Study of the Influence of Pigments in the Polymerization and Mechanical Performance of Commercial Dental Composites

Estudo Sobre a Influência dos Pigmentos na Polimerização e no Desempenho Mecânico dos Compósitos Dentais Comerciais

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ABSTRACT

Objective: The aim of this study was to analyze the influence of the amount of shade pigment and opacifying components in the polymerization and mechanical properties in two brands of Brazilian dental resin composites. Material and Methods: Master Fill microhybrid resin for enamel (colorless and shades A2, A4), and Opallis for enamel and dentine, both shades A2. The composite resins were photopolymerized by a LED device at 19.7 J. The mechanical properties were obtained through flexural resistance (FR), compression strength (CS) and Vickers microhardness (VM); the degree of conversion (DC) was obtained by FTIR and translucence was obtained by sphere espectrometer. Conclusion: The analysis indicated that resin with more color pigments (MA4) or more opacity components (ODA2) had low polymerization and poor mechanical properties than clearer (M) or more translucent (OEA2) resins, indicating that better performance can be obtained by increasing the radiation dose.

Keywords: Composite resin, shade, translucence, mechanical analysis.

INTRODUCTION

The varieties of composites resin currently available provide a very good aesthetic effect, therefore the restoration is practically imperceptible, besides correcting imperfections in the tooth. However, it is no use if the professional has excellent aesthetic restoring material if does not know to use it adequately. The manufacturer makes many tests before placing the product in the market, and therefore the recommendations of the same must be followed. Moreover, many institutions of research also make analyses and the results of the same ones are available. The professional must be very well informed about new products. Some resins, due to the shade and the opacity possess differentiated times of polymerization, and that, if they are not respected they can make the restoration to be compromised before the stated period.

There are reports indicating that the shade does not effect the polymerization[1,2] but there are others indicating the contrary[3-5], and there are still others indicating that it may be more dependent upon translucency than upon shade[6,7].

The translucency can be defined as the amount of light transmitted through an object and it constitutes one of the main factors to be considered in the appearance of the restorations. Due to the translucency it is possible to obtain a restoration with characteristics of naturalness, deceiving the observer[8].

For dental materials, sufficient radiopacity is required to allow the composite to be distinguished from the natural tooth on an X-ray photograph. The radiopacity of a compound increases with the use of heavy elements. Therefore, heavy elements compounds are applied. Conventional glass fillers contain heavy elements oxides such as BaO. Other ceramic fillers can also be used. The heavy elements oxides also increase the refractive index of the filler, providing a higher transparency. This increases the polymerization depth of the filling composite when it is irradiated with visible light by the dentist[9].

The pigments are important elements for dental composites, which are necessary to match the colour of the natural tooth. They have to be stable in the oral environment and the shade is not allowed to change over time. Oxidic pigments, such as ferric oxide (Fe_2O_3, red) or ferric hydroxide (FeOOH, yellow), are frequently used[9].

Our study aims to add information on the subject by analyzing two Brazilian resin brands: Opallis and Master Fill. The manufacturer of Opallis resin recommends the radiation time in relation to dental resin shade and their different degrees of translucence10. But several manufacturers recommend the same radiation time for all resins, independent of shade or degree of translucency.

Adequate polymerization of composite resin restorative materials is fundamental for optimal physical and chemical properties, as well as, for best clinical performance[11].

In order to test the hypothesis of the influence of the amounts of both pigment and opacifying components in the mechanical properties of the materials, analyses of mechanical strength and on the degree of conversion were performed.

Clinically the physical properties of a dental material are important. Hence, indirect tests such as Fourier Transform Infrared (FTIR) spectroscopy to determine the degree of cure of the resin may be considered as second best. The DC of the monomers in polymers of the resin is important in dentistry because the presence of unpolymerized material decreases the mechanical properties with consequent wear of the implant. In addition, the unreacted monomer may generate some pathological states[12]. The spectroscopy of FTIR is not useful to compare results of a resin with another one, but to get and to follow the conversion of monomer for polymer of one same resin, in the long run.

Mechanical properties including compression strength, flexural strength are important factors to be studied in restorative materials that are used where high biting forces and stresses can exacerbate inherent materials defects, resulting in inadequate fracture resistance of the materials[13]. The microhardness test has been the most popular method for investigating factors that may influence the depth of cure[7].

