

ANALYSIS THIN FILM POLYMER COMPOSITE OF POLYMETHYL METACRYLATE-REACTIVE MESOGEN DIACRYLATE AND ITS CONDUCTIVITY PROPERTIES

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ABSTRACT

This study aimed to investigate the impact of temperature during the UV curing process on the formation of a thin film polymer composite composed of polymethyl methacrylate-reactive mesogen diacrylate. The thin film polymer composite was successfully synthesized using the UV curing method, and the reactive mesogen diacrylate employed in this study was RM 82. Methyl methacrylate and RM 82 were polymerized through UV curing with photopolymerization additions using Benzoyl Peroxide as the initiator. The photopolymerization process was performed at various temperatures of 125°C, 150°C, and 200°C for 5 minutes. The thin film photopolymerized at 125°C and 150°C exhibited greater transparency than the thin film photopolymerized at 200°C. The FTIR results indicated the breakdown of the vinyl group and the triple bond. The FTIR spectrum displayed successful polymerization, with peak wave numbers of 1147 cm-1 and 1221 cm-1 for PMMA in the presence of C-O-C in the ester and a peak at 1465 cm-1. The SEM results demonstrated that the thin film photopolymerized at 200°C had become distorted. The XRD results revealed that the thin films of all variations were semi-crystalline. Meanwhile, the conductivity test revealed that the thin film photopolymerized at 125°C had a conductivity value of 2.095 x 10-12 S, while the photopolymerized thin film at 150°C had a conductivity of 2.14 x 10-12 S. Hence, the addition of reactive mesogen RM82 by 50% may increase the conductivity value of the thin film, making it a potential material for applications as a thin film polymer composite of PMMA-RM 82.

Keywords: reactive mesogen RM 82, polymethyl methacrylate, liquid crystal polymer

INTRODUCTION

Thin film polymer composites are attractive for various applications due to their unique properties, such as lightweight, transparency, and water resistance. In recent years, using two or more types of polymers in thin film manufacturing has become increasingly popular due to the ability to create multifunctional materials with enhanced properties. These thin films are widely used in various electronic devices, such as liquid crystal displays (LCDs) and organic light-emitting diodes (OLEDs). For instance, OLEDs consist of multiple layers of thin films, including a polymer layer, to emit light efficiently. [<u>1</u>, <u>2</u>, <u>3</u>] The use of thin film polymer composites continues to expand in various fields due to their unique properties, including mechanical flexibility, electrical conductivity, and thermal stability.

Reactive mesogen diacrylate, specifically RM 82, is a liquid crystalline monomer with desirable properties. It has been widely used in actuators and sensors as single or dual layers due to its ability to change in response to thermal, solvent, light, mechanical, or electrical stimuli [2]. By incorporating RM 82 into the polymer matrix, the resulting thin film polymer composite can exhibit these desirable properties. Polymethyl methacrylate (PMMA) is one of the materials commonly used as an optical substrate because of its excellent transparency, durability, weather resistance, and high index of refraction [3]. In addition to its optical properties, PMMA has good insulation properties, including low dielectric constant and low dielectric permittivity. Furthermore, the dielectric's surface can morphology affect the devices' performance, making PMMA an ideal material for optical substrates in various applications [4, 5, 6,7, 8].

Research on the polymer composite of PMMA-RM 82 has been gaining interest as it can potentially improve the properties of thin film polymer composites. The combination of RM 82 with PMMA can result in a material that gives properties suitable for sensor applications. Reactive mesogens are commonly used to make anisotropic optical films, and their combination with PMMA can result in a material that exhibits desirable optical, electrical, and mechanical properties. The resulting polymer composite can also be produced cheaply and processed and shaped easily compared to nanostructured materials, which can be challenging and expensive to manufacture [9, 10].

Combining methyl methacrylate with reactive mesogen diacrylate, specifically RM 82, can lead to a new material with desirable properties for various applications. The combination of PMMA and RM 82 can result in a polymer composite exhibiting excellent optical, electrical, and mechanical properties while being cost-effective and easy to process and shape. In addition, the resulting polymer composite can be utilized in sensor applications, making it a promising material for future research[11,12]. The combination of these two monomers has the potential to create a new material with superior properties for various applications. UV curing in the process of photopolymerization is a relatively new and innovative method of creating polymer composites that has the advantage of being quick and efficient [13,14].

