

Ultra-Violet Curable Resin Based on Palm Oil: Determination of Reaction Condition and Characterization of the Resin

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ABSTRACT: In this study, a UV curable resin based on palm oil was prepared by modifying palm oil with glycerol and glycidyl methacrylate (GMA). First, monoglyceride (MG) was prepared from a mixture of palm oil and glycerol at a ratio of 1 : 2 based on equivalent basis. The MG was treated with GMA to introduce unsaturated groups to MG chain via etherification between epoxy group (from GMA) and hydroxyl group (from MG). The amount of catalyst needed, reaction temperature, and time for this chemical modification were determined. The completion of reaction between GMA and monoglyceride was measured using Fourier transform infrared (FTIR) spectroscopy by monitoring the disappearance of the epoxy group in FTIR spectrum. An increment at peak 1630 cm^{-1} in FTIR spectrum was observed when more GMA was used in the chemical modification. Viscosity test and gel permeation chromatography analysis were carried out to characterize the resin produced. The resin was then cured using UV radiation and the properties of the cured resin were measured. From the results, the gel content of the cured resin was more than 97%. The hardness of the MG-GMA film was influenced by the amount of GMA used. © 2012 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 000: 000–000, 2012

KEYWORDS: ultra-violet curable resin; palm oil; hydroxyl number; gel content

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INTRODUCTION

Vegetable oils have attracted considerable attention as a raw material for polymeric material preparation. The main intention of using vegetable oil is to replace traditional use of petroleum-based raw material due to environmental issue. Vegetable oils such as linseed oil and tung oil have long found various uses in the paint and varnishes industries. Soybean oil, safflower oil, sunflower oil and canola oil have also been used in the preparation of resin.^{1–7}

In Malaysia, the total oil palm plantation area had been increased from 2.3 million hectares in 1997 to 4.1 million hectares in 2005.^{8–10} Palm oil is a nondrying oil which cannot be polymerized or dried when exposed to air. It can only be polymerized when an effective and suitable modification is conducted to introduce the appropriate reactive groups. Thus, it is believed that by attaching a functional group to palm oil molecule, the behavior of palm oil could be changed from noncurable palm oil to a curable product. The technology of palm oil in the coatings industry area is still new and constantly in development, hence, more research and developments need to be carried out. Besides, due to environment and legislation constraints, coatings industry is moving through a major revolu-

tion. Research efforts are directed towards reducing the volatile organic components (VOC) which has proven to have an effect on the environment and human health depending on the chemicals used. Radiation curing is now being increasingly used in various sectors of applications, mainly in the coating industry, graphic arts, and microelectronics, replacing conventional thermally curable solvent-based coatings, inks and adhesives. Besides the ecological aspect of minimizing the volatiles organic compounds (VOCs), ultra fast cure speed, room temperature operation, consequent low energy consumption, and high-quality end products are additional aesthetic and economic factors of noteworthy significance.^{3,5} Thus, UV curable coating is experiencing an important growth in the marketplace as well as in the research area.

Various studies have been carried out using vegetable oils for the preparation of radiation curable resins. Wan Rosli et al.² prepared epoxidized palm oil (EPO) and cycloaliphatic diepoxide which can be cured by UV radiation. Mei Yee¹¹ reported that radiation curable acrylates resin can be derived from epoxidized oils by reacting them with acrylic acid. EPO has also been shown to have a number of commercially realizable end uses such as additives in the plastic industry, as an effective starting material to make polyols and prepolymers in surface coatings

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formulations. Another invention for radiation curable product was palm oil-based acrylated polyester resins for wood coatings. According to Ali et al.,¹ palm oil incorporated with trimethylol propane triacrylate (TMPTA) or (1,6-hexanediol diacrylate) (HDDA) monomers were found to be most suitable for wood coatings application. Hilmi et al.¹² reported that acrylated palm oil could be employed as a prepolymer in free radical polymerization with encouraging results.

