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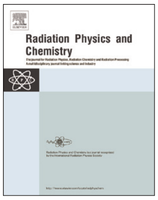

Influence of ionizing radiation on the mechanical properties of BisGMA/TEGDMA based experimental resin

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HIGHLIGHTS

- Interactions of ionizing radiation in BisGMA/TEGDMA experimental dental composites filled with sylanized silica.
- Process of post-curing by ionizing radiation at low doses.
- Effect of ionizing radiation on mechanical properties of experimental dental composites.

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ABSTRACT

Dental restorative composites are activated by visible light and the polymerization process, known as direct technique, is initiated by absorbing light in a specific wavelength range (450–500 nm). However this technique presented some disadvantages. If light is not inserted correctly, layers uncured can cause countless damage to restoration, especially with regard to mechanical properties. A clinical alternative used to reduce the shortcomings of direct application is the use of composite resins for indirect application. These composites are adaptations of resins prepared for direct use, with differences mainly in the healing process. Besides the traditional photoactivation, indirect application composites may be submitted to particular curing conditions, such as a slow curing rate, heating, vacuum, and inert-gas pressure leading to an oxygen-free environment. However few studies have been conducted on the process of post-curing by ionizing radiation at low doses. On this sense the purpose of this study was to evaluate possible interactions of ionizing radiation in the post-curing process of the experimental composites based on BisGMA/TEGDMA filled with silica Aerosil OX-50 silanized. Characterization of the experimental composites was performed by thermogravimetry analysis, infrared spectroscopy, elastic modulus and flexural strength. Statistical analysis of results was calculated by one-way ANOVA/Tukey's test. Cross-linking of the polymeric matrix caused by ionizing radiation, influenced the thermal stability of irradiated specimens. FTIR analysis showed that the ionizing radiation induced a post-cure reaction in the specimens. The irradiation dose influenced directly the mechanical properties that showed a strong positive correlation between flexural strength and irradiation and between modulus strength and irradiation.

1. Introduction

Composite resin used for adhesive dental restoration has as main advantages the realization of minimum cavity preparation and superior esthetics (Sun-Hong et al., 2010). Over the last decade, the constant search for esthetics has turned the utilization of the aforementioned materials as a replacement of the non-esthetic reparation materials. Furthermore, the use of dental amalgam is foregone by an increasing number of professionals due to the possibility of systemic mercury toxicity (Rathore et al., 2012).

Meanwhile, several experiments have been carried out over the last few years in order not only to improve the physical and mechanical properties of the material in question, but also to optimize its performance in terms of polymerization shrinkage and residual stress, high sorption or solubility, among others (Sideridou and Karabela, 2011; Calheiros et al., 2004).

Restorative composites react by a mechanism of free radicals which are generated by the chemical reaction of benzoyl peroxide

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with a tertiary amine, initiating the polymerization of methacrylic groups and forming a three-dimensional polymer matrix. They are activated by visible light, initiating their polymerization process by absorbing visible light within a specific wavelength (450–500 nm) (Hammouda, 2010).

In the direct technique, the composite is cured directly on the tooth by means of a curing light, however this technique presents some disadvantages. For instance, if the light source is not placed correctly, uncured or partially cured layers can cause countless damage restoration, especially with respect to the mechanical properties (Visvanathan et al., 2007). In the direct technique there is not the possibility of a post-curing process. Recently published investigations have shown that post-curing of dental resin restorative materials may improve properties such as flexural strength or wear resistance (Behr et al., 2005).

A clinical alternative used to reduce the shortcomings of direct application, is the use of composite resins for indirect application. These are adaptations of direct use resins that differ mainly in the curing process. In the indirect technique, the remaining tooth structure is reconstructed into a mold and the material is cured and post-cured under controlled laboratory conditions. This has been demonstrated to improve the mechanical properties such as hardness and flexural strength due to an increase in the degree of conversion (Lovell et al., 2001).

