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# Preparation of ENR/PVC/RH Composite Membrane for Water Permeation Application

Norfarhana Ab. Samad[a], Nazwa Jon[b], Mohd Azwan Shah Mat Lazim[b], Ibrahim Abdullah[b] and Rizafizah Othaman[b]

[a]*Department of Petrochemical Engineering,*
*Politeknik Pagoh Johor, Hab Pendidikan Tinggi Pagoh, 84500 Muar, Johor, Malaysia*

[b]*School of Chemical Sciences and Food Technology, Faculty of Science and Technology,*
*Universiti Kebangsaan Malaysia, 43600 Bangi Selangor, Malaysia.*

rizafizah@ukm.my

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**ABSTRACT**

Composite membrane from Epoxidized Natural Rubber/ Polyvinyl Chloride/ Rice Husk (ENR/PVC/RH) was prepared via phase inversion method. The porous membrane was developed that can be used for water permeation application. The effects of addition of RH on thermal stability, mechanical properties pores development, morphology, water absorption, and water permeation of membrane had been investigated. The thermogravimetric curve of ENR/PVC membrane was characterized by two peaks that correspond to thermal degradation of PVC at 250-300 °C and ENR/PVC at 250-500 °C. Thermal stability of the membranes improved with the addition of RH and the residual mass of ENR/PVC membrane increased with RH loadings. However, increasing RH loadings in ENR/PVC blends resulted in the reduction of tensile strength due to the presence of pores and fillers in the membranes. On the other hand, tensile modulus of the ENR/PVC/RH membranes increased with the incorporation of RH. SEM images showed that the ENR/PVC membrane with 5 wt% and 10 wt% of RH had pores on the surface which consequently enhances the water absorption, flux and permeability of the membranes. Hence, RH was capable improving thermal stability, mechanical properties , morphology and performance of the ENR/PVC membranes and potential for utilization in water separation process.

## 1. Introduction

Membrane is defined as a barrier between two or more phases of a mixture that allows one component of the mixture to permeate freely while hindering permeation of other components [1]. Membrane separation process are the excellent medium of separation without addition of adsorbent. This technology offer many advantages in terms of less energy requirement, environmental friendly, easy to operate and use compact equipment compared to the conventional water separation processes [2]. However, the most important part in membrane separation process is the performance of the membrane. Therefore, the suitable method and materials for water treatment application is highly needed. Porous structure is important to induce flux, permeability and selectivity in membrane to be applicable for water treatment. Various methods are used to prepare porous membranes such as sintering, stretching, track-etching, template leaching, phase inversion and coating [3]. The most commonly employed and commercially available method is phase inversion [4].

Polyvinyl chloride (PVC) is a polymer with outstanding properties that used to prepare microporous membranes due to it stiffness, flexible, durable, excellent resistance to abrasion and low cost. Epoxidized Natural Rubber (ENR) is a versatile elastomer that polar, high in strength, high oil resistance, and high tensile and tear strength. The blending of PVC with ENR is beneficial where the PVC is expected to impart high tensile strength, good chemical resistance whereas ENR have good tear strength and acts as a permanent plasticizer to PVC. This thermoplastic elastomer was reported to improve the physical and mechanical properties of material and to form miscible blend at any proportion. [5-8]. The use of rubber based membrane for various industrial separation process has been explored by a number of researchers [9, 10]. Chen et al. [11] studied the separation properties of alcohol-water mixture through silicone rubber membrane filled with zeolite silicalite- I by different treatment for pervaporation application while Achalpurkar et al. [12] studied the potential of amine substituted silicon rubber (ASR) membranes on gas permeation properties. However, no work has been done for rubber based membrane in water separation application.

