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FABRICATION OF POLYETHERSULFONE – POLYITACONIC ACID (PES-PIA) MEMBRANE FOR REMOVAL OF REACTIVE RED 120

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Abstract:

The hazardous and mutagenic properties of dye effluents discharged into water bodies have led to the development of membrane separation for water treatment. pH-sensitive membranes have been widely employed as they exhibit remarkable improvements in the permeability and separation performance of polyethersulfone (PES) membranes. In this study, the PES membranes incorporated with polyvinylpyrrolidone (PVP), pluronic F108, and polyitaconic acid (PIA) by phase inversion were fabricated and characterized. PIA at different weight percentages were introduced into the PES dope solution via blending technique. The dye removal capacity, chemical composition, and thermal stability were characterized using UV-Vis spectrometer, fourier transform infrared (FTIR), and thermogravimetric analyzer (TGA) respectively. The fabricated membranes showed excellent rejection of reactive red 120 (RR120), which is an anionic dye. The study evaluated the membrane performance of pure water permeation (PWP) along with RR120 dye rejection. M20-PPI1, with a weight percentage of PVP/Pluronic F108/PIA; 3%/1.5%/0.25%, exhibited the highest water flux of 12 L/m^{2}h and dye rejection of 82% at pH value of 2 for RR120 dye rejection. The findings propose that M20-PPI1 is an effective pH-sensitive membrane for dye rejection.

Keywords:

pH-Sensitive Membranes, Polyitaconic Acid (PIA), Reactive Red 120 (RR120), Pure Water Permeation (PWP), Dye Rejection

Introduction

Synthetic dyes have been widely used in the dyeing process and might contain hazardous and mutagenic properties. It is also hard to degrade where releasing them into the water bodies will result in adverse effects to health and environment including water pollution. Hence, the development of an ideal and efficient material and method is greatly crucial for dye removal from wastewater (Hosseinifard et al., 2020). Membrane separation had been widely recognized to resolve these environmental contamination issue (Mohammadnezhad et al., 2019).

Membrane technology has been extensively applied for various wastewater treatments. This is due to its selectivity and permeability properties as one of the promising separation techniques (Zhao et al., 2018). Previous research had proposed polyethersulfone (PES) as an effective aromatic polymer in membrane fabrication including nanofiltration (NF) membranes which exhibited high thermal and mechanical resistance (Ladewig & Al-Shaeli, 2017). Prior study reported that polyvinylpyrrolidone (PVP) is one of the various types of additives that had been included in the PES system, it acts as a pore former and structure controlling agents (Milescu et al., 2019). It is also found that the employment of various surfactants as an additive in the dope solution had been successful in fabricating highly efficient membranes with high selectivity (Bolandi et al., 2020). Hamzah and co-workers have employed pluronic F108, a non-ionic surfactant which led to the increase of water flux, membrane permeability and selectivity (Hamzah et al., 2020, 2018).

Fan, Purkait and co-workers also suggested that pH-sensitive membranes displayed excellent improvements towards the permeability and separation performance of polymeric membranes. The studies have stated that, functional groups such as carboxyl and pyridine displayed weak polyelectrolytes behaviors with a good pH-sensitive polymers. The utilization of polymerized itaconic acid, polyitaconic acid (PIA) into the membrane matrix fabrication would relatively produce this pH-sensitive properties. This is due to its highly functional polarity and polyelectrolyte properties of carboxylic acid groups (-COOH) and acetic acid groups (-CH_2COOH) in the PIA molecular structures (Fan et al., 2018; Purkait et al., 2018).

