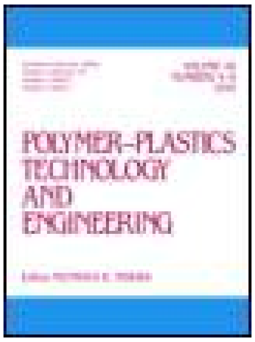

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Nano-silica/polysulfone asymmetric mixed-matrix membranes (MMMs) with high CO_2 permeance in the application of CO2/N2 separation

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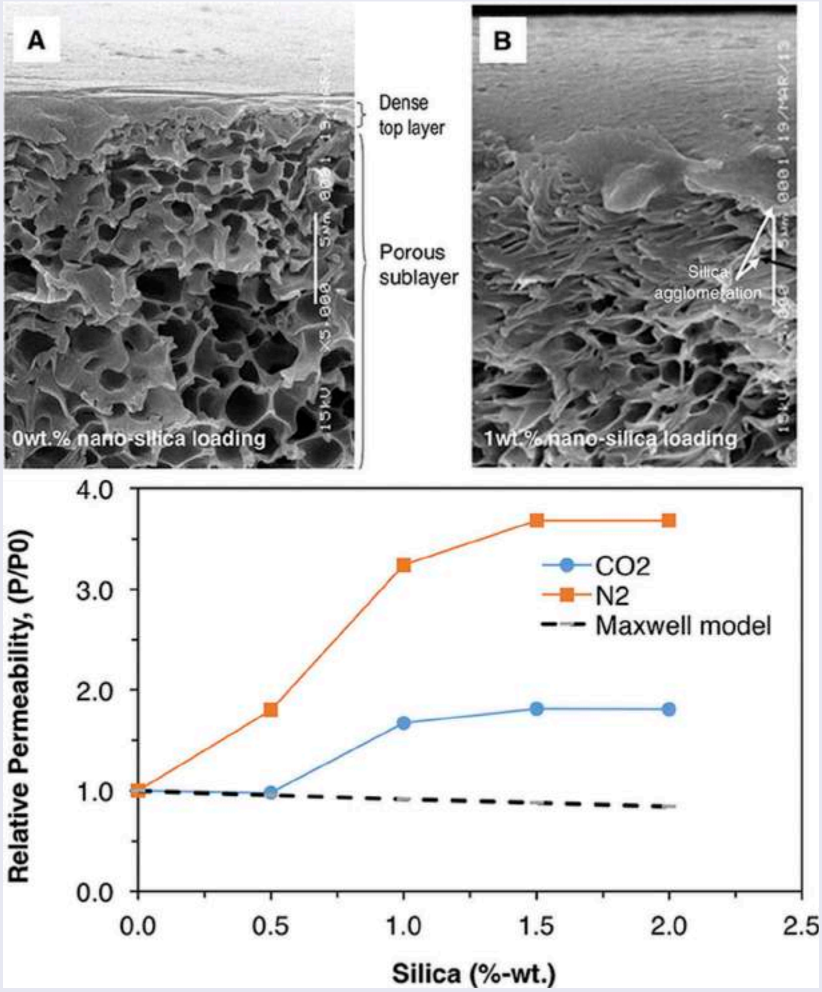
ABSTRACT

Nano-sized silica/polysulfone (PSf) flat sheet asymmetric MMMs with high CO_2 permeance for CO_2/N_2 separation were fabricated by dry/wet phase inversion method using N, N-dimethylacetamide (DMAc) and tetrahydrofuran (THF) as solvents and ethanol as additives. The results indicated that the addition of nano-silica on the polymer matrix resulted on reduced membrane performance due to void formation and particle agglomeration. Optimum membrane performance was obtained at the following fabrication parameters: 22 wt.% PSf, 31.8 wt.% DMAc, 31.8 wt.% THF, 14.4 wt.% ethanol, 20 s evaporation time, and 0 wt.% silica loading, with CO_2/N_2 selectivity of 15.6 and CO_2 permeance of 14.2 GPU.



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Introduction

Due to the increasing concern of climate change, reduction of green house gas emission, which largely consists of CO_2, has gained considerable attention for years. Research on the advancement of technology for selective CO_2 capture and separation has been extensive.[1–3] Conventional separation processes for CO_2 are adsorption with monoethanolamine (MEA) and cryogenic removal. Adsorption of CO_2 with MEA is a robust process with more than 90% CO_2 being captured. However, the drawbacks include significant energy requirement for MEA regeneration, degradation of MEA by trace components such as sulphur oxide and corrosion.[4] Cryogenic removal of CO_2 does not require any chemicals addition and can be operated at athmospheric pressure. Yet, as the process is conducted at very low temperature, the water content at the feed stream become critical parameter to avoid plugging of the cooling unit due to the ice formation.[4]

In recent years, research on membrane based gas separation technology have been significant due to its low energy demand, compactness, low maintenance requirement, flexibility in scale up, and easy to operate.-[5–13] Polymeric membranes are favourable due to their unique intrinsic transport properties, low cost, and flexibility in fabrication and engineering.[14, 15] The performance of polymeric membranes for gas separation is characterized by the gas selectivity and permeability. In general, there are three types of polymeric membranes for gas separation; dense, asymmetric and composite. Asymmetric membrane in particular, posses several advantages such as relatively high gas permeance due to the thin selective top-layer and good mechanical strength due to the presence of support sub-layer. However, defects in selective top-layer and sub-layer resistance become challenges in asymmetric membrane application. Pinnau and Koros, introduced dry/wet membrane fabrication method to overcome those challenges, which included convective or free standing evaporation of polymer solution prior to immersion in the precipitation bath. In this method, determination of membrane fabrication parameters, such as solvent evaporation time and solvent ratio, were crucial to control membrane thickness and avoid membrane defect.[16] Despite the optimum membrane thickness and the lack of membrane defects, trade-off between gas selectivity and permeability on polymeric asymmetric membranes application exists with Robeson's upperbound to be surpassed.

Recent advances on membrane for gas separation focused on mixed matrix membranes (MMMs) to overcome the selectivity and permeability trade-off. MMMs are consisted of two or more materials, one of which forms continuous phase known as matrix, while another material forms discrete phase, or filler. Polymers are usually used as the continuous phase and inorganic materials, such as zeolites, silica, carbon molecular sieves, nano metal oxides, and metal organic frameworks (MOFs) have been frequently used as the discrete phase in MMMs fabrication.[17, 18] In prior works on MMMs for gas separation, reduction of gas permeability were reported due to extended tortuous path as the filler particle was added to the polymer matrix. This prediction was postulated as Maxwell model. However, further work investigated the n-butane/CH_4 gas separation performance of PMP/nano-sized silica MMMs, showed contradictive results with Maxwell model. Compared to the PMP membrane, the n-butane/CH_4 selectivity was twice as much with the incoporation of nano-sized silica in the matrix of high-free-volume glassy polymers.[19] In addition, remarkable enhancement in n-butane permeability was also observed.

Ahn, et al investigated the O_2/N_2 and CO_2/CH_4 gas separation performance and permeation properties of MMMs prepared from polysulfone (PSf) and nonporous silica nanoparticles.[18] The addition of non-porous nano-sized silica, which had opposed properties with porous inorganic fillers, showed great potential to affect polymer chain packing in glassy and high-free-volume polymers. Due to the non-permeability of the nonporous silica particles, the incorporation of this filler into the polymer matrix did not directly contribute to the change of transport property. However, the particles altered the molecular packing of the polymer chains, resulting on improved gas permeation as well as the selectivity.

One-third of CO_2 emitted worldwide was produced by the combustion process in power plant. As the so-called flue gas has low CO_2 content, typically ranged from 3 to 15%, the gas flow rate to be processed is significant. In this particular condition, CO_2 permeance is an important parameter to justify the feasibility of MMM membrane for CO_2 separation. In this work, flat sheet MMMs (FSMMMs) were fabricated following dry/wet method with PSf as the continuous phase and colloidal nano-sized silica as the disperse phase. The effect of three membrane fabrication parameters, such as solvent ratio, force convective time and nano-silica loading, on the FSMMMs performance were investigated. The membrane fabrication parameters were varied following the Box-Behnken experimental design to optimize membrane formulation and provide statistical validation on the results.

