MÉTODO ALTERNATIVO DE FOTOPOLIMERIZAÇÃO DE RESINAS COMPOSTAS PARA TESTE DE FLEXÃO

Alternative method of photopolymerization of composite resin specimen for flexural strength test

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RESUMO

O teste de flexão é um dos mais utilizados em pesquisa de caracterização mecânica de compósitos resinosos para restauração dentária. A técnica ISO 4049 é feita em etapas com aplicação estática da luz. Para que toda a área do corpo de prova receba esta radiação é preciso que outras áreas recebam radiação extra. Na tentativa de sanar tal problema, esta pesquisa comparou o método da ISO com um método que utiliza um movimento constante (MC) durante a fotopolimerização. Foram produzidos 20 corpos de provas (CPs) com 25 x 2 x 2 mm, para cada grupo, sendo 10 polimerizados pela técnica estática recomendada pela normativa ISO-4049 e 10 polimerizados pela técnica de movimentação contínua (MC) para cada fotopolimerizador LED utilizado. Após análise estatística de Tukey apurou-se que não houve diferença estatística em relação à resistência à flexão (RF) e para o módulo de elasticidade em flexão. Porém, percebe-se, que o desvio padrão tanto da RF quanto do EF foram bem menores em MC do que em ISO. Sendo assim, a fotopolimerização em movimentação constante (MC) das amostras para o teste de resistência à flexão se mostrou como uma possível solução para o problema de sobreposição na técnica proposta pela ISO.

Palavras chaves: Resistência à Flexão, Módulo de Elasticidade, Resina Composta, Fotopolimerização.

ABSTRACT

The flexural resistence is one of the most used test in researchs of mechanical characterization of resin composites for dental restoration. ISO 4049 photopolymerization technique performed static application stages of light tip. To polymerize the entire area of the specimen some areas must receive extra radiation. These areas with extra radiation are called overlap areas. In an attempt to remedy this problem, this research compared the ISO method with a new method using constant movement (CM) during photopolymerization. Twenty specimens (SPs) were produced with 25 x 2 x 2 mm, for each group, 10 specimens being polymerized by the static technique recommended by the ISO-4049 and 10 specimens polymerized by the CM technique for each LED curing light used. After Tukey's statistical analysis, it was found that there was no statistical difference in relation to flexural strength (FS) and for the flexural modulus of elasticity (EF). However, the standard deviation of both FS and EF were much lower in MC than in ISO. Therefore, the photopolymerization by CM of the samples for the flexural strength test proved to be a possible solution to the problem of overlap of the technique proposed by ISO.

Keywords: Flexural Strength, Elastic Modulus, Composite Resin, Photopolymerization

INTRODUCTION

The flexural resistance is one of the most used tests in researchs of mechanical characterization of resin composites for dental fillings, due to the characteristics of flexural test where compression, traction and shear stresses are considered, as well as in the referred dental fillings.

Although this resistance is closely related to size, shape and amount of fillers of the composite (KIM, 2002; MUSANJE, 2004; HAHNEL, 2012, DIONYSOPOPULOS, 2016), three-point bending test is indicated by ISO (International Standards Organization) to evaluate resinous composites (RODRIGUES FILHO, 2006). The current technique standardized by ISO 4049 performed stages with static application of the light, moving the irradiating source in half the diameter of the active tip of the photopolymerizer, after the completion of each stage. Therefore, it is inevitable that to run the entire length of the specimen, some areas will receive extra polymerization. This “overpolymerization” of some areas can cause contraction, cracks and anisotropy, resulting in alteration of the microstructure, mechanical properties and great dispersion of the results, which can induce incorrect performance of the material in service (FERRACANE & MITCHEM, 1994; PICK et al., 2010; HAHNEL et al. 2012). On the other hands, the photopolymerization by a continuous movement method (application of light with constant speed) each part of the composite will receive the same energy density necessary for its polymerization, resulting in more stable values of flexural resistance of the materials. The polymerization process will occur much more homogeneously and consequently, with greater uniformity in the microstructure and properties. However, there is no way to apply this technique in daily clinic, and the reason to use the Continuous Movement Technique lies in production of more uniform samples for researchs in mechanical tests.

The objective of this research was to compare three points flexural resistance of a resin-based material photopolymerized by two methods, ISO 4049 and Constant Movement (CM).

