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# UV Curable Coatings: Effect of Diluents with 2, 2-Bis-[4-(2-hydroxy-3-acryloxy prop-1-oxy) phenyl] propane

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**Abstract :** Nine different formulations were prepared from the prepolymer, 2,2-bis-[4-(2-hydroxy-3-acryloxy prop-1-oxy)phenyl] propane with various functional diluents namely, monofunctional diluent, Di(ethylene glycol)ethylether acrylate, difunctional diluent, tri(ethylene glycol) dimethacrylate and trifunctional diluent, trimethylolpropane ethoxylate (1-ethoxylate/hydroxyl) triacrylate in the presence of photoinitiator, 2, 2-dimethoxy-2-phenylacetophenone. These solutions were coated on the glass plates and then cured under ultraviolet radiation. The kinetics of photopolymerization, percent double bond conversion and rate of polymerization were studied by Fourier Transform Infrared Spectra. The results show that increasing irradiation time increases degree of conversion, whereas the rate of polymerization increases suddenly and then decreases. The gel content (%), hardness and density of the photocrosslinked polymers were studied. The swelling coefficient, crosslink density and molecular weight between crosslinks of the photocured polymers were determined by swelling measurement using N, N'–dimethyl formamide as a solvent. It is found that density, hardness and the gel content increases with increase of functionality in the diluents.
**Keywords :** Ultraviolet radiation, photocrosslinked polymers, Fourier Transform Infrared spectra, percent double bond conversion, rate of polymerization, gel content (%).

## Introduction

In recent years, photopolymerization is widely used in several industrial fields, coatings, graphic arts and micro electronics[1-5]. An important advantages offered by this technology are high speed, reduced release of volatile organic compounds and low coast of equipments[6-7]. The photocrosslinked polymers have excellent mechanical properties, thermal properties, inertness to solvents and abrasion resistance due to high degree of crosslinking during polymerization[8-11].

An ultraviolet (UV) curable coating is composed of the prepolymer, photoinitiator and diluent. In the past years, many workers studied the formulation of curable systems which have been carried out to have effective photoinitiators and highly reactive diluent[12]. These two components in UV curing technology promote the cure speed, extent of polymerization and improve the final properties of the cured materials[13-15]. The diluent in the formulation of UV curing perform different functions that they act as a solvent for the prepolymer, as a controlling agent, to control the tack and viscosity and also as crosslinking agent. The diluent is used to determine nature of surface coating and photoresponce speed in conjunction with the photoinitiator. The functionality of diluent influences the hardness, scratch resistance, thumb twist resistance, reactivity and maximum degree of conversion[16].

The present work is to, study the effect of various diluents namely monofunctional diluent, di (ethylene glycol) ethylether acrylate (DEGEEA), difunctional diluent, tri (ethylene glycol) dimethacrylate (TEGDMA) and trifunctional diluent, trimethylolpropane ethoxylate (1EO/OH) triacrylate (TMPEOTA) on percent double bond conversion, rate of polymerization, swelling coefficient, crosslink density, molecular weight between crosslinks, density, hardness and gel content (%) with the prepolymer, 2, 2-bis-[4-(2-hydroxy-3-

acryloxypropoxy) phenyl] propane (BISGA) in the presence of photoinitiator, 2, 2-dimethoxy-2-phenylaceto phenone (Irgacure 651) under UV light exposure.

## Experimental

### Materials

The diluents, DEGEEA, TEGDMA, TMPEOTA, prepolymer, BISGA and photoinitiator, Irgacure 651 were purchased from Aldrich (U.S.A) and used without further purification. The solvent, N, N'–dimethyl formamide (DMF) used in the swelling measurement was a SRL assured product.