Materials and methods

Materials

PSf polymer was purchased from Solvay Advanced Polymers (Alpharetta, GA) under the trade name of ‘Udel Polysulfone P-3500 LCD’. The 40 wt.% dispersion of colloidal nano-silica was purchased from DuPont. The nominal size of nano-silica particles was 12 nm[20, 21] with net negative charge at pH above 1.6. BET surface area and the pore volume of the particles are 262 m^2/gram and 0.75 cm^3/gram, respectively. N,N-dimethylacetamide (DMAc) and tetrahydrofuran (THF) were used as less volatile and more volatile solvents in the membrane fabrication. The organic non-solvent used in this study was ethanol (EtOH). Tap water and methanol (MeOH) were used as coagulation medium during phase inversion process. All polymers and chemicals were used as received without further purification.

Membranes fabrication

Asymmetric PSf flat sheet membranes were fabricated by casting method. PSf particles with concentration of 22 wt% were firstly dissolved in DMAc, THF, and 14.4 wt% EtOH (nonsolvent). The composition of DMAc and THF was varied with the weight ratio of DMAc: THF of 0.75, 1 and 1.25. The solution was then mixed in a beaker glass using a magnetic stirrer at 60°C for 6 h to form homogeneous casting solution.

The dry/wet phase separation process was conducted to prepare asymmetric flat sheet membranes. Casting solutions were cast on a glass plate with a casting knife at ambient temperature of 27°C and relative humidity of 83%. The gap between the casting knife and the glass plate was 200 µm. Forced-convective evaporation was induced by blowing inert gas stream across the membranes surface for a certain period. Afterward, the casted membranes were immersed in water as the coagulation medium for 10 minutes and methanol for 2 hours. The membranes were then air-dried for 1 day. The air-dried membranes were further dried in desiccators for 1 day prior to gas permeation test and membrane characterization. The schematic diagram of asymmetric membrane preparation is shown in Figure 1a.

Asymmetric mixed matrix PSf flat sheet membranes were also prepared by casting method following the aforementioned procedure. The colloidal nano-silica was added into the DMAc-THF mixture and dispersed under ultrasonic treatment for 30 minutes to achieve better inorganic particles distribution. The polymer was then added and stirred at 60°C for 6 hours to form homogeneous casting solution. The solution was casted on a glass plate and after evaporation step, the membranes were immersed in the coagulation medium followed by air drying.

Gas permeation test

Single gas permeation tests using nitrogen (N_2) and carbon dioxide (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 ambient temperature of 27°C. Feed pressure was set at 1.7 bar while permeate side was set at atmospheric pressure. The experimental set-up of gas permation test is presented in Figure 1b. CO_2 and N_2 permeances were determined by recording the gas flux through the membranes and permeance were calculated following Eq. (1).

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

where $(P/l)_i$ is the permeance of gas i (GPU), Ji is the flux of gas i, and Δp is the pressure difference across membrane. Gas selectivity of the membranes were calculated following Eq. (2).

$$\alpha_{CO2/N2} = \; (P/l)_{CO2}/(P/l)_{N2} \tag{2}$$

To provide better understanding of the effect of nano-silica loading inside polymer matrix on the gas separation performance, the experimental results of gas permeation test were compared with ideal Maxwell equation model as shown in Eq. (3).[22]

$$P_f = P_o\left(1 - {}_f\right)/\left(1 - 0.5_f\right) \tag{3}$$

where P_f and P_o are the permeability coefficients of the inorganic-filled polymer matrix and of the pure polymer, respectively, and ∅f is the volume fraction of nano-silica particle in polymer matrix. This model predicts the reduction of gas permeability in hybrid system of non-porous filler and polymeric matrix due to the loss of polymer volume available for sorption and increased length of penetrant diffusion pathway.

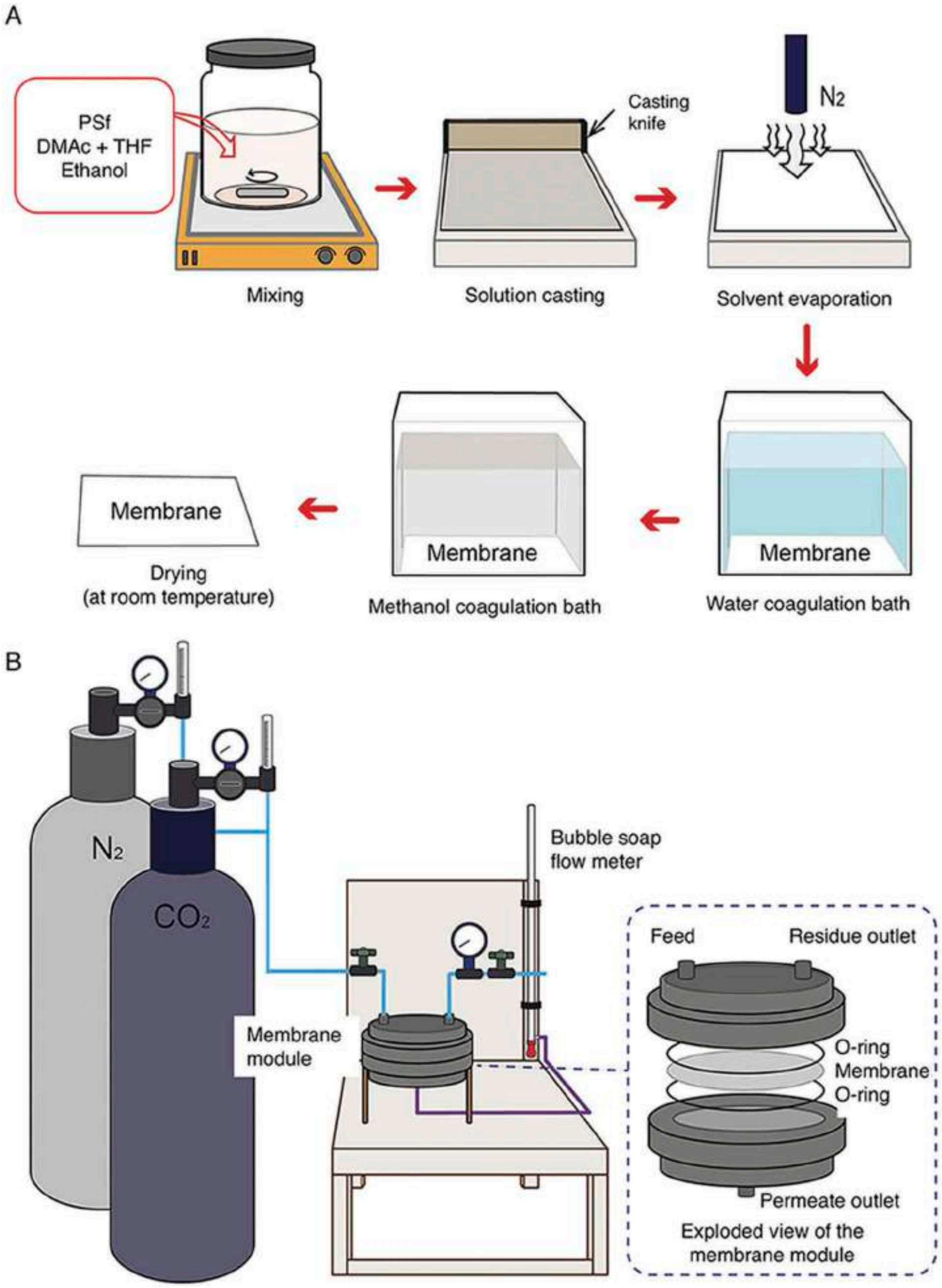

Figure 1. (a) Schematic of membrane preparation steps, and (b) Gas permeation experimental set up.