In this study, monoglyceride (MG) produced from palm oil is chemically treated with glycidyl methacrylate (GMA) to introduce unsaturated groups to MG (MG-GMA). Glycidyl methacrylate (GMA) is one of the monomers that can be used to chemically attach to various monomers/polymers due to its dual functionality—an acrylic group and an epoxy group on both ends of the molecule. GMA has easily transformable oxirane groups, which can also be modified and used to form a crosslinked structure via chemical reactions. Therefore, this monomer has been used to prepare several types of polymer; in coatings and adhesives as well as matrix for composite preparation.^{13–15} The MG modified with GMA (MG-GMA) was mixed with different percentages of photoinitiator to study the extent of reaction of the former. Other parameters studied relative to GMA modification of MG were reaction temperature, reaction time, and the amount of catalyst.

MATERIALS

A commercially available palm oil was used in this study. GMA and triethylamine were purchased from Sigma-Aldrich (M) Sdn Bhd. Photoinitiator, Irgacure 184 (Hydroxy-cyclohexyl-phenylketone) was obtained from Ciba Specialty Chemicals.

Preparation of MG and Palm Oil-Based Resin

MG was produced using alcoholysis process. Palm oil was treated with glycerol at a ratio of 1 : 2 (equivalent basis) in the presence of basic catalyst (0.1% sodium hydroxide) with a slow stream of nitrogen gas for 3 h at 220–235°C. Hydroxyl number of the MG was determined using phthalation reagent¹⁶ to calculate the percentage of hydroxyl groups available for etherification process. Hydroxyl (OH) number is defined as the number of milligram of potassium hydroxide equivalent to the hydroxyl content of the one gram of the sample. The hydroxyl group is esterified with a solution of phthalic anhydride in pyridine. The excess reagent is back titrated with standard sodium hydroxide solution and a blank is run on the reagents to determine the amount of anhydride consumed. The OH number and OH content can be calculated by using the equations shown below.

$$\text{OH number} = \frac{56.1 \times N.\text{NaOH} \times (\text{mL blank} - \text{mL sample})}{\text{sample weight (in gram)}}$$

$$\text{OH content (\%)} = \frac{\text{OH number}}{33}$$

The MG was then treated with GMA (based on equivalent weight) at different temperatures varied from 70 to 90°C with different amount of triethylamine (catalyst). In addition, 5% of hydroquinone was added to prevent premature polymerization of unsaturated group from GMA. The reaction time was also

varied to determine the time needed for completion of reaction. This was carried out using Fourier transform infrared (FTIR) spectroscopy.

Fourier Transform Infrared Spectrophotometer

The completion of reaction between MG and GMA was monitored using Nicolet's AVATAR 360 Fourier transform infrared (FTIR). The absorbance at 910 cm^{-1} representing the epoxy group of GMA was monitored. The scanning of the FTIR spectrophotometer was carried out from 4000 to 400 cm^{-1} with 64 scans for each sample. Conversion ratio (CR) of epoxy group is referred to the ratio of residual monomer function to the initial amount which is shown as below:

$$\text{Conversion (\%)} = 100 \times \frac{1 - A_t}{A_o}$$

where, A_t and A_o are the areas of the 910 cm^{-1} peak at time t and $t = 0$, respectively.

Viscosity Test

The Brookfield DV-II+ Pro Programmable viscometer was used to measure the viscosity of MG-GMA. It measures the torque required to rotate an immersed spindle in a fluid at a constant rotational speed and temperature (28°C). The measured torque is directly related to the viscosity of MG-GMA; which is automatically calculated and displayed by the viscometer.

Gel Permeation Chromatography

Weight average molecular weight (M_w) of MG-GMA was determined using Water 2414 gel permeation chromatograph, a differential refractive index meter as detector and a series of three styragel columns (2 styragel® HR 5E THF and a styragel® HR 4 Tetrahydrofuran (THF)). THF was used as an eluent, and polystyrene standards were employed for calibration. For the results to be valid, a sample must be kept at a constant temperature, achieved by keeping the columns constant at 35°C in a column oven, while all processed data were controlled by a Breeze software. All samples were dissolved in THF at concentration of 0.2% by weight. Each sample dissolved in THF was filtered through a 0.45- μm syringe filter prior to the injection. Totally, 20 μL of sample was injected into the column run time took \sim 45 min at a flow rate of 1.0 mL/min.

