

Research Article

Utilization of Rice Husk, an Abundant and Inexpensive Biomass in Porous Ceramic Membrane Preparation: A Crucial Role of Firing Temperature

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The influence of firing temperature on characteristics and bacterial filtration of the porous ceramic membrane prepared from rice husk (20 wt%) and kaolin has been investigated. As firing temperatures increased from 900 to 1100°C, the compressive strength of membrane increased from 555.3 N/cm² to 2992.3 N/cm², whereas the porosity decreased from 49.4 to 30.2% due to structural condensation and mullite formation. The condensation caused pore contraction that finally improved bacterial removal efficiency from ~90% to 99%. The results suggested that the porous ceramic membrane prepared from rice husk and kaolin should be fired at ~1050°C to attain both strength and filtration efficiency.

1. Introduction

Lack of access to clean water has been a critical issue in many parts of the world, particularly in poor and developing countries [1], and it is the cause of water-borne diseases such as diarrhea. Paradoxically, abundant water resources including lake, river, or stream are available in those countries; however, these sources are usually unsafe and required the application of treatment measures. Various water treatment technologies including boiling, chlorination, sand filtration, reverse osmosis filtration, porous ceramic membrane filtration, coagulation, and adsorption are considerably suitable for water purification. Among those technologies, water filtration using porous ceramic membrane (PCM) has emerged as a great candidate for water treatment [2–4]. The advantages of this method are its easy operation, easy cleaning, high durability, and affordable cost. PCMs can be fabricated in desired shapes from locally available clays and biomass [5]. Biomass sources like sawdust, wood flake, and

flour have been widely used as burning additives to prepare PCMs [3, 6–10]. In a research conducted by Zereffa and Bekalo, PCMs fabricated from clay with 5% grog and 15% sawdust and fired at 1000°C can remove 97.5% *E. coli* and 87.9% turbidity [11]. PCM fabricated by Bulatao and Micheal using the similar method but fired at 800°C can eliminate 96.5% *E. coli* and 82.1% turbidity, respectively [8]. Obviously, PCMs have a great potential application in the field of water filtration for bacterial removal.

Rice husk is an abundant agricultural waste with an estimation of 1.5×10^8 tonnes/year [12], but only a small fraction has been practically utilized [13]. It is estimated that 90% of rice is burned in open air or discharged into river and oceans, which may cause significant environmental consequences [14, 15]. Rice husk is a high energy source with a heating value of 15 MJ/kg [15], which has high potential for thermal energy production. Traditionally, rice husk is used as thermal energy for household cooking [16, 17], but nowadays, it is slowly replaced by gas and electric energy. Novel

technologies such as direct combustion, fast pyrolysis, alcohol production, and gasification have been investigated to create the added value to rice husk [12–15]. However, the deployment of those novel technologies is still limited; therefore, more practical measures that can create the added value to rice husk are required.

Recently, the utilization of rice husk to produce PCMs has attracted significant attention [18–23]; however, only few works reported on the fabrication of membranes for water filtration. Soppe et al. demonstrated that PCMs prepared from clay and rice husk can meet the requirement for household water filtration with >2-log reduction in *E. coli* concentration [21]. Some parameters such as rice husk/clay ratio, maximum kiln temperature, and rice husk particle size that may affect the filter’s performance were investigated. Porosity plays a significant role in controlling the filtration flow rate of PCMs. The porosity and pore size can be controlled by the rice husk/clay ratio and size of rice husk. Firing temperature has considerable influence on the characteristics and filtration performance of PCMs; however, this influence was investigated at firing temperatures $\leq 950^\circ\text{C}$ without elucidating the reason that may cause the increment in ceramic strength and filtration efficiency [21]. Previous investigation on the phase transition of ceramics from kaolin indicated that a major phase that contributes to the strength of ceramics is mullite. The formation of mullite phase begins at $\geq 900^\circ\text{C}$, but more noticeable at $\geq 1000^\circ\text{C}$ [24–26]. Therefore, it is necessary to study the phase transition of porous ceramics using rice husk as a pore-forming agent in a higher range of firing temperature. In this study, the critical effect of firing temperature on the characteristics and bacterial removal efficiency of PCMs prepared from kaolin and rice husk will be studied at a temperature from 900 to 1100°C .

