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Evaluation of the Performance of Nigerian Kaolin for Oil-Water Filtration Ceramic Hollow Fiber Membrane

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Membrane Technology has gained more interest in recent years for the treatment of oily wastewater. Among the membranes, ceramic membranes hold the prerequisite properties such as hydrophilicity, antimicrobial and thermal stability. In this study, ceramic hollow fiber membrane prepared from kaolin obtained from Kankara in Katsina State, Nigeria was investigated for the filtration of oily-wastewater treatment. EDS analysis of the kaolin showed adequate amount of Silica and alumina, 55% and 49% respectively suggesting that Kankara kaolin can be used in manufacture of ceramic membranes. The effect of different kaolin concentrations, 34, 35.5 and 37% on ceramic membrane formation was also investigated. AFM analysis of the hollow fiber ceramic membrane topography showed that 34 wt% kaolin membrane exhibited higher water permeability as compared to other membranes. This was attributed to the surface packing density and porosity of the membrane. Interestingly, 34% membrane also exhibited higher oil-water flux with rejection of more than 90%. Maximum oil-rejection and lesser fouling percentage were observed for 37 wt% membrane. This study suggests the possible utilization of low-cost ceramic membrane for oily wastewater treatment.

Keywords: Kaolin, Ceramic, Hollow-fiber-membrane, Oily-wastewater

1. Introduction

Currently, emphasis is given to low-cost ceramic starting materials for membrane fabrication process as known materials such as alumina and zirconia are relatively expensive. Kaolin is a clay mineral that is composed of mainly alumina and silica as its main constituents. Before now, it was used in the production of porcelain, support for gas separation and as catalyst substrates (Emani *et al.*, 2014; Hubadillah *et al.*, 2016; Hubadillah *et al.*, 2017) . It is chosen as the preferred raw material over the later mention ceramic materials due to it cheapness, crystal order, chemical composition, mineralogical content and abundancy in almost all countries. Also, investigation shows that kaolin provides low plasticity and high refractory properties to the membrane (Mittal *et al.*, 2011).

Recently, porous ceramic membrane developed from kaolin has attracted emergence interest into various purification/separation applications such as treatment of wastewater, this is due to kaolin's outstanding hydrophilic properties(Yang *et al.*, 2011). Vasanth *et al.*, (2013) fabricated ceramic membrane from kaolin (50%), quartz (25%), calcium carbonate (22%) and titanium oxides (3%). The fabricated membrane revealed a steady state flux with rejection of 87% for separation of oily wastewater. In another study, Kumar *et al*. (2015) fabricated a novel tubular ceramic membrane from kaolin, quartz, ball clay, feldspar and pyrophyllite via ram extrusion technique for microfiltration of synthetic oily wastewater. The study offered 53% porosity with lower average pore size of 0.309 µm, corrosion resistance and a stronger mechanical strength of 12 MPa and 99.98% of oil rejection was recorded. These features are recommended in treatment of oily wastewater. Madaeni *et al.* (2012) fabricated ceramic membrane from γ-Al_2O_3 for treatment of oily wastewater from petrochemical industry. The results obtained showed 100% oil rejection. In a similar study, Yeom *et al.* (2016) fabricated and investigated the effects of using kaolin, diatomite, talc, barium carbonate, bentonite and sodium borate as starting materials for flat sheet geometry. The fabricated membrane was used to treat oily water with oil concentration of 600 mg/L and hence the result obtained revealed 99.9% oil rejection. Kaolin stands out to be one of the most used amongst all ceramic materials used in ceramic membrane fabrication, this is due to the fact that it is inexpensive and has a porous structure, which gives high refractory features to membranes (Mittal *et al.*, 2011).

This study thus aims to investigate the performance of ceramic membrane fabricated using Nigerian kaolin via phase inversion and sintering technique for oil-water filtration application.

