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Study on the Effect of Sulfuric Acid Addition on the Porosity and Permeability Properties of Nata De Soya Porous Membranes

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Abstract

Porous nata de soya membranes are a promising filtration material due to their good mechanical and chemical properties. However, further modifications are necessary to enhance their filtration performance, one of which involves the addition of sulfuric acid. Therefore, the objective of this study is to investigate the effect of sulfuric acid addition on the porosity and permeability properties of porous nata de soya membranes. Porosity tests using Scanning Electron Microscope (SEM) were conducted to analyze the microstructure of the nata de soya membranes. The SEM characterization results provide detailed images of the membrane's pore structure, including size, distribution, surface area, and pore density. Additionally, permeability analysis was performed using Darcy's law. The results indicate that the addition of sulfuric acid affects the porosity and permeability of the membranes. Increasing the sulfuric acid concentration from 15% to 25% resulted in a decrease in porosity from 51% to 47%. The pore size increased from 0.35 μm at 15% to 0.86 μm at 25%. Furthermore, the surface area and pore density decreased with the increasing concentration of sulfuric acid, indicating a reduction in membrane effectiveness in filtration processes. The membrane's permeability decreased drastically from 2.55287 x 10^{-9} m² at 15% to 2.12739 x 10^{-12} m² at 25%, demonstrating that sulfuric acid causes pore closure and structural damage to the membrane, reducing its ability to allow fluid to pass through. The implications of this research suggest that porous nata de soya membranes are effective for microfiltration of bacteria (such as *E. coli*) and wastewater filtration.

Keywords: Membrane, Nata de Soya, Permeability, Porosity, Sulfuric Acid

1. Introduction

Porous membranes play a crucial role in various industrial applications, including separation, filtration, and purification[1-3]. The demand for effective and efficient membranes has driven continuous research to discover and develop new materials with superior performance. One material that has recently gained attention in membrane technology is nata de soya, a biotechnology product derived from soybean fermentation[4]. The researchers chose tofu and tempeh waste to be processed into a product called nata de soya membrane for several important reasons. First, tofu and tempeh waste is available in large quantities as a byproduct of the soybean processing industry, making its utilization beneficial for reducing waste issues and adding value. Second, tofu and tempeh waste still contains significant nutrients, such as protein, carbohydrates, and fats, which can be used as raw materials for producing nata de soya[5]. Third, processing tofu and tempeh waste into a useful product helps reduce negative environmental impacts, decrease pollution, and improve industrial waste management.

Fourth, transforming waste into economically valuable products like nata de soya membrane creates opportuneities for new business development, job creation, and increased community income. Fifth, the production process of nata de soya from tofu and tempeh waste is relatively simple and does not require advanced technology, making it easily applicable for small and medium enterprises. Lastly, nata de soya is a good source of natural fiber and can be used in various applications, including the food, pharmaceutical, and cosmetic industries [3,5]. For these reasons, utilizing tofu and tempeh waste to produce nata de soya membranes not only contributes to better waste management but also supports economic and environmental sustainability.

Nata de soya is known for its unique structure and good mechanical properties, making it a promising candidate for the fabrication of porous membranes [6,7]. The pores in the nata de soya porous membrane are measured for several important reasons related to the membrane's performance and applications. First, pore size and distribution significantly affect the filtration properties and permeability of the membrane. By measuring the pores, researchers can optimize the membrane's performance for specific applications, such as particle separation, contaminant removal, or liquid filtration. Additionally, pore measurement helps ensure the consistency and quality of the produced membrane, as membranes with uniform pores tend to have more predictable and reliable performance. The mechanical properties, such as the membrane's strength and flexibility, are also influenced by its pores. Therefore, by knowing the pore size, researchers can adjust the manufacturing process to achieve the desired mechanical properties. Various applications require specific pore sizes; for example, in water treatment, smaller pores may be necessary to filter bacteria. According to I. Gudavadze and E.-L. Florin (2022) , bacteria typically range in size from 0.2 to 2 micrometers (µm). Therefore, membranes with pore sizes around 0.2 µm or smaller are effective for filtering bacteria.

Porosity and permeability are two critical properties that determine the efficiency and effectiveness of membranes in various applications[9,10]. Porosity refers to the amount and size of pores within the membrane, while permeability relates to the membrane's ability to allow fluid flow through it [11,12]. These properties are interrelated and are vital for the membrane's performance in specific applications such as liquid separation, air filtration, and water purification [13,14]. One method that has proven effective in modifying material properties is the addition of certain chemicals during the manufacturing process [14]. Sulfuric acid, for example, has been used in various studies to modify the material's structure and enhance its functional properties [10,15]. In the context of nata de soya membranes, the addition of sulfuric acid is expected to alter the pore structure and improve filtration performance. Various studies have been conducted to explore the influence of chemicals on membrane properties. Li et al. [16] investigated the use of a multi-effect membrane distillation (MEMD) process to concentrate dilute sulfuric acid solutions. The study results showed that the performance ratio (PR) values in MEMD were significantly higher than those in traditional membrane distillation processes and other modifications.

