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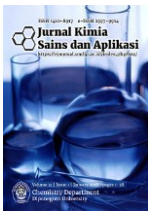

Effect of UV Curing Temperature on the Preparation and Properties of PMMA-Diacrylate RM82 Composite Membranes and Test of Water/Methanol Uptake

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Abstract

A composite membrane of Polymethylmethacrylate-reactive mesogen RM82 (PMMA-Diacrylate RM82) was prepared by UV curing at 110, 120, 130, 140, and 150°C. FTIR spectroscopy revealed peaks at 1086 cm^{-1} and 1147 cm^{-1}, indicating the presence of ester functional groups (C-O). An ether functional group (C-O-C) was observed at 1250 cm^{-1}, while aromatic structures were identified at 1605 cm^{-1}. XRD diffractograms showed high-intensity peaks at 2θ angles of 19.13°, 43.62°, and 72.42°, demonstrating that the membrane retained strong crystalline properties. SEM results showed a regular surface morphology with layered structures, whereas the membrane cured at 120°C exhibited an irregular morphology with coarse fragments. POM textures generally displayed sharp, elongated fibrous patterns. Water uptake was relatively low, with a maximum value of 34.2%, while methanol uptake ranged from 9.28% to 40.5%. The conductivity values of the composite membranes ranged from 3.01×10^{-7} to 4.24×10^{-7} S/cm. Conductivity consistently increased with higher UV curing temperatures and membrane thickness. This trend suggests that UV curing temperature enhances electrical conductivity by altering the molecular structure to create more effective conduction pathways.

1. Introduction

The process of making composite membranes using UV curing technology is strongly influenced by the curing temperature. In this process, monomers and other additives are irradiated and heated simultaneously. UV- cured temperature affects the structure and other properties of membrane composites, thereby influencing the membrane's performance in its application. A membrane composite was prepared by mixing two monomers for polymerization. One of the monomers has been studied before: methyl methacrylate, which has been polymerized to form polymethyl methacrylate. Therefore, in this article, the process of making a membrane composite between the monomer of methyl methacrylate and other materials is reported [1, 2].

Polymethyl methacrylate (PMMA) has several advantages, including good flexibility, high strength, and excellent dimensional stability. PMMA is a glass-like transparent thermoplastic polymer with chemical stability, weather resistance, and high optical transparency. This polymer is commonly used as a key component of positive resistance for electron and UV photolithography. However, PMMA has several disadvantages, namely poor heat resistance, weak mechanical surface, and low refractive index. Therefore, additional materials are needed that can be combined with PMMA [3, 4, 5].

The monomer added to the PMMA matrix is the reactive mesogen RM82, characterized by a six-carbon side chain and terminal acrylate double bonds. These monomers are frequently employed to synthesize macro-scale polymer networks via light-initiated polymerization [6, 7]. In this study, UV curing was utilized to polymerize the MMA and RM82 blend. A critical

parameter in this process is the curing temperature, which significantly influences the properties of the resulting polymer. Because both methyl methacrylate and RM82 contain acrylate double bonds that undergo addition polymerization, and each possesses a distinct melting point, the thermal environment directly dictates the efficiency of the polymerization process. The resulting composite membranes were subsequently characterized based on their water uptake, methanol uptake, and electrical conductivity [8, 9, 10].

Composite membrane technology facilitates the development of materials that integrate the synergistic properties of diverse monomers. Existing literature suggests that blending PMMA with secondary monomers can significantly enhance membrane performance and quality. Consequently, this study focuses on the fabrication and characterization of PMMA-based composite membranes. These membranes were synthesized via UV curing, with the monomer blends subjected to varying thermal conditions (60, 80, 100, 120, and 150°C). Comprehensive characterization was conducted to evaluate the molecular structure, surface morphology, and performance metrics, specifically through water and methanol uptake measurements.

2. Methods

2.1. Materials and Equipment

The materials used in this study included Polymethyl methacrylate (PMMA), reactive mesogen RM82, dimethylformamide (DMF), chloroform, methanol, benzoyl peroxide, sodium chloride, sodium hydroxide, sulfuric acid, and deionized water.