### Methods and measurement

The photocurable formulation containing a prepolymer, a diluent and a photoinitiator were irradiated under medium pressure mercury vapour lamp with intensity of 125 W/ $cm^2$. To study the effect of diluent, different formulations were prepared by mixing the prepolymer, BISGA (90%), photoinitiator, Irgacure 651 (2.5%) and different concentration of the diluent, 10%, 30% and 50% of DEGEEA separately. The same procedure was also followed for other two diluents, TEGDMA and TMPEOTA to carry out the reactions. The percent double bond conversion of the photocrosslinked samples were determined by using Bruker IFS 66V FT-IR Spectrometer. The gel content (%) and swelling studies were carried out using the solvent, DMF. The swelling coefficient, crosslink density and molecular weight between crosslinks were calculated. The relative hardness of the photocured polymers were determined by A Type-JIS K-6301 hardness tester.

## Results and Discussion

### Percent double bond conversion

The photocrosslinkable formulations and list of photocrosslinked polymers obtained from the prepolymer, BISGA with the monofunctional diluent (DEGEEA), difunctional diluent (TEGDMA) and trifunctional diluent (TMPEOTA) in the presence of the photoinitiator (Irgacure 651) are given in Table 1.

**Table 1. Photocrosslinkable formulations and list of photocrosslinked polymers obtained from the prepolymer, BISGA with various functional diluent**

| **Formulations** | | | **Irradiation time (min)** | | | | |
|---|---|---|---|---|---|---|---|
| **DEGEEA[a] (%)** | **BISGA (%)** | **Irgacure 651 (%)** | **1** | **2** | **3** | **4** | **5** |
| 10 | 90 | 2.5 | $M_1$ | $M_2$ | $M_3$ | $M_4$ | $M_5$ |
| 30 | 70 | 2.5 | $M_6$ | $M_7$ | $M_8$ | $M_9$ | $M_{10}$ |
| 50 | 50 | 2.5 | $M_{11}$ | $M_{12}$ | $M_{13}$ | $M_{14}$ | $M_{15}$ |
| **Formulations** | | | **Irradiation time (min)** | | | | |
| **TEGDMA[b] (%)** | **BISGA (%)** | **Irgacure 651 (%)** | **1** | **2** | **3** | **4** | **5** |
| 10 | 90 | 2.5 | $D_1$ | $D_2$ | $D_3$ | $D_4$ | $D_5$ |
| 30 | 70 | 2.5 | $D_6$ | $D_7$ | $D_8$ | $D_9$ | $D_{10}$ |
| 50 | 50 | 2.5 | $D_{11}$ | $D_{12}$ | $D_{13}$ | $D_{14}$ | $D_{15}$ |
| **Formulations** | | | **Irradiation time (min)** | | | | |
| **TMPEOTA[c] (%)** | **BISGA (%)** | **Irgacure 651 (%)** | **1** | **2** | **3** | **4** | **5** |
| 10 | 90 | 2.5 | $T_1$ | $T_2$ | $T_3$ | $T_4$ | $T_5$ |
| 30 | 70 | 2.5 | $T_6$ | $T_7$ | $T_8$ | $T_9$ | $T_{10}$ |
| 50 | 50 | 2.5 | $T_{11}$ | $T_{12}$ | $T_{13}$ | $T_{14}$ | $T_{15}$ |

a-monofunctional diluent; b-difunctional diluent; c-trifunctional diluent

The percent double bond conversion, DC (%) of the double bonds reacted was calculated from the following Equation[17].

$$DC(\%) = \left\{1 - \frac{\left(\frac{A_{acrylate\ C=C}}{A_{aromatic}}\right)\ crosslinked\ polymer}{\left(\frac{A_{acrylate\ C=C}}{A_{aromatic}}\right)\ prepolymer}\right\} \times 100$$

The decrease in the intensity of acrylated C=C absorbance ($A_{acrylate}$) at 1630-1645 $Cm^{-1}$ was monitored. The phenyl absorbance ($A_{aromatic}$) at 1600-1615 $Cm^{-1}$ was used as the internal standard. The prepolymer to diluent ratio followed for many industrial applications varies between10: 90, 30: 70 to 50: 50 by weight[18]. To formulate for the investigation on the cure characteristics, three different ratios were chosen. The effects of the various functional diluents such as DEEEA, TEGDMA and TMPEOTA on the percent double bond conversion are given in Table 2.