In regards to the traditional photoactivation, indirect application composites may additionally be subjected to particular curing conditions, such as a slow curing rate, heating, vacuum, and inert-gas pressure leading to an oxygen-free environment (Suresh, 2010). Slow curing rate allow for higher degree of conversion (Mehl et al., 1997), whereas heat polymerization provides greatly increased chain mobility resulting on the residual stress reduction, with a risk of superheating that may cause degradation of the composite (Viljanen et al., 2007). Photopolymerization with nitrogen pressure can extensively eliminate the entrapped oxygen before curing occurs (which improves conversion degree) and also reduces abrasion and yields better optical properties (Leinfelder, 2005). However, few studies have been conducted on the process of post-curing by ionizing radiation at low doses.

Ionizing radiation is a powerful tool in modifying and improving the properties of polymeric materials. The transferred high energy from incidental radiation is the origin a wide variety of excited state molecules and ions along tracks that may lead to subsequent reactions such as polymerization, crosslinking, grafting and degradation (Jipa et al., 2012; De Paoli, 2008; Spinks and Woods, 1990). Unlike ultraviolet radiation, the absorption of ionizing radiation takes place in a random manner. Ionizing radiation has a wavelength between 0.01 and 0.05 nm and consequently higher penetration amounts than ultraviolet (Cruz et al., 2009).

Published investigations have shown that post-curing of dental composite materials may improve properties such as flexural strength, wear resistance, fracture strength and micro hardness (Kamel and Neri, 1979; Von-Fraunhofer et al., 1989; Haque et al., 2001). However color changes measured lead to the expectation that electron beam irradiation at high doses (25–200 kGy) may have an unfavorable clinical appearance (Behr et al., 2005; Kolbeck et al., 2007). Therefore, the current paper aims to evaluate any possible applications of ionizing radiation (at low doses) for post-curing applications in BisGMA/TEGDMA experimental composites filled with Aerosil OX-50 silanized silica content, as well as any changes on its thermal and mechanical properties.

2. Materials/methods

2.1. Materials

The following materials were used in this study: BisGMA: (Bisphenol A bis(2-hydroxy-3-methacryloxypropyl)ether) manufactured by Esstech (Essington, USA); TEGDMA: (Triethyleneglycol Dimethacrylate) produced by Esstech (Essington, USA); Camphorquinone: (camphorquinone, 97%) provided by Sigma-Aldrich (Germany); DMAEMA: (2-(Dimethylamino)ethyl methacrylate), 98% purchased form Sigma-Aldrich (Germany); Silica: Aerosil OX-50 Sylanized provided by FGM, Brazil.

2.2. Methods

2.2.1. Experimental composite preparation

The experimental composites were prepared by manual mixing with incorporation of the inorganic filler in the polymeric matrix, as reported by Campos et al., (2014a) and Campos et al., (2014b).

Ten groups of experimental composites were prepared from a BisGMA/TEGDMA base (48%wt) filled with Silica Aerosil Ox-50 silanized (50%wt). The BisGMA was incorporated into TEGDMA in equal proportions (1:1) in all groups, as well the camphorquinone (1%wt) and the tertiary amine DMAEMA (1%wt). After manipulation of the polymer matrix, the inorganic filler was added in small amounts until the entire compounds incorporation. For a correct homogenization of the mixture, the experimental composite was manipulated for 5 min. The materials were individually weighed on an electronic scale digital precision, Shimadzu/model AY220.

Five groups were subjected to ionizing radiation without previous photopolymerization, except control group (not photopolymerized/not irradiated). Others five groups were photopolymerized for 40 s (using Kondorteck's Aigh-7A LED, at 470 nm wavelength, commonly used to the dental composite cure) and subjected to ionizing radiation twenty four hours after curing, except control group (photopolymerized/not irradiated). The description of the experimental composites can be found in table 1.