Preparation of Coating Films from Palm Oil-Based Resin

Palm oil based resin was mixed with different amount of photoinitiator (Irgacure 184) ranging from 1 to 5%. The optimum amount of photoinitiator to cure the prepared palm oil based resin was determined from the result of gel content. After the resin and initiator were well mixed, the mixture was coated on a glass plate and subsequently cured by IST Strahlentechnik UV machine with lamp type M-200-U2C, which is a medium pressure mercury vapor lamp (1–2 atm) generating UV-radiation in the range of 180–450 nm. The conveyor belt speed was set at 5 m/min.

Gel Content Determination

The gel content or degree of curing was calculated as follows (ASTMD2765-95, 1998). After UV irradiation, the film was conditioned for 24 h in a desiccator. Then, the prepared film was placed in a cellulose extraction thimble for soxhlet extraction

Table I. Hydroxyl Number and % OH of MG

Sample	Hydroxyl number	% OH
Palm oil	0	0
MG	312	9.45

with methyl ethyl ketone as solvent for 2 h. The film was taken out and dried in an oven and conditioned for 24 h before the determination of gel content using equation shown below.

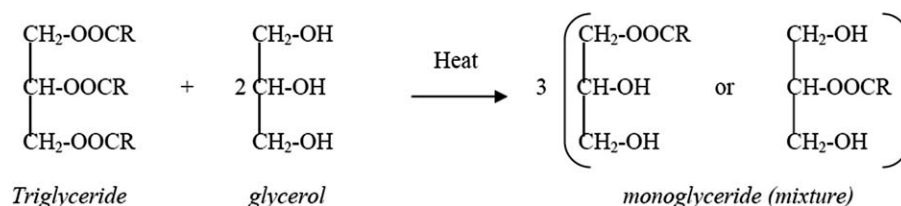
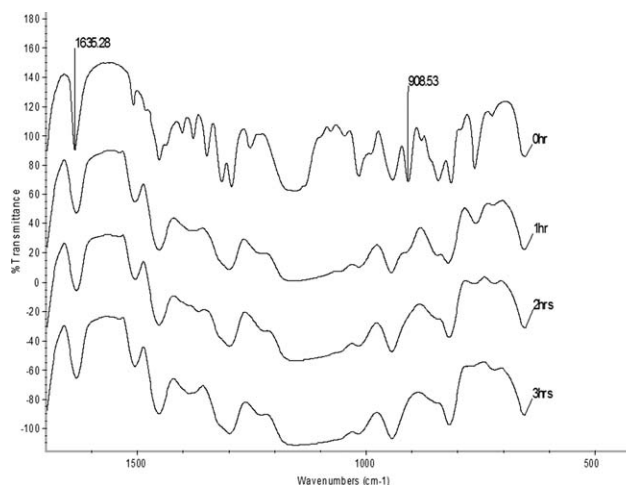
$$\text{Gel content (\%)} = \frac{\text{Weight after extraction}}{\text{Weight before extraction}} \times 100$$

Pendulum Hardness

The hardness of UV cured sample was determined according to the ASTM D4366-95 (1999) Standard Test Methods for Hardness of Organic Coatings by Pendulum Damping Test, test method A-König Pendulum Hardness Test. The test was performed with ERICHSEN-make Pendulum Damping Tester Model 299/300. In this test, a pendulum made contact with a coated panel through two steel balls. As the pendulum swung back and forth through a small angle, movement of the balls caused some deformation on the coating near the surface. The instrument was equipped with an automatic counter and acoustic signal. The digital value on the counter indicated the number of oscillations executed by the pendulum in changing the angle from 6 to 3°.

