Biaxial Flexural Strength of Direct Composite Resins Using Different Light-Curing Times and Post-Cure Method

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Abstract

The aim of this study was to evaluate the biaxial flexural strength of four composite resins of different compositions, light-cured in two stages, and then, post-cure using heat and pressure.

There were made 480 composite resin disks, being 120 disks each one. Of these 120 disks, 60 were light-cured for 20 seconds, the other 60 for 60 seconds. Then, half of each photoactivated group (n = 30) was subjected to post-cure using heat and pressure and the other half (n = 30) was not. Next, the disks were stored in distilled water for 24 hours before testing. After the statistical analysis, it was found that the composite Z100 and Filtek Z350 had a higher value of biaxial flexural strength in the groups of 20 and 60 seconds in the conventional method. In the condition of 20 seconds post-cure, the highest values are achieved by Z100 (18.65 MPa) and Filtek Z350 (24.71 MPa). While in the condition of 60 seconds post-cure the best result was achieved with the Z100 (64.88 MPa) and the lowest value was the IPS Empress Direct (7.56 MPa).

There was a statistically significant decrease in biaxial flexural strength in the Z100 that received the light curing for 20 seconds and post-cure (18.65 MPa), when compared to conventional polymerization (28.29 MPa). Therefore, there is no need to light-cure the composites exceeding the manufacturers recommended time, in the conventional method. The post-cure protocol promoted increase in biaxial flexural strength only for the Z100 light-cured for 60 seconds.

Experimental article (J Int Dent Med Res 2023; 16(3): 936-942)Keywords: Flexural strength, composite resins, light curing of dental adhesives, polymerization.Received date: 19 December 2022Accept date: 22 August 2023

Introduction

Among the restorative materials, the composite resin is one of the materials widely used in clinical restorative dentistry, as it caters to a cost-benefit ¹. Since its introduction in the late 1950s, by Rafael Bowen, to recent nanocomposites, composite resin has been the subject of much research, evolving in its chemical and physical properties ^{2,3}.

Its basic components are organic matrix, inorganic particles (filler) and bonding agent. Besides these, there are additional components, which are the coloring pigments, the initiatoractivator system (camphorquinone + tertiary amine) - responsible for triggering the

*Corresponding author: Camilla Lage Martins, Department of Dentistry, Pontifical Catholic University of Minas Gerais, Belo Horizonte, Minas Gerais, Brazil. E-mail: Camilla.lage@sga.pucminas.br polymerization of the composite - and stabilizers ^{3, 4-6}. The chemical properties are based on how the molecules interact, combine and change according to which they are exposed. The degree of conversion measured is the percentage of breakage of carbon double bonds converted to single bonds during polymerization of monomers ⁶⁻⁸. To succeed in restoration process, it is necessary that the resin undergoes an adequate degree of conversion, that is, good amount of resin monomers (60%) must be sensitized in the polymerization process ⁹.

To trigger the polymerization reaction, the composite resins have the combination of camphorquinone and a tertiary amine as a primer-activator system - which is used to accelerate the process. In addition to photoactivation, polymerization can be complemented by additional treatment, such as heat and pressure, which can be carried out under controlled environmental conditions. These additional treatments lead to an increase in the degree of conversion, an improvement in the

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mechanical properties, color stability and a reduction in the wear of these materials. ¹⁰⁻¹². The autoclave may be used for this purpose because its operation is based on the application of heat and pressure ¹³⁻¹⁵.

Within the physical properties, there is a subgroup based on the laws of mechanics that consist of the measures of resistance of the materials, if they undergo any change when exposed to a force, pressure or tension ¹⁶.

A resistance force is as a support material until the time at which plastic deformation or a fracture ⁶. Either the biaxial flexural strength test is a method in which the shaped sample disk is placed on top of a support, a three-legged support or on a ring, which supports the entire circumference of the sample ^{17, 18}. In this study, the mechanical test used disk-shaped specimens, supported on a hollow support with a 45° edge. Then they were subjected to mechanical loading, through the contact of a piston made of metal. The metallic piston descends in a controlled way to the edge of the lower support and forces the specimen up to the moment of its deformation.