Irradiation was carried out on a Gammacell model 220, with ^{60}Co source. Irradiation dose ranged from 0.25–2 kGy and dose rate was of 1.164 kGy/h^{-1}.

2.2.2. Thermogravimetry analysis (TGA)

The thermal stability and decomposition profile of the specimens was observed by thermogravimetry analysis ranging from 50 to 800 °C at the heating rate of 10 °C min^{-1}, on inert and oxidizing atmospheres on a Mettler Toledo TGA/SDT851.

Initially, groups of specimens that were subjected to ionizing radiation but had not been previously cured were analyzed. Afterwards, groups of specimens that were subjected to ionizing radiation after photopolymerization were analyzed.

Table 1
Description of experimental composites groups.

Group	Radiation dose (kGy)	Previously photopolymerized
1	–	No
2	0.25	No
3	0.5	No
4	1	No
5	2	No
6	–	Yes
7	0.25	Yes
8	0.5	Yes
9	1	Yes
10	2	Yes

2.2.3. *Fourier transform infrared spectroscopy-(FTIR)*

The experimental composites were analyzed by Fourier transform infrared spectroscopy to measure how the specimens absorb ionizing radiation, using attenuated total reflection (FTIR-ATR) on a ThermoNicolet 6700 FTIR spectrometer, with a scan ranging from 4000 to 500 cm^{-1}, 64 scans per minute in a non-controlled atmosphere. Samples of experimental composites were prepared according to the formulation pressed on the mold to have a perfect surface for reflectance infrared analysis on the FTIR-ATR. The optimum thickness of specimens was 0.50 mm. A Digital Caliper, Digital 6 in. model (0-150 'm) was used to check the samples' thickness.

2.2.4. *Flexural modulus and flexural strength*

Specimens $10 \times 2 \times 1$ mm were prepared using a split steel mold. The specimen was removed from the mold and subjected to three-point bending in a universal testing machine (Instron 5565 Canton, MA, USA), with 8 mm distance between the supports and a cross-head speed of 0.5 mm/min. Ten samples were tested for each group ($n=10$). Only the previously photopolymerized and irradiated groups were tested.

Based on the linear portion of the loadxdisplacement curve, flexural modulus was calculated according to the following formula (Boaro et al., 2010):

$$E = \frac{L \times D^3}{4 \times w \times h^3 \times d} x \ 10^{-3}$$

where E is the flexural modulus (GPa), L is the load recorded (N), D is the span between the supports, w is the width of the specimen, h is the height of the specimen and d is the deflection corresponding to L (all in mm).

Flexural strength was calculated according to the following formula (Boaro et al., 2010):

$$\sigma = \frac{3 \times F \times L}{2 \times b \times h^2}$$

where: σ is the flexural strength (MPa), F is the maximum load recorded before fracture (N), L is the span between the supports (mm), b is the width of the specimen (mm), h is the height of the specimen (mm).

3. Statistical analysis

Data for elastic modulus and flexural strength were analyzed using one-way ANOVA/Tukey's test. For both tests, the global significance level was 5%. Regression analyzes were performed to establish correlations among flexural modulus/flexural strength with absorbed dose.

4. RESULTS

4.1. *TGA*

The analysis results can be seen in Table 2, where mass loss was observed as function of temperature in the range from 50 to 800 °C at 10 °C min^{-1}.

Initially groups subjected to ionizing radiation, without preceding photopolymerization, were considered. In Fig. 1A, the control group (not cured/not irradiated) displayed the most significant mass loss in two different steps. The first step takes place between 100 and 319 °C, corresponding to a 21.9% mass loss and the second between 319 and 480 °C, corresponding to a 28.5% mass loss as commonly observed for BisGMA/TEGDMA matrix composites. A third step however, took place between 480 and 750 °C which could be related to the decomposition of silanols present in silica surface.

