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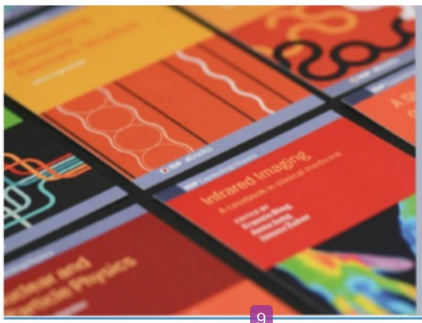

Performance of zeolite ceramic membrane synthesized by wet mixing method as methylene blue dye wastewater filter

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Abstract. Problem of pollution in water continues in Indonesia, with its manufacturing sector as biggest contributor to economic growth. One out of many technological solutions is post-treating industrial wastewater by membrane filtering technology. We presented a result of our fabrication of ceramic membrane made from zeolite with simple mixing and he. At 5% of (poring agent):(total weight), its permeability stays around 2.8 mD (10^{-14}m^2) with slight variance around it, attributed to the mixture being in far below percolating threshold. All our membranes achieve remarkable above 90% rejection rate of methylene blue as solute waste in water solvent.

1. Introduction

Problem of pollution in water continues in Indonesia. Growth of manufacturing sector, which is biggest contributor to economic growth, complicates this problem [1]. One particular problem of pollution in water is caused by textile industry usage of dye to colors its fabric. As such, it is necessary for dye wastewater to be treated before being let out to flow into the river. One possible treatment is by letting the wastewater through some selective membrane that acts, by size exclusion, as filter for the dyeing particle and molecule while letting water flow through it [2].

In this paper we presented our result on fabrication of ceramic membrane made from zeolite. Ceramic membrane is chosen because of its robustness and stability both against physical as well as chemical damage compared to other type of membrane (e.g polymer membrane) [6]. For the intended application, ceramic membrane, after it becomes clogged, also easier to clean by firing it in high temperature. While the constituent material, zeolite, is chosen because of it being “molecular sieve” hence having large surface contact area [3].

2. Methods

We fabricated our ceramics membrane by mixing ground zeolite powder with sol of suspended PEG4000 material in water as poring agent. The homogeneous mixture further pressed into the green body before being calcinated at 700°C for 10 hours. The resulting ceramic membrane then were tested

to measure its performance and its permeability, by letting test fluid flow through it as well as performing Vis-NIR spectrometry on the resulting filtrate.

Synthesis of poring agent begin by mixing PEG4000 powder and water in ratio of 2 : 5. The mixture further stirred to obtain a homogenous mixture of sol of suspended PEG4000 in water. This sol is to be used as poring agent to add more pores into our membrane to increase its permeability [7]. Synthesis of the membrane begin with grinding small zeolite chunks into powder and sieve the resulting powder to reduce variation in powder size. Varying amount of sol (3.5, 4, 4.5, 5, 5.5 gram) then added into 30g of ground zeolite powder. Mixture of zeolite powder and sol further mixed to obtain a homogenous mixture. The homogenous mixture then pressed to obtain the green body of the ceramic membrane. In the final step, green bodies are calcinated at 700°C to obtain zeolite ceramic membranes [8].

The membranes are tested by measuring its permeability and its rejection rate of pollutant inside test fluid. The test fluid itself is produced by adding 1g of methylene blue (MB) powder into 20L of water. The testing of permeability is based on Darcy's Law with pressure difference induced by hydrostatic pressure of 1.8kPa [4], can be seen in Figure 1.

$$Q = \kappa \frac{A(p_b - p_a)}{\mu L} = \kappa \frac{A\rho g \Delta h}{\mu L} \tag{1}$$

$$\kappa = \frac{\mu L}{A\rho g \Delta h} Q \tag{2}$$

Q: Debit flow (mL/h)
κ: permeability (mD = 10^{-14} m^2)
μ: water viscosity
A: membrane cross-section area
L: membrane thickness
$\rho g \Delta h$: hydrostatic pressure

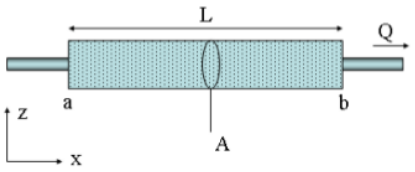

Figure 1. Darcy's law membrane model [4]

Vis-NIR spectroscopy further performed on the filtrate to measure the rejection rate of MB as pollutant by means of Beer-Lambert law.

$$c \propto A$$

$$\eta := \left(1 - \frac{c}{c_0}\right) = \left(1 - \frac{A}{A_0}\right) = \left(1 - \frac{\log T}{\log T_0}\right) \tag{3}$$

η: Efficiency /rejection rate
c: MB concentration
A/A_0: absorption of light/absorption of light by control
T/T_0: transmission of light/transmission of light by control

3. Result and Discussion

We obtained permeability of 2.73-2.82 mD ($10^{-14}m^2$) for PEG4000 fraction of 3.23-4.98wt%. Figure 2 shows plot of permeability against weight fraction of poring agent (PEG4000) inside green body.The plot shows the expected linear relationship between poring agent fraction and permeability. The poring agent contributes to permeability by making more pores inside the membranes. The more pores inside the membrane, the easier the test fluid to pass through the membrane, hence the increasing permeability [3]. Fitting the plot with line, will give an intercept about 2.59mD. Comparing the intercept value with slope of +0.04, shows a small variance albeit measurable linear relationship. This small variance may be attributed to, at 5% PEG4000 weight fraction, the porosity being much lower than the percolation threshold. Because of this, there's no end-to-end connection between the two sides of the membranes. The permeability further dominated by the bulk permeability of zeolite membrane instead of the permeability of the pores, hence the small variation of permeability [9,11].