The measurement of the specimen and the force required to deform it are noted and, subsequently, the biaxial strength value is determined by means of a mathematical formula. Thus, with this type of test can evaluate the biaxial strength resin made small increments.

More specifically, can evaluate a characteristic type of composite resin, one that has shade of enamel, which every company currently has. This increment can then be isolated and studied separately from the core of the material; therefore, the methodology of the present study was the biaxial flexural strength test of small increments of composite resin in the color of enamel. This intention was generated from the scarcity of studies in the literature that address the effect of different techniques on some type of mechanical property of composite resin.

With the knowledge acquired, the objective of this research was to evaluate the biaxial flexural strength on in direct composite resins that received photopolymerizations in two times of exposure to light and, also, with post-cure treatment through heat and pressure or not.

Materials and methods

Materials

Four direct composite resins brands for

enamel of different compositions were evaluated, as folow: Z100 [™] Resin (3M / ESPE, St Paul MN, United States), Filtek [™] Z350 XT (3M / ESPE, St Paul MN, United States), IPS Empress Direct (Ivoclar Vivadent, AG, Schaan/Liechtenstein, Germany) and Palfique LX5 (Tokuyama, Japan). Figure 1 shows the characteristics of these composites.

The photopolymerizer used was VALO® (ULTRADENT - USA), with LED base (Light Emitting Diode), power of 1,000 mW / cm².

The autoclave used is a steam under pressure Vitale Class (Biosafety Cristófoli - Brazil), with 12 liters capacity and power 1,200 Watts. This equipment complies with the NBR ISO 13485 standard.

The digital caliper to measure the thickness of the specimens was VONDER® - Model: PD 150 / 150mm.

The storage containers for the samples were Eppendorf microtubes (1.5 ml) and the substance chosen was distilled water.

				Composition
Composite	Manufacturer	Classification	Lot	(% Weight)
Z100™	3M/ESPE, St Paul MN <u>,</u> USA.	Micro-hybrid	1826700549	Ceramics treated silanized (80-90%); Triethyleneglycol dimethacrylate (TEGDMA) (1-10%); Bisphenol A diglycidyl ether dimethacrylate (Bis- GMA) (1-10%); 2-Benzotriazolyl-4- methylphenol (0.1416-0.145%).
Filtek™ Z350 XT	3M/ESPE, St Paul MN, USA.	Nanofiller	1816600592	Ceramics treated silanized (60-80%); Silane treated silica (1-10%); Urethane dimethacrylate (UDMA) (1- 10%); Bisphenol A polyethylene glycol diether dimethacrylate (Bis- EMA) (1-10%); Bisphenol A diglycidyl ether dimethacrylate (Bis-GMA) (1- 10%); Zirconia ceramic, surface modified with 3- methacryloxypropyltimethoxysilane, bulk material (1.96-5%); Polyethylene glycol dimethacrylate (PEGDMA) (<5%); Triethyleneglycol dimethacrylate (TEGDMA) (0.2364- 0.99587375%).
IPS Empress Direct	Ivoclar Vivadent, AG, Schaan/ Liechtenstein Germany.	Nano-hybrid	X20930	Urethane dimethacrylate (UDMA) (10- <25%); Ytterblum triffuoride (2.5- 10%); Tricyclodecane dimethanol dimethacrylate (2.5-10%); Bisphenol A diglycidyl ether dimethacrylate (Bis- GMA) (2.5-10%).
Palfique LX5	Tokuyama, Japan.	Supra nano-filler	E7201 e E7501	Triethyleneglycol dimethacrylate (TEGDMA) (1-10%); Bisphenol A diglycidyl ether dimethacrylate (Bis- GMA) (1-10%); Silica-zirconium

Figure 1. Technical characteristics of the tested materials

Sample making

The samples were made in a disk format using a silicone matrix, thereby standardizing the diameter (7 mm) and thickness (0.5 mm). Altogether, there were 480 composite resins disks, being 120 of each one (Figure 2.1).