2. Materials and Methods

2.1 Materials

The Nigerian kaolin clay was collected from Kankara local Government area of Katsina State in Nigeria. The area lies at Latitude 11° 55 and Longitude 7° 25 North-west. The kaolin clay powder was kept in the oven (60°C) overnight before use to completely remove any trace amount of moisture in it. The solvent used was N-2-methyl-2-pyrrolidone (NMP) because of its non-volatility in preparation of the kaolin-based ceramic membrane. Arlacel P135 (polyethylene glycol 30-dipolyhydroxystearate) was chosen as the dispersant and was purchased from Uniqema. Polyethersulfone (PESF) was the polymer binder used during the suspension preparation and was purchased from Amoco chemical.

2.2 Methodology

In order to achieve the best kaolin membrane, kaolin membrane was prepared by varying the kaolin content 34 wt.%, 35.5 wt. % and 37 wt. % and then, the membranes were subjected to sintering temperature of 1400 °C. Table 1 shows the ceramic dope composition and labeling of Nigerian kaolin membrane.

Table 1. *Composition of kaolin dope suspension*

Membrane/ Materials (wt.%)	NK34	NK35	NK37
Kaolin	34	35	37
NMP	60	59	57
PESf	5	5	5
Arlacel P135	1	1	1

2.3 Characterization of kaolin and ceramic membrane

The elemental composition of the kaolin membrane was evaluated using Energy Dispersive X-ray (EDX) Analysis. EDX was applied in detecting/characterizing the elemental surface composition of the membrane using scanning electron microscope (SEM; Hitachi, TM3000). Prior to image acquisition, the samples were coated with gold particles in order to enhance the visibility of the image. The morphology and roughness of the membrane surface were analyzed using atomic force microscopy (AFM: XE-100; Park System, Korea). The membrane was cut into small squares approximately 1 cm^2 and were glue on a glass substrate. The scanning scale is 10 µm x 10 µm.

2.4 Filtration analysis

2.4.1 Water Flux Analysis

The filtration system measurement for the membrane pure water was set up in a crossflow filtration mode. The filtration system was operated at 2 bars and 25 °C. The pure water flux was calculated using Eq. (1):

$$J_w = \frac{V}{t \times A} \qquad (1)$$

Where J_w (L/m^{2}h) is the pure water flux, t is the time (s), A is the total surface area of the membrane (m^2).

2.4.2 Oil-water treatment studies

The oil-water separation test was carried out using synthetic oily wastewater via a laboratory scale microfiltration system. The oil rejection coefficient R was calculated using the equation 2 below;

$$R(\%) = \frac{C_f - C_p}{C_f} \qquad (2)$$

Where C_f and C_p are the concentrations of feed and permeate respectively.

3. Results and Discussion

3.1 EDX chemical composition of the Nigerian kaolin membrane

The contents of the relative elements of the kaolin sample were determined by EDX spectra. As shown in Figure 1, the EDX data confirmed the presence of C, O, Al and Si in the membrane. From the spectra, the membrane had significant amount of Al, Si and O which are the major constituent of ceramic membrane.

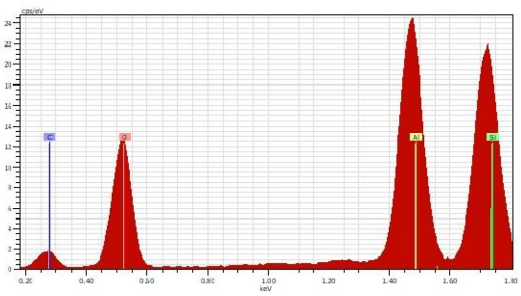

Figure 1. EDX spectra of the fabricated membrane showing: Oxygen (O), Aluminum (Al), Silica (Si) and Carbon (C)

3.2 AFM Topography analysis of ceramic hollow fiber membranes

AFM is an important tool to analyze the membrane surface roughness and arrangements. The fouling property depends on the surface roughness of the membrane. Figure 2 shows the AFM topography of NK (Nigerian Kaolin) membranes prepared at different concentrations (34, 35.5 and 37 wt.%). A valley-like structure was noticed for all the membranes. This is due to the higher roughness. The roughness decreases with the increase of kaolin concentration of the membranes from 34 to 37 wt.%. The higher roughness is due to the formation of dense porous structure and higher concentration of kaolin on the membrane surface. This suggest that NK37 membrane have lower pore structure than others. Thus, it is expected to retain the oil molecules on the surface.

