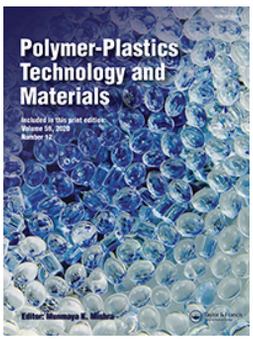

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Synthesis, characterization, and gas separation performances of polysulfone and cellulose acetate-based mixed matrix membranes

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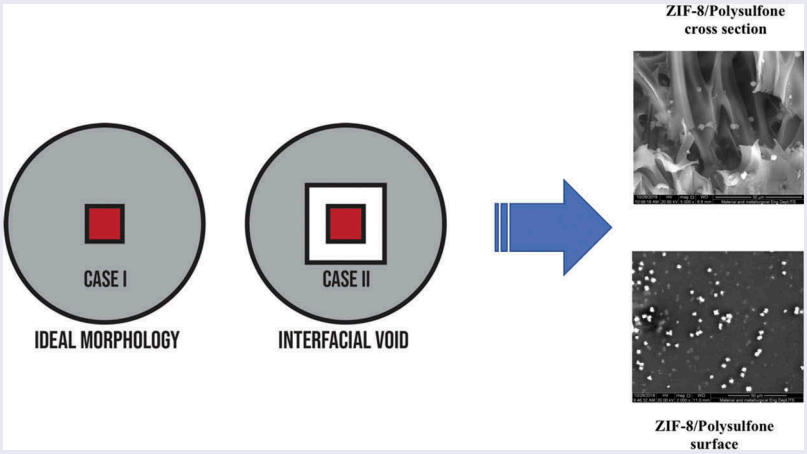
ABSTRACT

The combination of polymeric and inorganic fillers inside mixed matrix membranes (MMMs) becomes a hot research topic due to the gas permeability-selectivity trade-off in polymeric membranes. Until recently, the problem of voids hampers the real application of MMMs, hence deep understanding on polymer-particle compatibility is required. This study focuses on the synthesis and characterization of polysulfone and cellulose acetate-based MMMs that combined with ZIF-8 and TiO_2 particles. ZIF-8 dispersed more uniform than TiO_2. The crystallinity of MMMs was higher than pure polymeric membrane. In addition, micro voids in MMMs resulted a slight decrease in CO_2/N_2 selectivity (from 15 to 12).



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Polysulfone; cellulose acetate; ZIF-8; TiO_2; membrane

1. Introduction

Gas separation issue grows as an important topic in our daily life as the occurrence of several gases, such as carbon dioxide (CO_2), is considered as the main cause of greenhouse effect that increasing the possibility of climate change phenomenon. It is predicted that the concentration of CO_2 as the greenhouse gas in air will increase rapidly due to the rapid increase in economic and industrial development.[1] Energy production from fossil fuel burning produces large amount of CO_2. Recently, more than 85% of the energy worldwide is supplied by the combustion of fossil fuels.[2] Many countries and nations have tried to find and to optimize their natural resources to be used as alternative energies that can produce less emissions of CO_2 and friendlier to environment. However, based on evidence that most countries and nations still depend on fossil fuels as their main source of energies, it is very important to find and explore technologies to reduce and capture CO_2.

The main problem for gas separation materials and techniques is the little difference of properties among gases that need to be separated. For example, the kinetic diameters of CO_2 and N_2 are 3.30 and 3.64 Angstrom, respectively.[3] Therefore, it is urgent to find new material or to manipulate the molecular structure of material in order to separate gas mixture. Several researchers have developed different kinds of material that have been used in various unit operations like absorption, adsorption, and membrane separation. Among separation methods available today, membrane gains greater interest to be explored than other technologies as it offers smaller footprints as well as easier

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to be brought to larger scale than other technologies. Nevertheless, membranes available today should compromise their gas permeation and selectivity that can impede their wide application in industries.[4,5]

The trend of research on gas separation membrane is therefore directed on the development of membrane that can overcome the trade-off relationship between gas permeation and gas selectivity of membrane. The incorporation of inorganic particles into polymer matrix or mixed matrix membranes (MMMs) has been explored and investigated widely to be proposed as alternative approach to improve gas permeation and gas selectivity of the membrane at the same time.[5] However, there are several problems arise from the utilization of MMMs such as the formation of voids and defects as a result of incompatibility between inorganic particles and polymer materials as well as the increase of resistance because of the utilization of micrometer size of particles.[5,6] There are several techniques that can be applied to reduce the formation of voids and defects. Such techniques include the priming protocol and the functionalization of particles. In the priming protocol, the particles are firstly mixed with a small amount of polymer solution before dispersed thoroughly in the remaining polymer solution. On the other hand, the incompatibility issue between polymer and particles can be altered by adding functional groups by functionalization process, which can improve the dispersibility of the particles inside polymer matrix.[6] In addition, the importance of long-term operation process and membrane aging as well as plasticization of polymeric membrane in MMMs cannot be neglected as these phenomena determine the wider application of membrane in real gas separation processes.

Up to now, research on MMMs for gas separation process concentrates only on the synthesis on the membrane from one material of either polymer or inorganic particles. Further studies are needed to compare the synthesis of MMMs from different materials to investigate the effects of materials on the membrane characterization. This research studied the synthesis and characterization of MMMs from two different polymer matrices and two different particles. Both polysulfone and cellulose acetate were investigated as matrices for MMMs, and they were combined with commercial titanium dioxide (TiO_2) and inhouse prepared of Zeolitic Imidazolate Framework-8 or ZIF-8 particles. Each particle has different characteristic. TiO_2 particle is hydrophilic particle, whereas ZIF-8 is hydrophobic. ZIF-8, as one type of Metal Organic Frameworks (MOFs) material, theoretically will improve the dispersion of this particle inside polymer matrix as it has a combination of inorganic and organic materials. The difference in the characteristics between TiO_2 and ZIF-8 will determine the dispersion of particles inside polymer matrix, hence will determine the characterization results for each MMMs.

2. Materials and methods

2.1. Materials

Polysulfone polymer was purchased from Solvay Advanced Polymers (Alpharetta, GA) under the trade name of 'Udel Polysulfone *P*-3500 LCD'. Chemicals for cellulose acetate membrane preparation, such as: cellulose acetate (CA, CAS No. 9004-35-7), formamide (CAS No. 75-12-7), acetone (CAS No. 67-64-1), were supplied by Sigma Aldrich. The solvent for polysulfone membrane synthesis were dimethylacetamide (DMAc, CAS No. 127-19-5)), tetrahydrofuran (THF, CAS No. 109-99-9), and ethanol (CAS No. 64-17-5), which were purchased from Sigma Aldrich. TiO_2 (CAS No. 13463-67-7) particles was purchased from Sigma Aldrich. The chemicals for ZIF-8 particle synthesis were 2-methylimidazole (CAS No. 693-98-1), methanol (CAS No. 67-56-1), and zinc nitrate hexahydrate (CAS No. 10196-18-6) were kindly supplied by Sigma Aldrich. Pure CO_2 and N_2 gases were kindly supplied by PT Aneka Gas, Indonesia. All chemicals were used without prior purification.

2.2. ZIF-8 synthesis

ZIF-8 was synthesized using rapid room-temperature method.[7] In this method, ZIF-8 crystals were formed at ambient temperature using simple mixing of zinc nitrate hexahydrate and 2-methylimidazole chemicals in methanol as solvent. Reaction was held at room temperature for 1 h and crystals were separated from solution by centrifugation and washing with fresh methanol for two times.