Masturi et al. [17] examined the fabrication of ceramic membranes from Muntilan clay and sand using a sol-gel method with a titania coating on the surface. Polyethylene glycol (PEG) was used as a pore-forming agent. The findings indicated that the addition of sand increased membrane permeability by creating larger pores. Kesava and Dinakaran [18] reported enhanced ion exchange capacity and proton conductivity in membranes doped with sulfuric acid. Gao et al. [19] found that acid modification on cellulose acetate membranes not only improved pore structure but also increased water flow, which is an important indicator of membrane permeability. Building on the knowledge gained from previous research on the effects of chemicals on membrane properties, researchers are interested in exploring the impact of adding sulfuric acid on the permeability and porosity properties of nata de soya porous membranes.

2. Method

This study employs an experimental method with varying concentrations of sulfuric acid.

2.1 Fabrication of Nata De Soya Membrane

The first stage of the research involves mixing tofu and tempe waste with coconut water, which is used as a source of minerals and vitamins for the bacteria *Acetobacter xylinum*. The waste was taken from a tofu and tempe processing factory called "Rumah Inovasi Tahu-Tempe Sekarsari" (RITSS) located in Patemon village, Gunungpati district, Semarang city, Central Java. The mixture is then heated, filtered, and stirred in a stainless steel pan with a diameter of 40 cm. Subsequently, 2% granulated sugar, food-grade ammonium sulfate (chemical formula; $(NH_4)_2SO_4$, CAS 7783-20-2, brand YR-020, nitrogen (N) content 21%, purity 99%) at a concentration of 2 grams per liter and up to 2% table vinegar were added to condition the starter to an acidic pH.

The process of making the starter and nata de soya begins by heating the mixture of tofu and tempe waste with coconut water until boiling for 10 minutes, then cooling it to below 40°C for 1 hour. The cooled solution is then added with 10% liquid *Acetobacter xylinum* culture, stirred well, and poured into a sterile plastic tray to a depth of 4 cm. The tray is covered with clean newspaper and incubated for 8-14 days at room temperature. After incubation, the formed nata de soya samples, with a thickness of approximately 1.25-1.50 cm, are cleaned of the mucus layer and undergo cold pressing at room temperature with a pressure of 0.018-0.031 atm, followed by hot pressing at a temperature of 120-180°C with a pressure of 3-24.5 atm for 30 seconds. The samples, which have turned into rigid sheets of off-white color, were then immersed in a sulfuric acid solution (chemical formula; H_2SO_4, Specification; purity 98%, Impurities; Chloride (Cl) maximum 10 ppm, Nitrate (NO_3) maximum 5 ppm, Iron (Fe) maximum 50 ppm, Lead (Pb) maximum 50 ppm, Liquid form) with varying sulfuric acid concentrations of 15%, 20%, and 25%. A total of 1000 mL of the solution was used for immersion for 30 minutes. Afterward, the drying process is carried out using a hairdryer at 60°C for 15 until 20 minutes.

2.2 Porosity Test

Furthermore, characterization was conducted to determine the porosity of the porous membrane using Scanning Electron Microscopy (SEM). The electron microscope used was the Phenom Pro-X series. Morphological characterization of the membrane using SEM allows for a detailed examination of the morphology of the nata de soya membrane. This includes observation of membrane pores, pore size distribution, and surface structure.

The SEM images were further processed into three-dimensional models using OriginPro 2024 software. The modeling of SEM images into three-dimensional pore structures aims to provide a more detailed and accurate visualization of the membrane's microscopic structure [19]. Porosity measurements for each sample were analyzed using OriginPro, while average pore size calculations were performed using Microsoft Excel 2007. Additionally, the calculation of pore area and density was analyzed using Microsoft Paint and Microsoft Excel 2007. The total area of pores was determined by counting the number of pixels (as indicated in MS Paint) within the pore regions and converting them into area units based on the image resolution. Subsequently, pore density was calculated by dividing the total pore image area by the total area of the image [20].

$$\text{Pore Density} = \frac{\text{Total Pore Image Area}}{\text{Total Image Area}} \tag{1}$$

2.3 Permeability Test

The permeability of nata de soya membrane is measured using Darcy's law, which yields varying permeability values depending on the concentration of sulfuric acid and the duration of immersion. Darcy's law, as proposed by Matyka et al. (2008), explains the relationship between volumetric flow rate (Q) and certain parameters within a filtration system. It is expressed as $Q = kA\Delta P/(\mu\Delta L)$, where k is the filter permeability, A is the filter surface area, ΔP is the pressure difference, μ is the fluid viscosity, and ΔL is the filter thickness. Utilizing Darcy's law to determine the permeability of nata de soya membrane involves several structured and measurable steps.

