Mechanical Properties of Light-curing Composites Polymerized with Different Laboratory Photo-curing Units

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This study aimed to analyze the microhardness (KHN) and diametral tensile strength (DTS) of two hybrid resin composites (TPH Spectrum and Filtek Z250). To this end, the composites were polymerized with six laboratory photo-curing units (LPUs) and the results compared with an alternative polymerization method using conventional halogen light source in conjunction with additional polymerization in an autoclave (15 minutes/100ºC). LPUs were used following the manufacturers’ instructions. Diametral tensile strength and Knoop hardness tests were conducted for all groups (n=5). Data were statistically compared using ANOVA and Tukey’s test (α=0.05). Among the LPUs, the one that provided light curing in conjunction with heat and nitrogen pressure resulted in a significant increase in KHN and DTS of resin composites. Between the resin composites, Filtek Z250 showed higher hardness values than TPH Spectrum. It was concluded that the use of alternative polymerization with conventional light polymerization and autoclave was feasible with a wide implication for the general public in terms of reduced dental treatment cost.

Keywords: Resin composite, Laboratory photo-curing unit, Microhardness

INTRODUCTION

The polymerization of a resin composite occurs by the conversion of monomer molecules into a polymer network, accompanied by a closer packing of the molecules—thereby causing contraction in the composite⁶. When more intense light energy is used to polymerize a resin composite, more photons come into contact with camphorquinone photoinitiator molecules within the resin and more photoinitiator molecules are activated and raised to the excited state. In this excited state, the camphorquinone molecule collides with an amine, and a free radical is formed. The latter reacts with carbon to form a carbon double bond (C-C) of a monomer molecule, and thus polymerization is initiated⁷.

Based on the abovementioned polymerization mechanism, the degree of resin composite conversion ranges from 65 to 80%. This is so because the conversion of monomer to polymer is also dependent on others factors—such as concentration of activator, amount of inhibitor present, resin composite type, and light transmission through material⁸. On the other hand, by increasing the conversion level, composites may also undergo increased polymerization shrinkage and a higher stress level may be induced within the bond⁹. At this juncture, it should also be pointed out that under-polymerization is usually associated with inferior physical and mechanical properties—such as higher solubility⁹, susceptibility to abrasion⁹, color instability⁹, and post-operative sensitivity from unpolymerized monomer. Against this host of issues confronting polymerization, there is little doubt that adequate polymerization of composites is an important factor in ensuring good clinical performance.

In a study by Matsumura et al.⁸ whereby prosthetic composites were polymerized with different light sources, it was reported that hardness and water solubility were strongly influenced by the type of laboratory photo-curing unit used. It was also demonstrated that the use of a high-intensity laboratory photo-curing unit was effective not only for improving curing depth and wear resistance, but hardness and solubility as well⁸. With indirect composite systems, an important aspect is the possibility of using post-polymerization mechanisms associated with heat, pressure, or high light intensity⁹. With respect to materials, indirect and direct composites have similar compositions⁹, but in some cases, different components assure better physical properties that last longer⁹. Presently, easy and inexpensive techniques have been developed for direct composites in indirect restorations. Coupled with the employment of devices that are normally available at dentists’ offices to apply these techniques, better performance can now be obtained and harnessed from material properties, resulting in improved dental health⁹.

The first aim of this study was to test the hypothesis that laboratory photo-curing units
employing different polymerization methods—namely halogen lamp only, halogen lamp in conjunction with heat, halogen lamp in conjunction with heat and nitrogen pressure, and alternative polymerization method using halogen lamp and autoclave—could lead to statistical differences among the physical properties of composites. The second aim of this study was to test the hypothesis that material composition had a statistically significant influence on Knoop hardness and diametral tensile strength.

Therefore, it would be opportune to evaluate the influence of laboratory photo-curing units on the hardness and diametral tensile strength of two hybrid resin composites used to confect indirect restorations. The null hypothesis was that different polymerization devices would result in similar hardness and diametral tensile strength of composites.