In recent years, the incorporation of agro-based renewable materials in polymer matrix have received more attention that are evolving as potential alternative to mineral fillers in a wide variety industrial applications. Fillers from agricultural sources (such as kenaf, pineapple, rubber wood, palm oil empty fruit bunch) have been utilized to improve material properties of polymer composites mainly due to their low cost, low density, high specific strength and modulus, environmental friendly, fully biodegradable and renewable nature [13-16]. Rice Husk (RH) is a major agro-waste product and is abundantly available in Malaysia and neighbouring countries., RH can also be utilized as an alternative as a low cost filler to reduce the waste and improve the properties of the composite since RH is an excellent source of silica and lignocellulosic material. Ray et al. [17] reported that filler loading natural rubber (NR) membranes showed better toluene selectivities than unfilled membranes. Many studies have described the use of RH as filler in polymer matrix improved the thermal and mechanical properties of the composites [18-20]. It has been reported that the use of RH in natural rubber/ linear low density polyethylene (NR/LLDPE) composite results in an increase of tensile modulus and hardness [21]. The enhancement of modulus and hardness of the NR/LLDPE composite indicates the ability of RH to impart greater stiffness to the blends [13]. Similar observation had been reported by Premalal et al., 2002 [14]. Besides, the addition of RH enhances the water absorption in polymer matrix.

In this study, RH was selected as fillers with the ENR/PVC matrix. It was expected that RH would improve the thermal stability, mechanical properties and morphology of ENR/PVC membrane. The main objective of the present study to prepare porous membranes from ENR/PVC/RH by phase inversion method. The effects of RH loadings on the characterization and water permeation of ENR/PVC composite membrane were studied and discussed.

## 2. Experimental

*2.1 Materials*

ENR of 50 % epoxidation level (ENR-50) was obtained from Rubber Research Institute of Malaysia (RRIM) and Polyvinyl Chloride (PVC) from Industrial Resin (M) Ltd. Tetrahydrofuran

(THF) and sodium hydroxide (NaOH) from Merck, USA. Rice Husk (RH) was obtained from Merbuk MDF Sdn. Bhd., Malaysia.

*2.2 Sample preparation*

RH was grinded using a crusher machine and sieved to 45-63 µm size. The RH powder was pre-treated with 5 % NaOH for 2 h under constant stirring condition. Then the mixture was filtered and rinsed with distilled water until pH 7 was obtained. The pre-treated RH was dried in oven at 60 °C for 24 h. Then, the RH was re-grinded and sieved to the similar size (45-63 µm) and kept in a desiccator.

The ENR/PVC (40/60 by weight percent) matrix was prepared by melt blending in an internal mixer at 160 °C and a mixing rate of 50 rpm for 13 min. The blend was swelled in tetrahydrofuran (THF) and the solution was vigorously stirred for 24 h to homogenize the mixing. Various loading of RH (0, 1, 5, 10 wt % ) were added and the solution was stirred continuously for another 24 h. Then, they were degassed at room temperature by ultrasonic bath for 30 min to remove bubble. The solutions were casted on glass plate using a casting knife and immediately immersed in coagulation bath (water) for phase inversion process. The composite membrane was removed from the glass plate, rinsed with water and dried at ambient temperature. The thickness of the membranes was about 0.05-0.10 mm.

*2.3 Membrane characterization*

Thermal properties of the materials were analysed out using a thermogravimetric analyzer (Mettler Toledo TGA/SDTA 85f model). The thermogravimetric analysis was performed under nitrogen atmosphere at a heating rate of 10 °C/min.

The surface and cross sectional images of the membranes were obtained using a LEO model 1450VP scanning electron microscope (SEM) under low vacuum operation at 20 kV. The membranes were cut under liquid nitrogen and then sputter coated with gold.

In order to evaluate the water absorption and porosity of the membranes, the membranes were cut into 14.6 $cm^2$ discs. Then the membranes were dried in a vacuum oven at 60 °C for 24 h and then weighed as $W_d$. After that, the membrane was immersed in water for 24 h and weighed as $W_w$ after surface moisture was wiped. The percent water absorption and porosity of membranes were calculated as follows [2, 22]:

$$\text{Water absorption}\ (\%) = \frac{Ww - Wd}{Ww} \times 100 \quad (1)$$

$$\text{Porosity}\ (\%) = \frac{Ww - Wd}{Ah} \times 1000 \quad (2)$$

where $W_w$ and $W_d$ are the weights of wet and dry membranes, respectively; $A$ the membrane surface area ($m^2$) and $h$ the the membranes thickness (mm).