Firstly, the nata de soya membrane is installed in a filtration system that allows water to flow through it. The volumetric flow rate (Q) of water passing through the membrane is measured using a water pressure measuring device (*press gauge*), expressed in cubic meters per second (m^3/s). The pressure difference (ΔP) between the upstream and downstream sides of the membrane is expressed in Pascals (Pa). The effective area of the membrane involved in the filtration process is measured in square meters (m^2), while the thickness of the nata de soya membrane (ΔL) is measured in meters (m). The dynamic viscosity of the fluid at the operating temperature of 20°C is 1.002 mPa·s.

Once all the aforementioned parameters are known, Darcy's law is applied to calculate the permeability of the nata de soya membrane. The calculated permeability results are then utilized to analyze the performance of the nata de soya membrane. Permeability testing is conducted to determine the extent to which the nata de soya sample is capable of allowing fluid to pass from the top surface to the bottom surface.

$$k = \frac{Q\mu\Delta L}{A\Delta P} \tag{2}$$

Where k is the permeability of the sample (m^2), Q is the flow rate (m^3/s), μ is the dynamic viscosity of the fluid (mPa·s), ΔL is the difference in thickness of the sample before and after immersion in sulfuric acid (m), ΔP is the pressure (Pa), and A is the effective area of the membrane involved in the filtration process (m^2).

3. Result and Discussion

Porous membranes made from nata de soya are derived from a fermentation product originating from tofu and tempe waste. The manufacturing process involves cellulose fermentation produced by Acetobacter xylinum bacteria. During fermentation, cellulose fibers are arranged in a dense, orderly tissue. Nata de soya membranes exhibit an interconnected structure of cellulose fibers with pores formed due to tissue degradation through chemical or enzymatic processes. The porosity and permeability of these membranes vary significantly depending on the manufacturing process, including fermentation conditions and subsequent chemical treatments.

This study aims to investigate the effect of sulfuric acid addition on the porosity and permeability properties of porous nata de soya membranes. Porosity testing using Scanning Electron Microscope (SEM) is a highly effective method for analyzing the microstructure of a material, including nata de soya membranes [22,23]. SEM provides detailed visual representations of the surface and internal structure of the membrane, enabling researchers to measure pore size, shape, and distribution with great accuracy [24,25]. This method is crucial in the development and optimization of membranes as porosity significantly influences filter performance. SEM operates by utilizing emitted electrons directed towards the sample surface [26]. These electrons interact with atoms on the sample surface, producing signals that are then collected and analyzed to generate high-resolution images. SEM is capable of producing three-dimensional images of the sample surface, allowing for in-depth analysis of surface topography and morphology[14, 27]. The SEM characterization results for porous nata de soya membranes with sulfuric acid concentrations of 15%, 20%, and 25% are shown in **Figure 1**.

