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Influence of Light-polymerization Modes on the Degree of Conversion and Mechanical Properties of Resin Composites: A Comparative Analysis Between a Hybrid and a Nanofilled Composite

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Clinical Relevance

The nanofilled composite presented a lower degree of conversion, flexural modulus and flexural strength than the hybrid composite. On the other hand, the two materials had a similar hardness. The light polymerization mode influenced only the degree of conversion and the hardness of composites.

SUMMARY

This study analyzed the influence of the light polymerization mode on the degree of conversion (DC) and mechanical properties of two resin composites: a hybrid (Filtek P60) and a nanofilled composite (Filtek Supreme). The composites were light activated by three light polymerization modes (Standard-S: 650 mW/cm^2 for 30 seconds; High intensity-H: 1000 mW/cm^2 for 20 seconds and Gradual-G: 100 up to 1000 mW/cm^2 for 10 seconds + 1000 mW/cm^2 for 10 seconds). The DC(%) was measured by FT-Raman spectroscopy. Flexural strength and flexural modulus were obtained from bar-shaped specimens (1 x 2 x 10 mm) submitted to the three-point bending test. Microhardness was evaluated by Knoop indentation (KHN). Data were analyzed by ANOVA and Student-Newman-Keuls multiple range test and linear regression analysis. The results showed the following DC: H > S > G (p<0.0001) and hybrid > nanofilled (p<0.005). Correlation was found between DC and the radiant exposure (R^2 = 0.92).

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With respect to mechanical properties, only KHN was significantly influenced by the light polymerization mode, as follow: H>S=G ($p<0.0001$). The hybrid composite presented higher flexural strength and flexural modulus than the nanofilled composite ($p<0.0001$). No significant difference was found in KHN between the two composites ($p=0.1605$). The results suggest that nanofilled composites may present a lower degree of conversion and reduced mechanical properties compared to hybrid composites.

INTRODUCTION

Since resin composites were developed,[1] many efforts have been made to improve the clinical behavior of this aesthetic restorative material. Research conducted with this purpose has focused on polymeric matrix development[2-3] and inorganic filler particle features; that is, type, treatment and content.[4-5] Although both organic and inorganic phases might influence the material behavior, the filler particle features and rate are the most important factors related to improvement of the mechanical properties of resin composites. In this field, Kim and others[6] showed that the flexural strength, flexural modulus, microhardness and fracture toughness of resin composites were significantly influenced by the filler particle morphology and rate. Rodrigues Júnior and others[7] also found a correlation between the filler weight content, flexural strength and modulus of the elasticity of resin composites.

Recently, resin composites have been classified according to their filler particle size as hybrid (0.5-3 µm), microhybrid (0.4-1 µm) and microfilled (0.04-0.4 µm).[8] More recently, however, with the introduction of nanotechnology in dentistry,[9] a new class of resin composite, the nanocomposite, is available to clinicians. Filtek Supreme (3M ESPE, St Paul, MN, USA) is a typical nanofilled composite with a filler particle system that combines silica nanofillers with a primary particle size of 20 or 75 nm and zirconia-silica nanoclusters 0.6–1.4 µm in diameter. Its filler content is around 59.5 vol%, which is close to that of several hybrid and microhybrid composites.[9] According to the manufacturer, this nanocomposite presents physical properties similar to those of hybrid composites and is indicated for high-stress bearing posterior restorations. Until now, however, few studies have investigated the mechanical properties of this nanocomposite.[7,10-11]

The degree of conversion is a co-determinant of the mechanical properties of restorative resins.[12] A previously published study showed a significant correlation between the degree of conversion and hardness, modulus of elasticity and flexural strength of dental restorative resins.[13] Thus, particularly in stress bearing areas, adequate polymerization could be considered a crucial factor in obtaining good clinical performance of resin composite restorations. Factors, such as the filler particle size,[5] polymeric matrix[14] and radiant exposure generated by the light source,[15] can influence the degree of conversion of resin composites and thereby influence their mechanical properties.