2.3. Membrane synthesis and charaterizations

The synthesis of pure and MMMs was conducted by solution blending and phase inversion techniques. Cellulose acetate-based membranes were synthesized by mixing 20 wt.% of cellulose acetate with 33 wt.% formamide and 47 wt.% of acetone until all polymers were dissolved. For the purpose of MMMs synthesis, the particles of ZIF-8 or TiO_2 were dispersed under rigorous mixing with the polymer solution. Then, the membranes were casted on a glass plate followed by soaking inside water at room temperature. The annealing process was then conducted and produced membranes were characterized with Scanning Electron Microscope (SEM), X-Ray Diffraction (XRD), and Fourier Transform Infra-Red

(FTIR). SEM analysis was conducted for surface and cross section of the membranes. The cross section of the membranes was attained by cutting the membranes in liquid nitrogen. The membranes were then coated with Pd/Au coating before SEM and Energy Dispersive X-Ray (EDX) analysis. SEM was conducted by using Evo MA 10 Carl Zeiss SEM equipment that equipped with EDX analyzer. The crystallinity of the membranes was analyzed by using Empyrian XRD analyzer. FTIR for chemical analysis was conducted under Alpha FTIR for the surface of the membranes.

Polysulfone-based membranes were produced following procedure described elsewhere.[6] In the procedure, 22 wt.% of polysulfone were mixed with DMAc, THF, and 14.4 wt.% of ethanol. The solution was then mixed in a beaker glass using a magnetic stirrer at 60°C for 6 h to form homogeneous casting solution. For the purpose of MMMs synthesis, the particles with certain weight were added into the polymer solution. Then, the casting solutions were cast on a glass plate with a casting knife at room temperature. The casted membranes were then soaked in water for 10 min and methanol for 2 h followed by drying.

2.4. Gas permeation tests

Single gas permeation tests using nitrogen (N_2) and CO_2 gases were conducted to examine the gas separation performance of the membranes. Circular membrane discs with an effective permeation area of 13.5 cm^2 were used. Experiments were conducted at room temperature. Feed pressure was set at 1 bar gauge while permeate side was set at atmospheric pressure. The CO_2 and N_2 permeabilities were determined by recording the gas flux through the membranes and permeance were calculated following Eq. (1).

$$(P)_i = J_i/\Delta p_i x l \tag{1}$$

where $(P)_i$ is the gas permeability (Barrier), Ji is the flux of gas i, Δp is the pressure difference across membrane and l is the thickness of the membrane. Gas selectivity of the membranes (α) was calculated following Eq. (2).

$$\alpha_{\frac{CO_2}{N_2}} = \frac{P_{CO_2}}{P_{N_2}} \tag{2}$$

3. Results and discussion

The pure polymeric membranes and MMMs from this study were each assigned as polysulfone-based membrane and CA-based membrane and the discussion of each membrane is presented in the following section.

3.1. The morphology of membranes

The morphology of membrane was analyzed by scanning the surface and cross section of both pristine polysulfone membrane and MMMs using SEM. The SEM pictures are depicted in Figure 1.

The pure polysulfone membrane showed a relatively smoother surface compared to the surface of MMMs. The surface of MMMs contained the agglomerates of particles indicating the degree of dispersion of each type of particles inside the polymer matrix. ZIF-8 particles were produced from inorganic and organic chemicals, hence ZIF-8 has metal center connected by organic linkers. This unique property creates a unique result when ZIF-8 dispersed inside the polysulfone matrix. Thus, it is predicted that ZIF-8 will form smaller amounts of agglomerates compared to other pure inorganic particles, such as TiO_2. From the comparison of Figure 1c–e, TiO_2 particles formed large agglomerates inside polysulfone matrix. On the other hand, ZIF-8 particles in Figure 1e showed a relatively good dispersion inside the polymer matrix.

To confirm the agglomeration of TiO_2 particles, the cross section of TiO_2/polysulfone MMM in Figure 1c was then analyzed for its Ti element using EDX analysis. The EDX analysis is then presented in Figure 2. The agglomeration of TiO_2 particles can be detected by analyzing the distribution of Ti element. Figure 2 clearly shows the agglomeration of Ti element (yellow dot) inside the polymer matrix indicating the non-uniform distribution of particles. The agglomeration might occur due to the incompatibility of the particle and polymer matrix. Compared to ZIF-8, TiO_2 particles do not posses the organic linker that can improve the dispersion of particles inside the polymer matrix.

To confirm further the particle dispersion inside the polymer matrix, the cellulose acetate (CA)-based membranes were also scanned under SEM. The SEM results of the surface and cross section of CA-based membranes are shown in Figure 3.

Similar to pure polysulfone membrane, the pure CA membrane in Figure 3a also showed a smooth surface compared to its MMMs counterparts. The agglomeration of particles inside CA-based MMMs still occurred. (Figure 3b–e) However, similar to polysulfone-based MMMs, the ZIF-8 particles were agglomerated less than TiO_2 particles. The inorganic nature of TiO_2 particles hinder their possibility to interact with polymer matrix. Good interaction between particles and polymer will improve the degree of dispersion of particles inside polymer matrix. Thus, it will improve the possibility of membrane to produce good gas separation performances.