**MATERIALS AND METHODS**

Two photo-polymerizable hybrid resin composites (TPH Spectrum, Lot #20032373, Dentsply DeTrey, Konstanz, Germany; and Filtek Z250, Lot #20041370, 3M ESPE, St. Paul, MN, USA, Shade A3) were studied. Table 1 shows the compositions of these two materials. To analyze the effect of laboratory photo-curing units (Table 2)—versus an alternative polymerization method using a conventional halogen light source followed by additional curing in an autoclave—on the mechanical properties of these composites, Knoop hardness (KHN) and diametral tensile strength (DTS) tests were carried out. Five samples were made for each combination of restorative material and polymerization system for each test.

*Diametral tensile strength test*

Cylindrical specimens were prepared for diametral

<table>
<thead>
<tr>
<th>Composite</th>
<th>Composition</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filtek Z250</td>
<td><strong>Organic Matrix</strong>: Bis-GMA, Bis-EMA, and UDMA</td>
<td>3M ESPE, St. Paul, MN, USA</td>
</tr>
<tr>
<td></td>
<td><strong>Inorganic Filler</strong>: Rounded zirconia, silica (92% weight or 69% volume)</td>
<td></td>
</tr>
<tr>
<td>TPH Spectrum</td>
<td><strong>Organic Matrix</strong>: Urethane modified Bis-GMA</td>
<td>Dentply DeTrey, Konstanz, Germany</td>
</tr>
<tr>
<td></td>
<td><strong>Inorganic Filler</strong>: Barium glass and silica (77.5% weight or 59% volume)</td>
<td></td>
</tr>
</tbody>
</table>

**Table 1** Compositions and manufacturers of restorative materials used in this study

<table>
<thead>
<tr>
<th>Curing unit</th>
<th>Main polymerization mechanism</th>
<th>Wavelength (nm)</th>
<th>Light irradiance (mW/cm²)</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>EDG Lux</td>
<td>Halogen lamp (4 lamps)</td>
<td>400-440</td>
<td>250</td>
<td>EDG Ltda, São Carlos, SP, Brazil</td>
</tr>
<tr>
<td>Evolution</td>
<td>Halogen lamp (4 lamps)</td>
<td>420-620</td>
<td>800</td>
<td>Foto Ceram Ltda, Goiânia, GO, Brazil</td>
</tr>
<tr>
<td>Nitroceram</td>
<td>Nitrogen pressure 135° heat</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Optilux</td>
<td>Halogen lamp (1 lamp)</td>
<td>400-500</td>
<td>650</td>
<td>SDS Kerr, Orange, CA, USA</td>
</tr>
<tr>
<td>HP Curing Unit</td>
<td>Nitrogen pressure 155° heat</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Targis Quick</td>
<td>Halogen lamp (1 lamp)</td>
<td>400-500</td>
<td>400</td>
<td>Ivoclar Vivadent, Schaan, Liechtenstein</td>
</tr>
<tr>
<td>Targis Power</td>
<td>Halogen lamp 95° heat</td>
<td>400-580</td>
<td>400</td>
<td></td>
</tr>
<tr>
<td>UniXS</td>
<td>Xenon stroboscopic light (2 lamps)</td>
<td>320-520</td>
<td>1200</td>
<td>Heraeus Kulzer, Wahrheim, Germany</td>
</tr>
</tbody>
</table>

**Table 2** Details of laboratory photo-curing units, as provided by the manufacturers
Table 3  Mean KHN (MPa) values obtained with the different curing units, as analyzed by Tukey’s test (p<0.05)