METHODOLOGY

Specimens (SPs) with 25x2x2 mm were produced using a bipartite metallic matrix. For this, a composite resin widely found on the market was used. Before the photopolymerization a transparent polyester strip was placed on both of surfaces, upper and lower. During light curing a 0.1 mm thick glass plate was also placed to guarantee good flatness and the most polished surface possible, without decreasing the power light over the SPs.

Twenty SPs were produced for each group, 10 polymerized by the ISO-4049 static technique and 10 polymerized by Continuous Movement (CM) technique proposed by this research. Two LED photopolymerizers were used, totalling 40 specimens. The ISO technique recommends polymerization in parts, starting at the middle of the specimen and moving half the diameter of the active tip to each polymerization to each side until it polymerize the entire length of the CP, overlapping half the area of the guide tip at each light curing. The polymerization time in each step was 20s, as recommended by the manufacturer of the resin composite used.

The CM technique aims to avoid the overpolymerization that occurs in the ISO technique. The photopolymerizer was attached to a claw to keep the position fixed and stable. Once the surface of the guide tip was perfectly parallel to the sample surface, the movement speed of the INSTRON testing machine was calculated, so that each part of the sample received the same 20 s of light exposure recommended by the manufacturer (Equation 1).

$V = \Delta D / \Delta T$. (1)

Where:

V = INSTRON bar speed

ΔD = Diameter of guide tip

ΔT = Light curing time (20 s)

The speed used was 0.4 mm/s, because the displacement is equal to the diameter of the active tip of 8 mm and the time interval is 20 s of light curing. So, it was required about 60 s to light curing the entire specimen. However, it was considered that in the ISO technique the recommended overpolymerization doubles the time of exposure to light, therefore the MC technique would also have to double this time to 40 s. Thinking about the equivalence of the energy density emitted for each sample, it takes about 120 s to light cure the entire sample, in this case. However, the maximum operating time for LEDs 1 and 2 is 20 s. Thus, the sample was polymerized by the continuous movement (CM) technique for two times each surface, prioritizing the minimum of interruptions during light curing.

Once polymerized and removed from the matrix, the PCs were polished with 600 sandpaper. The three-point flexural test was performed at a speed of 1 mm/min. To measure the deformation of the samples, an LVDT (Linear Variable Displacement Transducer) was installed in contact with the lower face of the sample and in perfect alignment with the flexural teste device. Such device was produced especially for this test, ensuring the 20 mm space between the sample holders, recommended by ISO-4049.

The flexural strength calculation, σ, was done according to Equation 2:

$$\sigma = \frac{3FL}{bh^2} \quad (2)$$

Where:

F is the maximum load, in Newtons.

L is the distance in millimeters between the supports, 20 mm.

b is the width of the sample, 2 mm.

h is the height of the sample, 2 mm.

It was possible to calculate the flexural modulus due to the use of LVDT to accurately measure the strain of the sample, according to Equation 3:

$$E = \frac{F' \beta}{4bh^3 d} \quad (3)$$

Where:

F’ is the charge, in newtons.

d is the deflection, in millimeters, under load F1;

b and h are the same as the previous one.

Tukey's statistical analysis, with a 95% confidence level and confidence interval analysis was used to analyse the results.

After three points flexural test, some of the fractured samples were taken to SEM for fractographic analysis.

RESULTS

The results of three-point flexural strength (FS) and elasticity modulus (E) of composites polymerized by two photopolymerizers using the techniques of ISO and Continuous Movement (CM) are shown in Table 1.

Table 1 *Flexural strength (FS), elasticity modulus (E) and standard deviation values in brackets of RBCs polymerized by two photopolymerizers using ISO 4049 and continuous movement (CM) techniques.*

	GROUP I		GROUP II	
	FS (MPa)	E (GPa)	FS (MPa)	E (GPa)
ISSO	104,24	8,38	104,90	9,32
	(15,83)	(0,86)	(18,74)	(1,1)
CM	120,46	8,12	117,79	9,11
	(8,04)	(0,54)	(6,95)	(0,47)

After Tukey's statistical analysis, with a 95% confidence level, it was found that when the samples were polymerized by the static ISO technique and the CM technique, there was no difference for flexural strength (FS) as well as for elasticity modulus (E). However, the standard deviation of both the FS and E of groups I and II were much lower for CM than ISO. Such statements can also be seen in the graphs of figures 1 and 2. In the graphs of figures 3 and 4 where the diameter represents the amplitude of the standard deviation, we can see, even more easily, that the standard deviation of the group I is larger than the group II.