Membrane characterization

Morphology of the prepared membranes were observed under scanning electron microscope (SEM) JEOL JSM-6360LA equipped with energy dispersive spectrometer system JEOL JED-2300 at 5000 times magnification. Prior to the SEM characterization, the membranes were sputtered with gold. Membrane densities were measured by gravimetric analysis as described in the literature.[18] The weight of the membrane samples was measured by an analytical balance and the volume of membrane samples were obtained by dissolving the samples into the solvent. The measured density (ρ_M) was then calculated as the ratio of sample weight and volume. The theoretical density (ρ_T) was calculated using Eq. (4), with the polymer volume fraction (ϕ_p) equal to 1 - ϕ_f.

$$\rho_T = \rho_p \varphi_p + \rho_f \varphi_f \tag{4}$$

where ϕ_f is the volume fraction of the inorganic component. The excess specific volume fraction (ϕ_{ex}) was calculated using Eq. (5).

$$\varphi_{ex} = 1 - \rho_M / \rho_T \tag{5}$$

Table 1. Factors, their coded levels, and actual values as used in the experimental design.

	Actual values of coded levels		
Variables	−1	0	1
Solvent weight ratio, DMAc:THF (A)	0.75	1.00	1.25
Evaporation time (B), s	10	20	30
Nano-sized silica loading (C), wt.%	0	1	2

Experimental design

Box-Behnken experimental design for response surface methodology was used to investigate the influence of DMAc:THF solvent weight ratio (A), evaporation time (B), and nano-sized silica loading (C) on CO_2 permeance and CO_2/N_2 selectivity of the asymmetric PSf membrane. Box-Behnken design is an incomplete factorial design, which consists of a central point and middle points at each edges of the experimental design box. This design is more efficient compared to the three level factorial design and central composite design. As the factors in Box-Behnken design are at the middle level, combinations of factors at extreme conditions (e.g. highest or lowest levels) can be avoided.[23] Each operating conditions was set as independent variables with three variations and coded as −1 (low), 0 (medium) and 1 (high). The coded and actual values of the variables are presented in Table 1. The experiments were conducted in three blocks, in each of which two operating conditions were varied on low and high level while keeping one operating condition constant. Experiment on center point (i.e. at medium level of all operating conditions) was conducted in three replicates. Analysis of Variance (ANOVA) was performed to determine the significance of each operating parameters and interaction of operating parameters on the CO_2 permeance and CO_2/N_2 selectivity using statistical design software Minitab version 16.

Additional experiments with nano-silica loading of 0.5 wt.% and 1.5 wt.% were also conducted subsequent to the analysis of Box Behnken experimental design to provide better understanding on the effect of nano-silica loading to the PSf/silica MMMs performance.

Results and discussion

Summary of ANOVA

The effect of main parameters and their interaction on CO_2 permeance and CO_2/N_2 selectivity were determined statistically through the Box-Behnken of response surface methodology (RSM). The effect of three main parameters, such as solvent ratio (A), evaporation time (B) and nano-sized silica loading (C), on the gas separation performance were assesed by 15 randomized experiments. The regression equations of CO_2 permeance and CO_2/N_2 selectivity are presented in Eq. 6 and 7.

$$\begin{aligned} CO_2\ \text{permeance (GPU)} = {} & 51,28 - 63,9A - 1,454B \\ & +0,85C + 26,85A*A + 0, \\ & 0083B*B - 2,48C*C + 1, \\ & 30A*B + 9,62A*C + 0, \\ & 063B*C \end{aligned} \tag{6}$$

$$\begin{aligned} CO_2/N_2\text{selectivity} = {} & -19,15 + 39,41A + 0,70B - 1, \\ & 54C - 17,68A*A - 0,017B*B \\ & +0,58C*C + 0,053A*B - 1, \\ & 47A*C - 0,027B*C \end{aligned} \tag{7}$$

The statistical significance of regression equations (Eq. (6) and (7)) were checked by conducting the F-test, and the results are shown in Tables 2 and 3. The calculated

Table 2. Summary of ANOVA for CO_2 permeance as response.

Source	DF	Sum of square (adj)	Mean of square (adj)	F-value	P-value
Model	9	839.6	93.3	214.1	0.0
Linear	3	734.0	244.7	561.5	0.0
A	1	321.7	321.7	738.3	0.0
B	1	46.1	46.1	105.7	0.0
C	1	366.2	366.2	840.6	0.0
Square	3	38.7	38.7	29.6	0.0
A x A	1	10.4	10.4	23.9	0.0
B x B	1	2.5	2.5	5.9	0.0
C x C	1	22.8	22.8	52.3	0.0
2-way interaction	3	66.9	66.9	51.2	0.0
A x B	1	42.2	42.2	96.8	0.0
A x C	1	23.2	23.2	53.1	0.0
C x D	1	1.6	1.6	3.7	0.1
Error	5	5	5	0.4	
Lack-of-fit	3	1.8	0.6	3.6	0.2
Pure error	2	0.3	0.2		
Total	14	14			
		Model summary			
	R-sq	R-sq (adj)	R-sq (pred)		
	99.7%	99.3%	96.4%		

Table 3. Summary of ANOVA for CO_2/N_2 selectivity as response.

Source	DF	Sum of square (adj)	Mean of square (adj)	F-value	P-value
Model	9	1.0	0.2	47.3	0.0
Linear	3	1.2	0.4	107.9	0.0
A	1	0.2	0.2	45.7	0.0
B	1	0.0	0.0	10.9	0.0
C	1	1.0	1.0	267.1	0.0
Square	3	0.4	0.1	33.7	0.0
A x A	1	0.1	0.1	33.4	0.0
B x B	1	0.3	0.3	68.9	0.0
C x C	1	0.0	0.0	1.8	0.2
2-way interaction	3	0.0	0.0	0.2	0.9
A x B	1	0.0	0.0	0.5	0.5
A x C	1	0.0	0.0	0.0	1.0
C x D	1	0.0	0.0	0.0	1.0
Error	5	0.0	0.0		
Lack-of-fit	3	0.0	0.0	11.9	0.1
Pure error	2	0.0	0.0		
Total	14	1.6			
			Model summary		
	R-sq	R-sq (adj)	R-sq (pred)		
	98.8%	96.8%	82.3%		

F-values of the model for CO_2 permeance and CO_2/N_2 selectivity were 214.12 and 47.27, respectively. The probability value of both responses were equal to 0, which were lower than the significance level ($\alpha = 0.05$). This indicates the significance of the proposed model. Furthermore, the accuracy of the models were evaluated by the determination coefficient (R-sq) and the adjusted determination coefficient (R-sq(adj)) obtained from the ANOVA results. As shown in Table 2, the calculated R-sq for CO_2 permeance and CO_2/N_2 selectivity were 99,74% and 98,84%. This values were reasonably close to unity and suggested that more than 98% of the variability in the data can be explained by the models. The values of adjusted determination coefficient (R-sq(adj)) and predicted determination coefficient were close, indicated a high level of significance and accuracy of the model. The lack-of-fit P-values for both responses were higher than significance level which showed that the lack of fit is insignificant, indicating a good agreement between the models and experimental data.

The adequacy of the models can be evaluated from the residual pattern. Unstructured residual patterns were expected when the models were correct and the assumptions were satisfied. In particular, the residual should be unrelated to any other variable, including the predicted response. Figure 2(a and b) presents the residual versus the fitted value of the CO_2 permeance and CO_2/N_2 selectivity. Distinct and unstructured pattern moves from negative to positive were observed. The assumption of normally distributed data was also satisfied as the residual value distribute closely to normal distribution line (Figure 2c and d). This pattern indicates that the deviation between the corresponding values was small. Therefore, this response was acceptable.

Simultaneous effect of the membrane synthesis parameters on psf/nano-silica mmms gas separation performance

According to ANOVA results on Tables 2 and 3, all observed main variables (solvent ratio, evaporation time, and nano-silica loading), significantly affected CO_2 permeance and CO_2/N_2 selectivity. The effect of membrane synthesis parameters on CO_2 permeance and CO_2/N_2 were investigated by constructing the three dimensional (3D) response surface plots. Figure 3(a) shows CO_2 permeance as a function of nano-silica loading and solvent ratio at constant evaporation time. The CO_2 permeance increased with the increased of solvent ratio and nano-silica loading. The highest CO_2 permeance was observed at 2 wt.% nano-silica loading and 1.25 solvent ratio, while the lowest was observed at 0 wt.% nano-silica loading and 0.75 solvent ratio. The increase of CO_2 permeance was followed by the reduction of CO_2/N_2 selectivity, as presented in Figure 3(c). The highest CO_2/N_2 selectivity was observed at nano-silica loading of 0 wt.% and solvent ratio of 1.25, while the lowest was observed at 2 wt.% nano-silica loading and 0.75 solvent ratio.