MATERIAL AND METHOD

The characteristics of the two selected brands of microhybrid resin composites are presented in Table 1. Master Fill resins (Biodinâmica, Ibiporã, PR, Brazil) were used to analyze the effects of the amount of pigments responsible for the shade of the resin, and the Opallis resins (FGM, Joinville, SC, Brazil) were used to study the effect of the amount of opacifying components. Two of the Master Fill samples contain shade pigments grades A2 and A4 (A4 being darker than A2) in their formulations; one has no pigment and is colorless (M). Opallis samples EA2, DA2, where E stands for enamel and D for dentine restorations. The chemical composition of composite resins for enamel or for dentine differ in the amounts of opacificant components, the latter having the largest. All experiments were performed at 300 K. The resins were introduced in a specific mold required by the equipment in use were light-cured at 492 mW/cm2 by a light-emitting diode (LED) device (ULTRA BLUE - Dabi Atlante, Ribeirão Preto, SP, Brazil), and were calibrated with a radiometer (ECEL, RD-7, Ribeirão Preto, SP, Brazil).

Microhardness was measured in the digital Vickers hardness tester (model HVS-5, Laizhou Huayin Testing Apparatus Co., Shandong, China). Each batch had five specimens measuring 12.50 mm (diameter) x 1.85 mm (thickness). The top and bottom surfaces of each specimen were previously marked and photoactivated for 40 s (19.7 J), then they were wrapped in aluminum foil and stored for 24 h in a dark environment. Four impressions of each top and bottom surfaces were taken at 300 gf, for 15 s each. The polymerization percentage was obtained from the ratio between microhardness values for the top surface and for the bottom surface.

Flexural resistance and compression strength experiments were performed using the universal machine (model DL2000-EMIC, São José dos Pinhais, SP, Brazil). Each batch with five specimens was prepared for flexural resistance (2x2x25 mm) and compression strength (4x10 mm) diameter and height, respectively. The samples used for the compression strength measurements were irradiated for 40 s (19.7 J) on both surfaces. Those used to measure flexural resistance were irradiated in the center and at both ends (three points) on both surfaces. After radiation, the samples were immersed in distilled water at 37 ± 1 ºC and stored for 24 h before the tests.

All the obtained data were submitted to ANOVA statistical analysis and the Tukey post hoc tests at the preset significance level of 0.05.

The degree of conversion (DC) from monomer to polymer[14] was determined using an (Shimadzu-8300, Kyoto, Japan) FTIR spectrophotometer. For each group, five resin samples were photopolymerized for 40 s and stored for 24 h protected from the light and heat. After it, each sample was macered and mixed one part of resin with ten parts of KBr power salt. This mixture was placed into a pelleting device and then pressed in a hydraulic pressing device working at 4 tons in loading to obtain an adequate pellet to work. The FTIR spectra were obtained[15] in transmission mode with 32 scans and resolution of 4 cm^{-1}.

Table 1. Characteristics of the samples.

Resin composite	Manufacturer	Organic matrix	Filler composition	Indication/Shade (Batch n.)
Master Fill	Biodinâmica, Ibiporã, PR, Brazil	BIS-GMA, UDMA and TEGDMA monomers, pigments, camphorquinone, and viscosity controllers	Boro-silicate of Barium-aluminum glass, silica of medium size 0.6μ, presenting a weight of 79% inorganic load	Enamel A2 (441/08) Enamel A4 (1025/07) Enamel Colorless (PI 100/08)
Opallis	FGM, Joinville, SC, Brazil	BIS-GMA, BIS-EMA and TEGDMA monomers, pigments, camphorquinone, accelerator and stabilizers	Barium – Aluminum silicate silanized micro-particles and silicon dioxide in nanoparticles. Particles of medium size 0.5 μ, totaling 78.5% of weight of load for the enamel colors, 78.5 to 79.5% in the dentine colors	Enamel A2 (111207) Dentine A2 (221107)

For the analysis of the translucency, three specimens in record form for each group had been confectioned in steel molds inox with the dimensions of 15,5 mm of diameter and 1,0 mm of thickness. After separating in four quadrants and curing for 40 seconds, by source LED each quadrant, totalizing 160 seconds of exposition to the light, they had been inserted in spectrophotometer of sphere, X RITE, model SP 62 for the determination of the color parameters and opacity, that after being obtained they are inserted in the equation (1) for the determination of the translucency. The analyses work carried through in the Biodynamics company (Ibiporã, PR).

$$T=100\times[1-((Lp+16)/Lb+16)] \qquad (1)$$

Where "p" relates to the measurement of the composite on the deep black color, and "b" relates to the measurement of the composite on the deep white. Letter "L" represents the luminosity, being numbered from 0 to 100, where L=100 is white, L=50 is gray and L=0 is black.

RESULTS

The polymerization degree calculated from Vickers microhardness measurements is shown in Figure 1.

