Journal of Mechanical Engineering *Vol 21(3), 183-198, 2024*

Effect of Temperature on Zirconia Powder Synthesized from Amang Zirconium Oxychloride Precursor

*Nurul Asilah Md Amin[1], Istikamah Subuki[*1,2], Nurul Haiza Sapiee[1], Norin Zamiah Kassim Shaari[1]*

[1]School of Chemical Engineering, College of Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, MALAYSIA
[2]Industrial Metallurgy Research Group (IMReG), College of Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, MALAYSIA
[*]istikamah@uitm.edu.my

ABSTRACT

Silica (SiO2) is widely used in numerous applications, including ceramic membranes, and can be extracted from rice husks, which is an agricultural waste containing high amounts of silica. In fact, it is essential to reduce the vast amounts of discarded rice husks as open burning of the rice husk can have detrimental effects on the environment due to the release of hazardous pollutants. For the rice husk to be used, a suitable sintering temperature should be obtained to ensure the membrane synthesized has the desired porous structure. A high sintering temperature can affect the melting condition of the membrane's porous structure. While a low sintering temperature may not give the required porosity. Therefore, this study aims to evaluate the effect of sintering temperature on the pore size of the membrane fabricated from kaolin, polyvinyl alcohol, and rice husk ash. The rice husk was initially treated with hydrochloric acid (HCl) and heated at 900 °C to produce rice husk ash. The membranes fabricated were then sintered at 1000 °C to 1400 °C. X-ray fluorescence (XRF) results show that 71.36% of silica was successfully extracted from the rice husk. According to the XRD results, only two membranes sintered at 1300 °C and 1400 °C have crystallinity forms. Because the ceramic membrane contains a crystalline phase, it was determined that 1300 °C is an appropriate temperature for sintering. It also has a low absorption percentage (88.89%) and the desired porosity (69.93%).

Keywords: *Rice husk; Silica; Membrane; Sintering; Porosity*

ISSN 1823-5514, eISSN 2550-164X

https://doi.org/10.24191/jmeche.v21i3.27353

Received for review: 2023-10-16
Accepted for publication: 2023-12-08
Published: 2024-09-15

Introduction

The content and composition of rice husks from different areas may differ [1]. Generally, it has about 36% - 40% cellulose, 12% - 19% hemicellulose, 20% lignin, and 12% ash in rice husks, with 80% - 90% of the ash being silica [2]. The open burning of rice husks produces carbon dioxide (CO_2) and releases hazardous pollutants into the environment [3]. It has been demonstrated that treating rice husk with hydrochloric acid (HCl) during silica extraction increases silica purity. The result shows 97.64 wt% of silica (SiO2) from treated rice husk with HCl, which was the highest purity of silica compared with using other acids [4]. Amorphous silica and crystalline silica were formed from treated or untreated rice husk, with amorphous silica obtained by heating at 550 °C - 800 °C and crystalline silica at 900 °C - 1000 °C [5].

The use of a ceramic membrane in the filtration process was preferable compared to a polymeric membrane because the polymeric membrane has random chain scission, an unzipping depolymerization reaction, and disintegrates at higher temperatures [6]. All materials have unique characteristics, such as good chemical resistance (filter of organic solvents or hostile fluids like acids, bases, and oxidants), moderate thermal resistance (working temperatures of up to several hundred degrees), and enough mechanical strength (allowing pressures of around 10 bar - 100 bar) [7]. Low maintenance requirements, fouling resistance, and high-temperature stability help these systems have lower lifecycle costs [8]. The formulation of the ceramic membrane contains rice husk silica, which presents a possibility for low-cost ceramic membrane manufacture [9].

According to earlier studies, when temperatures rise, the crystalline silica membrane's porosity size rises [9]. Increasing the membrane sintering temperature makes the porous size of the membrane increase. However, increasing the sintering temperature to 1500 °C and above melts the membrane [9]. Which can close the pores of the membrane. Therefore, examining the membrane characterization and minimum porosity was required. For the membrane, 40% porosity and above was required for the ceramic membrane to minimize the pressure drop [9]. This study has proposed the fabrication of membranes by mixing the rice husk ash (RHA), kaolin, and polyvinyl alcohol (PVA) by setting the suitable sintering temperature range in observing the porosity of the membrane and the correlation between absorption percentage with porosity. Therefore, the objective of this study was to study the porosity of the membrane at different sintering temperatures. The study explored the potential for changing waste to wealth. Porosity was an important factor in ceramic membrane performance. Hence, this study will contribute useful findings for the feasibility of ceramic membrane application using rice husk.