Some light polymerization modes can now be used to start the resin composite polymerization reaction.[16] Modes that use high initial irradiance provide higher DC and better physical properties. However, when resin composites are submitted to initial high irradiance, a higher shrinkage stress may be induced during the polymerization reaction, increasing marginal gap formation at the cavity wall-resin composite interfaces.[17] In clinical practice, gradual polymerization modes have been introduced in an attempt to minimize the harmful effects of shrinkage stress developed by resin composites. These polymerization modes, in which the resin composites are first submitted to low irradiance followed by an increase in irradiance, allow shrinkage stress relief by polymer chain relaxation.[18] Published studies have shown that these polymerization modes did not reduce the degree of conversion of resin composites or resin polymers.[16,19]

Recently, Mehl and others[20] have shown that a gradual polymerization mode did not reduce the mechanical properties of resin composites. However, these authors did not correlate the mechanical properties evaluated with the degree of conversion presented by the materials.

This study compared the mechanical properties (Knoop microhardness, flexural strength and flexural modulus) and the degree of conversion of a traditional hybrid and a nanofilled composite polymerized with three light polymerization modes. The research hypothesis tested was that no significant differences would be found in the DC and the mechanical properties of the resin composites evaluated.

METHODS AND MATERIALS

Two resin composites, chosen in accordance with their type of filler particles, were tested: a hybrid (Filtek P60—P60) and a nanofilled composite (Filtek Supreme—Su) (3M ESPE). Both materials have the same polymeric matrix (Bis-GMA, Bis-EMA, TEGDMA and UDMA), differing only with respect to the filler particle type. Their compositions are described in Table 1.

All the specimens in this study were light activated with a quartz-tungsten-halogen unit (Optilux 501, Kerr, Danbury, CT, USA). Three light polymerization modes were used: Standard (S): 650 mW/cm^2 for 30 seconds (19.5 J/cm^2); High intensity (H): 1000 mW/cm^2 for 20 seconds (20 J/cm^2) and Gradual (G): 100 up to 1000 mW/cm^2 for 10 seconds + 1000 mW/cm^2 for 10 seconds (≈17 J/cm^2). The radiant exposure was calculated as the product of the curing unit irradiance by using a radiometer (Model 100, Demetron Inc, Danbury, CT,

Table 1: *Composition of the Resin Composites Used in This Study*

Resin Composite	Composition
Filtek P 60 (A3 shade)	Filler: 61 vol% silica/zirconia filler with mean particle size of 0.6 µm Polymeric matrix: Bis-GMA, Bis-EMA, UDMA TEGDMA
Filtek Supreme (A3B shade)	Filler: 59.5 vol% combination of aggregated zirconia/silica cluster filler with primary particles size of 5-20 nm, and nonagglomerated 20 nm silica filler Polymeric matrix: Bis-GMA, Bis-EMA, UDMA TEGDMA

USA) and the time of irradiation. For the Gradual mode, the radiant exposure was obtained by the sum of mean irradiance over the first 10 seconds multiplied by 10 seconds at 10 J/cm^2, corresponding to the radiant exposure in the last 10 seconds of light exposure.

Degree of Conversion—DC (%)

Raman spectra of the uncured and cured specimens of each resin composite were recorded by a Raman spectrometer (Nicolet FT-Raman 950, Thermo Nicolet Inc, Madison, WI, USA) operating with 120 scans at a resolution of 2 cm^{-1}. An increment of 1 mm of each resin composite was compressed between two polyethylene strips and two glass slides to produce a thin film (approximately 40 µm). Five films of each resin composite were then light activated in accordance with the three light-polymerization modes tested, with the light tip in contact with the glass slide. Raman spectra were recorded after 24-hour dry storage at 37°C. The DC (%) was calculated from the ratio between the peaks of the aliphatic C=C bond (1638 cm^{-1}) to the aromatic C=C bond (1608 cm^{-1}) obtained from the cured and uncured specimens using the following equation:

$$\text{DC}\ (\%) = 100 \times [1 - (R_{\text{cured}}/R_{\text{uncured}})] \qquad (1)$$

where R = peak at 1638 cm^{-1}/peak at 1608 cm^{-1}

Knoop Hardness Number (KHN)

