Microhardness Evaluation of Resin Composites Polymerized by Three Different Light Sources

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This study examined the surface microhardness of four kinds of resin composites with different fillers and resin matrices. Ten specimens of 2 mm thickness and 4 mm diameter of each resin composite were polymerized using a halogen light, a blue light-emitted diode, and a plasma arc unit. Microhardness evaluation was performed at top and bottom surfaces for each specimen using a Vickers microhardness tester. Furthermore, morphologies of the polished top surfaces of composites cured with blue light-emitted diode were observed using scanning electron microscopy. Results indicated that composites cured with halogen or blue light-emitted diode light yielded higher microhardness values, although it also appeared to depend on the type of composite cured. Plasma arc curing according to manufacturer’s instructions yielded the lowest microhardness values for all the materials. Among the materials tested, the nanofilled resin composite displayed the highest microhardness values for each curing regime.

Keywords: Microhardness, Resin composite, Light curing unit

INTRODUCTION

The popularity of esthetic tooth-colored restorations has promoted the use of light-activated resin composites. Newer resin composites have increased strength, owing to a higher filler content, improved filler technology, modifications in the organic matrix, and better polymerization⁵. Hybrid and microhybrid resin composites contain a broad range of particle sizes which can lead to high filler loading with resultant high strength. While they may contain a small fraction of filler particles in the nanometer particle size range, they also contain a range of substantially larger filler particles⁶. On this note, recent hybrid composites contain macrofillers of an average size of 0.1–6.0 μm and microfillers with a particle size of 0.01–0.05 μm⁷.

Due to the modification of fillers to extremely small particles, microfilled composites have been developed. Traditional microfillers are made from silica with an average particle size of 40 nm²⁵. Recently, nanofabrication technologies have been used to produce nanosized fillers based on building blocks on a molecular scale. This process has produced a commercial product with silica nanoparticles of 2 to 20 nm and zirconia-silica nanoclusters of 0.6 to 1.4 μm⁵⁶.

Adequate polymerization is of prime importance in obtaining optimal mechanical and physical properties as well as improved clinical performance of resin composite restorative materials⁷⁹. However, a number of factors determine the polymerization quality of a resin composite: material composition, operating features of light source, and photo-polymerization conditions¹⁰–¹⁴.

Polymerization of light-activated dental resin composites takes place through photoinitiators present in their compositions¹⁵. The majority of resin composites employ camphorquinone (CQ) as a photoinitiator with a maximum light absorption of 468 nm¹⁶–¹⁸. Upon activation by light energy, CQ will react with an amine-reducing agent to form free radicals. Resin composite is then polymerized with monomers or short-chain carbon groups converted to longer-chain polymers that are more stable, strong, and chemically resistant¹⁹. However, some resin composites contain other photoinitiators in addition to CQ, known as coinitiators, which absorb light at shorter wavelengths²⁰.

In response to the dramatic rise in the use of resin composite restorations, many different light curing units (LCUs) have emerged over the past few years²¹–³⁰. The quartz-tungsten-halogen LCU has been widely used as a practical method to cure resins. Halogen lamps have a broad bandwidth output which is filtered to deliver blue light at 400–500 nm²¹–²². Despite their popularity, the quartz-tungsten-halogen LCUs present several limitations such as gradual reduction in energy output due to bulb and filter degradation, limited depth of cure, and relatively long exposure time²¹–²².

To overcome such problems, light-emitting diode technology has been used to cure resin composites²³–²⁸. It has been reported that blue
light emitted diode-based LCU s had a narrow spectral range with a peak around 470 nm — which coincides with the optimum absorption wavelength for CQ activation\textsuperscript{2,7,15-17,21,28-31}. It has also been found that blue light-emitted diode source produced a degree of monomer conversion that was significantly higher than that obtained with a halogen source, even though all the sources were adjusted to produce the same irradiance (100 mW/cm\textsuperscript{2})\textsuperscript{23}.

In a bid to reduce polymerization time, plasma arc-based LCU s are now available. They produce light of a much greater intensity than conventional halogen LCU s\textsuperscript{2,7,15-17,21,28-31}. However, rapid initiation of polymerization may induce shorter chain lengths, thereby leading to a lower molecular weight and compromising the physical and mechanical properties of composite materials\textsuperscript{17}.