Methodology

Synthesis of the rice husk ash (RHA)

In this study, the rice husk was utilised for membrane fabrication. Rice husk weighing 5000 g (obtained from Berkat Padi Sdn. Bhd.) was soaked and washed with tap water and then dried in the oven for 24 hours at 110 °C. The drying of rice husk was to remove the water content in the rice husk that can influence the concentration of acid used. With a constant stirring at 650 rpm, the rice husk was soaked in 1 M hydrochloric acid (HCl) at 60 °C for 2 hours. Then, the rice husk was washed and soaked with tap water until the pH was between 7 - 8. The rice husk was dried in the oven for 24 hours at 110 °C [5]. Lastly, the rice husk was heated in the furnace at 900 °C for 2 hours to obtain rice husk ash (RHA). The heating rate of the furnace was set to increase at a rate of 5 °C/min [5]. The RHA composition was checked by using X-ray fluorescence (XRF) (model: Epsilon3-XL).

Preparation of the membrane

For membrane fabrication, the RHA was mixed with kaolin and polyvinyl alcohol (PVA) as binders according to the weight compositions as shown in Table 1.

Table 1: Composition of each component used for membrane fabrication

Components	Weight, %
Rice husk ash (RHA)	38.46
Kaolin	38.46
Polyvinyl alcohol (PVA)	23.08

Figure 1 depicts the overall fabrication process. The distilled water was added to the mixture of RHA, kaolin, and PVA. Then, the mixture was heated at 80 °C at 700 rpm for three hours to mix evenly. The white slurry was then dried in the oven for two hours at 100 °C to remove the water content. The shape of the membrane was fabricated in a rectangular shape [9]-[10]. Five membrane filtration samples were sintered using the high-temperature furnace (Thermoline/F46110CM). The five samples were heated at different temperatures. The sample was named rice husk membrane (RHM) and each sample was labelled as RHM-1, RHM-2, RHM-3, RHM-4, and RHM-5 with heating temperatures of 1000 °C, 1100 °C, 1200 °C, 1300 °C, and 1400 °C, respectively. The heating rate was 3 °C/min for 2 hours [9]. The membrane phase after the sintering process has been checked using X-ray diffraction (XRD) (model: Rigaku / Ultima IV). The scan speed used for analysis was 3 ^{o}/min and the scan range was from 20^o to 80^o with a scan-axis of 2θ. The porosity of the fabricated membrane was analyzed by using a Mercury porosimeter (model: Micromeritic/Autopore Iv 9510). The swelling percentage of the membrane was conducted by soaking the membrane in distilled water for 24 hours. Equation (1) was used to calculate the swelling percentage. The surface morphology of the membranes fabricated was analysed using a scanning electron microscope (SEM) (model: Hitachi TM3030).

$$\text{Swelling percentage (\%)} = \frac{\text{Final weight (wet)} - \text{Initial weight(dry)}}{\text{Initial weight(dry)}} \times 100 \quad (1)$$

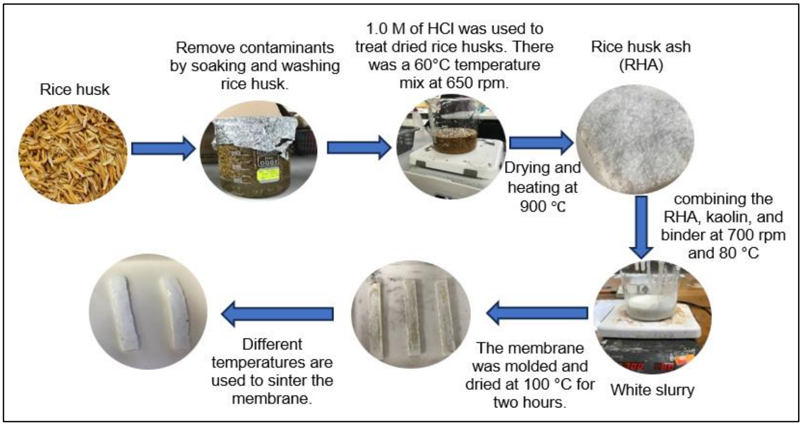

Figure 1: The flow method in synthesizing the ceramic membrane

Results and Discussion

Rice husk ash composition characterisation

From the heating process of the treated rice husk with hydrochloric acid (HCl) at 1 M, the white powder known as rice husk ash (RHA) was obtained, as shown in Figure 2. According to Shen *et al*. [1], the result of the thermochemical conversion of rice husk was rice husk ash. The conversion process conditions, including pyrolysis, gasification, or combustion, determine the physical-chemical properties of the finished products, including the ash. According to Zainal *et al*. [5], heating the rice husk at temperatures ranging between 900 °C to 1300 °C can produce crystalline silica powder. Besides removing carbonaceous wastes, pyrolysis at temperatures above 800 °C also causes the creation of crystalline silica [1]. Based on a study by Younesi *et al*. [11], crystalline rice husk ash was obtained when heating the treated rice husk at 700 °C. This study used a temperature of 900 °C to heat the treated rice husk, resulting in a white powder, as shown in Figure 2. Table 2 shows the composition of RHA.

From the XRF analysis as shown in Table 2, 71.36% of silica (SiO_2) was obtained as the main component in RHA. Another study gained 98.94% of silica by heating the rice husk at 800 °C and 99.5% at 600 °C, respectively [12]-[13]. The difference in silica composition obtained in the rice husk was due to the type of rice, the region's soil, and its climate [1].

