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Ceramic Membrane Surface Roughness Induced by Modified Phase Inversion: The Effect of Thermodynamic Properties

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ABSTRACT

Emphasizing upon the need to develop and target application within surface roughness of ceramic membrane, this work addresses the new method of 'modified phase inversion'. Phase inversion that commonly used in the fabrication of polymeric membrane was applied. The thermodynamic properties (composition and temperature) involved in the phase inversion system was studied. Knowledge of the surface roughness of ceramic membrane is very important towards it characterization and performance. In this study, the investigation towards the surface of ceramic membrane was characterized in term of surface roughness and morphology. Kaolin with 25µm was used as the composition of membrane preparation. The amounts of kaolin investigated were 60g, 70g and 80g respectively while the temperature was conducted assist room temperature and 3oC. Result of atomic force microscopy (AFM) showed that by decreasing phase inversion temperature improve the membrane surface roughness from 0.136 µm to 0.093 µm at 80g of kaolin content. Similar trend for the study towards the effect of kaolin composition. In addition, the SEM images explained that membrane surface morphology obviously change with the effect of temperature during phase inversion process. Experimental investigations inferred that te membrane performance is optimal for minimum combination of thermodynamic properties, temperature (room temperature) and kaolin composition (60g) at which conditions, PWF and mean pore radius were obtained as 80.1221 Lm-2h-1 and 0.112µm respectively. Membrane prepared at low temperature (3oC) possesses smallest pore size of 0.043µm under 80g of kaolin composition.

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INTRODUCTION

Ceramic membranes have exhibited potential application like separation and purification in order to achieve better environment. This is due to the high mechanical strength, chemical resistance and thermal (Kingsbury and Li, 2009). Phase inversion was first implemented by Loeb and Sourirajan in the production of high flux polymeric membranes (Loeb and Sourirajan, 1963). Recently, phase inversion method was applied to the fabrication of polymeric membrane. Nowadays, phase inversion have been extensively applied in the fabrication of ceramic membranes (Wei *et al*., 2008; Wang and Lai, 2012). This is due to the traditional method that applied in the fabrication of ceramic membrane requires long period to produce the final ceramic membrane. The asymmetric structure of ceramic membrane can be produced by the phase inversion system [Figure 1]. In addition, the thermodynamic properties of the phase inversion is important to produce membrane with desired characteristics. According to Harun *et al*., phase inversion is the system that was easily to be applied by changing the thermodynamic properties. It was reported that ceramic membrane fabricated at low temperature solve the problem of unsolidified membrane and resulting a better porosity (Harun *et al*., 2014). Thus, the fabrication method from Harun *et al* was applied in this work in order to study the effect towards membrane surface roughness and morphology.

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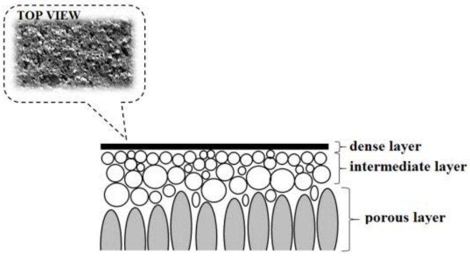

Fig. 1: Asymmetric structure of ceramic membrane.