The Figure 2 shows the evolution of the spectrum of the FTIR for the uncured and cured resins. The main vibrational modes have been previously identified[15] and are assigned to the Methacrylate C=C stretching mode at 1638 cm^{-1}, the C=C stretching of the aromatic group at 1609 cm^{-1}. An unpolymerized composite

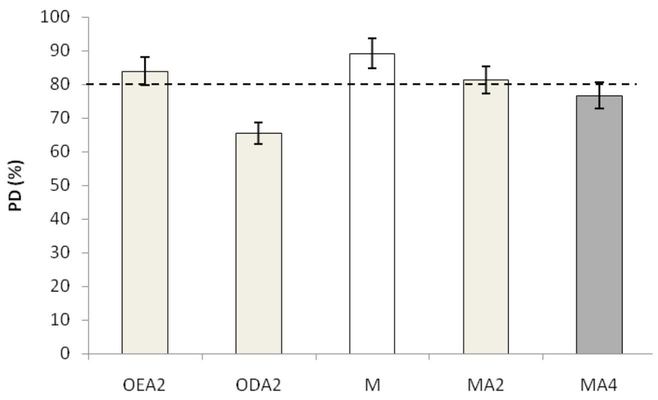

Figure 1. Polymerization degree of resins. Dotted line indicates the desirable minimum value.

Table 2: Experimental results.

Resins	Shade	DC (%)	FR (MPa)	CS (MPa)	VM (gfmm-^{2})
Master Fill (M)	M	55.0[a]	112.90[a] (13.50)	286.05[a] (15.90)	33.96[a] (1.12)
	MA2	55.5[a]	99.60[b] (10.93)	237.15[b] (6.15)	31.50[a] (0.70)
	MA4	47.0[b]	96,30[b] (1.84)	159,40[c] (3.00)	31.00[a] (0.58)
Opallis (O)	OEA2	44.6[c]	100.25[a] (9.26)	218.55[a] (14.00)	38.56[a] (1.57)
	ODA2	45.6[c]	91.94[ab] (4.50)	187.20[b] (2.70)	33.21[b] (1.90)

DC = Degree of Conversion; FS = Flexural Strength; CS = compression strength; MV = Vickers Microhardness; (SD) = standard deviation; M = colorless resin

*Letters indicate statistically similar groups for each resin, (p=0.05)

Table 3: Translucency

Resin	Shade	Translucence (%)
Master Fill	M	21,94[a]
	MA2	12,83[b]
	MA4	6,89[c]
Opallis	OEA2	12,81[b]
	ODA2	9,19[d]

paste sample was obtained and used as reference for the calculation of DC. Considering the intensity of the transmittance peak of the aromatic bond C=C (1608 cm^{-1}) as internal pattern and the peak related to C=C aliphatic (1638 cm^{-1}) were used to evaluate the changes during the polymerization. The degree of conversion is then calculated by using the following equation:

$$DC(\%) = 100*(1 - R\,cured / R\,uncured) \quad (2)$$

Where R = band height at 1638 cm^{-1} / band height at 1609cm^{-1}.

The FTIR spectrum (Fig. 2) shows changes in the relative intensities of the peak at 1638 cm^{-1} as a function of polymerization. Note in the Figure that the intensity of the peak at 1609 cm^{-1} corresponding to the aromatic group, remains constant and, therefore, not depending of the polymerization[16].

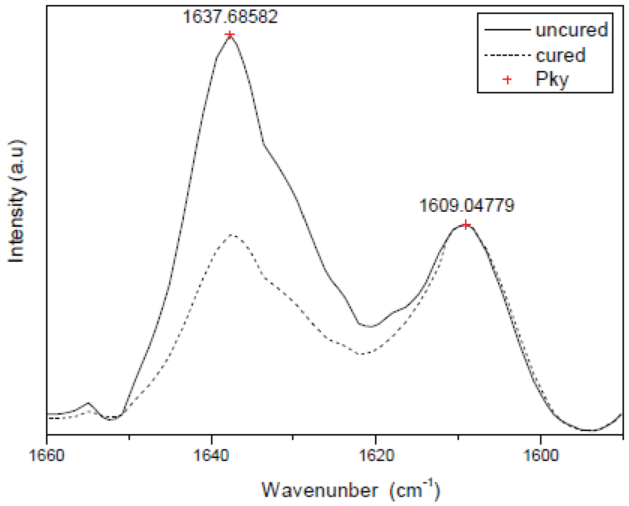

Figure 2. Characteristic FTIR spectrum of MA2 resin before and after exposure to visible light.

Results for the Degree of Conversion (DC) and analysis of mechanical performance of flexural resistance (FR), compression strength (CS), and Vickers microhardness (VM) and their standard deviation (SD) are given in Table 2.