Figure 2.1. Flowchart for making sample - total sample for each composite resin.

Of these 120 disks, 60 were light-cured for 20 seconds (manufacturer's recommendation), the other 60 were photoactivated for 60 seconds (exceeding this recommendation). Then, half of each photoactivated group (n = 30) was subjected to post-curing by heat and pressure and the other half (n = 30) was not (Figure 2.2). Subsequently, the disks were stored in distilled water for 24 hours before of the tests. With the resin spatula, the composite resin was accommodated on the mold and placed a strip of polyester on it simulating the polishing. An optical microscope coverslip was seated on the polyester strip and applied manual pressure on it, allowing possible excesses to flow and making the surface uniform. Then, the blade was removed and carried out the photopolymerization with VALO® (ULTRADENT - USA) at distance of 1 mm between the photopolymerizer and, the sample.



Figure 2.2. Flowchart for making sample - separation of the groups by time of light curing and post-cure.

Additional heat and pressure treatment

Half of all resin disks - 240 specimens, being 60 dikcs of each composite resin and of these 60, 30 disks each time (20 and 60 seconds) - were subjected to an additional heat treatment using an autoclave.

Each group of composite resin and photoactivation was properly packaged for use in an autoclave and a sterilization cycle was applied according to the instructions of the autoclave manufacturer for sterilization of dental instruments. The cycle corresponded to a heating time of 10 to 35 minutes, with a temperature of 134 ° C at a pressure of 216 kPa (2.2 kgf / cm2), sterilization time of 10 minutes and drying time of 35 minutes (maximum temperature drying temperature 120 ° C). This process is called post-cure.

Sample storage

Each disk was stored in an Eppendorf microtube (1.5 ml) completely filled with distilled water, sealed and stored in a cool room temperature for 24 hours. Immediately after removal of water, has measured the thickness of each test piece with a digital caliper (VONDER® - Model: PD 150 / 150mm) and their measurements were recorded on an appropriate sheet. Then, the test was performed.

Biaxial Flexural Strength Test

After obtaining samples, they were subjected to the biaxial flexural strength test. The samples were centered on the lower support edge (edge cast with 45°) of the device and, then being specimen subjected to mechanical load on the top center portion of the disk, through contact of a piston made of metal.

The metal piston descends in a controlled way to the edge of the lower support and forces the sample up to the moment of its deformation. The universal testing machine was equipped with a 500 N load cell (EMIC DL 500, Curitiba Paraná, Brazil) with constant descent programming at a speed of 0.5 mm / minute.

A computer attached to the testing machine recorded the load in Newtons (N) achieved at the moment of deformation of each sample. This value together with the thickness dimensions of each disk was applied to the following formula to calculate the biaxial flexural strength of each composite resin direct ¹⁹.

$$\sigma_{bf} = \frac{3P(1+v)}{4\pi t^2} \left[1 + 2ln\left(\frac{a}{b}\right) + \frac{1-v}{1+v} \left[1 - \frac{b^2}{2a^2} \right] \frac{a}{R^2} \right]$$

In this equation, we have:

P = load (N);

v = Poisson's ratio of the composite;

t = thickness of the composite disk (mm);

a = metal support diameter (mm);

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b = metal stem diameter (mm);

R = specimen radius (mm).

Being the Poisson's ratio (v) value defined as 0.27 $^{\rm 20}.$

The MegaPascal (MPa) values, within each experimental condition, were subjected to the Analysis of Variance one factor test and Tukey post-hoc test, with p<0.05, for the comparison between the light-curing for 20 and 60 seconds of the composite resins, in conventional and post-cure mode. The t test (p <0.05) was used to analyze potential differences in each time (20 or 60 seconds) within each polymerization condition: with and without postcure.