A dead-end stirred cell filtration system (Model 8200) connected with a nitrogen gas cylinder was used to determine the water flux and permeability of ENR/PVC membrane. Water flux and permeation measurements were carried out using distilled water at a constant time. The effective area of the membrane was 14.6 $cm^2$ and all experiments were conducted at room temperature. The operating pressure of the experiment was 1–5 bar. Water flux of the membranes was calculated as follows [2]:

$$\text{Flux} = \frac{Q}{A\,\Delta t} \quad (3)$$

where Q is the quantity of permeate water (L), A the membrane surface area ($m^2$) and $\Delta t$ the permeation time (h). A graph of water flux vs. pressure was plotted and the water permeability of the membrane was determined from the slope.

The mechanical properties of the membranes were measured by a universal testing machine (UTM) (Instron-5566 model) at a jogging rate of 50 mm/min at 25±2 °C with ±50 N loading. The samples were cut into pieces in a dumbbell-shaped that are specified by ASTM D412.

## 3. Results and Discussion

### *3.1 Thermogravimetric Analyzer (TGA)*

Figure 1 and 2 shows the results of thermogravimetric (TG) and differential thermogravimetric (DTG) analyses for ENR/PVC membranes with various amount of RH. The decomposition profiles of the ENR/PVC membrane can be characterized by two peaks corresponds to thermal degradation of PVC, starts at about 230 °C until 500 °C and ENR, starts at 300 °C until 400 °C, respectively [23]. In the DTG curve (Figure 2), three degradation peaks was observes. The first and second degradation peaks at 230-350 °C indicates the dehydrochlorination of PVC while the last degradation peak at 380-500 °C was due to the the decomposition of both ENR and PVC [24, 25]. Nair et al. [25] observed two dominant degradation peaks during TGA analysis of ENR/PVC blends where the first peaks correspondings to the elimination of HCl molecules from polyene chains of PVC and the second peak due to the fragmentation of polyisoprene chains of ENR and the residual polyene formed after the dehydrochlorination of PVC.

The TG curves in Figure 1 show the residual mass of the membranes increased with the increment of RH loadings. The residue of the ENR/PVC, ENR/PVC/RH-1%, ENR/PVC/RH-5% and ENR/PVC/RH-10% membranes are 4.6, 5.1, 5.3 and 6.3 %, respectively. The increased residual mass at high temperature is due to the presence of ash as well as lignin [26]. Besides that, the high silica content of the RH is also a factor that contributes to the high residual mass of membranes thus increasing the thermal stability of the membrane [27]. From the DTG curve in Figure 2, the shoulder observed at around 230-250 °C for PVC decomposition and sharp peaks at around 400-450 °C for ENR decomposition disappeared with RH loadings, indicates an interfacial interaction between RH and the polymer matrix.