To evaluate the properties of the PMMA-RM 82 polymer composite, the authors used several analytical techniques, including Fourier Transformed Infra-Red (FTIR), Scan Electron Microscopy (SEM), differential scanning calorimetry for thermal analysis, and XRD. These methods allowed for thoroughly characterising the polymer composite's structure, morphology, and thermal behaviour. Additionally, the authors measured the conductivity value of the PMMA-RM 82 composite using an LCR meter. This step is crucial to assess the material's suitability for sensor applications, where conductivity is a key property. This research contributes significantly to polymer specifically composites, in developing PMMA-RM 82 polymer composites. UV curing in the photopolymerization process and the thorough analytical characterization of the resulting material provide a new and valuable approach to creating and evaluating polymer composites with desired properties. The findings of this study can potentially open up new avenues for developing highperformance materials for various applications, including sensors, actuators, and optical films.

METHOD

Materials

The apparatus used in this study was 25 watts UV lamp with and wavelength of 365 nm, a CWC (Cano Wedge Cell) glass plate measuring 2x3 cm, a spatula, an analytical balance, a magnetic stirrer, hotplate stirrer, aluminium, black box, dropper, а thermometer, beaker. The chemicals used were methyl methacrylate (MMA), dimethylformamide (DMF), chloroform, (BPO) and benzoyl peroxide reactive mesogen (RM 82).

Preparation

The methyl methacrylate monomer was polymerized using the UV curing and reactive mesogen (RM82) to produce thin films. A cosolvent of dimethylformamide and chloroform in a 7:3 ratio was used to dissolve three grams of methyl methacrylate in a 30 mL glass beaker at room temperature, resulting in a 30% wt polymer solution. The RM82 solution was then combined with the MMA solution. A tip of the spatula (0.05 grams) of BPO initiator (Benzyl Peroxide) was added to the MMA-RM82 mixture at a temperature of 95oC with a speed of 250 rpm for 5 minutes. The resulting mixture was placed on a glass plate and polymerized using the UV curing method at various temperatures of 125°C, 150°C, and 200°C for 5 minutes.

Instrumentations

Thin film LCP PMMA-RM 82 were characterized using FTIR (Fourier Transform Infrared Spectroscopy) from Alpha II Bruker type, which aims to determine functional groups. XRD (X-Ray Diffraction) from XRD Empyrean Series 3 Panalytical type is carried out to determine the phase of thin films. SEM (Scanning Electron Microscope) from the Carl Zeiss type aims to determine the morphology. Thin films' electrical conductivity is characterized using an LCR Meter from the HIOKI IM3533 type, which aims to determine the electrical conductivity of thin films. The last characterization was the characterization of DSC (Differential Scanning Calorimetry) from DSC-60 C30935200137SA type with atmosphere Nitrogen, Flow rate 10 (mL/min), start temperature 30°C, temperature rate 10 (°C/min), hold temperature 300°C.

RESULTS AND DISCUSSION

Polymerizations of MMA-Diacrylate Resin

Photopolymerization of monomer methyl methacrylate with reactive mesogen 82 using UV curing technology called addition polymerizations. The initiator step produces free radical photopolymerizations in MMA and RM 82. The second step, each continuous process, is the propagation of two monomers. As a result, free radical monomer MMA and RM 82 are linking Figure 1 [11]. UV curing process at temperature variations of 125°C, 150°C and 200°C for 5 minutes each photopolymerized. The BPO initiator will be exposed to ultraviolet radiation energy and undergo homolytic cleavage of the C-C bond resulting in an uncharged radical species until the termination process ends. Furthermore, the liquid crystal monomer of RM will bind to PMMA [<u>12</u>, <u>13</u>, <u>14</u>]. Thin film LCP PMMA-RM 82 was photopolymerized for 5 minutes at a temperature of 125°C, 150°C and 200°C can be seen in Figure 2.



Figure 1. Photopolymerization illustration of polymer composite [14]





Thin film photopolymerized at 200°C is harder and stiffer than the photopolymerized thin film at 125°C and 150°C. Thin film photopolymerized at 200°C is also easier to remove from the substrate than thin film photopolymerized at 125°C and 150°C; this shows that thin film photopolymerized at 200°C dries faster and stiffens than thin film, which was photopolymerized at 125°C and 150°C. However, as can be seen, thin films photopolymerized at 125°C and 150°C are visually transparent more than those photopolymerized at 200°C [14].

