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Microhardness and Diametral Compression Strength of Single Fill Composite

Microdureza y Resistencia a la Compresión Diametral de Resina Compuesta Mono Incremental

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ABSTRACT: To evaluate Knoop microhardness and diametral compression strength at different polymerization times. A total of 80 cylindrical samples with Filtek resin™ One Bulk Fill were made to perform the *in vitro* study, using half of total samples (n=40) for each test and divided equally between groups (n=10). All samples were made according to ISO 4049 using 2 mm thickness Teflon matrix and a central orifice with 4 mm diameter. Four different times of light-curing in Standard mode suggested by the light-unit manufacturer were selected, therefore, group 1 samples (G1) were light-cured for 5 seconds; group 2 (G2) for 10 seconds; group 3 (G3) for 15 seconds, and group 4 (G4) for 20 seconds. A radiant display, which means a power in mW/cm^2 by time in seconds was expressed in Joules (J). The samples were stored in a dark container with distilled water at a temperature of 37°C for 48 hours before testing. One-way analysis of variance (ANOVA) with T-Test (LSD) of multiple comparisons of the mean values of Knoop hardness and diametral compression strength was performed, with significance index α=0.05. Regarding the Knoop microhardness test, G1 (35.73 ± 6.2) presented the lowest values, followed by G2, while G3 and G4 did not present statistical differences between them. For the diametral compression test, G1 (1387.76 ± 190.51) obtained the lowest value when compared to the other groups. G2, G3, and G4 did not present significant statistical differences. The different polymerization times influenced the Knoop microhardness and the diametral compression strength of Bulk Fill resin.

KEY WORDS: composite resins; hardness; stress, mechanical; physical properties.

INTRODUCTION

The development of light cured composite resins marked the modern era of restorative dentistry. The evolution in the mechanical properties of direct composites, as well as the improvement of light-curing devices provided the use of these materials with a wide range of treatments.

Restoration fractures are still constantly pointed out as causes of failures, even if these materials have undergone great evolution. Polymerization, the relationship of the resin with the quantity and size of particles are factors that are linked to such fractures, and can influence the compressive strength of the

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materials (Alkhudhairy & Vohra, 2016). The search for the improvement of these materials, or even to simplify their use points out those developments has still been a great challenge (Monterubbianesi *et al*., 2016).

Due to its mechanical properties, aesthetics, easy handling, short polymerization time, composite resin becomes the material of choice for restorations. The incremental technique requires a delicate and rigorous technique from the professional, as it requires increments of a maximum of 2 mm in the cavities of the dental elements (Son *et al*., 2017). However, they are materials that, even following a strict protocol, can cause failures in restorations due to possible "gaps" formed between the layers (Shibasaki *et al*., 2017).

Camphorquinone is a photo initiator that reacts with an amine reducing agent and upon reaching the specific wavelength of light it becomes activated, resulting in the formation of free radicals, in which the polymerization of the resin begins. Full polymerization is an important factor in the use of light cured composites, but it is still considered a challenge in clinical practice (Al Shaafi *et al*., 2011).

For better adaptation of the material into the cavity and reduction of the clinical time, Bulk Fill or single-fill resins were introduced to the market. Increments can be inserted uniquely into the cavity, up to 5 mm thick, which makes them an alternative to conventional restorative treatment (Tsujimoto *et al.,* 2017). In addition to simplifying procedures, due to the shorter clinical time, Bulk Fill resins provide better well-being for both patients and dentists (Tsujimoto *et al*.).

Single-fill resins propose a reduction of the polymerization shrinkage and a greater depth without the need to increase the irradiation time, and allows the filling of dental cavities in larger layers, from 4 mm to 5 mm thick (Pereira *et al*., 2018; Shimokawa *et al*., 2018). These composites can be light-cured in thicker layers with low polymerization shrinkage and good mechanical properties, because Bulk Fill resins are translucent (Fronza *et al*., 2017).

Several factors can influence the mechanical properties of composite resin (CR), among them: compressive strength, increment size, diametric stress resistance, microhardness, composition and even different polymerization times. Thus, this study evaluated the influence of different times on the polymerization of Bulk Fill resin in some of its physical properties.