With regard to the group 2 (0.25 kGy) the first step occurred between 168 and 378 °C (corresponding to 11.6% mass loss) and the second step between 378 and 480 °C (corresponding to 32.9% mass loss). For group 3 (0.5 kGy) the first step occurred between 155 and 366 °C (corresponding to 11.7% mass loss) and the second step between 366 and 480 °C (corresponding to 32.6% mass loss). For group 4 (1 kGy) the first step occurred between 150 and 368 °C (corresponding to 11.8% mass loss) and the second step between 366 and 480 °C (corresponding to 38.2% mass loss). For group 5 (2 kGy) the first step was identified between 153 and 370 °C (corresponding to 12.9% mass loss) and the second step between 370 and 480 °C (corresponding to 36.1% mass loss).

For previously photopolymerized groups it can be seen (Fig. 1B), that in all groups (including control group) the mass loss took place between 250 and 500 °C. This was due to the previous formation of bonds through photopolymerization. Thus it was only possible to notice the effects of ionizing radiation in the non-previously-photopolymerized specimens.

4.2. *FTIR*

In Fig. 2, the main bands related to the previously-photopolymerized and irradiated groups were identified. The peaks at 1720 and 1634 cm^{-1} correspond, respectively, to the stretching of carbonyl group (C=O) and to the stretching of double bonds between carbons (C=C). The peak at 1609 cm^{-1} corresponds to axial deformation of the C–C bonds and the peak at 1509 cm^{-1} to aromatic double C=C bonds, both present in BisGMA. The peak at 1251 cm^{-1} corresponds to a superposition of peaks from the axial deformation of ether groups (C–O–C) and from ester groups (COO) in the BisGMA/TEGDMA matrix.

The peak at 2300 cm^{-1} was attributed to the formation of CO_2 due to the decomposition of polymeric matrix in the irradiated groups. Analogous to the thermal decomposition, the most obvious source of carbon dioxide from both the resin and its

Table 2
Results of thermogravimetry analysis: Thermal stability and decomposition profile of the specimens.

Non-previously-photopolymerized					Previously-photopolymerized				
Groups (dose)	1st weight loss		2nd weight loss		Groups (dose)	1st weight loss		2nd weight loss	
	T1 (°C)	weight loss (%)	T1 (°C)	weight loss (%)		T1 (°C)	weight loss (%)	T1 (°C)	weight loss (%)
1 (Control)	100.9	22.0	318.5	28.5	6 (control)	250	68.4	500	31.6
2 (0.25kGy)	158.1	11.5	368.6	32.9	7 (0.25kGy)	250	66.5	500	33.5
3 (0.5 kGy)	155.7	11.6	366.1	32.6	8 (0.5 kGy)	250	66.1	500	32.9
4 (1 kGy)	150.8	11.8	368.4	38.2	9 (1 kGy)	250	66.9	500	33.1
5 (2kGy)	153.4	13.0	370.9	36.1	10 (2kGy)	250	66.6	500	33.4