<table>
<thead>
<tr>
<th>Curing Unit</th>
<th>Filtek Z250 Top</th>
<th>Filtek Z250 Bottom</th>
<th>TPH Spectrum Top</th>
<th>TPH Spectrum Bottom</th>
</tr>
</thead>
<tbody>
<tr>
<td>XL 3000 + autoclave</td>
<td>95.65 (10.22)</td>
<td>95.60 (8.83)</td>
<td>85.29 (6.03)</td>
<td>83.36 (4.57)</td>
</tr>
<tr>
<td>UniXS</td>
<td>101.2 (8.20)‡</td>
<td>100.92 (9.31)‡</td>
<td>77.40 (4.98)‡</td>
<td>77.61 (5.68)‡</td>
</tr>
<tr>
<td>EDG Lux</td>
<td>90.14 (8.56)‡</td>
<td>91.58 (12.96)‡</td>
<td>75.48 (5.75)‡</td>
<td>75.00 (4.92)‡</td>
</tr>
<tr>
<td>Evolution</td>
<td>94.35 (4.91)‡</td>
<td>93.08 (6.11)‡</td>
<td>75.72 (4.16)‡</td>
<td>75.39 (4.19)‡</td>
</tr>
<tr>
<td>Targis Quick + Targis Power</td>
<td>97.31 (4.59)‡</td>
<td>98.21 (6.92)‡</td>
<td>76.38 (3.73)‡</td>
<td>75.17 (3.97)‡</td>
</tr>
<tr>
<td>Evolution + Nitroceram</td>
<td>103.75 (5.18)‡</td>
<td>103.86 (5.95)‡</td>
<td>80.60 (5.11)‡</td>
<td>81.88 (3.08)‡</td>
</tr>
<tr>
<td>Optilux 500 + HP Curing Unit</td>
<td>106.58 (5.63)‡</td>
<td>96.79 (8.12)‡</td>
<td>81.64 (8.54)‡</td>
<td>80.58 (5.37)‡</td>
</tr>
<tr>
<td>Mean - irrespective of curing method</td>
<td>98.47‡</td>
<td>98.38‡</td>
<td>77.57‡</td>
<td>77.48‡</td>
</tr>
</tbody>
</table>

Different uppercase letters denote significant differences among resin composite surfaces, irrespective of the curing method (horizontal comparison only).
Different lowercase letters denote significant differences among polymerizing units (vertical comparison only).

tensile strength test according to ADA Specification No. 27. An aluminum mold of 3 mm height and 6 
mm diameter was used. TPH Spectrum and Filtek 
Z250 resin composites were inserted in three incre-
ments and polymerized in one direction only (from 
top of the samples) using the methods described in 
Table 3.

Group 1: Conventional polymerization with a 
halogen light source (XL 3000, 3M ESPE, St. Paul, 
MN, USA) at 850 mW/cm² for 40 seconds and post-
polymerized in an autoclave (Cristália, São Paulo, SP, 
Brazil) at 100°C for 15 minutes.

Group 2: Polymerization with a xenon strobo-
sopic light curing unit (UniXS, Heraeus Kulzer, 
Wehrheim, Germany) for 90 seconds and final polymer-
ization for three minutes in the same unit.

Group 3: Three minutes with a multifocal labo-
ratory halogen light source (EDG Lux, EDG Ltda, 
São Carlos, SP, Brazil), then final polymerization 
for seven minutes.

Group 4: Three minutes with a multifocal labo-
ratory halogen light source (Evolution, Foto Ceram 
Ltda, Goiânia, GO, Brazil), then final polymerization 
for seven minutes in the same unit.

Group 5: Polymerization with a halogen light 
source (Targis Quick, Ivoclar Vivadent, Schaan, 
Liechtenstein) for 10 seconds, and then final poly-
merization at 400 mW/cm² and 95°C for 25 minutes 
with a Targis Power (Ivoclar Vivadent) laboratory 
unit.