Nevertheless, PES membranes had displayed hydrophobic properties that greatly affect the membrane flux and causes fouling. Membrane filtration operations for wastewater treatment were prone to fouling due to the irreversible and reversible associations between the surface of membranes and numerous foulants. Thus, the modification of PES membranes either by chemical or physical means is vital, in order to enhance the hydrophilic properties of the fabricated membranes (Safarpour et al., 2016). The sole addition of additive was not sufficient in producing promising structural properties of the fabricated membranes by phase inversion approach. Therefore, the incorporation of other additives in the dope solution for membrane fabrication was required to overcome those limitations. No previous findings were found on the incorporation of PIA via blending into polymeric membrane matrix. Thus, the impact of the addition of PIA towards the PES matrix mainly in the membrane performance and structural properties were investigated in this research study. Furthermore, this newly formulated PES membranes were fabricated for the dye removal application. The findings of this study may have contributed new knowledge and findings through the incorporation of innovative materials in the membrane fabrication process for dye removal treatment. In this study, PES membranes incorporated with PVP, pluronic F108 and PIA by phase inversion were fabricated. The process started by blending the polymer, PES into the respective solvent, NMP to produce a well-dissolved polymer suspension. The PVP, pluronic F108 and PIA were incorporated in succession into the polymer suspension to produce a homogenous dope solution. The dope solution was then poured onto the glass plate and cast using a casting knife. After that, the dope solution was submerged into the coagulation bath containing a non-solvent, water. Finally, a cured polymeric membrane film was obtained. The dye removal capacity, chemical composition, and thermal stability were characterized using UV-Vis spectrometer, fourier transform infrared (FTIR), and thermogravimetric analyzer (TGA) respectively. Moreover, the PES membranes performance of the pure water permeation (PWP) was evaluated along with the RR120 dye rejection.

Methodology

Material

Polyethersulfone (PES), n-methyl-2-pyrrolidone (NMP) and polyvinylpyrrolidone (PVP) were purchased from Merck while pluronic F108, itaconic acid (IA) and reactive red 120 (RR120) dye were purchased from Sigma Aldrich. Ethanol and hexane were procured from HmbG Chemicals.

Preparation of Polyitaconic Acid (PIA)

Itaconic acid (IA) need to undergo polymerization process to produce polyitaconic acid (PIA). (Enchev, 2017) has proposed an effective procedure for the high conversion of itaconic acid polymerization. Firstly, 100 g of IA was half neutralized with NaOH in 50 mL cold deionized water by cooling it with ice water, which then produced a concentrated solution. This concentrated solution was purged with nitrogen gas to create inert condition and was heated to 90 °C to 100 °C. Next, 70 wt% of tert-butyl hydroperoxide (tBHP) which act as an initiator was injected to initiate the polymerization process. The polymerized solution became viscous with the increase in time. After the solution turned into brittle solid, the polymerization process completed. Afterwards, the PIA samples were cooled down and were ground using mortar and pestle. The PIA samples were then stored in a closed container.

Fabrication of Membrane

The PES membranes were fabricated via phase inversion approach. Firstly, PES was added gradually into the round bottom flask, containing NMP at 50 °C to 60 °C of heating under constant stirring. NMP was utilised as a solvent to dissolve PES for the phase inversion technique. This process was done in the fume hood until a homogenous polymer suspension was produced. PVP and pluronic F108 were subsequently added to the polymer suspension after all of the PES granules have dissolved. Different wt% of PIA samples obtained were also incorporated together into the dope solution by blending. The stirring of the dope solution was pursued for 8 hours at 50 °C to 60 °C of heating. The formulation of dope solution according to each membrane sample was tabulated in Table 1.

Table 1: Formulation of Dope Solution

Membrane	PES (wt%)	NMP (wt%)	H_2O (wt%)	PVP (wt%)	Pluronic (wt%)	PIA (wt%)
M20	20	75.00	5	0	0	0
M20-PPI1	20	70.25	5	3	1.5	0.25
M20-PPI2	20	70.00	5	3	1.5	0.50
M20-PPI3	20	69.75	5	3	1.5	0.75

The dope solution prepared was sonicated for 30 minutes to remove any air bubbles present. The dope solution was poured onto the glass plate and was cast slowly to the thickness of 100 µm to 150 µm by using a casting knife. The glass plate was soaked in distilled water (non-solvent) which acts as a coagulation bath. Later, it produced the membrane film. The membrane film was stripped carefully from the glass plate in the coagulation bath. After that, the membrane film was left immersed in the distilled water for 24 hours. Afterward, the membrane film was immersed in ethanol for another 24 hours and in hexane for 3 hours consecutively. The PES membrane film was then dried at room temperature.

Characterization and Performance Evaluation of Membrane

The membrane films were further characterized using UV-Vis spectrometer, fourier transform infrared (FTIR) and thermal gravimetric analyzer (TGA).

UV-Vis Spectrometer

UV-Vis spectrometer (Varian Cary 50) was operated at a wavelength range of 400 nm to 700 nm using a single beam configuration to investigate the absorbance of stock and filtered RR120 dye solutions.

