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EVALUATION OF MECHANICAL PROPERTIES OF Z250 COMPOSITE RESIN LIGHT-CURED BY DIFFERENT METHODS

AVALIAÇÃO DE PROPRIEDADES MECÂNICAS DA RESINA COMPOSTA Z250 FOTOATIVADA COM DIFERENTES MÉTODOS

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ABSTRACT

This study evaluated some mechanical parameters of Z250 composite resin using different light-curing methods. Ten specimens were prepared for each mechanical test group with different dimensions according to the test. Light-curing was performed by: a). continuous light (800mW/cm^2-40s); b). exponential light (0-800mW/cm^2-40s); c). intermittent light (2s-600mW/cm^2; 2s without light-80s); d). stepped light (10s-150mW/cm^2; 30s-650mW/cm^2); e). PAC (1320mW/cm^2-3s); f). LED (350mW/cm^2-40s). After 24 ± 1 h, the specimens were loaded at a crosshead speed of 0.5 mm/min until fracture. The mechanical properties were calculated and analyzed by ANOVA and Tukey test (5%). The results showed that the highest compressive strength values were found for the continuous, exponential, intermittent and stepped light methods, whereas PAC and LED obtained the lowest values. LED, stepped light, PAC, exponential and continuous light presented the highest values for diametral tensile strength. The intermittent light showed the lowest value, which was significantly lower than the value obtained for LED only. Flexural strength results were not significantly different between all light-curing methods. Finally, the highest modulus of elasticity values were obtained for LED, exponential, continuous and intermittent light, whereas PAC and stepped light showed the lowest values. The mechanical properties were affected by light-curing methods employed.

Uniterms: Composite resins; Light-curing methods, Compressive strength; Diametral tensile strength; Flexural strength; Modulus of elasticity.

RESUMO

Este estudo avaliou algumas propriedades mecânicas da resina composta Z250 usando diferentes métodos de fotoativação. Dez amostras foram preparadas para cada grupo, com diferentes dimensões de acordo com o ensaio. Os métodos de fotoativação foram: a) luz contínua (800mW/cm^2-40s); b) luz exponencial (0-800mW/cm^2-40s); c) luz intermitente (2s-600mW/cm^2; 2s sem luz-80s); d) dupla intensidade (10s-150mW/cm^2; 30s-650mW/cm^2); e) PAC (1320mW/cm^2-3s); f) LED (350mW/cm^2-40s). Após 24 ± 1 h, as amostras foram carregadas até fraturar (v=0,5 mm/min.). As propriedades mecânicas foram calculadas e os dados analisados estatisticamente (ANOVA e teste de Tukey 5%). Os resultados mostraram que, para resistência à compressão, os maiores valores foram encontrados com os métodos luz contínua, intermitente, exponencial e dupla intensidade, enquanto que PAC e LED obtiveram os menores valores. Os métodos LED, PAC, luz contínua, exponencial e dupla intensidade, mostraram os maiores valores para resistência à tração diametral, enquanto luz intermitente mostrou os menores valores, que diferiram apenas do LED. Para a resistência flexural, não houve diferença entre os métodos. Já, para o módulo de elasticidade, os maiores valores foram obtidos com LED, luz exponencial, contínua e intermitente, enquanto que PAC e dupla intensidade mostraram os menores valores. As propriedades mecânicas podem ser afetadas pelo método de fotativação utilizado.

Unitermos: Resinas compostas; Fotoativação; Resistência à compressão; Tração diametral; Flexão; Módulo de elasticidade.

INTRODUCTION

Light-cured composites resins are an important group of restorative materials in Dentistry and can be used to restore shape and function of anterior and posterior teeth. The mechanical properties of a restorative material are a crucial factor in their clinical performance. Strength data usually provided by manufacturers usually includes compressive, tensile and/or flexural strength. These properties are strongly related to the composition (filler content, organic matrix) of the material[3,8,11,12,20] and may be altered depending on the characteristics of the light source used for curing[10,13,19].

Dimethacrylate monomers are widely used as an organic matrix of composite resin and react via addition polymerization to form highly cross-linked structures when light of appropriate wavelength and intensity is supplied[12,25]. The addition of inert fillers to dimethacrylate resins can significantly improve properties such as compressive and flexural strength, hardness, and modulus of elasticity[4, 5,11]. For mechanical properties enhancement, an efficient coupling must be obtained between filler and resin matrix[4,11,15]. However, factors concerning resin composition are determined by manufacturers and cannot be altered by the clinicians. Thus, light radiation is the control instrument available to the operator for property tailoring and may contribute to determination of the final properties of the material and clinical performance.