**Table 2. Effect of diluent functionality and diluent concentration on the percent double bond conversion**

| **DEGEEA (%)** | **Percent double bond conversion** | | | | |
|---|---|---|---|---|---|
| | **Irradiation time (min)** | | | | |
| | **1** | **2** | **3** | **4** | **5** |
| 10 | 14 | 22 | 27 | 29 | 31 |
| 30 | 24 | 38 | 45 | 50 | 54 |
| 50 | 46 | 64 | 72 | 75 | 78 |
| **TEGDMA (%)** | **Percent double bond conversion** | | | | |
| | **Irradiation time (min)** | | | | |
| | **1** | **2** | **3** | **4** | **5** |
| 10 | 12 | 16 | 21 | 25 | 28 |
| 30 | 19 | 31 | 38 | 42 | 46 |
| 50 | 30 | 47 | 57 | 64 | 69 |
| **TMPEOTA (%)** | **Percent double bond conversion** | | | | |
| | **Irradiation time (min)** | | | | |
| | **1** | **2** | **3** | **4** | **5** |
| 10 | 10 | 14 | 17 | 22 | 26 |
| 30 | 15 | 27 | 34 | 39 | 41 |
| 50 | 21 | 36 | 45 | 51 | 54 |

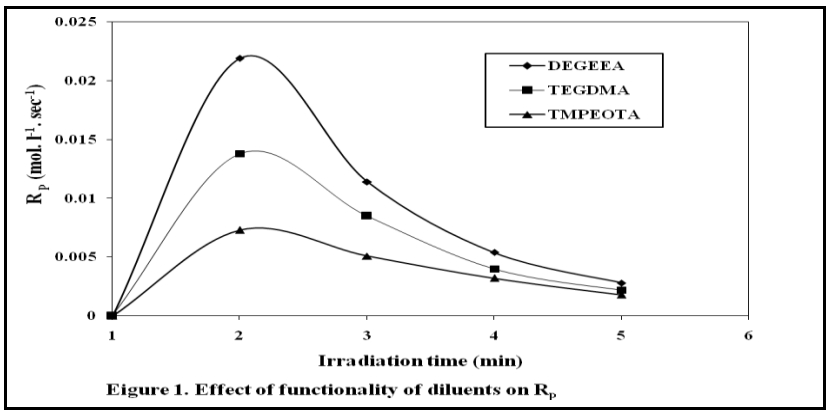
The results indicate that the percent double bond conversion of the diluents for DEGEEA is 78% and for TEGDMA and TMPEOTA is 69% and 54%, respectively. For monofunctional diluent, DEGEEA by keeping photoinitiator concentration (Irgacure 651, 2.5%) and irradiation time (5 min) as constant varying the prepolymer, BISGA to diluent, DEGEEA concentration as 90: 10, 7: 30 and 50: 50, the conversion was found to be 31%, 54% and 76%, respectively. The increase in percent double bond conversion may be due to the decrease in the viscosity of the system, which is more favorable for the reaction. In a similar way, the same trend was observed for the diluent, DEGEEA, TEGDMA and TMPEOTA. By comparing the rate of conversion form the diluent, DEGEEA, TEGDMA and TMPEOTA, the order of reactivity of diluent is as follows: tri < di < mono[19].

**Rate of polymerization ($R_p$)**

Rate of polymerization ($R_p$) was calculated using by the following Equation[19].

$$R_p = [M_0] \times \left[\frac{(A_{810})t_1 - (A_{810})t_2}{(A_{810})t_0\ (t_2 - t_1)}\right]$$

where, [Mo] is the monomer concentration before irradiation, $(A_{810})\ t_0$ , $(A_{810})\ t_1$ and $(A_{810})\ t_2$ represent the absorption due to carbon-carbon double bond before and exposure during $t_1$ and $t_2$ time, respectively. The effect of functionality of the diluents (DEGEEA, TEGDMA and TMPEOTA) by keeping diluent concentration (30%) and photoinitiator concentration (2.5%) constant on rate of polymerization is given in Figure 1.




**Eigure 1. Effect of functionality of diluents on $R_p$**

The rate of polymerization depends on the bond conversion. The monofunctional diluent, DEGEEA has higher $R_p$ than TEGDMA and TMPEOTA which may be due to higher percent double bond conversion of DEGEEA than that of TEGDMA and TMPEOTA.