The effect of evaporation time and solvent ratio on membrane performance is shown in Figure 3(b and d) with increased CO_2 permeance at higher solvent ratio. In this work, solvent ratio indicates the ratio of less volatile solvent (DMAc) to more volatile solvent (THF). Therefore, during solvent evaporation step, membrane fabricated with solvent ratio of more than 1 formed thinner top-layer due to less solvent being evaporated and less polymer concentration at the outermost skin region in the nascent membranes. This led to lower mass transfer resistance and resulted on much

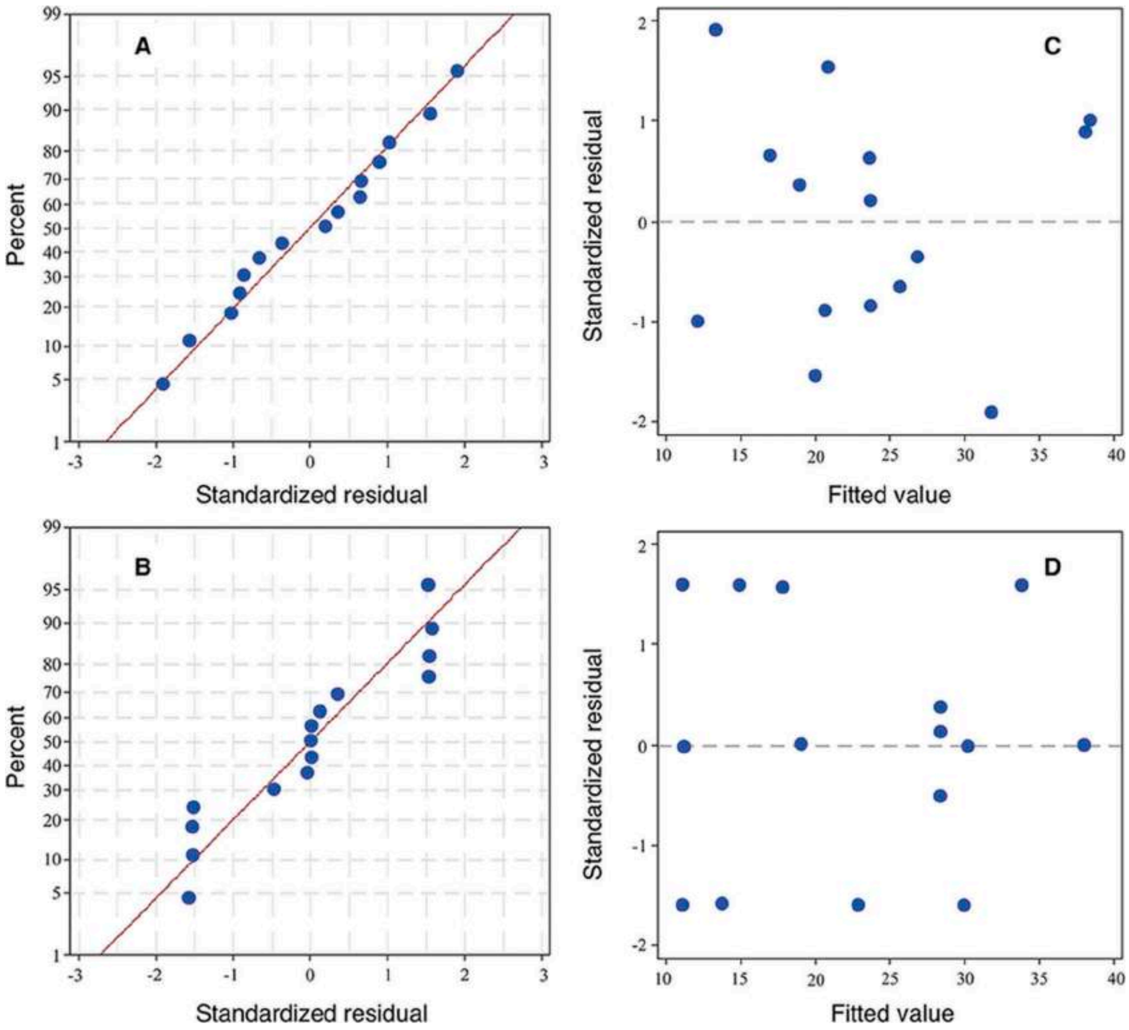

Figure 2. Normal plot of standardized residual for (a) CO_2 permeance and (b) CO_2/N_2 selectivity and standardized residual for (c) CO_2 permeance and (d) CO_2/N_2 selectivity.

increased CO_2 permeance. However, thinner top-layer is generally not defect-free, indicated by reduced CO_2/N_2 selectivity (Figure 3(d)).

While the focus of this work is to fabricated MMMs membrane with high permeance, membrane selectivity is also of importance for process efficiency. The results in Figure 3(c) shows selectivity reduction with the addition of nano-silica. On the experiments without nano-silica loading, solvent ratio and evaporation time influence the CO_2/N_2 selectivity as indicated in Figure 4. Highest CO_2/N_2 selectivity of 15.61 was observed at solvent ratio of 1 and evaporation time of 20 s. At experiment with evaporation time of 10 s, insufficient evaporation time led to lower polymer concentration at the outermost skin region and resulted on defects formation on membrane top-layer. Longer evaporation time of 30 s led to higher polymer concentration and thicker top-layer, which inhibited the transport of gases through the membrane. At the optimum membrane fabrication parameters, the CO_2 permeance was 14.22 GPU.

The SEM image of the asymmetric PSf membrane fabricated following dry/wet phase inversion method (22 wt.% PSf; 31.8 wt.% DMAc; 31.8 wt.% THF; 14.4 wt.% ethanol; and evaporation time = 20 s) is presented in Figure 5(a) and in accordance to previous result observed by Pesek and Koros.[4] The cross section image of the membrane showed a relatively well-defined dense skin layer with invisible defects. The approximate dense skin layer thickness was 1.2 µm. The supported membrane layer had a highly porous open-celled sub layer containing macrovoid. The morphology of MMMs with 1 wt.% and 2 wt.% nano-silica loading are shown in Figure 5(b) and (c), respectively. The incorporation of nano-silica particles into

