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LED LIGHT EFFECT ON KNOOP MICROHARDNESS OF CONVENTIONAL COMPOSITE RESINS AND BULK FILL

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ABSTRACT

Objective: Evaluate the Knoop microhardness (KHN) of conventional and bulk fill composites light cured in different layers. **Materials and Methods:** Eighty specimens were prepared, of which forty in 8x2mm thickness and forty in 8x4mm thickness, according to the resins tested: Opus Bulk Fill (FGM); Filtek Bulk Fill (3M); Filtek Z350 XT (3M); Opallis (FGM). The material was prepared according to ISO 4049.The photoactivation was performed with the LED device for 40s. The specimens obtained were submitted to the Knoop microhardness test. **Results:** Thus, the values obtained were submitted to Kruskall Wallis test, followed by Dunn test, $p < 0.05$. When evaluating the top and bottom surfaces, the top surfaces showed statistically superior results to the bottom surfaces in all resins. From the micro hardness of the 8x2mm samples evaluated, only Filtek Bulk Fill and Filtek Z350 XT resin had acceptable top / bottom ratio (≥0.8). Of the 8x4mm specimen only the validated bulk fill resins presented this acceptable microhardness ratio and were statistically similar to the 8x2mm base. **Conclusion:** It was observed that, according to the methodology used, only the bulk fill composite resins achieved an acceptable polymerization and microhardness at 4mm depth.

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INTRODUCTION

Composite resins have become the first choice for direct aesthetic restorations (Mm *et al.*, 2016). Historically, composite resins have been light cured in 2mm thick increments, but recently, manufacturers have introduced resin-based bulk-fill composites, and it has been claimed that they can fill cavities up to 4–6 mm at once to reduce the length of the clinical procedure. There is evidence that underpolymerized composite resins are responsible for restoration failure due to the increased risk of fracture, secondary caries or excessive wear (Prince *et al.,* 2015). Moreover, when the composite resin is not satisfactorily polymerized, it becomes more prone to release greater amounts of chemicals in the body (Durner *et al.,* 2012). Arbitrary increase in light exposure times in an effort to avoid underpolymerization can damage pulp and surrounding tissues, as light curing increases the temperature of the tooth and surrounding oral tissues (Gomes *et al.,* 2013).

Thus, dentists and light curing manufacturers need the knowledge to provide light that can polymerize composite resins at a clinically relevant exposure time and thickness. Two common methods for determining the degree of polymerization of a resin are: degree of conversion using infrared radiation spectroscopy (Finan *et al.,* 2013) and microhardness tests (Tarle *et al.,* 2015). Most publications report a good correlation between the degree of conversion and microhardness tests (Erickson *et al.,* 2014; Mm *et al.*, 2016). The Knoop microhardness test is a relatively simple test that provides an accurate reproducible assessment of the degree of polymerization of the composite resin and a linear relationship has been shown between Knoop microhardness, Young's modulus and the viscosity of the composite resin (Li *et al.,* 2009). Thus, changes in Knoop microhardness measurements can be used as an efficient method to evaluate polymerization characteristics (Erickson *et al.,* 2014). The Knoop microhardness test uses a low intensity load on a

rhombohedral-shaped diamondindenter with a larger and smaller diagonal. When the Knoop indenter is removed from the test material, elastic recovery (dimensional change) occurs mainly on the smaller diagonal, leaving the longest diagonal virtually unchanged (Mm *et al*., 2016). In addition, the narrow width of the Knoop indentator tip allows indentations to be arranged closer to each other or closer to the sample edge than when the Vickers indenter is used (Shahdad *et al.*, 2007). Bulk fill resins have recently been introduced allowing larger increments to be adequately polymerized in only one light exposure, thus reducing the insertion time (Bucuta&Ilie, 2014; Ilie *et al.,* 2013). Some researchers report that although there were significant differences between conventional and bulk fill composites, it could be recommended to polymerize them in 4 mm increments (Ilie *et al.,* 2013). Others have warned that some study protocols may overestimate the resin polymerization depth determined by hardness tests (Flury *et al.,* 2012) and there has been concern that bulk fill resins may not be adequately polymerized to a depth of 4 mm (Jang *et al.,* 2015).

Objective

The aim of this study was to evaluate the Knoop microhardness (KHN) of conventional and bulk fill composite resins light-cured in different thicknesses. The null hypothesis tested was that there is no significant difference between Knoop microhardness values in two bulk fill resin thicknesses when exposed to high power wide spectrum LED light for 40s.