**Density of photocrosslinked polymer**

Density of the photocured polymers were measured by using the Formula.

$$\text{Density} = \left(\frac{a}{a-b}\right) \times d$$

where, a and b are weight (gm) of photocrosslinked polymer in air and isopropyl alcohol respectively and d is density of isopropyl alcohol. The density data of the photocrosslinked polymers are given in Table 3. Results show that the increasing irradiation time, functionality and diluent concentration increase density, which may be due to the increasing crosslink density.

**Table 3. The influence of diluent functionality and diluent concentration on density of photocrosslinked polymer**

| Functionality of diluent | DEGEEA (%) | Density (g. $dm^{-3}$) | | | | |
|---|---|---|---|---|---|---|
| | | Irradiation time (min) | | | | |
| | | 1 | 2 | 3 | 4 | 5 |
| Mono | 10 | 0.813 | 0.819 | 0.825 | 0.828 | 0.832 |
| | 30 | 0.820 | 0.827 | 0.831 | 0.834 | 0.838 |
| | 50 | 0.832 | 0.836 | 0.840 | 0.843 | 0.849 |
| Di | TEGDMA (%) | Density (g. $dm^{-3}$) | | | | |
| | | Irradiation time (min) | | | | |
| | | 1 | 2 | 3 | 4 | 5 |
| | 10 | 0.840 | 0.844 | 0.850 | 0.854 | 0.861 |
| | 30 | 0.845 | 0.853 | 0.861 | 0.866 | 0.869 |
| | 50 | 0.851 | 0.859 | 0.869 | 0.873 | 0.877 |
| Tri | TMPEOTA (%) | Density (g. $dm^{-3}$) | | | | |
| | | Irradiation time (min) | | | | |
| | | 1 | 2 | 3 | 4 | 5 |
| | 10 | 0.846 | 0.849 | 0.855 | 0.858 | 0.867 |
| | 30 | 0.850 | 0.856 | 0.864 | 0.872 | 0.875 |
| | 50 | 0.855 | 0.863 | 0.876 | 0.880 | 0.885 |

**Swelling study**

Photocrosslinked polymer (50 mg) was added with DMF (7 ml). The amounts of maximum swollen sample were found out by employing different time intervals (6, 12, 18, 24, 30, 36, 42, 48, 54 and 60 hrs). The swelling studies were carried out in DMF at 25 ± 0.05 ℃ . The amount of maximum swelling was obtained by plotting the amounts swollen sample versus the swelling time. The swelling coefficient (Q) was calculated using the following formula[20].

$$Q = \frac{\text{Weight of in the swollen polymer}}{\text{Weight of in the swollen polymer}} \times \frac{\text{Density of DMF}}{\text{density of polymer}}$$

The crosslink density (υ) and molecular weight between crosslinks (Mc) were determined by using Flory-Reihner's Equation[20].

$$\upsilon = \frac{Vr + x V_r^2 + \ln(1 - Vr)}{d_r V_0 \left(V_r^{\frac{1}{3}} - \frac{V_r}{2}\right)}$$

where, υ = crosslink density (effective number of moles of crosslinked units per gram of crosslinked polymers, $V_r = 1/ (1+Q)$, the volume fraction of polymer, $d_r$ = density of polymer, x = polymer-DMF interaction parameter (lattice constant 0.34, for $\delta_s = \delta_p$, $V_0$ = molar volume of DMF. The solubility parameter of the solvent, DMF ($\delta_s$), which imparts maximum swelling for the polymers, was taken as the solubility parameter of the polymer ($\delta_p$).

$$\text{Molecular weight between crosslinks (Mc)} = \frac{1}{\upsilon}$$

The swelling coefficient, crosslink density and molecular weight between crosslinks of the crosslinked polymers obtained from BISGA with various functional diluents are presented in Table 4. The result shows that as irradiation time increases, crosslink density increases, whereas swelling coefficient and molecular weight between crosslinks decreases. When diluent concentration varied from 10% to 50%, the crosslink density increases, whereas swelling coefficient and molecular weight between crosslinks decreases. In this study, it is predicted that the viscosity of the system affects the mobility of the propagating macro-radicals, consequently high viscosity systems may show reduced reactivity due to the mobility restrictions of the propagating macro radicals and hence crosslink density decreases. With respect to the functionality of the diluents form mono to tri, crosslink density increases, whereas the swelling coefficient and molecular weight between crosslinks decreases.

