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APPLICATION OF KAOLIN-ZEOLITE CERAMIC FILTER MEMBRANE IN RECOVERING WASTE OIL INTO USABLE OIL

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ABSTRACT

Several membrane materials are continuously being developed to produce efficient and environmentally friendly filtration media. This study examines the application of kaolin-zeolite-based ceramic filter membranes in the process of refining used cooking oil into usable oil. The main materials used are kaolin, zeolite, and PVA as a binder. The variations in the kaolin:zeolite composition used are 50%:50%, 60%:40%, 70%:30%, 80%:20%, and 100%:0% with sintering times of 1, 2, and 3 hours at a temperature of 1000°C. Membrane characterization includes density, porosity, flux, morphology tests using a Scanning Electron Microscope (SEM), as well as the removal of Free Fatty Acids (FFA) in used cooking oil. The results showed that the composition of 50% kaolin and 50% zeolite with a sintering time of 3 hours produced the best performance with a density of 1.32 g/cm³, porosity of 56.00%, flux of 143.93 L/m².hour, and the removal capacity of ALB up to 70.6%, reducing the ALB content from 4.56% to 1.34% according to SNI standards. SEM analysis showed a macroporous structure (0.57–3.02 µm) that supports filtration, while the adsorptive properties of zeolite play a role in reducing polar compounds. These results prove that kaolin-zeolite ceramic membranes have the potential as an alternative technology in used cooking oil recovery.

Keywords: Ceramic Membrane, Kaolin, Zeolite, Sintering, Flux, Free Fatty Acid, SEM.

1. INTRODUCTION

1.1 Background

Used cooking oil is a household and industrial waste product largely generated from cooking activities. Repeated use of cooking oil at high temperatures can damage its chemical structure, producing harmful compounds such as free fatty acids (FFA) and other polar compounds that are carcinogenic (Nuryanti et al., 2020). If not managed properly, used cooking oil disposal can pollute the environment, particularly soil and water (Raharjo & Indarto, 2022). Therefore, efforts to recover used cooking oil into more usable oil are crucial to reduce negative environmental impacts while providing added value.

The used cooking oil recovery process using a kaolin-zeolite-based ceramic filter membrane not only aims to filter physical impurities but also reduces the levels of certain chemical compounds, such as free fatty acids. Kaolin serves as the main component of the ceramic, providing a strong structure, while zeolite has adsorption properties that enable it to bind polar compounds. The combination of these two materials creates a membrane with optimal performance for the purification process, while utilizing locally sourced, environmentally friendly, and affordable materials (Rahman et al., 2019).

The challenge in processing used cooking oil lies in its high contaminant content, such as polar compounds and free fatty acids. One widely developed solution is the application of ceramic membrane technology based on natural materials such as kaolin and zeolite. These materials are known for their good mechanical properties, high temperature stability, and superior adsorption capacity, enabling them to filter impurities and improve the quality of used cooking oil. The use of kaolin-zeolite based membranes also offers economic advantages due to the abundant and inexpensive raw materials (Santoso et al., 2020).

This study describes a technique for fabricating macroporous ceramic filters using a polymer template, such as polyurethane foam. In this process, a ceramic slurry is prepared beforehand and the polymer template is dipped into it. The template serves as a temporary structure to form the macropores in the ceramic filter. After drying and sintering, the polymer template is burned off, leaving the desired pore structure. A gypsum mold serves as a molding container during drying and to maintain the final shape of the ceramic membrane (Maspupah et al., 2020).

In this study, kaolin and zeolite were used as membranes to recover used cooking oil into quality oil that meets health standards.

2. RESEARCH METHODS

Research methodology

2.1 Research Place

This research was carried out at the Process Unit Laboratory, Pilot Plant Laboratory, Operations Unit laboratory of Lhokseumawe State Polytechnic.

2.2 Tools and Materials

2.2.1 Tools used

Equipment used in this research includes analytical scales, beaker glass, griding mill, measuring cup, spatula, pressure pump, measuring pipette, burette, furnace (oven), caliper, aluminum foil, hot plate stirrer, stirring rod, membrane mold, ball pipette, sieve.

2.2.2 Materials used

The ingredients used in this research include Used cooking oil, kaolin, zeolite, polyvinyl alcohol (PVA), sodium hydroxide solution (NaOH), phenolphthalein indicator (PP), 96% ethanol, 0.1 N KOH solution, and distilled water (aquades).