2. Materials and Methods

2.1. Materials. Kaolin samples from Truc Thon (Hai Duong, Vietnam, Table 1) were provided by a local supplier. Rice husk was collected from a local source in Hai Duong, Vietnam. Kaolin samples were dried and ground to the particle size $\leq 63\ \mu\text{m}$ while rice husk was crushed until the size of $\leq 0.5\ \text{mm}$. Conventional agar and the strains of *E. coli* ATTC 25922 were purchased from Merck.

2.2. Porous Ceramic Membrane Preparation. In a typical PCM preparation, a desired amount of kaolin and rice husk (20–38 wt% in dry ceramic mixture including kaolin and rice husk) was mixed with water (approximately 27 wt% of dry ceramic mixture) and incubated in 48 h. The mixture was then filled in a steel cylindrical mold (60 mm diameter and 20 mm height) and pressed by a hydraulic press machine at about $500\ \text{kg}/\text{cm}^2$ to form filter disk samples. The prepared samples were dried at room temperature for 5 days and at 105°C for one day followed by firing in a furnace. Firing process was programmed in 3 steps involving the increase of temperature from room temperature to 200°C , then to 550°C , and to a firing temperature in a range of 900 to 1100°C at the ramping rate of $3^\circ\text{C}/\text{min}$; at the end of each step, an isothermal period

TABLE 1: Composition of a kaolin sample.

No.	Chemical composition	Kaolin (wt%)
1	SiO_2	63.88
2	Al_2O_3	25.2
3	MgO	1.58
4	K_2O	2.47
6	CaO	2.1
7	TiO_2	0.31
8	MnO	0.02
9	Fe_2O_3	2.63
10	Others	1.78

of 2 h was applied. Samples were finally cooled down to room temperature for further experiments.

2.3. Characterization. Porosity of porous ceramic membrane is determined by a saturated water method [27]. Dry filter sample is weighed, soaked in water for 24 hours, and taken out to check the water saturated mass.

$$P = \frac{(m_s - m_d)}{\rho_w V_F} \times 100\%, \quad (1)$$

$$V_m = \frac{(m_s - m_{iw})}{\rho_w}, \quad (2)$$

where P is porosity (%), m_s is weight of water-saturated membrane (g), m_d is weight of dried membrane (g), ρ_w is density of water ($1000\ \text{g}/\text{L}$), V_m is volume of membrane (L) that can be determined by equation (2), and m_{iw} is weight of ceramic membrane in water (g), which is determined by weighing filter underwater.

Ceramic membrane specimen with the size of the 60 mm in diameter and 20 mm thickness was used for compressive strength measurement. Cross-sectional surface of the specimen was ground smoothly then mounted on 300 kN compressive strength testing machine followed by applying a load slowly until specimen was broken. Compressive strength (N/cm^2) was calculated by dividing the load (N) to cross-sectional area (cm^2).

X-ray diffraction (XRD) patterns were collected on the XRD Panalytical Empyrean instrument Cu-K α source operating at a voltage of 45 kV, current of 40 mA, and scanning step of $0.017^\circ/\text{s}$ with a scanning range from 10 to 80° . The morphology of adsorbent was studied by scanning electron microscopy (SEM) using a Quanta 650 microscope. Nitrogen adsorption/desorption study was investigated using Micromeritics TriStar V6.07 A. Samples were first crushed into small bead and then degassed at 320°C for 5 h prior to analysis. FTIR spectra were collected on a Nicolet iS10 FTIR spectrometer (Thermo Fisher Scientific) using a KBr pellet method. TG-DTA were conducted on STA 409PC-NETZCH under air atmosphere from room temperature to 1000°C with the ramping rate of $10^\circ\text{C}/\text{min}$.