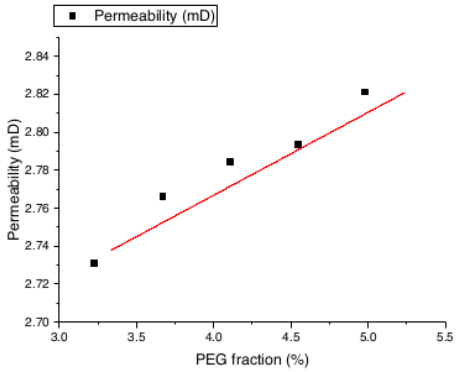

Figure 2. Plot of permeability vs PEG4000 weight fraction

Using Beer-Lambert law with Vis-NIR spectroscopy data, we obtain the rejection rate of our filter. All of our filters achieve a remarkable above 90% rejection rate. However, upon closer inspection of the data, the rejection rate doesn't vary monotonically with variable parameter, PEG4000 weight fraction. We have predicted that higher poring agent ratio would reduce rejection rate as it increases permeability, but the data does not show such is the case. It may be attributed to that the rejection rate has achieves a saturating value, which is the rejection rate of the bulk. Because the much lower porosity than the percolation threshold, as previously stated, fluid flows through the bulk instead of pores most of the time, i.e. the porosity isn't significant enough to affect membrane's bulk properties including permeability and rejection rate (Figure 3). In the case of permeability there's still small effect measured, however in case of rejection rate a random undetermined factor dominates. One particularly strong factor that may causes the non-monotonic or fluctuating rejection rate may be attributed to inhomogeneity. We may then consider 2 cases of inhomogeneous membrane with same porosity but different pores configuration [5]. In Figure 4, two different pores configurations having the same porosity have different rejection rate performance attributed to their pores configuration. Right configuration allows more fluid to pass through the bulk medium. As more fluid pass through the bulk medium more pollutant (MB) is rejected. However as long as the debit flow (coupled with pressure) is below a critical value, the two differing configurations don't affect its permeability as much as it affects its rejection rate (Table 1).

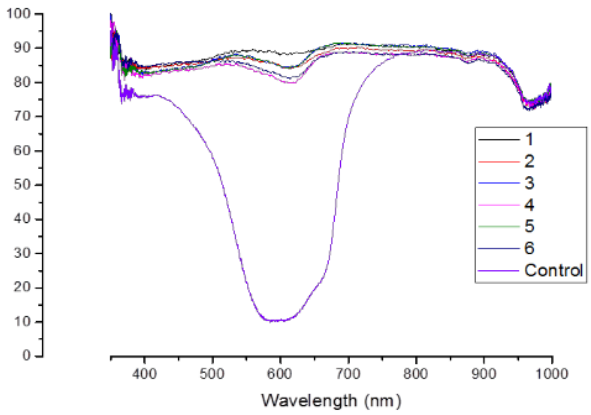

Figure 3. Vis-NIR spectroscopy result

Table 1. Rejection rate vs PEG4000 content

PEG4000 content (%)	Rejection rate η (%)
3.23	92.7
3.67	92.7
4.11	90.4
4.54	92.8
4.98	91.3

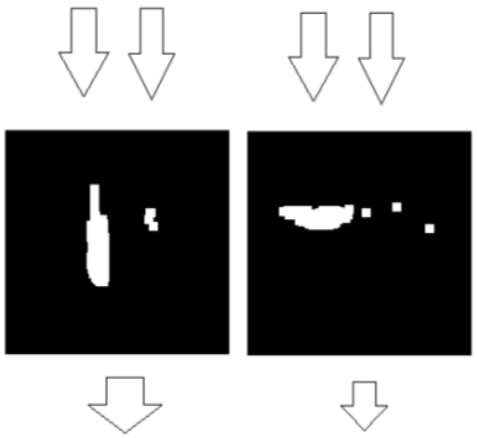

Figure 4. Left: pores form network of entry-to-exit ends; Right: pores form network perpendicular to that of entry-to-exit direction; Left filter has lower rejection rate to that of right filter, because most MB capture is performed by the bulk part

4. Conclusion

A zeolite ceramic membrane has been fabricated using wet mixing method (by sol). From the membranes we obtain permeability of 2.73-2.82 mD (10^{-14}m^2) for PEG4000 fraction of 3.23-4.98wt%. The filter rejection rate to methylene blue (as waste for test fluid) all achieve above 90%, hence its high possibility as feasible methylene blue based dye wastewater.

Acknowledgment

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