MATERIAL AND METHOD

Study Design. This study was an experimental research, in which the factor under study was the light curing time (at four levels). Having as variable the response to Knoop microhardness, measured in Kilograms/force per square millimeter (Kgf/mm^2) and the diametral compression strength measured in Newtons (N), both of a quantitative nature as shown in Figure 1.

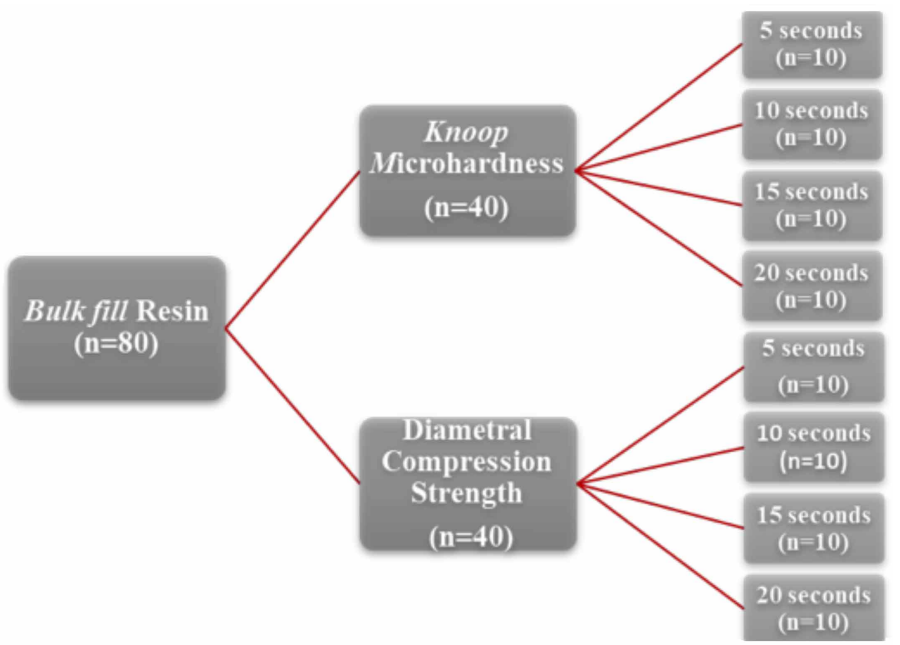

Fig. 1. Experimental design.

Sample Making. A total of 80 cylindrical samples with Filtek resin™ One Bulk Fill were prepared to perform this *in vitro* study (3M ESPE, St. Paul, MN, EUA) (Table I), using half (n=40) for each test performed and divided equally between groups (n=10). Teflon matrix of 2 mm thickness and a central orifice with 4 mm of diameter were used following the norms of ISO 4049 (International organization for standardization, 2009). All samples were standardized and checked with a thickness gauge (Golgran dental instruments, São Caetano do Sul, Brazil).

On a black background, a glass plate and a transparent polyester matrix (Fava, São Paulo, SP, Brazil) were placed, followed by the Teflon matrix and thus filled in a single increment with the aid of a composite resin spatula Suprafill 1 (Golgran dental instruments, São Caetano do Sul, Brazil). Afterwards, a new polyester matrix and glass slide were placed on the filled set. The entire set was maintained with digital pressure for 10 seconds for homogenization (Fig. 2) and flattening the surface of the Bulk Fill resin (Fig. 3).

Table I. Filtek Resin™ One Bulk Fill characteristics.

Material	Manufacurer	Composition	Inorganic Charge	Type	Lot
Filtek™ One Bulk Fill	3M ESPE, St. Paul, USA	Treated silanized ceramics, aromatic urethane dimetacrylate, dimetacrylate diurethane (UDMA), itebium fluoride (YbF3), silane treated silica, 1.12-Dodecandicrylate (DDDMA), treated silonium zirconia, water.	76.5% by weight 58.5% in volume.	Nanoparticulate	1824700218

Source: 3M ESPE.

Fig. 2. Homogenization of Bulk Fill resin.