2.4. Bacterial Removal Efficiency. To evaluate the bacterial removal, water was spiked with *E. coli* ATCC suspension ($\sim 2.1 \times 10^4$ CFU/mL) and filtered through a ceramic disk with a constant flow. An aliquot (0.1 mL) of filtered water was taken and stretched over agar plates for cultivation at 37°C for 24–48 h to count for bacterial cells that remained. The bacterial removal efficiency was determined as a percentage of bacteria eliminated by filtration.

3. Results and Discussion

3.1. Porous Ceramic Membrane Characterization. The porosity of a ceramic membrane could be significantly affected by rice husk content used as a pore-forming agent; increasing rice husk would produce high porosity but the strength could be reduced. Thus, porosity and strength of a ceramic membrane should be compromised to maximize its performance. To investigate the effect of rice husk additives on the strength and porosity, the rice husk contents in dry ceramic mixture (including rice husk and kaolin) were varied from 20 to 38 wt% while the firing temperature was kept constant at 1000°C. As the rice husk contents increased from 20 to 35 wt%, the porosity increased from 45.5% to 56.6%, respectively (Figure 1(a)). To further understand the porous structure of membrane, nitrogen adsorption and desorption study was conducted and an adsorption/desorption isotherm of a ceramic membrane prepared with 20% rice husk and fired at 1000°C is shown in Figure 1(b). The isotherm has a hysteresis loop of type H3 according to IUPAC classification. This revealed that the ceramic membrane is associated with macropores [28]. The porosity of pore size in the range of 1.7–300 nm is only 0.025 cm³/g, and the surface area of membrane is 4.1 m²/g. Obviously, the pores with size < 300 nm account for a very small fraction in the total porosity of the membrane.

Undoubtedly, higher rice husk content helps improve the porosity of PCMs thanks to more space that rice husk created after being burned out. Higher porosity was expected to benefit water filtration flow rate, but, unfortunately, it caused significant reduction in the strength of the membrane. At rice husks contents ≥ 35 wt%, PCMs were broken apart while installing into the filter or during filtration. As the rice husks decreased to 25 wt%, PCMs can be installed into the filter; however, they have a potential of damage during maintenance, whereas PCMs with 20 wt% rice husks were steady without cracking during installation, filtration, and maintenance. The membranes can be removed for cleaning and reinstalled without damage; therefore, 20 wt% rice husks were selected to investigate the effect of firing temperature on the characteristics of PCMs. Accordingly, the variation in compressive strength as a function of firing temperature was investigated and the results are exhibited in Figure 2(a). It is clear that the compressive strength increased along with the elevation of firing temperature; however, the strength increment rate slightly varied at different temperature ranges. Strength inclined almost linearly from 555.3 N/cm² to 1597.0 N/cm² in the temperature range from 900°C to 1050°C, with a rate of 6.6 N/cm²·°C. Nevertheless, when the temperature elevated from 1050°C to 1100°C, the compressive strength increased to 2992.3 N/cm² with a

rate of 27.9 N/cm²·°C. This suggests that the possible ceramic phase transition occurred at $\geq 1050^\circ\text{C}$, particularly the formation of mullite phase that resulted in the improvement of compressive strength.

The phase transition due to the raising firing temperature was studied by X-ray diffraction, and results are shown in Figure 2(b). Peaks at 16.4°, 30.9°, 33.1°, 35.4°, and 40.8° corresponding to the diffraction of mullite phase (PDF 01-076-2578) are relatively weak intensity at 950°C but become profound at 1000°C and 1050°C. Previous works reported that mullite can be formed at $>900^\circ\text{C}$ [24–26] and have an important role in the development of mechanical strength in ceramics [29–31]. The XRD study revealed that the mullite phase was likely formed at temperatures $\geq 950^\circ\text{C}$. This results explain the reason why the compressive strength of ceramics increased more rapidly at a firing temperature range from 1050°C to 1100°C.