Several studies have demonstrated that the degree of conversion of double bonds is a co-determinant of the mechanical properties of the resulting polymer. Also, the degree of conversion is dependent on the total light incident on the material in order to activate the photo-initiator and start the chain reaction[2,3,8,10,12,13,20,23].

The main light curing units available in the market today include: halogen lamps light, plasma arc lights (PAC) and the light emitting diode (LED). These light curing devices are different in nature with respect to light intensity, exposure time required to achieve a given amount of polymerization, and spectral emission. Halogen lamps used in conventional curing units generate a white light (full visible wavelength spectra) when the tungsten filament is heated to high temperatures. This light must be filtered to emit only the blue spectrum. Traditionally, these lamps are used to supply a high intensity for the light sources[16,25]. In recent years, alternative light-curing methods utilizing these lamps have been suggested, such as stepped light or step-cure[2,17], exponential light or ramp-cure [23], and intermittent light or pulse-cure[17,24].

PAC sources also emit continuous visible light spectrum, but at higher intensities (> 1000 mW/cm^2). However, PAC light has a more symmetric light distribution of around 470 nm when compared to halogen lamps. Due to the high light intensity emitted by these sources, manufacturers recommend exposure times between 1 and 3 seconds[10,13,18,23]. Finally, LED devices present a narrow and symmetric wavelength spectrum[9], but supply lower light intensities than halogen and PAC sources.

The inherent differences between each light-curing method may provide different initiation and polymerization rates, and may result in polymeric matrixes with dissimilar densities of cross-linking[10,21,23]. Consequently, variations in the final mechanical properties are possible when different light sources are considered for resin composite polymerization.

The aim of this study was to evaluate the effects of different light-curing methods on compressive strength, diametral tensile strength, flexural strength, and modulus of elasticity of a universal resin composite. The hypothesis was that mechanical properties of Z250 composite resin would be affected as a function of the light-curing method.

MATERIALS AND METHODS

The composite resin used in this investigation was Filtek Z250. The composition and batch is listed in Table 1.

The composite was tested for compressive strength, diametral tensile strength, flexural strength, and modulus of elasticity. For the compressive and diametral tensile strength testing, the composite was placed into silicon matrixes (compressive strength: h = 6 mm, d = 3 mm; and diametral tensile strength: h = 3 mm, d = 6 mm), with increments varying between 1.5 – 2.0 mm in thickness, cured after placement of each increment. The matrix was placed on a glass slab with a clear polyester strip for material's placement. The last increment was also covered with a polyester strip and pressed with a glass slab to accommodate the composite into the matrix. For flexural strength and modulus of elasticity testing, the material was placed into an acrylic matrix (l = 25 mm, h = 2 mm, w = 2 mm) which was placed on a metallic base and a polyester strip. These were subsequently covered by another polyester strip and pressed with a glass slide. The light radiation was initially performed at the center, and then

TABLE 1- Composition of Z250 composite resin (according to manufacturer)

Organic Matrix	Filler Type	Filler % (vol)	Filler Size (μm)	Batch
Bisphenol-glycidyl methacrylate (BisGMA), urethanethyl dimethacrylate (UDMA) and Bisphenol-polyethylene glycol dimethacrylate (BisEMA). Camphorquinone (initiator)	Zirconia/silica	60	0.19 – 3.3	2XX 2005-09

moved to the next section until all specimens had been irradiated. Light-curing methods employed in this study were: a) continuous light; b) exponential light; c) intermittent light; d) stepped light; e) PAC, or; f) LED.

