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Polysulfone/TiO_2 Thin Film Nanocomposite for Commercial Ultrafiltration Membranes

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Abstract

Due to low water fluxes, commercial ultrafiltration (UF) membranes used in water treatment need to be improved. High-quality UF membranes were fabricated from polysulfone (PSF)/titanium dioxide (TiO_2) nanocomposite fibers as substrates using the spray pyrolysis method. The influence of nano-TiO_2 on the UF nanocomposite membrane was studied. Scanning electron microscopy (SEM), contact angle, and porosity were evaluated to characterize the mechanical characteristics of the membranes. The results show that adding TiO_2 to the substrates increased the hydrophilicity and porosity of the substrates. The pure water flux of the Thin Film Nanocomposite (TFN) membrane manufactured utilizing a PSF substrate coated with 0.1 wt% TiO_2 nanoparticles (denoted as Pc 0.1) improved at a rate of 35.28 l/m^2.h, and for a PSF substrate coated with 0.2 wt% TiO_2 nanoparticles (denoted as Pc 0.2) improved at a rate of 44 l/m^2.h. Additionally, increasing TiO_2 nanoparticle loading to 0.1 and 0.2 wt. percent resulted in higher water flow over 20 l/m^2.h PSF commercial membrane. The results of the UF performance show that Pc 0.2 membrane offered the most promising results, with a high-water flux than commercial membranes without nano-TiO_2 (Pc).

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Introduction

The Use of Nanoparticles (NPs) in polymeric membranes made them increase rapidly due to their unique properties such as nanoscale size, wide surface area, high reactivity, and degree of functionalization for water and wastewater treatment[1]. Domestic and industrial effluents are responsible for a large part of pollution on the planet [2]. The implementation of clean technologies on water treatment minimizes the negative impacts on the environment and improves water resources[3]. However, the inappropriate disposal of these effluents has caused concern for the scientific community, which challenges applying repairing processes for these environmental damages [4]. Conventional wastewater treatment processes include biochemical methods, centrifugation, ultracentrifugation, thermal treatments, and each of these processes has severe limitations, whether energetic, treatments (thermal and mechanical), or chemical [5]. Techniques that have been receiving more attention due to

their energy efficiency use membranes as an active principle for their operation, because it presents a clean technology, simplicity of operation, and has broad applicability, in addition to being possible to combine with other processes [6].

Membranes are semi-permeable separation barriers that act as a kind of filter, which can restrict, totally or partially, the transport of one or more chemical species present in the phases [7]. Furthermore, the pores of the membranes are responsible for the properties and the countless applications of the membranes, promoting separations between the particles and fractioning molecules of different molar masses [8]. They are prepared by using various techniques such as phase inversion, evaporation, or extrusion. For example, depending mainly on the materials that make up the membrane as well as various types of application, ranging from classical pressure processes, such as Microfiltration – MF, Nanofiltration – NF, Ultrafiltration – UF, and Reverse Osmosis RO to more recent emerging processes such as membrane contractors [9].

Among polymeric membranes, those produced with polysulfone (PSF) as the main component have been widely used to treat industrial effluents due to their desired properties such as stability, high mechanical strength, and ease of modification [10]. The modification of PSF membranes presents an excellent opportunity to improve their performance in wastewater treatment [11].

Until now, studies on nanomaterials and hydrophilic macromolecules used in the modifications of PSF ultrafiltration/microfiltration membranes for application in water treatment were extensively analyzed [12]. These modified membranes exhibited a remarkable improvement in water permeability, salt rejection, and anti-fouling characteristics of PSF modified membranes compared to pure PSF membranes[13]. However, because PSF, in its pure form, presents a very hydrophobic characteristic as the main limitation, modifications are necessary to improve its properties[14]. Therefore, the addition of inorganic components to the polymeric solution in the preparation of membranes are widely used to obtain nanocomposite membranes through the addition of inorganic nanoparticles such as clay[15], oxide of zinc (ZnO)[16], titanium dioxide (TiO_2)[17], graphene oxide (OG)[18], and inorganic salts, with the aim of improvement of the morphological, mechanical, and flow properties of polymeric membranes[19]. The addition of titanium dioxide nanoparticles to polysulfone membranes will make changes significant in the morphological structure, increasing the amount and size of pores present in the top surface and cross-section of such nanocomposite membranes[20]. In addition, it will promote resistance to scale formation and improvements in permeability[21]. Selectivity, mechanical and chemical resistance give the membranes the desired properties, thus favoring their application for the treatment of liquid effluents such as the greywater treatment. Thus, studies of insertion of nanoparticles in obtaining hybrid membranes applied in the treatment of greywater effluents represent an important contribution to academia and society, in addition to serving as support for future work.