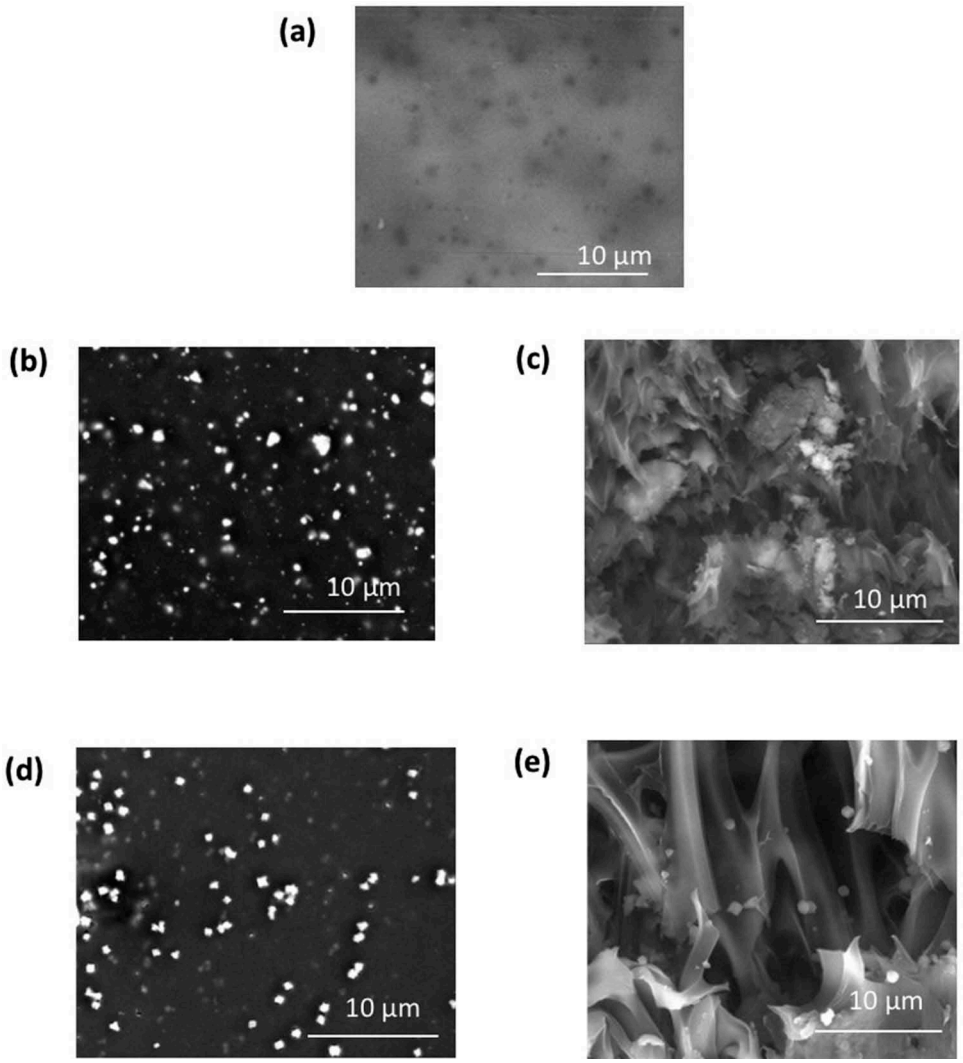

Figure 1. The SEM pictures of: (a) the surface of pure polysulfone membrane; (b) the surface of 5 wt% TiO_2/Polysulfone MMM; (c) the cross section of 5 wt% TiO_2/Polysulfone MMM; (d) the surface of 5 wt% ZIF-8/Polysulfone MMM; and (e) the cross section of 5 wt% ZIF-8/Polysulfone MMM.

3.2. Chemical analysis of pure polymeric membranes and MMMs

To confirm the interaction between the particles and polymer matrix, FTIR analysis were conducted by comparing the FTIR spectra of pure polysulfone membrane and MMMs. FTIR will be able to inform not only the functional groups inside the membranes, but also can predict the nature of chemical bond between the particles and polymer. The formation of hydrogen bond between ZIF-8 particles and polymer inside MMMs was reported in a previous study,[8] and the bond improves the mechanical strength and plasticization resistance of the MMMs. Hence, in this study, the results of chemical analysis using FTIR of polysulfone-based membranes as representative polymer in this study are presented in Figure 4a.

The FTIR spectrum of pure polysulfone membrane shows C = C bonds of aromatic at wave number of 1637.28–1583.05 cm^{-1}. The C-O bond of ether was detected at wave number of 1234.70 cm^{-1}, while C-H bonds at aromatic and aliphatic rings were found at 3065.85 and 2967.08 cm^{-1}. In addition, the asymmetric and symmetric bonds of S = O were detected at 1363.68 and 1168.17 cm^{-1}, respectively.

To confirm the formation of chemical bond between the particles and polysulfone matrix, analysis on FTIR spectra of MMMs in Figure 4b–c were then conducted. Figure 4b shows the C = C bond inside TiO_2/polysulfone MMM as detected at around 1637.49–1583.30 cm-1. The C-H bond on aromatic and aliphatic rings was formed at 3067.25 cm-1 and 2967.31 cm-1, respectively. In addition, the C-O, symmetric and asymmetric S = O bonds were detected at 1235.27, 1168.25, and 1363.76 cm^{-1},

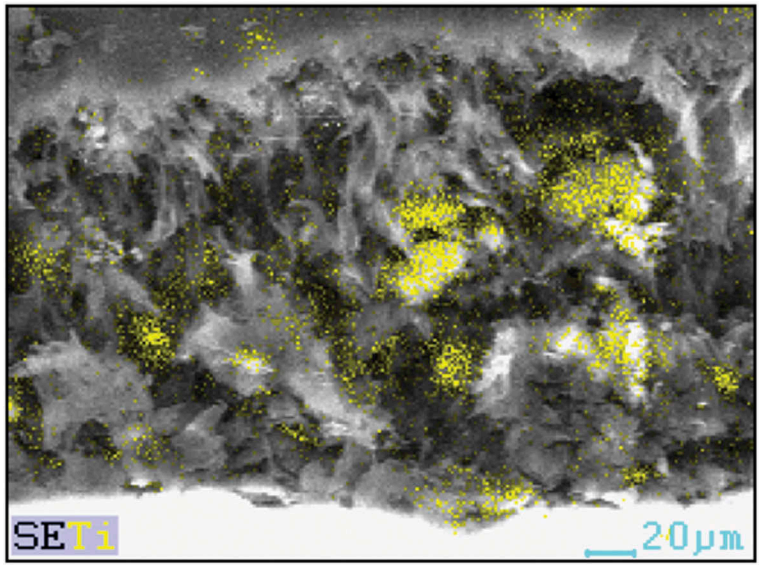

Figure 2. The EDX mapping of the cross section of TiO_2/Polysulfone membrane.

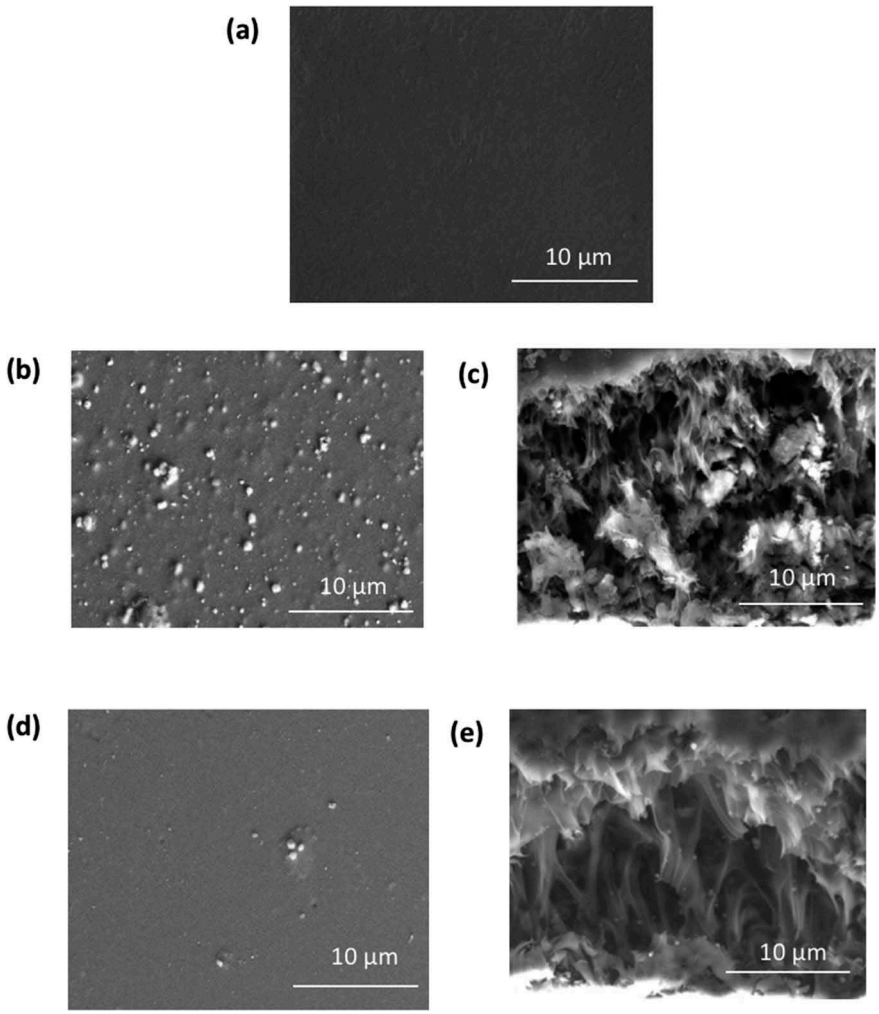

Figure 3. The SEM pictures of: (a) the surface of pure cellulose acetate (CA) membrane; (b) the surface of 5 wt% TiO_2/CA MMM; (c) the cross section of 5 wt% TiO_2/CA MMM; (d) the surface of 5 wt% ZIF-8/CA MMM; and (e) the cross section of 5 wt% ZIF-8/CA MMM.

