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The Curing Efficiency and Mechanical Properties of Light-Activated Acrylic Resin

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Summary: The aim of this investigation was to determine degree of conversion, and flexural and compressive strength of photocurable acrylic resin. The acrylic resins based on bis-phenol A glycidyl dimethacrylate (BisGMA) and triethylene glycol dimethacrylate (TEGDMA) were formulated and then irradiated with red light to form a solid crosslinked polymer. Various post curing conditions were studied to investigate their effects on curing efficiency. Degree of conversion was analyzed by means of Fourier Transform Infrared Spectroscopy (FTIR) to monitor the quantity of remaining acrylic double bond in the cured resin. Three-point bending and compressive strength were tested using Universal Testing Machine (UTM) to evaluate the mechanical performance. Our investigation demonstrated that the formulated acrylic resins were possibly polymerized under irradiation with red light and degree of conversion and mechanical properties were closely correlated.

Keywords: light-activated acrylic resin; photopolymerization

Introduction

Photoinitiated acrylic resins have been widely used as biomaterials for several decades. The most used photoinitiator, especially in dental materials is camphorquinone (CQ), coupled with tertiary amine as a reducing agent. This photoinitiator has a light absorbing range between 400–500 nm corresponding with blue light. The use of blue light has been largely accepted in medical field although it evidently caused some damage to retina.[1–2] In the field of tissue engineering, red light which has less energy is then a proper light source than blue light since it does no harm to human tissues. Using red light emission between 500–700 nm, the photocurable polymer seeded with human living cells could be polymerized without cell damaging. In this study, the photocurable acrylic resins, sensitive to red light were formulated. The effects of post-curing on degree of conversion and mechanical properties as well as the stability of the light-cured acrylic resins were investigated.

Experimental Part

Photocurable Acrylic Resin and Specimen Preparation

Bis-phenol A glycidyl dimethacrylate (BisGMA, Esstech) and triethylene glycol dimethacrylate (TEGDMA, Esstech) at the weight ratio of 7/3 BisGMA/TEGDMA were blended with 0.1-wt% photoinitiator (H-Nu-Blue 640 from Spectra Group Ltd., USA). The resins were further added with certain chemicals such as benzoyl peroxide (BPO, Merck), cumene hydroperoxide (CPO, Fluka) and N,N-dimethylaminoethyl methylacrylate (DMAEMA, Fluka).

To prepare specimens, the resin for a given formulation was irradiated with red light at wavelength of 630 nm for 30 min. The specimens were then kept at room temperature for 1 day before post curing at 70 °C or 100 °C for 1 h.

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Degree of Conversion Measurements

Degree of conversion was measured using Fourier Transform Infrared Spectrometer (Perkin-Elmer). The cured specimens in disc shape with 6 mm in diameter and 3 mm in thickness were ground in liquid nitrogen to produce fine powder. The powder was mixed with a high purity potassium bromide (KBr) and pressed into a transparent pellet. The uncured resins were smeared between the KBr windows. The degree of conversion (DC) was calculated from the peak height ratio of (C=C)1640 cm^{-1}/(C=C) 1610 cm^{-1} of cured and uncured samples as follows:

$$\mathrm{DC}\,(\%) = 100 - (\text{peak height ratio}_{\text{cured}} / \text{peak height ratio}_{\text{uncured}} \times 100) \quad (1)$$

Flexural Strength and Flexural Modulus Measurements

The rectangular bar-shaped specimens, 35 mm in length, 5 mm in width, and 3 mm in thickness were placed on a three-point bending fixture with a span length of 20 mm. The force was then applied using Universal Testing Machine (Model 4502, Instron Corp.) at a crosshead speed of 1.0 mm/min. Four specimens were tested for each of resin formulation.

Compressive Strength and Compressive Modulus Measurements

The square-shaped specimens, 10 mm in length and width, and 3 mm in thickness were subjected to load at a crosshead speed of 10.0 mm/min using Universal Testing Machine (Model 4502, Instron Corp.). Six specimens were tested for each of resin formulation.

Results and Discussion

Degree of Conversion

IR spectra of cured and uncured samples are shown in Fig. 1. As polymerization proceeded, the peak intensity at 1640 cm^{-1} representing aliphatic double bond reduced.

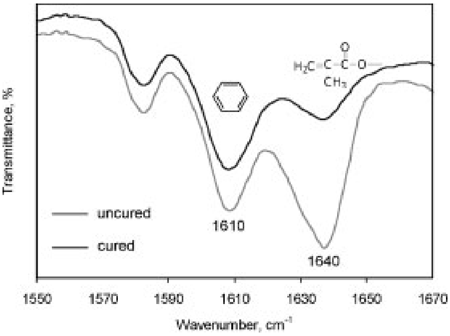

Figure 1.
IR spectra of cured and uncured samples.

DC indicating the extent of polymerization was then determined from the reducing amount of aliphatic double bond with reference to aromatic double bond (1610 cm^{-1}) which did not evolved in the polymerization reaction.

The Effect of Post Curing

Degrees of conversion of the cured resins at different post curing conditions are reported in Table 1. The results showed an increase in DC as post-curing temperature increased (sample No.1–3). At high temperature, the remaining unreacted monomers were possibly leached out or further polymerized which resulted in increasing conversion.