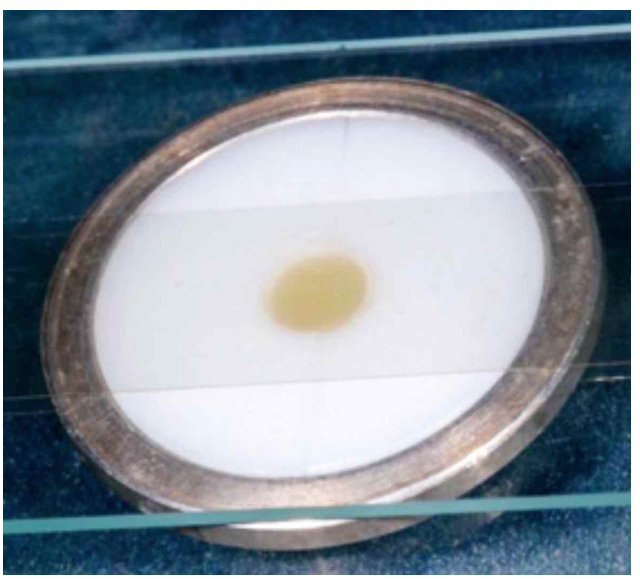

Fig. 3. Planning the resin increment on the teflon matrix.

The Valo diode (LED) photoactivator emitting light source (LED) (Ultradent, South Jordan, UT 84095, United States) with the 10 mm diameter circular shaped light outlet tip was used for the polymerization of all samples. The light-curing in Standard mode was used with irradiance energy of 1000mW/cm^2 which was suggested by the light-unit manufacturer (Fig. 4).

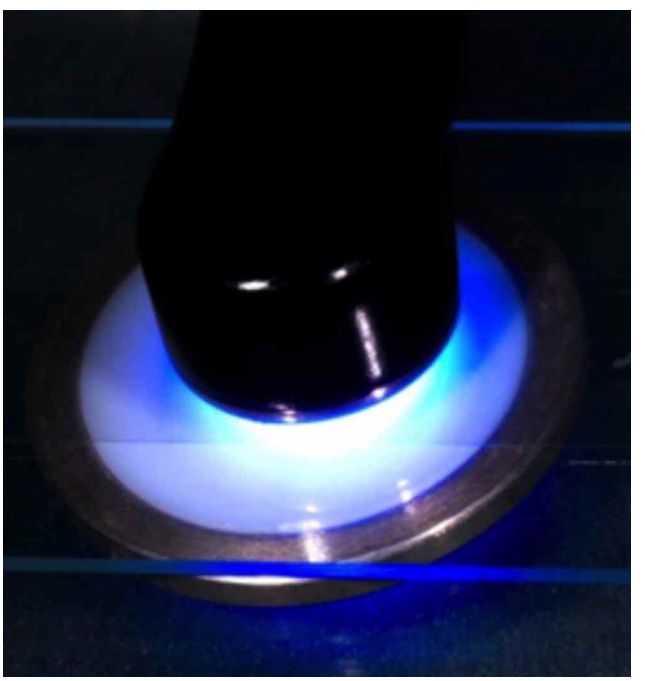

Fig. 4. Bulk Fill resin light curing.

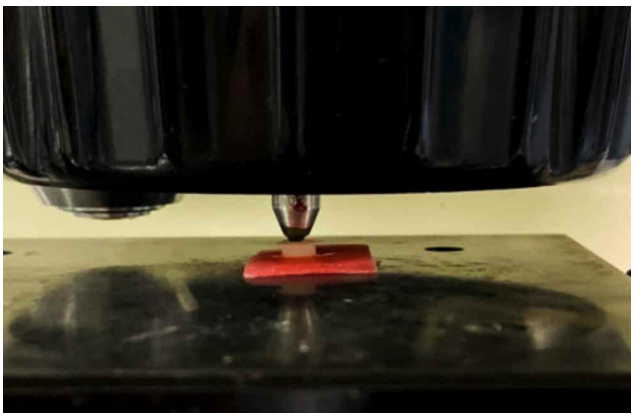

Fig. 5. Knoop microhardness test.