The curing efficiency of a light source can be assessed by evaluating the mechanical, physical, and chemical properties of the resin composite cured. Among the properties studied, the depth of cure measurement has shown clear relevance to the clinical aspects of composite curing\textsuperscript{11,14,17,23}. In determining the depth of cure, measurement of the degree of C=C conversion is considered to be the most reliable technique. However, microhardness evaluation is also suggested because of high correlation between the two methods for a certain resin material\textsuperscript{2,9,12,14,16,24,30}. Therefore, the aim of this study was to compare the microhardness of four composite resins with different chemical compositions cured with quartz-tungsten-halogen, blue light-emitted diode, and plasma arc light sources.

**MATERIALS AND METHODS**

**Composites**

Four light-activated dental resin composites of the same shade (A2), but with different filler contents and compositions were selected to determine the curing efficiency of light curing units. The list of composites, including their brand names, manufacturers, and compositions (resin matrix, filler size, filler type, and filler content), are shown in Table 1. The composite Durafill VS was a microfill, Gradia Direct microhybrid, Solitaire 2 a packable hybrid resin composite, and Filtek Supreme a nanocomposite based on a combination of nanoparticles and nanoclusters. Solitaire 2 contained a coinitiator in addition to the standard initiator CQ\textsuperscript{26,32}, while others contained only CQ as the photoinitiator.

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Restorative materials used in this study</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material</td>
<td>Filtek Supreme</td>
</tr>
<tr>
<td>Manufacture</td>
<td>3M ESPE, St. Paul, MN, USA</td>
</tr>
<tr>
<td>Type</td>
<td>Nanocomposite</td>
</tr>
<tr>
<td>Resin Matrix</td>
<td>BIS-GMA, BIS-EMA(6), UDMA, TEGDMA</td>
</tr>
<tr>
<td>Filler type</td>
<td>nanosilica and zirconia/silica nanoclusters</td>
</tr>
<tr>
<td>Average particle size</td>
<td>nano silica: 5-20 nm</td>
</tr>
<tr>
<td>Filler volume %</td>
<td>58-60</td>
</tr>
<tr>
<td>Filler weight %</td>
<td>78.5</td>
</tr>
<tr>
<td>Co-initiators</td>
<td>no</td>
</tr>
</tbody>
</table>

*Say et al\textsuperscript{15}, **Peris et al\textsuperscript{13}, ***Beun et al\textsuperscript{6} ****Uhl et al\textsuperscript{32} other data are as disclosed by the manufacturers.

Bis-GMA: Bisphenol A diglycidylmethacrylate; Bis EMA(6): Bisphenol A polyethylene glycol diether dimethacrylate; Bis-GA: Bisphenol A glycidilpolyacrylate; UDMA: Urethane dimethacrylate; TEGDMA: Triethyleneglycoldimethacrylate.
Table 2 Light curing units and conditions tested in this study

<table>
<thead>
<tr>
<th>Light source</th>
<th>Helilux DLX</th>
<th>UltraLight PB-070</th>
<th>Apollo 95E Elite</th>
</tr>
</thead>
<tbody>
<tr>
<td>Light source</td>
<td>Halogen</td>
<td>LED</td>
<td>PAC</td>
</tr>
<tr>
<td>Intensity (mW/cm²)</td>
<td>750</td>
<td>1000</td>
<td>1600</td>
</tr>
<tr>
<td>Energy density (J/cm²)</td>
<td>30</td>
<td>10</td>
<td>4.8</td>
</tr>
<tr>
<td>Wavelength of emission (nm)</td>
<td>400-500</td>
<td>440-480</td>
<td>460-490</td>
</tr>
<tr>
<td>Curing time (s)</td>
<td>40</td>
<td>10</td>
<td>3</td>
</tr>
</tbody>
</table>

Light curing units
The composites were cured with LCUs of a conventional quartz-tungsten-halogen lamp (QTH; Helilux DLX, Ivolear Vivadent, Schaan, Liechtenstein), a blue light-emitted diode (LED; UltraLight PB-070, Fine Vision Electronics Co. Ltd., Sanchung City, Taipei County, Taiwan), and a plasma arc (PAC; Apollo 95E Elite, Dental Medical Diagnostics Systems Inc., Orange, CA, USA). Light intensities given by the light unit manufacturers, as well as energy densities and curing time recommended by the restorative manufacturers are listed in Table 2.