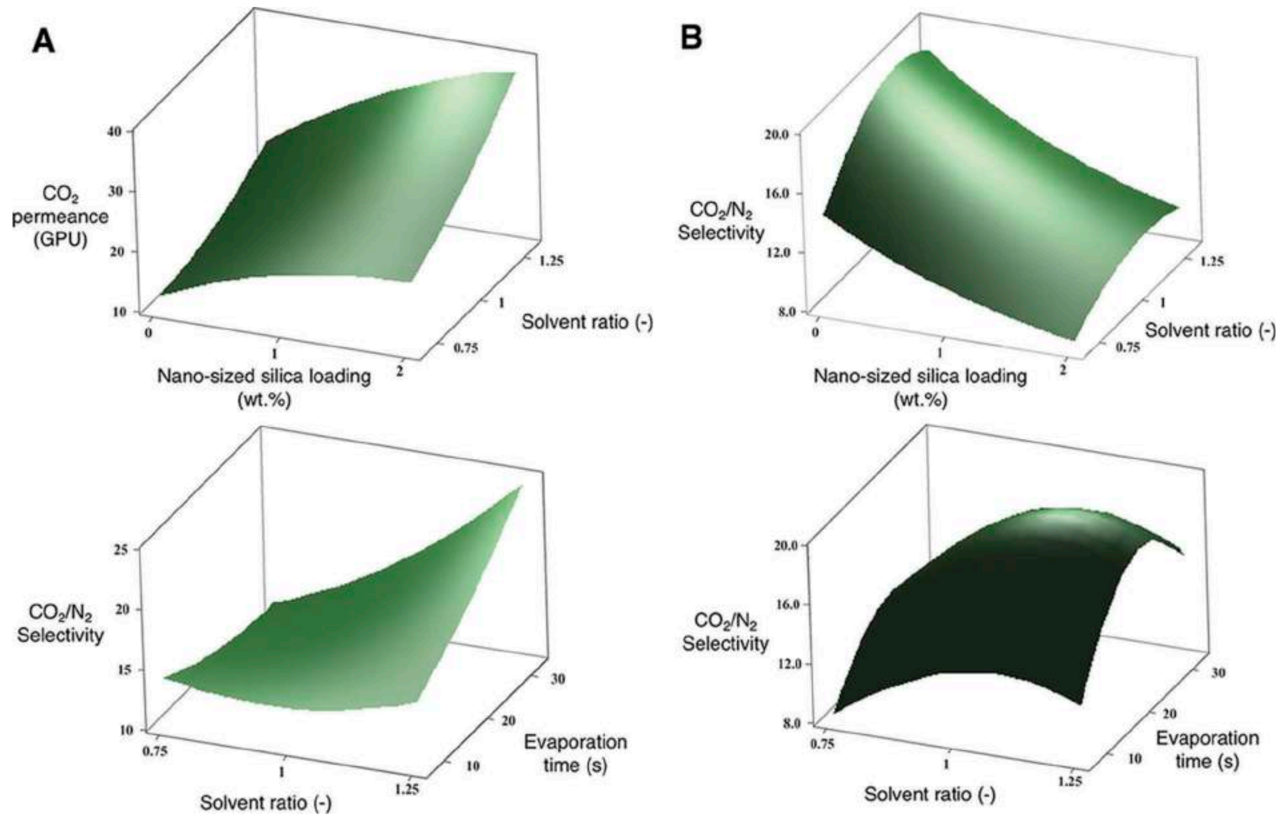

Figure 3. Effect of (a) nano-silica loading and DMAc:THF solvent ratio (evaporation time = 20 s), (b) DMAc:THF solvent ratio and evaporation time (nano-silica loading = 1 wt.%) on CO_2 permeance and effect of (c) nano-silica loading and DMAc:THF solvent ratio (evaporation time = 20 s), (d) DMAc:THF solvent ratio and evaporation time (nano-silica loading = 1 wt.%) on CO_2/N_2 selectivity.

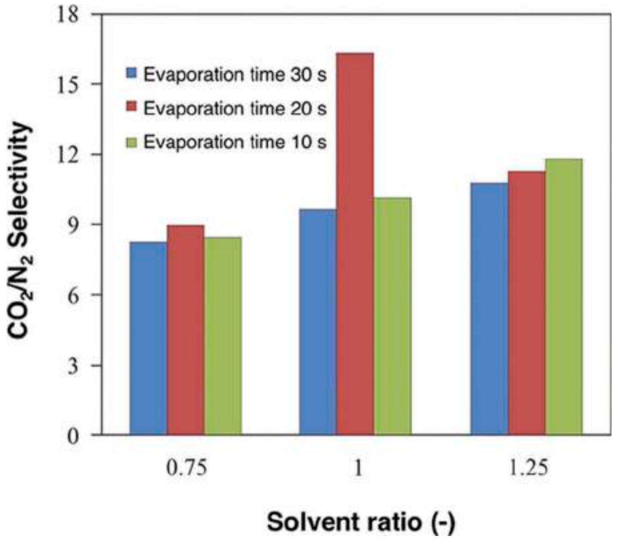

Figure 4. Effect of DMAc:THF solvent ratio and evaporation time on CO_2/N_2 selectivity without nano-silica loading.

polymer matrix induced particle agglomeration, which increased with the increase of nano-silica particles concentration, as indicated by the black arrows.

The incorporation of nano-silica in PSf asymmetric membranes provided significant effect on membrane gas separation performance as indicated by blue and orange line in Figure 6 (A). The results from this study demonstrated that the presence of relatively low nano-silica loading resulted on significant increase in CO_2 permeance (up to 181.2% compared to pristine PSf membrane). However, the enhancement of N_2 permeance was more eminent, thus resulting on 49% decrease in CO_2/N_2 selectivity. For comparison, previous study on dense PSf membrane incorporated with 20 vol% nano-silica showed an increase in CO_2 gas permeability up to 310% with 62% decrease in CO_2/CH_4 selectivity.[16] This was due to the microvoid formation in dense skin layer as the organic and inorganic materials were poorly interacted.

Relative gas permeance calculated using Maxwell equation for MMMs is also presented in Figure 6(a). The results indicated that the model could not be applied in this work. This equation predicts lower gas permeability of nonporous inorganic particles filled membrane compared to unfilled membrane. An increase in volume fraction of inorganic particles leads

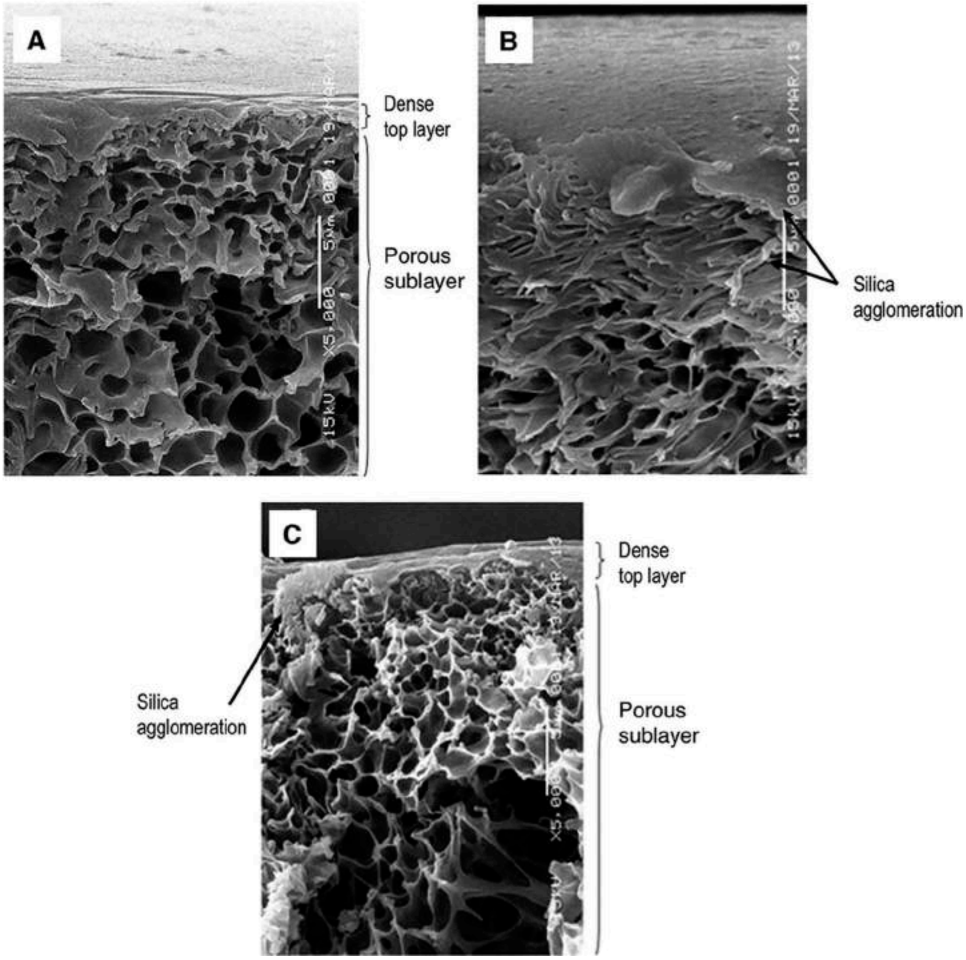

Figure 5. (a) Cross section SEM images of asymmetric PSf membrane with 0wt.% nano-silica loading, (b) MMMs PSf/nano-silica with 1wt.% nano-silica loading and (c) MMMs PSf/nano-silica with 2wt.% nano-silica loading. (PSf = 22 wt%; DMAc = 31.8 wt%; THF = 31.8 wt%; Ethanol = 14.4 wt%; and evaporation time = 20 s).