Thermodynamic properties of phase inversion involved many parameters such as temperature and composition. In fabrication of ceramic membrane via phase inversion, every parameters are very important in producing desired structure. These have been reported elsewhere (Lalia *et al*., 2013). In formation of porous polymeric membrane, the term phase inversion was used to describe the phase changing of the membrane fabrication mechanism. Conceptually, membrane formation by phase inversion system can be described by ternary diagram which consist of three components; polymer, solvent and non-solvent. Prior to fabrication of ceramic membrane via phase inversion, the system can be explained by the exchange between the solvent and the non-solvent coagulant bath which induces the precipitation of polymer in the suspension and thus consolidate the ceramic material (Baker, 2004). Previously, concentrations and temperature represent for the polymer solution is called Flory-Huggins model (Flory, 1953) which can be described as in Figure 2. In most cases however, the thermodynamical properties can be described reasonable through a Flory-Huggins model at constant temperature and pressure, when concentration dependent interaction parameters are used (Pouchly *et al*., 1969). In contrast of preparing ceramic suspension, there is one additional component which are raw material or powder. Due to this, Kingsbury *et al* have proposed a viscous fingering mechanism which explain the finger-like formation in ceramic membrane structure (Kingsbury *et al*., 2009). This phenomenon was further studied by Wang *et al* in verify unique features of the viscous fingering phenomenon in alumina/PES/NMP suspension (Wang *et al*., 2012). However, this phenomenon investigated towards membrane cross section only and their interpretation did not exclude the possibility that other properties also involved before the immersion precipitation method of phase inversion. In addition, the membrane surface also plays an important role in membrane characterizations and performances. In this study, we are proposed a new method as “modified phase inversion system” to demonstrate that the thermodynamic properties (composition and temperature) is also responsible for the surface membrane morphology.

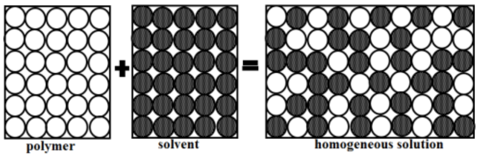

Fig. 2: Flory-Huggins Theory Diagram.

Early studies of this phenomenon originated from observations of unbalanced solidified of ceramic membrane prepared via phase inversion (Harun *et al*., 2014). Most investigations on fabrication of ceramic membrane via phase inversion systems were carried out in hollow fiber shape (Othman *et al*., 2010; Dong *et al*., 2014; Pan *et al*., 2012). The mechanism of phase inversion is still the same, however, it affects the outer surface of membrane which therefore disturbed the membrane characterizations and performances. Thus, the proposed method of ‘modified phase inversion’ in this study perhaps solve this problem. Among various parameters of membrane fabrication, thermodynamic properties such as composition and temperature are commonly used. According to Das and Maiti, low temperature leading to better homogeneity, high purity and controlled chemical composition (Das and Maiti, 2009). In addition, temperature also affect the viscosity of suspension which change the mechanism of demixing process during the phase inversion system. Recently, similar phenomenon has been observed by Shohur. M.F when preparing polymeric membrane due to the effect of different non-solvent and temperature of coagulant medium (Shohur, 2014). Phase separation mechanisms can generally be subdivided in three main categories depending on the parameters that induce demixing. By posing a change in one of these parameters at one particular side of the film, asymmetric boundaries are posed on the polymer film which can be expressed in the resulting structure. By changing the temperature at the interface of the polymer solution, heat will be exchanged and demixing can be induced (temperature induced phase separation or TIPS). The original polymer solution can also be subjected to a reaction which causes phase separation (reaction induced phase separation) (RIPS). The most used technique is based on diffusion induced phase separation (DIPS). By contacting a polymer solution to a vapour or liquid, diffusional mass exchange will lead to a change in the local composition of the polymer film and demixing can be induced. For the interpretation of membrane formation, the term delayed and instantaneous demixing are further studied in relation to its diffusional mechanism. Composition also is one of the thermodynamic properties in phase inversion. According to Zeng *et al*, surface roughness of ceramic membrane is strongly affected by composition (Zeng *et al*., 1997). In the phase inversion process induced by immersion precipitation, commonly a polymer solution cast onto a suitable support such as glass plate which then immersed directly into non-solvent coagulant bath. In this work, the non-solvent coagulant bath used is distilled water. The same method was applied in the ‘modified phase inversion’ method by changing the immersion method. In this work, the casting solution was dried for 24h before directly immersion method. Subsequently, this mechanism can be explained by the delay demixing phenomenon which formed a thicker dense layer (Wienk, 1996).