The phase transformation can be further asserted by TG-DTA as shown in Figure 3(a). Both kaolin and rice husk/kaolin showed weight loss at around 100°C which reflects the moisture vaporization. The kaolin sample had a weight loss at 450°C–600°C with an endothermic peak at 519°C, which related to the removal of structural water in kaolin to form metakaolin and the condensation of $\equiv\text{Si-OH}$ to form siloxane [32]. Whereas the rice husk/kaolin mixture showed thermal decomposition at lower temperature from 230°C to 600°C, besides an endothermic peak at 519°C, it had two exothermic peaks at 346°C and 453°C corresponding to the release of volatile matter in rice husk and the rice husk combustion [33]. Particularly, an exothermic peak was found at 993°C in both samples that belongs to the formation of mullite crystal [32]. The thermal decomposition of rice husk and condensation of $\equiv\text{Si-OH}$ are also observed on the FTIR spectra of the sample (Figure 3(b)). A remarkable vibration at 2930 cm⁻¹, 3621 cm⁻¹, and 3697 cm⁻¹ corresponding to C-H bonding and -OH group in rice husk disappeared after firing at 1050°C indicating the burning out of rice husk. Moreover, a peak at 915 cm⁻¹ was significantly reduced suggesting the condensation of $\equiv\text{Si-OH}$ groups.

Besides the mullite formation, the PCM structure becomes more condensed at higher firing temperature reflecting via the increase in the diameter shrinkage (Figure 4(a)) and reduction in the porosity (Figure 4(b)). The diameter shrinkage increased almost linearly with temperature, from 1.7% to 9.2%, as temperature increased from 900°C to 1100°C, whereas the porosity decreased slowly at a temperature from 900 to 950°C, 49.4% at 900°C to 48.1% at 950°C, but more rapidly at temperature $> 950^\circ\text{C}$, which remained only 30.2% at 1100°C. The reduction in porosity can also be observed clearly in SEM images. SEM images of PCMs fired at 950°C and 1050°C (Figure 5) showed that a porous structure is formed by the connection of primary particles (quartz and mullite) and voids created by burning off rice husk. Large pores 20–30 μm are found in the sample fired at 950°C but hardly observed at $\geq 1050^\circ\text{C}$. Obviously, the improvement of porous ceramic strength along with increasing firing temperature is related to structural condensation and mullite formation. Structural condensation caused noticeable diameter shrinkage and reduction in porosity. These may hinder the