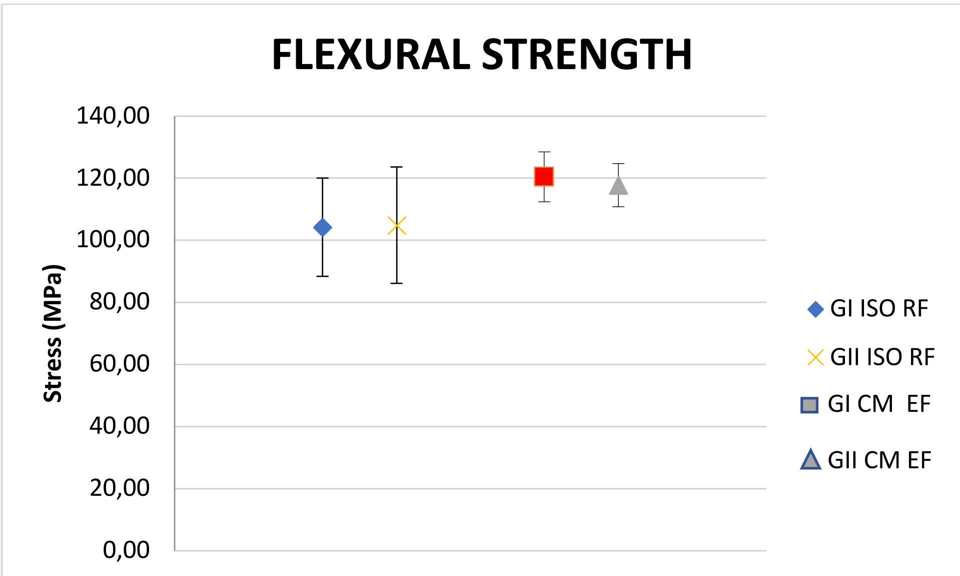


Figure 1*: Graph of flexural strengths of groups GI and GII polymerized by the techniques of ISO and MC.*