MATERIALS AND METHODS

Eighty specimens were made for this study: forty with 8 mm in diameter and 2 mm in thickness, and another forty with 8 mm in diameter and 4 mm in thickness. They were divided into 8 experimental groups (n = 10) according to the materials and thicknesses tested, denominated:

- Group 1 (n = 10): FILTEK BULK FILL composite resin, 3M, 8x2mm;
- Group 2 (n = 10): OPUS BULK FILL composite resin, FGM, 8x2mm;
- Group 3 (n = 10): FILTEK BULK FILL composite resin, 3M, 8x4mm;
- Group 4 (n = 10): OPUS BULK FILL composite resin, FGM, 8x4mm;
- Group 5 (n = 10): FILTEK Z350 XT composite resin, 3M, 8x2mm;
- Group 6 (n = 10): OPALLIS composite resin, FGM, 8x2mm;
- Group 7 (n = 10): FILTEK Z350 XT composite resin, 3M, 8x4mm;
- Group 8 (n = 10): OPALLIS composite resin, FGM, 8x4mm;

On a glass coverslip a polyester strip was placed and, over this, the matrix made from addition silicon (Adsil Putty Soft - Coltene) for insertion of the composite resin. Prior to the placement of the matrix, a graphite marking was performed on the surface to represent the base of the specimens. Then, the internal cavity of the matrix was filled with the previously selected restorative material, in color A2, in a single increment using a non-stick dental composite spatula. To standardize the top surface of the composite resin cylinder with the base, a second polyester strip was placed over the matrix. Then a glass coverslip was also placed to better accommodate the composite resin and to obtain flatter surfaces. The curing light (KAVO WIRELESS, 1100mW / cm² and 420-480 nm, Australia) was activated for 40s through the glass cover by touching the tip of thelight curing apparatus on it. After photoactivation was completed, the glass cover slip and polyester strip were removed and the specimens were stored in distilled water. They were kept in black plastic vials (photographic film packaging) for 24 hours in an oven (STERILIFER SX 1.A, Diadema - São Paulo, Brazil) at 37°C and therefore free of light until Knoop microhardness test. Hardness measurements were obtained after 24 hours from the specimens obtained in a Future Tech microdurometer (MICROHARDNESSTESTER, Future Tech FM-800, Future Tech Corp., Tokyo 140, Japan) for Knoop hardness evaluation. Knoop hardness was measured on the base and top surfaces at five equidistant points, totaling ten penetrations per specimen, using a load of 10 grams for 20 seconds. After each indentation, the larger diagonal referring to the rhombus (indentation) was measured with the help of the vertical bars present in the microdurometer display (Figure 1).

Figure 1. (a) Matrix made from addition silicone. (b) Light curing according to ISO 4049, 2009. (c) Proof bodies. (d) Future Tech brand microdurometer

An average of the five indentations for each surface was made and transformed into Knoop Hardness Number (KHN). The mean Knoop hardness values for each group were calculated and the results tabulated and subjected to statistical analysis.

RESULTS

The results were initially submitted to the normality curve adherence test (Shapiro - Wilk test) with negative result. Thus, the selected values were submitted to Kruskall Wallis test, followed by Dunn test, $p < 0.05$. The values obtained as well as the statistical results for the evaluated experimental groups can be observed in Table 1. When evaluating the top and bottom surfaces, the top surfaces showed statistically superior results to the base surfaces in all resins evaluated. From the micro hardness of the 8x4mm samples evaluated, only the bulk fill

Table 1. Means and standard deviation of surface microhardness (Knoop) values of experimental groups for top and bottom surface

Composite Resin	Surface		
	Top	Base 8x2mm	Base 8x4mm
Filtek Bulk Fill	42.4545 ± 5.1864^{Aa}	37.4300 ± 3.4644^{Ab}	37.6480 ± 3.7808^{Ab}
Opus Bulk Fill	37.2850 ± 5.7267^{Ba}	25.2460 ± 3.4094^{Bb}	31.0320 ± 3.6678^{Bb}
Filtek Z350 XT	47.8590 ± 0.3995^{Aa}	39.6760 ± 8.2720^{Ab}	26.4780 ± 4.1030^{Bc}
Opallis	30.1120 ± 4.2652^{Ca}	20.7260 ± 4.0843^{Bb}	14.2560 ± 2.9954^{Cb}

In each column, different capital letters mean statistically significant difference; on each line different lower case letters signify a statistically significant difference ($p < 0.05$).