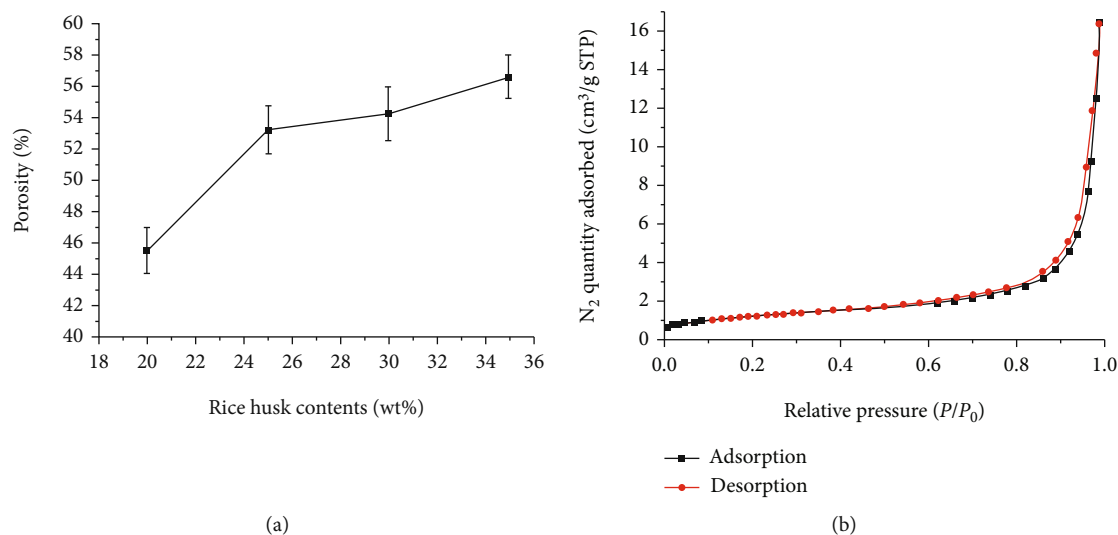


FIGURE 1: Variation in the porosity of PCMs with rice husk content (wt%) in a ceramic mixture fired at 1000°C (a) and nitrogen adsorption/desorption isotherm of a PCM sample prepared from a ceramic mixture containing 20 wt% rice husk fired at 1000°C (b).

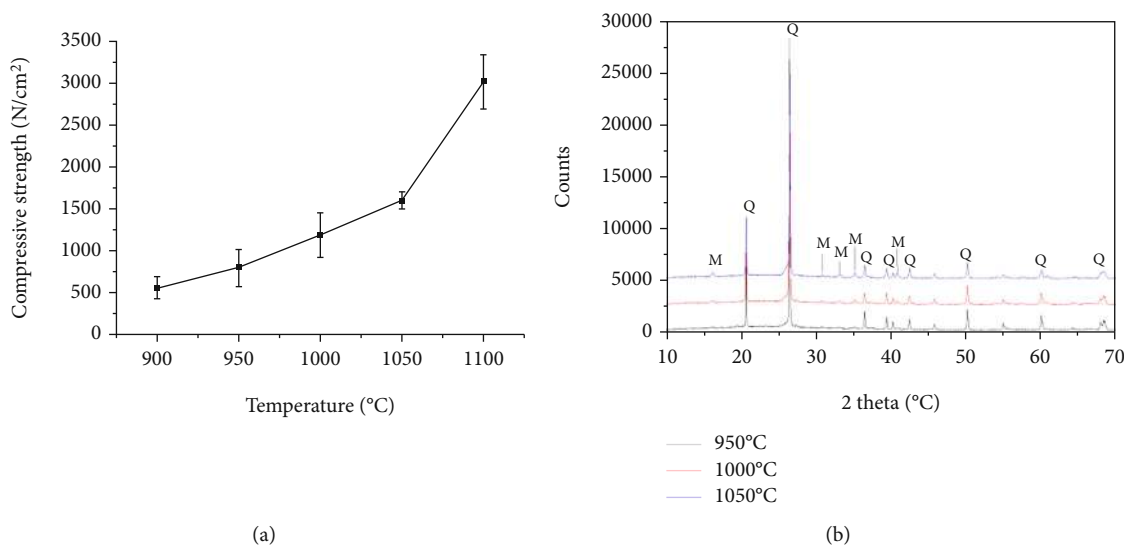


FIGURE 2: Variation in the compressive strength (a) and X-ray diffraction (b) of PCMs with firing temperatures.

membrane production at large scale and decrease the filtration ability of the membrane; therefore, firing temperature should be optimized to minimize the effect of structural variation on the membrane's performance.

3.2. Bacterial Removal Efficiency. It is apparent as shown in Figure 6 that the removal efficiency of membrane was significantly improved with increasing firing temperature. *E. coli* removal efficiency reached >90% as PCMs were sintered at 900°C and increased to >99% at 1050°C. This removal efficiency is comparable with that of ceramic membrane prepared from clay, laterite, and rice husk and fired at 800–900°C in a previous study, which reached a removal efficiency of approximately 99% [21]. To further evaluate its stability, a

membrane fired at 1050°C has been used for the filtration of water spiked with *E. coli* ($\sim 2.1 \times 10^4$ CFU/mL) for 3 days (at least 6 h/day). Interestingly, no significant drop in the bacterial removal efficiency was observed after 3 working days. Obviously, there is a strong relation between porous structure of a PCM and its removal efficiency. Probably, the porous structure was more condensed and large pores created by burned rice husk were constructed to smaller sizes at higher firing temperature resulting in the improvement of *E. coli* removal efficiency. According to the guideline reference level recommended by the World Health Organization, a household water treatment technology that eliminates 2 log of bacteria (equal to 99% removal) can meet the requirement for drinking water at a protective level [34]. Thus, for PCMs