Figure 2: The rice husk ash (RHA) synthesized

Table 2: Composition of RHA from XRD data

RHA contents	Composition, %
SiO_2	71.3637
P_2O_5	0.3537
K_2O	0.0537
CaO	0.1943
TiO_2	0.0030
MnO	0.0070
Fe_2O_3	0.0237
CuO	0.0040
ZnO	0.0037
SrO	0.0020
Ag_2O	0.0020

Effect of sintering temperature on membrane phase

Based on Figure 3, the XRD pattern for RHM-1, RHM-2, and RHM-3 was similar which indicates that the membrane phase was in the amorphous phase. Meanwhile, membrane RHM-5 (1400 °C) and RHM-4 (1300 °C) have crystalline phases because it has a broadening peak. The peak was $2\theta = 22.2°$ for RHM-4 and $2\theta = 22.14°$ for RHM-5. It shows that increasing sintering temperature changes the amorphous membrane to a crystalline membrane. This finding was similar to that obtained by Nayak *et al.* [14], where the phase of the amorphous membrane has turned into a crystalline membrane at a sintering temperature of 1300 °C. In contrast, it was different from another study which indicated that the crystalline phase can be observed at a sintering temperature of 1150 °C and 1000 °C [15]-[16]. This discrepancy may be due to the number of component mixtures used in fabricating the membrane which triggers altered values of crystalline sintering temperature.

As a result, it demonstrates that not all sintering temperatures can yield crystalline membranes; instead, it may rely on the composition utilized to make the membranes. RHA, kaolin, and PVA were combined for this study, and each component may have its sintering temperature to enter the crystalline phase. For the kaolin membrane sintered at a temperature of 800 °C, the membrane showed partial crystallization and the crystallization form increased with increasing sintering temperature [17]. From XRD analysis, the crude powder of kaolin has shown that the crystalline phase was in the form of quartz [18]. The sintering temperature of the kaolin membrane at 1100 °C was changed the crystalline form of the membrane to the mullite phase [18]. It shows that the kaolin has crystallinity form.

With the siloxane (Si-O-Si) and silanol (Si-OH) groups in rice straw ash, PVA will form a potent secondary bond [19]. This is because PVA has a hydroxyl group in each iteration of the polymer, which causes PVA and silica to have a three-dimensional structure [19]. The PVA will function as a binder between the particles, enhancing the silica membrane's green strength [19]. The polymer PVA was partly crystalline [20]. Previous studies have demonstrated that as rice husk concentrations were increased, the percentage crystallinity of membrane-containing PVA decreased [21]. According to other studies, the peak gets sharper when more cellulose nanocrystals from rice husks are present. When the number of cellulose nanocrystals at 1.0 percent in the PVA-containing membrane increases, the peak then decreases [22]. As a result, it demonstrates how the mixing of each component's amount affects the membrane's crystallinity.

The crystalline membrane has greater hardness resistance [5]. There were a few types of crystalline forms, for this membrane most of the crystalline formed was in cristobalite and low quartz. Most phase crystalline for RHM-5 was in the cristobalite phase and for RHM-4 phase of crystalline was in cristobalite and quartz. Other researchers, the type of crystalline that has been found in rice husk membranes were mullite and quartz [15]. According to Olivia *et al.* [23], PVA and cellulose can affect the crystallinity of the membrane.

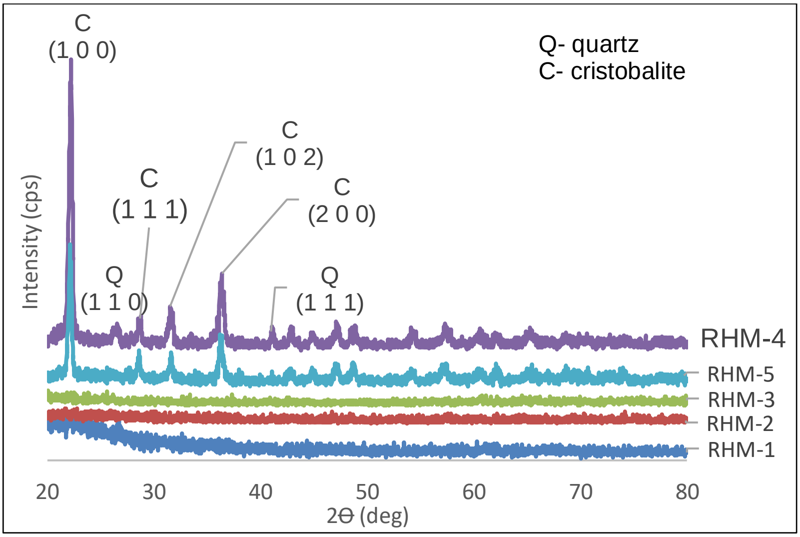

Figure 3: XRD analysis on membrane fabricated

Effect of sintering temperature on membrane porosity

The porosity of crystalline rice husk membrane (RHM) after sintering at different temperatures was analyzed, and Figure 4 shows the membrane pore size distribution. Membrane RHM-1 (1000 °C) has the highest mercury intrusion compared to RHM-5 (1400 °C). This result shows similarities with previous research from Hubadillah *et al.* [9], where the sintering temperature increased as the intensity of the mercury intrusion reduced.

