Influence of Polymerization Light Intensity on the Degree of Conversion of Composite Materials (Part I)

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Summary

Degree of conversion of composite materials is an important factor that influences both quality and longevity of the composite restoration during polymerization. Besides the components of the composite material, degree of conversion is greatly influenced by properties of the light source and polymerization time. The purpose of this study was to compare the effect of three polymerization modes of various intensities (medium, exponential and standard) of Elipar Trilight halogen curing (3M/ESPE, Seefeld, Germany) unit on samples of Tetric Ceram (Vivadent, Schaan, Liechtenstein) and Filtek Z 250 (3M/ESPE, Seefeld, Germany) on the surface and at 2 mm depth. For determining the degree of conversion, Fourier transform infrared spectroscopy (FTIR) was used. Results of the degree of conversion measurement showed the highest degree of conversion when using light source of highest intensity as well as a higher degree of conversion on the surface than at 2 mm depth regardless of the type of composite material and light intensity used for polymerization.

Key words: *composite materials, polymerization light source, degree of conversion.*

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Introduction

Light curing composite resins are polymerized by visible light in the 400-500 nm spectra. Polymerization of composite resins depends on various factors such as absorption and dispersion of light in the material itself, color and opacity of the composite material, type, ratio and size of inorganic filler particles, organic composition of the material, concentration and type of photo initiator, intensity of emitted light and polymerization time (1).

Some studies showed that dark shades of composite material exhibit a lower degree of conversion in relation to light shades (2). However, Ferracane et al. (3) proved that the degree of conversion of dark shades light polymerizing composite material equals the degree of conversion of light shades of light curing materials and that the degree of conver-

sion depends less on shade itself, but more on translucency of the material itself.

Sufficient polymerization of composite material represents a key factor in the longevity and quality of composite filling. Inadequate polymerization, whether because of thicker composite layer or inadequate light source, as a consequence has lower monomer to polymer conversion and higher portion of non-reacted double bonds which decrease the physical properties of the filling, increases water absorption and solubility and causes discoloration of the filling.

On the contrary, polymerization of composite materials using high intensity polymerization devices will sufficiently harden composite resin, but will also lead to greater polymerization shrinkage and stress resulting in microcracks as a consequence of shrinkage as well as greater increase in temperature that can compromise pulp vitality (4).

Intensity of the halogen polymerization device is difficult to determine based only on bright blue light intensity on the end of the optical cable. Hardness of the surface of the filling, often used in clinical practice as a subjective parameter for determining polymerization of composite materials, is also not a good parameter. Research showed that both weaker and stronger polymerization devices will harden the surface equally well but difference will be apparent at layers deeper than 2 mm where devices of weaker intensities will show almost no polymerization (5).

The degree of conversion of composite materials is measured by direct and indirect methods. Indirect methods encompass "scraping"and surface hardness, while direct methods include Infrared Spectroscopy and Laser Raman Spectroscopy. Direct methods are not used as often as indirect because they are more expensive, more complex and more time consuming. The degree of conversion measured by the mentioned methods varies on average between 43.5 to 73.8% (5, 6).

The purpose of this study was to determine the degree of conversion of two composite materials on the surface and 2 mm depth using FTIR, after polymerization with halogen curing unit with three polymerization modes of different intensity.

Materials and methods

In the study materials Tetric Ceram (TC) (color A1) (number B49177) and Filtek Z 250 (F) (color A1) (number 20000222) were used. Samples of the material were polymerized with Elipar Trilight halogen curing unit whose wavelengths of emitted light ranges between 400-515 nm. Elipar Trilight device has three operation modes:

1. "Medium mode" (ETM) - uniform light intensity of 450 mW/cm^2.
2. "Exponential mode" (ETE) - polymerization starts at 100 mW/cm^2 intensity and for 15 seconds gradually rises to 800 mW/cm^2 where it remains until the end of 40 seconds.
3. "Standard mode" (ETS)- uniform intensity of light at 800 mW/cm^2.

For the degree of conversion measurements a total number of 120 samples were prepared. For polymerization with each light source ten samples were prepared for each light source for both TC and F totalling in 30 samples for each material for the surface and 30 samples for each material for 2 mm depth. Samples polymerized with ETM mode were a control group.