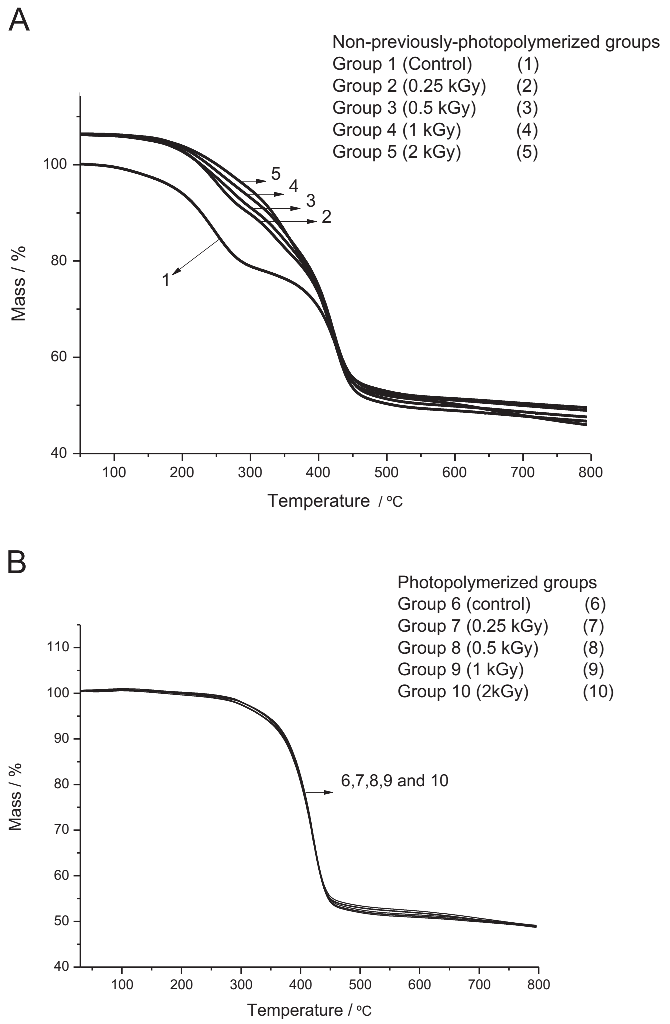

Fig. 1. Thermal degradation curve of irradiated and non-previously-photopolymerized groups (1A). Thermal degradation curve of irradiated and photopolymerized groups (1B).

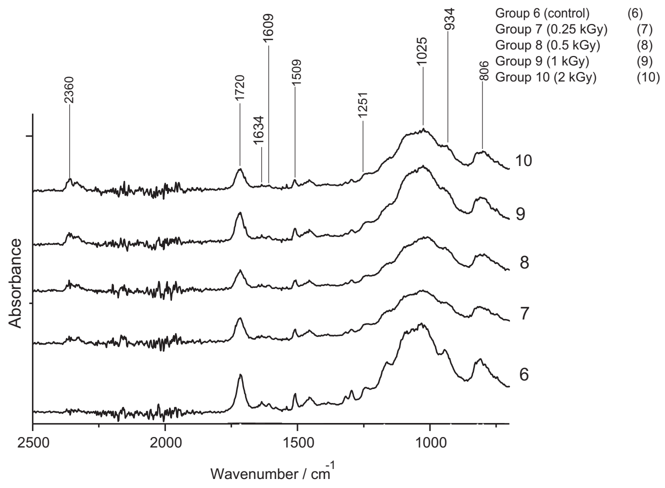

Fig. 2. FTIR spectra of irradiated and photopolymerized groups.

Table 3
Means (Standard deviation) of flexural modulus (GPa) and flexural strength (MPa) of irradiated and photopolymerized groups. In the same column, means followed by the same uppercase letter are statistically similar.

Groups (dose)	Flexural modulus (GPa)	Flexural strength (MPa)
6 (control)	2.1 (0.5) B	83.4 (8.7) D
7 (0.25kGy)	2.6 (0.7) AB	84.3 (9.3) CD
8 (0.5 kGy)	2.7 (0.8) AB	99.0 (14.2) BC
9 (1 kGy)	2.7 (0.6) AB	106.0 (11.9) B
10 (2 kGy)	3.0 (0.7) A	123.0 (13.3) A

decomposition products (acrolein and allyl alcohol) was oxidation due to the presence of a small amount of oxygen in the system (Grace and Guy, 1985).

As for the inorganic filler, the peak at 1025 cm^{-1} corresponds to the antisymmetric stretching vibration of the Si–O–Si bond. The peak at 934 cm^{-1} corresponds to the stretching of the Si–OH groups and that at 797 cm^{-1} corresponds to symmetric stretching of the Si–O–Si bond (Ray and Bhowmick, 2004).

4.3. Flexural modulus and flexural strength

The results of the flexural modulus and flexural strength of previously-photopolymerized and irradiated groups are presented in Table 3.