Analysis of Polymer PMMA-Diacrylate Resin

Analysis group function of thin film LC polymer of PMMA-RM 82 using Fourier Transformed Infrared Spectroscopy (FTIR). Based on the FTIR spectrum in Figure 3 peak in low absorption at 2921 cm⁻¹ for PMMA and a strong absorption at 2900 cm⁻¹ for MMA containing CH₃. Furthermore, an absorption wave of 3100 cm⁻¹ appeared in MMA, indicating the vinyl groups' presence. However, this absorption was not present in PMMA because the vinyl had broken in PMMA [15,16, 17].



Figure 3. FTIR Spectrum of Thin Film LCP PMMA-RM 82

Furthermore showed a fairly sharp peak at wave numbers 1140 cm⁻¹-1240 cm⁻¹ for MMA and 1147 cm⁻¹, 1221 cm⁻¹ for PMMA in the presence of C-O-C in the ester group to MMA and PMMA. The peak in 1465 cm⁻¹ indicated which dominates the group function of C-H, and absorption peaks are at a wave number of 1670 cm⁻¹ for a C=C double that in methyl methacrylate molecule. According to the literature on the FTIR spectrum for RM 82, absorption appears at a wave number of 2861 cm⁻¹-2900 cm⁻¹ which has the presence of C-H alkanes. Furthermore, absorption appears at wave numbers 1741 cm⁻¹- 1600 cm⁻¹ which develops C=O ester. Aromatic C=C is seen at the absorption of 1500 cm⁻¹. Absorption at the wave number of 1435 cm-1 dominates CH₂ [18] [19].

The polymerisation process was successful, based on the polymer spectra

obtained at temperatures of 125°C, 150°C, and 200°C. However, the peak at 3100 cm-1 did not indicate the presence of a vinyl group since there was no absorption wave number for it. This suggests that the double bond in the vinyl group was broken. Moreover, the absence of absorption wave numbers at 1435 cm⁻¹ and 1670 cm⁻¹, which are found in RM82 and PMMA, respectively, indicate the cleavage of the CH2 bond in RM82 and the central C=C in PMMA. Conversely, a new absorption wave number at 1438 cm⁻¹ was observed in the thin film photopolymerized at 200°C, demonstrating that the CH2 bond was not broken in the photopolymerized thin film at this temperature [20]. Photo SEM PMMA-RM 82 in magnifications are 10000x and 15000x, shown in Figure 4.



Figure 4. Morphology surface of thin film LCP PMMA-RM 82 in 10000x and 15000 magnification

Analysis of surface morphology of PMMA-RM 82 that resulted in uv curing process at a temperature of 125°C, foto image of SEM with 10000x and 15000x magnification showed a smooth surface with little agglomeration. The thin film photopolymerized 10000x at 150°C with and 15000x magnification on SEM showed a uniform pattern on the surface compared to the photopolymerized thin film at 125°C and 200°C. This uniform pattern that long particles on the surface are assumed to be RM82. This is evidenced by the shape of the molecule, such as rods or discs, as a characteristic of reactive mesogens. Previous research showed that rod-shaped molecules are reactive mesogens and a reactive mesogen morphology resembling bars is seen using SEM [21].

Polymer PMMA-RM 82 that photopolymerized at 200°C showed a very smooth surface and appeared as if there were no long particles from the surface RM 82. However, regions some were not homogenous. Maybe some chains in the polymer broke out because the uv curing temperature of 200°C was high. Therefore, it maybe has been deformed or damaged on the surface of that polymer. This is observed following the FTIR spectrum, which shows that there is no peak for the CH₂ bond, and it is assumed that RM82 has been deformed <u>[21.22]</u>.



Figure 5. XRD diffraction pattern of LCP PMMA-RM 82 (T=125, 150, 200)

5 shows the XRD Figure diffractogram characterizing the crystallinity property of the PMMA-RM82 polymer. The diffractogram indicates that pure PMMA has dominant and wide peaks at 20 13.20, with low-intensity peaks at 31° and 35°. In contrast, the RM82 diffractogram has typical diffraction peaks at 20 18.95, 20.39, 22.26, and 23.28, which are pointed and have very high intensity, demonstrating the crystalline nature of RM82. The XRD diffractogram for the polymer PMMA-RM82 produced by UV curing at temperatures of 125°C, 150°C, and 200°C shows a sharp crystalline peak with high intensity at 2θ 44.53° and a blunt peak with very high intensity at 2θ 16.83[°] [22].