According to Bat-Amgalan *et al.* [24], the pore size distribution of the supports sintered at low temperatures (950 °C - 1000 °C) was smaller than that of the supports sintered at high temperatures (1050 °C – 1150 °C). When the sintering temperature increases, it produces larger pores size. When looking at the results obtained from this study for the pore diameter range between 6 nm - 5000 nm, as shown in Figure 4, most of the RHM-1 (1000 °C) pore size was in the range of 6 nm to 1300 nm while the RHM-5 (1400 °C) in the range of 200 nm to 3000 nm. This shows that the membrane was sintered at a temperature range of 1000 °C to 1200 °C, showing a small diameter of the pore. Meanwhile, when the sintering temperature of the membrane increased from 1300 °C to 1400 °C, it showed a big pore size diameter. The particle diffusion that results from the silica melting phenomenon at higher temperatures was the cause of this [9]. This result shows that increasing sintering temperature increases the pore size, similar to that found by Hubadillah *et al.* [9]. In addition, large pores may be produced because rising sintering temperature encourages particle development. However, tiny pores may overlap as large pores develop [24].

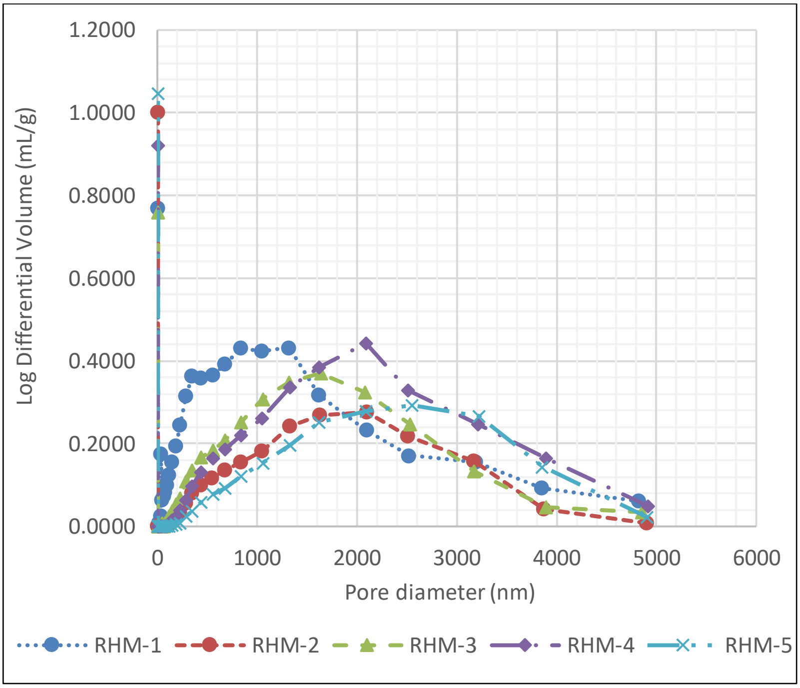

Figure 4: Membrane pore size distribution

From Figure 5, all membranes have more than 40% porosity. According to Hubadillah *et al*. [9], porous ceramic membranes have a significantly higher porosity above 40% to reduce pressure loss. High pure water flux was created by the ceramic membrane with a porosity more significant than 40%, which was desired for water treatment applications [9]. The application of the membrane defines the porosity needed for a particular membrane. The majority of rice husk membranes were intended for use in water filtration because of their high porosity.

Moreover, the membrane's pore size can be changed to target particular pollutants, including bacteria, viruses, and organic particles [25]. The smallest pore size of the ceramic membrane fabricated in this study was larger than 6 nm. Therefore, the fabricated membrane was suitable for microfiltration because the desired pore size for this type of membrane filtration was more than 50 nm [26]. Although the pore size range required for ultrafiltration membrane was between 2 nm to 50 nm [26], the membrane fabricated in this study was not suitable for this type of membrane because it has a high pore size which was greater than 50 nm, and the smallest pore size which was 6 nm. Therefore, the sintering temperature used to fabricate the membrane in this study has the potential for water microfiltration applications due to porosity exceeding 40% and pore size larger than 50 nm.

Examining membrane ability in terms of porosity alone was insufficient because various other aspects need to be considered for ceramic membrane characteristics. Based on Figure 5 the porosity membrane RHM-1 was higher than other membranes. It has 75.25% porosity. High porosity and high-strength ceramics were required; however, the high porosity yields poor-strength materials [27].