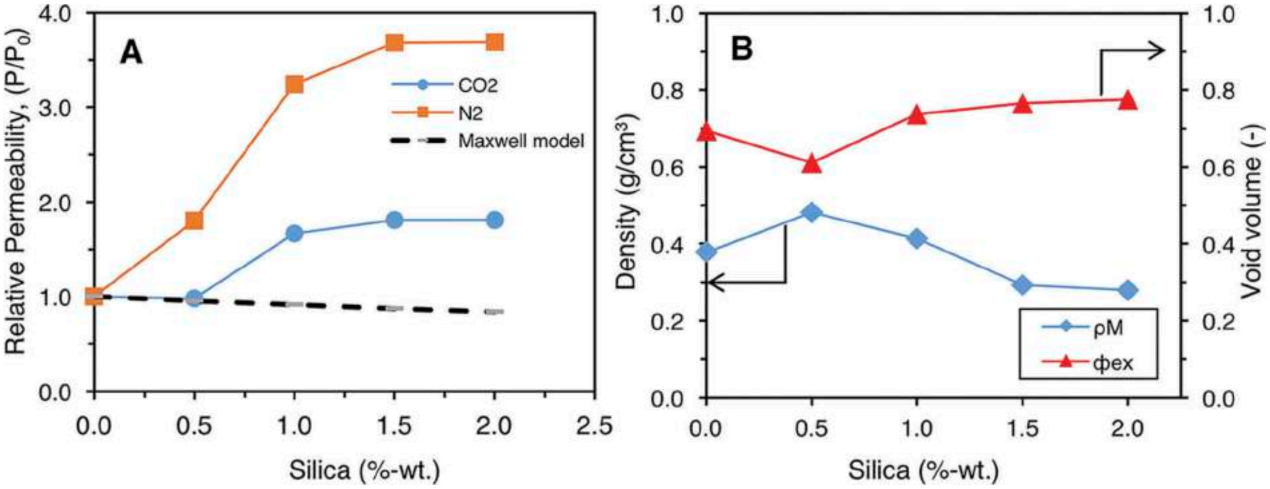

Figure 6. (a) Enhancement of relative permeance of PSf–nano-silica MMMs to a variety of penetrants as a function of nano-sized silica loading (PSf = 22 wt%; DMAc = 31.8 wt%; THF = 31.8 wt%; Ethanol = 14.4 wt%; and evaporation time = 20 s). The CO_2 and N_2 permeance at experiments with 1wt.% and 2wt.% nano-silica loading were 14.22 GPU and 0.91 GPU, respectively. (b) Void volume fraction and density of polysulfone/nano-silica MMMs as a function of nano-silica loading.

to decrease in gas permeability due to the decrease in diffusivity by increasing the tortuosity in the polymer matrix. According to Aroon et al.[24], the Maxwell equation is applicable to dilute suspension of spheres and can only be applied for low particle loadings (less than 20 wt.%). This model based on the assumption that the streamlines around particles are not affected by the presence of nearby particles. In addition, the ideal Maxwell equation actually assumes negligible interaction between fillers and polymer matrix. These phenomena are in agreement with previous study[18], which stated that introduction of nano-silica causes a substantial change of free volume (i.e., quantity of nanospace). Pinnau et al.[25–27] also reported that the addition of nonporous nano-silica to rigid, glassy high-free-volume polymers (e.g., PMP and PTMSP) increased the total free volume of the membrane.[25–27] Void volume in the mixed matrix asymmetric PSf membrane was formed due to the incompatibility of silica – polymer interfacial and particles agglomeration. The fraction of void volume in membrane structure can be determined by the density of the membrane. The increase in membrane void volume can be explained by lower measured density as indicated in Figure 6(b), which shows enhancement of excess void volume with the increase in nano-silica volume fraction due to the incompatibility of silica – polymer interfacial.

Comparison of membrane performance in this study and literature

The gas separation performance of PSf membranes fabricated in this study, particularly those with highest CO_2 permeance (nano-silica loading of 2.wt%) and highest selectivity (nano-silica loading of 0.wt%), were compared with several membranes in selected literatures, as shown in Table 4. Membranes performance in this study and literature were determined by single gas permeation tests. The highest CO_2 permeance of membranes fabricated in this study was obtained in test using 2%.wt nano-silica loading MMM, which was more than four times higher than PSf membranes in literature. However, the CO_2/N_2 selectivity of 2%.wt nano-silica loading MMM was significantly lower at 7.7. The highest CO_2/N_2 selectivity of membranes fabricated in this study was obtained in test using membrane without nano-silica loading. Compared to PSf membranes in the literature, the selectivity of membrane without nano-silica loading was approximately 30% lower and the CO_2 permeance was more than two-fold higher. Therefore, as the CO_2 permeability of the membrane facbricated in this study was significantly higher, it can potentially be applied for gas separation operation at high feed flow rate despite its lower selectivity.

Conclusion

Performance of PSf-based asymmetric membrane for CO_2/N_2 separation can be tailored by optimizing membrane fabrication parameters, such as solvent ratio, evaporation time and nano-silica loading. The solvent ratio, evaporation time and nano-silica loading had significant effect on membrane selectivity and permeance. The results of experiments designed following Box-Behnken method indicated reduced membrane performance with the addition of nano-silica due to more significant N_2 permeance increase compared to CO_2. At relatively low nano-silica loading there was a significant increase in CO_2 permeance to 181% and a decrease in CO_2/N_2 selectivity to 49%. This was due to the increase of void volume in membrane dense top layer structure as the result of polymer/nano-silica interphase incompatibility and particle agglomeration. The optimum membrane performance (selectivity of 15.6 and CO_2 permeance of 14.2 GPU) was observed at the following membrane fabrication parameters: 22 wt.% PSf, 31.8 wt.

Table 4. Comparison of PSf membrane performance in this study and literature.

Membrane material	Feed pressure (atm)	Temperature (°C)	P (CO_2) (Barrer)	P (N_2) (Barrer)	Selectivity (CO_2/N_2)	Reference
PSF	10/5	35	5.6	0.3	22.4	[28]
PSF-F	10/5	35	4.5	0.2	22.5	[29]
PSF-O	10/5	35	4.3	0.2	21.5	[29]
PSF-P	10/1	35	6.8	0.3	21.3	[30]
PSF-AP	2	35	8.1	0.3	29.2	[31]
PSF-M	1	35	2.8	0.1	25.5	[30]
This work*	1.7	27	17.1***	1.1***	15.6	-
This work**	1.7	27	30.9***	4.0***	7.7	-

*membrane fabrication parameters: PSf = 22 wt%, DMAc = 31.8 wt%, THF = 31.8 wt%, Ethanol = 14.4 wt%, evaporation time = 20 s, nano-silica loading = 0wt.%.

**membrane fabrication parameters: PSf = 22 wt%, DMAc = 31.8 wt%, THF = 31.8 wt%, Ethanol = 14.4 wt%, evaporation time = 20 s, nano-silica loading = 2wt.%.

**Approximate membrane thickness in this work is 1.2 µm.

% DMAc, 31.8 wt.% THF, 14.4 wt.% ethanol, 20 s evaporation time and 0 wt-% nano-silica loading. Comparing the performance of membrane fabricated at optimum condition in this study with the membranes in literatures, 32% reduction of membrane selectivity was observed. However, more than twice higher CO_2 permeance was obtained using membrane in this study. This highlight the opportunity of using the membrane fabricated in this study in application with high flow rate.