In this work, the effect of ‘modified phase inversion’ method was studied towards ceramic membrane surface roughness. AFM and SEM were utilized to elucidate the changes in surface roughness and surface morphology which results from the effect of thermodynamic properties (composition and temperature) via phase inversion system. AFM is a relatively recent technique with a resolution which can reach atomic dimensions for flat surfaces (Albrecht and Quate, 1987). In addition, this technique produces topographical images by scanning a sharp tip, at the end of a cantilever, over the target surface (Binnig *et al*., 1987). According to Li, the AFM have much better function over the SEM as it can zoom images much smallest as 1nm that that of SEM which is around 100nm only. In addition, AFM can produce three dimensional image which SEM can only produce two dimensional images.

Surface roughness and surface morphology of ceramic membrane is also significantly affecting the application involved such as filtration. They play an important role in determining the characteristics of membrane fouling (Zhong *et al*., 2013). Rough surface is more easily to be fouled due to large surface area. In addition, the membrane flux decreased due to higher surface roughness. Therefore, methods to control the ceramic membrane surface roughness is so important. Refer to Corneal *et al*., surface morphology can be affected by surface modification (Corneal *et al*., 2010). However, surface modification involved many complicated steps and it required long period. In addition, it can change the original characteristic of the ceramic membrane. To reach the global aim of the present work, the pure water flux (PWF) performance and mean pore radius was investigated to relate within the effect of surface roughness. The PWF was determined the volume of water that drips penetrate the membrane layer at the time period specified. The PWF was investigated using Archimedes’ principle by using equation 1:

$$PWF = \frac{Q}{A\ x\ \Delta t} \tag{1}$$

where Q is permeate volume (L), A is effective membrane area ($0.00159m^2$) and Δt is permeate time (0.167h).

Filtration process divides into four stages like Microfiltration, Ultrafiltration, Reverse Osmosis, and nanofiltration as illustrated in figure 3. The different between those processes basically base on their pore molecules size and separation capability. In this work, the type of membrane fabricated was investigated by using mean pore radius (r_m). Mean pore radius r_m (μm) on the other hand was determined using the filtration velocity method. According to the Guerout–Elford–Ferry equation, r_m (Basri *et al.*, 2011) could be experimentally determined by equation 2:

$$Mean\ pore\ radius, r_m = \sqrt{\frac{(2.9-1.75\varepsilon)x\ 8\eta lQ}{\varepsilon\ x\ A\ x\ \Delta P}} \tag{2}$$

where η is the water viscosity (8.9× 10−4 Pa s), ℓ is the membrane thickness (m), Q is the volume of the permeate water per unit time ($m^3\ s^{-1}$), ε is porosity calculated at previous study (Harun *et al*., 2014), A is the membrane effective area ($0.00159m^2$) and ΔP is the operational pressure (0.1 MPa).

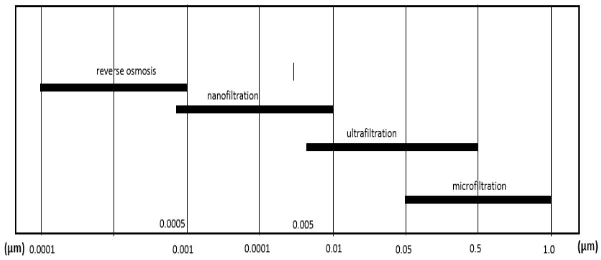

Fig. 3: Type of membrane based on their pore size (Mulder, 1996).

MATERIALS AND METHODS

Materials:

Dope solutions were prepared using PES (UDEL P1700) as polymeric material, NMP (MERCK) as solvent, sieve 25µm kaolin as raw material and distilled water was used as non-solvent additive and non-solvent bath. All chemicals purchased in this work were used without further purification.

Ceramic membrane preparation:

The 85g of NMP was taken in 100mL beaker and the 15g of PES was slowly added into the NMP solution. The solution was kept stirrer with 500 rpm on the hot plate of 60°C for 3 hours until they completely dissolved.

Table 1: Composition of the suspension.