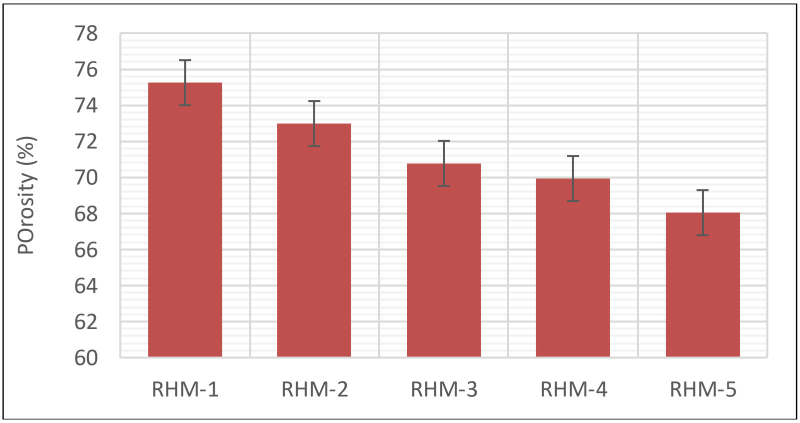

Figure 5: Membrane porosity percentage (%)

Effect of sintering temperature on swelling percentage

The swelling or absorption percentage was the percentage of water uptake in the membrane. From Figure 6, the membrane that has low swelling percentages was RHM-5 (88.24%). Meanwhile, RHM-1 has the highest swelling percentages compared to other membranes. From previous research, the swelling percentage of the membrane was decreased by increasing the composition of rice husk ash in the membrane [28].

Based on the swelling percentage of membrane sintered at different temperatures, elevating the sintering temperature was frequently found to reduce the absorption of water. When the sintering temperature was at 1200 °C and above, a drastic reduction in water absorption occurred. The result from Prasad *et al.* [15], has shown that the water absorption percentage drastically reduces when the temperature increases from 1100 °C to 1150 °C. From this study, the water or swelling percentage of membrane RHM-1 was the highest, and the percentage was decreased when the sintering temperature of the membrane was increased.

The absorption of the membrane correlates well with the porosity of the membrane. The volume of gaps has a noticeable impact by increasing the amount of water absorbed [29]. In samples with pores, water absorption increased approximately linearly with time [29]. From the previous study on decreasing membrane porosity, the water absorption also decreases [30]. By comparing results from porosity and swelling percentage, the membranes followed the effect of porosity on water absorption, as the membrane's porosity decreases and water absorption decreases. However, a high absorption percentage reduces the pure water flux of the membrane [31]. Therefore, a membrane with high water absorption can reduce water flux on the membrane where high water flux was required for ceramic membrane, but it also requires high selectivity.

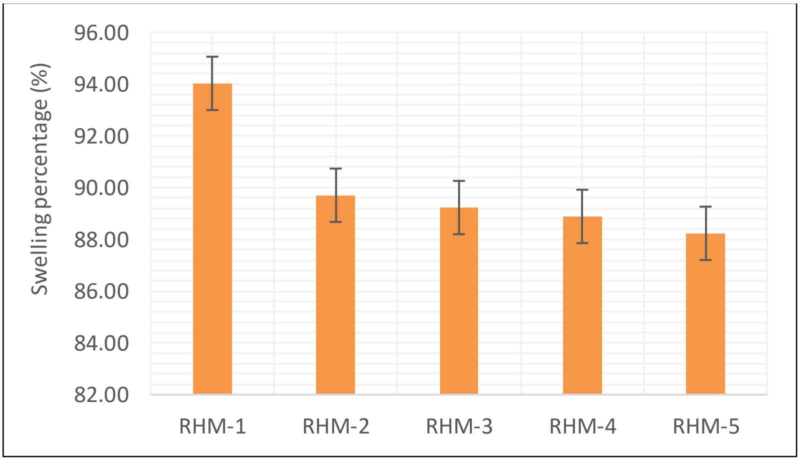

Figure 6: Membrane swelling percentage (%)

Lastly, Figure 5 shows that RHM-1 had the highest porosity of any membrane, with a value of 75.25%. In comparison to other membranes, RHM-1 exhibited the highest absorption percentage as a result. This demonstrates that the water flux of the RHM-1 may be minimal, and RHM-5 may have a significant water flux.

Analysis of membrane surface morphology

The surface morphology of the fabricated membranes was analyzed, and Figure 7 presents the results for the five different membranes fabricated. The pores on the membrane were densely arranged, however, the distribution of the pores was not arranged consistently. These atoms or ions were grouped in a regular three-dimensional structure with a distinct and ideal lattice [9]. Furthermore, the structure of silica produced was in quartz form [9]. Quartz was a significant crystalline phase in almost all triaxial whiteware compositions [15].

Regarding the membrane pore size, the pore size of each membrane was not uniform. At 500x magnification, the porosity on the membrane part was not visible. The membrane consists of open and closed pores. Open pores link to the material's surface, whereas closed pores are pockets of vacuum space within a bulk material. Open pores are essential for molecule separation, adsorption, and catalysis research. Thermal insulators and structural applications are the most common applications for closed pores [32]. However, for RHM-5 (1400 °C), the porous structure was seen as more significant compared to the other sintering temperature. The pore structure was more open and clearly seen. This was probably because the PVA used to hold the rice husk ash structure was evaporating due to the current high temperature during the sintering process. For the RHM-1 (1000 °C) membrane, smaller pore sizes were observed, as shown in Figure 7. The result can be related to pore size distribution (Figure 4), where the pore size increased due to increasing sintering temperatures [9].

