Correlation Between Degree of Conversion and Light Transmission Through Resin Composite Samples

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Povezanost stupnja konverzije i transmisije svjetla kroz uzorak kompozitnog materijala

Summary

Quantitative analysis of residual double bonds or free radicals in the polymer network is the most reliable method for determining the degree of conversion. In this study, measurements of the degree of conversion and light transmission of three different composite resin materials and two different shades (the lightest and the darkest shades) were evaluated. The correlation between the measurements of the degree of conversion and light transmission was observed. Absorption and scattering by filler particles would continue the light attenuation through a composite resin specimen and consequentially reduce the degree of conversion. There is a dependence on the amount of filler loading and composite resin shade. The results confirmed the hybrid materials and lighter shades to provide higher conversion than the microfilled composite resins as well as darker and more opaque shade. It was also found that illumination by a curing unit of low output (230 mW/cm²) should be prolonged and gradual increments should not exceed one mm.

Key words: *composite resins, degree of conversion, light transmission*

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Introduction

The efficiency of polymerization of visible-light cured composite resins is reflected by the number of double bonds in the methacrylate groups that have reacted during the polymerization process. (1) The degree of conversion could be defined as the extent to which monomers react to form polymers or as the degree to which carbon double bonds (C = C) are converted into carbon single bonds (C — C) (2). During the radical polymerization, the monomers polymerize to form a three-dimensional network containing double bonds and free radicals (3). It is known that residual double bonds in polymers make them less resistant to degradation reactions (4). Adequate curing of composite resins is of paramount importance to ensure optimal clinical performance (5). There are some reliable and relevant methods to determinate the conversion rate of a composite resin specimen, such as Electron Spin Resonance (ESR) (3, 6), Dynamic Mechanical Thermal Analysis (DMTA) (7), Infrared Spectroscopy (IR) (8, 9), Multiple Internal Reflection (MIR) (10), Attenuated Total Reflection (ATR) Infrared Spectroscopy techniques (11), Laser Raman Spectroscopy (12, 13) and Fourier-Transform-Raman Spectroscopy (14), Fourier-Transform Infrared Photoacoustic Spectroscopy (15) and Fourier-Transform Infrared Spectroscopy (FTIR) (16, 17). Some authors analyzed the relationship between the degree of conversion and the surface hardness (18, 19), thickness of scraped sample (20), strength (21, 22) and translucency (23).

The aims of this study were to determine correlation between the degree of conversion and the filler loading amount in resin composites as well as the dependence between the degree of conversion and shade of the material; and to compare the degree of conversion values with the light transmission measurements through the same types and shades of composite resin specimens.

Materials and methods

The tested materials were microfilled composite resins Helioprogress and Heliomolar 20 and 24 and hybrid composite materials Tetric 20 and 24 (Vivadent, Schaan, Liechtenstein). Two experiments were made to measure the degree of conversion and light transmission.

Measurements of the degree of conversion

The intention was to simulate the composite resin wafer at a particular depth. To ensure this, well cured overlays of 1.0 and 2.0 mm thickness were used. They were cured in a Spectramat PM 1830 (Ivoclar/Vivadent, Schaan, Liechtenstein), two minutes on each side. A thin wafer of uncured composite was placed between two Mylar sheets and pressed by 100 bar. A Heliolux II (Vivadent, Schaan, Liechtenstein) was used for curing the composite resin specimens through 1.0 and 2.0 mm thick overlays. The light output of the curing unit was 230 mW/cm^2, which was tested by a Curing Radiometer, Model 100 (Demetron Research Corporation, Danbury, CT, USA). A cured overlay was placed over the uncured specimen between the two Mylar sheets, and the fiber optic tip was clung to the overlay and illuminated for 40 s. The same experiment was repeated three times for both shades of each material. Cured specimens were stored in the dark at temperature of 37 °C for 24 h. After separation from the Mylar sheets, thin specimens were measured. The degree of conversion was measured using a FTIR Spectrometer (PERKIN ELMER 1600 series FTIR 1640) with eight scans at a resolution of 4.0 cm^{-1}. This method determines the ratio of aliphatic C = C absorption at 1640 cm^{-1} to aromatic C = C absorption at 1582 cm^{-1}. The aromatic C = C absorption spectrum was used as an internal standard. The spectrum of the uncured resin had been previously obtained under the same conditions as the cured specimens. The conversion rate of the cured composite resin specimen was calculated from the equivalent aliphatic/aromatic molar ratio of the cured (C) and uncured (U) specimens (24).

$$\% \text{ conversion} = (1\text{-}C/U) \times 100\%$$

The measured values of the degree of conversion were than analyzed by t-test II: paired data, at the levels of $p < 0.05$ and $p < 0.01$.