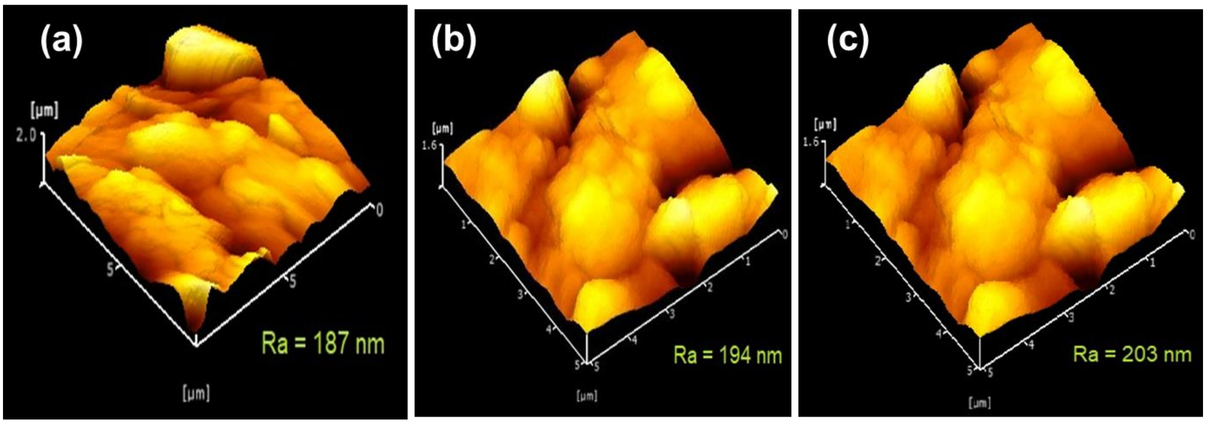

Figure 2. AFM Topography of NK membranes (a) NK34, (b) NK35.5 and (c) NK37.

3.3 Oil-water filtration of ceramic hollow fiber membranes

Figure 3 shows the synthetic oil-water flux for the NK membranes made at different concentrations. The flux increases with increase in Transmembrane Pressure (TMP). Among the membranes, NK34 membrane holds the highest oil-water flux. The desired membrane for the oil-water separation should hold higher flux without compensation of oil-rejection and lower fouling propensity. The higher flux is due to formation of large pores as compared to other membranes. This phenomenon is due to packing arrangement of kaolin. The flux also decreases with the increase of NK concentrations. It was also observed that the flux increases linearly with respect to TMP for other NK35.5 and NK37 membranes. This is due to the lesser oil molecules which are adsorbed on the membrane surface. In the case of NK34 membrane, oil attaches easily due to higher pore size of the membranes. Therefore, it blocks the membrane surface and this was also evident with fouling analysis. Higher flux of 67% was observed for NK34 membranes as shown in Figure 4. While Figure 5 shows the oil-rejection data of NK membranes and the higher retention of 91% was observed for NK37 membrane. This was attributed to the formation of dense porous structure due to increase in kaolin concentration. Among the membranes, NK37 membrane hold lower fouling and higher oil rejection for the synthetic oil-water solution.

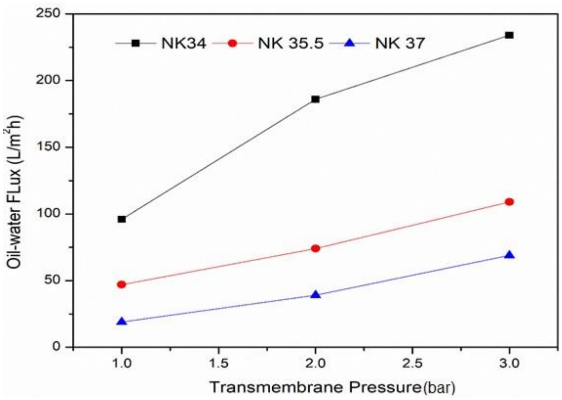

Figure 3. Effect of Transmembrane Pressure on oil-water flux of NK based different concentration membranes.

