	3.531 (0.405) A
R3	1.486 (0.150) B	2.445 (0.390) AB	2.468 (0.481) AB	2.530 (0.402) AB	2.626 (0.365) A

Abbreviations: R1, Filtek Z250 XT; R2, TPH 3 Spectrum; R3, Opallis; T, time.
[a] *Statistically significant difference ($p < 0.05$, by the analysis of variance [ANOVA]–Tukey test) within a column between different materials. Dissimilar letters in one row designate radiopacities that differed ($p < 0.05$, by the ANOVA–Tukey test) between different treatments.*

Bethesda, MD, USA). A trained evaluator collected three areas of the same size in regions of interest (ROIs) on the center of each resin specimen and on each step of the wedge. For an accurate delimitation of each ROI, the image was enlarged. All data relating to pixel intensity values were tabulated and then converted into mmAl using a linear regression function. The minimal adjustment was found to be $R^2 \geq 0.9$.

Data in mmAl were tabulated and then statistically analyzed using an analysis of variance (ANOVA). For statistical analysis, there was no dissociation of polymerization and photoaging factors once the methodology was longitudinal for each specimen. All statistical analyses were conducted with a significance level setting of 5% (α=0.05).

RESULTS

Table 4 shows the values of radiopacity from different resin composites in terms of their Al-equivalent thicknesses. TPH 3 Spectrum (R2) resin composite showed higher values of radiopacity, Filtek Z250 XT (R1) resin composite showed intermediate values, and Opallis (R3) resin composite showed lower values. However, despite the differences in radiopacity values, only at the first time (T1) did the higher values of radiopacity of the TPH 3 Spectrum resin composite differ statistically significantly (p=0.0000) from others. After application of the treatments (from T2 on), all radiopacity values were brought closer (p-values to T2=0.0507, T3=0.0536, T4=0.0502, T5=0.0501) because resin composites Filtek Z250 XT (R1) and Opallis (R3) had a variation in radiopacity by a consecutive increase from the first time (T1). In Filtek Z250 XT (R1), this increase in radiopacity was statistically significant (p=0.0318) compared with that at the third time (T3). In Opallis (R3), the radiopacity was statistically significant (p=0.0447) at the fifth time (T5). Conversely, in TPH 3 Spectrum (R2), the radiopacity was more stable after the treatments, without significant differences (p>0.05).

DISCUSSION

In this short-term laboratory study, the radiopacities from different microhybrid resin composites were longitudinally evaluated under the action of time, from the first uncured stage to further late stages under polymerization and with induced photoaging. This methodology for evaluation of radiopacity differed from other previous studies commonly found in the literature[3,5,7-10] because radiopacity has always been evaluated in a static mode, transversely. In the current study, conversely, radiopacity was assessed more dynamically under the action of treatments over time. Moreover, against expectations and partially rejecting the null hypothesis, this property was changeable with the type of composite used and underwent changes according to the properties of each material. Thus, under the action of polymerization and photoaging, this property may be subject to changes that typically vary according to the chemical characteristics of each material.

Radiopacifying agents, which differ in their constituents, combinations, concentrations, and particle sizes, possess distinct x-ray attenuation coefficients that cause variation in radiopacity.[3-5,8,9,11-13] An ideal radiopacifier should be inert to content and be nonhazardous, possessing an adequate attenuation coefficient to enable its visualization on radiographic images without compromising other material properties such as wear resistance, degree of conversion, and polymerization shrinkage.[11,14-16] However, due to trade secrets, the specific chemical identity and/or exact percentage (concentration) of each of these chemical components of commercially available resin composites is not always accessible.

Fundamentally, all dental resin composites contain an organic matrix and inorganic fillers as ingredients. All physical properties of the resin composite are critically influenced by both the chemical structure of the monomers used in the matrix phase and the properties of their fillers. The final radiopacity of the composite is therefore largely

derived as a result of the sum of radiopacities from these different ingredients,[14] with the inorganic fillers causing a higher attenuation of x-rays. Amirouche and others[14] described all the theoretical background information about attenuation coefficients of x-rays by the dental materials according to the atomic number of their radiopacifying agent and their spatial distribution, where a high atomic number results in radiopaque images and their high concentration per area results in more radiopacity in the image. Valente and others[11] reported that the size of the inorganic fillers could not change the radiopacity when comparing dental resin composites of similar elemental composition of the filler systems but with micron- and submicron-sized monomodal glass filler particles. However, these authors emphasized that in spite of a similar elemental composition, there was a higher concentration of filler in the submicron composite. It is probable that this increase in concentration of the submicron particles promoted a compensation in attenuation, which was consequently able to promote similar radiopacity to that caused by the higher particles. In the current study, all materials evaluated were commercially available microhybrid resin composites, which are still very popular and widely used in several countries. They had particles relatively similar in size, with average values that vary from 0.1 to 0.6 μm, but had dissimilarities in elemental compositions. There are no studies in the available literature using such an approach comparing microhybrid, nanohybrid, and nanofilled resin composites, preventing any such supposition in this regard. Thus, for a better understanding on this subject, further study, using not only microhybrid, which was a limitation of the present study, but also nanohybrid or nanofilled composites, should be performed.