Unpolymerized sample of the composite material was placed between two celluloid Mylar foils (2x2 cm in size) and pressed between two inox plates (diameter 2 cm) in a hand press at 107 Pa pressure to 0.1 mm thick.

For the surface degree of conversion measurements, blue light source was placed on the upper foil of the unpolymerized sample, placed between two foils and polymerized (40 for seconds TC, 20 seconds for F).

For measurements at 2 mm depth, over layers 2 mm thick were made for each material separately: unpolymerized sample was placed in the inox ring (diameter 1 cm) 2 mm thick, covered with Mylar foil from the upper and lower side and pressed in a hand press between two inox plates to 2 mm thickness. Over layer prepared in this way was polymerized in Spectromat PM 1831 (Ivoclar/Vivadent, Schaan, Liechtenstein) polymerization device that emits low intensity blue light so over the layer of composite material is polymerized for two minutes from each side.

For measurements at 2 mm depth, the unpolymerized sample of the composite material, prepared in the same way as the sample for measuring conversion at the surface of material, was covered with 2 mm thick over layer and light source was placed on the over layer. The samples were polymerized in the same way as when measuring surface conversion and the degree of conversion was measured after polymerization and separation from the Mylar foil (7, 8).

For determining the degree of conversion, FTIR spectra of polymerized samples and spectra of one unpolymerized sample TC and F composite sample needed as a parameter for calculating the degree of conversion, were recorded. FTIR spectra were recorded at room temperature and resolution 4 cm-1 with 20 repeat scans per sample using Perkin-Elmer spectrometer (model 2000) (Beaconsfield, Bucks, UK). Recording and analysis of the absorption spectra was performed using IRDM program (Infrared Data Manager). For calculating the degree of conversion, Rueggeberg method was used (9). Based on aliphatic/aromatic molar ratio of polymerized (P) and unpolymerized (U) sample, the degree of conversion was calculated using the following formula (9):

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

The statistical methods used in the study were descriptive statistical methods ANOVA and Post-hoc test for difference in the degree of conversion regarding light source, t-test for dependant samples for difference in the degree of conversion on the surface and at 2 mm depth in relation to the composite material tested.

Results

Results of the degree of conversion measurements obtained by statistical data analysis are shown in Tables 1-3. The degree of conversion for composite materials varies from 61 to 69% for surface polymerization, and from 57 to 66% for samples polymerized at 2 mm depth.

ANOVA and Post-hoc tests were used for observing the differences in the degree of conversion regarding each polymerization mode for F and TC materials (Table 1). 1. When material F was used the results showed there was a difference in the degree of conversion considering the polymerization mode on the surface and at 2 mm depth, highest degree of conversion on the surface and at 2 mm depth being when ETS mode was used and lowest when ETE mode was used. Post-hoc Scheffe test showed no difference on the surface between ETM and ETS ($p>0.05$), and at the 2 mm depth between ETM and ETE ($p>0.05$). 2. When the TC material was used it was also shown that difference exists in the degree of conversion regarding the polymerization mode. Post-hoc test did not determine difference on the surface only between ETM and ETS ($p>0.05$). At 2 mm depth no significant difference was observed between ETM and ETE modes ($p>0.05$). For TC the highest degree of conversion was also observed for ETS polymerization mode while lowest surface degree of conversion was observed when using ETE mode and at 2 mm depth when using ETM mode.

The difference in the degree of conversion was also studied with regard to specific composite material using t-test for independent variables (Table 2). 1. When using ETM polymerization mode difference in the degree of conversion was found between two materials ($p<0.05$) and at 2 mm depth ($p<0.05$) . TC had a higher degree of conversion for both surface and 2 mm depth. 2. When ETE polymerization mode was used, there was a difference in the degree of conversion between the two materials tested both on the surface and at 2 mm depth. TC had a higher degree of conversion at both depths. 3. For ETS polymerization mode there was a difference in the degree of conversion for both materials tested on the surface and at 2 mm depth. TC had a higher degree of conversion at both depths.