For SEM images at 2500x magnification (Figure 8), the state of the porous membrane was more clearly visible. The pore size for the RHM-5 (1400 °C) membrane was seen as large. As there were no finger-like voids, the membranes were considered symmetrical [9]. The pores coalesced at a high sintering temperature, resulting in the formation of these large pores. At high temperatures, some of the pores may have vanished [33]. Therefore, high sintering temperatures caused the pore size to increase, which was attributed to small pores interacting with one another [33].

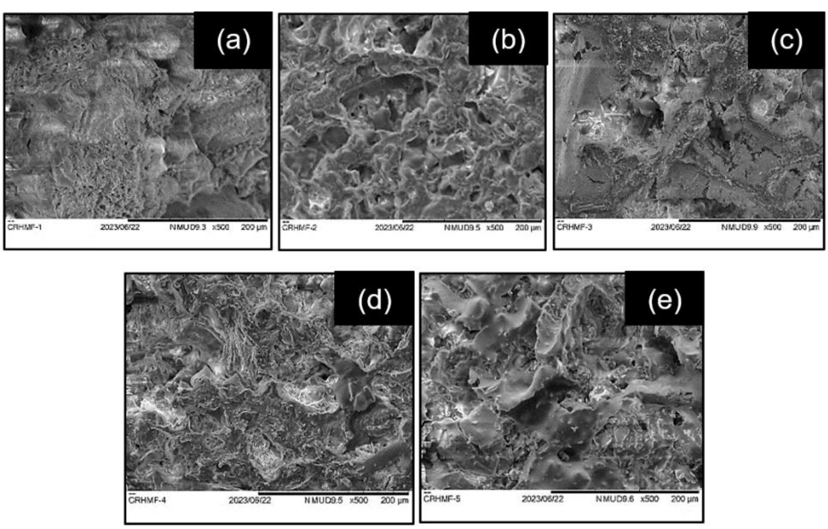

Figure 7: Membrane surface SEM image at 500x magnification; (a) 1000 °C, (b) 1100 °C, (c) 1200 °C, (d) 1300 °C, and (e) 1400 °C

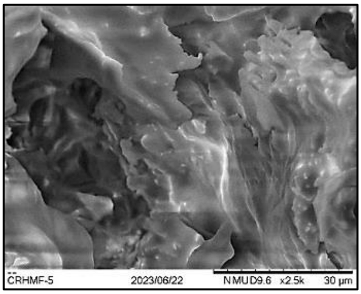

Figure 8: Membrane surface SEM image at 2500x magnification for RHM-5

On the other hand, the performance of the fabricated membrane has yet to be tested in a particular application. For any application, it has not been confirmed that the range of membrane pore sizes achieved in this study can be acceptable. Each application may have its own desired porosity and pore sizes required for effectiveness and it includes the value of swelling percentage. Therefore, further analysis should be tested on the membrane fabricated to look at its potential in specific applications.

Conclusions

In this study, about 71.36% of silica has been obtained from rice husk ash. From the study, it can be concluded that the membrane's pore size increases by increasing the sintering temperature. Besides that, all of the membrane's porosity exceeded 40%, and the swelling percentage of the membrane was reduced by increasing the sintering temperature. The sintering temperature of the membrane fabricated the membrane 1300 °C has been observed as a suitable temperature for sintering the ceramic membrane due to having a crystalline phase. Moreover, the RHM-4 has a desired porosity requirement for the ceramic membrane, which was 69.93%, where this value exceeded the minimum porosity required for ceramic membrane filtration. The swelling percentage for RHM-4 was 88.89%. Although other membranes' swelling percentage was lower, increasing the sintering temperature up to 1400 °C can affect the mechanical strength. Moreover, the pore size of membrane RHM-4 was also suitable for microfiltration membranes.

Contributions of Authors

The authors confirm the equal contribution in each part of this work. All authors reviewed and approved the final version of this work.

Funding

This work received no specific grant from any funding agency.

Conflict of Interests

All authors declared that they have no conflicts of interest.

Acknowledgement

The authors would like to thank the College of Engineering, Universiti Teknologi MARA for the facilities.