Measurements of light transmission

The composite resin specimens used for these measurements were 25 mm in diameter and 1.0 mm thick. They were cured in a Spectramat PM 1830, 2 min on each side. Three repeated measurements for each sample were performed. For transmission measurements, a Minolta Chroma Meter CT-310 was used. Illumination was provided by a pulsed xenon arc lamp, and the double-beam feedback system was used for greater accuracy. The pulse duration was 0.002. Calibration was done through distilled water. The mean transmission value of the light through the medium was 99.97%. Results obtained for the degree of conversion and light transmission were compared and analyzed.

Results

Figure 1 shows a comparison of the degree of conversion of the three composite materials: Helioprogress (HP), Heliomolar (HM) and Tetric (T) at the depths of 1.0 and 2.0 mm. They

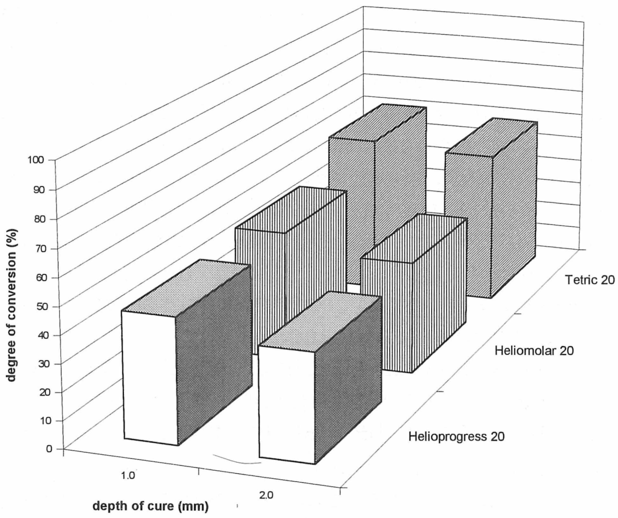

Figure 1. *Comparison between the degree of conversion of the light shade (20) of Helioprogress, Heliomolar and Tetric at the depths of 1.0 and 2.0 mm*

Slika 1. *Usporedba stupnja konverzije Helioprogressa, Heliomolara i Tetrica svijetle boje (20) na dubini od 1.0 i 2.0 mm*

differed in the amount of filler loading, but were of the same shade (light shade -20). The highest degree of conversion was obtained for Tetric 20 (61.07± 0.05) at the depth of 1.0 mm. The mean value achieved by Heliomolar 20 (48.49 ± 0.02) was higher than that for Helioprogress 20 (45.40 ± 0.01). At the depth of 2.0 mm, the values were lower. Tetric 20 showed the highest conversion rate (58.91 ± 0.07), but the values were not significantly different from those obtained at the depth of 1.0 mm. Heliomolar 20 showed a lower mean value (42.04 ± 0.14), which was significant at $p < 0.05$. Helioprogress 20 achieved the lowest values (38.94 ± 0.11).

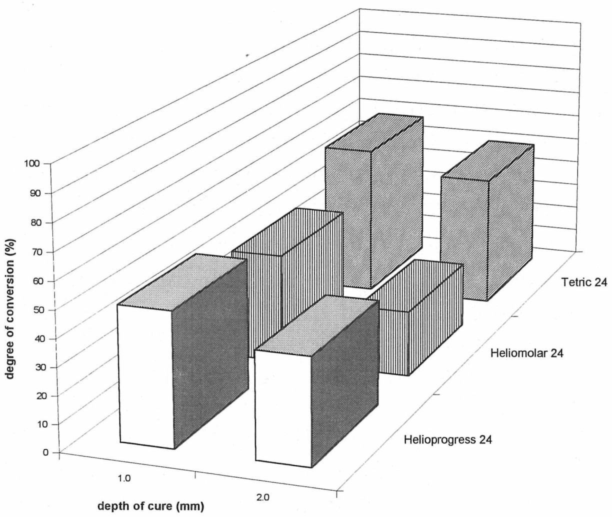

Figure 2. *Comparison between the degree of conversion of the dark shade (24) of Helioprogress, Heliomolar and Tetric at the depths of 1.0 and 2.0 mm*

Slika 2. *Usporedba stupnja konverzije Helioprogressa, Heliomolara i Tetrica tamne boje (24) na dubini od 1.0 i 2.0 mm*

Figure 2 shows a similar comparison for HP, HM and T 24 (dark shade) at the depths of 1.0 and 2.0 mm. All values were lower than those obtained for the light shade. The degree of conversion for Tetric 24 (57.96 ± 0.29 at 1.0 mm and 49.94 ± 0.34 at 2.0 mm depth) was higher at both depths than for Helioprogress (48.72 ± 0.05 at 1.0 mm and 38.51 ± 0.10 at 2.0 mm depth) and Heliomolar 24 (40.83 ± 0.13 at 1.0 mm and 24.49 ± 0.14 at 2.0 mm depth). The poorest conversion was recorded in Heliomolar 24. At the depth of 2.0 mm, the values were significantly lower ($p < 0.01$) than those obtained at the depth of 1.0 mm.