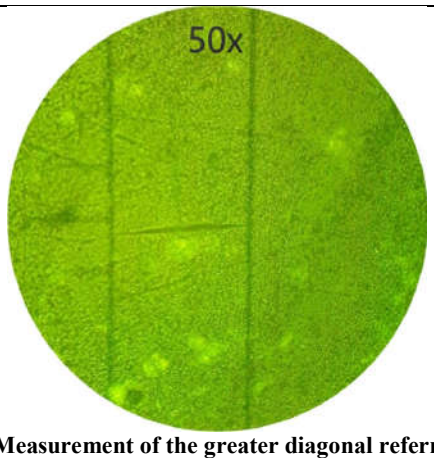

Figure 2. Measurement of the greater diagonal referring to the diamond (indentation), with the aid of the vertical bars present in the microdurometer display

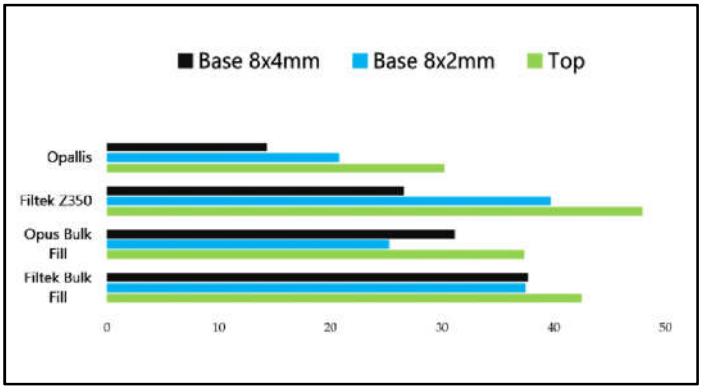

Figure 3. Mean surface hardness (knoop) values

Table 2. Composition and classification of resins used in the study

Composite Resin	Inorganic Particle Size	norganic content % by weight)	Organic Content
Filtek Bulk Fill (3M ESPE Dental Products, St. Paul, USA)	Nanohybrid	Agglomerated and non-agglomerated particles of: 20 nm silica, 4 to 11 nm zirconia, and agglomerated particles: ytterbium trifluoride of 100 nm - 76.5%	Bis-GMA, BISEMA, UDMA, TEGDMA, Procrylat Resins
Opus Bulk Fill (FGM, Santa Catarina, Brasil)	Nanohybrid	Silanized silicon dioxide (silica), stabilizers and pigments - 79%	Monômerosuretanodimetacrilatos, estabilizantes
Filtek Z350 XT (3M ESPE Dental Products, St. Paul, USA)	Nanoparticulate	Primary silica (non-agglomerated with 20nm average size) and zirconia agglomerateand silica with particles ranging from 5 to 20nm, forming clusters from 0.6 to 1.4 μm - 78.5%	Matriz de Bi-GMA, Bis-EMA, TEGDMA
Opallis (FGM, Santa Catarina, Brasil)	Microhybrid	Silanized Barium-Aluminum silicate glass, pigments and silicas, at 79%	Bis-GMA, BisEMA, TEGDMA

resins presented acceptable top/base ratio (≥0.8) and statistically similar to the base of the 8x2mm samples. About the conventional resins, none reached acceptable microhardness at the base of the 8x4mm specimens and only Filtek Z350 XT demonstrated acceptable microhardness at the base of the 8x2mm samples.

DISCUSSION

Properties such as polymerization degree, absorption, solubility or hardness are interdependent. These properties depend on the nature of the monomer, the degree of conversion, the type, the morphology and the percentage of filler particles, which strongly affect the behavior of restorations (Kusgoz*et al.,* 2011), being important parameters to characterize materials such as composite resins (Janda*et al.,* 2006). The major disadvantages of conventional composite resins are the stresses that occur as a result of polymerization shrinkage and the polymerization depth limited to approximately 2mm. To overcome these problems, it is recommended to use oblique incremental techniques for insertion of composite resin with 2mm thick layers (Kusgoz*et al.,* 2011).