The PMMA-RM82 polymer is semicrystalline, as shown by the XRD diffractogram's broad peaks, indicating the presence of both amorphous and crystalline properties. However, the lack of sharp peaks with high intensity suggests that the crystalline nature of RM82 is still present in the PMMA-RM82 thin film. This confirms that PMMA and RM82 are well mixed in a 30%wt MMA and 50%wt RM82 ratio, with no dominant trait for either PMMA or RM82.

Conductivity Properties of Polymer PMMA-Diacrylate

The conductivity properties of polymer PMMA-Diacrylate Resin known by the measure of film polymer are conductance (S), Capacitance (F), and conductivity (S/cm). Therefore, the phenomenon of conductivity properties can be recommended for applications of thin film polymer PMMA-Diacrylate resins[23].

Curing Temperatures	Conductance (S)	Capacitance (F)	Conductivity (S/cm)
125°C	4,19 x 10⁻ ⁶	3,44 x 10 ⁻⁷	2,095 x 10 ⁻¹²
150°C	4,28 x 10 ⁻⁶	1,07 x 10 ⁻⁷	2,14 x 10 ⁻¹²
200°C	4,30 x 10 ⁻⁶	2,59 x 10 ⁻⁸	2,15 x 10 ⁻¹²

Table 1. PMMA-Diacrylate thin film conductance, capacitance and conductivity data

The conductivity value of PMMA-RM 82 was determined by an LCR meter. The frequency range used in the measurement is 42 Hz-50 Hz; from the measurement results obtained, the conductance value of the thin film. The thin film conductivity can be calculated from the conductance value by:

 $\sigma = G \frac{L}{A}....(1)$

σ, G, L and A are conductivity (Siemens/cm), conductance (Siemens), thickness (cm) and sample surface area (cm²). Polymer materials are generally insulators. This can be seen from the very small conductivity value of the polymer material. For example, based on research conducted, PMMA has a conductivity value of 5x10⁻¹⁸ S/cm [24, 25, 26].

Based on Table 1, it can be seen that the conductivity of the PMMA-RM82 thin film is greater than the conductivity of pure PMMA. lt can be seen that the photopolymerized thin film at 125°C has a conductivity value of 2.095 x 10-12, the photopolymerized thin film at 150°C has a conductivity of 2.14 x 10⁻¹², while the photopolymerized thin film at 200°C has a conductivity of 2.15 x 10⁻¹². Adding RM82 by 50% can increase the conductivity value of the thin film. Variations in UV curing

temperature only slightly affect the conductivity value to be greater [27, 28, 29].

Based on the chromatogram data, polymer PMMA-RM 82 was made at a temperature of 150°C and showed a DSC peak at 423.45°C. Properties that do not respond to the temperature modulation are those that involve time-dependent (kinetic) transitions, which include crystallization, decomposition, evaporation and chemical reactions (including curing).[30 31],

CONCLUSION

Photopolymerization of Methyl Methacrylate with Reactive Mesogen 82 using the Uv Curing Method has been successfully photopolymerized. The results of FTIR characterization did not indicate the presence of vinyl groups in the absorption at wave numbers 3100 cm⁻¹ and 1670 cm⁻¹. This indicates a severance of the double bond in the vinyl and C=C groups, so it can be assumed that a new bond is formed on the PMMA-RM82 thin film. Increasing the photopolymerization temperature increases the intensity of the wavenumber. SEM characterization showed that the photopolymerized thin film at a temperature of 125°C had molecules agglomerated (agglomeration). The photopolymerized thin film at 150°C showed a uniform pattern on the

surface. The thin film that was photopolymerized at 200°C had been deformed or had been damaged. XRD results of the thin film showed broad peaks indicating that the thin film was semi-crystalline. Therefore, thin Film PMMA-RM82 does not have the potential to be a conductor. The DSC peak temperature of thin film PMMA-RM is 423.45°C. Among thin films that were photopolymerized at temperature variations of 125°C, 150°C and 200°C, thin films with photopolymerization temperature variations of 150°C had the most uniform surface morphology and only a little agglomeration.

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