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CHEMICAL PROPERTIES AND SURFACE MORPHOLOGY OF ENR/PVC FILLED CELLULOSE GRAFTED PMMA MEMBRANE

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ABSTRACT. *The ENR/PVC thin film has great potential as a membrane due to its ideal owing to unique characteristics such as freestanding, high-pressure resistance, and durability over time, but low porosity. This research aims to investigate inside into chemical and morphological properties of the developed composite epoxidized natural rubber with polyvinyl chloride-filled cellulose grafted polymethyl methacrylate (ENR/PVC/cellulose-g-PMMA) membrane. Solution blending of 60:40 wt. % ENR/PVC 10 % w/v of filler was mixed homogeneously for 24 h, stirring in THF solution. The membranes were cast onto a glass plate, and the phase inversion technique was used to prepare ENR/PVC/Cell-g-PMMA membranes. The characterization of chemical properties was carried out by Fourier Transform İnfrared Spectroscopy (FTIR) to determine functional groups related to bond breaking and the formation of new bonds of the species during grafting copolymerization and membrane fabrication. Variable Pressure Scanning Electron Microscopy (VPSEM) was conducted to determine surface morphology and textural properties of fillers and membranes. Furthermore, it contributes to porosity and the formation of pores on the membrane. FTIR spectrum shows that absorption peaks around the range 1735-1725 cm^{-1} of the carbonyl ester -C=O functional group have been detected and prove the success of the grafting method between cellulose and PMMA. The rough surface of fibers, formation of open pores, and interspace structure between ENR/PVC matrix and filler affirmed the addition of cell-g-PMMA fillers caused by film-filler and filler-filler interfaces interaction, as shown by the VPSEM micrograph. In summary, the chemical properties and morphological analysis give useful information on the effectiveness of potential ENR/PVC/Cell-g-PMMA membranes in developing standalone, highly pressure-resistant, porous, and elastic membranes.*

INTRODUCTION

Membranes are used extensively in a variety of industries, including water purification, petrochemical refining, pharmaceutical manufacture, mining, building, and space exploration (Norfarhana *et al.,* 2022). They are divided into organic (polymeric) and inorganic (ceramic, metallic, and zeolite) forms depending on their component ingredients. Organic membranes are made from synthetic or natural polymers such as polytetrafluoroethylene (PTFE), polyamide-imide (PAI), polyvinylidene difluoride (PVDF), polyether sulfone (PES), and polyacrylonitrile (PAN) (Arman Alim & Othaman, 2018; Norfarhana *et al.,* 2022). Several factors, including the composition of the feed solution, the operating environment, the features of the application, and the need for separation, influence the choice of these polymers. Compared to organic membranesstability andmbranes are easier to clean after fouling, offer greater chemical and thermal stability, and are less susceptible to microbiological deterioration. Nonetheless, the reason for their initial greater costs is the exact thickness specifications required to withstand different pressure drops (Castro-Muñoz, 2020).

To tackle these issues, new polymers with enhanced mechanical and pressure-tolerant qualities have been created, inorganic fillers or nanoparticles have been added to boost strength, freestanding membranes have been made via phase inversion or electrospinning, and mixed matrix membranes (MMMs)—a blend of organic and inorganic components—have been employed (Castro-Muñoz, 2020). Nevertheless, there are often trade-offs between cost, complexity, and performance due to these improvements. Resolving these obstacles is essential to progressing the field, especially in pressure-resistant, robust, and standalone organic membrane advancement, which is still an active area of membrane science and technology research (Farahbakhsh *et al.,* 2021).

A composite membrane with several advantages has been created by combining epoxidized natural rubber (ENR) and polyvinyl chloride (PVC). This composite's hybrid structure of plastic and rubber makes it possible to create a freestanding single-layer membrane that does not require support material. This membrane differs from other composites due to the strength and flexibility of rubber and the porous nature of PVC plastic (Norfarhana *et al.,* 2022). The porosity of PVC and the strength and flexibility of rubber work together to enhance the overall functionality of the ENR/PVC composite membrane. While the PVC plastic contributes porosity—which is necessary for filtration applications—the rubber component provides flexibility and durability (Ismail *et al.,* 2015).