In the current study, the material with higher radiopacity was TPH 3 Spectrum (R2), probably due to titanium dioxide and inorganic iron oxides, which have higher atomic numbers. Silica and ceramic, the primary radiopacifying agents of resin composites Filtek Z250 XT (R1) and Opallis (R3), have the lowest atomic numbers, causing the lowest x-ray attenuation. However, in the current study, statistically significant differences in radiopacity were observed only at the first time (T1), when the composites were yet uncured. After polymerization, composites Filtek Z250 XT (R1) and Opallis (R3) did not differ significantly from TPH 3 Spectrum (R2). Thus, after polymerization, all radiopacities were statistically similar. The same situation was seen in previous studies[3,8,10,12,17] in which, despite clinical radiopacity having had variation and causing changes in the diagnostic accuracy,[3] no statistically significant difference was observed among them.

The increase in radiopacity observed in composites Filtek Z250 XT (R1) and Opallis (R3) after polymerization could be justified by polymerization shrinkage that causes closer spatial approximation between particles of radiopacifiers. The polymerization process can promote an overall reduction in the distance between the molecules of matrix because monomers react to form a covalent bond resulting in volumetric shrinkage in the final polymer network.[15] Evidence has been reported in the literature[11,15,18,19] that the volumetric shrinkage of composites is proportional to its degree of conversion. The ideal for mechanical properties would be a composite having a minimal polymerization shrinkage with an optimal degree of conversion. Unfortunately, however, the degree of conversion depends on several complex interconnected factors, ranging from chemical characteristics of the resin composites used to methodological and environmental interferences. The inorganic compounds have important roles in determining the physical and mechanical properties of resin composites.

In relation to the behavior of attenuation of radiation by resin composites over time, under the influence of induced photoaging, two distinct patterns observed in the current study can be described. One pattern was shown by composites Filtek Z250 XT (R1) and Opallis (R3), whose behavior was the increase in radiopacity over time, from the first stage after polymerization. This behavior of increasing radiopacity over time could be initially justified by the polymerization shrinkage described before that causes a closer approximation between particles, promoting the highest attenuation of radiation. In the study by Lau and others[16] it was observed that there is a continuing shrinkage even after the end of the 40-second photoactivation, with the shrinkage strains measured at 10 minutes being significantly greater than those measured at 40 seconds. However, in addition to initial polymerization shrinkage, a more important cause of dimensional change could be a bias of methodology, which was caused during the photoaging process because the specimens were maintained under a condition of relative humidity and were exposed to light. In this case, the resin composites Filtek Z250 XT (R1) and Opallis (R3), both with bisphenol-A glycidyl methacrylate, a hydrophilic compound, might have had a higher dimensional change by the absence of immersion in

water added at dehydration caused by the environment with 65% (±5%) relative humidity and by the light exposure, which may interfere in the hygroscopic expansion causing an over-shrinkage.[20,21] The other pattern was shown by composite TPH 3 Spectrum (R2), in which the radiopacity remained constant over time after applying the treatments. This behavior, on the contrary, could be justified by the highest proportion of inorganic fillers in the matrix, which might cause a higher tolerance to water sorption[20] and, consequently, smaller dimensional changes due to bias of methodology, being therefore more stable to treatments.

Furthermore, it must be emphasized that the results of laboratory evaluations are not directly applicable to the oral environment; there are several huge challenges in which dental restorative materials must withstand the widely varying conditions, including temperature fluctuations, continuous exposure to moisture, mechanical stresses, and a more extensive aging protocol. Thus, further studies comparing longitudinally the radiopacity of different materials under the influence of hydrothermal cycling and controlled clinical trials are necessary to substantiate the validity of the present results. However, an implicated direct outcome of the current study is related to the time for evaluation of radiopacity in future studies. Thus, time is a variable that should be strictly controlled between preparation of specimens and analysis of radiopacity in all groups.

CONCLUSION

The null hypothesis of the present study was partially rejected for the reason that photoactivation and photoaging processes influenced the radiopacity, but changes occurring in the degree of radiopacity were dependent on the composition and chemical characteristics of each composite used. Thus, an increase of resin composite radiopacity over time may occur, causing interference in radiographic diagnosis using digital radiographic images.

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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