RESULTS AND DISCUSSION

Table I depicts hydroxyl number of Palm oil and MG prepared from palm oil and glycerol. The result indicates that no hydroxyl group is available in palm oil. After it was undergone alcoholysis process, the resulted MG had about 10% hydroxyl group. It is expected as shown by Figure 1 where 2 moles of glycerol are able to produce 3 moles of MG which each possess two hydroxyl groups, i.e. primary hydroxyl group and secondary hydroxyl group. This hydroxyl group is then subjected to reaction with GMA prior to curing process. According to Senila Güner et al.,¹⁷ there are two methods which can be used to produce alkyd resin from vegetable oil, i.e., alcoholysis process and fatty acid process. For alkyd resin preparation, alcoholysis process or monoglyceride process involves two stages. In the first stage, free hydroxyl group will be produced which will then be esterified by a polyacid for polymer preparation in the second stage. As for fatty acid process, polyacid, polyalcohol, and fatty acid will be mixed and heated in a single stage to obtain the alkyd resin. Alkyd resin prepared from fatty acid process has

**Figure 1.** Preparation of MG using alcoholysis process.**Figure 2.** FTIR spectra of MG and GMA mixture at 90°C with 2% TEA.

higher viscosity, good drying, and hardness properties.¹⁷ However, alcoholysis process was chosen because the temperature of the reaction in the second stage can be controlled to prevent premature polymerization of unsaturated group from GMA or MG-GMA before it was exposed to UV radiation.

Alcoholysis (transesterification) process is a chemical reaction between triglycerides and alcohol/glycerol in the presence of catalyst to produce mono-esters and glycerol. This reaction actually results in a mixture of unreacted glycerol, monoglyceride (MG), diglycerides (unconverted vegetable oil). This process has been widely used to reduce the high viscosity of triglyceride. The composition depends on the ratio of glycerol to oil and on catalyst, temperature, and time. In general, the reaction is not taken to equilibrium.^{18–22}

DG has two fatty acid chains whereas MG has one fatty acid chain attached to the glycerol molecule. In other words, DG is a di-ester whereas MG is a monoester. Hence, it can be said that MG and DG has two hydroxyl groups and one hydroxyl group available, respectively for further chemical reactions. Typically, this reaction is an additional or condensation reaction to produce a condensation polymer. The reactant may carry reactive functional groups which may interact with MG or DG and subsequently produce a reactive polymer.

Figure 2 represents different FTIR spectra of MG and GMA mixture (GMA/MG ratio = 2) with variable reaction time at a temperature of 90°C with 2% of triethylamine (TEA) as a catalyst. In these spectra, transmittance peak at 910 cm⁻¹ which represents epoxy group is monitored. It can be seen that the

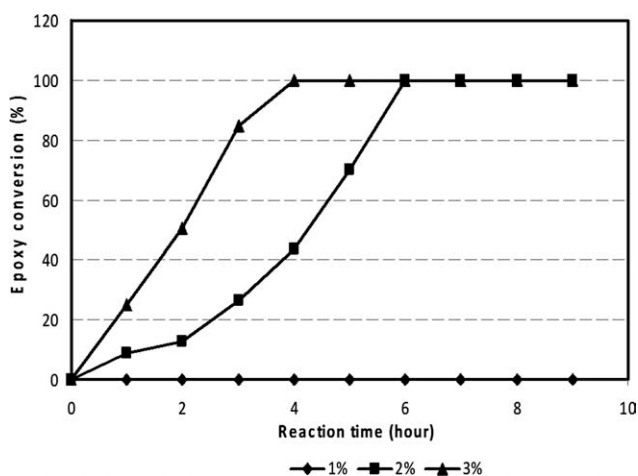


Figure 3. The effect of reaction time and percentage of catalyst (TEA) on the percentage of epoxy conversion.

transmittance peak at 910 cm^{-1} disappears after the mixture is heated for 1 h. In addition, the intensity of transmittance peak at 1635 cm^{-1} , representing unsaturated group ($\text{C}=\text{C}$) is also reduced. From this observation, it can be said that the epoxy group from GMA has been successfully attached to MG via etherification process. The spectra show that the completion of reaction between MG and GMA is detected at 2-h reaction for 90°C with 2% of TEA. It is also interesting to note that some unsaturated groups from GMA or MG-GMA have reacted, although with the presence of hydroquinone in the mixture.