A search of the literature revealed that few studies deal with TiO_2/PSF in the UF process. The current work addresses this research gap by producing TiO_2NPs loaded PSF for Greywater (GW) treatment using the UF procedure. In this study, TiO_2NPs are produced using a spray pyrolysis technique, then incorporated into a PSF commercial membrane (Pc) with polymer solution at various concentrations of TiO_2 with weight 0.1% (Pc0.1) and 0.2 % (Pc0.2). SEM, XRD, contact angle, and Pure Water Flux, were used to characterize the commercial membranes.

2. Experimental Procedure

2.1. Materials

The chemicals used in this study and suppliers are represented in Table 1.

Table 1: The chemicals materials, specifications and suppliers.

No.	Chemicals	Specifications		Supplier
1	**Polysulfone polymer pelts (PSF)**	Specific Gravity(g/cm^3)	1.23	Huaian Ruanka, China
		Water Absorption (%)	0.2	
		Modeling Shrinkage Rate (%)	0.7	
2	**Titanium Dioxide Nanoparticles (TiO_2)**	Empirical formula	TiO_2	Skyspring Nanomaterials
		Crystal structure	Anatase	
		Purity (%)	> 99.5	
		APS (nm)	10-30	
		SSA (m^2/g)	> 50	
3	**Dimethylformamide solvent (DMF)**	Chemical formula	C_3H_7NO	Sigma Aldrich
		Molar mass (g·mol^{-1})	73.095	
		Appearance	Colorless liquid	
		Density (g/ml)	0.95	
4	**Tetrahydrofuran solvent (THF)**	Chemical formula	C_4H_8O	Sigma Aldrich
		Molar mass (g·mol^{-1})	72.107	
		Appearance	Colorless liquid	
		Density (g/ml)	0.89	
		Molar mass (g·mol^{-1})	78.13	
		Appearance	Colorless liquid	
		Density (g/ml)	1.1004	
5	**Polyvinylpyrrolidone solvent (PVP)**	Chemical formula	$(C_6H_9NO)n$	Sigma Aldrich
		Molar mass (g·mol^{-1})	2,500	
		Appearance	white to light yellow	
		Density (g/ml)	1.2	

2.2. Membrane Type Selection

The membranes used are polysulfone polymer types. In the current study, two types of membranes were used (i.e., commercial) purchased from Zhangjiagang Chuntai Environmental Protection Mechanical Engineering, China, and self-prepared characteristics features of commercial polysulfone membrane as shown in Table 2.

Table 2: Specifications of PSF commercial membrane**.**

Parameter	Unit	Value
Material	**—**	Polysulfone (PSF)
Origin	**—**	China
Area	cm^2	15 x 12
Porosity	%	48.3 ± 2.6
Mean pore radius	Nm	6.9 ± 0.56
Pure water flux	l/m^{2}h	19.7 ± 3.2
Mean surface roughness	Ra-nm	2.9 ± 0.23
Layer thickness	Mm	200
Fabrication method	**—**	NIPS
Price	$	65 for 1 Piece

2.3. Instrumentations

Spray pyrolysis (SP) (Invents, Turkey) is the process in which a thin film is deposited by spraying a solution on a heated surface. The device consists of many specifications, as shown in Figure 1.