			Thermodynamic properties	
Sample	NMP (g)	PES (g)	Kaolin (g)	Temperature
S1	85	15	60	3°C
S2	85	15	70	3°C
S3	85	15	80	3°C
S4	85	15	60	Room temperature
S5	85	15	70	Room temperature
S6	85	15	80	Room temperature

Then, the 6g of distilled water was slowly added into polymer dope under constantly agitation. After that, the desired amount of sieved kaolin was subsequently added and kept stirred for at least 10 hours to make sure the dope was completely became homogeneous solution. The desired amounts mentioned were 60, 70g and 80g. To remove air bubbles contained in the dope, the solution was left in the oven with 60°C and to avoid local precipitation occurred. If local precipitation occurred, the agitation was continued until the dope became homogeneous again. After that, the ceramic suspension was cast on a glass plate with casting knife and the thickness of the membranes was controlled within 1.5mm by using masking tape at each side of the glass plate (Sarbatly, 2011). The cast film was left under two conditions: 1) room temperature; 2) ice box, 3°C for 24 hours. Then, the membranes were immersed in distilled water overnight to remove NMP and then dried at room temperature. Accordingly, the ceramic membranes green body was heated using furnace (*PLF 140/5*) at 800°C for 3 hours in order to completely remove the binder and another 1100°C for 3 hours to for fusion and bonding occur. The furnace is set up 100°C for every 1 hour before reached hold temperatures. The compositions of the suspensions used are list listed in Table 1. The main stages of the preparation process required for the modified phase inversion membrane used in this work are described in Figure 4.

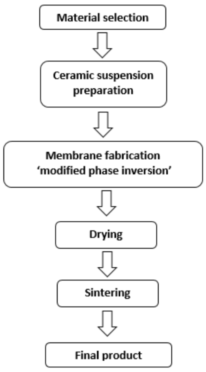

Fig. 4: Main stages of modified phase inversion ceramic membrane.

Atomic Force Microscopy (AFM):

All AFM experiments were carried out using XE-100 Park at Mint-SRC, UTHM. AFM samples preparation started with cutting a 1cm x 1cm of ceramic membrane before sintering procedure. Measurement were performed on dry membrane samples under ambient atmospheric conditions. Roughness is reported as average roughness (R_a).

Scanning Electron Microscopy (SEM) examination:

In a similar method as for the AFM samples, SEM samples were prepared by cutting 1cm x 1cm before sinter. Then, the membrane samples were immersed in liquid nitrogen, fractured carefully then coated with thin platinum prior to SEM analysis.

RESULTS AND DISCUSSION

Surface morphology characterization:

The AFM images showed the representative 3-D surface morphologies. Table 2 and Figure 5 revealed the surface roughness of the membrane within the effect of composition and temperature. Table 2 list the mean roughness parameter (R_a), root-mean-squared roughness (R_q) and the point average roughness (R_z).

For the membrane at kaolin composition of 60g, the surface roughness is 0.218 µm and 0.306µm, at different temperature, respectively. Compared to membrane at 80g, the surface roughness is 0.093µm and 0.136µm, respectively. According to the AFM measurements, the surface roughness of the ceramic membrane decreased with increased of kaolin composition. Refer to Zeng *et al*., surface roughness of ceramic membrane is strongly affected by composition (Zeng *et al*., 1997). This is due to the larger surface area provided by the ceramic membrane at low kaolin composition (60g). In fact, crystal structure of kaolinite is the reason of the membrane roughness.

In contrast, the surface roughness of the ceramic membrane prepared at low temperature (3°C) is less rough compare to the ceramic membrane prepared at room temperature. This phenomenon is due to the evaporation process that takes place easily at lower temperature that have high humidity (Harun *et al*., 2014). In addition, the kaolin particles freeze at low temperature and packed closely together which reduce surface area of the ceramic membrane.