Laboratory apparatus included a diffusion cell, an analytical balance, a magnetic stirrer, a hotplate stirrer, a thermometer, a pycnometer, glass plates, and standard laboratory glassware. The membrane properties were characterized using Polarization Optical Microscopy (POM), Fourier Transform Infrared (FTIR) spectroscopy, Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), and an LCR meter.

2.2. Experiment

This study employed an experimental approach. The initial stage involved preparing PMMA–diacrylate RM82 composite membranes via solution casting. Methyl methacrylate (MMA) was polymerized by UV curing at 110, 120, 130, 140, and 150°C. The resulting membranes were characterized by FTIR, SEM, and XRD. In addition, water and methanol uptake, swelling behavior, and conductivity were evaluated.

The PMMA–diacrylate RM82 composite membrane was synthesized by first preparing a 70 wt% MMA solution: 7 g of MMA was dissolved in 3 mL of chloroform and homogenized. Separately, a 30 wt% RM82 solution was prepared by dissolving RM82 in 3 mL of chloroform until homogeneous. The two solutions were then combined at a 70:30 wt% ratio in a mixed solvent system of DMF and chloroform (7:3 v/v).

The resulting solution was further stirred at 200 rpm for 5 minutes using a magnetic stirrer. Benzoyl peroxide (BPO) was added as an initiator. The mixture was then cast onto a glass plate to form a 100 µm-thick film. UV curing was performed at 110–150°C for 15 minutes [11, 12].

Water and methanol uptake tests were conducted at room temperature (25°C). The membranes were first dried for 24 hours to obtain the dry weight (W_d). The samples were then immersed in water or methanol for 24 hours and weighed to obtain wet weight (W_w). Conductivity measurements were carried out using an LCR meter at room temperature for all PMMA–diacrylate RM82 composite membranes.

3. Results and Discussion

3.1. Synthesis of Composites of PMMA-Diacrylate RM82

PMMA is an amorphous polymer belonging to the acrylate family. It is transparent and colorless, with a glass transition temperature in the range of 100–130°C and a density of approximately 1.20 g/cm^3 at room temperature. PMMA exhibits low water absorption (~0.3%) and a linear mold shrinkage of 0.003–0.0065 cm/cm. It also shows good resistance to UV radiation and maintains stable thermal properties, tolerating temperatures up to ~100°C. In addition, PMMA possesses excellent optical properties, with a refractive index of 1.49, and demonstrates good biocompatibility. Chemically, it is resistant to most dilute laboratory reagents, although it is susceptible to hydrocarbons, esters, chlorinated ketones, and aromatic compounds [13, 14]. PMMA can function as a pore-forming agent, enabling the formation of uniform pore structures suitable for polymer electrolyte membrane (PEM) fuel cell applications. Its low density and relatively low cost make it an attractive material. Furthermore, PMMA can act as a mechanical support matrix and assist in regulating transport properties within the membrane. When coated with conductive materials, it may also contribute to electrical and thermal conductivity, making it a potential candidate for current collector components in fuel cells [15].

The fabrication of PMMA–diacrylate RM82 composite membranes involved two main stages: solution preparation and UV curing. In the first stage, a 70 wt% MMA solution was prepared and mixed with DMF and chloroform in a beaker. RM82 powder was weighed separately and then combined with the MMA solution. BPO was added as an initiator to promote polymerization. The mixture was stirred using a magnetic stirrer on a hot plate at 80°C for 5 minutes until a homogeneous solution was obtained [16].

In the second stage, the prepared solution was cast onto a glass substrate and UV-cured. The curing process was carried out for 15 minutes under UV irradiation at different temperatures (110, 120, 130, 140, and 150°C) to evaluate the effect of temperature on membrane formation and properties.