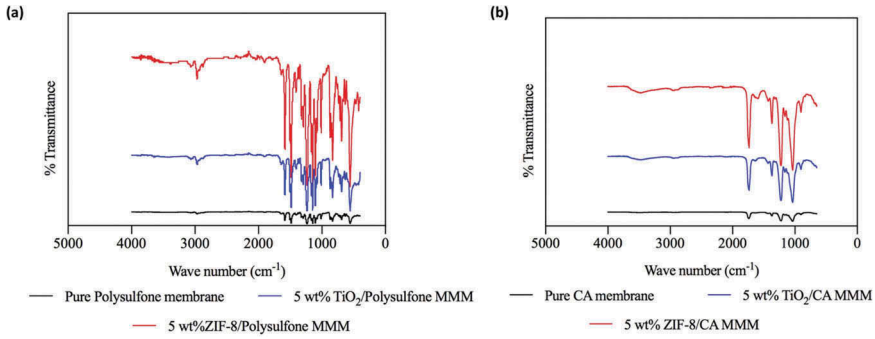

Figure 4. FTIR spectra of (a) Polysulfone (Psf)-based membranes and (b) Cellulose Acetate (CA)-based membranes.

respectively. The incorporation of TiO_2 particles did not affect or form new peaks indicating the nature of particles only as fillers inside the membranes. In addition, the agglomeration of TiO_2 particles inside polysulfone matrix solely occurred due to the incompatibility between the particles and polymer.

ZIF-8-based MMM showed a similar trend with TiO_2-based MMM as there were no additional peaks detected from the FTIR spectrum of ZIF-8-based MMM shown in Figure 4c. This is an indication that ZIF-8 particles was functioned as filler and did not chemically interact with polysulfone. Less agglomeration of ZIF-8 inside the polysulfone matrix compared to TiO_2 indicated the advantages of using ZIF-8 inside MMMs because the 'organic' nature of ZIF-8 particles improves the dispersion of ZIF-8 in the polymer matrix. In terms of cellulose acetate-based membranes, FTIR spectra presented in Figure 4b showed similar trends with polysulfone-based membranes. The indication of the formation of chemical bond between polymer and particles cannot be detected under FTIR.

3.3. The degree of crystallinity analysis of membranes

The incorporation of particles inside MMMs will affect the crystallinity of the polymer matrix. The crystallinity of the membranes in this study was analyzed by XRD and the degree of crystallinity of each membrane was calculated using Eq. (3).

$$DOC = \frac{A}{A'} x100\% \tag{3}$$

where DOC is the degree of crystallinity, A is the area of all peaks, and A' is the total area of the XRD pattern. The XRD pattern can provide information about the nature of materials under analysis including the position of the peak/crystalline of the material and the possible amorphous nature of the materials.

The XRD patterns of polysulfone and cellulose acetate-based membranes are depicted in Figure 5. The polysulfone membrane presented peaks at 17.41°, 17.52°, 17.7°, and 17.73°, which are in accordance with previous study.[9] In addition, the XRD pattern of polysulfone also showed an amorphous nature of the polymer, which was indicated by the spread peaks at around 20° and 45°. These amorphous regions will provide flexibility to the polymer when it interacts with inorganic fillers in MMMs. The XRD pattern of pure CA membrane showed signature peaks at 9.2°, 10.8°, 13.5°, 17.9°, and 23.7° with amorphous regions at around 10° and 18°. The incorporation of inorganic particles into polymer matrix might present additional peaks and increase or decrease the peaks intensity of the polymer.

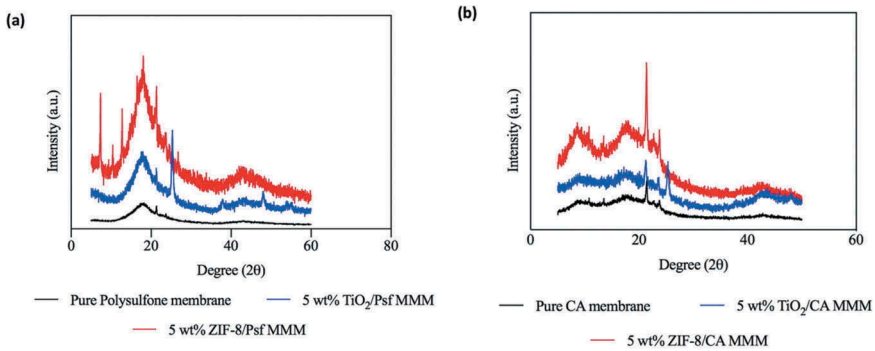

Figure 5. XRD patterns of (a) Polysulfone (Psf)-based membranes and (b) Cellulose Acetate (CA)-based membranes.

The XRD patterns of TiO_2/polysulfone and ZIF-8/polysulfone membranes with 5 wt% particle loading are presented in Figure 5a. Based on the patterns in Figure 5a, after the incorporation of TiO_2 particles, the signature peaks of TiO_2 were detected at 2θ of 25°, 37.82°, 47.97°, 54.25°, and 55.9° which were in accordance to previous study.[10] Similar trend was also occurred for ZIF-8-based membranes. The signature peaks of ZIF-8 were detected at 7,34°, 10,40°, 12,72°, 16,47°, 18,05°, 21,3°, 23,72°, 24,49°.[8] The signature peaks of these particles clearly increased the degree of crystallinity of MMMs. As the DOC of each membrane was analyzed and calculated using Eq. (1) using Origin software, it is very interesting to know the impact of the presence of particles to the degree of crystallinity of the membranes. The degree of crystallinities of pure polymeric and MMMs are presented in Table 1.

In general, the incorporation of ZIF-8 and TiO_2 particles improved the degree of crystallinity of the membranes. The type of particle slightly influences the improvement of the degree of crystallinity of the membranes. The addition of TiO_2 provided higher improvement of crystallinity than ZIF-8 particles. The agglomeration of TiO_2 inside MMMs contributed to the improvement of the degree of crystallinity of the MMMs as the agglomeration might affect the XRD patterns of the MMMs. However, this should be confirmed by further analysis. In addition, the increase in particle concentration from 5 to 10 wt% enhanced the degree of crystallinity of the MMMs. This will affect the mechanical strength of the membranes as higher concentration of particles made the membranes more fragile.

As ZIF-8-based MMMs provided better dispersion of particles inside polymer matrix, further experiments on gas separation performances using pure gas of CO_2 and N_2 were conducted using ZIF-8-based membranes.

3.4. Pure gas separation performances of ZIF-8-based MMMs

To know the gas separation performances of the membranes, pure gas of CO_2, N_2, and CH_4 were employed in a gas permeation set up. The gas separation performances of ZIF-8-based membranes are presented in Tables 2 and 3.

The presence of ZIF-8 particles clearly increases the gas permeability through the membranes. ZIF-8 has pore diameter around 3.4 Angstrom that is suitable to separate CO_2 from N_2. The kinetic diameters of CO_2 and N_2 are 3.4 and 3.6 Angstrom, respectively. In addition, ZIF-8 has relatively large surface area that improves its adsorption capacity to certain gas. However, the linker flexibility of ZIF-8 particles is also reported and it decreases its capability to discriminate gas mixture, especially gases that have a relatively close kinetic diameter, such as CO_2 and N_2. This was indicated by the decrease of CO_2/N_2 selectivity of ZIF-8-based MMMs in this study as can be seen in Tables 2 and 3. In addition, the decrease of gas selectivity of the membranes was also attributed by the formation of micro voids during membrane synthesis. The micro voids inside the MMMs were mainly caused by the incompatibility between the particles and polymer matrix. Even though ZIF-8 has organic linker, the aggregation of ZIF-8 particles was still possible in this study thus decrease the gas selectivity through the membranes.