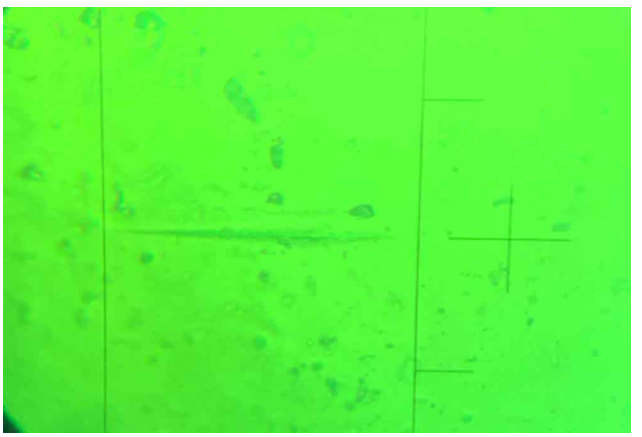

Fig. 6. Photomicrography of indentation.

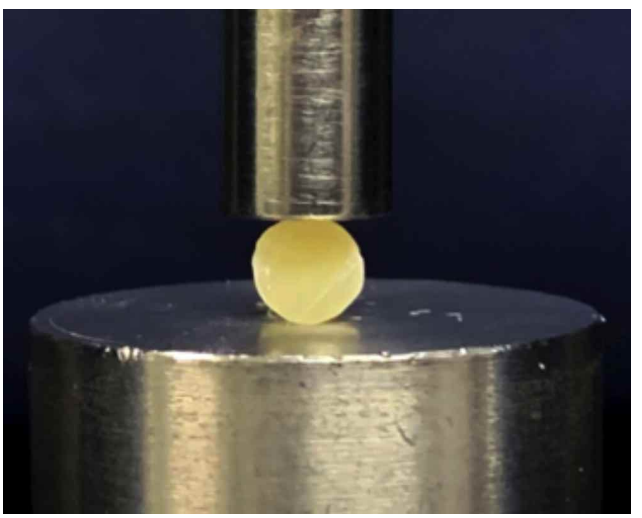

Fig. 7. Diametral Compression Test.

Hence, the samples from group 1 (G1) were photopolymerized for 5 seconds; group 2 (G2) for 10 seconds; group 3 (G3) for 15 seconds, and group 4 (G4) for 20 seconds. The radiant exposure, which means the power in mW / cm^2 times the time in seconds, was expressed in Joules (J). Soon after photo activation, the samples were stored in a dark container with distilled water in a laboratory oven at a temperature of 37 °C for 48 hours.

Microhardness Test. The Knoop hardness values were measured at the top in regions of the sample that did not present porosity/bubbles, using the HMV 2000 digital microdurometer device (Shimadzu, Tokyo, Japan) with 50 g load calibration applied during 10s (Fig. 5). Three indentation regions were demarcated, making a total of 10 measurements per group (Fig. 6).

The values obtained in micrometers were converted into the number of Knoop microhardness (KHN - Kgf / mm^2), by the device's software. For each sample, the average of the three indentations was obtained

Diametral Compression Tests. The test was performed using a Universal Testing Machine (EMIC DL2000, São José dos Pinhais, PR, Brazil), coupled to a 10 mm diameter rod with a flat end. The positioning of the specimens was in the center of the rod, which exerted diametral compression force at a speed of 1mm/minute towards the base of the device until the material failure (Fig. 7). The collected data recorded the maximum compression force in Newtons (N).

Statistical Analysis. The results were tabulated and analyzed using the Bioestat Software 5.3 (Federal University of Pará, Belém, Brazil) showing normal and homogeneous distribution. Variance analysis (ANOVA) of a criterion with T-Test (LSD) of multiple comparisons of the mean values of both Knoop microhardness and diametral compression strength with significance index α=0.05 was performed.

