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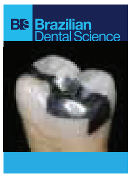

ORIGINAL ARTICLE

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Analysis of flexural strength of composite resins polymerized by 2nd and 3rd generation leds

Análise da resistência flexural de resinas compostas polimerizadas por led 2ª e 3ª geração

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ABSTRACT

Objective: This study aimed to investigate the mechanical properties of three composite resins using 2nd and 3rd generation LED-based light-curing devices. **Material and Methods:** Sixty specimens distributed according to the type of resin (Group Z350 - nanoparticle composite resin [Filtek Z350 / 3M ESPE]; Group AP - microhybrid composite Amelogen Plus/Ultradent; Group DF - Group composite resin Durafil l/Heraeus Kulzer), and the light-curing device (Group 2ndG - 2nd generation LED-based curing unit at power density of 500 mW/cm^2; Group 3rdG - 3rd generation LED-based curing unit at power density of 1100 MW/cm^2). The specimens were stored in a dark, dry container at 37 °C in an incubator for 24 h and submitted to the mini-flexural test on universal test machine (EMIC) to determine the elastic modulus and flexural strength using a three-point test. The data were submitted to two-way ANOVA (Resin Composite X LED) and Tukey test (5%). **Results:** Concerning to flexural strength (in MPa), ANOVA showed significant in Tukey test for the interaction between the factors: Group Z350/3rdG - 105.36a; Group AP/3rdG – 81.49ab; Group DF/3rdG – 66.43bc; Group AP/2ndG – 66.13bc; Group DF/2ndG: 60.61bc; Group Z350/2ndG: 47,19c. With regard to the modulus of elasticity (in GPa), the results obtained were: Factor resin composite - Group Z350 (8.85a) > Group AP (7.61b) > Group DF (1.94c); Factor LED - Group 3rdG (7.13a) > Group 2ndG (5.14b). **Conclusion:** It was concluded that the 3rd generation LED (1100 mw/cm^2) significant increased the means of the flexural properties of composites. It was also concluded that the result of flexural properties of composites depends on the resin material tested.

KEYWORDS

Composite resin; Flexural strength; Modulus of elasticity.

RESUMO

Objetivo: Este estudo teve o propósito de investigar as propriedades mecânicas de 3 resinas compostas, utilizando dois fotopolimerizadores à luz de LED com diferentes densidades de potência. **Material e Métodos:** Sessenta espécimes (2x2x12 mm) de 3 marcas comerciais de resina composta (n = 20): Grupo Z350 - resina composta nanoparticulada Filtek Z350(3M ESPE), Grupo AP - resina composta microhíbrida Amelogen Plus (Ultradent), Grupo DF - resina composta microparticulada Durafil (Heraeus Kulzer). Em seguida, os grupos foram divididos em 2 subgrupos de acordo com LED utilizado para fotopolimerização, variando a densidade de potência: Subgrupo 500 - 500 mw/cm^2 (2ª geração); Subgrupo 1100 - 1100 mw/cm^2 (2ª geração). Os espécimes foram armazenados em recipiente escuro e seco à 37 °C em estufa por 24 h e submetidos ao ensaio de mini-flexão na máquina de Ensaio Universal EMIC para determinar o módulo de elasticidade e resistência à flexão 3 pontos. Os dados foram submetidos aos testes ANOVA dois fatores (Resina composta X LED) e Tukey (5%). **Resultados:** Para a resistência flexural, ANOVA mostrou diferenças significantes para a interação entre os fatores: Grupo Z350/1100 (em Mpa) - 105,36a; Grupo AP/1100 - 81,49ab; Grupo DF/1100 - 66,43bc; Grupo AP/500 - 66,13bc; Grupo DF/500: 60,61bc; Grupo Z350/500: 47,19c. Para o módulo de elasticidade, ANOVA mostrou diferenças significantes para o fator Resina composta: Grupo Z350 (em GPa): 8,85a; Grupo AP: 7,61b; Grupo DF: 1,94c; e para o fator LED: Subgrupo 1100: 7,13a; Subgrupo 500:5,14b. **Conclusão:** O LED de 3ª geração (1100 mw/cm^2) demonstrou aumentar significantemente as propriedades flexurais das resinas compostas, e o tipo de partícula de carga da resina composta parece influenciar diretamente nas propriedades flexurais das resinas compostas.

PALAVRAS-CHAVE

Resina composta; Resistência à flexão; Módulo de elasticidade.

INTRODUCTION

The composite resin restorations for both anterior teeth and posterior teeth are constantly being subjected to considerable flexural/functional stress [1]. Thus, one way to assess one of the indispensable prerequisites for its use as a restorative material is the mechanical resistance to fracture, by using the flexural strength test [2].