Microhardness testing
Ten specimens of each composite were cured with each LCU. The composites were inserted into circular Teflon molds (height: 2 mm; inner diameter: 4 mm; outer diameter: 8 mm), and confined between two opposing polyester strips (Mylar, Moyo Union Broach, York, USA). A glass slide (1 mm thick) was then placed on the mold and excess material was extruded. The composite surface was irradiated through the glass slide and Mylar strip. After which, the Mylar strips were discarded and the specimens were stored in distilled water at 37°C for 24 hours. Prior to the microhardness measurement, the samples were polished flat using a sequence of 600-, 800-, and 1200-grit silicon carbide papers and Sof-Lex discs (3M ESPE, St. Paul, MN, USA). The specimens were then blotted dry.

By means of a digital microhardness tester (Shimadzu HMV-M3, Kyoto, Japan), a 300-g load was applied through the Vickers indenter with a dwell time of 15 seconds. Three indentations were made at top and bottom surfaces respectively, and the measurements were taken at different points on each specimen. A mean Vickers hardness number (VHN) and hardness ratio were then calculated and tabulated using the following formula: Hardness ratio = VHN of bottom surface/VHN of top surface.

Statistical analysis
Mean values and standard deviations of microhardness values were calculated using a SPSS statistical software program (Version 10.0, SPSS Inc., Chicago, USA). The data were subjected to statistical analysis among the composites as well as among light sources using two-way analysis of variance (ANOVA). Where significant differences were present, Tukey’s post hoc test was applied to examine pairwise differences at a significance level of 0.05.

Scanning electron microscopic observation
To clarify the size and distribution of filler particles of the resin composites tested, one specimen from each material of 1 mm thickness and 4 mm diameter was prepared in the same way as for the microhardness test and cured with LED LCU. The top surface of each composite specimen was polished as described above, and the polished surfaces were gold sputter-coated. Then, photomicrographs were taken using a scanning electron microscope (SEM) (Jeol JSM 6400, Noran Instruments, Japan) at ×5,000 and ×10,000 magnifications.

RESULTS
The means and standard deviations of the VHN values of top and bottom surfaces are shown in Table 3. Hardness ratios obtained after polymerizing composite resins with each of the three light curing units were also given.

A complete microhardness evaluation by two-way ANOVA indicated clearly that one of the LCUs had the best curing effect on a particular composite type used (F=112.57 for the top surface and F=65.75 for the bottom surface). Then, for all the materials cured with the three light sources, the values obtained for the bottom surface were found to be lower than those of the top surface.

In terms of the curing efficiency of different LCUs at the top surface of a given composite material, Tukey’s post hoc test showed that there were statistically significant differences among the microhardness values cured with QTH, LED, and PAC LCUs (p<0.05), except for Gradia Direct resin composite material. Durafill VS and Solitaire 2 resin composites cured with QTH LCU, and Filtek Supreme and Gradia Direct resin composites cured with LED LCU yielded the highest microhardness values, when compared to other specimens of the same resin composite cured with other LCUs. PAC LCU appeared to produce the poorest cure, giving the lowest VHN values for all materials. However, when Gradia Direct resin composite was cured with QTH or PAC, no statistically significant differences were noted (p>0.05).
### Table 3  Mean Vickers microhardness values and standard deviations (SD) for each resin composite and LCU evaluated