Results

Table 1 shows the values in MPa of undergoing photoactivation groups only in the conventional manner without the post cure procedure. In the 20-second group, Z100 (28.51 MPa) and Filtek Z350 (25.89 MPa) achieved the highest values for resistance to biaxial flexion, statistically different from the other groups. Within 60 seconds, the same result occurred - Z100 (30.61 MPa) and Filtek Z350 (26.04 MPa). There was no statistically significant difference between the times of 20 and 60 seconds in any of the photopolymerized resins.

	20 seconds	60 seconds
Z100	28.51 (9.83) ^{Aa}	30.61(9.39) Aa
Filtek Z350	25.89 (8.16) ^{Aa}	26.04 (13.76) Aa
Palfique LX5	12.19 (3.46) ^{Ba}	13.69 (4.53) ^{Ba}
IPS Empress Direct	13.25 (4.52) ^{Ba}	16.59 (4.03) ^{Ba}

Table 1. Biaxial flexure strength (MPa) and standard deviation (in parentheses) of composite resins subjected to two curing times in the conventional method.

^{A,B}Averages and standard deviations in columns followed by the same capital letters are not considered statistically different from each other (p> 0.05). P values were obtained using 1-Way ANOVA and Tukey post-hoc test. ^{a,b} Average and standard deviation on the lines followed by the same lowercase letters do not show statistically significant differences.

In Table 2, the biaxial flexural strength values of the composite resins subjected to lightcure and post-curing process are available. It is observed that in the condition of 20 seconds post-cure the highest values are achieved by composites Z100 and Filtek Z350 (18.65 MPa and 24.71 MPa, respectively), while in the condition of 60 seconds and post-cure the best result was achieved with Z100 (64.88 MPa). This

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material obtained a high result, showing a statistically significant difference in relation to the other composites. The Z100 also showed a statistically higher value in the condition of 60 seconds post-cure, when compared to 20 seconds post-cure.

The IPS Empress Direct showed the lowest value (7.56 MPa) among the materials of the condition 60 seconds with post-cure. This value was also considered lower and significantly different from the 20-second polymerization condition followed by the post-curing process (15.85 MPa).

	20 seconds post-cure	60 seconds post-cure
Z100	18.65 (6.58) Ab	64.88 (28.34) ^{Aa}
Filtek Z350	24.71 (14.30) Aa	21.00 (9.38) ^{Ba}
Palfique LX5	13.54 (5.09) ^{Ba}	13.59 (4.99) BCa
IPS Empress Direct	15.85 (4.03) ^{Ba}	7.56 (2.53) ^{Cb}

Table 2. Biaxial flexural strength (MPa) and standard deviation (in parentheses) of composite resins subjected to two curing times followed by the post-cure step.

^{A,B}Averages and standard deviations in columns followed by the same capital letters are not considered statistically different from each other (p> 0.05). P values were obtained using 1-Way ANOVA and Tukey post-hoc test. ^{a,b} Average and standard deviation on the lines followed by the same lowercase letters do not show statistically significant differences.

Table 3 shows the comparison of the materials within the time of 20 seconds submitted by two polymerization methods (conventional and with post-cure). There was a decrease in the biaxial flexural strength, statistically significant, in the Z100 composite that received the post-cure process (18.65 MPa), when compared to conventional polymerization (28.51 MPa). The other resins do not show statistical differences between their polymerization methods.

	20 seconds	20 seconds post-cure
Z100	28.51 (9.83) ª	18.65 (6.58) ^b
Filtek Z350	25.89 (8.16) a	24.71 (14.30) ª
Palfique LX5	12.19 (3.46) ^a	13.54 (5.09) ª
IPS Empress Direct	13.25 (4.52) ª	15.85 (4.03) ª

Table 3. Biaxial flexural strength (MPa) and standard deviation (in parentheses) of composite resins subjected to a time of 20 seconds.