According to the International Standards Organization (ISO) [3], the flexural strength is the mechanical resistance condition known as a failure caused by the tension of the resin material measured by its curvature [1,2,4,5]. The clinical relevance of this property is present primarily in the act of mastication, when there are different masticatory forces, which induce various stresses, both in the tooth and the restoration [1,2].

In addition to the flexural strength, another important parameter supplied by the mechanical flexural test is the modulus of elasticity, which describes the relative stiffness or hardness of a material measured by the reduction in the elastic region of the deformation/stress diagram. It is the principle of the lower deflection for a given value, the greater is the value of the modulus of elasticity. [2,6]. Its importance is because different clinical situations require a restorative material with different elastic moduli.

The most widely used equipment for light-curing the composite resins are light-emitting diodes or LEDs. The light is generated through the use of gaseous semiconductors, usually gallium nitrate, which generate blue light required for photoactivation [7,8]. These devices have the advantage of not emitting infrared radiation into the composite resin and the tooth, producing low heat, thereby reducing the deterioration of the internal components over time, and having greater clinical duration [7-9].

The LED devices can be classified into 1st, 2nd, and 3rd generation, and this classification relates to the number of LEDs, the power density emitted, and the spectral range [7]. The first LED devices launching into the market were named 1st generation LEDs, which had several low emission light LEDs together, resulting in low power density devices (< 150 mW/cm^2) and had absorption spectrum between 450 and 490 nm, with a peak of 470 nm coinciding with maximum absorption spectrum of the photo initiator used in most composites, the camphorquinone (468 nm); however, it resulted in lower curing efficiency of composite resins [7,8,10-13]. The 2nd Generation LED devices have only one LED (higher surface area) or “microbeam” (chip) LEDs, which emit output power between 300 and 1000 mW/cm^2 and blue wavelength (450 to 490 nm), compatible with the sensitization of camphorquinone [7,8]. The 3rd generation LEDs have “microbeam” LEDs that emit different wavelengths between 375 nm (violet) to 510 nm (blue), creating a wide spectral range, and deliver higher power of 1000 mW/cm^2 [7,8]. These 3rd generation LEDs allow that both camphorquinone and other photo initiators (PPD/phenyl propanedione; TPO/Alkyl phosphinic oxidemono oxide mono and BAPO/ bis-alkyl phosphinic oxide) are sensitized during photoactivation [7,8,13].

The success of the restoration depends, among several factors, on the adequate curing of composite resin. Both 2nd and 3rd generation devices allow an effective polymerization of the composite resin restorations [13]. Notwithstanding, the increase in light intensity may result in higher degree of conversion of the composite, and the greater the degree of conversion of the composite, the greater is its mechanical properties [10,11], e.g., flexural strength and module of elasticity.

Thus, this study aimed to evaluate the flexural properties of three direct composite resins with different types of particles light-cured by LED units with different power densities. The tested hypotheses were: (1): composite resins, are different with respect to flexural properties; (2) the different power densities differ from each other regarding the flexural properties.

MATERIAL AND METHOD

Sixty specimens were made of composite resin, which are divided into 3 groups according to the type of resin composite used, as follows:

- Group FK: nanoparticle resin composite Filtek Z350(3M ESPE, St. Paul, MN, USA), shade A3;
- Group AP: microhybrid resin composite Amelogen Plus (Ultradent, South Jordan, UT, USA), shade A3;
- Group DL: microfilled resin composite Durafill (Heraeus Kulzer, GMbH, Kg, Germany), shade A3.

All tested resins, manufacturers, compositions and classifications are shown in Table 1.

The composites were inserted at a single increment into prefabricated bipartite metal matrix with dimensions of 12 mm x 2 mm x 2 mm [5] placed on a glass plate. A colorless polyester strip (FAVA, São Paulo, SP, USA) was positioned on the metallic matrix and then pressed by a second glass plate. After the removal of the glass plate, the excess material was removed with the aid of a spatula.

Then, the composites were photo-activated through the LED device by varying the intensity of light, divided into 2 subgroups (n = 10):

- Subgroup 2ndG: 2nd generation LED device (Emitter A, Schuster LTDA, Santa Maria, RS, Brazil) at average power density of 500mW/ cm^2, measured by a radiometer (Curing Radiometer Model 100, Demetron Research Corporation, Danbury, CT, USA), LED tip diameter of 8 mm.
- Subgroup 3rdG: 3rd generation LED device (Demi LED Light Curing System- Kerr Corporation, Middleton, WI, USA), at average power density of 1100 mw/cm^2, measured by a radiometer (Curing Radiometer Model 100), LED tip diameter of 8 mm.