References

[1] Y. Shen, P. Zhao, and Q. Shao, "Porous silica and carbon derived materials from rice husk pyrolysis char," *Microporous and Mesoporous Materials*, vol. 188. pp. 46-76, 2014. https://doi.org/10.1016/j.micromeso.2014.01.005

[2] H. S. Hafid, F. N. Omar, J. Zhu, and M. Wakisaka, "Enhanced crystallinity and thermal properties of cellulose from rice husk using acid hydrolysis treatment," *Carbohydr Polym*, vol. 260, pp. 1-12, 2021. https://doi.org/10.1016/j.carbpol.2021.117789

[3] Muhammad Abdulhadi, "Removal of Zn (II) ions Using Activated Carbon Prepared from Rice Husk Treated with Sodium Hydroxide," pp. 1-33, 2011, [Online]. Available: https://utpedia.utp.edu.my/id/eprint/311/

[4] K. A. Matori, A. Wahab, and S. Aziz, "Producing Amorphous White Silica from Rice Husk," *MASAUM Journal of Basic and Applied Sciences*, vol. 1, no. 3, pp. 1-5, 2009. [Online]. Available: https://www.researchgate.net/publication/236839923

[5] N. S. Zainal, Z. Mohamad, M. S. Mustapa, N. A. Badarulzaman, and A. Z. Zulkifli, "The ability of crystalline and amorphous silica from rice husk ash to perform quality hardness for ceramic water filtration membrane," *International Journal of Integrated Engineering*, vol. 11, no. 5, pp. 229-235, 2019. https://doi.org/10.30880/ijie.2019.11.05.029

[6] A. Islam, B. Praveen Chakkravarthy Raghupathy, M. V. Sivakumaran, and A. Kumar Keshri, "Ceramic membrane for water filtration: Addressing the various concerns at once," *Chemical Engineering*

Journal, vol. 446, no. 4, pp. 137-386, 2022. https://doi.org/10.1016/j.cej.2022.137386.

[7] M. Messaoudi, M. Douma, N. Tijani, and L. Messaoudi, "Study of the permeability of tubular mineral membranes: application to wastewater treatment," *Heliyon*, vol. 7, no. 4, pp. 1-10, 2021. https://doi.org/10.1016/j.heliyon.2021.e06837.

[8] A. Abdullayev, M. F. Bekheet, D. A. H. Hanaor, and A. Gurlo, "Materials and applications for low-cost ceramic membranes," *Membranes*, vol. 9, no. 9. no. 105, pp. 1-31 2019. https://doi.org/10.3390/membranes9090105.

[9] S. K. Hubadillah *et al.*, "Fabrication of low cost, green silica based ceramic hollow fibre membrane prepared from waste rice husk for water filtration application," *Ceramic International*, vol. 44, no. 9, pp. 10498-10509, 2018. https://doi.org/10.1016/j.ceramint.2018.03.067.

[10] N. S. I. Chik, N. Z. B. Kassim Shaari, N. A. B. Ramlee, and M. R. Bin Abdul Manaf, "Extraction of Silica from Rice Husk Ash and Its Effect on the Properties of the Integral Membrane," *ASM Science Journal*, vol. 17, pp. 1–13, 2022. https://doi.org/10.32802/asmscj.2022.1035.

[11] H. Younesi and Z. Ghasemi, "Preparation and characterization of nanozeolite NaA from rice husk at room temperature without organic additives," *Journal of Nanomaterials*, vol. 2011, pp. 1-8, 2011. https://doi.org/10.1155/2011/858961.

[12] I. J. Fernandes *et al.*, "Characterization of silica produced from rice husk ash: Comparison of purification and processing methods," in *Materials Research*, Universidade Federal de Sao Carlos, vol. 20, pp. 519-525, 2017. https://doi.org/10.1590/1980-5373-mr-2016-1043.

[13] W. H. Kwan and Y. S. Wong, "Acid leached rice husk ash (ARHA) in concrete: A review," *Materials Science for Energy Technologies*, vol. 3, pp. 501-507, 2020. https://doi.org/10.1016/j.mset.2020.05.001.

[14] J. P. Nayak and J. Bera, "Effect of sintering temperature on phase-formation behavior and Mechanical properties of silica ceramics prepared from rice husk ash," *Phase Transitions*, vol. 82, no. 12, pp. 879-888, 2009. https://doi.org/10.1080/01411590903471564.

[15] C. S. Prasad, K. N. Maiti, and R. Venugopal, "Effect of substitution of quartz by rice husk ash and silica fume on the properties of whiteware compositions," *Ceram Int*, vol. 29, no. 8, pp. 907-914, 2003. https://doi.org/ 10.1016/S0272-8842(03)00035-X.

[16] K. A. Matori *et al.*, "Effect of Temperature on Ceramic from Rice Husk Ash," *International Journal of Basic & Applied Sciences IJBAS*, vol 9, no. 9, pp. 1-8, 2009. [Online]. Available: https://www.researchgate.net/publication/265526358

[17] S. Zaiou *et al.*, "Sintering of anorthite based ceramics prepared from kaolin DD2 and calcite," *Ceramica*, vol. 62, no. 364, pp. 317-322, 2016. https://doi.org/10.1590/0366-69132016623642015.

[18] F. de Santana Magalhães, E. de Paulo Ferreira, L. P. Bessa, C. S. D. Costa, M. G. A. Vieira, and M. H. M. Reis, "Fabrication of kaolin hollow fibre membranes for bacteria removal," *Processing and Application of Ceramics*, vol. 14, no. 4, pp. 303-313, 2020. https://doi.org/10.2298/PAC2004303M.