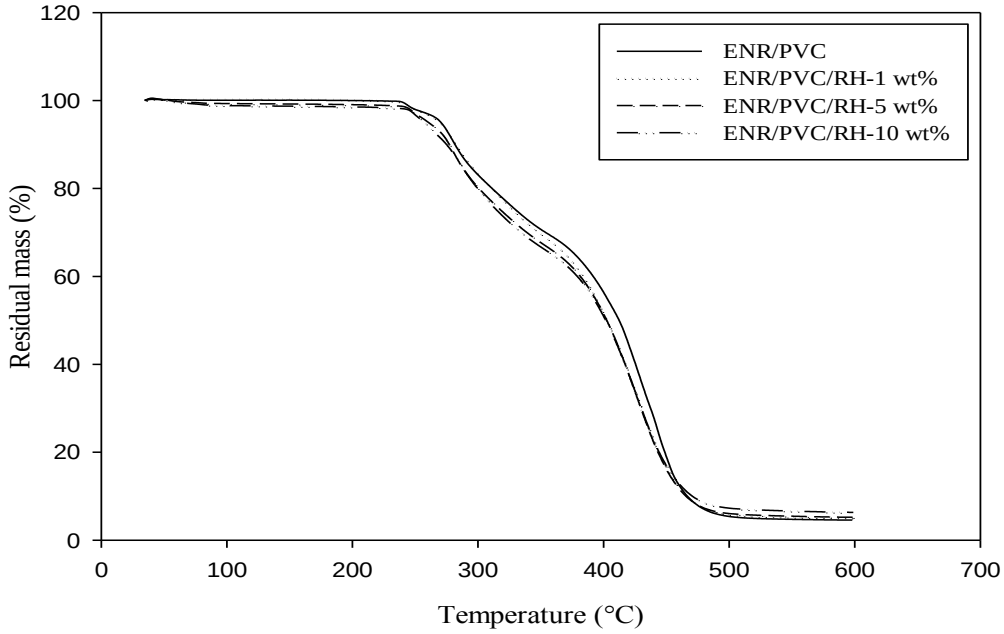


Figure 1. TGA curves for ENR/PVC membrane with various amount of RH

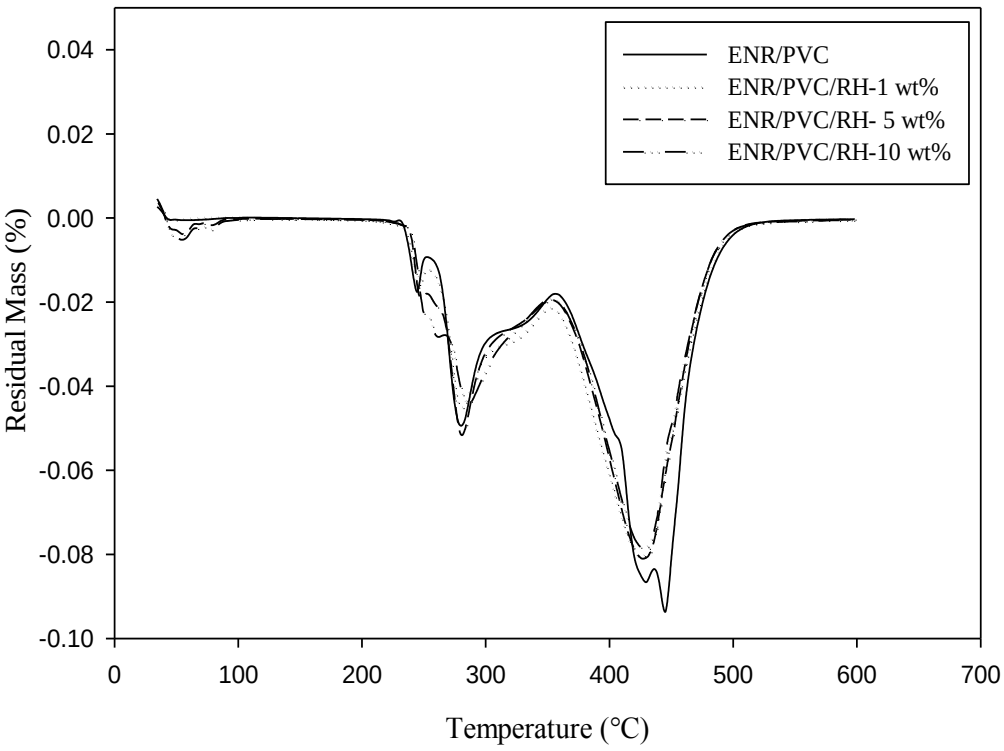


Figure 2. DTG curves for ENR/PVC membrane with various amount of RH

*3.2 Morphology (SEM)*

Figure 3 and Figure 4 represent the SEM micrograph of the surface and cross-section of the membranes , respectively. The pores development is clearly seen in ENR/PVC with addition of 1, 5 and 10 % RH membranes as compared to the smooth ENR/PVC and the number of pores enhances with the increasing of RH loadings. This pores were developed when the casting solution is immersed directly into a non-solvent bath. When the solvent exchanged with water, the space after water evaporation would turn into pores in the ENR/PVC/RH membranes [4, 28]. The phase inversion process is possible to prepare pores structure of thermoplastic elastomer composite membrane that possible used for water separation process.