^{a,b}Averages and standard deviation in the lines followed by the same lowercase letters do not show statistically significant differences according to the test-T with a significance level of 0.05.

Table 4 provides the comparison between composite resins within 60 seconds submitted to

different polymerization protocols. The Z100 shows a statistically significant increase in biaxial flexural strength when subjected to the post-cure process. For IPS Empress Direct, the post-curing process (7.56 MPa) caused a reduction in the biaxial flexural strength value, when compared to the conventional method (15.85 MPa).

	60 seconds	60 seconds post-cure
Z100	30.61(9.39) b	64.88 (28.34) ^a
Filtek Z350	26.04 (13.76) ^a	21.00 (9.38) ^a
Palfique LX5	13.69 (4.53) a	13.59 (4.99) a
IPS Empress Direct	16.59 (4.03) ^a	7.56 (2.53) ^b

Table 4. Biaxial flexural strength (MPa) and standard deviation (in parentheses) of composite resins subjected to a time of 20 seconds.

^{a,b}Averages and standard deviation in the lines followed by the same lowercase letters do not show statistically significant differences according to the test-t with a significance level of 0.05.

Discussion

In this study, the results show that the method was not always effective in increasing the biaxial resistance. It is likely that chemical formulations explain these results. Thus, the polymerization of each brand and in each experimental condition is, in turn, dependent on the chemical composition of the restorative materials.

The organic matrix of composite resin is made of aromatic and/or aliphatic dimethacrylate monomers - such as bisphenol A diglycidyl ether dimethacrylate (Bis-GMA), Bisphenol А polyethylene glycol diether dimethacrylate (Bisdimethacrylate EMA) urethane (UDMA), triethylene glycol dimethacrylate (TEGDMA) and ethylene glycol dimethylacrylate (EGDMA) generating high strength and rigidity 3, 5, 6, 21. When polymerizing initiate, monomers that have C=C bonds and intermolecular spacing of 0.3 to 0.4 nm are converted into chemical covalent C-C bonds between different monomer units, with the distance between molecules dropping to 0.15 nm ⁷. It is interesting to know that long linear chains of monomers maintained by covalent C-C bonds present high flexibility given the ability of this bond to rotate when some effort reaches its limits. Something that could prevent rotation is the greater number of crosslinks between the polymeric chains.

The inorganic part is composed of mineral filler (quartz, silica, barium, zinc, strontium and zirconium glasses, aluminum and lithium borosilicate glasses), whose basic function is to increase the mechanical properties of the composite resin and minimize the amount of organic matrix. This reduce the polymerization contraction, coefficient of thermal expansion and water absorption ^{5, 6, 22}. Therefore, there needs to be a balance between organic matter and inorganic load to arrive at an ideal composite.

During the polymerization, the degree of conversion is a measure of the percentage of the breakage of the double carbon bonds are converted into single C-C bonds. Among the factors that influence the conversion is the distance between the light and the composite resin, the size of the increment of the composite resin and its opacity ⁶⁻⁸. When the photoinitiator system is sensitized with the blue light used to cure the material, camphorquinone reaches an excited state and interacts with the amine and both are changed into free radical substances. Only free radicals can carry out the polymerization reaction ^{3, 4, 6, 23, 24} and this event is crucial to determine the clinical behavior of the restorative treatment as well as the mechanical properties of the composites. The quality of the formed polymeric network will depend on the organic content, but crediting the quality of the restorations and their mechanical properties only to this is a mistaken idea. These mechanical properties are relevant to the durability of composites ^{25, 26} and are related to the filler matrix, which includes factors such as shape, type, number, and distribution of inorganic particles. Restorative materials must have mechanical integrity to withstand the forces acting in the oral cavity for a long time, so it is important to study these properties for the clinic, since one of the biggest failures is due to fracture, due to the quality of the technique ^{18, 27}.