The regression analyzes are presented in Figs. 3 and 4.

Regarding the flexural modulus, the statistical analysis revealed significance of the analyzed factor ($p=0.044$). The higher irradiation dose (group 5–2 kGy) resulted in higher flexural modulus when compared to control group (group 1). No statistical differences were observed among the other groups, or the control group. Even though there were no statistical differences among groups, the regression analysis revealed a positive relationship between flexural modulus and absorbed dose ($R^2=0.781$).

Regarding flexural strength, the statistical analysis revealed significance of the analyzed factor ($p=0.000$). The control group (group 1) presented the lowest value of flexural strength, and it was statistically similar to group 2 (0.25 kGy). As irradiation increased the values of flexural strength also increased and that was confirmed by the regression analysis that presented a strong positive correlation between flexural strength and absorbed dose ($R^2=0.952$).

5. Discussion

The presence of two thermal degradation steps in the thermogravimetric analysis (TGA) of the BisGMA/TEGDMA polymeric matrix has been previously observed. According to Karabella and Sideridou (2011), this can be attributed to the heterogeneous polymer architecture, mainly owing to the formation of primary rings during photopolimerization. They believe that the first

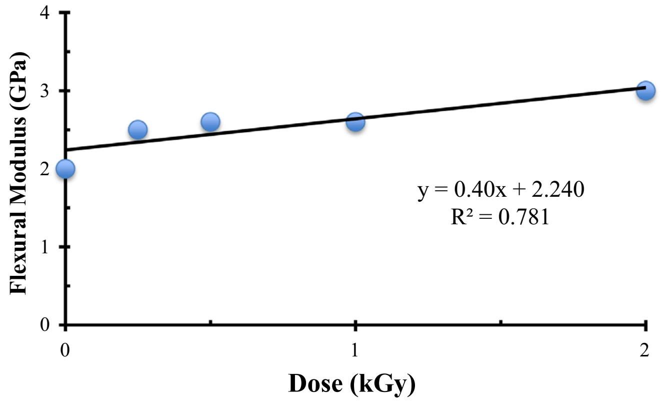

Fig. 3. Scatter plot and regression analysis between flexural modulus and irradiation of photopolymerized and irradiated groups.

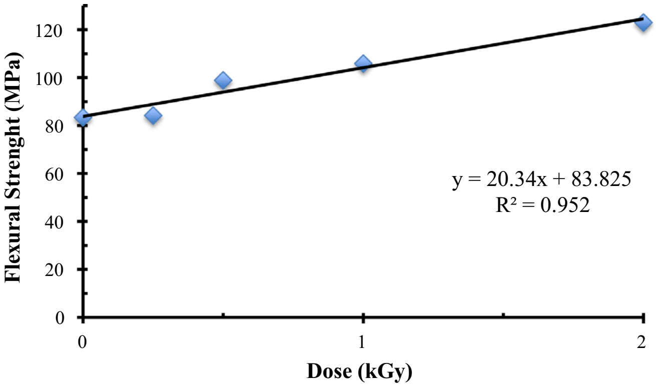

Fig. 4. Scatter plot and regression analysis between flexural strenght and irradiation of photopolymerized and irradiated groups.

degradation step (occurring at lower temperature) corresponds to the breaking of the primary rings and the secondary step to the breaking of the main chain. The authors also pointed out that the third step is related to the superficial silanol condensation in the silica.

When comparing the groups of non-previously-photopolymerized irradiated composites (with the exception of the control group), it can be observed that the use of ionizing radiation had some influence on the thermal degradation profile, regardless of the applied dosage, and irradiation makes them gradually more temperature-resistant due to radiation-induced polymerization and cross-linking. However, for specimens that have been photopolymerized, subsequent irradiation has no influence on the observed thermal properties.