Nevertheless, treating wastewater with a high oil content can be less successful when using the ENR/PVC composite alone. Methyl methacrylate (MMA) can be grafted onto cellulose to enhance membrane performance in water-oil filtration (Shamsuddin *et al.,* 2013). This alteration boosts the membrane's hydrophobicity and creates sufficient porosity for effective water and oil separation. This research aims to understand the chemical and morphological properties of ENR/PVC/Cell-g-PMMA membrane to develop an effective membrane for oil-water separation. Inside chemical properties analysis, FTIR was used to determine how the functional group affects bond breaking and formation of new bonds in the graft copolymerization reaction of modified cellulose. Furthermore, this study focuses on identifying the relationship between surface morphology and pore formation during the fabrication of membranes. This characteristic is very important to boost the membrane's hydrophobicity and porosity, resulting in a more effective membrane for water-oil separation applications.

MATERIALS AND METHODS

Materials from several sources are used in the production of ENR/PVC thin films with cell-g-PMMA filler. Epoxidized natural rubber with 50 % epoxidation level (ENR50) was supplied by Guthrie Polymer Ltd., Thailand. High molecular weight polyvinyl chloride (PVC 97000), tetrahydrofuran (THF), and methyl methacrylate (monomer) were purchased from Sigma Aldrich, Germany. As previously documented, the Technical Association of the Pulp and Paper Industry (TAPPI) method was used to extract cellulose from pineapple leaves. Following the protocol previously described in the literature, this extracted cellulose is then functionalized with PMMA by graph-copolymerization (Figure 1A) (Shamsuddin *et al.,* 2013).

Development of ENR/PVC Thin Films

The thin film manufacturing process consists of two main stages: matrix preparation and thin film molding. The ENR/PVC matrix was prepared using a solution mixing process. Epoxidized natural rubber (ENR) was dissolved in THF to produce a homogenous solution. 6 g of ENR was soaked overnight in 80 mL of THF, agitated until homogeneous, then mixed with 4 g of powdered polyvinyl chloride. The mass ratio of ENR to PVC was 60:40, whereas the ratio of matrix to THF solvent was 1:8 (Ismail *et al.,* 2020).

Phase reversal and solution moulding were used to generate the ENR/PVC/Cell-g-PMMA thin film. 10% Cell-g-PMMA fillers were introduced to a homogenous ENR/PVC matrix solution during the solution moulding process to examine their effects on the thin film properties. To make sure the fillers were dispersed equally, the mixture was stirred. After that, the solution was poured onto a glass surface and smoothed with a 0.035 mm-thick casting knife.

Reversing a liquid phase into a solid state is known as phase reversal. To remove the THF solvent and enable the creation of the ENR/PVC/Cell-g-PMMA thin film, the leveled solution was rapidly evaporated and then immersed in distilled water. Before being removed from the glass plate and allowed to dry at room temperature until sufficiently mature for additional analysis, the membrane was allowed to finish phase reversal for about ten minutes (Mod *et al.,* 2019).

Characterization of Filler and ENR/PVC Thin Films

A Perkin Elmer Fourier Transform Infrared Spectroscope Spectrum 400 FTIR/FT-NIR equipped with a Spotlight 400 Imaging System was used to examine the interaction between the filler and the membrane matrix. The functional groups showing how the filler interacts with the matrix were found using this technique. Using a Leo 1450 VP model Scanning Electron Microscope (SEM) running at 15 kV, the morphology, texture, and interaction of the thin films with filler were investigated. The preparation of the samples involved putting them on conductive adhesive tape, gold sputter-coating, and VPSEM observation. All the characterization methods were followed by Shamsuddin *et al.* (2013).