The photoactivation was performed on two points on the top of the metal matrix for 20 s at each point, according to the manufacturer's instructions (Figure 1). The specimens were prepared by a single operator to achieve standardization.

Table 1 - Resins used in this study.

Name	Manufacturer	Average size of particles	% Filler	Composition	Classification
Amelogen Plus	Ultradent, South Jordan, UT, USA	0.7 µm	76%	Bis-GMA, boron glass, aluminum, barium silicate (particle size from 0.4 to 0.7 microns). Photo initiator: camphorquinone	Microhybrid
Filtek Z350	3M ESPE, St. Paul, Mn, USA	75 nm	78.5%	Bis-GMA), bis-EMA, primary non-agglomerated silica (average size 20 nm) and clusters of zirconia and silica (particles 0.6 to 1.4 microns) Photo initiator: camphorquinone	Nanoparticulate
Durafill	Heraeus Kulzer, GMbH, Kg, Germany	0.04 µm	54%	Urethane dimethacrylate (UDMA), highly dispersed silicon dioxide (0.02 to 0.07 microns), pre-polymerized particles (10-20 microns) Photo initiator: camphorquinone	Microfilled

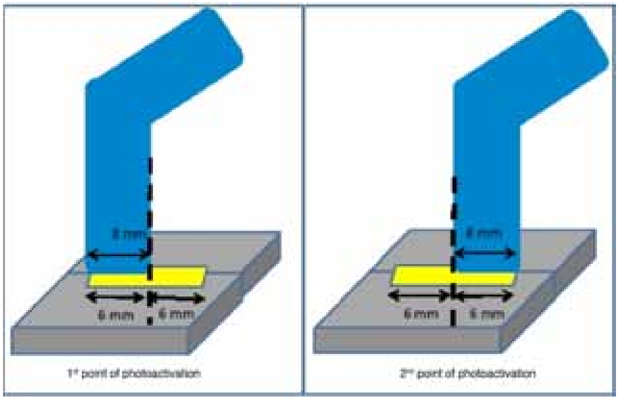

Figure 1 - Illustrative picture of the light-curing of the specimens.

Then the specimens were stored in plastic containers in dark for 24 h at 37 °C in a bacteriological incubator (ECB 11 Digital - Odontobrás, Ribeirão Preto, SP, Brazil).

Then, the specimens were subjected to modified flexural test [1,2,5], to determine the values of flexural strength and modulus of elasticity. The specimens were placed in a three-point test device containing two parallel surfaces apart from each other by 9 mm and subjected to load equidistant to support points, at a rate of 0.75 mm/min in a universal testing machine EMIC (DL-2000,São Jose dos Pinhais, PR, Brazil)

The results of the flexural strength were obtained in N and converted to MPa by using the formula from ISO 4049 specification, described below:

$$\sigma = 3FI/2bh2$$

Where σ is the flexural strength (MPa), F is maximum load supported (N), I is the length between the supporting points (9 mm), b is the width of the prism (2 mm), and h is the thickness of the prism (2 mm).

To calculate the elastic modulus following formula was applied:

ấ=I3 x F1 /4fbh3

Where I is the length between the supporting points, b [2 mm] and h [2 mm] are respectively the width and the height of the specimens, F1 [N] is the load and f [mm] is the deflection of the bar (in the elastic phase).

The flexural strength and modulus of elasticity values were submitted to two-way ANOVA (Composite resin X LED) and Tukey test at a significance level of 5%.

RESULTS

Flexural strength

ANOVA showed a p-value < 0.05 for the factors Composite Resin and LED and the interaction between the factors.

Table 2 presents the results of Tukey test for interaction between factors. The resin composite Filtek Z350 associated with the 3rd generation LED showed flexural strength values significantly greater than all other resins, regardless of the light source. The resin composite Amelogen Plus associated with the 3rd generation LED showed flexural strength values significant higher than those of the resin composites Filtek Z350 and Durafill, associated with the 2nd generation LED. The resin composite Filtek Z350 associated with the 2nd generation LED exhibited flexural strength values significantly smaller than those of the other resins, regardless of the light source, except for resin composite Durafill associated with 2nd generation LED.

Modulus of elasticity

ANOVA showed a p-value < 0.05 for the factors Composite Resin and LED, however, for interaction between factors no statistically significant differences were found (p > 0.05).