RESULTS

The results obtained are shown in Table II. The Knoop microhardness test, G1 (35.73 ± 6.2) presented the lowest values p<0.001, followed by G2, while G3 and G4 did not present statistical differences between them p> 0.05. For the diametral compression test, G1 (1387.76 ± 190.51) obtained the lowest value when compared to the other groups p<0.001. G2, G3, and G4 did not present significant statistical differences p>0.05.

Table II. Knoop microhardness and diametral compression test. Average ± standard deviation.

	Knoop microhardness test (Kgf/mm_)	Diametral compression test (N)
G1 5 J/cm^2	35,73 ± 6,2^C	1387,76 ± 190,51^B
G2 10 J/cm^2	49,66 ± 6,1^B	1552,51 ± 226,71^A
G3 15 J/cm^2	58,22 ± 6,4^A	1664,62 ± 164,11^A
G4 20 J/cm^2	63,68 ± 8,3^A	1709,59 ± 164,65^A

ANOVA of a criterion and T-Test (LSD). Different letters indicate statistical difference p>0.05.

DISCUSSION

With the development of single-increment resin, mechanical characteristics such as high chewing resistance, has been considered an objective for clinical practice in restorative treatments. For this research, the two null hypotheses were rejected, since the different polymerization times influenced both the Knoop microhardness and the diametral compression strength.

In a simplified way, the concepts of light sources on clinical results of restorations should be understood High-powered LED photopolymerizers significantly imply the complete polymerization of certain materials, so that there is no failure through the quality of polymers formed (Farahat *et al*., 2016; Alkhudhairy *et al*., 2017).

Factors such as translucency of the material, the type of resin, the exposure time, significantly interfere in the polymerization of composites. The degree of conversion of a material is measured indirectly through the microhardness test, by means of significant results on the depth of polymerization, both on the upper and lower surfaces of the samples (Abed *et al*., 2015; Fronza *et al*.; Gonçalves *et al*., 2018; Alrahlah, 2018). In this study, it can be observed that for the same material, the lowest radiant exposure (5J) obtained the lowest results, both for microhardness and diametral compression strength.

There was a change in the molecular base of Bulk Fill resins, which decreased or replaced Bis-GMA, moving to a monomer of lower viscosity, or to the use of monomers of greater molecular weight, such as Bisphenol-EMA (Bisphenol-A-glycidil metacrylate), TEGDMA (Dimetacrycrylate of triethylene glycol), EBPDMA (Ethyl 4-dimethyl aminobenzoate) and UDMA (Diuretano-dimethcrycrycrylate) (Lima *et al*., 2019; Rizzante *et al*., 2019; Tauböck *et al*., 2019). In this line of reasoning Zorzin *et al*. (2015) report that the association of Bis-GMA monomers with UDMA or TEGDMA will increase the degree of conversion, creating a network of highly rigid polymers, which consequently increase the polymerization of the resin at depths.

The incorporation of new photoinitiators influences the resin curing process, such as increasing its ability to transfer light at its depths (Gomes *et al*., 2018; Ilie, 2019). Filtek resin™ One Bulk Fill has unique monomers in its composition, additional fragmentation (AFM) and aromatic dimethacrylate urethane (AUDMA) (Bucuta & Ilie, 2014). Therefore, this explains why the resin does not present statistically significant differences in groups 2, 3, and 4 in the diametral compression test, as the monomers were introduced to reinforce the initiation, as it has satisfactory characteristics regarding the regularization of polymerization, and its ability to deeply polymerize single-fill materials.

Similar results were obtained in previous studies conducted by Farahat *et al*., where they evaluated two Bulk Fill resins at different polymerization times, 20 and 40 s, and observed that by increasing the polymerization time, they obtained a significantly higher result on the degree of conversion of the samples. However, the success of using Bulk Fill resins is linked to exposure time.

The statistical differences observed in the Knoop microhardness test, where Group 1 (35.73 ± 6.2) obtained lower results, which can be answered due to the polymerization being only at the top of the sample. Obtaining similarity in the study by Karacolak *et al*. (2018), which evaluated 11 resins, and all groups, radiant energy decreased with increasing thickness. And found no significant difference at the top of the samples.