2.3 Experimental Treatment Design

2.3.1 Fixed Variables

- Sintering temperature: 1000°C
- Waste cooking oil volume: 1 L
- Membrane thickness: 0.6 cm (6 mm)

2.3.2 Independent Variables

- Kaolin: 50%, 60%, 70%, 80%, 100%
- Zeolite: 50%, 40%, 30%, 20%, 0%
- Sintering Time: 1, 2, and 3 hours

2.3.3 Dependent Variable

1. Density Test
2. Porosity Test
3. Flux Test
4. SEM Test
5. Free Fatty Acid Test

2.4 Experimental and Testing Procedures

2.4.1 Procedure for Making Kaolin-Zeolite-Based Ceramic Membranes

1. Prepare kaolin and zeolite in a specific ratio, for example, 50% kaolin and 50% zeolite. Weigh these ingredients using an analytical balance.
2. Mix the kaolin and zeolite in a glass beaker and stir until thoroughly combined using a spatula or manual stirrer.
3. Add a binder, such as silicone or Polyvinyl Alcohol (PVA), to make the paste denser and more malleable. Mix until homogeneous.
4. Pour the mixture into the ceramic membrane mold. Press the mixture evenly to avoid air bubbles in the membrane.
5. After drying, remove the membrane from the mold and clean the surface with distilled water to remove dust and any remaining unused material.
6. Transfer the dried membrane to a furnace and bake at 1000°C for 1, 2, and 3 hours to activate the pore structure and increase the membrane's durability.
7. After heating, allow the membrane to cool to room temperature.

Visually inspect the membrane surface to ensure there are no cracks or defects.
8. Store the finished ceramic membrane in a dry, tightly closed place to prevent moisture damage.
9. Analyze the finished membrane, including density, porosity, and filtration performance. Ensure the membrane meets the standards for use in removing free fatty acids from used cooking oil.

2.4.2 Free Fatty Acid/FFA Procedure
1. Take 5 mL of used cooking oil before and after filtration into a beaker.
2. Add 96% ethanol (10 mL) and 3 drops of phenolphthalein indicator.
3. Titrate with 0.1 N KOH solution until the pink color persists for 30 seconds.
4. Record the volume of KOH used.

3. RESULTS AND DISCUSSION

3.1 Research Results

Table 3.1 Data from Test Results and Observation Analysis

Kaolin-Zeolit Compotion	Sintering Time (hour)	Density (g/cm³)	Porosity (%)	Flux (L/m².jam)
50:50	1	1,37	26,80	68,87
	2	1,35	48,80	125,36
	3	1,32	56,00	143,93
60:40	1	1,19	35,59	91,47
	2	1,3	51,64	132,77
	3	1,11	42,99	110,47
70:30	1	1,26	35,48	91,18
	2	1,32	42,91	110,26
	3	1,23	28,14	72,33
80:20	1	1,3	30,77	79,12
	2	1,2	39,03	100,29
	3	1,24	55,54	142,73
100:0	1	1,19	34,02	87,40
	2	1,19	46,27	118,86
	3	1,17	31,30	80,45

3.2 Discussion

Research on the manufacture of kaolin-zeolite based filter membranes in used cooking oil recovery has been carried out testing including density tests, porosity test results, flux tests, SEM tests and Free Fatty Acid tests.

3.2.1 Density Test

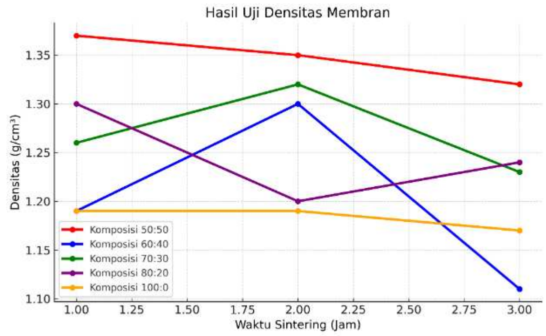


Figure 3.1 Graph of the Density Test Results on Membranes

From the density test data above, the highest density was recorded in the 50:50 composition with 1 hour sintering, while the lowest density was found in the 60:40 composition with 3 hours sintering at 1.11 g/cm³. This decrease in density indicates an increase in the number of cavities or pores in the membrane, which is closely related to the increase in porosity.

From the test results, it can be seen that the density value of the ceramic membrane decreases with increasing sintering time for most samples. For example, at a 50:50 composition, the density decreases from 1.37 g/cm³ at 1 hour of sintering to 1.32 g/cm³ at 3 hours of sintering. This indicates that the longer sintering process causes the material to become more porous due to the melting of fine particles that form cavities within the membrane structure.

3.2.2. Porosity Test

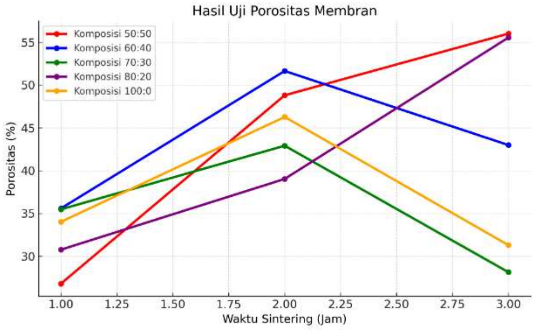


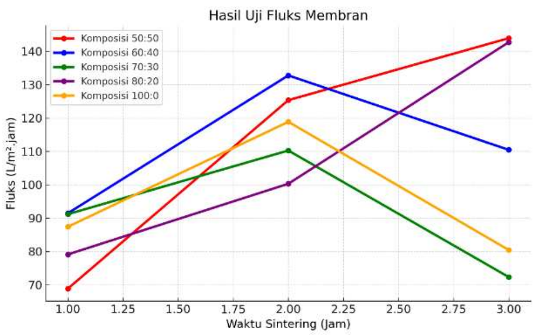
Figure 3.2 Graph of the Porosity Test Results on Membranes

From the porosity test data above, the 80:20 composition, sintered for 3 hours, showed the highest porosity at 55.54%, followed by the 50:50 composition, sintered for 3 hours, at 56.00%. This indicates that a combination of kaolin and zeolite with a balanced ratio, or a slight kaolin predominance, can produce a highly porous structure when sintered for longer periods.