RESULTS AND DISCUSSION

Chemical Interaction and Grafting Mechanisms

The chemical interaction of filler and matrix was analyzed by using peak intensities of certain functional groups in the sample. Figure 1A shows the FTIR spectrum of Cell, Cell-g-PMMA, ENR/PVC metrics, and ENR/PVC/Cell-g-PMMA. The absorption peak between 3300 - 3400 cm^{-1} refers to the vibrational stretching of hydroxyl groups, -OH, and hydrogen bonds that exist between cellulose molecules (Rosa et *al.* 2010), as well as the water absorption peak (Mohd *et al.,* 2021). While the absorption peak around 1650-1630 cm^{-1} refers to the stretching of the –OH group from cellulose (Sheltami *et al.,* 2012). Note that the -OH intensity of cell-g-PMMA was slightly decreased compared to cellulose due to the transformation of the -OH stretching bond into the ester carbonyl group.

The grafting process of cellulose and PMMA was successful due to the formation and transformation of a few functional groups. The peak 1736 cm^{-1} refers to the stretching vibration of ester carbonyl, C=O bond, belonging to PMMA that grafted onto the cellulose backbone (Sheltami *et al.,* 2012). The formation of the aldehyde (HC=O) functional group was detected by the stretching vibration of the CH bonding at 2776 cm^{-1}. Transformation of the C-C bond into HC=O and C-OH initiated by Ce (IV), preferably at the C_2-C_3 glycol unit by graft polymerization, occurred during the initiation and propagation step as described in Figure 1B.

The absorption spectral features observed within the wavenumber range of 3000-2000 cm^{-1} across all membrane spectra were indicative of CH stretching vibrations. Specifically, the peak at 1450 cm^{-1} corresponded to CH bending modes within the thin film, as reported by Nor *et al.* (2013). Additionally, the asymmetric and symmetric stretching vibrations of the CO functional group are characterized by an absorption peak at 1254 cm^{-1} and a range between 1110-1000 cm^{-1}, respectively. The presence of epoxy rings, C-O-C, and C-Cl bonds in ENR/PVC compounds was signified by absorption peaks at 876 cm^{-1} and 699 cm^{-1}, as suggested by Ismail *et al.* (2015), Nor *et al.* (2016) and Jon *et al.* (2017).

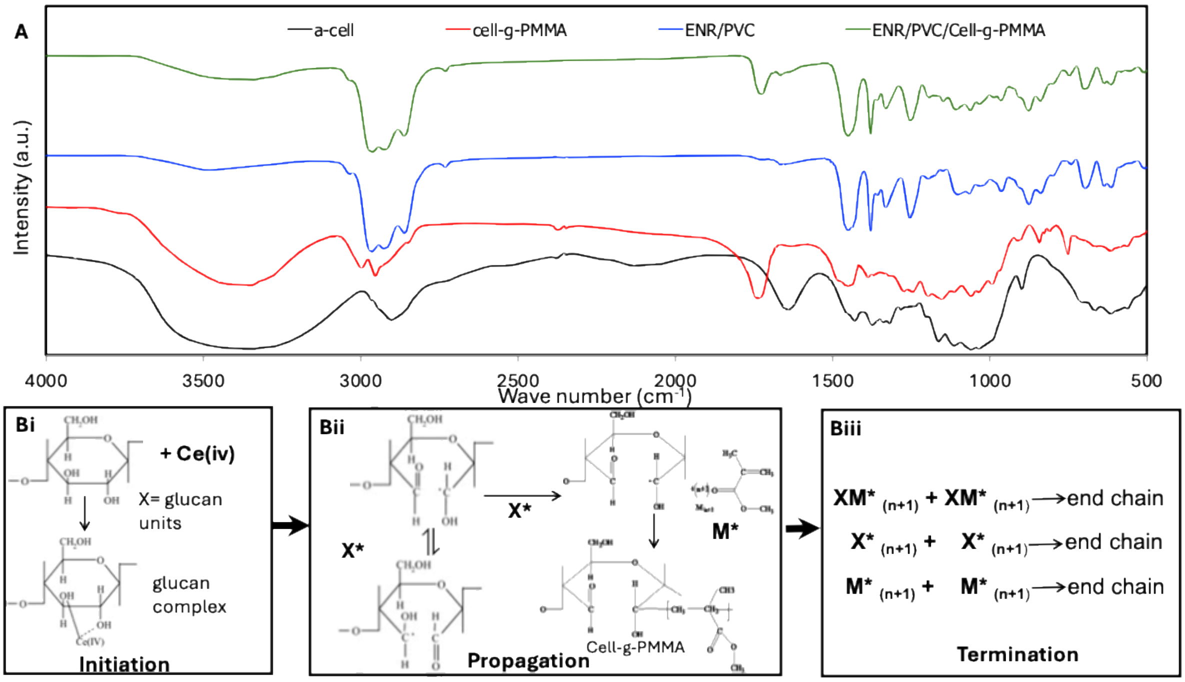

Figure 1. A) FTIR spectra; and B) Grafting of cellulose and PMMA.