Group 6: Three minutes with a multifocal labo-
ratory halogen light source (Evolution), final polymer-
ization for seven minutes in the same unit, then 
post-polymerization in a Nitroceram unit (Foto 
Ceram Ltda, Goiânia, GO, Brazil) under 413 MPa 
pressurized nitrogen and at temperature raised to 
135°C for 20 minutes.

Group 7: Conventional polymerization with a 
halogen light source (Optilux 500, Kerr Corporation, 
Orange, USA) for 40 seconds and additional polymer-
erization (HP Curing Unit, Kerr Corporation, 
Orange, USA) was done under 413 MPa pressurized 
nitrogen at temperature raised to 135°C for 20 min-
utes.

Prior to final polymerization, samples were 
removed from the metal mold and allowed for post-
polymerization free of metal. The specimens were 
then stored in a dark container in distilled water at 
37°C for 24 hours prior to testing. A compressive load 
was applied on the diametral surface of the 
samples to obtain diametral tensile strength at a 
crosshead speed of 0.5 mm/minute in a testing 
machine (EMIC 2000 DL, São José dos Pinhais, PR, 
Brazil).

Knoop hardness test
For Knoop hardness test, five cylindrical specimens 
for each group were made as described for the 
diametral tensile strength test. The samples were 
placed in a plastic cylinder, with their long axis per-
pendicular to the latter’s surface. The specimens 
were embedded in acrylic resin with the top and bot-

top surfaces of each sample exposed. Then, the sur-
faces were ground with a series of silicon carbide 
papers (Sof-Lex, 3M ESPE, St. Paul, MN, USA) and 
polished with diamond paste to produce a smooth,
uniform surface. Knoop hardness was determined with a microhardness tester (HMV-2000, Shimadzu Corporation, Kyoto, Japan) by applying a load of 50 g for 30 seconds. Five indentations were made on each sample (top and bottom), and mean values were calculated for the top and bottom surfaces of each sample.

Statistical analysis
Results of diametral tensile strength (MPa) were analyzed initially by one-way ANOVA and Tukey’s test (p<0.05) to detect differences among the materials. Then, two-way ANOVA (7×2) and Tukey’s test were used to detect differences between the laboratory photo-curing units and alternative polymerization using conventional photo-curing and autoclave. Results of Knoop hardness test on the top and bottom surfaces of each group were analyzed by Student’s t-test (p<0.05). The mean values of top and bottom surfaces together, which represented the general Knoop hardness value for each sample, were submitted to ANOVA and Tukey’s test (p<0.05) to verify the differences among the groups.

RESULTS
Figures 1 and 2 present the mean diametral tensile strength (DTS) and Knoop hardness (KHN) values of resin composites as a function of laboratory polymerization method.

One-way analysis of DTS showed differences among the groups analyzed in this study (p<0.001). On the overall, Tukey’s test (p<0.05) showed that TPH Spectrum had lower DTS than Filtek Z250.

![Fig. 1](image1)

Fig. 1 Means of diametral tensile strength vs laboratory photo-curing units. (Uppercase letters represent Tukey’s categories of Filtek Z250 and lowercase letters compare differences among TPH Spectrum values.)

![Fig. 2](image2)

Fig. 2 Means of Knoop hardness (MPa) vs laboratory photo-curing units analyzed by Tukey’s test (p<0.05). (Different uppercase letters denote significant differences among categories of Filtek Z250, while different lowercase letters denote significant differences among TPH Spectrum values.)
Two-way ANOVA ($7 \times 2$) was applied to verify the differences among the effects of each laboratory curing method and the possible interaction between composites and polymerization methods. Following which, Tukey’s test was applied to the DTS data for the interaction between these factors. It was shown that TPH Spectrum polymerized by an Evolution unit resulted in lower DTS values; as for Filtek Z250, the laboratory curing unit had no influence (Fig. 1).

A similar analysis was performed for KHN. On the overall, Tukey’s test showed higher KHN values for Filtek Z250 than TPH Spectrum, irrespective of the curing method. Microhardness verified on the top and bottom surfaces of the samples showed no significant differences between them in all groups (Table 3).