For the continuous light curing method, the curing tip was positioned close to the brass matrix/restorative composite. Light-curing was performed for 40 seconds with a light intensity of 800 mW/cm^2, using an Elipar Trilight curing unit (3M-ESPE, Seefeld, D-82229, Germany). For the exponential light technique, the same curing unit was used, however, the light intensity began at zero, increasing gradually to 800 mW/cm^2 (40 mW/cm^2 per second in the first 20 seconds and 800 mW/cm^2 for more 20 seconds), with a total exposure time of 40 seconds. Curing with the intermittent light method was performed using a curing unit developed in the Dental Materials Department, Piracicaba Dental School, UNICAMP, which provided 2 seconds of light with intensity of 600mW/cm^2 and 2 seconds without light. The total exposure time was 80 seconds. The stepped light method was performed using a XL 2500 curing unit (3M-ESPE, Seefeld, D-82229, Germany), which provided an initial 10-second exposure to the activating light with an intensity of approximately 150 mW/cm^2, maintaining a distance of nearly 2.0 cm from the curing tip. The curing tip was then positioned close to the brass matrix/restorative composite, resulting in an increased light intensity of 650 mW/cm^2, which was maintained for additional 30 seconds. For the PAC technique, the Apollo 95 E curing unit was used (DMD, Westlake, Village, CA 91362, USA), which according to manufacturer’s information achieved an intensity of 1320 mW/cm^2. The light exposure time was 3 seconds. Finally, for the LED method, an Elipar Freelight curing unit (3M-ESPE, Seefeld, D-82229, Germany) was used to light-cure the composite, providing an intensity of 350 mW/cm^2 for 40 seconds. The light intensity of all curing units but the Apollo 95E curing unit was measured with a radiometer (Curing Radiometer, model 100, Demetron/Kerr, Danbury, CT 06810, USA).

After light-curing, the specimens were separated from the matrix and stored in a dark container during 24 ± 1 hours. The compressive and diametral tensile specimens were then subjected to compressive force in a Universal Testing Machine (Model 4411, Instron Corp., Canton, MA, USA) at a crosshead speed of 0.5 mm/min until failure. The compressive strength (CS) was calculated by dividing the failure load (F) by the cross-sectional area, i.e.: $CS = F/\pi R^2$, where R is the radius of the cross-section of the specimen. The diametral tensile strength (DTS) was calculated using the equation: $DTS = 2F/\pi Dt$, where F is the failure load, D the diameter, and t the height of the specimen.

For the flexural strength (σ), the bar-shaped specimens were placed on two supports that were 20 mm apart. A force was applied at mid-span at a crosshead speed of 0.5 mm/min until failure and the flexural strength calculated: $s = 3Fl/2bh^2$, where F is failure load, l the distance between the supports, b the width, and h the height of the specimen. The modulus of elasticity (E) was calculated from the results of the three-point bending test. Ten specimens were used for each test method, with the exception of the modulus of elasticity test, which was calculated from the three-point bending test.

Analysis of variance (ANOVA) and Tukey test at a significance level of 95% were used for statistical analyses.

RESULTS

The compressive strength, diametral tensile strength, flexural strength, and modulus of elasticity results are presented from Tables 2 through 5 respectively.

Table 2 shows the results of the compression strength of Z250 composite resin. Continuous, exponential, intermittent and stepped light methods revealed the highest compression strength values (not significantly different). The lowest values were shown by the PAC and LED techniques (not significantly different), which were significantly lower than those obtained through other methods.

The results for diametral tensile strength are listed in Table 3 and showed that there was no statistical difference between the LED, stepped light, PAC, exponential light and continuous light methods. The lowest value was found for the intermittent light, which presented a significantly lower value than LED.

TABLE 2- Means of compressive strength of Z250 composite resin

Light-curing method	Compressive strength (MPa)	
Continuous light	298.32 ^{a}	(20.98)
Exponential light	282.9 ^{a}	(28.50)
Intermittent light	280.6 ^{a}	(30.11)
Stepped light	269.44 ^{a}	(36.55)
PAC	225.12 ^{b}	(38.54)
LED	213.91 ^{b}	(38.30)

Means followed by different letters are statistically different at 5% by the Tukey test.
( ) Standard Deviation.

TABLE 3- Means of diametral tensile strength of Z250 composite resin

Light-curing method	Diametral tensile (MPa)	
LED	66.35 ^{a}	(12.36)
Stepped light	63.52 ^{ab}	(9.95)
PAC	59.73 ^{ab}	(13.77)
Exponential light	58.88 ^{ab}	(10.85)
Continuous light	57.91 ^{ab}	(12.97)
Intermittent light	49.95 ^{b}	(10.53)

Means followed by different letters are statistically different at 5% by the Tukey test.
( ) Standard Deviation.

The flexural strength values (Table 4) showed no statistical difference between all light-curing methods. Finally, Table 5 presents the results of the modulus of elasticity calculations, which demonstrated that the LED, exponential, continuous, and intermittent light methods had significantly higher values compared to the PAC and stepped light methods.

