Fabrication of ceramic membrane from local raw materials for treatment different wastes

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Abstract. Ceramic membrane is a membrane with high chemical and physical stability. In addition, it is very stable chemically, thermally and mechanically and it has a very high selective process without phase transformation. In this work successful fabrication of ceramic membrane through dry compaction method at rather low temperature from, local and available materials such as; local kaolin, fly ash from palm oil mills, chipboard powder wastes, and sand was achieved. Materials used were classified into 4 different composites with different proportions since ratios of the materials significantly affect the membrane properties. All samples were fabricated using mould mainly prepared to make cylindrical shape and pressing machine at approximate pressure of 10kN. After fabrication, all samples were subjected to further processing to increase the membrane physical, chemical and thermal properties. Sintering was applied to achieve this goal. Gradual increase in temperature was applied during the sintering process till it reached 1000 °C for almost 16 hrs. After the samples get into the desired shape, they are tested for different contaminants. UV-spectrophotometer was used to evaluate the removal efficiency of dyes whereas conductivity meter for the removal efficiency of ions. Best results obtained for the treated samples for methylene blue, methylene red and sodium chloride ion, were 96, 76 and 44% respectively. Throughout this study, it can be concluded that ceramic membrane provides physical treatment which is much better than chemical treatment as it does not produce any by-product.

1. Introduction
The concept of membrane filtration is a process where membrane acts as a gate to allow only certain elements to flow (permeate) through while other elements (concentrate) remain in the system. By using this technique, unwanted particles and some contaminants can be eliminated from the water through the porous media. This type of membrane separation technology is in demand because of the environmental regulations and clean water request from the consumers. Waste disposal expense and material recovery can be reduced by using membrane thus altogether ensuring the economic benefit. Variations of membranes like micro-filtration, ultra-filtration and nano-filtration are used accordingly in water and wastewater treatment.

There are two types of membrane which are ceramic and polymer. Ceramic membrane is a type of artificial membrane that is made up from inorganic materials such as alumina, titanium, zirconia oxides, silicon carbide or some glassy materials. This type of membrane has the advantages of high temperature resistance, high acid and alkali resistance, homogeneous pore-size distribution, steady chemical property, high strength, large flux, long lifetime, antipollution, simple structure, small floor area, few
matching equipment, easy installation, no chemical additives, high operating efficiency, high management and automation, and so on [1-6].

Although the treatment efficiency by membrane technology depends on many factors, including membrane pore size, surface change of the membrane, trans-membrane pressure and flux, matrix water chemistry, and bio-fouling, the membrane pore size has been considered to be the most critical parameter affecting the performance for turbidity removal and the physical removal of biological contaminants [7].

It is well known that all industries produce wastes that need to be treated before their disposal into the open environment such as lakes, rivers and seas as a result avoiding environmental contamination and maintaining sustainability [8]. Hence, the main target of this paper is to fabricate durable ceramic membrane made up mainly from local available materials such as kaolin, clay, fly ash by dry compaction method and to utilize the product for treatment industrial wastes.

2. Methodology
Total of 20 composites were prepared to assess the ability for the produced ceramic membrane to remove contaminants. Best results were obtained only from four composites in table 1.

| Raw Materials                | Composite 1 (g) | Composite 2 (g) | Composite 3 (g) | Composite 4 (g) |
|------------------------------|-----------------|-----------------|-----------------|-----------------|
| Sand                         | 20              | 22.5            | 16.5            | 17.5            |
| Kaolin                       | 20              | 15              | 16.5            | 17.5            |
| Silicon carbide              | 20              | 7.5             | 17              | 18              |
| Fly ash from chipboard       | 4               | 7.5             | 5.5             | 5.5             |
| industry                     |                 |                 |                 |                 |
| Fly ash from palm oil        | 4               | 7.5             | 6               | 5.5             |
| waste                        |                 |                 |                 |                 |
| Boric acid                   | 3.5             | 7.5             | 6.5             | 5.5             |
| Sodium metasilicate          | 3.5             | 7.5             | 7               | 5.5             |
| Total (g)                    | 75g             | 75 g            | 75g             | 75g             |

The first step in preparation is the weighing of raw material based on their formulations to create specific ceramic membrane. Considerable mistakes when weighing raw material can affect the result significantly. Precaution also needed to reduce any error. Since some chemicals can be harmful if inhaled or contact with skin, rubber glove and goggle are recommended to be used.
Fabricating ceramic membrane is done by converting ceramic membrane raw material mixture into ceramic membrane product via dry compaction method. After producing homogeneous mixture powder, shaping was carried out by using hydraulic compressor to pack the mixture inside the mould as shown in figure 1. Thoroughly packing with plastic hammer is very important to ensure tight and gas-free sample in order to avoid cracks during sintering process. In order to create hollow ceramic membrane a rod was fixed on the bottom of the mould as shown in figure 1a. Once packing finished the sample is ready for sintering process.