Table 2 - Flexural strength results (MPa) of Tukey test for interaction between the factors

Resin composite	2nd generation LED	3rd generation LED
Filtek Z350	47.19(±12.51) d	106.36(±13,71) a
Amelogen Plus	70.13(±16.57) bc	81.49(±18,84) b
Durafill	60.61(±9.93) cd	66.43(±8,34) bc

* Means followed by the same letters do not show statistically significant differences.

Table 3 shows the results of Tukey test for factor resin composite.The composite resin Filtek Z350 (Group Z350) exhibited mean values of elastic modulus higher than those of other composites. The resin composite Amelogen Plus (Group AP) showed higher mean values of modulus of elasticity than that of resin composite Durafill (Group DF).

Table 3 - Results of the modulus of elasticity (in GPa) of the Tukey test for resin composite factor

Resin Composite	Mean (± SD)	Homogeneous groups *
Filtek Z350	8.85(±2.01)	A
Amelogen Plus	7.61(±1.76)	B
Durafill	1.94(±0.54)	C

Table 4 displays the results of the Tukey test for factor LED. The 3rd generation LED presented values of modulus of elasticity significantly higher than those of the 2nd generation LED.

Table 4 - Results of the modulus of elasticity (GPa) of the Tukey test for factor LED

LED	Mean (± SD)	Homogeneous groups *
3rd Generation	7.13(±3.59)	A
2nd Generation	5.14(±2.65)	B

DISCUSSION

The first hypothesis tested in this study was accepted because the resin composites Amelogen Plus (microhybrid) and Filtek Z350 (nanoparticulate) had flexural strength mean values significantly higher than those of resin composite Durafill (microparticulate). The microparticulate resin composites have filler particles about 300 times smaller than one quartz particle, of the order of 0.04 microns and are made of fumed silica or colloidal silica [1,2,14]. Clinically, they behave very well when used in anterior area with direct aesthetic involvement and in surfaces near or in contact with the gingival tissues. Since its physical and mechanical properties are lower than traditional resins, their use in areas on masticatory stress becomes limited, in addition to present higher water sorption, high coefficient of thermal expansion, high polymerization shrinkage, low modulus of elasticity and low tensile strength [9].

Hybrid or microhybrid resins are a mixture of microparticles with macroparticles, thus presenting features of both types of materials. Most has about 10 to 20% by weight of colloidal silica microparticles and 50 to 60% of heavy metal glass macroparticles, totalizing a filler percentage between 75% and 80% by weight and filler particle sizes ranging from 0.04 μm to 10 μm, which gives high resistance to fracture [2,9].

The nanoparticulate direct resin composites were introduced recently in dental market, in order to meet the growing demand for a universal restorative material, that is, one that could be used in both anterior and posterior teeth [9]. These resins have the filler particles of approximately 0.02 nm, which involves two types: nano-sized and nano-agglomerated. This technology aims to give the material various properties, such as improved polishing, better radiopacity, durability, color excellence, surface smoothness similar to that of microhybrid resins, particularly as regards their mechanical properties. Due to the small size of its particles and the high filler content, the nanoparticulate resins have high wear resistance and adequate resistance to fracture in areas of high masticatory stress [2].

In this study, lower flexural strength values were obtained with the resin composite Durafill showing statistically significant differences when compared to Filtek Z350 and Amelogen Plus. These values can be justified by the increasing in the amount of inorganic filler incorporated into the resin matrix of the composite because the composites Durafill, Amelogen Plus, and Filtek Z350 respectively have in their compositions about 54%, 76%, and 78.5 % by weight of total inorganic filler. Corroborating the results of this study, authors [15-18] suggested that the increase of the flexural properties of resin materials is directly proportional to the increase of the amount of inorganic filler by weight. Consequently, it can be expected that a composite resin with large amount of filler shows excellent mechanical properties as observed in this study.

The second tested hypothesis in this study was accepted for the flexural strength as the 3rd generation LED promoted flexural strength values significantly higher than those of 2nd generation LED. The photoactivation by the 3rd generation LED promoted flexural strength values significantly higher than those of 2nd generation LED, probably due to the higher degree of conversion of the composite, thus improving its mechanical properties. According to Santos et al. [19] the higher the light intensity, the greater is the number of photons present; and the greater the number of photons, the greater is the number of camphorquinone molecules that will reach the excited state to react with the amine to form free radicals. Thus, the higher the intensity, the greater is the extent of polymerization composite resin which is a favorable factor for increasing the strength of the direct restorative material [20].

The resin composite Filtek Z350 associated with 3rd generation LED showed flexural strength values significantly higher than those of the other resins. Such results demonstrated an excellent association between the use of a composite resin with nano technology (nanoparticulate with nano-agglomerated) with high inorganic filler content (78.5%) and the use of higher light intensity, which probably resulted in higher conversion of monomers into polymers, and, consequently, increased its mechanical properties when compared to other composites.