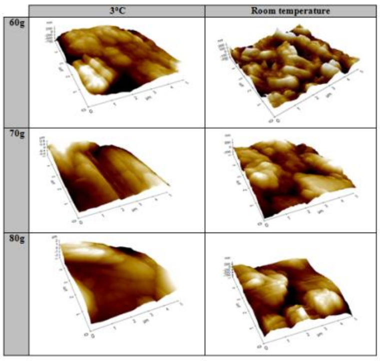

Fig. 5: AFM images and membrane surface roughness (R_a) value of ceramic membranes, (a) 3°C/60g; (b) room temperature/60g; (c) 3°C/70g; (d) room temperature/70g ;(e) 3°C/80g; (f) room temperature/80g.

Table 2: Average ceramic membrane surface roughness via AFM.

	R_a (µm)	R_q (µm)	R_z (µm)
S1	0.218 ± 0.03	0.282 ± 0.05	2.255 ± 1.13
S2	0.113 ± 0.07	0.153 ± 0.07	0.137 ± 1.07
S3	0.093 ± 0.05	0.099 ± 0.02	0.101 ± 0.98
S4	0.306 ± 0.06	0.417 ± 0.06	0.531 ± 0.37
S5	0.298 ± 0.03	0.312 ± 0.05	0.311 ± 0.12
S6	0.136 ± 0.03	0.170 ± 0.08	1.001 ± 1.23

Figure 3 presents the SEM images of the membrane prepared at different temperature and composition. Obviously, surface morphologies of ceramic membrane were strongly influenced by the composition and temperature. From the SEM images, figure 6(e) shows the smoothest image of ceramic membrane surface and give the lowest value of surface roughness (R_a = 0.093 µm). Various application of ceramic membrane was referred to its unique characteristics. In some application, membrane required to have hydrophobic or hydrophilic characteristic. Refer to Kujawa *et al*, hydrophobicity of the ceramic membrane can be achieved when the membrane surface roughness decreased (Kujawa *et al*., 2014).

Permeation result:

The effect of temperature of prepared ceramic membrane on PWF via modified phase inversion system was evaluated as shown in figure 6. It is shown that the PWF decreases when the kaolin content increases at low temperature. The same results indicated by the membrane prepared at room temperature. The trend of PWF shown in figure 7 is expected by the relation of porosity result (Harun *et al*., 2014) that increases too. Zaini *et al*., also claimed that porosity has a direct relationship with the flux permeation and that was the key for the membrane performance (Yunos *et al*., 2014). In this work, the 60g kaolin of ceramic membrane prepared under low temperature is 43.6551 $Lm^{-2}h^{-1}$ while at room temperature is 80.1221 $Lm^{-2}h^{-1}$. The slowest PWF was illustrated from the membrane prepared at 80g under low temperature. This is due to the surface roughness of membrane which is the smoothest compared to others prepared membranes (figure 5 and 6). The aim of this experiment was to study the effect of thermodynamic properties towards membrane surface roughness.

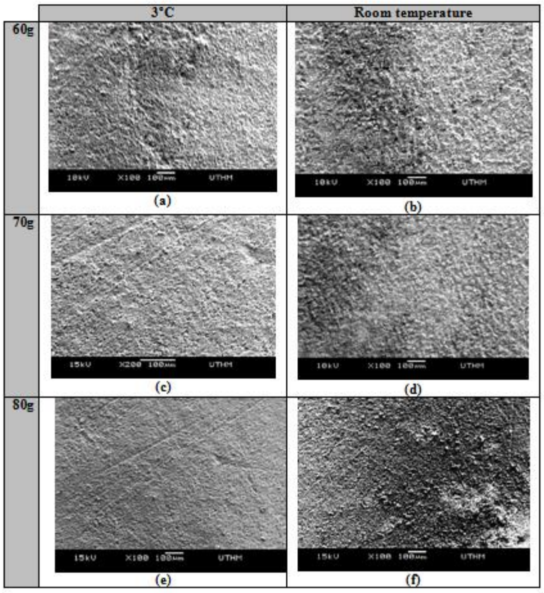

Fig. 6: FESEM images of ceramic membranes, (a) 3^{o}C/60g; (b) room temperature/60g; (c) 3^{o}C/70g; (d) room temperature/70g ;(e) 3^{o}C/80g; (f) room temperature/80g.

