Mechanical properties of nanofilled and microhybrid composites cured by different light polymerization modes

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This study compared nanofilled and microhybrid composites polymerized by different light polymerization modes, and the effects these modes had on the mechanical properties of the composites. The following mechanical properties were measured: Vickers microhardness numbers, diametral tensile strength, flexural strength, and flexural modulus. The filler content (Wt%) present in the resins was investigated. Data were treated statistically by ANOVA and Tukey’s test (P ≤ 0.05) and the interaction of the inorganic content with the mechanical properties was analyzed by Pearson’s correlation (P ≤ 0.05). The nanofilled material showed a higher percentage of inorganic filler and better mechanical properties when compared to the microhybrid composite. The correlation of Wt% with the tested properties was positive for all but the flexural strength test. Different light polymerization modes were shown not to have a significant influence on the mechanical properties of the composites in this study.

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esin-based composite (RBC) fillers display either a spheroidal or an irregular morphology, which influences the filler content, orientation, and distribution within the resin matrix. The addition of nanosized or spherical particles in RBC systems enables the highest filler content due to the incorporation of free nanosized particles and clusters. As a result, a nanofilled composite acts as a single unit conferring greater resistance with improved physical and mechanical properties. Nanoclusters can provide damage tolerance and can modify the inherent failure mechanisms found in RBC systems, such as fractures and wear, by promoting a greater reinforcement in the resin matrix. Nanoclusters are useful for improving wear resistance and for reducing the possibility of fracture because the thickness and arrangement of the silane agents around and within the interstices of a nanocluster’s structure fills voids and gaps commonly found in resins with irregular particles. However, nanoclusters do exhibit a greater tendency toward multiple fractures compared to conventional fillers and exhibit a comparatively higher variability of pseudo-modulus and load prior to and at fracture, which may modify the damage tolerance of the overall RBC system. The size, morphology, and constituents of filler particles employed in dental RBCs have been shown to influence the properties of the resultant material.

According to the manufacturer, Filtek Z350 contains a combination of nanoparticles, including non-agglomerated silica of 20 nm and zirconia/silica nanoclusters (freely-linked), consisting of clusters of primary particles of zirconia/silica between 5-20 nm. The size of the agglomerated particles varies from 0.6 to 1.4 μ and the percentage of filler is 78.5% by weight.

Several factors can affect the mechanical properties of a composite resin, such as chemical composition, light source, amount of emitted radiation distance from the tip of the light source, and photoactivation mode. An increase in nanocomposite inorganic filler can also influence the mechanical properties of an RBC, such as flexural strength, flexural modulus, compressive strength, and microhardness.

Several protocols have been developed with different light curing units, promoting one or more activation sequences with a soft start. Variations on this technique include the progressive activation (when the light intensity is increased gradually), which can be step-by-step (soft start), linear or exponential (ramp) and pulse delay activation, which is performed with pulses separated by a time interval. The aim of this study was to perform a comparative analysis of Vickers microhardness numbers (VHNs), diametral tensile strength (DTS), flexural strength (FS), and flexural modulus (FM) of a nanoparticle composite versus a microhybrid resin using different photo activation modes. Additionally, the filler content of the composites was identified and correlated with the mechanical properties.

Materials and methods

Two resin composites, selected in accordance with their type of filler particles, were tested: a microhybrid (Charisma, Heraeus Kulzer) and a nanofilled composite (Filtek Z350, 3M ESPE) both in shade A3. Compositions and manufacturers are described in Table 1. The composites were light activated with an Ultralux (Dabi Atlante) with an intensity of 400 mW/cm² and with an Elipar Freelight 2 (3M ESPE) with an intensity of 900 mW/cm².

The intensity of the curing light bulbs—Demetron LC Curing Light (Kerr Corporation) and Demetron LED Radiometer (Kerr Corporation)—was measured before making the samples from each experimental group. The light polymerization modes used were as follows: conventional (QTH) continuous exposure to 400 mW/cm² for 40 s; conventional (LED) continuous exposure to 900 mW/cm² for 20 s; and exponential (LED) exposure for 5 s, followed by exposure to 900mW/cm² for 15 s.
Table 1. RBCs used in study.

<table>
<thead>
<tr>
<th>Product</th>
<th>Composition</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filtek Z350</td>
<td>Primary particle size of 5-20 nm and non-agglomerated 20 nm silica filler. Monomer matrix-GMA, Bis-EMA, UDMA, TEGDMA</td>
<td>3M ESPE</td>
</tr>
<tr>
<td>(A3 shade)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Charisma</td>
<td>Particle size of 0.2-0.07 µm. Average particle size of 0.7 µm Barium glass, aluminum fluoride. Monomer matrix: Bis-GMA, TEGDMA</td>
<td>Heraeus Kulzer</td>
</tr>
<tr>
<td>(A3 shade)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Microhardness**

The VHN specimens (n = 5) were made using a teflon split mold (8 mm diameter, 2 mm height). The composites were inserted in a single increment and were covered with a polyester strip and a glass slide to promote a flat surface. The specimens were photoactivated and stored at 37°C for 24 hours, followed by grinding in a polishing machine (DPU-10, Struers, Inc.) with 1200 grit SiC paper. Three VHN measurements were taken with a hardness tester (HMV-2, Shimadzu Corporation) using a load of 0.98 N for 30 s on the top and bottom of each sample.