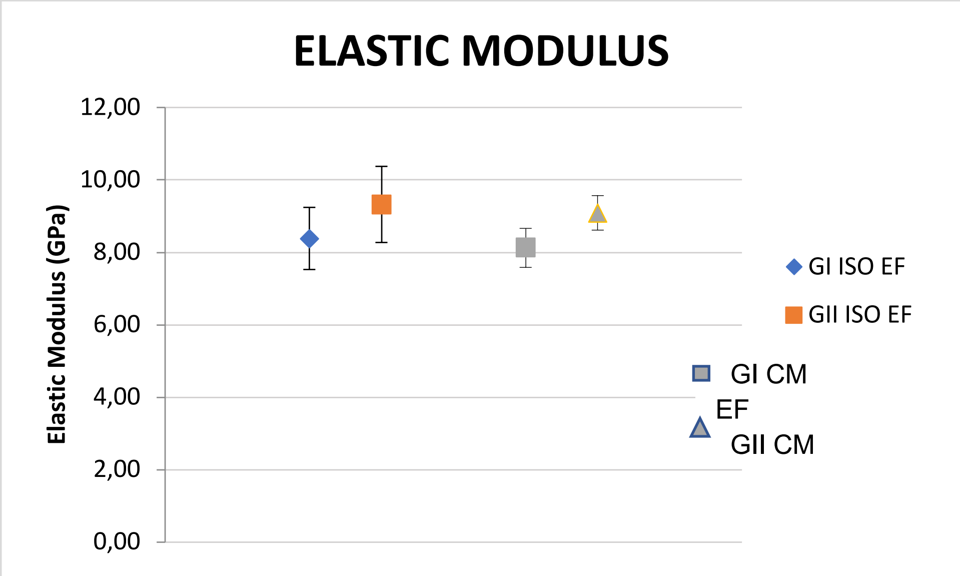


***Figure 2**: Graph of the elasticity modulus of groups GI and GII polymerized by the techniques of ISO and MC.*

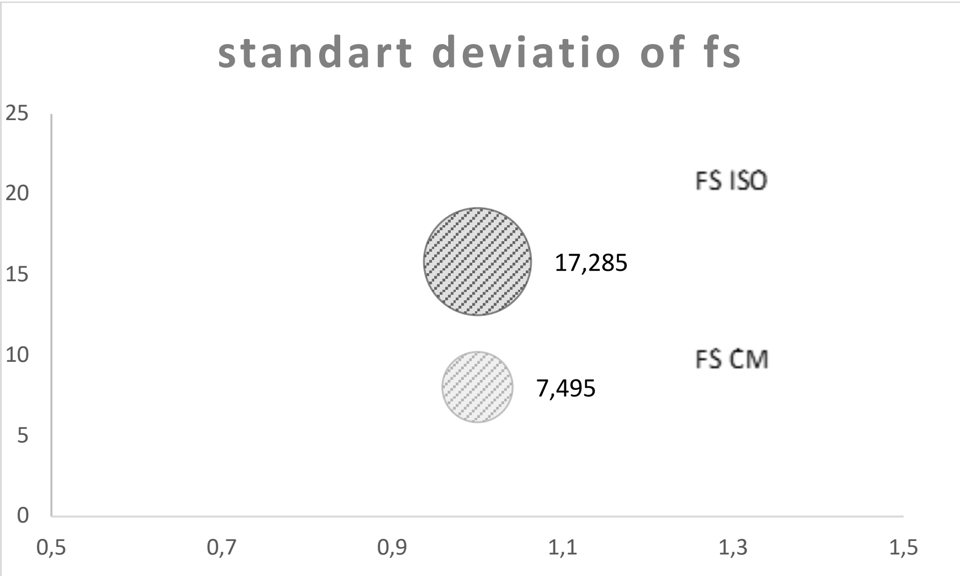


Figure 3: Graph of the standard deviations of the flexural strengths of the ISO and MC techniques.

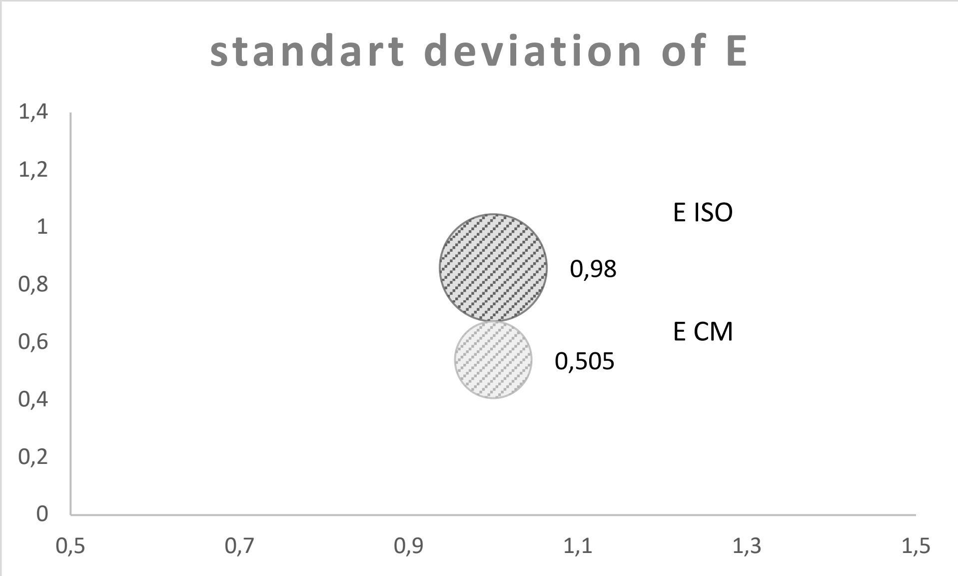
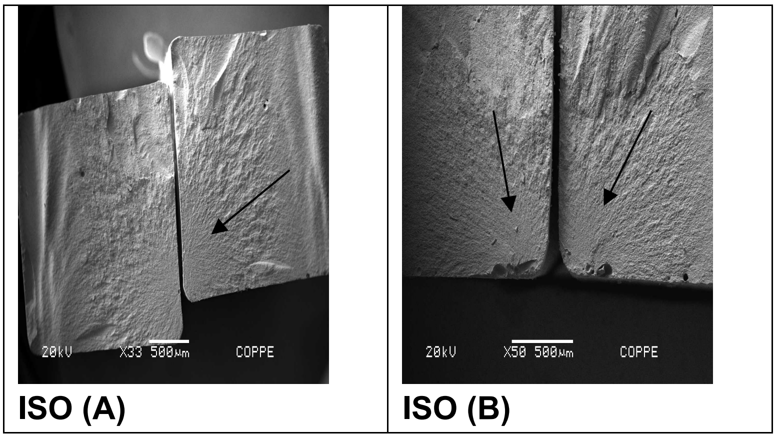
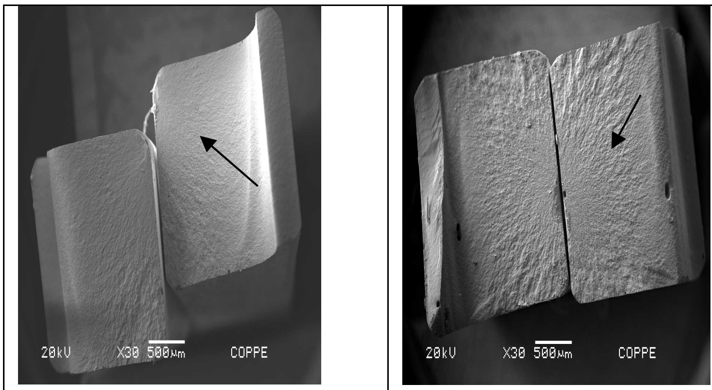