Figure 3 shows the effect of time and amount of catalyst added on epoxy conversion, which is based on the area of the 910 cm^{-1} peak at 70°C . It can be seen that no reaction has taken place with 1% of TEA throughout 9 h of reaction. With 3% TEA, 100% epoxy conversion is achieved in 4 h. This shows that the reaction between epoxy group from GMA and hydroxyl group from MG or MG-GMA has completed. Hence, it can be said that 3% of TEA is sufficient to complete the reaction in 4 h at a temperature of 70°C . Figure 4 depicts the effect of reac-

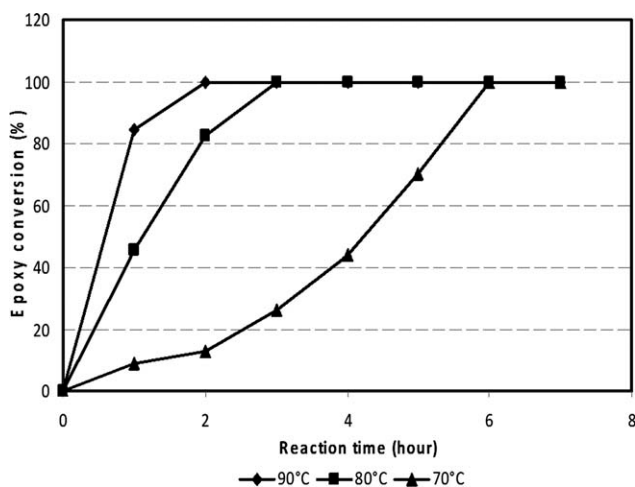


Figure 4. The effect of reaction time and temperature on the percentage of epoxy conversion with 2% of TEA.

Table II. Summary of the Effect of Temperature and Amount of Catalyst on MG and GMA Reaction Time

Temperature ($^\circ\text{C}$)	% of Catalyst (triethylamine)		
	1	2	3
90	6 h	2 h	2 h
80	>15 h	3 h	3 h
70	-	6 h	4 h

tion time and temperature on the percentage of epoxy conversion based on the mixture GMA/MG ratio = 2 and 2% of TEA. From the result, it is noticed that the reaction is complete at the end of 2 h at 90°C . The summary of the effects of the parameters is presented in Table II. Thus, the results indicate that temperature and the presence of catalyst affect the percentage of epoxy conversion in MG-GMA reaction. Through theory of collision, the increase in temperature may increase the possibility of both MG and GMA to come together and react. While, the presence of catalyst, in this case TEA, may reduce the activation energy, thus, facilitating the reaction of both components. Tertiary amine (TEA) was used as a catalyst because it has been found to enhance the tendency of etherification process.²³ The proposed interaction between GMA and MG is shown in Figure 5. The unsaturated group from GMA introduced on MG will then be subjected to polymerization as it is exposed to UV radiation or radical initiator produced by photoinitiator.

In the subsequent study, MG-GMA was prepared using the selected condition (90°C , 2 h, 2% of catalyst) and different ratios of MG to GMA. The objective of this study was to determine the effect of GMA/MG ratio on the properties of MG-GMA resin. When more GMA is added to the mixture, it is believed that GMA would interact with the hydroxyl group generated from the reaction between GMA and MG as depicted in Figure 6.

Table III depicts the viscosity of MG-GMA resin prepared from different ratio of GMA/MG . It is noticed that the viscosity of the MG-GMA resin increases as the amount of GMA is increased to a certain extent (GMA/MG ratio = 2), after which it decreases. By looking at the result of average molecular weight of MG-GMA resin prepared from different GMA/MG ratio (Table IV), a similar trend is observed. According to La Scala and Wool,²⁴ the molecular weight of oil can affect its viscosity, i.e., as the molecular weight increases, the viscosity is increased. This may come in the form of physical chain entanglements as well as chemical or intermolecular interactions, such as hydrogen bondings and dipole-dipole interactions. Hence, it can be said that the increase of viscosity at the initial stage is attributed to the increase in weight average molecular weight (M_w) when more GMA is employed to treat MG. However, the viscosity decreases when GMA/MG ratio is more than 2 (GMA/MG = 2.5). It is also noticed that the M_w of MG-GMA resin produced with GMA/MG ratio of 2.5 is lower than the ones prepared with GMA/MG ratio of 2. The reduction of MG-GMA M_w at GMA/MG ratio of 2.5 is attributed to the presence of homopolymer of GMA. The formation of GMA homopolymer in MG-GMA resin is due to the homopolymerization of