Fourier Transform Infrared (FTIR)

The membrane films were characterized using FTIR spectroscopy (Nicolet iS50) to give information on the functional groups present in the PES membranes. FTIR spectroscopy was operated using attenuated total reflection (ATR) technique at wavenumber, λ ranging from 400 cm^{-1} to 4000 cm^{-1}.

Thermal Gravimetric Analyzer (TGA)

TGA (Mettler Toledo) was used to characterize the distribution of chemical components in the membrane formulation along with the thermal stability of the membrane samples. The characterization was based on the decomposition curve shift. This was done by placing about 15 mg samples into the platinum crucible. TGA was operated under a nitrogen atmosphere with a heating rate of 20 °C/min and a flow rate of 50 mL/min at a temperature range of 30 °C to 1000 °C.

Pure Water Permeation (PWP)

PWP was utilized to evaluate the pure water flux and permeability of every membrane film that were produced (Lau & Ismail, 2017). The dead-end filtration system was pressurized to several pressure ranging from 3 to 7 bars containing membrane film with 30 mL distilled water in the pressurized cell. The water permeation was evaluated by quantifying the water passing through the membrane film or water flux, (Jw) at a rate of L/m^2h. The water permeation was computed using Eq. (1). Where Qp was the volume of permeate (L), A was the active surface area of membrane film (m^2), and Δt was the duration time of the permeation procedure (h).

$$Jw = \frac{Qp}{A\Delta t} \quad (1)$$

Dye Rejection

The dye removal has also proceeded using the dead-end filtration system in pressurized conditions ranging from 3 to 7 bars. The membrane was firstly soaked in distilled water of different pH values of 2, 4, 6 and 8 for 24 hours before testing. Afterward, the dye rejection was tested with RR120 dye solution of matching pH values. This was based on the method that had been conducted by (Fan et al., 2018). The RR120 dye rejection percent were calculated using Eq. (2), where Cp and Cf were denoted as the concentration of solute in permeate and feed consecutively.

$$R(\%) = 1 - \frac{Cp}{Cf} \times 100\%$$

(2)

Results

Fourier Transform Infrared (FTIR)

Figure 1 depicts the FTIR spectra of pure PES membrane (M20) and PES-PIA membranes incorporated with PIA (M20-PPI1, M20-PPI2 and M20-PPI3). M20-PPI1, M20-PPI2 and M20-PPI3 showed broad and strong peaks ranging from 3403.74 cm^{-1} to 3431.32 cm^{-1} indicating O-H stretching vibration of itaconic acid. The intensity of O-H bands was slightly increased and may be due to the increment of PIA concentration incorporated in M20-PPI1, M20-PPI2 and M20-PPI3. At the wavenumber ranging from 2871.70 cm^{-1} to 2873.28 cm^{-1}, the PES-PIA membranes depicted medium peaks indicating C-H stretching vibration of Pluronic F108 and PVP. Additional small peaks were observed at the wavenumber ranging from 1715.23 cm^{-1} to 1718.43 cm^{-1} for PES-PIA membranes. This indicates the C=O stretch of PIA. The result was supported by findings of Wang et al., (2015) and Wei et al., (2018) which verify the incorporation of IA and carboxyl group into their compound. The slight increment of C=O bands intensity for M20-PPI1, M20-PPI2 and M20-PPI3 may be due to the increasing PIA concentration added into the PES-PIA membranes. Moreover, all samples showed medium to weak peaks ranging from 1577.13 cm^{-1} to 1577.62 cm^{-1} specifying C=C stretching vibration of the phenyl structure of PES. From the wavenumber 1071.62 cm^{-1} to 1235.62 cm^{-1}, strong peaks were observed for all samples indicating the C-O-C stretch of ether linkage of PES. The pure PES membrane and PES-PIA membranes also manifested C-H bending vibration of disubstituted, para phenyl structure of PES at the wavenumber ranging from 834.59 cm^{-1} to 870.78 cm^{-1}.