From the figures, more pores were developed with the increasing of RH loading. This results shows the RH potential to develop pores of the ENR/PVC membrane. Besides that, Wu et al., 2009 [29] revealed that RH performed as pore former during fabrication of porous glass–ceramic scaffolds. This also confirm by porosity result that show as increased amount of RH, the percentage of porosity also increased. However, the distribution of pores on the membranes surface are heterogenous. It may be due to agglomeration of RH particles and poor adhesion between RH and matrix. Hussein et al., 2011 [30] reported as the filler content increases, the formation of agglomerations increases. This is due to the difficulties of achieving a homogeneous dispersion of filler at high filler content which in turn resulted in heterogenous pores.

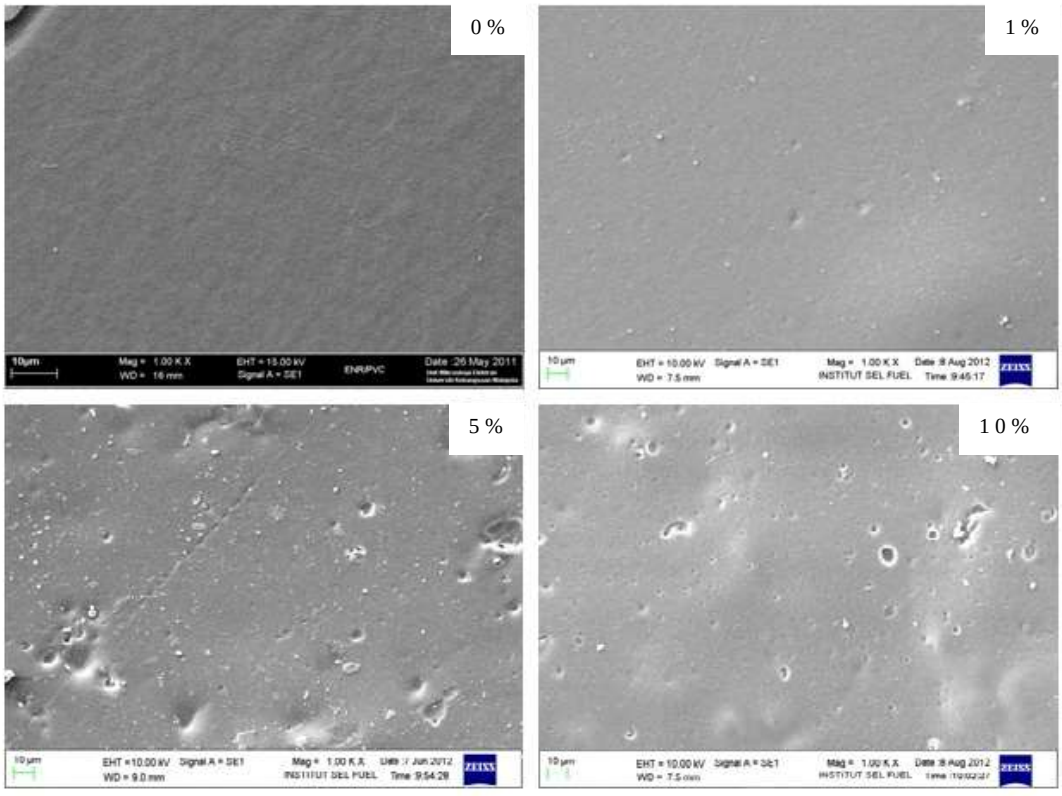


Figure 3. Effects of RH loading on the surface of ENR/PVC membrane at magnification of ×1000