Table III. Viscosity of MG-GMA Prepared from Different Ratio of GMA/MG

GMA/MG ratio	1.0	1.5	2.0	2.5
Viscosity (cPs)	769 (± 3)	1702 (± 5)	4381 (± 13)	4160 (± 14)

Figure 7 depicts FTIR spectra of MG-GMA resin (uncured) and cured MG-GMA. It can be seen that transmittance peak at 1634 cm^{-1} disappears after it is exposed to UV radiation in the presence of photoinitiator. This indicates that the unsaturated group in MG-GMA has been consumed during polymerization process. In addition, the intensity of transmittance peak around 2900 cm^{-1} (due to stretching of C—H bonds) has increased due to the presence of more C—H bond in cured MG-GMA.

Table V shows the gel content or the degree of curing of MG-GMA after it is exposed to UV radiation with different percentages of photoinitiator. No significant trend is observed as the percentage of photoinitiator added is varied. However, the hardness of cured MG-GMA is affected by the amount of photoinitiator added. It is shown in Figure 8 that the hardness of the cured MG-GMA increases with the increment of percentage of photoinitiator except for GMA/MG of 1. This is attributed to the amount of radicals formed by photoinitiator. When more photoinitiator is employed, more radicals are generated upon UV radiation exposure. Hence, more radicals are available to react with unsaturation sites from MG-GMA and subsequently producing harder films. It is noted that there is no change in hardness for those with GMA/MG of 1, as the photoinitiator is increased. This probably indicates that a minimum amount of

Table IV. Weight Average Molecular Weight of MG-GMA Prepared from Different Ratio of GMA/MG

GMA/MG ratio	M_w
1.0	1951
1.5	2178
2.0	2637
2.5	2364

photoinitiator (1%) is sufficient to react with all unsaturation sites available. These results also show that those with more GMA (1.5, 2.0, and 2.5), display an increasing trend in hardness as the photoinitiator is increased. This indicates that the unsaturation sites in GMA respond well to the presence of more photoinitiators and produce more reactions and crosslinkings which manifested in the increase of hardness.

CONCLUSIONS

A UV curable resin based on palm oil was successfully prepared by treating Palm oil with glycerol to produce MG, which was subsequently subjected to reaction with GMA. The results indicated that unsaturation sites were successfully attached to the MG after it was treated with GMA and the resulting product could be cured by UV radiation in the presence of a photoinitiator. The viscosity of MG-GMA resin was strongly influenced by the GMA/MG ratio employed. Higher ratio of GMA/MG will produce MG-GMA resin with higher viscosity. However, the presence of GMA homopolymer reduced the viscosity of MG-GMA resin. As for the percentage of photoinitiator, no