The resin composites were inserted in bulk in a Teflon split mold measuring 3 mm in diameter and 2 mm high. The mold was covered with a polyester strip and a glass slide (0.7 mm thick) and the resin composite was light activated. Five specimens were prepared in each experimental group. After 24-hour dry storage at 37°C, the light-irradiated surfaces of the specimens were wet ground in a polishing machine (DPU-10, Struers, Copenhagen, Denmark) with 1200 grit SiC paper. Five Knoop indentations (50 g for 15 seconds) were made on the irradiated surfaces of each specimen (Micromet 2003, Buehler, Lake Bluff, IL, USA).

Flexural Strength and Flexural Modulus

The resin composites were inserted into a bar-shaped steel split mold (1 x 2 x 10 mm) positioned over a polyester strip. After filling the mold to excess, the material surface was covered with a polyester strip and glass slide and compressed with a device (500 g) to extrude excess material. The specimens were light activated from the top by two overlapping footprints, using the three light-polymerization modes tested. Ten specimens were prepared in each experimental group. The specimen dimensions were measured using a caliper (MPI/E-101, Mitutoyo, Tokyo, Japan). After 24-hour dry storage at 37°C, the specimens were submitted to three-point bending, with a 6 mm span between the supports, in a universal testing machine with a 50 N load cell (DL 10000, Emic, Curitiba, PR, Brazil) at a crosshead speed of 0.5 mm/minute. The Flexural modulus (FM) was calculated from the linear portion of the load/deflection curve and the flexural strength (FS) from the load at break by the following standard equations:

$$FM = \frac{l^3F}{4wh^3d}\ (\text{GPa}) \qquad (2)$$

$$FS = \frac{3lF}{2wh^2}\ (\text{MPa}) \qquad (3)$$

where l is the support span length (mm), F is the load (N), F1 is the failure load (N), w is the specimen width (mm), h is the specimen height and d is the deflection (mm) at load F.

Statistical Analysis

The statistical analysis was performed using Statgraphics 5.1 Software (Manugistics, Rockville, MD, USA). For all the variables studied, the mean and standard deviation were calculated from the values obtained for each specimen. The data were submitted to the Shapiro-Wilk test to check the distribution normality and the Levene test for variance homogeneity. After the normality of distribution was proved, the DC and mechanical properties data were analyzed by two-way ANOVA, one-way ANOVA and Student-Newman-Keuls multiple range test. Data were also submitted to two-dimensional regression analysis, with each mechanical property and the radiant exposure as the dependent variables and the DC as the independent variable. All statistical analysis was performed at a significant level of α=0.05.

RESULTS

The results of the DC, KHN, FS and FM are presented in Table 2. With respect to DC (%), two-way ANOVA detected a significant influence for the resin composite (p<0.005) and light polymerization mode (p<0.0001). The Student-Newman-Keuls test showed that the highest DC was obtained by the hybrid composite polymerized with the H mode. The two composites with the S mode and the nanofilled composite with the H mode

Table 2: *Mean Values (Standard Deviation) for DC, KHN, FS and FM of Groups Studied*