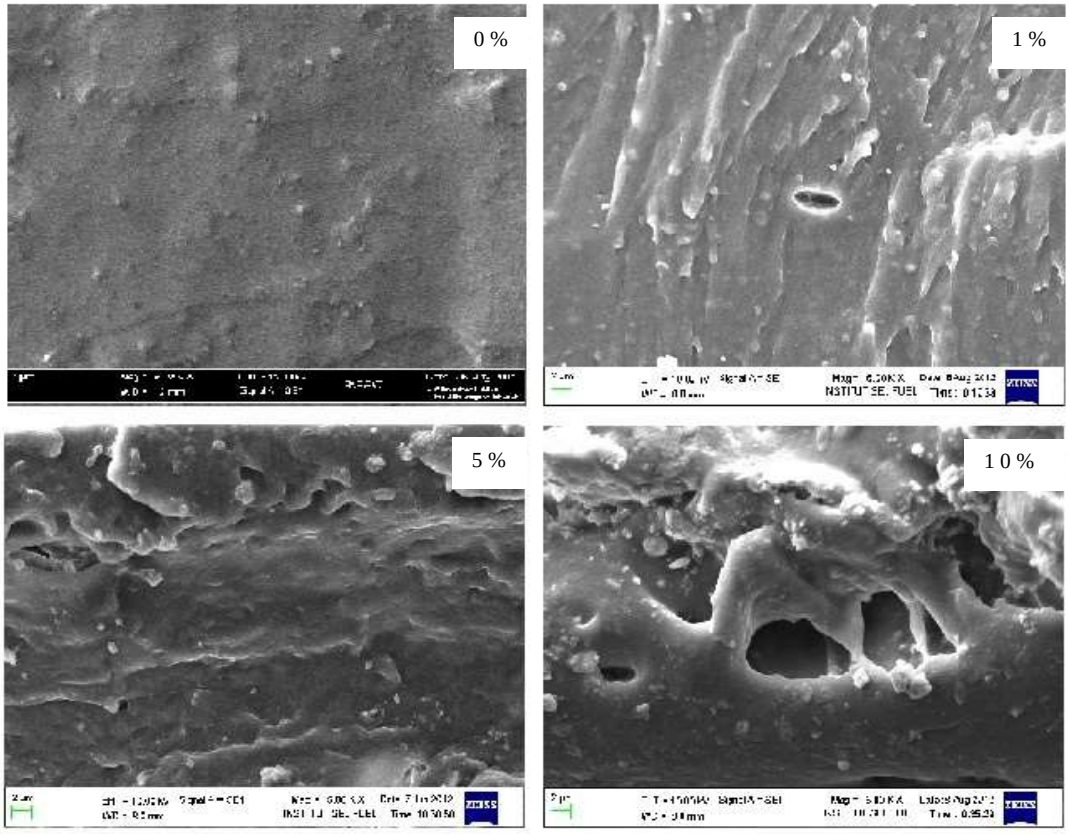


Figure 4. Effects of RH loading on the cross-section of ENR/PVC membrane at magnification of ×5000

*3.3 Water absorption and porosity of membranes*

The water absorption and porosity of ENR/PVC membranes with various amount of RH loading is shown in Table 1. The water absorption increased when the RH loading increased. The increasing in water absorption with RH loading is due to the hydrophilic nature of RH as cellulose fibres [18]. Percentage of water absorption depends on the nature of the fibre [31]. As the RH loading increased, the cellulose content increased, which in turn resulted in the absorption more water. This results agrees with those from Hardinnawirda and SitiRabiatull Aisha, 2012 [32] where the water absorption increased when RH filled unsaturated polyester resin (UPR) composites. Moreover, the water absorption increased due to the presence of voids in the natural filler polymer composites. These voids formed because of the poor adhesion between hydrophobic matrix, ENR/PVC and hydrophilic fillers, RH. When the membrane exposed to the moisture, the hydrophilic RH will swells resulted in the de-bonding of filler and matrix that formed voids.

As shown in Table 1, presence of RH increased the porosity of ENR/PVC membrane. The increase in porosity with RH loading is due to the phase inversion process in which the solvent was exchanged with water, and as it happens the space after water evaporation would turn into pores in the ENR/PVC/RH membranes. Wu et el., 2009 [29] reported that RH had potential as pore former agent. Hence, in this study RH not only as reinforcing filler but helps in the development of pores on the ENR/PVC membrane.