Surface Morphology and Textural Properties

Surface morphology and pore production on the fillers and membrane were assessed by analyzing the morphological and textural features. The resulting micrographs were employed as well to investigate the effects of grafting alterations and the interactions between the filler and the membrane. Figure 2i shows the top surface morphology of the cellulose filler without any modification under 5000× magnification. Clearly, a smooth surface fiber was obtained with a fiber diameter of around 4.5 µm. Alteration by grafting polymerization of PMMA onto the cellulose backbone drastically changes the surface morphology of the fibers. The rough surface with a flaky texture was obtained in Figure 2ii with an increasing diameter of the fibers around 10 µm. These were due to grafting PMMA coated on top of the fibers' surface to give extra diameter to the modified fibers.

Membrane fabrication of ENR/PVC/Cell-g-PMMA prepared by solution casting and phase inversion method was exhibited in Figure 2iii, representing the surface area of the membrane at a magnification of 1000×. A previous study by Nor *et al.* (2016), Jon *et al.* (2017), and Norfarhana *et al.* (2017) found that the surface micrograph of the ENR/PVC membrane reveals a smooth, phase-stable, and uniform morphology with no observable pore formation on either the surface or cross-section. This indicates that the ENR/PVC blend achieves phase compatibility without requiring additional synchronization to ensure homogeneity. However, the incorporation of cell-g-PMMA filler into the ENR/PVC matrix induces notable alterations in both the surface morphology and the cross-sectional structure of the membrane. Following the introduction of cell-g-PMMA filler, the membrane surface exhibited the formation of fine pores, which transitioned the previously smooth and uniform surface of the ENR/PVC to a more textured appearance. Although the membrane surface became slightly irregular, the cross-sectional view did not reveal any translucent pores. Additionally, the cross-sectional analysis (Figure 2iv) of the membrane revealed the formation of pores between the filler and the membrane, as well as between individual filler particles. The interactions at the interface between the membrane and the filler, as well as among the filler particles themselves, contributed significantly to the increased development of these pores within the membrane.