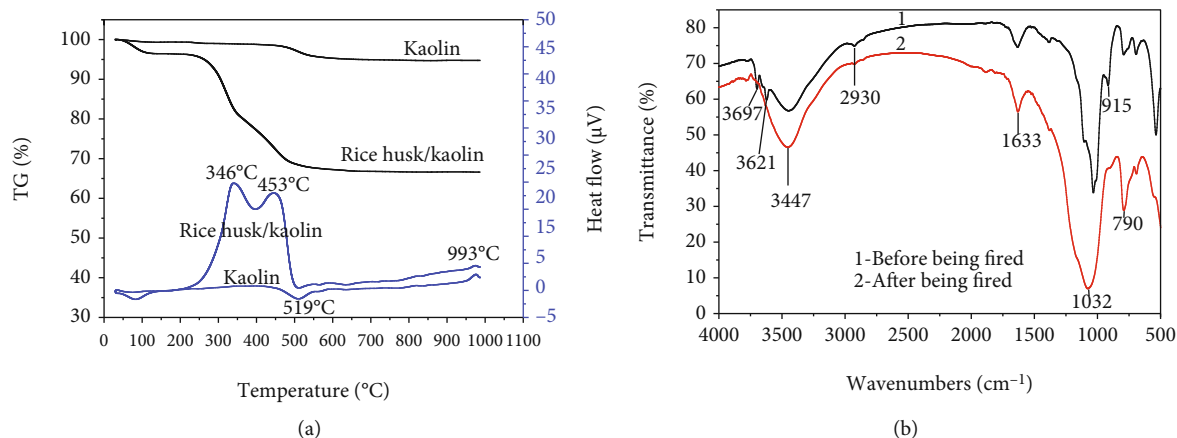


FIGURE 3: TGA and DTA profiles of kaolin and ceramic mixture containing 25% rice husk (a) and FTIR spectra of ceramic samples before and after being fired (b).

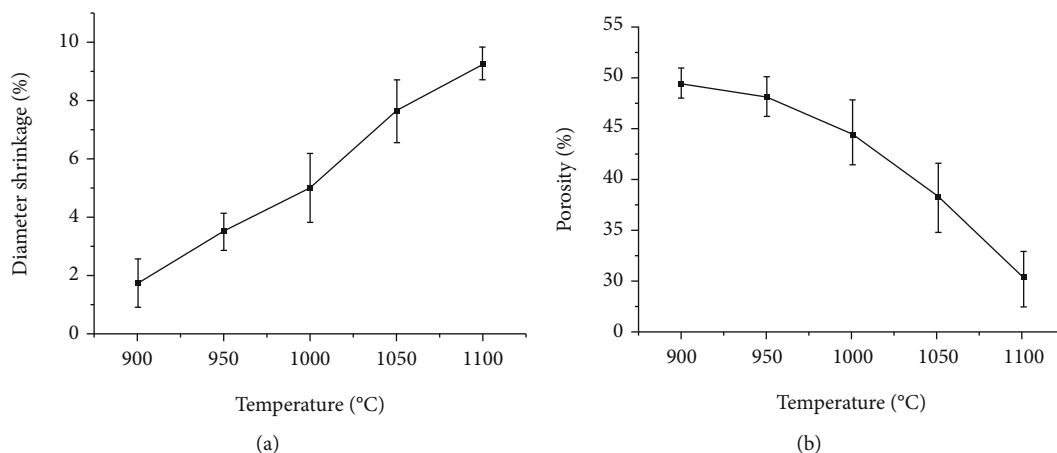


FIGURE 4: Variation in diameter (a) and porosity (b) of PCMs as a function of firing temperature.