**Table 4. The effect of diluent functionality and diluent concentration on swelling coefficient, crosslink density and molecular weight between crosslinks**

| **DEGEEA (%)** | **Swelling coefficient (Q)** | | | | | **Crosslink density (υ × $10^{-5}$)** | | | | | **Molecular weight between crosslinks (Mc × $10^3$)** | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | **Irradiation time (min)** | | | | | **Irradiation time (min)** | | | | | **Irradiation time (min)** | | | | |
| | **1** | **2** | **3** | **4** | **5** | **1** | **2** | **3** | **4** | **5** | **1** | **2** | **3** | **4** | **5** |
| 10 | 8.6 | 8.4 | 8.1 | 7.4 | 6.9 | 8.9 | 9.5 | 9.9 | 11.2 | 13.1 | 11.2 | 10.6 | 10.0 | 8.9 | 7.7 |
| 30 | 8.3 | 7.3 | 7.3 | 6.7 | 6.2 | 10.6 | 10.6 | 11.9 | 13.7 | 15.6 | 10.4 | 9.4 | 8.4 | 7.3 | 6.4 |
| 50 | 6.9 | 6.3 | 5.9 | 5.7 | 5.6 | 13.3 | 15.3 | 16.8 | 17.6 | 18.5 | 7.6 | 6.5 | 6.0 | 5.7 | 5.4 |
| **TEGDMA (%)** | **Swelling coefficient (Q)** | | | | | **Crosslink density (υ × $10^{-5}$)** | | | | | **Molecular weight between crosslinks (Mc × $10^3$)** | | | | |
| | **Irradiation time (min)** | | | | | **Irradiation time (min)** | | | | | **Irradiation time (min)** | | | | |
| | **1** | **2** | **3** | **4** | **5** | **1** | **2** | **3** | **4** | **5** | **1** | **2** | **3** | **4** | **5** |
| 10 | 8.4 | 8.0 | 7.3 | 6.5 | 5.8 | 9.4 | 10.3 | 11.6 | 14.2 | 17.4 | 10.7 | 9.8 | 8.6 | 7.0 | 5.7 |
| 30 | 7.6 | 7.4 | 6.2 | 5.7 | 5.1 | 11.0 | 11.3 | 15.4 | 18.1 | 22.7 | 9.1 | 8.8 | 6.5 | 5.5 | 4.4 |
| 50 | 5.9 | 5.4 | 5.1 | 4.9 | 4.4 | 17.2 | 19.6 | 21.9 | 23.6 | 28.1 | 5.8 | 5.1 | 4.6 | 4.2 | 3.5 |
| **TMPEODA (%)** | **Swelling coefficient (Q)** | | | | | **Crosslink density (υ × $10^{-5}$)** | | | | | **Molecular weight between crosslinks (Mc × $10^3$)** | | | | |
| | **Irradiation time (min)** | | | | | **Irradiation time (min)** | | | | | **Irradiation time (min)** | | | | |
| | **1** | **2** | **3** | **4** | **5** | **1** | **2** | **3** | **4** | **5** | **1** | **2** | **3** | **4** | **5** |
| 10 | 8.0 | 7.4 | 6.7 | 6.0 | 5.5 | 9.8 | 11.6 | 12.6 | 16.8 | 19.1 | 10.2 | 8.7 | 7.9 | 6.0 | 5.2 |
| 30 | 6.7 | 6.4 | 5.8 | 5.4 | 4.7 | 13.7 | 14.6 | 17.0 | 19.7 | 24.6 | 7.3 | 6.9 | 5.9 | 5.1 | 4.1 |
| 50 | 5.9 | 5.7 | 5.2 | 4.6 | 4.3 | 17.2 | 18.2 | 21.4 | 26.1 | 26.1 | 5.8 | 5.5 | 4.7 | 3.8 | 3.4 |