<table>
<thead>
<tr>
<th>Resin composites</th>
<th>Top surface</th>
<th>Mean±SD Bottom surface</th>
<th>Hardness Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Duraﬁll VS</td>
<td>QTH</td>
<td>33.40±1.13&lt;sup&gt;a&lt;/sup&gt;</td>
<td>25.33±5.27&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>LED</td>
<td>22.30±0.38</td>
<td>16.78±2.07&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>PAC</td>
<td>20.11±1.85</td>
<td>16.26±3.75&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>Filtek Supreme</td>
<td>QTH</td>
<td>66.79±5.09</td>
<td>59.38±4.92&lt;sup&gt;c&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>LED</td>
<td>70.80±4.89</td>
<td>64.89±6.57&lt;sup&gt;c&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>PAC</td>
<td>53.60±3.03</td>
<td>35.98±5.14&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>Solitaire 2</td>
<td>QTH</td>
<td>51.04±2.43</td>
<td>40.51±3.53</td>
</tr>
<tr>
<td></td>
<td>LED</td>
<td>40.69±0.56&lt;sup&gt;b&lt;/sup&gt;</td>
<td>32.85±2.09&lt;sup&gt;f&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>PAC</td>
<td>32.10±1.94</td>
<td>27.61±2.47&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>Gradia Direct</td>
<td>QTH</td>
<td>29.76±2.72&lt;sup&gt;aA&lt;/sup&gt;</td>
<td>21.08±4.53&lt;sup&gt;cD&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>LED</td>
<td>38.00±4.55&lt;sup&gt;b&lt;/sup&gt;</td>
<td>31.55±6.35&lt;sup&gt;f&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>PAC</td>
<td>27.16±2.69&lt;sup&gt;A&lt;/sup&gt;</td>
<td>17.52±1.99&lt;sup&gt;D&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

<sup>n=10 specimens per experimental condition.</sup>

The values underlined are the maximum top surface hardness.

By the two-way ANOVA: \( F = 112.57 \) for top surfaces; \( F = 65.75 \) for bottom surfaces; \( F = 3.991 \) for hardness ratio at \( P = 0.000; p<0.05. \)

Means labelled with the identical letters in the columns are not significantly different by the Tukey’s Test (\( p>0.05 \)). The same small letters are for the comparison of different resin composites cured with the same light source; the same capital letters for the same resin composite according to the light curing methods, respectively. Others not superscripted differ statistically by the Tukey’s Test (\( p<0.05 \)).

Composites yielding high microhardness values at their top surface also gave high bottom surface microhardness values. For the same resin composite cured with different light sources, the bottom surface yielded different microhardness values (\( p<0.05 \)). However, the bottom surface hardness values of Duraﬁll VS and Gradia Direct did not change after curing with QTH and PAC LCUs. Similar results were also obtained for Filtek Supreme after curing with QTH and LED LCUs (\( p>0.05 \)).

The comparison of VHN values of different composite materials cured with the same light source yielded statistically significant results at both (top and bottom) surfaces (\( p<0.05 \)). The nanocomposite, Filtek Supreme, yielded the highest microhardness values, independent of the LCU used. The hybrid resin composite, Solitaire 2 seemed to be the second hardest material. However, microhardness values of this material were not significantly different from those of microhybrid resin composite, Gradia Direct, at both surfaces when they were cured with LED LCU. Similar result was found for Filtek Supreme of which the bottom surface hardness was not significantly different from that of Solitaire 2 when cured with PAC LCU (\( p>0.05 \)). As for the microﬁlled resin composite, Duraﬁll VS, the VHN values of both surfaces when cured with QTH LCU were not significantly harder than those of Gradia Direct when cured with the same light source (\( p>0.05 \)). However, with the use of LED and PAC LCUs, the VHN values of this material at the top surface showed a significant decrease when compared to those of Gradia Direct resin composite (\( p<0.05 \)). As for the PAC curing of Duraﬁll VS and Gradia Direct, similar bottom surface microhardness values were yielded (\( p>0.05 \)).

Hardness ratio ranged between 0.65±0.04 and 0.92±0.03. In particular, LED curing of all the resin composites, except with Duraﬁll VS, yielded a score exceeding 0.8. The highest hardness ratio was found for Filtek Supreme cured with LED LCU, while the lowest score was Gradia Direct cured with PAC LCU.

In Fig. 1, the SEM image showed that Filtek Supreme had spherical particles. Large agglomerations of nanoclusters were clearly seen, while individual nanoparticles sheared off from the zirconia-silica nanoclusters. In Fig. 2, it was apparent that Duraﬁll VS had fine particles (pyrogenic SiO<sub>2</sub>) which were much smaller than the large particles of splinter polymers. The SEM image of Gradia Direct (Fig. 3) was markedly different from that of Filtek Supreme, showing low levels of agglomeration. It seemed that a single-phase polymer matrix had a more dominant effect than the particles. In the SEM image of Solitaire 2 (Fig. 4), irregular-shaped particles were found. The amorphous and crystalline-like phases appeared to coexist with the crystalline-like
phase embedded in the amorphous phase.