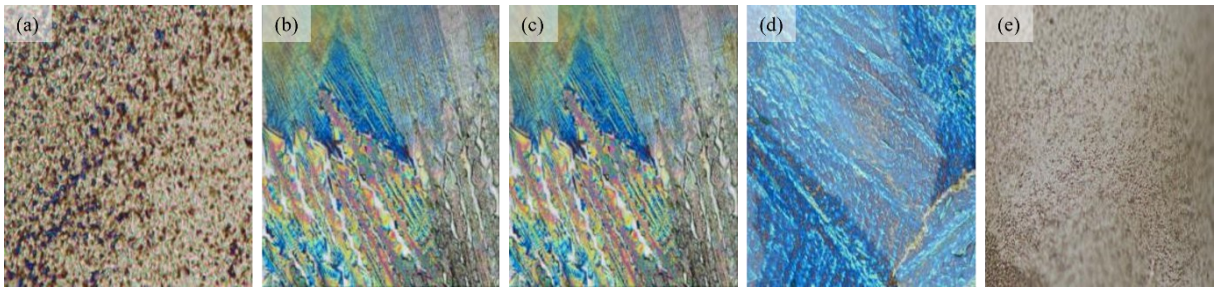

Figure 1. POM images of PMMA–diacrylate RM82 composite membranes at different curing temperatures: (a) 110°C, (b) 120°C, (c) 130°C, (d) 140°C, and (e) 150°C

A membrane composite of PMMA-Diacrylate RM82 was obtained in this research, with variations in UV curing temperature, as shown in Figure 1. The picture shows a pattern of fibers or lines that are quite sharp and elongated. It is characteristic of the crystal orientation in the membrane, indicating molecular orientation or partial crystallization of the PMMA membrane, whereas RM82 helps regulate membrane morphology, allowing the formation of more directional molecular orientations in some areas. Dark and light areas indicate a variation in the thickness or density of the structure. Darker areas are likely to have a more regular molecular orientation (crystalline), while lighter areas may represent amorphous regions or lower crystallinity [17, 18, 19].

3.2. Characterizations of Membrane Composite PMMA-Diacrylate RM82

Based on Figure 2, the FTIR spectra of the membranes cured at temperatures ranging from 110 to 150°C exhibit similar spectral patterns, indicating no significant changes in the main chemical structure. The FTIR spectrum of PMMA–diacrylate RM82 shows several characteristic absorption peaks corresponding to specific functional groups. Peaks observed at 1086 cm^{-1} and 1147 cm^{-1} are attributed to C–O stretching vibrations of ester groups. The absorption band at 1250 cm^{-1} is associated with C–O–C stretching, indicating the presence of ether linkages. A peak around 1440 cm^{-1} corresponds to the alkene group (C=H) that may originate from the C-H bond on the aliphatic and aromatic chains at PMMA-RM82. Additionally, the absorption peak at 1605 cm^{-1} is assigned to C=C stretching in aromatic rings.

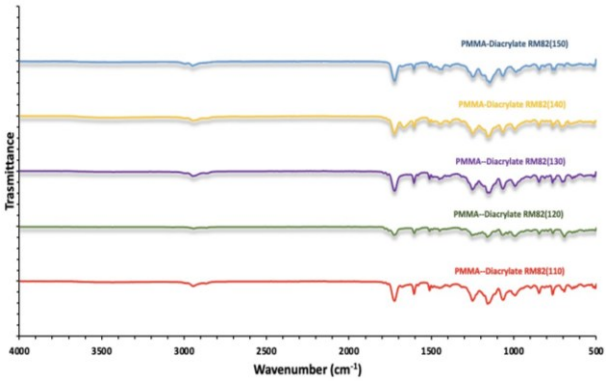

Figure 2. FTIR spectra of PMMA–diacrylate RM82 composite membranes at different UV-curing temperatures: 110, 120, 130, 140, and 150°C