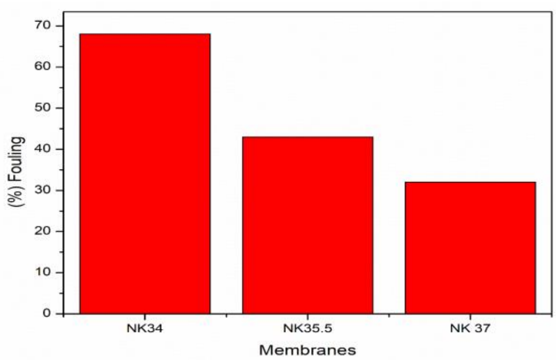

Figure 4. Effect of oil fouling percentage on oil-water solution of NK based different concentration membranes.

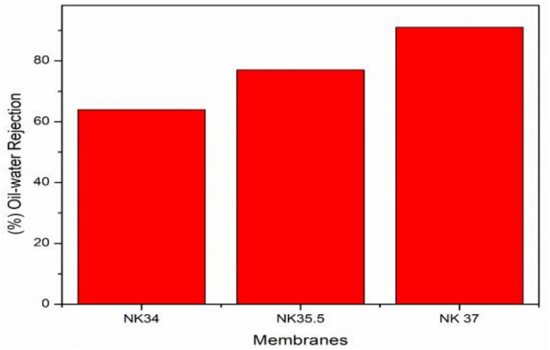

Figure 5. Effect of real time effluent oil-filtration analysis.

Figure 6 shows the NK37 membrane flux for the oil-water effluent analysis. The flux decreases with the increase in time for real time oil effluent. The decline is due to binding of oil solutes to the membrane separation layer. The fouling and oil rejection were found to be 45% and 78% respectively. Flux and oil rejection decreased as compared to synthetic effluent. This is due to the high oil concentration and impurities in the feed. The results are consistent with the works of Emani *et al. (*2014) and Hudillah *et al.* (2017) and indicates that the low-cost kaolin membranes with further modification can be employed for oil-water separation.

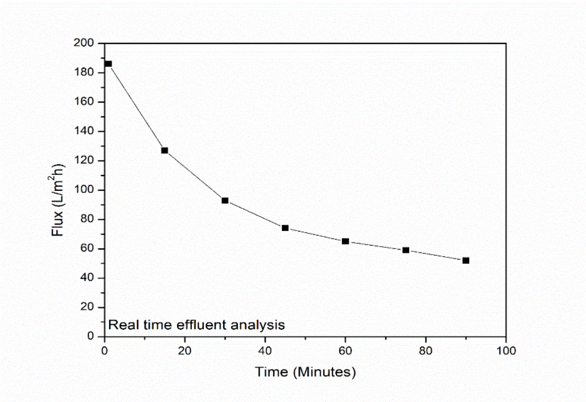

Figure 6. NK37 membrane flux performance of real time oil-water effluent (Transmembrane Pressure-3 bar).

4. Conclusion

Performance studies of NK based membranes of three different concentrations (34, 35.5 and 37 wt%) were successfully tested for oil-water filtration studies. AFM topography analysis showed that higher concentration membranes (37 wt% NK) exhibit higher surface roughness, lower pore size and irregular surface arrangements. Synthetic oil-water filtration studies showed that NK34 membrane exhibited higher flux with lower oil rejection. Interestingly, NK37 membranes showed lesser fouling and higher oil rejection for synthetic oil effluent. NK37 membranes also tested with real time effluent showed that the flux declined with increase in time. The low-cost NK membranes also showed consistent filtration properties with respect to different cycles and batch. Generally, the results reveal that the NK based membrane exhibits good properties and with further modification are good materials for filtration application.

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Conflict of interest

The authors declare no conflict of interest.

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