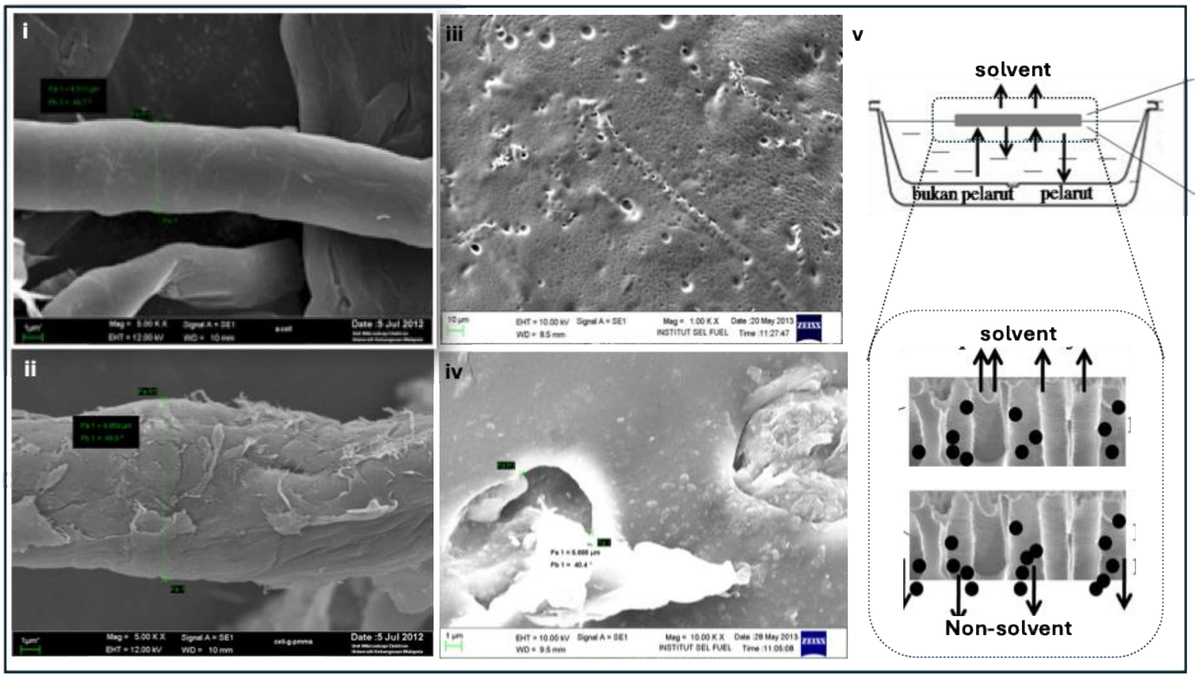

Figure 2. VPSEM micrograph and phase inversion method.

DISCUSSION

Grafting Mechanisms

Graft copolymerization of cellulose involves the synthesis of synthetic polymer branches (grafts) that impart specific characteristics to the cellulose while preserving its inherent properties. Through this modification process, new functional attributes are introduced to the cellulose, enhancing its performance without compromising its original features. The graft polymerization reaction between cellulose and methyl methacrylate monomer, which yields Cell-g-PMMA, is initiated by cerium ammonium nitrate (CAN). The proposed schematic of the three-step grafting reaction process—initiation, propagation, and termination—is depicted in Figure 1B. The mechanisms were inspired by Kumar *et al.* (2011).

For methyl methacrylate to graft polymerize onto cellulose, nitric acid is an essential ingredient. Cerium ions react with nitric acid in aqueous solutions as shown by the following equations:

$$Ce^{4+} + H_2O \rightarrow Ce\,(OH)_3^{3+} + H^+ \quad (1)$$
$$2Ce(OH)_3^{3+} \rightarrow Ce\text{-}O\text{-}Ce^{6+} + H_2O \quad (2)$$

In aqueous solutions, cerium ions can exist in the forms of Ce^{4+}, $Ce\,(OH)_3^{3+}$, and $Ce\text{-}O\text{-}Ce^{6+}$. These species' presence depends on the concentration of nitric acid used in the reaction. The ions Ce^{4+} and $Ce\,(OH)_3^{3+}$ exhibit smaller sizes and are more effective in forming complexes with cellulose compared to $Ce\text{-}O\text{-}Ce^{6+}$ (Arthur *et al.,* 1966; Hermans, 1962; Kumar *et al.,* 2011). Cerium ions are also involved in the oxidation process during the termination of monomer chains, as illustrated by the following equation (Arthur *et al.,* 1966; Kumar *et al.,* 2011; Nada *et al.,* 1989):

$$Ce^{4+} + M_n^* \rightarrow Ce^{3+} + H^+ \quad (3)$$

Nitric acid was used to dissolve CAN and create Ce^{4+} ions during the initiation stage. A Ce^{4+}-cellulose coordination complex was then formed because of these Ce^{4+} ions attacking the hydroxyl (OH) groups on the C_2 and C_3 carbons of cellulose (Hermans, 1962). The bonding in this compound is weak and unstable. Ce^{4+} ions are used in the oxidation process of different alcohols and substrates with hydroxyl groups when perchloric and nitric acids are present (Hermans, 1962; Kedzior *et al.,* 2016; Wohlhauser *et al.,* 2020). Subsequent investigation indicates that the hydroxyl group at C_6 and the glycol C_2-C_3 locations are the main locations where free radicals originate (Hermans, 1962; Nada *et al.,* 1989). The diol group oxidizes six times faster at the glycol C_2-C_3 than it does at the hydroxyl C_6, according to a study on the relative oxidation rates of the two groups. This shows that the glycol C_2-C_3 locations exhibit the most $[Ce]^{4+}$ oxidation, with very little reaction taking place at the main hydroxyl group C_6 (Boujemaoui *et al.* 2019).