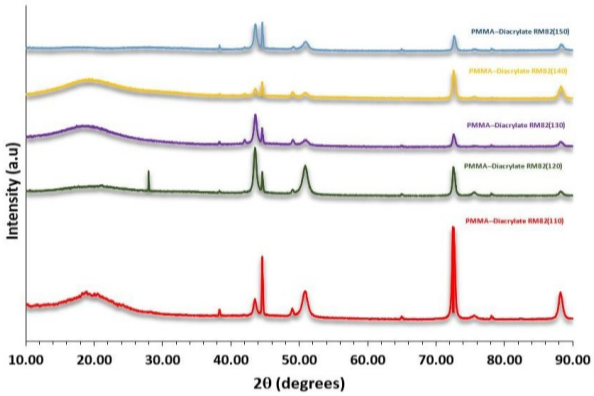

Figure 3. XRD diffractograms of PMMA–diacrylate RM82 composite membranes at different UV-curing temperatures at 110, 120, 130, 140, and 150°C

The strongest absorption peak at about 1720 cm^{-1} indicates the presence of a carbonyl group (C=O), which can be derived from the ester group in PMMA-Diacrylate RM82. There is a wavenumber peak of 2949 cm^{-1} that tightens the C-H bond of the alkane group. The absence of 3100 cm^{-1} waves in this spectrum indicates that the vinyl group in MMA has been damaged because MMA is successfully synthesized into PMMA. The polymerization results at 110, 120, 130, 140, and 150°C are considered successful because there is no significant change at the peak of the wave, and MMA is successfully synthesized into PMMA [20].

At 110°C, Figure 3 shows several high-intensity peaks at 2θ values of 19.13°, 43.62°, and 72.42°. The PMMA–diacrylate RM82 membranes prepared at UV-curing temperatures of 110–150°C exhibit similar diffraction features, indicating the presence of semi-crystalline domains. As the curing temperature increases, the peak intensities gradually decrease. At 120°C and 130°C, the reduction in intensity is noticeable but not significant, and the characteristic peaks at 19.13°, 43.62°, and 72.42° remain observable, although less sharp compared to 110°C. A similar pattern is observed at 140°C, with no substantial change in peak intensity. However, at 150°C, a more pronounced change occurs, marked by the disappearance of the peak at 2θ = 19.13°. Overall, the XRD results suggest that increasing curing temperature reduces the degree of crystallinity of the PMMA–diacrylate RM82 membrane, although the changes occur gradually and are not highly significant [21, 22].