DISCUSSION

Light-cured composite resins are basically composed by a resin matrix, inorganic fillers, and a coupling agent. Thus, composition characteristics of these materials have an influence upon their final properties and clinical performance. The filler content, size, type, and distribution, as well as coupling between particles and matrix are also factors that influence mechanical properties such as strength and modulus of elasticity[4,5,11]. However, the degree of conversion of double bonds, for a given monomer system, is a co-determinant of mechanical properties of the resulting polymeric matrix[3,8,12,20]. As the degree of conversion of double bonds is dependent on incident activation light characteristics, the choice of light curing unit and method may have important effects on the mechanical properties of composite resin.

The present study investigated four mechanical properties: compressive strength, diametral tensile strength, flexural strength, and modulus of elasticity using different light-curing methods to cure the Z250 composite.

Three light sources and six light-curing techniques were used: 1). halogen lamps, which were employed for: 1.a- continuous light, 1.b- stepped light, 1.c- intermittent light and, 1.d- exponential light; 2). PAC, and 3). LED.

The results obtained in this study (Tables 2–5) showed differences in the mechanical properties for Z250 composite when light-cured by different methods. The compressive strength values demonstrated no statistical difference for continuous, exponential, intermittent, and stepped light methods, which were higher than those for PAC and LED (Table 2). The explanation for this result may be related to energy density, which is the product of the output of the curing unit and the time of radiation [18]. The energy density is an indication of the total light energy to which the material is subjected [23]. The PAC and LED devices present the lowest energy densities, 3.96 and 14 J/cm^2, respectively. However, there is a difference in the order of magnitude between the energy densities of these two methods. Yet, these methods represent the two extremes of the relationship between light intensity and exposure time. While the PAC presents a very high light intensity (1320 mW/cm^2) for a few seconds (3 s), the LED supplies a lower intensity (350 mW/cm^2), but at longer exposure time (40 s). The highest compressive strength values were found for the light-curing methods that employ halogen lamps, which supply an intermediate light intensity, but provide higher energy densities due to exposure time (21 – 32 J/cm^2). These results are in agreement with previous studies in the literature, which suggest that light intensity does not significantly affect the composite resin properties as long as a constant energy is used[13,14,22].

The highest value for diametral tensile strength was obtained for LED, followed by stepped light, PAC, exponential and continuous light (no significant difference between them), while the lowest value was found for intermittent light (significantly lower then LED) (Table 3). This finding demonstrated a difference in the behavior of the Z250 composite in relation to compressive strength *vs.* diametral tensile strength, when light-cured by different methods. Previous studies have already demonstrated no correlation between these mechanical properties[6,7,23]. According to Brosh, et al.[6] high compressive strengths are not correlated to the material's ability to withstand tensile loads. When tensile stresses develop in a restoration, crack development and propagation within the material occurs, jeopardizing the restoration clinical outcome.

In the present study, the specimens for compressive and diametral tensile strengths were prepared utilizing silicon molds, which did not offer resistance to the displacement of the specimen, minimizing the formation of cracks and flaws within the material bulk and surface during their preparation. As the same material was used in all conditions tested, characteristics concerning the light source and curing process itself might explain these differences.

Although light intensity and energy density are usually

TABLE 4- Means of flexural strength of Z250 composite resin

Light-curing method	Flexural Strength (MPa)
Intermittent light	172.97 ^{a} (19.63)
Exponential light	168.78 ^{a} (12.79)
LED	164.36 ^{a} (14.25)
Stepped light	163.16 ^{a} (31.44)
PAC	155.23 ^{a} (15.87)
Continuous light	154.10 ^{a} (23.40)

Means followed by different letters are statistically different at 5% by the Tukey test.
( ) Standard Deviation.