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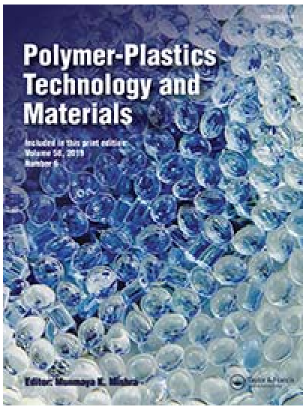

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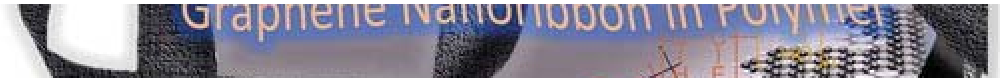

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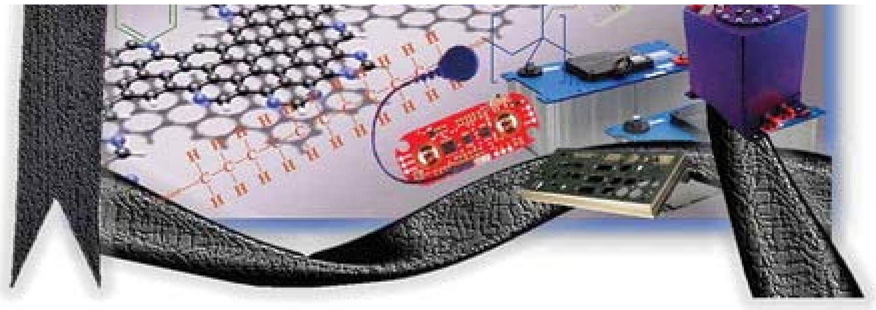

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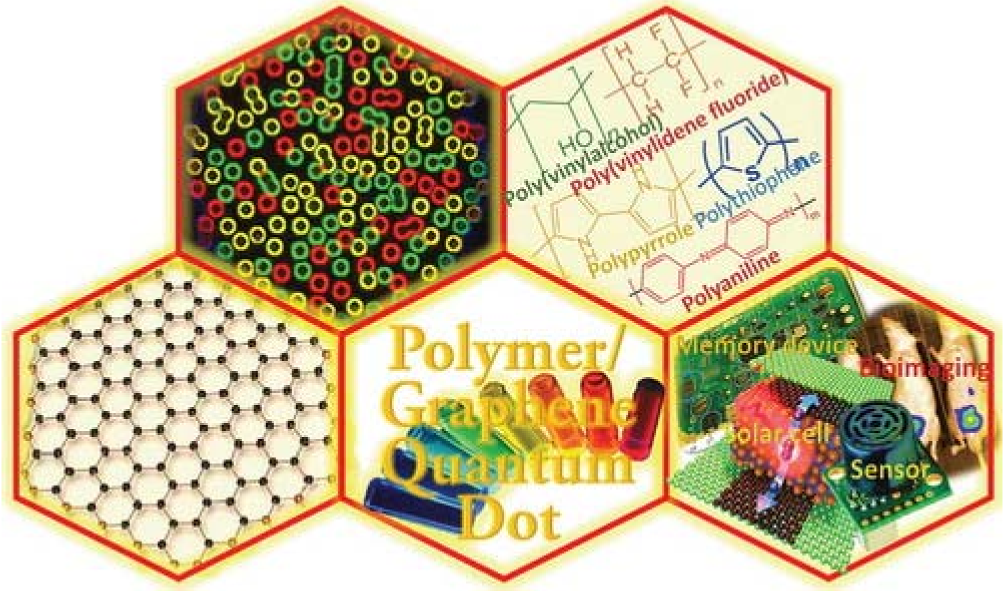

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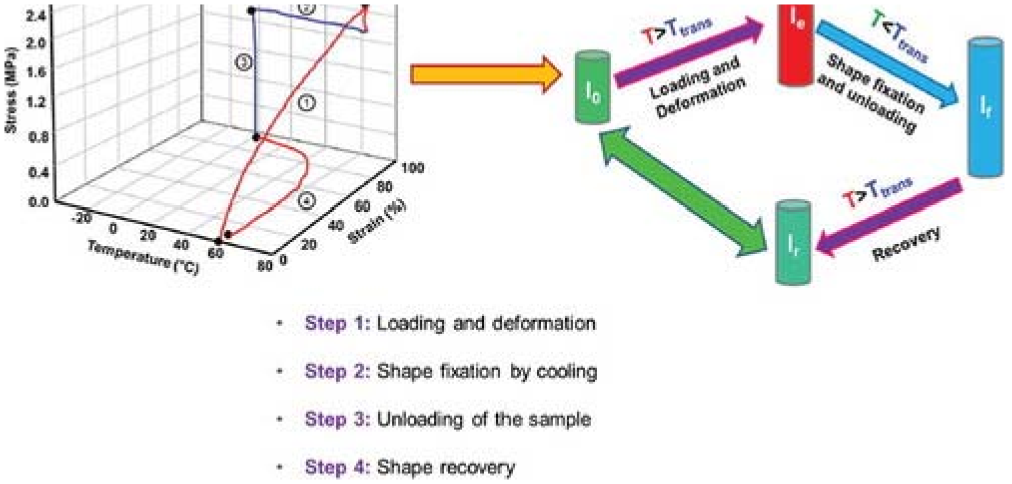

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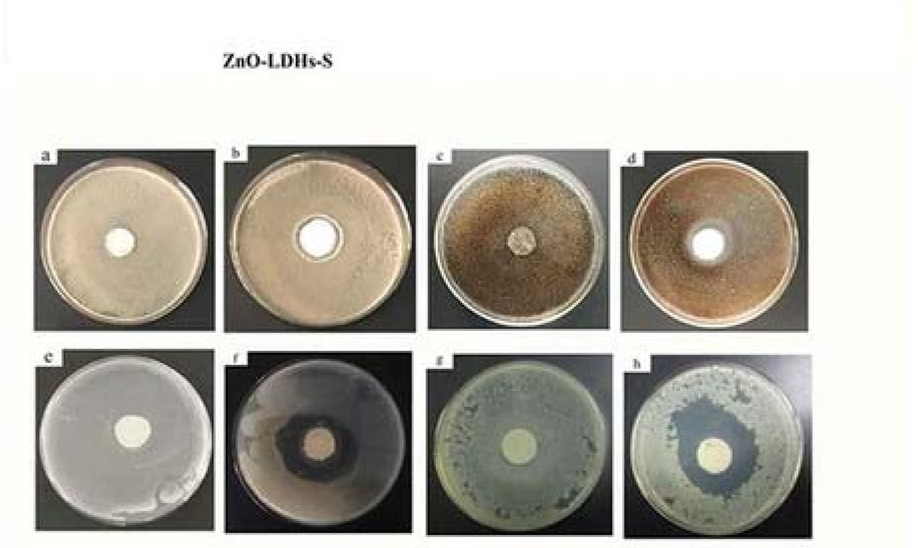

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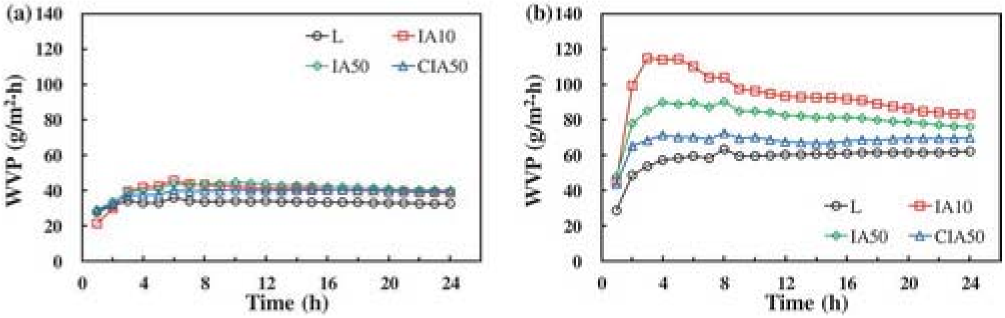