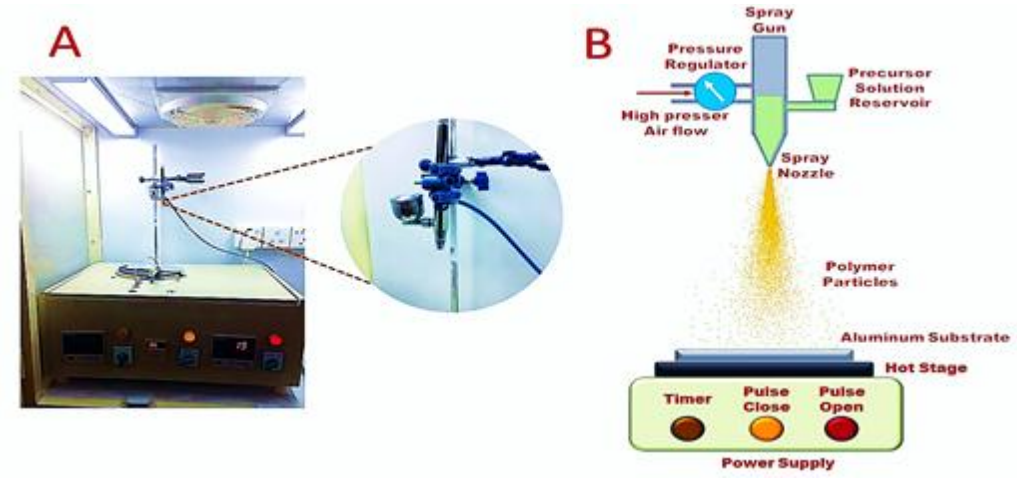

Figure 1: (A) Spray Pyrolysis device, (B) Device Diagram.

2.4. Preparation of Polysulfone Polymer Solution

Table 3 summarizes the specifications of the dope solution used in fabricating the spray pyrolysis process. This membrane was prepared before the dope solution preparation, and a vacuum oven was used to dry up the PSF pellets for a minimum of 3 h at about 90 ^{o}C to remove the moisture [22]. PSF particles were added to (25 g) and 0.025 g of PVP in (100 ml) of the mixture of DMF: THF as 70%:30% to enhance membrane mechanical properties due to improved solvent-induced fusion of interfering junction points. Solvents were converted from concentration in milliliters to grams using the following equation [23]:

$$\rho = \frac{m}{V} \quad (1)$$

Where; **ρ** = Density in g/ml for each solvent**, m** = Mass of solvent in gram unit/g, and **V** = Volume of solvent in milliliter unit /ml. The resulted mixture was stirred for 2 hours at 700 rpm and 70 ^{o}C. After that, a pale-yellow solution was obtained and then sonicated using ultrasonic (40 kHz) for 1 h at room temperature before the spray pyrolysis process started. This technique cleared any trapped air bubbles and got a clear homogeneous solution.

Table 3: The composition of spray pyrolysis dope solution preparation.

Spray Pyrolysis	PES (g)	PVP (g)	DMF (g)	*THF (g)
PSF	25	-	66.5	26.7
PSF/PVP	25	0.025	66.5	26.7

2.5. Preparation of PSF-TiO_2 (0.1 and 0.2 g) Solution

Initially, (0.1 or 0.2 g) of TiO_2 nanoparticles were dispersed in (25 ml) of DMF: THF as 70%: 30% using the ultrasonic device for 2 h at 50 ^{o}C. Then, the resulted dispersed solution of TiO_2NPs was added carefully to 100 ml of the solution under vigorous stirring at room temperature. After that, the mixture was sonicated for 10 h at 50 ^{o}C to produce a white clear homogeneous solution of PSF-TiO_2.