TABLE 5- Means of modulus of elasticity of Z250 composite resin

Light-curing method	Modulus of Elasticity (GPa)
LED	11.57 ^{a} (1.14)
Exponential light	11.00 ^{a} (0.89)
Continuous light	10.90 ^{a} (0.92)
Intermittent light	10.68 ^{a} (1.82)
PAC	9.04 ^{b} (0.94)
Stepped light	8.92 ^{b} (1.03)

Means followed by different letters are statistically different at 5% by the Tukey test.
( ) Standard Deviation.

related to higher degree of conversion and mechanical properties[3,7,8,12,14,19,22], these properties are not specifically related to spectral emission. Thus, each light source may exert dissimilar influences upon the Z250 composite, the photo-initiator of that being camphorquinone, which has an absorption peak at a wavelength of 468 nm[9]. The light curing units used in this study present great differences in relation to spectral emission. The halogen lamps are characterized by a wide wavelength that ranges between 380 – 510 nm, with a wavelength peak at 484 nm[9]. These light sources emit a significant quantity of energy towards the red end of the spectrum, i.e., they generate heat[10,13]. The PAC curing units supply high light intensity, at a narrow and symmetric wavelength of around 470 nm (440 – 500 nm), but these are used for just a few seconds[13]. Finally, the LEDs supply a low light intensity[9] and present a narrow and symmetric wavelength that lies between 450 – 490 nm, with a wavelength peak at 466 nm.

Since the molar absorption of camphorquinone is a function of wavelength, different emission profiles will have non-equivalent effects on the initiator[13]. In fact, energy is not the main issue, but the correct wavelength that is supplied to the photo-initiator. This energy requires no more than one photon, in a proper wavelength, to achieve the excitation of the camphorquinone. The efficiency of absorption clearly varies with wavelength and is never 100%, thus, the efficiency of the creation of free radicals will also be less than 100% [16]. However, the creation of the excited state of the photo-initiator reduces the concentration of excitable molecules and, therefore, the efficiency of absorption declines. The photo-initiator system may rapidly become saturated when the radiation is increased and, as a consequence, no further improvement in the rate of polymerization initiation is possible. Furthermore, at high rates of free radicals generation, mutual annihilation and reduced reaction chain lengths occur, leading to poorer network formation and poorer mechanical properties [16]. It is possible that over-exposure may be detrimental if a high-radiation lamp is chosen [16]. Lovell et al. [13] examined the effect of initiation rate using different types of light sources on the conversion and flexural strength of a resin mixture model that did not contain filler particles. The authors found that although the specimens were exposed to similar absorbed photon flows (controlled by the use of appropriate filters), the polymerization rates were not equivalent. This may have occurred due to nature of the light sources utilized in that study: a PAC curing unit that supplies a high light intensity (1800 mW/cm^2) for a few seconds (1 – 18 s); and a halogen lamp, which emitted a filtered light between 440 – 510 nm (200 mW/cm^2) at various exposure times (3 – 80 s). These findings suggested that mutual annihilation and reduced reaction chain length really occurred.

Compressive and tensile stresses form the basis of flexural tensions. Hence, the flexural strength might be able to predict more clearly the characteristics of the material from a practical point of view. The results of this study indicated that flexural strength values were not affected by the curing light sources (Table 4). However, the modulus of elasticity, which was calculated from the three-point bending test, showed higher values for LED, exponential, intermittent and continuous light than those for PAC and stepped light (Table 5). These findings may depict that the modulus of elasticity of the Z250 composite is a more sensitive indicator of differences between light-curing methods than flexural strength.

The lowest modulus of elasticity values were obtained for the PAC and stepped light and probably resulted due to differences in polymerization kinetics and network formation for the two methods employed. The PAC source supplies a high intensity, which theoretically may have resulted in two limitations. First, the photo-initiator system may rapidly become saturated by the high light intensity supplied, resulting in no improvement in the initiation rate and annihilation of the free radicals [16]. Second, faster polymerization rates produced by high intensity sources might result in shorter chains with lower molecular weight and less cross-linking. Higher molecular weight may be an important factor since it may yield tie molecules[18,21]. For the stepped light, another phenomenon may have occurred. The low intensity supplied in the initial 10 seconds (presenting wide wavelength) provides the lowest polymerization rates, which may result in more linear polymeric chains with reduced density of cross-linking. However, lower initiation and polymerization rates in initial stages of the reaction may lead to an extensive primary cyclization, creating microgels, promoting higher local conversion, phenomenon that may lead to reductions in the effective cross-linking densities, compromising the mechanical properties[1].

Finally, in terms of mechanical properties, the results of this study are not conclusive. The findings showed that each property revealed a different behavior as a function of the light-curing method considered. Unfortunately, the macroscopic testing performed in this study cannot confirm all hypotheses mentioned, these only described the behavior of the Z250 as a function of the light-curing method. Due to the complexity of this subject, microscopic testing added to polymeric characterization techniques may better describe the network structure as a function of polymerization conditions and may be subject of further research.

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