Figure 4: Graph of the standard deviations of the Elasticity Modules (E) of the ISO and CM techniques.

FRACTOGRAPHY

The analysis of fractured surfaces showed different patterns of fracture progression between the ISO and CM technique. Figure 5 shows mirrored areas and more defined Hackles lines for samples polymerized by ISO techniques than for samples polymerized by CM technique.





MC (A) MC (B)

Figure 5: Fractographies of samples polymerized by ISO and MC techniques. (A) Group I, (B) Group II. The arrows indicate the mirrored areas, followed by the Hackle lines.

DISCUSSION

First of all, it is necessary to point out that the method of continuous polymerization has not been found in the literature by the present author. This application in continued moviment, however, showed excellent homogeneity when the flexural strength test was analysed. Such homogeneity is considered of fundamental importance in the prediction of the clinical performance of composites, due to the compression and traction stresses on the specimen during the flexural test (YILMAZ et al., 2007). According to BELLI et al. (2014), such mechanical behavior is analogous to masticatory efforts. In addition, the flexural strength should not vary when the same material is polymerized by different methods (YILMAZ et al., 2007). In the case of this study the samples were polymerized by two LEDs with wavelengths compatible with camphorquinone (JANDT & MILLS, 2013), but with different average irradiance and light power distribution along the tip- guide, which can cause variations in flexural strength.

When the graphs in figures 1 and 2 were analysed, the GI and GII, cured by the CM method, obtained less standard deviation (Table 1), suggesting more homogeneity in the light-cured microstructure. Therefore, the CM method seems to polymerize the samples more homogeneously, being able to replace the static method with overpolymerization proposed by ISO-4049. Another alternative to avoid the irregular photopolymerization would be to use the biaxial test with a cylindrical sample. According to PICK (2010), in this type of test the shape of the specimen is better adjusted to the shape of the guide tip due to its cylindrical shape. So, the tension is made in the centre of the sample and defects located at the edges do not influence the results.

According to HAHNEL et al. (2012), the size of the specimen proposed by ISO-4049 for the three-point flexural strength test is easy to perform, however, the tensile stresses are concentrated on the lower surface, accentuating effects of the condition of this surface on the results of the test. This fact agrees with PICK (2010), who reported great influence of defects located on the surface of the specimen in the results. So, they concentrate tensions and reduce the flexural resistance.

The fractographic analysis in the specimens of this study revealed a material with some pores, where the fractures started on or near the surface being accompanied by a clear mirrored area followed by Hackle lines more in samples polymerized by the ISO technique than by the CM technique. Thomaídis (2013) and Hongyi Fan et al 2017 also found in their research.

According to the norm for fractographic analysis in ceramics, C 1322 - 05b (ASTM, 1984), well-defined mirrored areas followed by radial Hackle lines reveal a fine-grained microstructure. XXXXXX Distinct fracture mechanisms between the polymerized specimens using the ISO and CM technique (Figure 5). Within this same reasoning, the specimens in ISO, for having more Hackles lines, show a subcritical growth of slow crack characteristic of a plastic deformation, suggesting less polymerization in the inner layers (PICK et al, 2010; GHAVAMI-LAHIJI et al, 2018). This is in line with the greater irregularity of the data, due to its greater standard deviation, in the present study. This greater irregularity is directly related to the polymerization technique proposed by ISO.

CONCLUSION

- The irregularity in the distribution of luminous power throughout the area of the guide tip impairs the mechanical properties of polymerized composites.

- Photopolymerization in constant movement (CM) of the samples for the flexural strength test proved to be a possible solution to the problem of overlap in the technique proposed by ISO.

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