Sintering was carried out in a furnace. The sintering temperature was programmed to 150 °C at the rate of 5 °C/min for a period of 2 h and followed by a heating rate of 5 °C/min for another period of 5 h at 300 °C, then by the same rate till 500 °C and finally to 700 & 1000 °C. The total Sintering time was almost 16 hrs. After that the furnace allowed for gradual drop in the temperature at rate of 5 °C/min till reach room temperature. Details of the sintering process temperature and time are shown in figure 2 and final product of the ceramic membrane is shown in figure 3.
Filtration was carried out by using ceramic membrane as a filter that consist of container in the upper part contains the contaminant. From down it was connected with vacuum pump to receive the filtrate. Two equipment were used to evaluate the filtrate before and after treatment. The UV-Spectrophotometer and conductivity meter. UV-spectrum was used to evaluate the removal efficiency of dyes namely methylene blue and methylene red (MB and MR). The conductivity meter was used to evaluate the removal efficiency of ions (NaCl) in the solution. Under cross-flow filtration set-up, rejection concentration usually evaluated, however in this work for simplicity removal efficiency was calculated based on initial and final concentrations.

3. Results and discussion

3.1. Removal of Methylene Blue (MB)

Under cross-flow filtration set-up, rejection concentration usually evaluated, however in this work for simplicity removal efficiency was calculated based on initial and final concentrations. The formula to calculate the percentage removal is stated as below:

\[
\text{Percentage removal (\%) } = \frac{A - B}{A} \times 100\%
\]

Where:

- \(A\) = Absorbance of methylene blue before filtration
- \(B\) = Absorbance of methylene blue after filtration

During sintering process in order to remove moisture, temperature was kept below 150 °C for almost 3 hrs. Further removal of moisture was achieved at 300 °C or below for another 3 hrs. From 500 °C till 700 °C all volatile organic carbon (VOCs) in the composites supposed to be evaporated. Boric acid and sodium metasilicate were used as a binder whereas chipboard and fly ash at high temperature tend to produce porosity and their ratios effect the pore size of the membrane.

From the experiments conducted, the maximum removal efficiency of methylene blue was noticed for composite 3 and reach to 76.34 % as shown in figure 4. Minimum removal efficiency was noticed for composite No 4 that was 66.67%. This could be attributed to the components used as a binder and also components responsible to form the porosity of the membrane. Slight change in the ratios for these components achieved significant change in the removal efficiency.
3.2. Removal of Methylene Red (MR)

Based on equation (1) removal efficiency for methylene red was calculated. As depicted in figure 5, the removal efficiency of red methylene reached more than 96% for composite 3, whereas the minimum removal efficiency for the same sample filtrate was obtained by composite No 4.

From the results obtained for methylene blue and methylene red, it is clear that ratios of the binders and porosity elements are the main reason for the obtained results.

3.3. Removal of Sodium Chloride (NaCl)

The pore size of reverse osmosis (RO) membrane is 0.0001 μm which is smaller than the size of the sodium chloride Molecule which is 0.0007 μm and will not let it through. Nor will it let through germs and viruses, and organic molecules which are very much larger than the RO pore. RO membrane purifies H₂O (0.000275 μm in size) from almost all chemicals allowing water to pass through and molecules
smaller than the water molecule, like CO\textsubscript{2} gas. Smaller molecules than water is generally not harmful to health as shown in figure 6.

![Diagram showing pore size compared with molecules, bacteria, and virus approximate values and figure is not to scale.](image)

**Figure 6.** Pore size compared with molecules, bacteria, and virus approximate values and figure is not to scale.

In order to evaluate the removal efficiency of the produce membrane, equation (1) was used in similar manner for calculation removal efficiency for NaCl. According to figure 7, composite No 2 shows higher removal efficiency than other composites. Almost 45\% of the salt was removed from this sample. Even though sintering process for all samples was carried out under the same conditions, composites ratios could be the reason for the show results. As shown in the same figure, minimum removal was occurred for composite No 1.

![Bar chart showing sodium chloride (NaCl) removal.](image)

**Figure 7.** Sodium Chloride (NaCl) removal.
4. Conclusion

Successful fabrication for the ceramic membrane was achieved by using local raw materials. Almost complete removal for MR was obtained whereas 76% & 44% removal efficiency was obtained for MR and NaCl respectively. Even though less than 50% removal was achieved for the NaCl, merits for using ceramic membrane is the cost compared with using desalination process that involve high temperature plus the easy way to fabricate the produced ceramic membrane. Moreover, the used technique in this work could be used to study different composites with different ratios to achieve the pore size required for complete and successful separation.

It is recommended to perform more characterization for the pore size of the produce membrane, whether removal occurred due to filtration process or due to adsorption on the surface of the membrane. Special care is needed during fabrication of ceramic membrane such as purity and ratios of the raw materials, packing process, and sintering temperature. The best way is to start with low temperature and increase the temperature gradually until it reached the desirable temperature to avoid cracks in the product.

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