The complex then reduces to Ce^{3+} ions, which causes free radicals to be produced at the C_2 or C_3 locations of the glucan repeat units. This starts the propagation stage (Arthur *et al.,* 1966; Nada *et al.,* 1989). The graft polymerization process is then started when the free radicals react with the vinyl monomers that are in the reaction mixture. These free radicals target the double bonds ($-C=CH_3$) in the methyl methacrylate monomer, leading to the formation of new bonds. Free radical transfer also occurs within the methyl methacrylate chain, resulting in the formation of polymethyl methacrylate. The free radical attack continues, facilitating chain extension until the polymerization reaches the chain termination phase. Chain termination is achieved when the free radicals interact with each other, producing a neutral polymer.

FTIR spectrum of cellulose and Cell-g-PMMA proves that the bond breaking and new bond formation during the propagation step occurred. Decreasing intensity of the OH functional group of PMMA compared to cellulose shows the formation of Ce^{4+}-cellulose coordination complex, which attacks the OH functional group mainly at the C_2 and C_3 positions. Furthermore, the formation of an aldehyde functional group (HC=O) during the bond breaking of the C_2-C_3 position and the existence of the ester carbonyl group, C=O, from PMMA grafted on the cellulose backbone.

Relationship Between Textural Properties and Pore Formation

The pore formation in ENR/PVC/Cell-g-PMMA membrane can be attributed to three principal mechanisms. Firstly, pore formation occurs during the phase inversion process involved in membrane production. During this phase, solvent evaporation from the membrane surface, coupled with the substitution of part of the solvent by water (a non-solvent), leads to pore creation (Lalia *et al.,* 2013). Post-drying, the residual water vaporizes, resulting in the formation of fine pores. Figure 2v illustrates this phase inversion mechanism, highlighting solvent evaporation and non-solvent replacement as critical factors in pore generation. As solvent evaporation and water substitution proceed, the mixture undergoes a transition to a solid phase during aging, leading to the formation of pores as the remaining water evaporates (Mod *et al.,* 2019).

The second mechanism involves the interfacial interactions between the filler and the polymer matrix. Effective filler-polymer interactions ensure that the filler particles are uniformly coated with the ENR/PVC mixture. Conversely, poor interactions create gaps between the filler and the matrix, which manifest as functional pores within the thin film. Micrographs (Figure 2) provided proof of this phenomenon, showing that insufficient interaction between the cellulose filler and the ENR/PVC matrix resulted in pores that were unevenly distributed and sized (Shamsuddin *et al.,* 2013). The third process concerns the interactions between the filler particles in the matrix. Particles agglomerate when filler concentration rises, forming filler clusters. These clusters are close to one another, which causes spaces between them to form pores (Nor *et al.,* 2016). Increased filler content makes these agglomerations more likely, which increases the pore size of the membrane and its porosity.

CONCLUSION

Critical information about the mechanisms of graft copolymerization and pore formation is provided by the surface morphology and chemical properties of the filler (cell-g-PMMA) and the ENR/PVC/Cell-g-PMMA membrane. The ester carbonyl (C=O) group of PMMA and the formation of aldehyde (HC=O) functional groups, indicated by the stretching vibration of CH bonds at 2776 cm^{-1} and an absorption peak at 1736 cm^{-1}, respectively, were detected by FTIR spectroscopy as proof of the successful grafting of PMMA onto the cellulose backbone. VPSEM showed that grafting PMMA onto cellulose resulted in rough, flaky surface roughness and an increase in fiber diameter from about 4.5 µm to 10 µm. The addition of cell-g-PMMA filler to the ENR/PVC matrix caused the membrane's surface to become more textured as tiny pores began to form. Pores were found within and between filler particles as well as between the filler and the membrane matrix, according to cross-sectional examination. The improved pore development within the membrane was largely caused by interactions at the interfaces between the filler, the membrane, and the filler particles. All things considered, the grafting of PMMA onto cellulose and its incorporation into the ENR/PVC membrane changed the chemical composition and surface morphology, leading to a rise in porosity.

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