However, not all increases in sintering time resulted in the highest porosity; some compositions fluctuated, such as the 70:30 composition, which actually decreased in the third sintering cycle (28.14%). This could be due to shrinkage of the material's microstructure due to over-sintering, which causes the pores to begin to close again.

Porosity is an important parameter in determining the membrane's ability to transmit fluids. Data shows that porosity tends to increase with increasing sintering time. At a 50:50 composition, porosity increased from 26.80% to 56.00% as sintering time increased from 1 to 3 hours.

3.2.3. Flux Test



Picture. 3.3 Graph of the Porosity Test Results on Membranes

From the flux test data above, the results show that flux increases with increasing porosity. The 50:50 composition with 3 hours of sintering had the highest flux of 143.93 L/m².h, in line with its highest porosity (56.00%). Meanwhile, the lowest flux occurred with the 50:50 composition with 1 hour of sintering, at 68.87 L/m².h.

This significant increase in flux indicates that membranes with high porosity allow for higher fluid flow rates. However, excessive porosity can also risk reducing the membrane's mechanical strength. Therefore, a balance between flux, porosity, and physical strength is an important consideration in selecting the optimal composition and sintering time..

The flux test describes the membrane's ability to flow water per unit area and time. Based on the results of a simulation calculation with a filtrate volume of 1 liter, a time of 4 hours, and an effective area of 0.00363 m², an initial flux of 68.87 L/m².h was obtained for a porosity of 26.80%. Next, the flux was calculated in proportion to each porosity value.

3.2.4 Free Fatty Acid Test

Table 3.2 ALB% Analysis Results Data and ALB% Allowance

Komposisi	ALB Awal (%)	ALB Sesudah (%)	Penyisihan ALB (%)
50:50%	4,56	1,34	70,6
60:40%	4,56	1,41	69,1
70:30%	4,56	1,56	65,8
80:20%	4,56	1,72	62,3
100:0%	4,56	1,88	58,8

Based on Table 4.2, the Free Fatty Acid (FFA) content of used cooking oil before the filtration process was recorded at 4.56%, which exceeds the quality threshold for edible cooking oil according to the Indonesian National Standard (SNI) (maximum 0.3–2.5%). This indicates that the used cooking oil was no longer suitable for consumption in its original state.

After undergoing filtration with a kaolin-zeolite-based ceramic membrane, the FFA content was successfully reduced to 1.34–1.88%, with a removal percentage of 58.8%–70.6%. The 50:50 composition variation yielded the best results with a removal rate of 70.6%, followed by 60:40 at 69.1%, 70:30 at 65.8%, 80:20 at 62.3%, and 100:0 at 58.8%.

This pattern indicates that the higher the zeolite content in the membrane, the better the FFA removal capacity. Zeolite acts as an adsorbent due to its surface area and pores that can capture free fatty acid molecules. A 50:50 composition demonstrates optimal performance due to the balance between the mechanical properties of kaolin (as a binder/ceramic former) and the adsorptive properties of zeolite.

These results align with research by Rahmawati et al. (2020), which found that the combination of kaolin and zeolite was effective in reducing FFA levels through filtration and adsorption mechanisms. Therefore, it can be concluded that varying the kaolin-zeolite composition significantly influences the membrane's effectiveness in removing FFA from used cooking oil.

4. CONCLUSION

4.1 Conclusion

Based on the results of the research that has been carried out, the following conclusions can be drawn:

1. The analysis of the effect of kaolin variations on the quality of kaolin-zeolite ceramic filter membranes showed that increasing the kaolin composition resulted in higher membrane density. A composition of 50% kaolin produced an optimum density of 1.32 g/cm³, indicating good mechanical strength to support membrane structural stability.
2. The analysis of the effect of zeolite variations on the quality of kaolin-zeolite ceramic filter membranes showed that adding zeolite increased the porosity and adsorption capacity of the membrane. A composition of 50% kaolin: 50% zeolite produced a porosity of 56.00% and supported high filtration performance in the refining process of used cooking oil.
3. The analysis of the effect of sintering time on membrane porosity characteristics showed a significant effect on pore formation. Sintering time of 3 hours gave the best results with a flux of 143.93 L/m².hour and was able to remove free fatty acids (FFA) up to 70.6%, reducing the FFA content from 4.56% to 1.34%, according to the SNI standard for cooking oil (<2.5%).

4.2 Suggestions

Based on the conclusions drawn, the following suggestions are given:

Future researchers hope to be able to Conduct additional parameter testing for further research, it is recommended to add testing on other parameters such as compressive strength, resistance to high temperatures.

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