Groups	DC (%)	KHN	FS (MPa)	FM (GPa)
P60S	52.9 (2.0)^{a}	103.7 (11.4)^{a}	225.4 (19.3)^{a}	11.0 (0.8)^{a}
P60H	69.0 (2.0)^{b}	141.4 (7.5)^{b}	209.5 (17.7)^{a,b}	10.5 (0.8)^{a}
P60G	47.0 (1.4)^{c}	94.9 (5.9)^{a}	221.3 (22.9)^{a}	10.5 (1.1)^{a}
SuS	55.3 (2.1)^{a}	99.2 (4.0)^{a}	173.7 (30.4)^{c}	8.8 (0.7)^{b}
SuH	53.8 (1.9)^{a}	135.0 (4.3)^{b}	185.8 (28.7)^{b,c}	9.1 (0.6)^{b}
SuG	49.0 (1.7)^{c}	94.1 (8.3)^{a}	190.2 (19.2)^{b,c}	8.8 (0.6)^{b}

Within each column, values with same superscript letters are not statistically different (α=0.05)

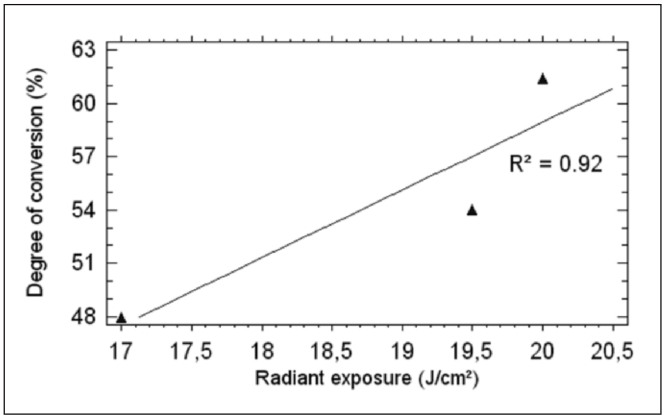

Figure 1. *Correlation between DC and the radiant exposure applied by the three light-polymerization modes.*

were not statistically different. A significantly lower DC was presented by both resin composites polymerized with the G mode. Figure 1 shows a correlation (R^2=0.92) between DC and the radiant exposures applied by the three light polymerization modes.

As regards KHN, two-way ANOVA showed statistical differences only for the light polymerization mode (p<0.0001). No difference in KHN was found for the resin composite (p=0.1605) and the double interaction between the light polymerization mode and the resin composite (p=0.1086). The Student-Newman-Keuls test showed the following results for individual groups: P60H=SuH > P60S = SuS = P60G = SuG.

The results of the two-way ANOVA for flexural strength detected statistical significance only for the resin composite factor (p<0.0001). No significant differences in FS were detected between the light polymerization modes (p=0.5300) and the interaction between the two factors (p=0.1583). The multiple comparison of individual means showed that the FS obtained for hybrid composite with the three light polymerization modes were higher than that presented by the nanofilled composite, while the FS values for the nanofilled composite with the H and G modes were statistically similar to those presented by the hybrid composite when polymerized with the H mode.

As regards the flexural modulus, the two-way ANOVA showed statistical differences only for the resin composite independent factor (p<0.0001). The light polymerization mode (p=0.7312) and the double interaction (p=0.2110) presented no statistical significance. The Student-Newman-Keuls test showed that the FM obtained for the hybrid composite with the three light polymerization modes were similar to and higher than those presented by the nanofilled composite, which showed no statistical difference between them.

No correlation was found between the DC and the mechanical properties (p>0.05).

DISCUSSION

Over the last few years, several studies related to the degree of conversion and mechanical properties of resin composites have been developed.[12-13,19-24] However, few have focused on the correlation between these resin composite characteristics.[12,22] Currently, resin composites are widely used in restorative dentistry, and specifically, in posterior restorations; the material is constantly under masticatory stresses. From this viewpoint, resin composites with good mechanical properties must be selected for this purpose.