2.6. Membrane Fabrication

The spraying procedure worked by loading 10 ml from the prepared stock solution of PSF, PSF-TiO_2 0.1 g, or PSF-TiO_2 0.2 g were packed separately in the device nebulizer content. Compressed air was allowed to flow into the nebulizer sector from a height of 30 cm on a carrier material to nebulize the polymer solution. The nebulized polymer drops were collected on the aluminium substrate on the hot stage at 70 °C with an open: close pulse to 2:10 seconds. Table 4 summarizes the optimized Spray Pyrolysis Nanomeaterials (SPNMs) parameters. The generated random nanofibers were collected on a plate collector covered by aluminum foil upon completing the SP procedure. Subsequently, the resultant SPNMs were dipped in a deionized water bath for three days to eliminate any left-over solvents or Polyvinylpyrrolidone solvent (PVP). Finally, the nanofibrous mats were dried

in the air at room temperature for one day before storing them in a desiccator cabinet (Secador) for further treatment and instrumental characterization. This is the same mechanism used to make commercial membrane coatings by adding the same concentrations of nano-titanium oxide, as shown in Figure 2.

Table 4: Spray Pyrolysis conditions of SPNMs.

Spray Pyrolysis Parameters	SPNMs
Volume of solution	10 ml
Open: Close Pulse	2:10
The temperature of the collecting stage	70°C
Membrane fabrication time	18 h

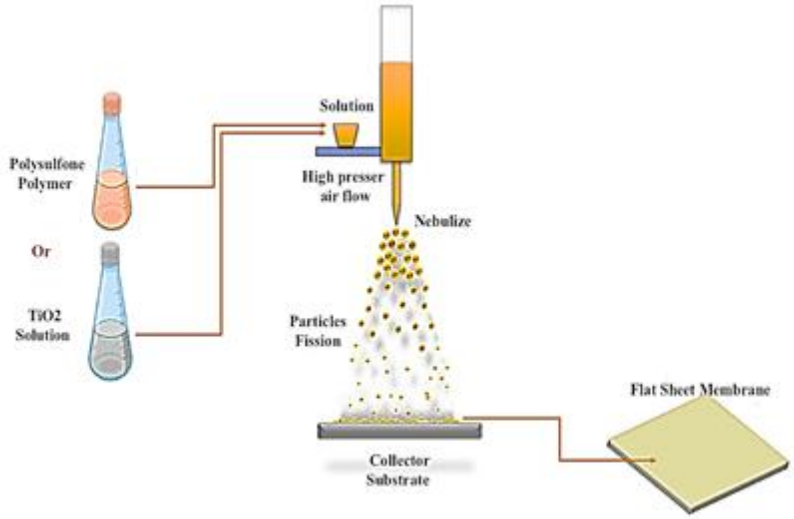

Figure 2: diagram of Polysulfone membrane fabrication.

2.7. Characterization of Nanofiber Substrates and TFMs Membranes

The morphological studies of the PSF /TiO_2 substrate and the membranes were conducted by a scanning electron microscope (EO Elektronen-Optik-Service GmbH, Germany). The energy dispersion of X-ray (XRD-6000, Shimadzu Japan) was used to determine the TiO_2 nanoparticle distribution in the Thin Film Nanocomposites (TFNs) membranes. Water contact angles of the nanofiber substrates were investigated based on the sessile drop method and determined using a contact angle instrument (FI-02130 Espoo, Finland). All tests were carried in (Physics and Chemistry Department/Nano-science and Technology /Al Zahra University /Iran).

Porosity was determined using the following Equation (2) [24]:

$$\textbf{Porosity (\%)} = \frac{\frac{wtw-wtd}{\rho k}}{\frac{wtw-wtd}{\rho k}+\frac{wtd}{\rho p}} \textbf{ x 100} \qquad \textbf{(2)}$$

Where; **wtw**: is the weight of the membrane in the wet state, **wtd**: is the weight of the membrane in the dry state, **ρk:** is the isopropanol density, and **ρp:** is the polymer density. Each value of porosity is the average of three different measurements.