Mechanical properties were found dependent on DC, as observed from the increase of flexural modulus from 526 MPa (56.96% DC) to 1417 MPa (71.88% DC) when heated sample from 70 °C to 100 °C. As a result, the post curing of specimen was chosen to do at 100 °C to obtain high curing

Table 1.
Degree of conversion at different post curing conditions

No.	Added Chemicals (-wt% of resin)	Post curing condition	DC (%)
1	None	No heat	45.28
2	None	Heat at 70 °C	56.96
3	None	Heat at 100 °C	71.88
4	0.2% BPO	Heat at 100 °C	64.63
5	1.0% CPO	Heat at 100 °C	70.63
6	8.0% DMAEMA	Heat at 100 °C	71.22
7	8.0% DMAEMA, 1.0% CPO	Heat at 100 °C	82.17

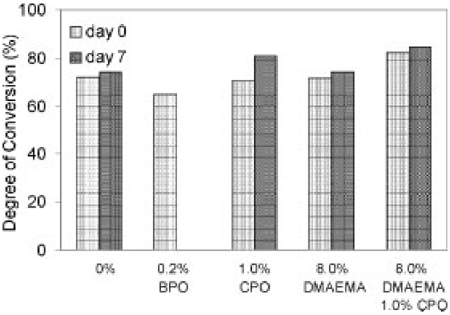

Figure 2.
Degree of conversion of the cured resins at day 0 and day 7.

efficiency. DC was further increased by adding DMAEMA, coupled with CPO into the photocurable resin (sample No.7). The addition of either DMAEMA or CPO (sample No.5–6) had no significant effect on DC whereas the addition of BPO (sample No.4) reduced DC. BPO was found to deactivate photoinitiator, as observed from the change in resin color with time. This resulted in poor photoinitiation, and hence poor DC.

The Stability of the Photocurable Resins

The stability of the photocurable resins was determined by measuring DC and mechanical properties of freshly prepared resins in comparison with 7 day-old resins. DC, flexural and compressive properties of each formulated resin are presented in Figure 2–4, respectively.

All resins exhibited good stability, except the one added with BPO which could not be cured after storing the resin for 7 days. This was a result of the reaction between BPO and the photoinitiator as mentioned earlier. Expectedly, the resin added with DMAEMA/CPO which had the highest DC yielded the best flexural and compressive properties. In contrast, the DMAEMA added resin showed very poor mechanical properties uncorrelated with its

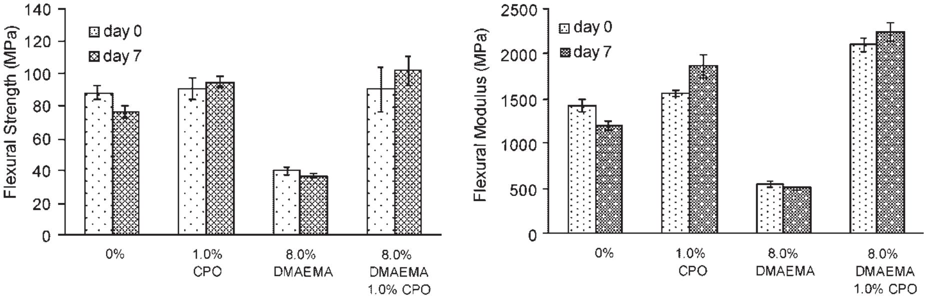

Figure 3.
Flexural strength and modulus of the cured resins at day 0 and day 7.

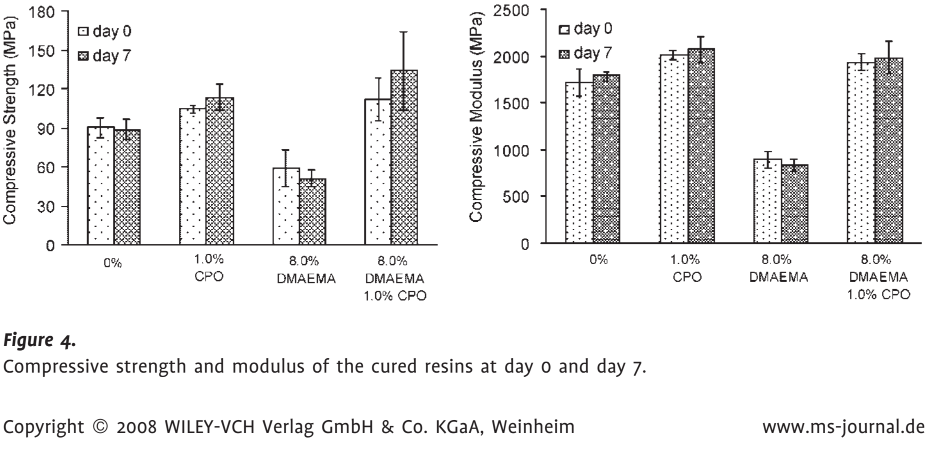

Figure 4.
Compressive strength and modulus of the cured resins at day 0 and day 7.

DC. It implied that DMAEMA without CPO did not take part in polymerization but behaved as a plasticizer reducing mechanical strength.

Conclusions

The formulated acrylic resins using H-Nu-Blue 640 as a photoinitiator could be polymerized when irradiated with red light. DC and mechanical properties of the cured resins were found closely correlated. Increased DC by post curing the specimens at high temperature (100 °C) led to the increase in mechanical properties. This conversion was further increased when DMAEMA/CPO was added.

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