Amoung the resins of masterfill, the colorless one is more translucent, it is followed by A2 and A4, and, between the Opallis resins, A2 indicated for the enamel is more translucent than that the indicated for the dentine, as show in Table 3.

DISCUSSION

During the photopolymerization process, the light that passes through the composed resin is reflected, absorbed and spread[17]. Thus, light intensity is attenuated and the effect is reduced with the increase of depth[18]. In this way, the depth on polymerization depends on the irradiance, time of exposition and others several factors such as composition of the material[19], color of composite[20] and translucence[21]. The transmission of light through darker materials is reduced because of the opacity of the material[22]. Thus, composites with darker colors tend to absorb greater amount of light than clearer colors[23] and promotes greater interference in the transmission of the light when compared with composites of clearer colors; causing reduction of the intensity of the light that crosses the composite during the photopolymerization, thus, the intensity of the light that arrives at the deepest layers of the restoration, affect the mechanical properties of the composite[24].

The Master Fill resins were analyzed with the intention of verifying the influence of the amount of pigments, responsible for the shade of the sample, in the properties of the material, for that resins for enamel were used, whose main composition difference in the products is the addition of shade pigment which is absent in the colorless M, a little in MA2 and a little more in MA4.

In Table 2 it is verified that DC, FR and CS results indicated a performance smaller for the darkest Master Fill resin MA4; the analysis VM showed that there were no statistically significant differences amoung resins, however in Figure 1, the polymerization degree (PD) was found to decrease significantly with darker shades being in agreement with the work of Lui et al.[25] (2006). This fact is due to the presence of the pigments used to offer dark shades to the restoring materials, because, possibly they absorb the light that passes through the resin and they harm its polymerization, acting as selective filters for certain light wavelengths[5].

The Opallis (OEA2 and ODA2) resins were studied with the intention of verifying the influence of the amount of opacificant component responsible for translucence or opacity of the sample. The difference between enamel and dentine application is the amount of opacificant component, which is larger for the last one.

The FTIR results indicate the amount of monomer converted in polymer (Table 2). It can be noted that the DC is related to the mechanical properties of the material.

The analysis of the flexural resistance (FR) of the ODA2 presented small numeric difference when compared to OEA2 resin; however, in the analysis of the compression strength and Microhardness Vickers, the ODA2 resin presented performance smaller and the polymerization degree (Fig. 1) did not reach the minimum value suggested by Watts et al.[18] (1984).

Studies by Firoozmand et al.6 (2009) also indicated that the opacity of the resin and the curing light strongly influence the degree of microhardness of the composite.

In the resins with more pigments or more opacificant components the light must penetrate deeply to polymerize properly. However, the amount of shade pigments and translucence control substances will block the light proportionally. Futhermore, it cannot reach adequate polymerization depth making the resins more susceptible to generate smaller amount of free radicals[26] because they reduce the amount of available light that the initiators need for the polymerization. Acting inversely in mechanical performance, because are directly related to the amount of radicals[27]. Such results suggest an inversely proportional relationship among the amount of additive of shade or translucence controls substances, and polymerization[26].

CONCLUSIONS

Both the hypotheses on the pigments for the coloration and on the opacity components amounts influence in the polymerization and the mechanical properties of resins were observed.

Based on the data found in the present study, results indicated that darker or more opaque dental resins were influenced by the amount of pigments and opacity components because presented they lower performance compared to clearer or more translucent resins, due to the role of these substances in reducing the light intensity required by the initiator agent of polymerization. We suggest that the radiation time recommended by the manufacturer must be increased for resins with more pigments (darker) and/ or more opacity components and that is respected.

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RESUMO

Objetivo: O objetivo deste estudo era analisar a influência da quantidade de pigmentos de cor e de componentes opacificantes na polimerização e nas propriedades mecânicas em duas marcas Brasileiras de resinas compostas dentárias. Material e Métodos: Resina Master Fill, microhíbrida, para esmalte (incolor e nas cores A2 e A4) e Opallis para esmalte e dentina, ambas na cor A2. As resinas foram fotoativadas por um aparelho diodo emissor de luz (LED) a 19.7 J. As propriedades mecânicas foram obtidas por resistência flexural, força de compressão, microdureza Vickers; o grau de conversão foi obtido por FTIR e a translucidez foi obtida por meio do Espectrômetro de Esfera. Conclusões: As análises indicaram que resinas com mais pigmentos de cor (MA4) ou mais componentes opacificantes (ODA2) mostraram baixa polimerização e pobre qualidade mecânica quando comparadas às mais claras e/ou mais translúcidas, indicando que a dose de irradiação deve ser aumentada para obter melhor performance.

PALAVRAS-CHAVE: Resina composta, cor, translucidez, análises mecânicas.

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