However, it was observed that the resin composite Filtek Z350 associated with the 2nd generation LED exhibited the lowest results of flexural strength, significantly smaller than those of the other composites tested, except for the resin composite Durafill associated with the 2nd generation LED. Such results are surprising because they demonstrate that the intensity of light exerted a significant influence on the flexural strength of resin composite Filtek Z350, and the same was not true for resin composites Amelogen Plus and Durafill because such resins presented similar values of flexural strength for both tested light intensities.

Furthermore, according to the ISO 4049 specification the minimum value of flexural strength for composites must be 50 MPa, and Filtek Z350 associated with the LED light intensity of around 500 mW/cm^2 (2nd Generation) showed mean values of 47.19, below the mean determined by ISO 4049. Thus, for Filtek Z350, there may be need for indication of the use of high light intensities so that it can have appropriate mechanical properties. Such results contradict studies that claim that the 2nd and 3rd generation LED devices provide an effective polymerization of the composite resin restorations [13,21,22].

The composite resin Amelogen Plus associated with the 3rd generation LED showed flexural strength values significantly greater than those of the composites Filtek Z350 and Durafil l associated with 2nd Generation LED. The increased light intensity may result in higher degree of conversion of the composite, and the greater the degree of conversion of the composite the higher is their mechanical properties [10,11,22], e.g., flexural strength and modulus of elasticity.

With regard to the modulus of elasticity, the first hypothesis tested in this study was accepted because the nanoparticulate resin composite Filtek Z350 showed the highest flexural modulus values than those of the other tested resins. Furthermore, microhybrid composite Amelogen Plus showed the highest flexural modulus values than those of the composite resin Durafill. The flexural strength test allows to obtain the results of the flexural modulus, also called modulus of elasticity, and the modulus of elasticity values (measured in the GPa) are directly proportional to the flexural resistance values (measured in MPa) [1,2]. As explained above, increasing the amount of filler significantly improves all the mechanical properties of the composites [15-18], justifying our findings in which the composite Filtek Z350 (78.5% percent of inorganic filler) had the highest modulus of elasticity values than those of composites Amelogen Plus (76%) and Durafill (54%).

The second hypothesis tested in this study was accept for the modulus of elasticity as the 3rd generation LED presented mean values significantly higher than those of the 2nd generation LED. As previously explained, the larger the power density or intensity of light on the composite resin, the greater is the degree of conversion and hence the extent of composite resin polymerization, increasing its mechanical properties [19,20]. Probably the power density of 1100 mW/cm^2 resulted in higher conversion of monomers into polymers in the organic matrix of the composites than that of the power density of 500 mW/cm^2, increasing the modulus of elasticity of the group activated by the 3rd Generation LED when compared with the group activated by 2nd Generation LED.

Changes in the composition, such as the size and quantity of inorganic filler particles incorporated into the organic matrix of the resin composites, determines changes in their mechanical properties, allowing the professional diversifying the clinical application. The composite resins with nanotechnology have shown excellent results in relation to physical, mechanical, and aesthetic properties in laboratorial studies [1,5,14,23]. Notwithstanding, our findings demonstrated that such resins exhibit reduced flexural strength and modulus of elasticity when cured by 2nd generation LEDs compared with the 3rd generation LED. Therefore, the performance of nanoparticulate composite resin Filtek Z350 both in vitro and in vivo studies at immediate and long term should be further researched, observing its physical and mechanical behavior by varying the different light intensities.

CONCLUSIONS

According to the results obtained in this study, it can be concluded that:

- Resin composites Filtek Z350 (nanoparticle) and Amelogen Plus (microhybrid) showed higher flexural strength than resin composite Durafill.
- The photoactivation by 3rd generation LED promoted flexural strength values and modulus of elasticity means significantly greater than the photoactivation by 2nd generation LED;
- The composite resin Filtek Z350 associated with 3rd generation LED showed flexural strength mean values significantly greater than those of all other resins, regardless of the light source;
- The resin composite Amelogen Plus associated with the 3rd generation LED showed flexural strength values significantly smaller than those of the composites Filtek Z350 and Durafill associated with the 2nd generation LED;
- The resin composite Filtek Z350 associated with the 2nd generation LED showed flexural strength values significantly smaller than all other resins, regardless of the light source, except for resin composite Durafill associated with the 2nd generation LED;
- The resin composite Filtek Z350 showed higher modulus of elasticity than those of the other composites; the resin composite Amelogen Plus exhibited mean values of modulus of elasticity higher than those of composite Durafill.

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