DISCUSSION

Hardness is generally correlated with mechanical strength, rigidity, and resistance to intraoral softening. It is also associated with the degree of monomer conversion, which plays an important role in determining the clinical success of a dental restorative material\[28,32\]. Against this background, this study used hardness to evaluate the curing performance of different LCU technologies on resin composites with different chemical compositions. Shade A2 was selected to minimize the effects of colorants on light polymerization, and 2-mm-thick composite specimens were used to ensure a uniform polymerization\[30\]. Since a minimum intensity of 400 mW/cm² has been suggested for routine polymerization\[13,33\], LCUs greater than this intensity together with manufacturers’ recommended curing times for resin composites were used. The effectiveness of cure cannot be assessed by top surface hardness alone. Bottom surface hardness is more critically affected by light intensity, and is thus a better gage on the effectiveness of cure of a composite\[34\]. Against this background, bottom surface hardness was also measured.

Irrespective of the LCU used, bottom surface hardness values were lower than those of the top surface of all the materials tested. In this study, there was sufficient light to activate the CQ initiator and there were no overlying composites to interfere with light transmission. Therefore, composite cure at the top surface was not as dependent on
light intensity as the bottom surface. Indeed, as light passes through the bulk of a composite, its intensity is greatly decreased due to the absorption and scattering of light by filler particles and resin matrix. This decrease results in a gradation of cure such that it decreases from top surface inward. This then accounted for the difference between top surface hardness and bottom surface hardness of all the materials cured with each light source. As for the differences among the materials, they could be attributed to their different filler contents. This was because it has been stated that a higher filler proportion could make light penetration more difficult.

It has been suggested that bottom/top surface hardness ratio is an important criterion to verifying the efficiency of cure in deeper areas when compared to the surface located closest to the light source. Theoretically, hardness at the bottom surface of a cured resin composite should be at least 80% (0.8) of the hardness at top surface. In the present study, blue LED curing of all the resin composites (except for microfilled composite), QTH curing of the nanocomposite, and PAC curing of the hybrid resin composite yielded ratios exceeding 0.8. Apart from these aforementioned curing regimes, the rest of the curing regimes did not fulfill this criterion.

Results of the present study were in good agreement with those of previous studies, where variables such as size, shape, distribution, and content per volume of the filler particles in the matrix have been shown to possess a direct influence on material strength, hardness, and modulus of elasticity of light-cured resin composites.

From the results, it appeared that higher hardness values were achieved with smaller filler particles, as particles impart rigidity. On this note, the nanocomposite yielded the highest VHN values at both surfaces regardless of the light source used. In the SEM images (Figs. 1–4), the difference between the nanoclusters and small nanometer-size particles of nanocomposite and the larger particles of the other products could be clearly seen. Individual nanoparticles of the nanocomposite sheared off from the zirconia-silica nanocluster agglomerates (Fig. 1).

To explain the results, one point to consider was the volume content of fillers. Since the filler volume content of nanocomposite was lower than that of hybrid resin composite, the resin-filler volume ratio of nanocomposite was higher than that of hybrid resin composite. As nanofillers have larger contact surface area with the organic phase when compared to microfilled composites, higher wetting of the filler by the resin was expected in the nanocomposite. Consequently, the material hardness was improved. The nanocomposite had a filler content of 78.5% by weight and 58–60% by volume, including non-agglomerated/non-aggregated 20-nm silica filler particles and loosely bound agglomerated zirconia-silica nanoclusters. The latter consisted of primary zirconia-silica filler particles of 5–20 nm in size. Cluster particle size ranged from 0.6 to 1.4 μm. The agglomerates act as a single unit enabling high filler loading and high strength. In a recent study by Beun et al., it was found that the filler shape was spherical—which meant that these advantages were present: increased filler load in composite, as well as enhanced fracture strength since mechanical stresses tend to accumulate on angles and protuberances. These abovementioned factors might thus explain why the nanofilled resin composite gave the highest VHN values among the materials tested.