Some authors show high biaxial strength of Z100 composite resin polymerized in a conventional way when compared to indirect composite resins from laboratory and CAD/CAM, and temporary acrylic resins ^{26, 28}. That was similar to the results of the present study. The same result was found in this study for the time of 60 seconds (29.76 MPa in the conventional way and 64.88 MPa with post-polymerization, as seen in table 2 and 3). The 20-second postcuring group of Z100 (table 3) may have resulted in lower strength due to excessive brittleness from a not so well converted polymers, since the fabricant demand 40 seconds of cure. That is, the resin became fragile, without any mechanism for stretching its polymers. In this case, a polymerization time lesser that it is recommend by the manufacturer cannot be used. For example, one could decrease the time of light exposition hoping that the pos-cure process would provide a restorative material able to withstand mechanical loading. One composite showed another behavior. That is the case of IPS Empress Direct. Sixty seconds of post-cure method gave only 7.56 Mpa, statistically different from the conventional polymerization (16.59 Mpa). Heat and pressure excess may had degraded the polymer chain. A vitreous transition assay together with FTIR may provide more evidences of these results. Benalcázar Jalkh et al. ²¹ in the year 2019 for the Z350 resin found the same result. However, other works find higher values in the mechanical properties with the composite resin Z350 ²⁸. The present study verified that for the resin Z350 there was no improvement when increasing the exposure time and/or submitting the material to the post-cure process. The Palfigue resin showed the same behavior, but with the difference of always presenting lower values of biaxial resistance than the Z350, except in the condition of 60 seconds plus the post-cure effect.

Differences between materials can be explained by differences in organic and inorganic composition and the amount of them in each resin. The way in which the organic system captures light, its ability to convert monomers into polymers are factors that govern the effect of further polymerization. Parallel to the initial effects of the application of light, the behavior of the material after the application of post-curing polymerization methods is conducted by the ability of the residual monomers, those who did not participate in the polymer chains at the beginning of the cure, to react with each other, or with the already grown polymer chains.

Cross-linking is usually mediated by low molecular weight monomers, with smaller dimensions than other resinous materials and which manage to remain between two larger linear chains ³. TEGDMA - present only in the Z100 resin - is a type of monomer that is used in the resin for this purpose. A higher amount of TEGDMA determines a resinous material that has little effect on an additional curing process ²⁹. This result verified by the authors mentioned above partially contradicts what was observed in the present study; however, 20 seconds of

exposure to light probably generated a material with a higher amount of non-reacted monomers (TEGDMA among them). This mixture may had become insensitive to the application of heat and pressure thereafter.

After 60 seconds, a dense polymeric network is immediately formed for Z100 and the other materials. Despite this, only Z100 responded positively when post-cure method was applied, with a greater increase in the biaxial strength value gain compared to other brands of composite resins. The heat used on the postcure method perhaps cause some internal expansion so a residual monomer of Z100 could bond to a formed polymer. Differences in the organic composition and even the filler of the materials may have been the cause of this different response. Assuming this reasoning, heat and pressure could not produce an increase in biaxial resistance in this case. Nevertheless, this allegation needs to be carefully verified by other studies, especially those that are willing to analyze the chemical structure of composite resins.

Conclusions

Within the limits this study, it was concluded that the post-cure method promoted increase in biaxial flexural strength in only one composite resin (Z100), in 60 seconds.

In the conventional method, the composite resins in this study polymerized for 60 seconds showed no statistical difference in the biaxial flexural strength value, as compared to 20 seconds. Considering these aspects, there is no need to light-cure the composite resins exceeding the manufacturer's recommendation time. Except for the post-cure protocol with Z100 composite.

Acknowledgements

This study was only possible thanks to the entire structure offered by Pontifical Catholic University of Minas Gerais. The authors declare no conflicts of interest.

Declaration of Interest

The authors report no conflict of interest.

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