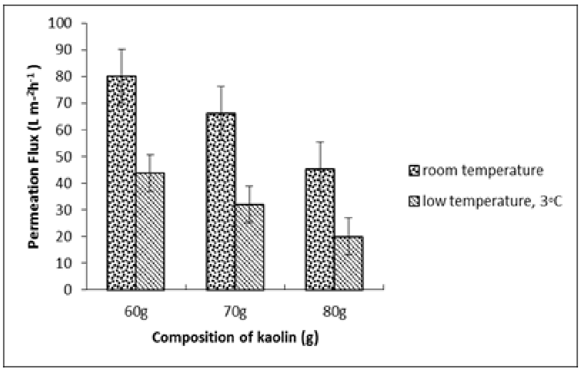

Fig. 7: Comparing thermodynamic properties of modified phase inversion ceramic membrane.

From the literature review at previous section, the surface roughness of membrane have strong relationship with the flux performance. For all cases, it has been observed that distinct membrane flux, profiles have been obtained for combinations of composition and temperature with the membrane flux (figure 7) and pore size (figure 8). According to Emani *et al*., 2014, substantial enhancement in membrane fouling did not occur with the enhancement of these two parameters. Similar flux profiles have been obtained within the membrane surface roughness by Zhong *et al*., 2011.

Mean Pore Radius:

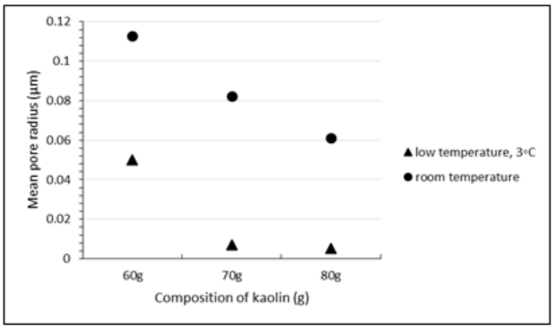

Fig. 8: Effect of thermodynamic properties towards mean pore radius (r_m).

Figure 8 shows membrane mean pore radius investigated from the Archimedes principle. From the mean pore radius trend, it was clearly seen that the addition of kaolin composition has reduced the membrane pore size in which the pore size decreased from 80g prepared membrane by 36% and 46% with kaolin composition of 70g and 80g respectively under room temperature. In contrast, the pore size decreased in low percentage for membrane prepared at low temperature (3^oC). The pore size decreased from 80g by 11% and 25% with kaolin composition of 70g and 80g respectively. This could be due to rapid solvent exchange that took place during modified phase inversion at room temperature resulting in leaching of kaolin particles and causing the formation of bigger pores.

Conclusion:

With limited literature data, this work emphasized upon the need to develop new method of 'modified phase inversion' towards ceramic membrane fabrication. The modified phase inversion was studied thermodynamically. The thermodynamic properties that have been studied in this work were temperature and composition. Membranes prepared under low temperature (3^oC) possessed smooth surface with smallest pore size. These characteristics were decreased the PWF performance. However, this membrane was deemed to have good integrity towards rejection performance and membrane fouling that will be further studied. In contrast, prepared membranes under room temperature indicated better PWF performance subjected to the bigger pore size and rougher surface significantly. Thus, it is apparent that temperature do have relationship with the membrane surface roughness. Comparing the other thermodynamic properties, composition, also shows the significant effect towards membrane surface roughness. Overall, the combinations of thermodynamic properties (temperature and composition) possesses the effect towards membrane surface roughness (with the comparison between optimal rough and smooth membrane surface of 0.306 ± 0.06 µm and 0.093 ± 0.05 µm respectively) which provided a slightly difference PWF of 80.1221 Lm^{-2} h^{-1} and 21.0173 $Lm^{-2}h^{-1}$ respectively. Inferentially, this work was further highlighted upon the need to prepare the ceramic membrane via 'modified phase inversion' towards others membrane performance. Thus, in the near future, research shall target upon the rejection and fouling performance.

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