One of the most important parameters involved in resin composite light activation is the radiant exposure, calculated as the product of the irradiance, and the time of irradiation provided by the light unit. In the current study, since the radiant exposures generated by the three light polymerization modes were close to the same value, a similar DC was expected for the three experimental conditions. However, a significant effect of the light polymerization modes on the DC was found, with the H mode presenting higher DC than the S mode, whose DC was higher than that of the G mode. The highest DC was presented by the hybrid composite polymerized with the H mode (Table 2). A reasonable explanation for this result can be based on the higher initial irradiance applied by the H mode (1000 mW/cm^2).[25] This high irradiance could have developed a greater rise in exothermic heat in the hybrid composite polymeric matrix, providing the polymer chains with greater mobility, consequently extending the DC.[23,25-26] On the other hand, the DC of nanofilled composite with the H mode was lower than that presented by the hybrid composite and similar to the DC in groups polymerized with the S mode (P60S, SuS). Since both resin composites have the same polymeric matrix, this behavior could be explained by the filler particle size of the nanofilled composite. Arikawa and others[27] showed that, at a wavelength of 470 nm, the light transmittance of Silux Plus, that is, a microfilled composite with similar filler particles to that of Su, was significantly lower than that presented by a hybrid composite. According to these authors, the light that passes through the resin composite is scattered by the small filler particle and the light transmittance is reduced. Based on this, it is possible for the non-agglomerated silica nanoparticles with a mean size of 20 nm to have caused a light-scattering effect in the nanofilled composite. Thus, the light intensity might have been attenuated and the DC decreased.

Findings from other studies disagree with the DC results of the current research, as they reported that a fixed radiant exposure, rather than high irradiance, was the crucial factor in the degree of conversion of resin composites.[15,25] A recent study, however, showed that resin composites light activated with a radiant exposure between 6 and 36 J/cm^2 presented similar degrees of conversion.[23] Moreover, Danesh and others[28] showed that light units that used high irradiance, instead of producing a lower radiant exposure, generated a high degree of conversion in resin composites. These conflicting findings suggest that further investigations into this research field are required

A linear correlation was found between the DC and the radiant exposure applied by the three light polymerization modes (Figure 1). This finding agrees with recent studies.[25,29] A reasonable explanation for this result can be based on the polymerization kinetics theory of light activated resin composites.[30] The rate of free radical production is influenced by irradiance absorbed by the photoinitiator and the quantum yield for initiation. Although the radiant exposures generated by the three light polymerization modes used in the current study were approximately the same, the initial irradiances were different (H > S > G). It is possible that the high irradiance of the H mode could have provided more photons for absorption by camphorquinone, increasing the rate of free radicals available for the polymerization reaction. The same concept could be applied to explain the differences between the S and G modes. It is important to emphasize that this correlation was analyzed, taking into account the average of the DC between the two composites. On the other hand, no correlation was found between the DC and the mechanical properties evaluated. Since previous studies have proven a correlation between the degree of conversion and some physical properties of resin composites,[12-13,22,29] this finding was unexpected. However, Chung[31] found no correlation between the degree of conversion, Knoop Hardness and diametral tensile strength of posterior resin composites and claimed that the filler concentration and resinous matrix played a more important role in determining the properties of resin composites. Based on this finding, it is reasonable to assume that the similarity between the polymeric matrixes and the filler content of the resin composites evaluated contributed to the inexistence of correlation between the DC and the mechanical properties in the current study.

Among the mechanical properties analyzed, only KHN was influenced by the light polymerization mode. The specimens light activated with the H mode (P60H = 141.38 and SuH = 135.02) presented higher KHN than the other groups (P60S = 103.71, SuS = 99.15, P60G = 94.87 and SuG = 94.13). This finding agrees with a previous study[32] that showed an increase in irradiance leads to a superior hardness in resin composites. In addition, according to Rahiotis and others,[16] light-curing units emitting high irradiance during the first 10 to 15 seconds of photopolymerization may cause rapid network formation at the superficial layer of the resin composites. Thus, the hardness may be improved. Obviously, this was the case for specimens that were light activated with the H mode. Some previous studies have indeed shown that the polymer structure is influenced by the initial irradiance during photoactivation.[14,33] High irradiance, as was the case for the H and S modes, would form a more cross-linked polymer network. On the contrary, gradual irradiance of the G mode would create a tail-head polymerization process, producing a more linear polymer that has low hardness.[34]