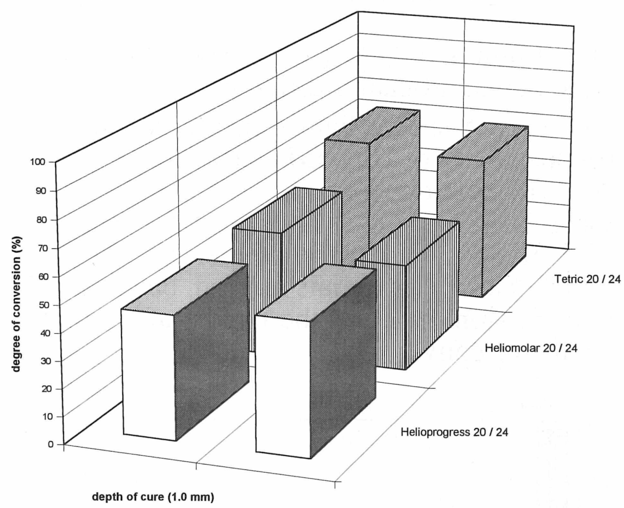

Figure 3. *Comparison between the degree of conversion of the light and the dark shades of the same material (HP, HM and T) at the depth of 1.0 mm*

Slika 3. *Usporedba stupnja konverzije svijetle i tamne boje istog materijala (HP, HM i T) na dubini od 1.0 mm*

Figure 3 presents a comparison between the light and dark shades of the same product at the depth of 1.0 mm. Light shades of all the three materials produced a higher degree of conversion than dark shades, which was significant for Heliomolar 20 ($p < 0.05$).

The same comparison at the depth of 2.0 mm is shown in *Figure 4*. All results were lower than at the depth of 1.0 mm. Heliomolar 24 showed significantly lower values ($p < 0.01$) at the depth of 2.0 mm as compared to the depth

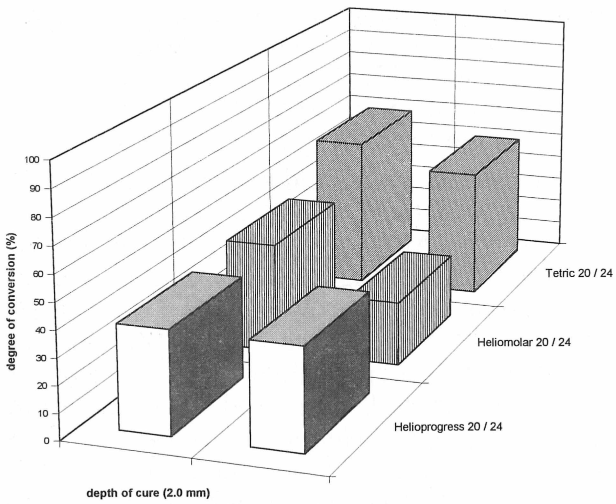

Figure 4. *Comparison between the degree of conversion of the light and the dark shades of the same material (HP, HM and T) at the depth of 2.0 mm*

Slika 4. *Usporedba stupnja konverzije svijetle i tamne boje istog materijala (HP, HM i T) na dubini od 2.0 mm*

of 1.0 mm and T 24 ($p < 0.05$). Other values were not significantly different.

Figure 5 shows light transmission through the specimens of HP, HM and T (shades 20 and 24). The best transmission value was obtained for T 20 (11.86) and the lowest for values of light transmission were higher for the light shade in all the materials studied, because light transmission is one of the factors affecting the degree of conversion.

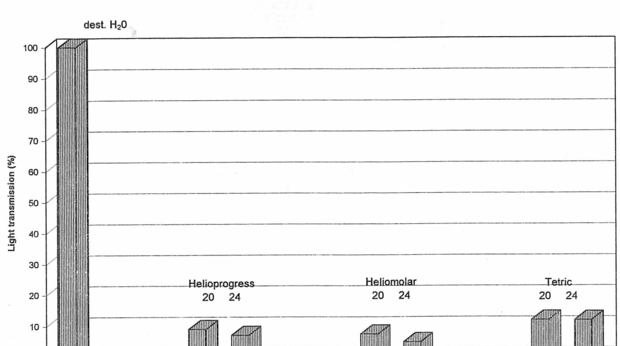

Figure 5. *Light transmission through distilled water and specimens of Helioprogress, Heliomolar and Tetric (shades 20 and 24)*

