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The Effect of Curing Time on the Degree of Conversion of Poly-acid Modified Composite Resin (Compomer) "An *In Vitro* study"

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Abstract

Aims to evaluate the effect of light intensity on the degree of conversion of polyacid-modified composite resin.

Materials and methods: Fourteen samples were prepared and divided randomly to four groups according to Light Intensity. Fourier Transform Infrared Spectrometer (FTIR) is used in order to measure the degree of conversion.

Results and discussion: Analysis of variance (ANOVA) and Duncan multiple range test were used for analysis. The study showed that at 1mm depth the polyacid-modified composite resin has a difference in the degree of conversion at different intensities. As the light intensity increased, the degree of conversion measure was increased.

Keywords

Degree of conversion; FTIR, composite resin; Light Intensity

Introduction

Degree of conversion defined as the ratio between aliphatic (cured or reacted) and aromatic (uncured or unreacted), will affect many properties. Thus, it has been postulated that the degree of conversion plays an important role in determining the ultimate success of restoration [1-3].

The two most widely used techniques to assess the extent of polymerization in direct aesthetic dental restorative materials have been the physical determination of surface hardness and the direct chemical analysis of conversion by mid-infrared (mid-IR) spectroscopy [4-6].

The degree of conversion of restorative 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 [7].

Fourier Transformation Infrared Spectroscopy (FTIR) has been proven to be a powerful technique and has been widely used as a reliable method, as it detects the C=C bond stretching vibrations directly before and after curing of materials. However, to measure the DC of bulk material by FTIR, the procedure is time consuming, as the polymerized specimens need to be pulverized [8,9] the working principle of the FTIR is to find the absorption of energy at a wavelength or wave number to investigate the chemical structure of the material being tested. The machine can emit a wide range of infrared radiation [10].

Many factors influence the degree and adequacy of the polymerization process, such as the type and relative amount of monomers, filler and initiator/catalyst as well as the shade and translucency of the material, its temperature during polymerization, the wavelength and intensity of the incident light, and the irradiation time [11]. Moreover, the polymerization process of composite resins apparently continues for some time after irradiation (the so-called post-cure) [12].

Degree of conversion in photoactivated materials is related to the energy density delivered by the light unit (expressed in J/cm^2). Considering energy density as the product of the power density (expressed in mW/cm^2) by the exposure time (in seconds), it should be possible to obtain similar conversions using different combinations of these two parameters [12]. The use of low power densities has become widespread in clinical practice, as several studies have shown that the use of continuous low intensity curing routines, as well as those characterized by the reduced power density at the initial seconds, may lead to significant reductions in microleakage and gap formation in composite restorations [13].

This study aims to evaluate the effect of light intensity on the degree of conversion of polyacid-modified composite resin.

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Materials and Methods

The material used in this study was visible light-curing compomer restorative material Glasiosite Caps (VOCO/Cuxhaven, Germany) (Figure 1).

Fourteen samples were prepared and divided randomly to four groups according to light intensity. The degree of conversion is measured by using FTIR Spectrometer (Fourier Transform Infrared Spectroscopy) (Bruker 27, TENSOR, Germany) (Figure 2).

Group A (Light intensity groups)

Group A_1: Ten samples were cured with 20 sec. at intensity 410mW/cm^2.

Group A_2: Ten samples were cured with 20 sec. at intensity 490mW/cm^2.

Group C (control group): Ten samples were cured with 20sec. at intensity 450mW/cm^2.

Uncuring group: Ten samples remain uncured.

Sample preparation

For the evaluation of the ratio of conversion of the reacted C=C (aliphatic) to the unreacted C=C (aromatic), compomer samples were prepared by condensing the compomer material into a Polyethylene mold (Figure 3) (1mm depth and 5mm in diameter) placed on glass slab. After placing the compomer into the mold, it was covered by a celluloid strip and cured by conventional QTH light curing unit, at a distance of 1mm from sample to light cure tip (with exception of the thickness of the celluloid strip). For different intensities (mW/cm^2) [14,15]. The power intensity of the curing lights were 410 and 490 mW/cm^2, was controlled by a manual Intensity-Changeable device (Figure 4) and Digital Multimeter (Figure 5), as measured by means of a radiometer, the specimens were cured at 450 mW/cm^2 for 20 sec., after curing the material was removed from the Polyethylene mold and placed in a closed dark container then stored in the incubator for 24 hours at 37°C. Then after 24 hours the samples were removed from the incubator and separately crushed and grinned manually by the piston and mortar into a powder then the powder was mixed with potassium bromide at a weight percentage of 1:5. After mixing, the resultant powder was poured into a metal mold and compressed into a disc shape by Bruker press at a load of 10 tons and the samples become ready for measurement. While the uncured samples were prepared by placement on a special cell supplied by the manufacturers of Fourier transform infrared spectroscopy (FTIR), and becomes ready for measurement [16].