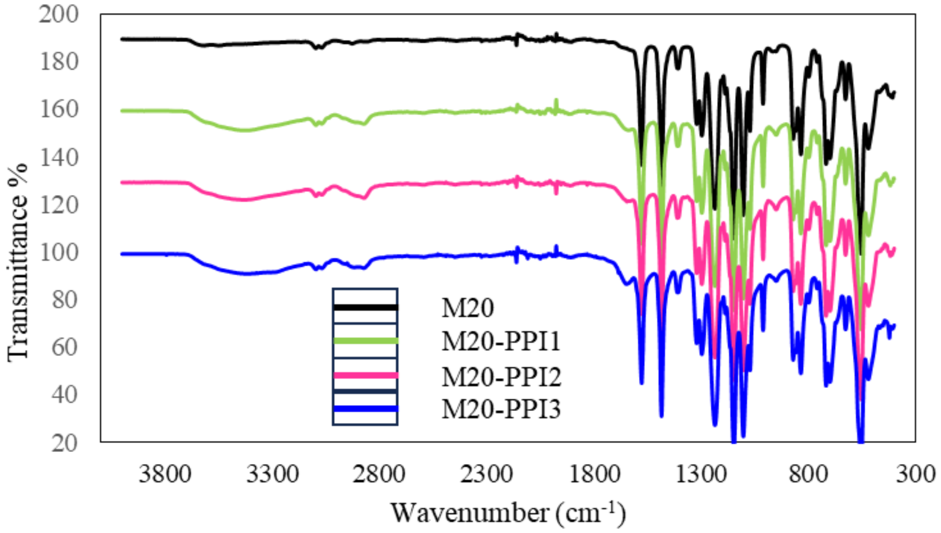

Figure 1: FTIR Spectra of Pure PES Membrane (M20) and PES-PIA Membranes Incorporated with PIA (M20-PPI1, M20-PPI2 and M20-PPI3)

Thermal Gravimetric Analyzer (TGA)

Figure 2 illustrates the TGA thermogram of pure PES membrane (M20) and PES-PIA membranes incorporated with PIA (M20-PPI1, M20-PPI2 and M20-PPI3). The pure PES membrane displayed mainly two stages of the TGA curve while the PES-PIA membranes samples depicted mainly three stages of the TGA curve. The first stage of decomposition started at the temperature of 40 °C to 120 °C with about 4% weight loss occurring at 110 °C for M20-PPI1 and at 120 °C for M20-PPI2 and M20-PPI3. This was due to the loss of water molecules and hydroxyl groups attached to the PES-PIA membranes. The second stage took place at the temperature ranging from 400 °C to 580 °C with approximately 52% weight loss observed at 570 °C for M20-PPI1, M20-PPI2 and M20-PPI3 and at 580 °C for M20. This indicated the decomposition of polymer chain cleavage of the pure PES membrane and the PES-PIA membranes. The last stage was from 580 °C to 1000 °C with around 12% weight loss for all samples depicting the carbonization of the whole polymer chain. Basically, the temperature of 570 °C for M20-PPI1, M20-PPI2 and M20-PPI3 and 580 °C for M20 were the main decomposition temperature. Although the thermal stability of PES-PIA membranes was slightly decreased from the pure PES membrane, they still displayed high thermal stability with a constant decomposition temperature between the samples, M20-PPI1, M20-PPI2 and M20-PPI3. Basically, the incorporation of PIA has produced PES-PIA membranes with considerably high thermal stability. The observations were supported by the previous thermal characterization studies Rowley & Abu-Zahra (2019) and Sakthivel et al., (2016).

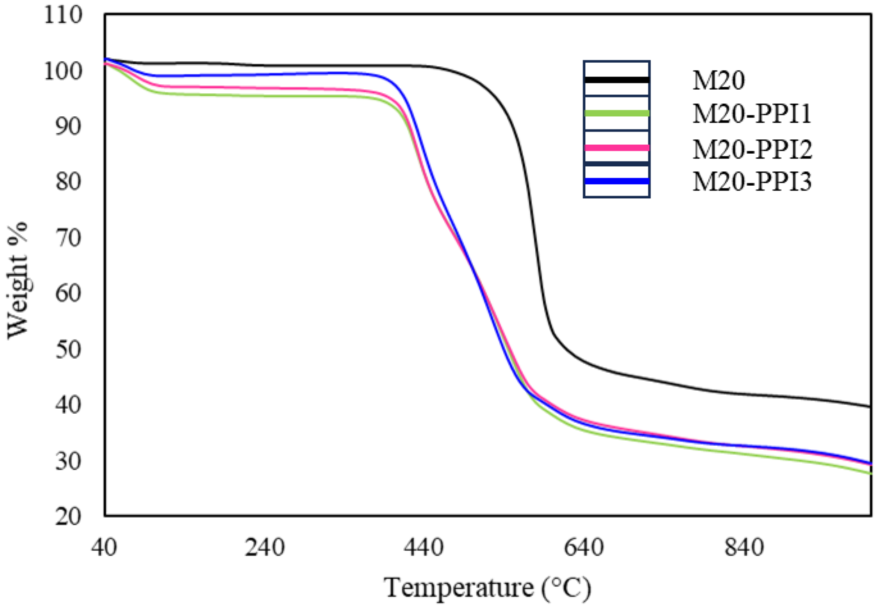

Figure 2: TGA Thermogram of Pure PES Membrane (M20) and PES-PIA Membranes Incorporated with PIA (M20-PPI1, M20-PPI2 and M20-PPI3)