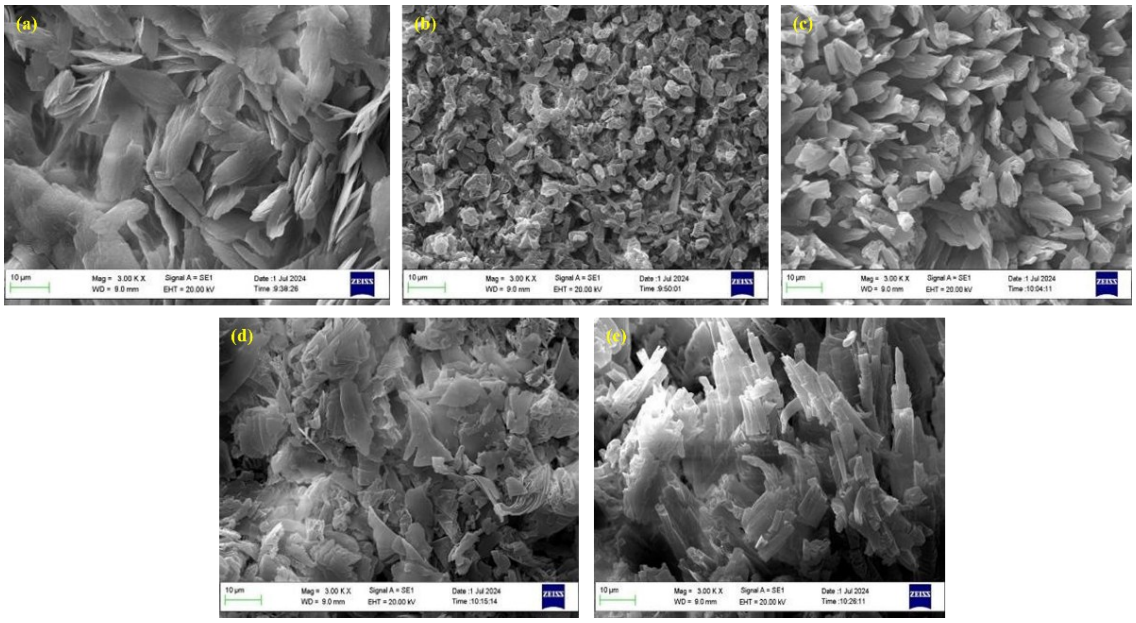

Figure 4. SEM images of PMMA–diacrylate RM82 composite membranes at different UV-curing temperatures: (a) 110°C, (b) 120°C, (c) 130°C, (d) 140°C, and (e) 150°C

Figure 4 shows that the surface morphology of the PMMA–diacrylate RM82 composite membranes varies with UV-curing temperature. At 110°C and 120°C, the membrane surfaces appear uneven, with non-uniform material distribution and the presence of small rod-like features. Layered or sheet-like structures are also observed, indicating a predominantly amorphous morphology at lower curing temperatures.

At 130°C and 140°C, the membranes exhibit relatively smoother surfaces with a more homogeneous particle distribution. The presence of finer particles and a more dispersed structure suggests an increase in surface roughness. In addition, more defined rod-like features begin to appear, which may be associated with the alignment of diacrylate RM82, known to exhibit a rod-shaped mesogenic structure. At 130°C, some regions exhibit more ordered, directional features, suggesting the onset of structural organization. At 150°C, the morphological changes become more pronounced. The membrane surface shows clearer rod-like structures and larger pore formations compared to those prepared at lower temperatures. The presence of cavities across all samples confirms the formation of porous structures within the composite membranes [23, 24].

Overall, the SEM observations indicate that increasing the UV-curing temperature promotes structural organization and pore development in the PMMA–diacrylate RM82 membranes. This trend is consistent with the XRD results, which suggest changes in structural ordering with increasing temperature. However, the transition appears gradual, indicating partial rather than complete crystallization [25, 26].

3.3. Water and Methanol Uptake

Water and methanol uptake tests were conducted to evaluate the absorption capacity of the PMMA–diacrylate RM82 composite membranes. This parameter is important for assessing membrane performance, particularly in applications involving mass transport. The test was performed by immersing the membrane samples in distilled water and methanol for 24 hours. It is hypothesized that higher curing temperatures may enhance the uptake capacity due to increased pore formation within the membrane structure. The results of water and methanol uptake are presented in Figure 5.

The membrane composite PMMA-Diacrylate RM82 was made at a UV curing temperature of 110°C; the water and methanol uptake percentages are relatively low. This shows that at these temperatures, the membrane structure remains very dense and exhibits little absorption. Furthermore, at the highest UV-curing temperature, 150°C, water uptake reaches 34.2%. This improvement shows that the structure of the membrane has changed significantly, making it easier for water to be absorbed from the membrane. Then, the methanol uptake test at 110°C shows a percentage of 9.28%, which is quite low, and at 150°C shows a percentage of 40.5%. The largest change occurred between 130°C and 140°C, where increases in water and methanol uptake were significant. Overall, the results are in accordance with the hypothesis [27, 28].

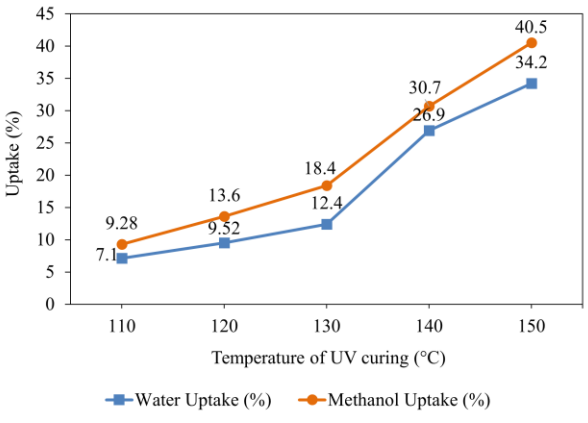

Figure 5. Water and methanol uptake of PMMA–diacrylate RM82 composite membranes at different UV- curing temperatures