Table 1

Water absorption and porosity of the membranes

| Membrane type | Water absorption (%) | Porosity (%) |
|---|---|---|
| ENR/PVC | 7.1 | 9.9 |
| ENR/PVC/RH-1wt% | 10.6 | 16.4 |
| ENR/PVC/RH-5wt% | 12.4 | 19.9 |
| ENR/PVC/RH-10wt% | 16.4 | 26.9 |

*3.4 Water flux and permeability of the membranes*

Water permeation experiment is among the most important methods to determine the structure and morphology of ENR/PVC/RH membranes. The results of the water flux and water permeability of the membrane are shown in Figure 5 and Table 2, respectively. No water permeation was detected for ENR/PVC and ENR/PVC/RH-1% membranes showing that rubber based membrane was not permeable to water. For ENR/PVC membranes with 1% RH no water was permeated because just a few pores were developed on the surface that not allowed the water permeation. By adding RH for 5% and 10% RH, the membrane became permeable to water since more pores were generated through the membrane can be as seen in the SEM micrograph. The water flux of both membranes increased with pressure. This is common phenomenon for a pressure driven membrane.

According to Table 2, permeability of water increased with RH loading. RH as hydrophilic filler increases the hydrophilicity of the membrane and enhances the water permeation. Moreover, as supported by SEM images, RH changes the morphology of the membrane by developing pores in the membrane to induce water permeation. Thus, the permeability increased due to the formation of pores on the membrane surface.

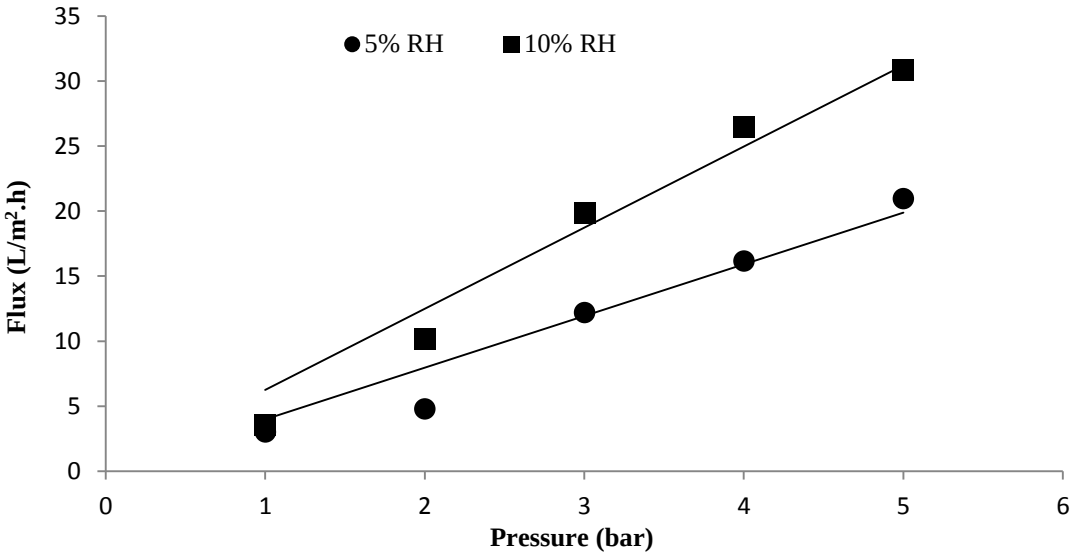


Figure 5. Effects of RH loading on water flux of ENR/PVC membrane

Table 2

Permeability of the membranes

| Membrane type | Permeability (L/m$^2$.h.bar) |
|---|---|
| ENR/PVC | - |
| ENR/PVC/RH-1wt% | - |
| ENR/PVC/RH-5wt% | 3.98 |
| ENR/PVC/RH-10wt% | 6.24 |

*3.5 Mechanical Properties*

The effect of RH loading on the mechanical properties of ENR/PVC membrane is shown in Figure 6. The addition of RH exhibit low tensile strength of the membranes. This phenomenon can be attributed to the membrane structure. As seen from the SEM micrographs, the development of pores on the surface may be affected the reduction of tensile strength of the membranes. The development of pores on the membranes surface results in poor interfacial adhesion between filler and matrix which reduced the tensile strength. Besides, the reduction of the tensile strength may be due to the incorporation of filler (RH) in the matrix which disrupts the elasticity of the polymer membranes [33,34]. This demonstrates that the fillers had hardened the composites and reduced their ductility.