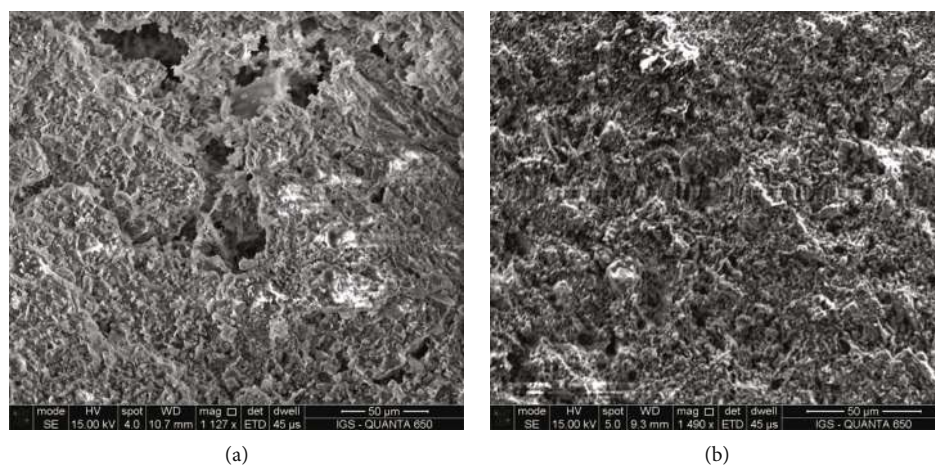


FIGURE 5: SEM images of samples fired at 950°C (a) and 1050°C (b).

prepared from kaolin and 20 wt% rice husk (particle size ≤ 0.5 mm), only membranes fired at $\geq 1050^\circ\text{C}$ can meet the requirement for a household water treatment technology.

This confirmed the importance of firing temperature, which influences not only the strength but also the filtration efficiency of PCMs.

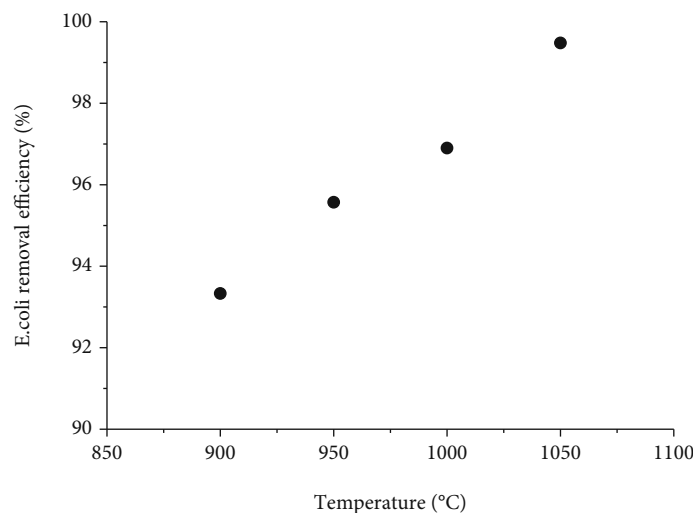


FIGURE 6: Bacterial removal efficiency by PCMs fired at different temperatures.

4. Conclusion

This study demonstrated an important role of firing temperature in controlling characteristics and filtration performance of PCMs prepared from rice husk and kaolin. Increasing firing temperatures from 900°C to 1100°C generated stronger ceramic membranes with a compressive strength increased from 555.3 to 2992.3 N/cm², most likely thanks to the formation of mullite phase. The firing temperature increase caused the ceramic structure condensation, and as a consequence, its porosity decreased from 49.4 to 30.3%, while the diameter of ceramic membrane decreased from 1.7 to 9.2%, respectively. The pore size decrease due to firing temperature increment improved bacterial removal efficiency, which was ~90% at 900°C then reached 99% at 1050°C. The study revealed that the optimum firing temperature should be ~1050°C to balance between strength and filtration efficiency.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

Acknowledgments

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