In this study, the difference in the degree of conversion between the surface and at 2 mm depth was also analyzed individually for TC and F materials for all three polymerization modes using t-test for dependent variables. (Table 3). 1. In the case of F composite material there was a statistically significant difference ($p<0.05$) in the degree of conversion for all three polymerization modes between the surface and 2 mm depth (higher degree of conversion was always at the surface). 2. TC composite material had statistically significant difference for all three

polymerization modes between the surface and at 2 mm depth ($p<0.05$) (higher degree of conversion, same as in the case of F at the surface).

Figures 1-4 represent absorption spectra of polymerization extent for both composite materials for all three polymerization modes on the surface and at 2 mm depth.

Discussion

The intensity of emitted light can be greatly decreased by various factors: variations in electric current, damaged or dirty filters, damaged and old optical fibers, damaged reflectors, break in fibro-optical fibers, repeated refills without previous complete discharge (batteries), stopping the polymerization before prolapsed designated time interval, damage or contamination of the tube as well as its damage by disinfective means, autoclave etc. (10).

Transmission or passage of light through the composite material and dispersion and refraction of light that results from such passage are phenomena caused by composition of the material which greatly influences depth of light passage and therefore the degree of conversion and depth of polymerization of the composite material. Greater dispersion causes reduced passage of light to the deeper sections of the material. Transmission of light through composite material increases with increase in wavelength from 400-700 nm, while dispersion increases with decrease in wavelength. As the light passes through composite material, it disperses on small filler particles and thus reduces the light passage. Greater dispersion of light inside the material causes smaller light transmission on smaller wavelengths. In order to avoid dispersion and light absorption in the material, the material should have approximately the same refraction index as organic matrix and filler, and the differences in sizes and distribution of inorganic filler particles should not be too great (1, 2).

Degree of conversion is proportional to the light intensity (11, 12). On the surface of the composite material where there is no above layer to interfere with the transmission of light, even the low intensity light sources can reach the degree of conversion equal to the high intensity light sources (13, 14). This study also confirmed the mentioned hypothesis because results show larger variance and differences between the degree of conversion at the surface and 2 mm depth. Literature states that a dominant factor for surface polymerization is not intensity of the polymerization device but exposure time. On the contrary, at 2 or 3 mm depth factors that influence composite material polymerization quality are thickness, exposure time, light intensity and their interaction (15).

In this experiment, polymerization mode of highest intensity, ETS, showed highest degree of conversion for both materials tested on the surface (TC 69.09%, F 64.67%) and at 2 mm depth (TC 65.60%, F 62.49%). Interestingly, at the surface level, polymerization by ETM mode for TC (68.77%) reached a higher degree of conversion compared to TC ETE mode (65.19%), while at 2 mm depth, results were almost the same. For F composite material, since according to manufacturer's instructions it needs to be polymerized for 20 seconds, at the surface better results were accomplished using ETM mode (64.15%) on the surface than ETE mode (62.35%) according to expectations because ETE mode has very low initial intensity (100 mW/cm^2) in relation to ETM mode.

Unterbrink and Muessner (16) and Uno and Asmussen (17) recommend devices with lower initial intensity to ensure slower polymerization, longer molecular chains with greater possibility of flow of the material during hardening. These authors showed that composite materials polymerized in this fashion achieve a sufficient degree of conversion and better properties than those polymerized with a polymerization device of higher intensity and shorter exposure time (18).

Insufficiently polymerized composite resin material decreases clinical value and longevity of the filling, and because of insufficient monomer to polymer conversion, non-reacted components can be toxic to the pulp and surrounding tissue. Monomer citotoxicity decreases during polymerization and after completion of the polymerization, it becomes non toxic. Therefore, this reason as a possible pulp irritant is excluded, and the main focus concentrates on infiltration of microorganisms through the pores created by polymerization shrinkage as well as temperature rise during polymerization because of light source

heat on one side and the dynamic of chemical reaction in the material itself on the other side (19).

Every clinical practitioner should be able to ensure by proper choice sufficient polymerization of the composite resin material with maximum decrease of unwanted side effects of the polymerization process.

Conclusion

Results of this study show that:

1. Composite materials polymerized by higher intensity polymerization modes produced a higher degree of conversion: highest degree of conversion was achieved with ETS polymerization mode both on the surface and at 2 mm depth for both composite materials tested.
2. Degree of conversion was higher on the surface than at 2 mm depth regardless of the composite material used and polymerization mode.