The ductility of the composites had reduced with the presence of the fillers and increased their stiffness [31]. From the graph, the tensile modulus of the membrane increased with the addition of RH. The increasing of tensile modulus with increasing amount of RH into the matrix may be due to better dispersion of the filler which results in good interfacial adhesion between filler and matrix. Syafri et al., 2011 reported the incorporation of RH into the matrix improved the stiffness of the composite membranes [21]. The addition of RH into the ENR/PVC matrix reduced the polymer chain mobility, consequently producing more rigid composites that decreased the tensile strength and increased the tensile modulus.

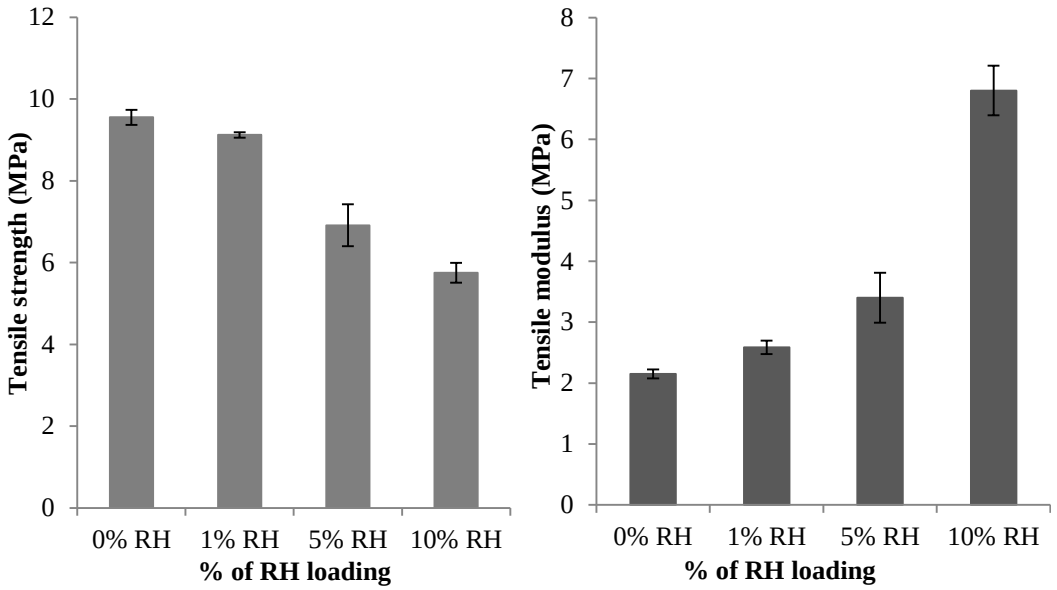


Figure 6. Effects of RH loading on the mechanical properties of ENR/PVC membrane

**4. Conclusions**

The effect of RH loading on the characterization and performance of ENR/PVC thin film composite membrane was investigated. It was found as increase in the RH loading will increase the thermal stability and water absorption of ENR/PVC/RH membranes. The water uptake was due to the increased cellulose content. Moreover, the water absorption increased due to the presence of pores in the membrane. Higher loading of RH caused more pores to be developed on ENR/PVC membranes, thus increasing the porosity, flux and permeability for the membrane to be applicable for water separation. Although the RH and the pores developed caused a reduction the tensile strength of the membranes, at the same time the tensile modulus increased since the incorporated RH acts as a reinforcing filler. It is concluded that RH enhanced the thermal stability, morphology and performance of the ENR/PVC thin film composite membrane towards water permeation.

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