Independent of the type of LCU used, a hybrid resin composite was found to be the second hardest material. The amorphous and crystalline-like phases appeared to coexist with the crystalline-like phase embedded in the amorphous phase (Fig. 4). This material contained SiO2 filler which integrated part of the resin matrix within the porous filler particles. This thus resulted in a firm bond between the filler particles and the matrix, which might also play a role in hardness.

Regardless of the LCU used, the microfilled and microhybrid resin composites yielded lower microhardness values at both surfaces than the other two materials. In particular, the lowest microhardness values were obtained for the microfilled resin composite cured with PAC LCU. The microfilled resin composite used in the current study had the lowest filler content (51.3% by weight; 40% by volume), and it has been found to comprise large aggregates of prepolymerized silica microfillers embedded in an organic matrix. Typically, microfillers are made from fumed silica prepared by a pyrogenic process. The structure of microfillers results in low filler loading. Most manufacturers thus add prepolymerized filled resin particles to increase filler loading. Despite using this process, microfilled composites have a substantially lower filler loading than hybrid composites, resulting in lower strength. Additionally, residual methacrylate groups bind the prepolymerized particles to the resin matrix. The effectiveness of this bond is impacted by the amount of residual double bonds on the surface of these particles. During the polymerization of the prepolymerized fillers, the reaction is driven to near completion. Hence, the bond of the prepolymerized filler particles to the resin is weaker than desired and breakdown frequently occurs at this interface. Our findings of this material were in parallel with those of other studies where microfilled resins were found to possess lower hardness values when compared to hybrid and nanofilled resin composites.

Besides filler type and filler loading, it should be borne in mind that the hardness of composites might
also be dependent on other factors such as the size of and distance between filler particles. The size of microfillers is extremely small — even smaller than that of the indenter of microhardness tester. Therefore, microfiller particles might not provide efficient reinforcement. With microhybrid resin composite, the regular size of filler particles ranged from 1 to 10 μm with average particle size at 0.85 μm. The SEM image of microhybrid resin showed that a single-phase polymer matrix reinforced with regular-sized filler particles (Fig. 3) had a more dominant effect than the particles — which had a dominant effect in microfilled resin composite. As a result, microhybrid resin composite had a higher hardness comparatively. Moreover, the small-sized particles that filled the gaps between the large-sizes particles of microfilled resin composite seemed to make polymerization more difficult (Fig. 2), thereby causing another setback to material hardness.

Inter-particle spacing might influence the physical properties of a resin composite: the smaller the distance, the higher the composite hardness. It has been stated that filler particles which were packed very closely protected the softer resin matrix from abrasives, thus reducing wear. On this account, reduced inter-particle spacing in composites was achieved by both decreasing the particle size and increasing the volume fraction of filler particles. In the current study, the overall results indicated that composite type had a significant effect on hardness values, because the composites used varied in their composition and type, size, and loading of fillers.

Apart from the characteristics of filler particles, the light source has been shown to influence the polymerization capacity of resin composites. The intensity and bandwidth of light output, as well as the curing time, are thought to be important parameters. In the present study, every material cured by QTH, LED, and PAC LCUs yielded microhardness values which were significantly different. Upon examining the VHN values, it became obvious that halogen-cured microfilled and hybrid resin composites yielded the highest values, whereas nanocomposite and microhybrid resin composites yielded the highest values when cured with LED LCU. However, PAC curing decreased the surface hardness of these materials. These results were generally compatible with the results of previous studies which evaluated the curing efficacy of light sources. There was also a good agreement that one of the light sources used in this study had the best curing effect on a particular composite type.

Statistical evaluation clearly showed that the LED LCU did not polymerize the microfilled and hybrid composites to the same extent as the QTH LCU. The latter material, Solitaire 2, had a coinitiator, 2,2-dimethoxy[1,2]diphenyletanone (DBM), in addition to the standard photoinitiator, CQ. The LED LCU might not have activated the coinitiator because of its narrow spectral range, which was 440–480 nm in the current study. This meant that in the LED curing of such materials, only the CQ photoinitiator participated in the polymerization process. Against this background, a greater degree of polymerization was thus achieved with QTH LCU, which has a broad spectral range of 400–500 nm.