Photo-curing units that were used in conjunction with heat and pressure curing (HP Curing Unit and Nitroceram) or polymerization with a xenon lamp (UniXS) resulted in higher KHN. On the other hand, specimens subjected to curing with only a halogen lamp (EDG Lux and Evolution) showed lower KHN values. As for the alternative polymerization method using conventional photo-curing and autoclave, it caused TPH Spectrum to yield a higher KHN value—a curing performance similar to that obtained with laboratory photo-curing units HP Curing Unit, Nitroceram, and UniXS. However, with Filtek Z250, the alternative polymerization method led to a lower KHN value—a curing performance similar to that obtained with Targis, EDG Lux, and Evolution units (Fig. 2).

**DISCUSSION**

The hypotheses tested in this study were partially rejected. Microhardness was influenced by laboratory photo-curing units, and the use of an alternative polymerization method with a halogen lamp and an autoclave on conventional composites resulted in comparable or superior mechanical properties. On the other hand, there were no differences in diametral tensile strength between the different material compositions.

Resin composites are currently one of the most widely used materials in restorative dentistry. They can be described as inorganic particles packed within an organic matrix and joined together by a silane coupling agent. Mechanical properties of resin composites are vastly influenced not only by their chemical composition, but also by the degree of monomer conversion. A light-sensitive material contains camphorquinone to react with a reducing agent when activated, and activating it to polymerization requires sufficient light intensity and a suitable wavelength. It is well known that inadequate polymerization adversely affects the mechanical properties of composite materials in terms of strength, stiffness, color stability, hardness, and wear resistance. However, when post-polymerization methods are used, the material properties can be improved. The laboratory curing units used in this research had different light sources, magnitudes of generated heat during polymerization, and post-polymerization methods.

This study evaluated the polymerization performance of laboratory photo-curing units by determining the Knoop hardness and diametral tensile strength of two resin composites. The physical properties of a restorative material provide an indication of how the material will function under stress in an oral environment. On this note, Knoop hardness measurement of resin composites is an indirect method that indicates the strength under compressive loading. Indentation, according to Xu et al., offers information that may be more relevant to applications that involve localized, non-uniform deformation or point contacts, such as dental occlusal contacts with surface asperities or third bodies during chewing and wear. While microhardness focuses on surface hardness characterization, diametral tensile strength provides information about the bulk properties of a restoration and gives an indication of its resistance to the lateral forces generated during function by occlusal contact. Thus, both Knoop hardness and diametral tensile strength are important properties of restorative materials used in posterior teeth.

The composition of conventional direct hybrid composites used in this study presented basic components similar to those of indirect resin composites. The main difference between these two types of material lies in the possibility of employing high light intensity and other post-polymerization methods with the indirect technique. In terms of Knoop hardness, Filtek Z250 resin—which had a BisGMA-based monomer phase with UDMA and inorganic filler particles composed of zirconium glass—showed generally better values than TPH Spectrum. The latter resin had an organic matrix based on urethane modified Bis-GMA and inorganic filler particles composed of barium glass and silica. It should be noted that a composite’s hardness is influenced by several factors, such as composition of the organic matrix, type and amount of filler particles, and also the degree of conversion. With all these factors taken together, the higher Knoop hardness values for Filtek Z250 might be attributed to differences in the filler and organic matrix compositions between the two hybrid resin composites. In terms of diametral tensile strength, there were no statistical differences between the two material compositions. It was possible that the diametral tensile strength test was not sensitive enough to differentiate hybrid composites. This test was designed for analyzing macrostructures,
and thus changes in diametral tensile strength—owing to small differences in material composition—might not be duly measured or reflected.