In conventional composite resins, the depth of polymerization is limited due to light attenuation, which leads to light reflection from the surface of the material, dispersion by filler particles and absorption by photoinitiators. Faced with these limitations, bulk fill resins have emerged from the need to reduce the clinical working time for direct composite resin restorations and by accepting the application of 4mm thick layers while maintaining a satisfactory degree of conversion and reducing the polymerization shrinkage (Par *et al.,* 2015). Bulk fill resins generally include small amounts of fillers material to reduce light scattering, as light penetration is closely related to theamount of particles present (Ilie*et al.,* 2013). They have modifications in their translucency/opacity to allow adequate conversion of monomers to polymers, even with the insertion of 4mm increments and the addition of photoinitiators with greater light absorption (Junior *et al.,* 2014). However, the polymerization depth as established by the ISO 4049 method seems to be overestimated for bulk fill composites. For this reason, it is recommended to use specific microhardness measures to determine the polymerization depth (Flury *et al.,* 2012). In addition, microhardness data for a specific material provides information on its wear, polishability and abrasive effect on antagonist teeth (Marovic

et al., 2013). Of all tests performed separately or in combination, the microhardness of the Knoop test proved to be the best predictor of conversion (Rueggeberg & Craig *et al.,* 1998). In addition, it is the most commonly indicated method for evaluating polymeric materials, such as composite resins, as it minimizes the elastic recovery effect often observed in these materials because, in this type of impression, elastic recovery would mostly affect the smaller diagonal. and consequently less influence the results obtained. For this reason, currently the Knoop tip is the most used when studying these materials (Shahdad *et al.,* 2007). However, Pearson's correlation test showed a positive correlation between Vickers and Knoop hardness number (Moore *et al.,* 2008). Acceptable polymerization depth is reached when the top / bottom microhardness ratio is greater than 0.8 (80%) (Moore *et al.,* 2008). In our study, Opallis resin showed no acceptable polymerization in either base. Filtek Z350 XT resin had acceptable polymerization only on the 2mm base, and only Filtek Bulk Fill (3M) resin met this requirement on all bases (2mm and 4mm). However, both Filtek Bulk Fill (3M) resin and Opus Bulk Fill (FGM) resin did not present statistically significant differences between the 2 and 4 mm bases and both were clinically acceptable at depths up to 4 mm, corroborating previous studies that stated that larger variations were not observed in the surface values at 2 or 4mm (Tarle *et al*., 2015) (Bucuta & Ilie, 2014) (Jang *et al.,* 2015) (Par *et al.,* 2015) (Kelic*et al*., 2016) (Walter, 2013) (Van Ende *et al.,*2013) (Li *et al.,*2015) (Karacolak *et al.,*2017).

However, studies state that the use of large amounts of this resin resulted in lower hardness values in the cervical surfaces of class II restorations (Poskus*et al.,*2004), with microhardness levels lower than the upper surface in all specimens, regardless the light curing technique used (Ilie*et al.,*2013). This difference can be explained by different methodologies used. There are studies in which, without polishing, the hardness test was performed on the outer resin layer of the specimen and obtained lower microhardness values (Li *et al.,*2009) (Ilie*et al.,*2013), but when it was decided to perform the wear and polish of the samples of the superficial layer satisfactory microhardness results were obtained (Kelic*et al*., 2016). Moreover, when applying bulk fill resins to deep cavities it is useful to increase the irradiation time period or to use a high power light source to provide sufficient energy to the lower layers of the restoration (Michaud *et al*., 2014) as was the case with this research. As for the difference in microhardness values between materials, these can be attributed to differences in inorganic filler (Table 2), since the type and size of filler particles used in composite resin is one of the most important factors affecting light penetration through the material. This is due to the difference in refractive index, which is responsible for light scattering at the interface of the organic matrix and charge particle, commonly called the gap region. (Bucuta & Ilie, 2014). In addition, each bulk fill resin adopts different strategies to achieve high light transmission, which may be the use of specific polymerization modulators, the use of silane-coated filler content, improved translucency or the use of more powerful initiator systems (Ferracane, 2011).

It was also observed that the 40s light curing time in both bulk fill resins was satisfactory for the restoration to achieve greater microhardness. However, there is still no consensus on the absolute energy value required to obtain optimal polymerization for each bulk fill composite resin, as this value depends on the translucency, type and shade of the composite resin, as well as the type of photoinitiator (Shortall *et al*., 2008), long-term clinical studies are recommended to evaluate the performance of these materials.

Conclusions

Within the limitations of this in vitro study, it can be concluded that:

- Only the bulk fill composite resins evaluated in this study have an acceptable microhardness at 4mm depth;
- Not all conventional composite resins have acceptable polymerization at 2mm depth.

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