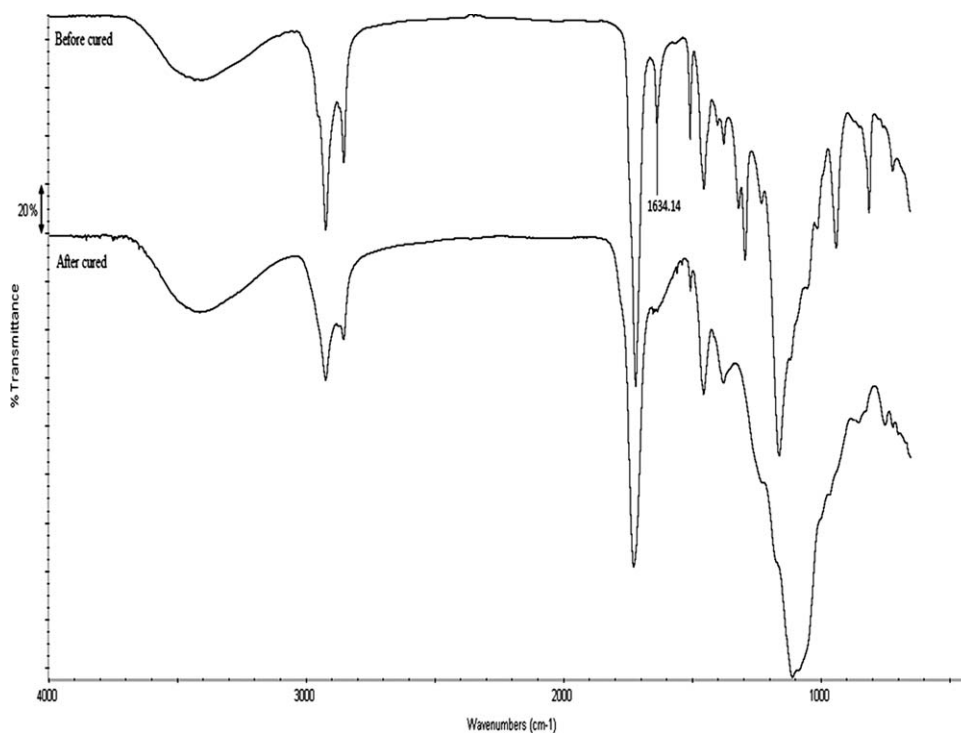
**Figure 7.** FTIR spectra of MG-GMA resin and cured MG-GMA.

Table V. Gel Content of MG-GMA

GMA/MG ratio	Photoinitiator (%)				
	1	2	3	4	5
1.0	99.16 (\pm 0.23)	98.54 (\pm 0.13)	97.68 (\pm 0.51)	98.73 (\pm 0.32)	98.65 (\pm 0.51)
1.5	99.43 (\pm 0.17)	98.78 (\pm 0.39)	98.06 (\pm 0.21)	99.46 (\pm 0.06)	97.76 (\pm 0.23)
2.0	99.48 (\pm 0.22)	99.65 (\pm 0.26)	98.48 (\pm 0.36)	98.76 (\pm 0.14)	99.38 (\pm 0.04)
2.5	98.98 (\pm 0.45)	99.72 (\pm 0.13)	98.59 (\pm 0.24)	99.23 (\pm 0.12)	99.50 (\pm 0.11)

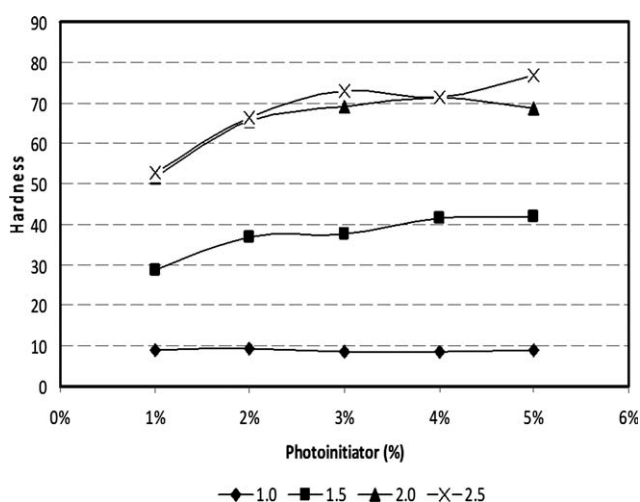


Figure 8. Pendulum hardness of MG-GMA film prepared from different percentages of photoinitiator and GMA/MG ratio.

significant change was noticed in gel content when different amounts of photoinitiator was used for MG-GMA curing process. Meanwhile, a significant trend was noticed in the film hardness when different amounts of photoinitiator were employed. This could be probably due to the amount of radicals produced by the photoinitiator as it was exposed to UV radiation.

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