Although there was a significant difference in DC, the light polymerization modes did not influence the FS and FM (Table 2). This result conflicts with a recent study by Vandewalle and others,[35] who showed that radiant exposure influenced the flexural strength and flexural modulus of Z250 resin composite, which has a polymeric matrix and filler particles similar to P60. In the Vandewalle and others study, however, the radiant exposure ranged from 4000 to 24,000 mJ/cm^2. Based on this, the authors speculated that, in the current study, the low difference among the radiant exposures generated by the three light polymerization modes (S=19.5, G=17 and H=20 J/cm^2) was not sufficiently sensitive to produce significant differences in FS and FM. Moreover, different from the effect on hardness, which is a surface property, the rapid polymerization produced by the high irradiance at the superficial layer may cause changes in the optical properties of resin composites and reduce light transmittance throughout the bulk material.[16] Moreover, Price and others[36] showed that, for several resin composites, there was an exponential decrease in light energy transmitted (mW/cm^2) as the specimen thickness increased. Specifically, for P60, these authors found a decrease of about half, from 0.5 mm to 1 mm, and of one-third, from 0.5 mm to 2 mm. Based on these findings, the authors speculated that, in the current study, the DC may have decreased because of the thickness of the bar-shaped specimens used for the FS and FM evaluation. From this viewpoint, FS and FM, which are properties that involve three-dimensional material behavior, may not have been influenced by different initial irradiances generated by the light polymerization modes used.

The FS and FM of the nanofilled composite were lower than that of the hybrid (Table 2). Previous studies have shown a strong correlation between the volumetric filler content, Young's modulus and flexural strength of resin composites.[4,37] Since the two composites used in the current study have the same polymeric matrix and approximately the same volumetric filler content, it can be assumed that this result would be related to the filler particle features of nanofilled composite. Based on earlier discussion, the light scattering effect produced by the non-agglomerated silica nanoparticles with a mean size of 20 nm may have decreased the DC of Su. Consequently, its FS and FM were lower.

Although the H mode presented a statistically better DC, the data from Table 2 shows that this result was strongly influenced by the P60H group. On the other hand, the DC of SuH was similar to the DC of the groups polymerized with the S mode (P60S, SuS). As previously discussed, the light polymerization mode did not influence the FS and FM. Taking into account that light polymerization modes that use high initial irradiance may develop a high shrinkage stress at the cavity wall-resin composite interfaces,[17,20] it is reasonable to advocate that the S mode could be more suitable for light polymerizing resin composites.

This study focused on the comparative analysis between a traditional hybrid and a nanofilled composite, which can be considered to be representative of a new generation of restorative materials. According to its manufacturer, this composite is indicated for both anterior and posterior restorations. The results obtained, however, showed that the DC, FS and FM of the nanofilled composite were statistically lower than that of the hybrid. Based on these findings, it is reasonable to infer that, although the filler incorporating technology of this nanofilled composite differs from that of traditional microfill resin composites (for example, pre-polymerized filler is not used), the light-scattering produced by the nanofiller may interfere negatively with its properties.

CONCLUSIONS

The results obtained in the current study led to rejection of the research hypothesis. The nanofilled composite presented lower DC, FS and FM than the hybrid composite. Furthermore, the DC was influenced by the light polymerization mode. Among the mechanical properties evaluated, only hardness was influenced by the light polymerization mode. A correlation was found between DC and the radiant exposures applied by the three light polymerization modes. In addition, further *in vitro* tests and clinical studies are necessary to add knowledge regarding the behavior of the nanofilled composite evaluated.

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