The QTH LCU used in this study had a light output intensity (750 mW/cm²) lower than the other two LCUs (1000 mW/cm² for LED; 1600 mW/cm² for PAC). However, it had the highest energy density at 30 J/cm², as compared to 10 J/cm² for LED and 4.8 J/cm² for PAC (light energy = light intensity × curing time). Therefore, it was reasonable and logical to presume that the QTH unit would result in a higher degree of polymerization, thereby rendering composites with superior microhardness. However, the results in this study did not completely support this conjecture. On the contrary, the partial fulfillment of the conjecture in this study showed that two factors were inexplicably intertwined in influencing composite hardness: material composition and light characteristics of the light curing unit.

Another important criterion in comparing the curing efficiencies of LCUs is the hardness ratio of materials. In the present study, different hardness ratios of the same material were obtained when cured with different LCUs. Results revealed that the ratios of bottom surface hardness value/maximum top surface hardness value of all materials cured with QTH, LED, and PAC LCUs were less than 0.8, except for nanocomposite and microhybrid resin composite cured with LED LCU. Specifically, the hardness ratios of nanocomposite and microhybrid resin composite cured with QTH and LED LCUs were found to be 0.84 (59.38/70.80), 0.92, and 0.55 (21.08/38.60), 0.82, respectively — whereby the maximum top surface hardness value was obtained when these two materials were cured with LED LCU.

LED LCU has an emission spectrum similar to the absorption spectrum of CQ photoinitiator. This spectral homogeneity thus allows complete usage of the emitted light by LED LCU, which otherwise does not happen with halogen or plasma arc curing. It has been shown that blue light at different parts of the absorption spectrum of CQ produced different levels of curing efficiency, and that light near the absorption peak was more effective in curing. In light of this curing behavior, it thus explained why LED LCU rendered nanocomposite and microhybrid resin composite with higher microhardness values, as compared to QTH light curing of the same materials. This could be due to the convolution of the
absorption spectrum of the CQ photoinitiator present in these composites and the matching emission spectrum of the LED LCU\textsuperscript{20}.

At the topmost layer, sufficient light energy reached and activated the CQ photoinitiator, thereby initiating polymerization. With continued exposure, it helped to sustain the activation of photoinitiator molecules near the surface. Hence, exposure duration was another determining factor of the polymerization quality of resin composites\textsuperscript{15}. PAC LCUs with high light intensities were introduced to shorten the time spent by dentists at curing resin composites\textsuperscript{14,16,20,21}. However, the use of this LCU led to lower hardness values in this study. It could be argued that the short exposure time of PAC LCU led to such lackluster performance. Furthermore, the activation wavelength of the resin composites might be slightly outside the narrow wavelength spectrum of PAC\textsuperscript{7}. It should also be mentioned that with the greater light intensity of PAC LCU, a greater amount of free radicals might be generated for a given volume of a material. Rapid polymerization could reduce interpenetration of incompletely polymerization units\textsuperscript{17}. Therefore, when curing resin composites, an extended curing time might be required even with PAC to attain sufficient degree of polymerization.

It could be concluded that the microhardness of resin composites varied according to the type of resin composite as well as the light curing unit. In this study, the curing efficiency of different light sources was determined only by hardness test. To verify their true performance in deeper areas, a further study that entails infrared spectroscopic analysis may be necessary.

CONCLUSIONS

Within the scope of this study, the following conclusions were drawn:

1. Among the resin composites tested, the Filtek Supreme nanocomposite showed the highest microhardness values followed by the hybrid resin composite, Solitaire 2 — regardless of the light curing unit used.

2. The effect of light sources on microhardness was found to be material-dependent: blue light-emitted diode curing yielded the highest values for nanocomposite (Filtek Supreme) and microhybrid resin composite (Gradia Direct), whereas quartz-tungsten-halogen lamp curing gave the highest microhardness values for hybrid (Solitaire 2) and microfilled (Duraflill VS) resin composites.

3. Plasma arc curing according to manufacturer’s instructions gave the lowest microhardness values for all the resin composites tested.

4. Microhardness values at the bottom surface were found to be lower than those of the top surface for all the materials cured with the three light sources.

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