Pure Water Permeation (PWP)

Figure 3 displays the water flux of pure PES membrane (M20) and PES-PIA membranes incorporated with PIA (M20-PPI1, M20-PPI2 and M20-PPI3). From the graph, the pure PES membrane, M20 showed incredibly low water flux with less than 0.1 L/m^{2}h. This was due to the hydrophobic properties of the PES polymeric membrane. M20-PPI1 on the other hand depicted the highest water flux with 11.73 L/m^{2}h at the lowest incorporation of PIA which was 0.25%. It was found that the incorporation of a small amount of PIA has produced PES-PIA membranes with significantly high-water flux. This might be because of the largest pore size present in M20-PPI1 compared to the other PES-PIA samples which were M20-PPI2 and M20-PPI3. The incorporation of 0.5% PIA of M20-PPI2 has led to a distinct decline in water flux to 0.42 L/m^{2}h. This was due to the reduction of the interspaces of pure PES chains due to the incorporation of increasing PIA which led to the decrease in the size of pores. However, a slight increase in the water flux of M20-PPI3 with 0.51 L/m^{2}h may be due to the uneven pore distribution within the pore networks. It can be observed that the incorporation of only a small amount of PIA into the PES-PIA membranes has improved the pure PES membrane permeability and hydrophilicity. The reasonings made were based on the pore size analysis investigation from previous findings (Fan et al., 2018).

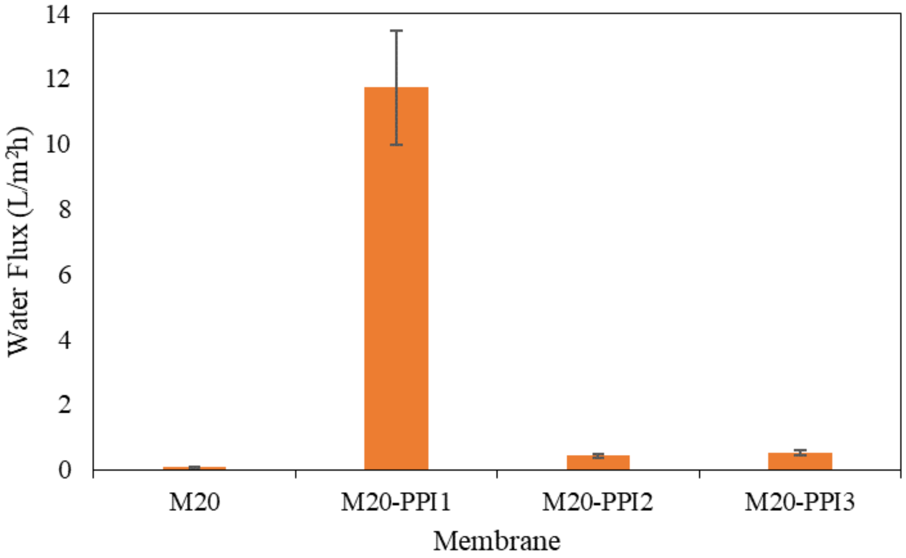

Figure 3: Water Flux of Pure PES Membrane (M20) and PES-PIA Membranes Incorporated with PIA (M20-PPI1, M20-PPI2 and M20-PPI3)