Table 1. The degree of crystallinities of various membranes.

Type of membrane	Degree of Crystallinity (%)	
	Cellulose Acetate	Polysulfone
Pure Membrane	13.54 ± 0.001	28.70 ± 0.005
5 wt% of ZIF-8 MMMs	19.55 ± 0.001	32.49 ± 0.003
10 wt% of ZIF-8 MMMs	20.6 ± 0.005	35.67 ± 0.004
5 wt% of TiO_2 MMMs	21.21 ± 0.005	33.24 ± 0.005
10 wt% of TiO_2 MMMs	22.53 ± 0.002	38.38 ± 0.003

Table 2. Pure gas separation performances of various ZIF-8/polysulfone-based membranes.

ZIF-8 loading (wt%)	CO_2 (Barrer)	N_2 (Barrer)	CO_2/N_2
0	17.1	1.1	15.6
5	18.5	1.5	12.3
10	20.3	1.6	12.69

Table 3. Pure gas separation performances of various ZIF-8/cellulose acetate-based membranes.

ZIF-8 loading (wt%)	CO_2 (Barrer)	N_2 (Barrer)	CO_2/N_2
0	10.2	1.05	9.71
5	12.3	1.34	9.18
10	15.4	1.73	8.90

4. Conclusions

The synthesis of MMMs using different types of polymers and inorganic particles was reported in this study. Both polysulfone and cellulose acetate-based membranes were successfully synthesized by mixing the polymers with TiO_2 and ZIF-8 particles. ZIF-8 with its organic linkers showed more uniform distribution inside the polymer matrix compared to TiO_2 particles. The aggregation of particles was detected for both particles with less aggregation detected for ZIF-8 particles. FTIR analysis confirmed no specific chemical bonds formed between both particles and polymers. Furthermore, the degree of crystallinity of the polymer matrix was improved after the incorporation of TiO_2 and ZIF-8 particles. CO_2/N_2 gas separation of ZIF-8-based membranes showed an increase in gas permeability and a slight decrease in gas selectivity. This confirmed the gate flexibility of ZIF-8 particles

and also the formation of micro voids inside the membranes.

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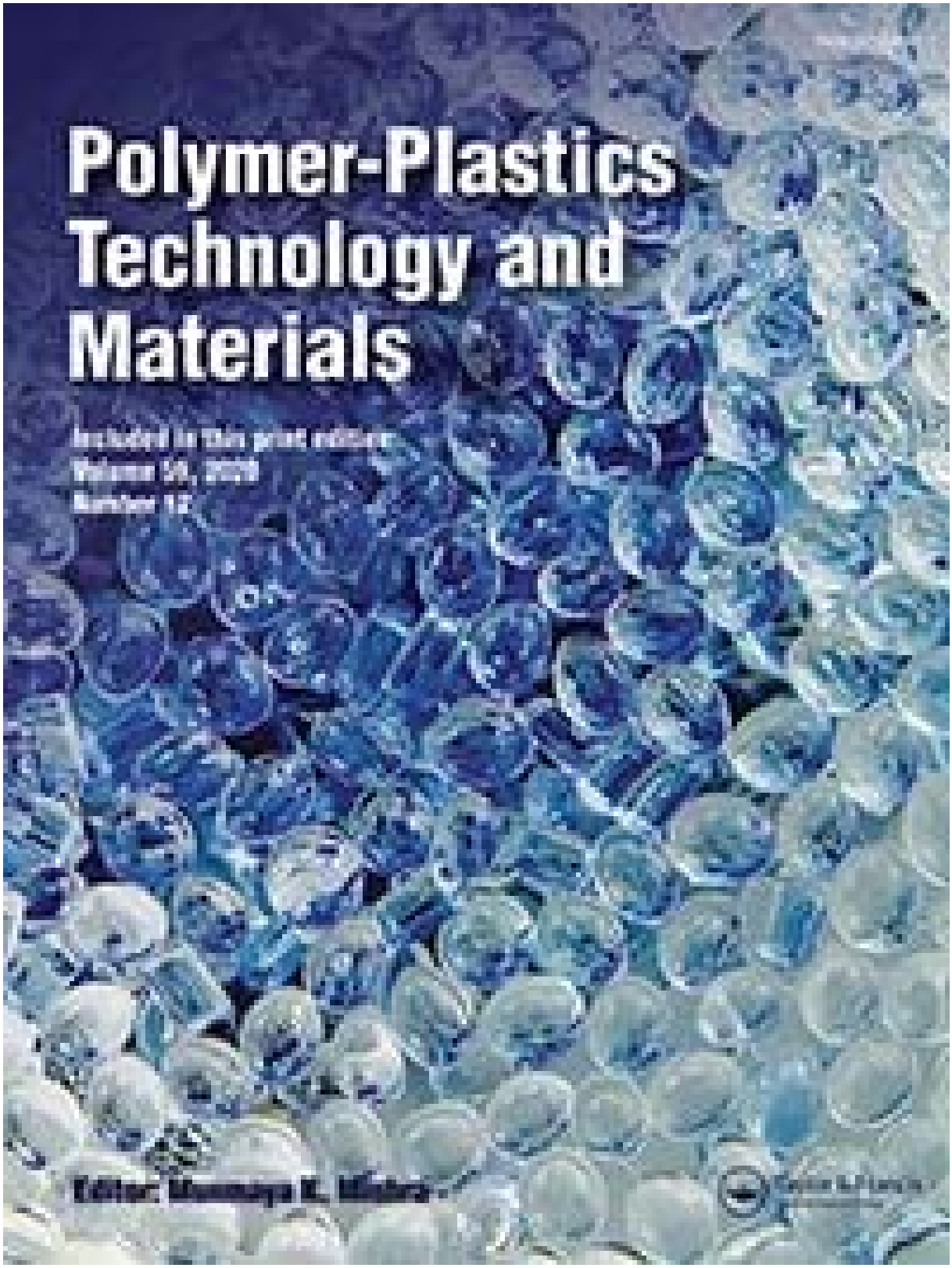
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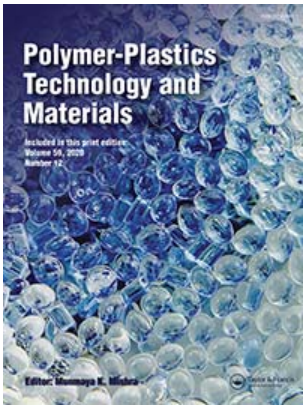