2.8. Performance of The Membranes

2.8.1. Pure Water Flux and Rejection

Flux and rejection measurements were performed in cross-flow ultrafiltration mode. Three ultrafiltration membranes were used in separate processes. The flat sheet membrane was initially washed with deionized water before the experiment. Then, a Polysulfone flat sheet membrane cut into a square sheet with a surface area of 18 cm^2, was placed inside the membrane module, as shown in Figure 3. The membrane module consists of four vents, input and output for both feed and permeate. The membrane module was connected to a vessel, which contained feed solution, and a pressure gauge was placed at the membrane module inlet. Pure water flux was determined using deionized water as feed solution at pressure 2 bar followed by measurement of peat water flux,

where the membrane was operated for two hours. Permeate was collected periodically (15, 30, 45, 60, 75, 90,105, and 120 minutes) for 22 days. Pure water flux (PWF) is calculated by the following equation (3) [24]:

$$J_{w1} = V/A*T \quad (3)$$

Where; $\mathbf{Jw_1}$ = PWF ($m^3/m^2 \cdot s$), **V** = volume of permeate (m^3), **T** = permeation time (s), and **A**= membrane surface area (m^2).

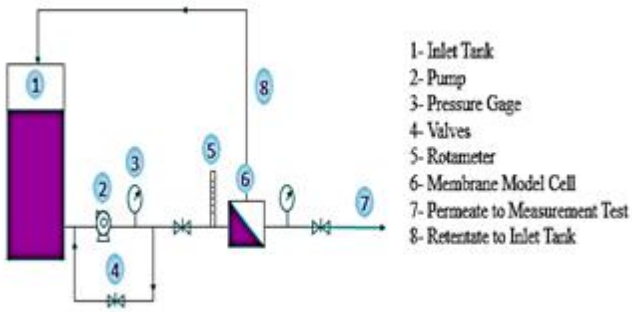

Figure 3: Schematic diagram of ultrafiltration experimental system.

3. Result and Discussion

3.1. Scanning Electron Microscopy (SEM)

The SEM measurement of the membrane reveals that Pc 0.1 and Pc 0.2 are composed of irregularly shaped pores within a size range of 0.7- 7 micrometer, which is larger than that found in the Pc membrane. This indicates that the substrate Pc0.1 and Pc0.2 membranes are structurally better than Pc, which has smaller pore sizes, resulting in high pollutant hindrance. Furthermore, after adding titanium dioxide nanoparticles, it was observed that the number of pores was reduced by increasing the proportion of nano titanium dioxide because the oxide nanoparticles block the pores, as shown in Figure (4 a-c).

S. H. Maruf *et al.* showed that a membrane with a lower roughness has a stronger antifouling ability [25]. Pc 0.2 membrane coating with nano-TiO_2 would therefore be expected to improve antifouling performance. The SEM results were in agreement with the results of previous research [26].

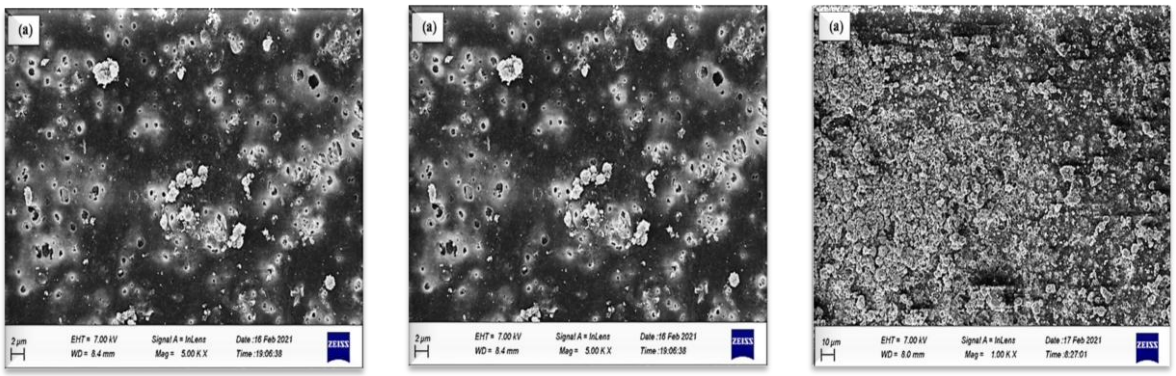

Figure 4: SEM images of a. Pc, b. Pc0.1, and c. Pc0.2.