Dye Rejection

Figure 4 shows the rejection % of reactive red 120 (RR120) of pure PES membrane (M20) and PES-PIA membranes incorporated with PIA (M20-PPI1, M20-PPI2 and M20-PPI3) at varying pH. The pure PES membrane, M20 displayed constant RR120 dye rejection of 45%. This specified that the RR120 dye rejection of the M20 was pH independent. The RR120 dye rejection % decreased along with the increase of pH for the blended PES-PIA membranes. The RR120 dye rejection of M20-PPI1 at pH values of 2,4,6 and 8 were 82%, 48%, 42% and 1% respectively. For M20-PPI2, the RR120 dye rejection were 35%, 17%, 6% and 3% at pH 2,4,6 and 8 respectively. The RR120 dye rejection % at pH values 2,4,6 and 8 were 28%, 12%, 10% and 3% for M20-PPI3 respectively. The findings of the study exhibited that the blended PES-PIA membranes showed the highest RR120 dye rejection at a pH value of 2 with 82%, 35% and 28% for M20-PPI1, M20-PPI2 and M20-PPI3 membranes, respectively. On the other hand, the lowest RR120 dye rejection could be observed at pH value of 8 with 1%, 3% and 3% for M20-PPI3, M20-PPI2 and M20-PPI1 respectively. The hydrophobic benzene rings of RR120 and the aliphatic backbones of PES-PIA have a weak attraction for one another, hence, the major driving factor behind the RR120 rejection was electrostatic interaction. The findings supported the PES-PIA pH-sensitive behaviours. The high concentration of H^+ at low pH condition protonated the carboxyl groups present in PIA. The positively charged PES-PIA membrane interacted with the negatively charged RR120, which resulted in higher RR120 rejection at lower pH. The carboxyl groups present in PIA eventually underwent deprotonation as the pH values increase. This led to more negatively charged condition which resulted in the decrease of RR120 rejection at higher pH. This was due to the repulsion of both negatively charged PES-PIA membranes and RR120. The presence of pH-sensitive properties in the blended PES-PIA membranes has therefore been proven. The prior investigations by Lim et al., (2021), Wei et al., (2018) and Zhang et al., (2021) validated the observations made.

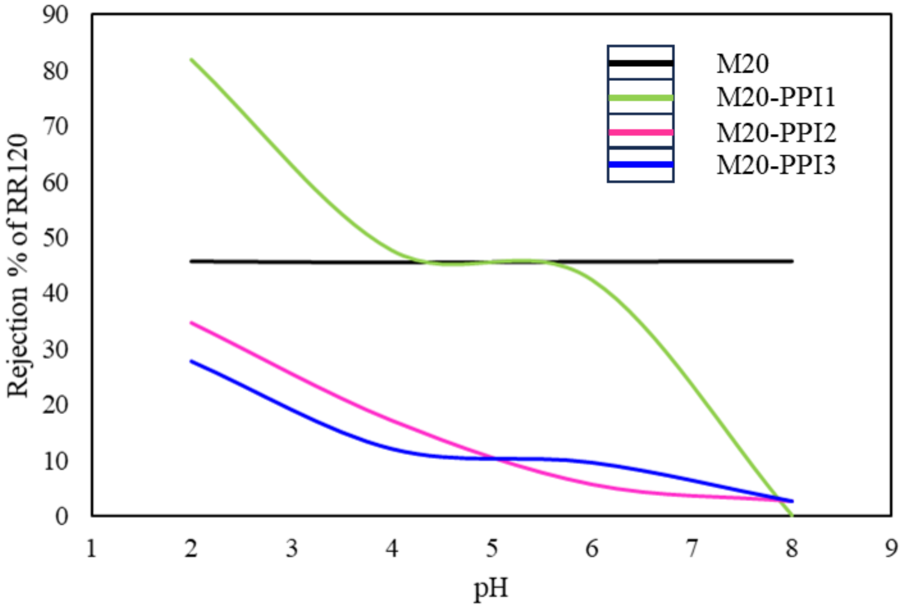

Figure 4: Rejection % of Reactive Red 120 (RR120) of Pure PES Membrane (M20) and PES-PIA Membranes Incorporated with PIA (M20-PPI1, M20-PPI2 and M20-PPI3) at Varying pH.

Conclusions

From this study, PES membranes incorporated with PVP, pluronic F108 and PIA were successfully fabricated and characterized. PES membranes performance in terms of PWP and RR120 dye rejection were also successfully evaluated. The study concluded that the FTIR analysis has confirmed the incorporation of PIA into the blended PES-PIA membranes. In addition, the TGA analysis has proved that the incorporation of PIA has produced PES-PIA membranes with considerably high thermal stability. The study also showed that M20-PPI1 with a weight percentage of PVP/Pluronic F108/PIA of 3%/1.5%/0.25%, exhibited the highest PWP of 12 L/m^{2}h and the highest RR120 rejection of 82% at pH value of 2. Moreover, the incorporation of PIA into the PES matrix has produced blended PES-PIA membranes with pH-sensitive characteristics.

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