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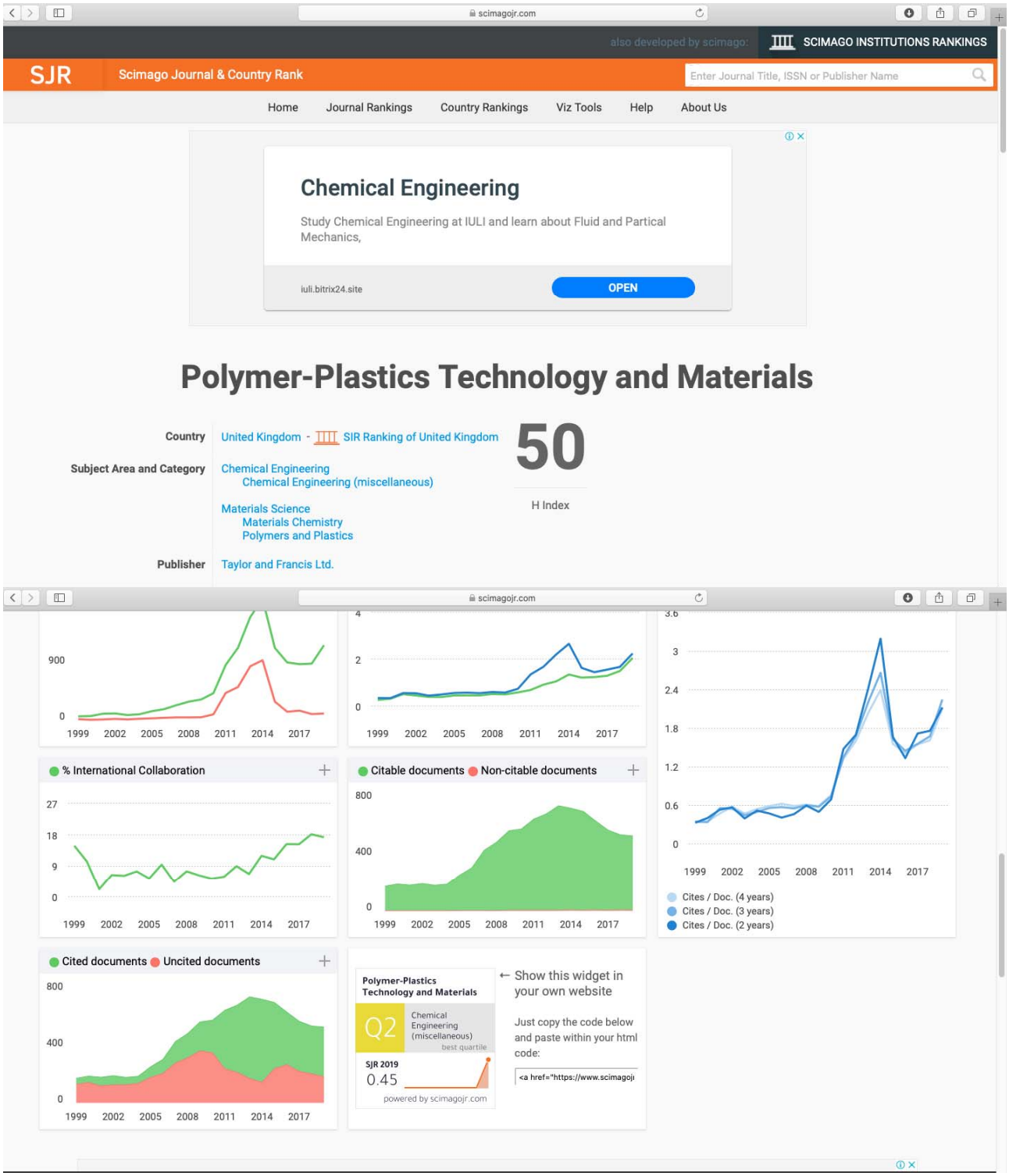

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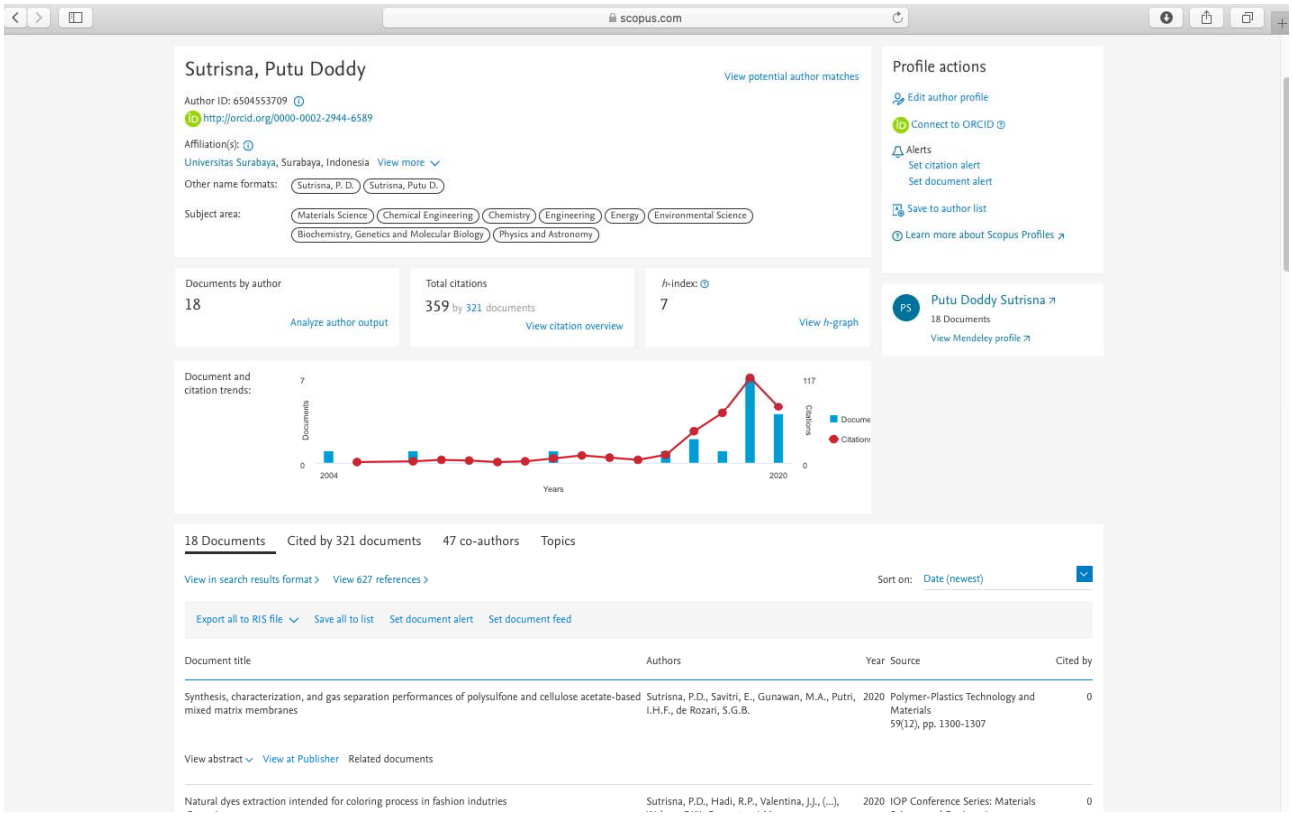