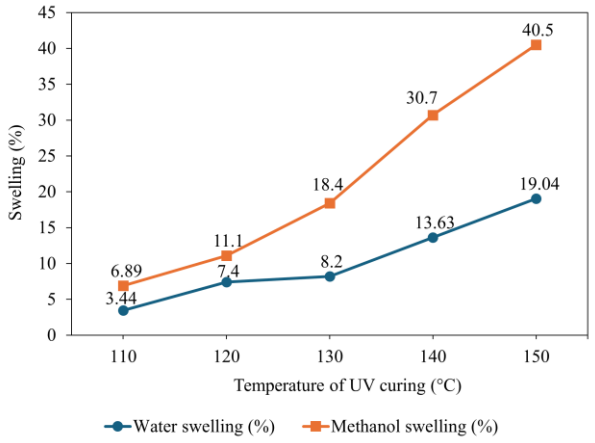

Figure 6. Water and methanol swelling of PMMA-diacrylate RM82 composite membrane

Table 1. Electrical resistance and conductivity of PMMA–diacrylate RM82 composite membranes at different UV- curing temperatures

UV-curing temperature (°C)	Resistance (Ω) (× 10^7)	Conductivity ($S.m^{-1}$) (× 10^{-7})
110	2.58	3.01
120	2.57	3.36
130	2.54	3.67
140	2.51	3.87
150	2.5	4.24

The swelling behavior of the membranes was evaluated in water and methanol to determine the extent of dimensional change after immersion. The membrane thickness was measured before and after 24 hours of soaking. The results (Figure 6) indicate that membranes cured at higher temperatures exhibit greater swelling, consistent with the water and methanol uptake results. This suggests that increased curing temperature enhances solvent absorption, leading to an increase in membrane thickness [29, 30].

The conductivity of the membranes was measured using an LCR meter. The measurement was based on the resistance values obtained, from which the conductivity was calculated. All tests were performed on membranes that had been immersed for 24 hours.

Based on Table 1, increasing the UV-curing temperature from 110°C to 150°C results in a gradual decrease in resistance (from 2.58 × 10^7 to 2.50 × 10^7 Ω) and a corresponding increase in conductivity (from 3.01 × 10^{-7} to 4.24 × 10^{-7} $S{\cdot}m^{-1}$). This trend indicates that higher curing temperatures enhance charge transport within the membrane. The improvement in conductivity may be associated with structural changes, such as increased chain mobility, improved interfacial contact, or the formation of more continuous transport pathways, which are consistent with the observed morphological and structural evolution. However, further increases in temperature may not necessarily lead to continued improvement, as excessive heating can cause structural degradation or loss of functional properties [31, 32].

4. Conclusion

PMMA–diacrylate RM82 composite membranes were successfully prepared via UV curing at temperatures of 110–150°C. POM observations revealed elongated, fiber-like textures with alternating dark and bright regions, indicating variations in molecular orientation and structural density. SEM analysis showed that the membranes predominantly exhibited amorphous morphology at lower temperatures, while higher curing temperatures promoted the appearance of more organized rod-like structures and increased pore formation. FTIR spectra confirmed the presence of characteristic functional groups, including ester (C–O) at 1086 and 1147 cm^{-1} and carbonyl (C=O) at ~1720 cm^{-1}. The absence of vinyl-related absorption indicates successful polymerization of MMA into PMMA. XRD analysis revealed diffraction peaks at $2\theta \approx 19.13°$, 43.62°, and 72.42°, suggesting the presence of semi-crystalline domains. Increasing curing temperature led to a gradual reduction in peak intensity, indicating a slight decrease in structural order. In addition, methanol uptake ranged from 9.28% to 40.5%, demonstrating that higher curing temperatures enhance solvent absorption, which is consistent with increased porosity and morphological changes in the membrane.

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