[19] S. Wahyuningsih, A. H. Ramelan, D. T. Wardoyo, S. Ichsan, and Y. R. Kristiawan, "Influence of Polyvinyl Alcohol (PVA) Addition on Silica Membrane Performance Prepared from Rice Straw," in *IOP Conference Series: Materials Science and Engineering*, Institute of Physics Publishing, vol. 333, no. 1, pp 1-10, Apr. 2018. https://doi.org/10.1088/1757-899X/333/1/012085.

[20] O. N. Tretinnikov and S. A. Zagorskaya, "Determination Of The Degree Of Crystallinity Of Poly(Vinyl Alcohol) By Ftir Spectroscopy." *Journal of Applied Spectroscopy,* Vol. 79, pp. 521-526, 2012. https://link.springer.com/article/10.1007/s10812-012-9634-y

[21] E. M. Abdel Bary, Y. A. Soliman, A. Fekri, and A. N. Harmal, "Aging of novel membranes made of PVA and cellulose nanocrystals extracted from Egyptian rice husk manufactured by compression moulding process," *International Journal of Environmental Studies*, vol. 75, no. 5, pp. 750-762, 2018. https://doi.org/10.1080/00207233.2018.1456862.

[22] Y. Hussein *et al.*, "Plant nanocellulose and its composite hydrogel membranes-based polyvinyl alcohol/hyaluronic acid for biomedical applications: Extraction, characterization, and in vitro bioevaluation," *Journal of Applied Pharmaceutical Science,* vol. 11, no. 1, pp. 049-060. 2021. https://doi.org/10.7324/JAPS.2021.110105.

[23] J. P. de Oliveira *et al.*, "Cellulose fibers extracted from rice and oat husks and their application in hydrogel," *Food Chemistry,* vol. 221, pp. 153-160, 2017. https://doi.org/10.1016/j.foodchem.2016.10.048.

[24] M. Bat-Amgalan, N. Miyamoto, N. Kano, G. Yunden, and H. J. Kim, "Preparation and Characterization of Low-Cost Ceramic Membrane Coated with Chitosan: Application to the Ultrafine Filtration of Cr(VI)," *Membranes (Basel)*, vol. 12, no. 9, pp. 1-15, 2022. https://doi.org/10.3390/membranes12090835.

[25] L. Hamoudi, D. E. Akretche, A. Hadadi, A. Amrane, and L. Mouni, "Comparative Study of Ceramic Membranes Developed on Different Algerian Natural Clays for Industrial-Effluent Filtration," *Minerals*, vol. 13, no. 2, pp. 1-19, 2023. https://doi.org/10.3390/min13020273.

[26] M. Qiu, X. Chen, Y. Fan, and W. Xing, "1.11 Ceramic Membranes," in *Comprehensive Membrane Science and Engineering*, Elsevier, 2017, pp. 270-297. https://doi.org/10.1016/b978-0-12-409547-2.12243-7.

[27] L. Wang *et al.*, "High-strength porous alumina ceramics prepared from stable wet foams," *Journal of Advanced Ceramics,* vol. 10, no. 4, pp. 852-859, 2021. https://doi.org/10.1007/s40145-021-0479-9.

[28] N. Zamiah Kassim Shaari, A. Syasya Mohd Shukri, and J. Jai, "Effects of Rice Husk Ash Loadings on the Tapioca Starch-based Film Composite," *International Transaction Journal of Engineering*, vol. 12, no. 9, pp. 1-9, 2021. https://doi.org/10.14456/ITJEMAST.2021.185.
[29] C. Castro López *et al.*, "Effect of porosity and hydrostatic pressure on water absorption in a semicrystalline fluoropolymer," *Journal of Materials Science*, vol 51, pp. 3750-3761, 2016. https://doi.org/10.1007/s10853-015-9692-7
[30] F. T. Owoeye, A. P. Azodo, and S. B. Udo, "Performance evaluation of ceramics microfiltration membrane for water treatment," *Journal of Mechanical Engineering and Sciences*, vol. 7, no. 1, pp. 1115-1126, 2014. https://doi.org/10.15282/jmes.7.2014.10.0108.
[31] Y. O. Raji, M. H. Dzarfan Othman, N. A. H. S. Md Nordin, and A. F. Ismail, "Preparation and Characterization of Bentonite - Based Ceramic Hollow Fiber Membrane," *Journal of Membrane Science and Research*, vol. 7, no. 2, pp. 85-94, 2021. https://doi.org/10.22079/JMSR.2020.121902.1346.
[32] G.Q. Lu and X. S. Zhao, "Nanoporous materials : science and engineering." *Series of Chemical Engineering*, Vol. 4, pp. 1-192, 2004. https://doi.org/10.1142/p181
[33] M. Mohamed Bazin, N. Ahmad, and Y. Nakamura, "Preparation of porous ceramic membranes from Sayong ball clay," *Journal of Asian Ceramic Societies*, vol. 7, no. 4, pp. 417-425, 2019. https://doi.org/10.1080/21870764.2019.1658339.