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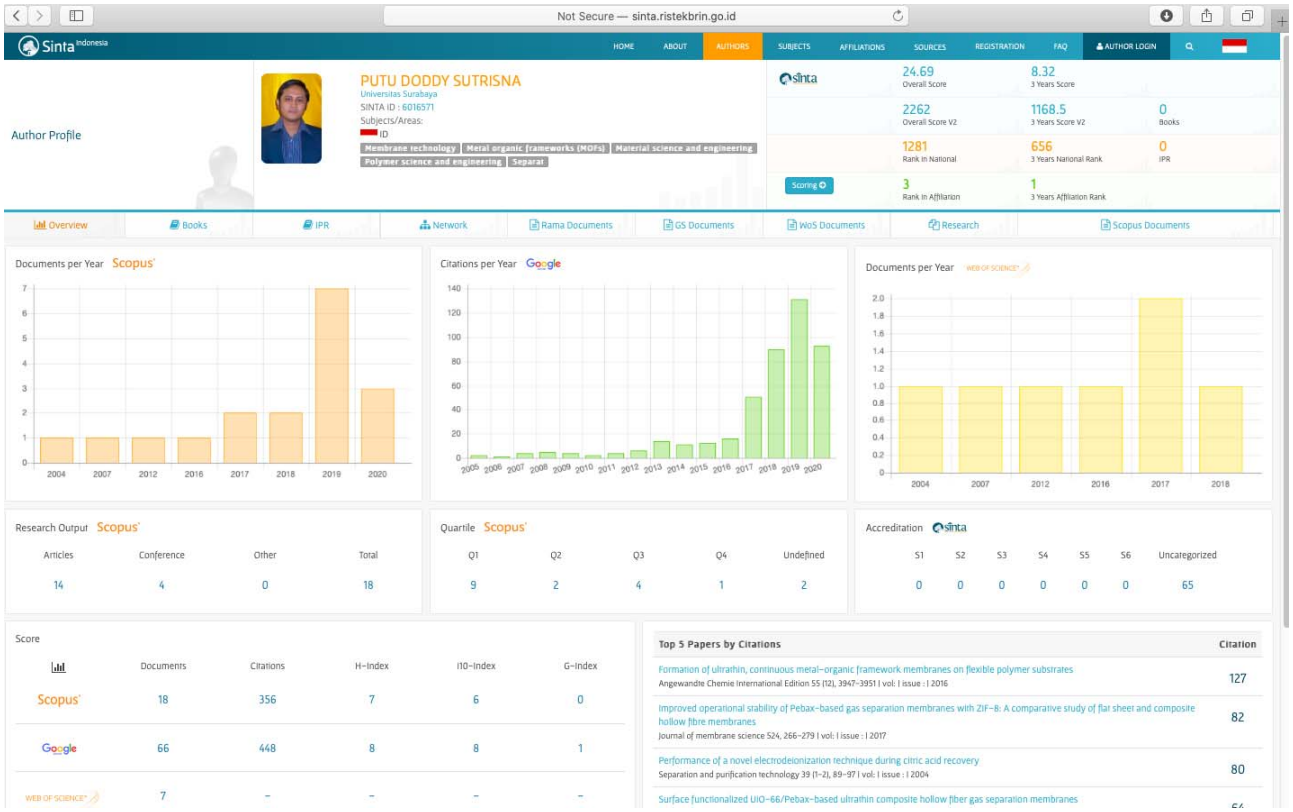

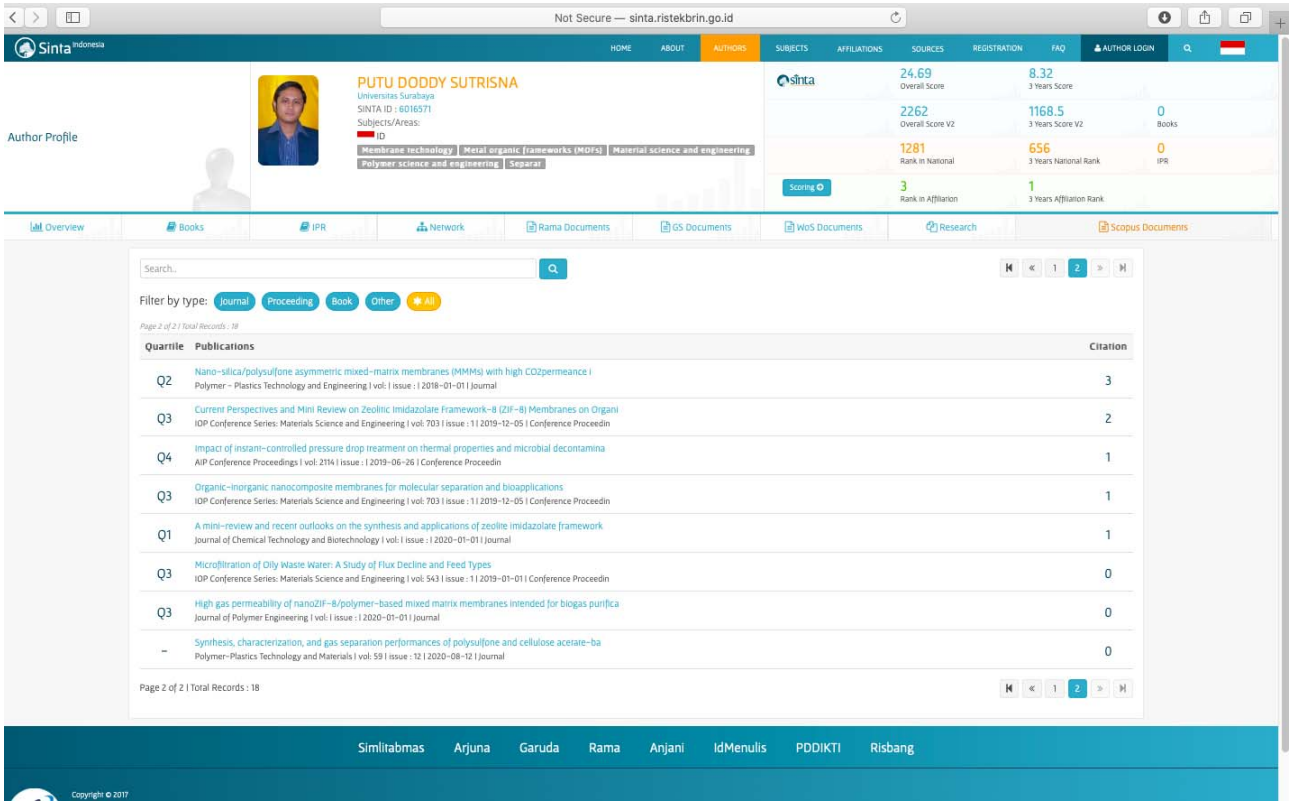

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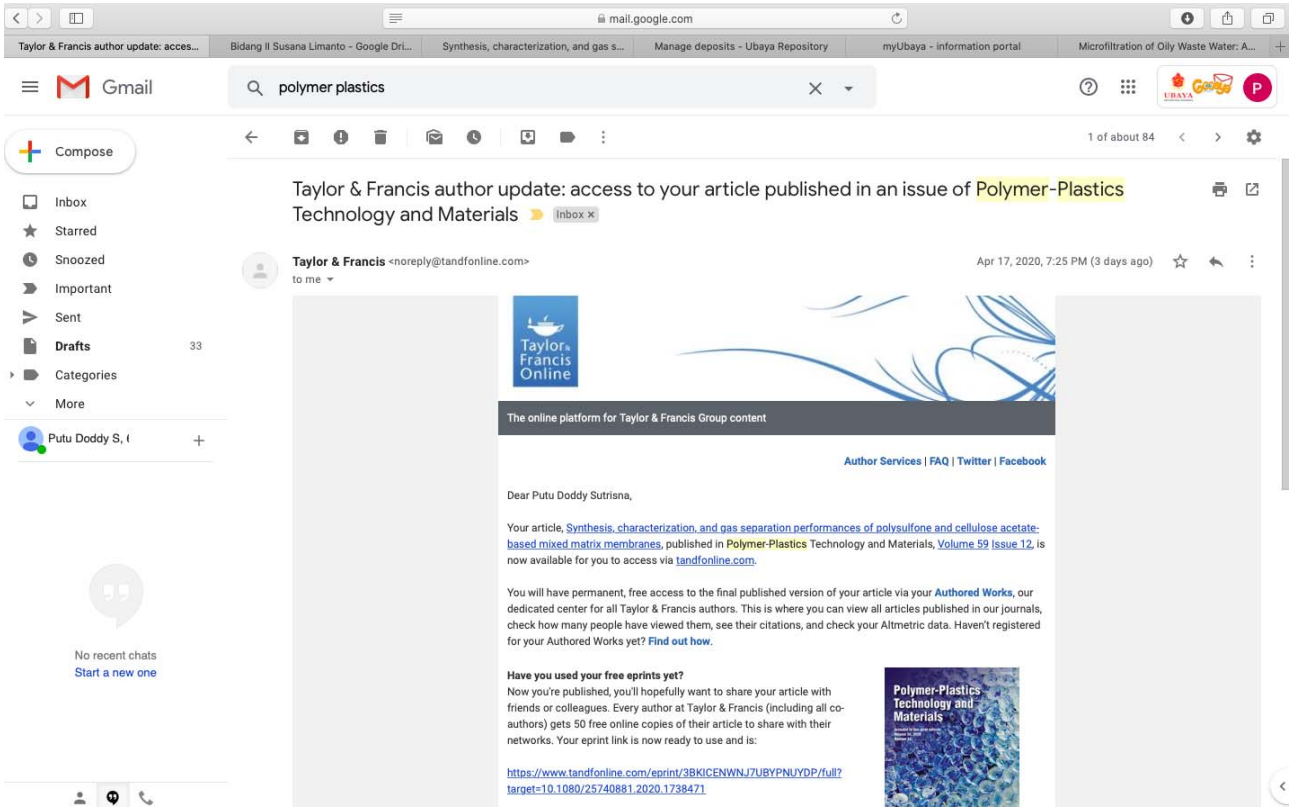