**Gel content (%)**

100 mg of the photocrosslinked polymer was added to 75 ml acetone and the mixture was allowed to stand for 72 hrs. The swollen sample was isolated by the Soxhlet extractor and subjected to vacuum drying for sufficiently long time to ensure complete drying. The gel content (%) was determined by using the Formula[12].

$$\text{Gel content (\%)} = \left(\frac{W_a}{W_b}\right) \times 100$$

where, $W_a$ and $W_b$ are weights of original and extracted sample respectively. The influence of various diluent functionality and diluent concentration on the gel content (%) are shown in Figure 2-4.

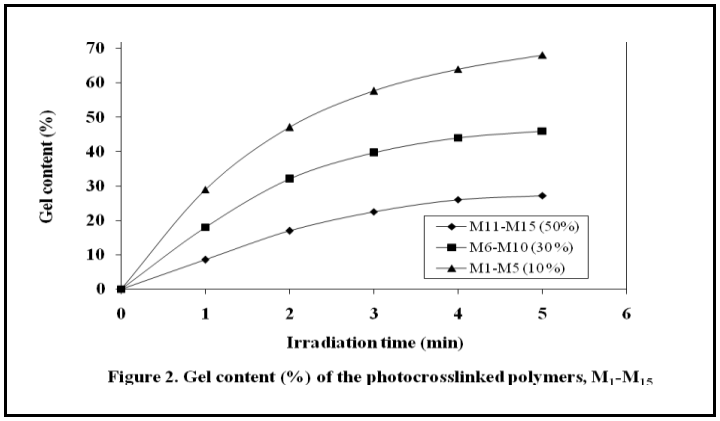



**Figure 2. Gel content (%) of the photocrosslinked polymers, $M_1$-$M_{15}$**

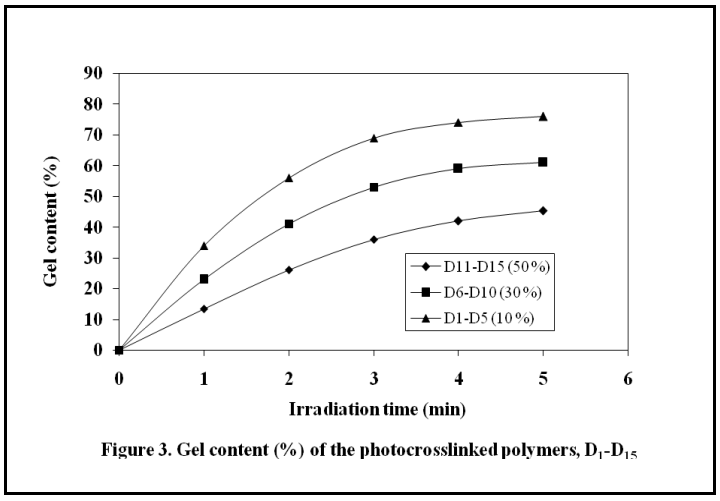



**Figure 3. Gel content (%) of the photocrosslinked polymers, $D_1$-$D_{15}$**

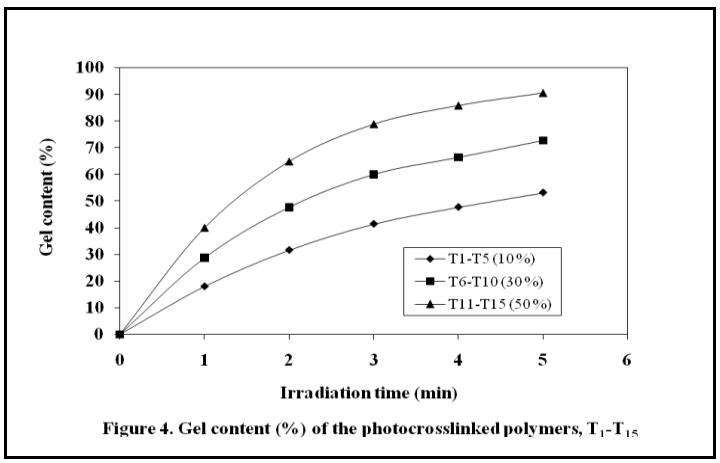



**Figure 4. Gel content (%) of the photocrosslinked polymers, $T_1$-$T_{15}$**

It can be seen that the gel content (%) increases with increasing irradiation time. The observation shows that the percent gel content is proportional to the percentage crosslinking. For mono functional diluent, DEGEEA by keeping photoinitiator concentration (Irgacure 651, 2.5%) and irradiation time (5 min) as by varying the concentration of the prepolymer, BISGA to diluent, DEGEEA as 90: 10, 70: 30 and 50: 50, the gel

content (%) was found to be 68%, 46% and 26%, respectively. The decrease in gel content may be due to increase in crosslink density. In similar way, the same trend was also observed for the diluents of TEGDMA and TMPEOTA. It was observed that if the functionality of diluents increases the gel content also increases.

**Hardness testing**

The relative hardness of the photocured polymer films was determined by A-Type- JIS K-6301 hardness tester. For this study, a polymer thickness of 15 mm was used. The readings were taken at several places on the same polymer sample by pressing the needle of the hardness tester. The average value was taken for the hardness of the sample under tester. The effect of various diluent functionality and diluent concentration on hardness of the photocrosslinked polymers are presented in Table 5.

**Table 5. The effect of diluent functionality and diluent concentration on hardness of photocrosslinked polymers**

| **Functionality of diluent** | **DEGEEA (%)** | **Hardness (degree shore)** | | | | |