3.2. X-Ray Diffraction (XRD) Analysis

The X-ray measurement of commercial polysulfone membranes showed the presence of several peaks, which it could not be determined for any of the commercial ingredients. Other peaks were due to other components that increase the membrane's strength. However, X-ray measurement showed the presence of the main peak of the carbon-polymeric structure at 16.68 degrees, which proves the presence of polysulfone within the composition, as shown in Figure 5a. The X-ray measurement of commercial membranes containing titanium oxide, in Figure 5 b-c, showed the presence of the polymer crest, but it was weaker than that of polymers containing titanium dioxide. In addition, X-ray measurement showed the characteristic peak of the titanium oxide, which appeared at approximately 25 degrees in both membranes. These results were in agreement with the results of previous research [27].

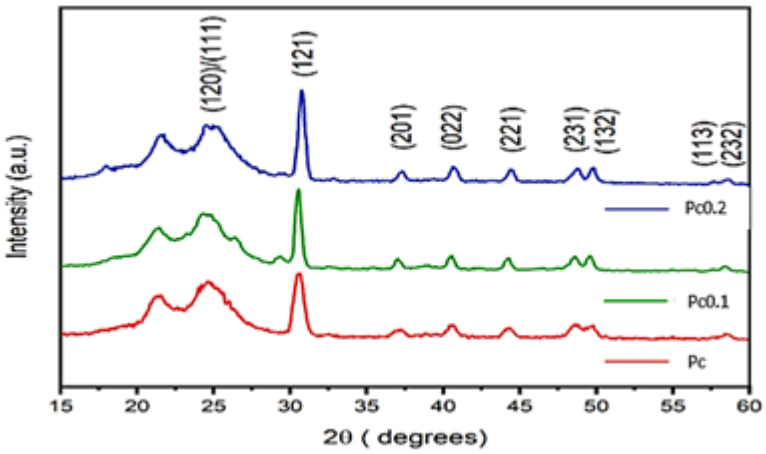

Figure 5: XRD patterns of Pc, Pco.1, and Pc 0.2.

3.3. Contact Angle Wettability Analysis

The addition of TiO_2 nanoparticles to spinning fluid increased the hydrophilicity of TFN substrate. The contact angle of support layer dropped from 65.4° for Pc to 33.7° for Pc 0.1 and 28.6 for Pc 0.2, indicating an increase in hydrophilicity. The surface wettability was shown to be impacted by surface energy and shape. The contact angle has been averaged five times to limit the influence of morphology. Table 4 shows that the total porosity of the substrate for all membranes. Low porosity resulted from uniform dispersion at low TiO_2 concentrations, and large porosity resulted from aggregation at high TiO_2 concentrations. The substrate thickness increased with the amount of TiO_2 nanoparticles.

In line with previous research [26], the average fiber diameter increased with the loading of TiO_2 nanoparticles. Thickness and porosity were closely connected to membrane performance. The best substrates for Pco.2 membranes are thin and porous, as shown in Figure 6.

Table 4: Effect of TiO_2 concentration on the properties of PSF substrates.

	Contact Angle	Porosity %
Pc	**65.4**	**51**
Pc0.1	**33.7**	**69**
Pc0.2	**28.6**	**55**