In order to compare the mechanical properties and the degree of polymerization of Bulk Fill resins, Czasch & Ilie (2013) observed that there were no improvements in increasing the polymerization time from 20 to 40s in composites of 4 mm. This can be explained due to the characteristics of the material, as well as high-power light-curing devices positively interfere in the polymerization process of composites.

This study obtained statistically significant results in the Knoop microhardness test, since the polymerization of the material occurred only at the top, due to the samples having a thickness of 2mm, with no need for polymerization at the bottom, since the light from the high-power photopolymerizer would penetrate to the bottom of the samples. In contrast, Son *et al*. state that polymerization at the top in Knoop microhardness measurements did not have different results, but when different thicknesses of the samples were compared at the bottom 2, 4 and 6 mm they obtained different results.

Another factor considered critical in relation to the polymerization of Bulk Fill resins, is the radiant energy fully received by there (J/cm∑), because the low exposure to photons will result in a low polymerization. This joint polymerization at the bottom and top of the restorations becomes more important (Van Ende *et al*., 2017; Karacolak *et al*.; Romão *et al*., 2018; Peixoto *et al*., 2019; Veloso *et al*., 2019). Contrary to Knoop microhardness results of this study, in which it occurred only at the top of the samples, because the reduction in stages of the hardness in the resin matrix body also decreases the degree of conversion of the composites.

Corroborating with obtained results, Lima *et al*. (2018) analyzed the polymerization depth of Bulk Fill resins, where the radiant energy of 20 J obtained greater similarity and satisfactory results in the analyzed studies. That is, the total energy dose is influenced by the irradiance offered.

It is evident, therefore, that despite having monomers that favor the degree of conversion of Bulk Fill resins, insufficient amounts of irradiating energy can negatively influence some physical properties of these composites. Given the technical limitations for performing this work, the measurement of microhardness in the bottom of samples when at depths greater than 2 mm, can be suggest for future studies.

CONCLUSION

It can be concluded from this study that: The different polymerization times influenced the Knoop microhardness of the tested Bulk Fill resin. In addition, the different polymerization times influenced the diametral compression strength.

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RESUMEN: El objetivo de este trabajo fue evaluar la microdureza Knoop y la resistencia a la compresión diametral en diferentes tiempos de polimerización. En el estudio realizado *in vitro*, se analizó un total de 80 muestras cilíndricas con la resina Filtek™ One Bulk Fill, utilizando la mitad (n=40) para cada prueba realizada y dividida en partes iguales entre los grupos (n=10). Todas las muestras se tomaron de acuerdo con ISO 4049 utilizando matrices de teflón de 2 mm de espesor y orificio central con 4 mm de diámetro. Se seleccionó cuatro veces el fotocurado de manera estándar, sugerido por el fabricante. Por lo tanto, las muestras del grupo 1 (G1) se fotopolimerizaron durante 5 segundos; grupo 2 (G2) durante 10 segundos; grupo 3 (G3) durante 15 segundos y grupo 4 (G4) durante 20 segundos. La exposición radiante, que indica la potencia en mW / cm^2 a lo largo del tiempo en segundos, se expresó en julios (J). Las muestras se almacenaron en un recipiente oscuro con agua destilada en una estufa a una temperatura de 37°C durante 48 horas antes del ensayo. Se realizó para comparaciones múltiples de los valores medios, análisis de varianza (ANOVA) de un criterio con la prueba T (LSD) tanto de la dureza de Knoop como de la compresión diametral con un índice de significación α=0.05. Con respecto a la prueba de microdureza de Knoop, G1 (35,73 ± 6,2) tuvo los valores más bajos, seguido de G2, mientras que G3 y G4 no mostraron diferencias estadísticas entre ellos. Para la prueba de compresión diametral, G1 (1387.76 ± 190.51) obtuvo el valor más bajo en comparación con los otros grupos. G2, G3 y G4 no presentaron diferencias estadísticamente significativas. Los diferentes tiempos de polimerización influyeron en la microdureza de Knoop y la resistencia a la compresión diametral de la resina compuesta mono incremental.

PALABRAS CLAVE: resinas compuestas, dureza, estrés mecánico, propiedades físicas.

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