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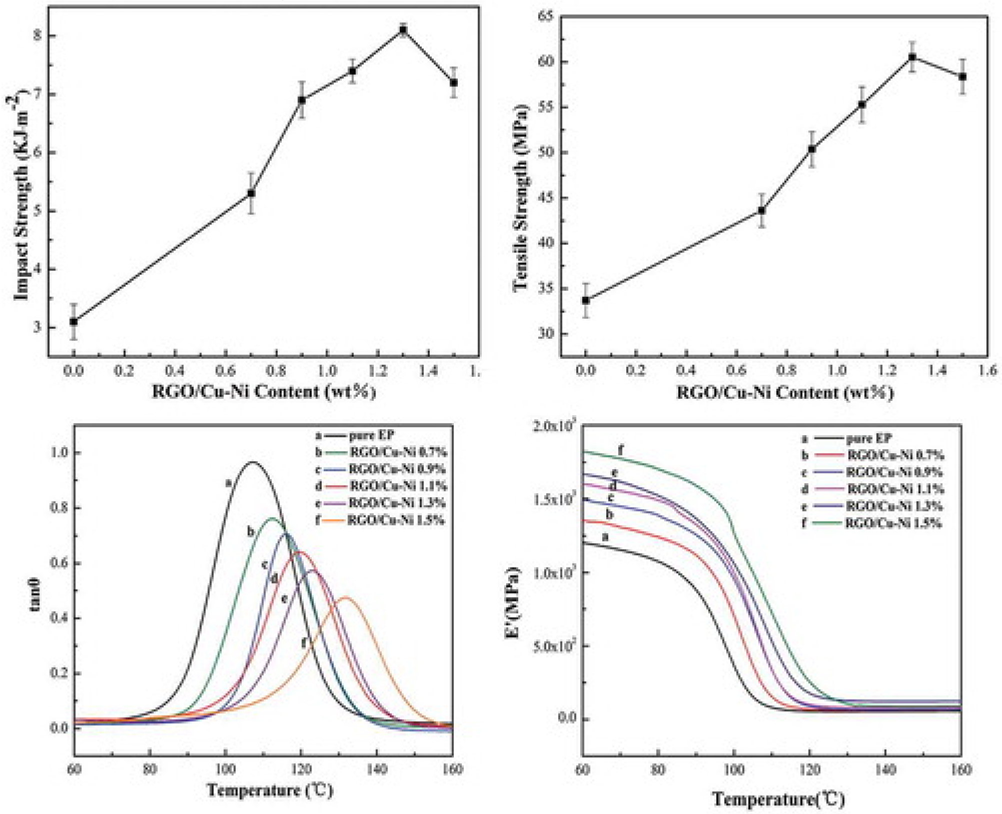

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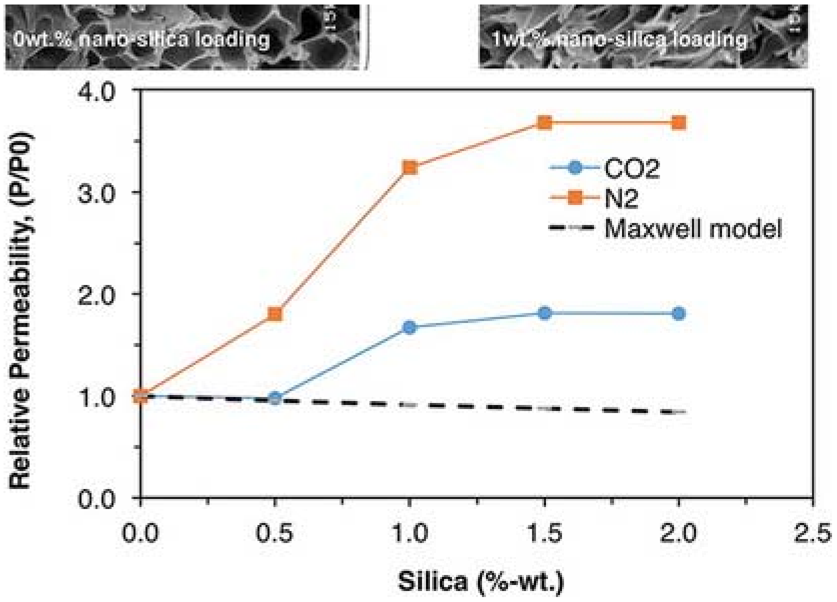

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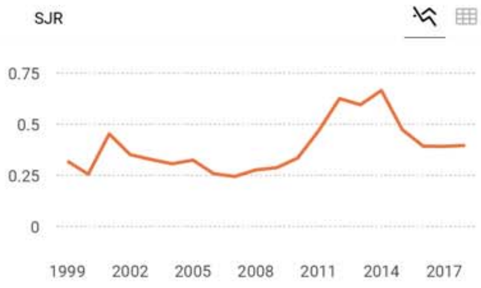

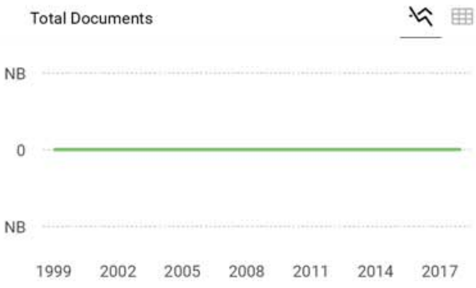

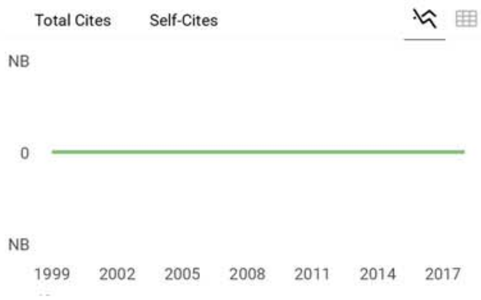

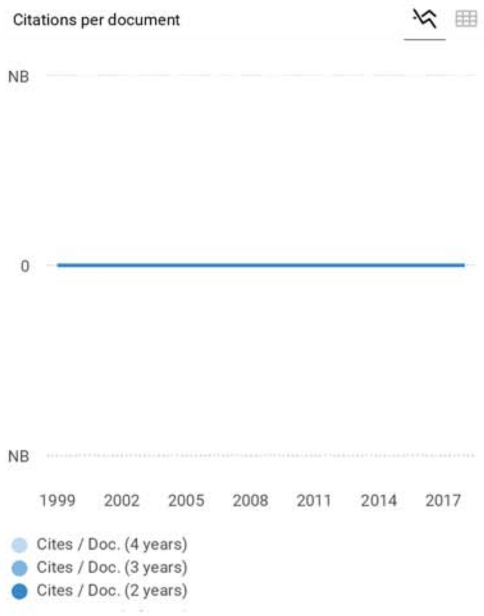

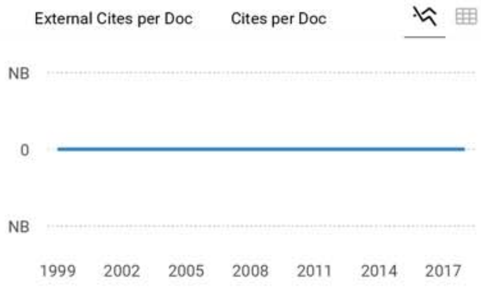

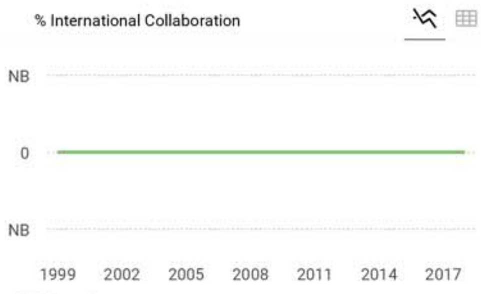

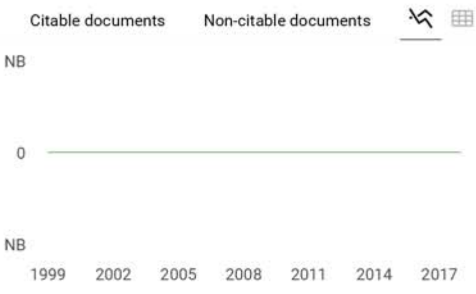

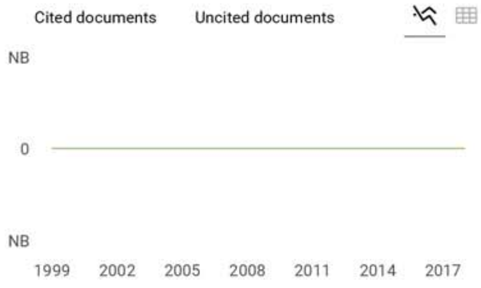

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