Figure 1: Light-curing compomer

Figure 2: Fourier transform infrared Spectroscopy

Figure 3: Polyethylene ring

Figure 4: Intensity-changeable device

Figure 5: Digital multimeter

Measurements of degree of conversion

The degree of conversion of the irradiated samples was measured

by Fourier Transform Infrared Spectroscopy (FTIR). The degree of conversion on the tested samples was calculated by the ratio between aliphatic (1637 cm^{-1}) and aromatic (1608 cm^{-1}) carbon double bonds peaks. The degree of conversion was calculated according to the formula [17,18]:

DC=**[** 1-{(aliphatic C=C)/(aromatic C=C)} of polymer **/** {(aliphatic C=C)/(aromatic C=C)} of monomer **]** × 100

Results and Discussion

The number of samples, mean and standard deviation of groups cured with different curing intensities (410 and 490 mw/cm^2) for the degree of conversion, were arranged respectively in (Table 1).

One-way analysis of variance (ANOVA test is a statistical method very commonly used in checking the significance and adequacy of the calculated linear regression model) showed the effect of time on the degree of conversion. It was obvious from this analysis that there was a significant difference ($p < 0.05$) in degree of conversion of poly-acid modified composite resin, with the exception that there was no significant difference in the degree of conversion of poly-acid modified composite resin between group C and group A2 as shown in (Figure 6).

This study showed that at 1mm depth, the polyacid-modified composite resin has a difference in the degree of conversion and the amount of residual monomer at different intensities. As the light intensity increased, the degree of conversion measure was increased.

Sufficient polymerization of restorative material represents a key factor in the longevity and quality of the filling. Inadequate polymerization, whether because of thicker filling layer or inadequate light source, as a consequence has a 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 [7].

Polymerization of restorative materials using high intensity polymerization devices will sufficiently harden resin, but will also lead to greater polymerization shrinkage and stress resulting in micro-cracks as a consequence of shrinkage as well as a greater increase in temperature that can compromise pulp vitality. The intensity of the halogen polymerization device is difficult to determine based only on bright blue light intensity at the end of the optical cable [7].

Any wavelengths bellow 430nm and above 500nm are not utilized in the electron promotion of the ketone groups in CQ and therefore it can be said that CQ ignores these wavelengths. The unwanted wavelengths do produce additional heat, affecting the kinetics of the reaction and may thereby influence the reaction. Conventional light sources produce a white light, which is then filtered in an effort to remove the unwanted wavelengths [3,19].

Another explanation for the effect of light intensity was found when several samples from this study were polymerized in low intensity (390mw/cm^2), they have a degree of conversion less than 10% with high residual monomer.

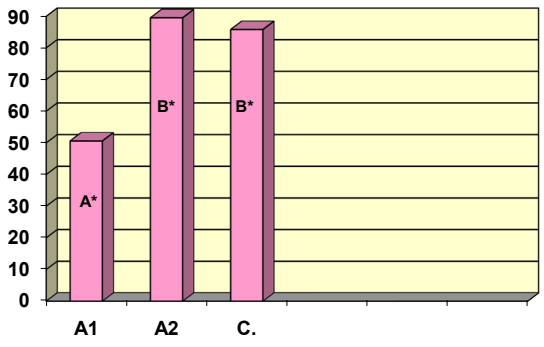

Figure 6: Histogram of the degree of conversion

Name	Number	Mean	Std.deviation
Group A_1	10	50.62	14.79
Group A_2	10	89.52	1.46
Group C	10	85.83	2.79

Table 1: Mean, standard deviation and number of groups utilized in degree of conversion

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