|---|---|---|---|---|---|---|
| | | **Irradiation time (min)** | | | | |
| | | **1** | **2** | **3** | **4** | **5** |
| **Mono** | 10 | 70 | 73 | 78 | 80 | 85 |
| | 30 | 66 | 70 | 75 | 78 | 80 |
| | 50 | 61 | 65 | 68 | 72 | 77 |
| **Di** | **TEGDMA (%)** | **Hardness (degree shore)** | | | | |
| | | **Irradiation time (min)** | | | | |
| | | **1** | **2** | **3** | **4** | **5** |
| | 10 | 76 | 79 | 81 | 84 | 90 |
| | 30 | 71 | 75 | 79 | 86 | 89 |
| | 50 | 63 | 74 | 76 | 83 | 85 |
| **Tri** | **TMPEOTA (%)** | **Hardness (degree shore)** | | | | |
| | | **Irradiation time (min)** | | | | |
| | | **1** | **2** | **3** | **4** | **5** |
| | 10 | 80 | 83 | 85 | 89 | 94 |
| | 30 | 75 | 78 | 82 | 90 | 92 |
| | 50 | 72 | 76 | 79 | 87 | 90 |

The result shows that the hardness increases with increasing irradiation time is due to increasing of the crosslink density of the polymer. For a mono functional diluent, DEGEEA by keeping photoinitiator concentration (Irgacure 651, 2.5%) and irradiation time (5 min) constant by varying the concentration of the prepolymer, BISGA to diluent, DEGEEA as 90: 10, 70: 30 and 50: 50, the hardness values were found to be 85, 80 and 77, respectively. The decrease in hardness value may be due to decrease in crosslink density of the polymer. In similar way, the same trend was observed for the diluents of TEGDMA and TMPEOTA. It was observed that if the functionality of diluents increases then there is an increase in the hardness, which is also due to increase in crosslink density of the polymer.

## Conclusion

The conclusion of the present work may be summarized as follows: percent double bond conversion increased with increasing irradiation time as well as diluent concentration and it was observed that the prepolymer from the diluents, TEGDMA and TMPEOTA had lower double bond conversion than DEGEEA. The rate of polymerization increased suddenly and then decreased with increasing irradiation time and diluent concentration. The hardness, density, gel content (%) increased with increasing irradiation time and functionality of diluent. It was also found that the increasing diluent concentration, hardness and gel content (%) decreased. The swelling study showed that the crosslink density increases with increasing irradiation time, diluent concentration and functionality of diluent, whereas swelling coefficient and molecular weight between crosslinks decreased.

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