Slika 5. *Transmisija svjetla kroz destiliranu vodu i uzorke kompozitnog materijala (HP, HM i T) svijetle i tamne boje*

Discussion

The curing unit used for photopolymerization of composite resin specimens was of a rather low output (230 mW/cm^2). It was used on purpose because in clinical conditions, many factors affect the intensity of light-curing units. Some of these are breakage in the flexible glass bundles, frosting and blackening of the bulb, filters damaged by heat, careless handling of light guides, residual composite on the tips, and many other factors. All these factors diminish the light output of the curing unit (25). It is known that even a very poor light source is able to polymerize the surface of a restorative resin just as well as an effective one (26). This was the reason for excluding surface measurements and to perform them at the depths of 1.0 and 2.0 mm.

The aim was to evaluate whether a curing unit with 230 mW/cm^2 light output is capable of polymerizing the bottom of a 2.0-mm thick composite layer. The results obtained for T, the light and dark shades, were sufficient at both depths, but the conversion values of HP and HM at the depth of 2.0 mm were insufficient.

The analysis of the light transmission measurement results revealed a correlation between the degree of conversion and light transmission. This might be explained as follows: the intensity of the light source and the attenuating power of the material influence the degree of conversion (27). Absorption and scattering of the light by filler particles would continue the attenuation of the light through the composite resin specimen (28). The transmission of the light itself is influenced by the light wavelength and refractive indices of the resin and fillers, as well as by the size, shape and amount of filler particles. Thus, darker and more opaque shades might be expected to have a lower degree of conversion (29, 30).

The previously mentioned results confirmed the presence of dependence on the amount of filler content and on the pigment. The results revealed that microfilled resins (HP and HM) showed lower transmission values than the hybrid resin (T). Light scattering is related to the size and shape of filler particles. It is made up to three phenomena: surface reflection, refraction and difraction. According to Ruyter and Rysaed (31), maximal scattering occurs at a particle diameter of approximately $\chi/2$, i. e. about 0.25 μm (the wavelength ranges between 450 and 500 nm). Microfilled materials used in this study contain particles sized between 0.04

and 0.2 μm. The SiO_2-agglomerated filler particles are very close to the critical size of one-half of the wavelength, which can optimize the scattering and reduce the transmission and degree of conversion. To avoid great scattering and absorption, it would be useful to have a material containing the filler and resin matrix with similar refractive indices, with not too wide a range of particle size distribution in hybrid composites and more regular shape of filler particles. The pigmenting of the materials also greatly influences the absorption. Yellow pigment absorbs blue light which induces polymerization (32). It is known that unnecesarily high camphorquinone concentration leads to attenuation of radiation through the material (7).

Conclusion

1. Composite resins with a higher filler loading content provide a greater degree of conversion (hybrid composite resins);
2. lighter shades of the material allow better transmission of the light through the material and consequentially better degree of conversion;
3. better results are obtained at a lower depth (1.0 mm);
4. the intensity of light source is a critical factor at deeper layers; and
5. illumination by curing units with low light output has to be longer and composite layers have to be thinner (1.0 mm).

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POVEZANOST STUPNJA KONVERZIJE I TRANSMISIJE SVJETLA KROZ UZORAK KOMPOZITNOG MATERIJALA

Sažetak

Najpouzdaniji postupak određivanja stupnja konverzije jest kvantitativna analiza zaostatnih dvostrukih sveza ili slobodnih radikala u polimerskoj mreži. Svrha ovoga rada bila je procjena ovisnosti stupnja konverzije i transmisije svjetla kroz različite uzorke kompozitnih materijala. Stupanj konverzije određivan je Fourier-transform infracrvenom (FTIR) spektroskopijom, a transmisija svjetla Minolta Chromametrom. Za polimerizaciju kompozita rabljena je standardna halogena žarulja Heliolux II snage 230 mW/cm². Rezultati potvrđuju međusobnu ovisnost prethodno nave-denih parametara kao i ovisnost stupnja konverzije o količini anorganskog punila i boji kompozitnog materijala. Znano je da hibridne kompozitne smole ostvaruju viši stupanj konverzije od onih s mikropunilom. Materijali tamnih boja ne osiguravaju dostatnu polimerizaciju dubljih slojeva kom-pozitnog ispuna. Stoga je, u tim slučajevima, nužno rabiti žarulje jače snage, produžiti vrijeme izloženosti svjetlu i postavljati tanje slojeve kompozitnog materijala (1 mm).

Ključne riječi: *kompozitni materijali, stupanj konverzije, transmisija svjetla*

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