Under the experimental conditions of this study, the results showed that different laboratory photo-curing units could lead to different physical properties in resin composites when analyzed by Knoop hardness and diametral tensile strength tests, and that units with a similar polymerization method presented the same curing performance. Except when compared with the UniXS unit, specimens polymerized with HP Curing Unit and Nitroceram presented higher KHN values than those polymerized with other laboratory units, regardless of material type. The latter devices operated at a temperature of 135 °C and under 413 MPa pressurized nitrogen. These results thus suggested that the use of light curing in conjunction with heat and pressure curing could increase the segmental chain vibration amplitude, allowing near radicals and methacrylate groups to collide, thereby increasing monomer conversion\(^{22,23}\). With increased conversion level, the mechanical properties of composites were then likewise improved.

In the same way, alternative polymerization using conventional photo-curing and autoclave post-curing method tended to yield higher DTS and KHN values, mainly in specimens confined with the TPH Spectrum composite. This suggestion might be explained by the loading process used in the present study. According to Brosh et al.\(^{10}\) on indirect composite techniques, pressure might be applied by means of hyperbaric atmosphere. Further, Wilson and Norman\(^{20}\) reported that the application of a 6-bar pressure before photo-polymerization produced significantly fewer voids than layering or bulk packing technique. In this study, the autoclave method was performed at 100 °C for 15 minutes. This was because it was reported that optimal post-curing effect was rendered under the conditions of 110 °C temperature for 10 to 60 minutes\(^{22}\). As for the laboratory unit protocol, it entailed a temperature range between 95° and 120 °C with time variation between six and 15 minutes\(^{8,10}\).

Another important factor that controls the curing performance of photo-curing units is the intensity of the light source\(^{10}\). Specimens polymerized with UniXS showed good DTS and KHN values. This was chiefly because this laboratory unit was equipped with two cylindrical xenon stroboscopic lamps which functioned at a light intensity of 1200 mW/cm\(^2\). Martos et al.\(^{28}\), in their evaluation on the effects of hydrolytic degradation of composites polymerized by HP Curing Unit (nitrogen pressure and 135 °C heat) and UniXS (Xenon stroboscopic light), suggested that heat and pressure treatment with additional light exposure did not affect KHN. In the present study, although UniXS also resulted in similar KHN, the employment of halogen lamp in conjunction with heat and pressure resulted in higher KHN for TPH Spectrum. The xenon lamp has a remarkably high light intensity compared with conventional halogen lamps\(^{28}\), but the key advantage of applying a xenon light source for resin composite polymerization lies in its reduced irradiation time when compared with other laboratory units.

It was thus shown that when subjected to polymerization with a conventional halogen light source in conjunction with post-curing in an autoclave, the two hybrid resin composites showed similar—if not superior—DTS and KHN values when compared with curing by laboratory photo-curing units. The satisfactory mechanical properties obtained indicated that the autoclave was a feasible alternative for confecting indirect restorations with similar functionality but with reduced final treatment cost, because these equipment and materials are typically present in a dentist's office. Nonetheless, despite the encouraging results obtained in this study, other properties attained by this method—in terms of conversion degree, marginal adaptation, and wear resistance—should be further investigated. It must also be put into perspective that this study was merely an in vitro characterization of material properties. As such, a long-term and controlled clinical study is needed to confirm the clinical efficiency of this alternative.

CONCLUSIONS

Based on the methodology developed in this study and the subsequent data analysis, it was possible to conclude that:

1. Knoop microhardness was influenced by the composition of resin composites and by the type of laboratory photo-curing unit used.
2. Similar KHN values were obtained on the top and bottom surfaces of all samples.
3. Different laboratory photo-curing units could lead to different physical properties in resin composites, and that units with a similar polymerization method presented the same curing performance.
4. The use of light curing in conjunction with heat and pressure curing improved the mechanical properties of resin composites.
5. The use of alternative polymerization with conventional photo-curing and autoclave was shown to be feasible with a wide implication for the general public in terms of reduced dental treatment cost.
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