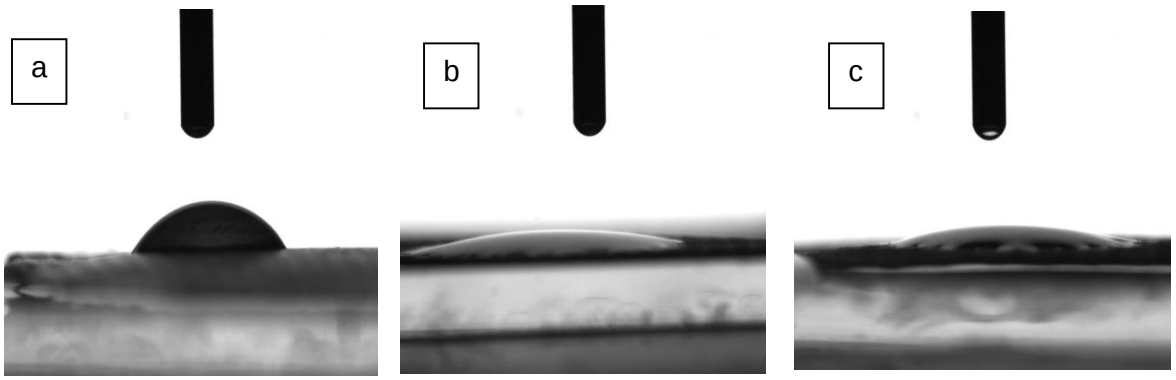

Figure 6: Contact angle measurements commercial membranes (a) Pc (b) Pc0.1, and (c) Pco.2.

3.4. Performance of the Membranes

3.4.1. Pure Water Flux (PWF)

Figure 7 presents the water flux of different commercial membranes evaluated by the cross-flow UF process. The experiments were conducted using distilled water at a flow rate of (1.32 l/min) and pressure of (2 bar and 2 h) for the total operation. The results showed that the Pc0.1 and Pc0.2 membranes substrates from TiO_2/PSF nanocomposite substrates had much higher water flux than the Pc membrane without TiO_2. When adding 0.1% TiO_2, the water fluxes of the membrane improved 19% , but adding too much TiO_2 water flux was only slightly higher. The last results show that the structural improvements properties of TiO_2/PSF substrates could minimize the transport resistance against water permeation. This expectation stems from the hydrophilic nature of TiO_2NPs, which will cause an increase in the hydrophilicity of membranes surface [28] and change the membrane surface morphology and internal structure. Average pure water fluxes (PWF) for Pc, Pc0.1, and Pc0.2 are 20, 35.28, and 44 l/m^2.h, respectively.

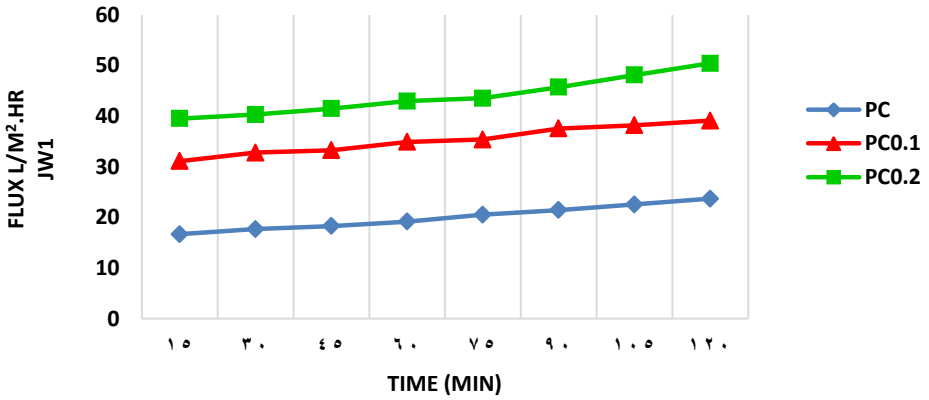

Figure 7: Pure Water Flux of PSF membranes without and with different TiO_2 concentrations.

4. Conclusion

This study concluded that incorporating TiO_2/PSF homogeneous spinning dope could be spray paralysis successfully. The substrates of UF membranes were fabricated with different average fiber diameters. It was found that TiO_2 nanoparticles impacted the morphology and structure of the substrates. Furthermore, the mechanical strength of TiO_2/PSF substrate decreased as the TiO_2 loading increased. Yet, incorporating TiO_2 in fibers could enhance the wettability of the TFN membranes. Also, results showed that the water fluxes of the TFN membrane prepared from the nanocomposite substrate increased with increasing TiO_2 concentrations. Thus, there is a high potential of the present PSF/TiO_2 mixed-matrix fiber mats as a substrate material to improve the water flux of the UF membrane.

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