**Diametral tensile strength**

The DTS specimens (n = 10) were shaped and stored in the same way as the VHN samples. Compressive tests to evaluate the diametral tensile were performed in a universal testing machine (DL 500, EMIC) with a load cell of 500 KgF at a crosshead speed of 0.05 mm/min. After each compressive test, the fracture load (F), in newtons (N), was recorded and the DTS (σ) (in MPa) was calculated as follows: \( \sigma = \frac{2F}{\pi dh} \), \( d = \) diameter (6 mm), \( h = \) height (2 mm) and \( \pi = 3.1416 \).

**Flexural strength and flexural modulus**

The FS and FM specimens (n = 10) were evaluated according to ISO 4049.19 Samples of 2 x 2 x 25 mm were prepared using a teflon split mold. The resin composite was covered with a polyester strip and glass slide. The tip of the light guide was placed over the center of the mold and the curing light was activated according to the group analyzed. Subsequently, the adjacent sections on either side were irradiated until the full length of the sample was polymerized. After irradiation, the specimens were removed from the molds and stored in water at 37°C for 24 hours. The samples were positioned in a 3-point bending apparatus on 2 parallel supports separated by 20 mm and loaded until fracture with a 500 KgF load cell at a crosshead speed of 0.05 mm/min in a DL 500.

Microhybrid resin, regardless of the light polymerization mode employed.

**Filler content**

The methodology used for experiments measuring the true content of filler was performed according to the American Society for Testing and Materials (ASTM) standard test measurement for rubber (13 ASTM D 297). Samples of each material were subjected to combustion under an oxygen atmosphere to eliminate the organic matrix. A precision balance (BG 1000, Gehaka) was used to weigh 2 g (W0) of each material, which was placed in a porcelain crucible and inserted into a muffle furnace (Q318S, Quimis) at 650°C for 3 hours. After that time, the porcelain crucible was removed from the oven and cooled in a desiccator for 90 min. Subsequently, the samples were re-weighed (W1) and the weight of the filler fraction was determined using the following formula: \( W_{\%} = \frac{W1}{W0} \times 100 \).

**Statistical analysis**

For all variables studied, the normality was checked by a Lillifors test, and the results were then analyzed statistically using 1-way analysis of variance (ANOVA). All variables showed homogeneity and normality. The parametric Tukey’s test was then applied at a significance level of 5%. The mechanical properties were correlated with the values of the filler content by Pearson’s correlation (\( P \leq 0.05 \)).

**Results**

The results of the VHN tests are presented in Table 2. Tukey’s test (\( P < 0.5 \)) showed that the nanofilled resin presented VHN means higher than those of the microhybrid resin, regardless of the light polymerization mode employed.

The results of DTS, FS, and FM are presented in Table 3. Statistical analysis (Tukey’s test, \( P < 0.05 \)) of the overall average of each composite showed no statistically significant differences between the averages of DTS, FS, and FM when photoinitiated by different methods of curing. Experiments testing the true content of the inorganic filler showed that the nanofilled composite presented 78.0 W\% of filler content, and the microhybrid presented 66.0 W\% of filler content. Pearson’s correlation showed positive relationship with VHN tests (\( r = 0.9922 \)).
and \( P < 0.001 \), DTS (\( r = 0.9635 \) and \( P = 0.0020 \) ), and FM (\( r = 0.4 \) and \( P = 0.027 \) ).

In the FS test, despite the slightly positive Pearson’s correlation (\( r = 0.14 \), no significant differences were found among the materials analyzed (\( P = 0.45 \)), suggesting that there was no relationship between the two variables.

### Discussion

The hardness of a resin composite is influenced by its constitution, which includes the type and matrix composition and the amount and particle shapes.\(^{20}\)

Previous literature suggests that a composite is properly polymerized when the maximum hardness of the bottom surface is \( \geq 80\% \) of the hardness value on the top surface.\(^{21,22}\) Given this, this study found that both materials showed effectiveness in the light polymerization modes, as the VHN mean value difference between the top and bottom surfaces was \( <20\% \). The light polymerization modes evaluated reached hardness values that were \( \geq 80\% \) of the hardness values of the upper surfaces, revealing that the photopolymerization modes were effective in curing all composites.