In group 1 (control group), the first step of mass loss took place at a temperature around 60 °C inferior to that of the irradiated groups. The second step of mass loss took place at a temperature approximately 50 °C lower for the control group in comparison to that for the irradiated specimens, which were all similar to each other. Such greater thermal stability of the irradiated specimens might be a consequence of cross-linking of the polymeric chain caused by ionizing radiation. According to the Behr et al. (2005) functional groups of silane coupling agents (from inorganic filler) and functional monomers (from polymeric matrix) might also be influenced by electron irradiation by activating even more possible linkage spots for the matrix of the composite.

In the FTIR spectrograms the consumption of residual monomers in the polymer matrix was evidenced by the decrease of the peaks referents to the monomer groups. A reduction of intensity in the peaks at 1634 and 1609 cm^{-1} (related to the C=C bonds in the polymeric matrix) was observed for the photopolymerized irradiated specimens in comparison to the control group. This could be related to the breaking of double bonds and grafting of residual monomers (post-cure polymerization). The main differences observed refer to peaks below 1720 cm^{-1}, which are related to both organic and inorganic parts of the composite. Such a result is in accordance with a previous work in which the author reports several chemical groups from dental composites with absorption below 1800 cm^{-1} show sensitivity to gamma radiation in a random way (Cruz et al., 2009).

Behr et al. (2005), observed that post-curing treatment of commercial dental composites by ionizing radiation (with doses from 25 to 200 kGy) can improve their properties (i.e.: fracture toughness, Vickers hardness and resistance against wear). Color changes were however observed. In another study, the author reports that the use of radiation caused an increase in hardness of the composites, as well as an associated reduction in water absorption and solubility (Haque et al., 2001). Vaishnavi et al. (2010) observed that mechanical properties of dental composites irradiated with electron beam (1kGy) were comparable to other post-curing systems, and higher doses would further optimize the material properties.

It was suggested that such behavior is possible as a consequence of exposure to radiation, due to its capacity in reorganizing residual chemical groups of low molecular weight inside the polymer matrix so that such effect could be beneficial (Von Fraunhofer et al., 1989). Other authors attested that the radiation absorption process leads to the formation of excited states and ionization, creating new reactive species Hsiue and Chang (1993). In 2009, Cruz et al., reported that even minimal exposure to ionizing radiation can promote chemical changes in commercial photopolymerized dental composites.

With regards to the silica used as filler in the preparation of the experimental composites, Behr et al. (2009) reported that the flexural strength of experimental composites filled with silanized silica could be improved by irradiation. The coupling agent poly (methylphenylsilane), when submitted to radiation, undergoes intermolecular crosslinking and forms a three-dimensional network (Herden et al., 1998). Ray and Bhowmick (2004) observed that the irradiation of silica improved its hydrophobic nature and reduced the surface polarity, which lowered its trend to form clusters.

The post-curing reaction of the polymeric matrix, together with the lower tendency of the filler to form clusters and the radiation effect in the coupling agents (as a result of exposure to ionizing radiation) can explain the different values presented in this study, with respect to the flexural strength and modulus strength of the irradiated specimens (in comparison to the control group). It was also found that the dose of radiation employed influenced the results directly, showing a strong positive correlation between flexural strength and irradiation ($R^2 = 0.952$) and a positive correlation between modulus strength and irradiation ($R^2 = 0.781$).

6. Conclusion

Cross-linking of the polymeric matrix caused by ionizing radiation, influenced the thermal stability of the irradiated specimens (non-previously-photopolymerized), if compared to the control group. FTIR analysis showed that the ionizing radiation (at doses from 0.25 to 2 kGy) induced a post-cure reaction and modification in the previously-photopolymerized irradiated specimens. The radiation dose influenced directly the results, which showed a strong positive correlation between flexural strength and irradiation dose, and a positive correlation between modulus strength and irradiation dose. Thus the use of irradiation showed effect in the post curing process of dental composites of BisGMA/TEGDMA filled with silanized silica.

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