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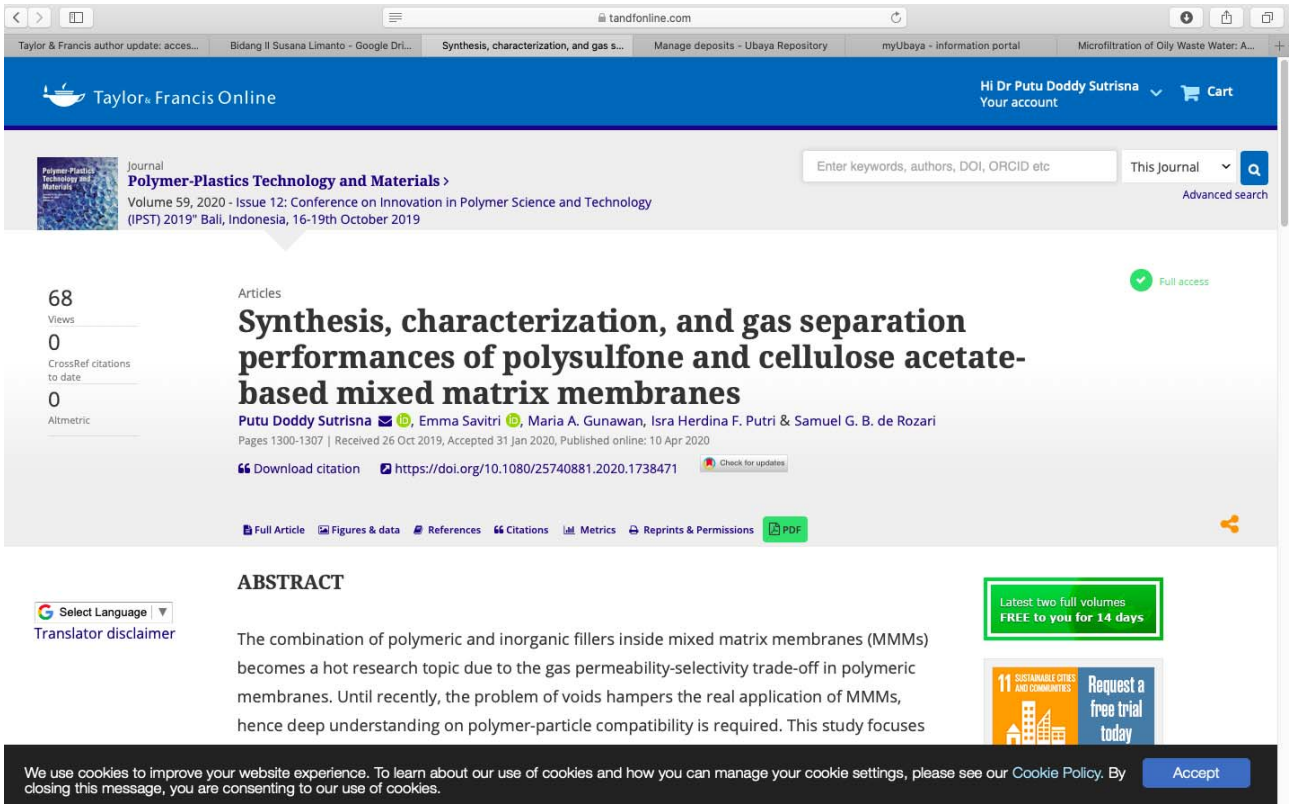

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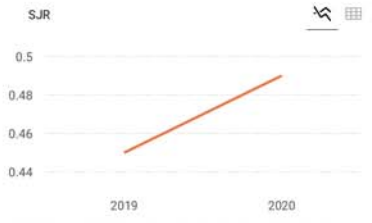

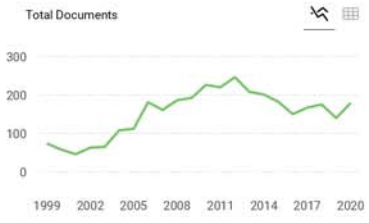

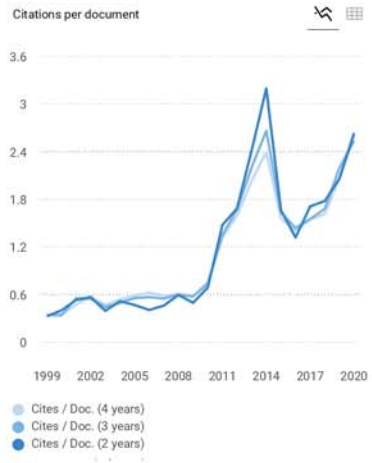

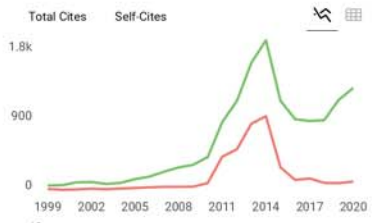

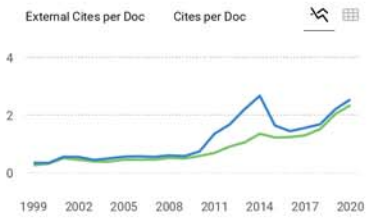

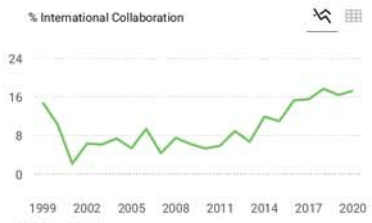

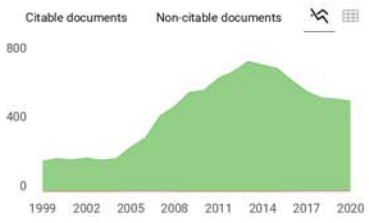

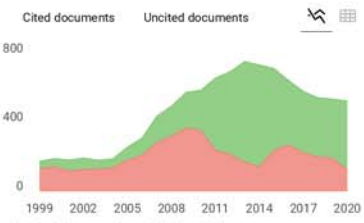

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