Statistical analysis showed that the nanofilled resin presented higher VHN values than did the microhybrid resin (Table 2): these results agree with studies conducted by Beum et al and Lu et al, but differed from a study by da Silva et al.\(^{1,11,22}\)

A strong correlation was identified between the VHN and the inorganic weight percent present in both composites, indicating that the amount of filler particles influenced and improved this property, thus corroborating the results of Mota et al.\(^{21}\)

Filler shape and size influences the inorganic content rise: spherical particles of regular size are responsible for this increase.\(^{21}\) A greater percentage of inorganic filler may improve the mechanical, physical and chemical properties of restorative resin-based materials.\(^{21}\)

DTS provides an important indication of the ability of the restorative material to withstand tensile stress generated during mastication. High diametral strength values are important for greater efficiency in supporting occlusal forces.\(^{10}\) The DTS results for the nanofilled composite were statistically higher than those of the microhybrid resin (Table 3), showing a strong correlation with the percentage of inorganic filler content. Some studies that evaluated resin-based materials found a positive relationship of this property with the physical-chemical composition of the materials.\(^{10,12,23}\)

In accordance with Specification No. 27 of the American Dental Association, the results of the diametral tensile test must present an average of at least 24 MPa.\(^{24}\) In this study, the results were higher than this value, revealing the effectiveness of both composites in relation to this property. The correlation of the percentage of filler content with this property showed a strong positive interaction.

Flexural strength is important for characterizing brittle materials because this type of test generates complex tensions by combining tensile stress, compression and shear. The clinical relevance of this property relates mainly to the chewing act, when different biting forces induce various tensions, both in the tooth and the restoration. Therefore, high DTS and FS values are important for the efficacy of restorative materials supporting the occlusal forces of posterior teeth.\(^{10}\)

The minimum flexural strength value required for restorative materials is 80 MPa.\(^{15}\) The materials used in this work achieved a better result regardless of the type of composite and light polymerization mode applied. This diverged from the studies of da Silva et al, Rodrigues et al, and Melander et al, where either no significant differences between nanofilled and microhybrid composites were observed or mycrohybrid resins showed better results compared to nanohybrid resins.\(^{11,18,25}\)

The flexural modulus, elastic modulus, or Young’s modulus is an important parameter obtained by the flexural test, which describes the stiffness of the material. Clinical situations require different composite materials with different flexural moduli. A class V cavity requires a restorative material with a low modulus to prevent displacement during chewing activity. On the other hand, a relatively high modulus is better for posterior restorations, such that they may withstand occlusal forces and preserve the adhesive interface.\(^{18}\)

The organic matrix of Filtek Z350, which consists of Bis-GMA, Bis-EMA, and UDMA with small amounts of TEGDMA, was developed by the manufacturer to reduce polymerization shrinkage.\(^{2}\) The presence of TEGDMA in the resin matrix has been associated

### Table 3. Mean values (SD) for DTS, FS, and FM of RBCs.

<table>
<thead>
<tr>
<th>Composite resin</th>
<th>Light Polymerization Mode</th>
<th>Tests</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>DTS (MPa)</td>
</tr>
<tr>
<td>Nanofilled (Filtek Z350)</td>
<td>Conventional (QTH)</td>
<td>54.39a (13.65)</td>
</tr>
<tr>
<td></td>
<td>Conventional (LED)</td>
<td>55.85f (9.98)</td>
</tr>
<tr>
<td></td>
<td>Exponential (LED)</td>
<td>56.87h (17.18)</td>
</tr>
<tr>
<td>Microhybrid (Charisma)</td>
<td>Conventional (QTH)</td>
<td>50.70k (9.44)</td>
</tr>
<tr>
<td></td>
<td>Conventional (LED)</td>
<td>50.32n (15.52)</td>
</tr>
<tr>
<td></td>
<td>Exponential (LED)</td>
<td>50.14q (10.77)</td>
</tr>
</tbody>
</table>

Different lowercase and uppercase letters on the row for each test were statistically significant (5% significance level by Tukey’s test).
with a decrease in the flexural strength of the material, while, paradoxically, it has been associated with an increased FM. 26
The FM values for the nanofilled resin were higher and significantly different from those recorded for the microhybrid resin (Table 3). These results corroborate those of Beun et al and Rodrigues et al, but diverged from the results observed by Melander et al. 18, 25
Upon evaluating the correlation of inorganic filler present in composite resins with the FS properties, no statistically significant relationship was found, suggesting that the size and shape of the particles do not influence this property. These findings differ from the Rodrigues et al study which showed that the filler content significantly interfered with the FS of the composites. 18
Statistical analysis of the FM of the resins showed a strong association with the filler content, revealing that the filler content increase directly influenced this property, which is in agreement with other studies. 1, 14, 18, 26 The VHN, DTS, FS and FM values among light polymerized materials tested by different modes of curing showed no statistically significant differences, as previously indicated in other studies. 1, 14, 22, 27 The energy density used for the conventional QTH and LED light polymerization modes was 16 J/cm3 and approximately 14.5 J/cm3 in exponential LED mode, which could explain the fact that they were not identified as different within this study.

Conclusion
This study revealed that the nanofilled resin performed better in all mechanical properties tested than did the microhybrid resin. The different curing units and the various photocuring modes, using approximately the same energy density, did not affect the mechanical properties of the composites evaluated. The type of composite seems to have more relevance in determining the mechanical properties than the choice of photocuring mode.

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