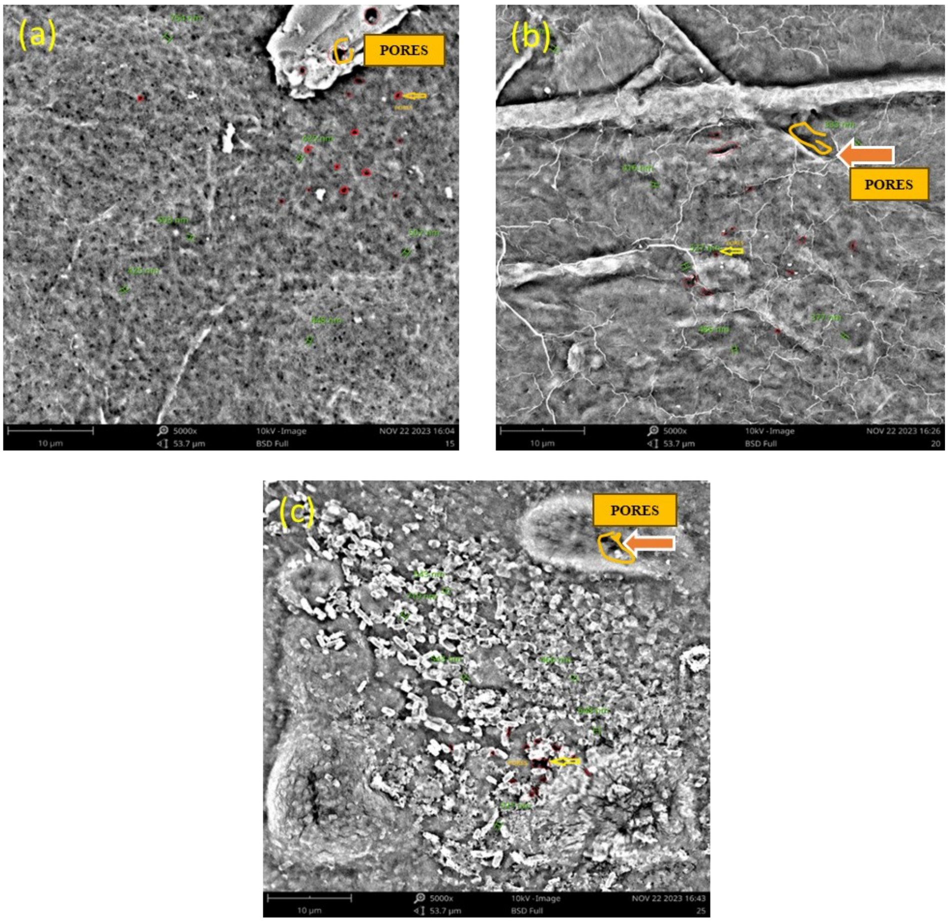


Figure 1. SEM surface test results of nata de soya membrane at a magnification of 5000x at concentrations (a) 15%; (b) 20%; (c) 25%.

The results of SEM testing provide highly detailed images of the pore structure in nata de soya membranes. Several key parameters analyzed include pore size, pore distribution, pore area, and pore density. The use of SEM in testing the porosity of nata de soya membranes yields valuable data on the membrane pore structure. This information is crucial for optimizing production processes and enhancing membrane performance in filtration applications.

3.1. Analysis of Porosity Results

According to Shlekhin [28], surface porosity of the membrane is defined as:

$$\emptyset = 1 - \frac{Vsolid}{Vtotal} \tag{3}$$

the equation defines the surface membrane porosity, which is the ratio of the volume of open pores (Vsolid) to the total volume of the membrane (Vtotal). In this context, surface porosity (∅) indicates how much empty space or pores are available on the membrane surface relative to its total volume. Thus, a high surface porosity value indicates that more pore space is available for interaction with fluids or particles passing through the membrane [29]. The test results can be seen in **Figure 2** and **Figure 3**.

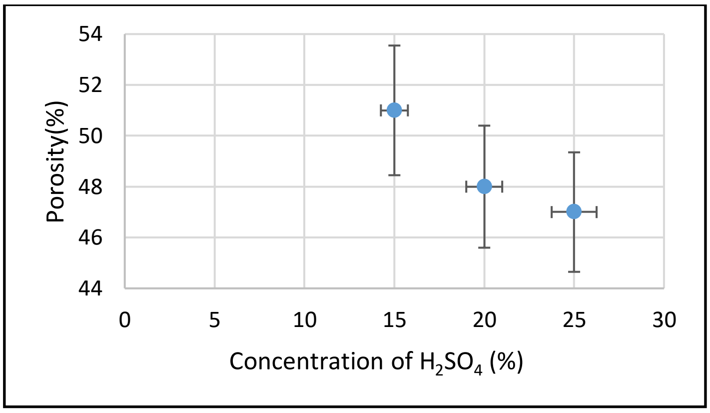
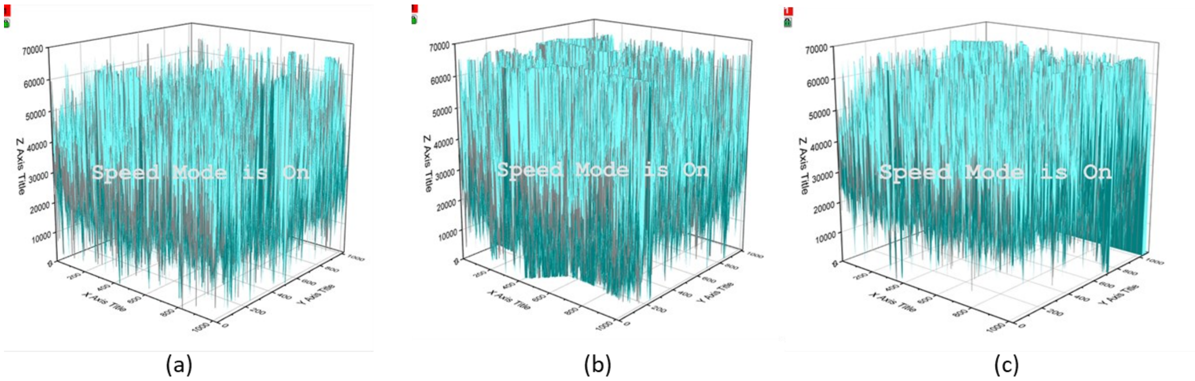


Figure 2. The effect of sulfuric acid variation on the porosity of nata de soya membranes.



(a) (b) (c)

Figure 3. 3D SEM modeling of porous nata de soya membranes at concentrations (a) 15%;(b) 20%;(c) 25%

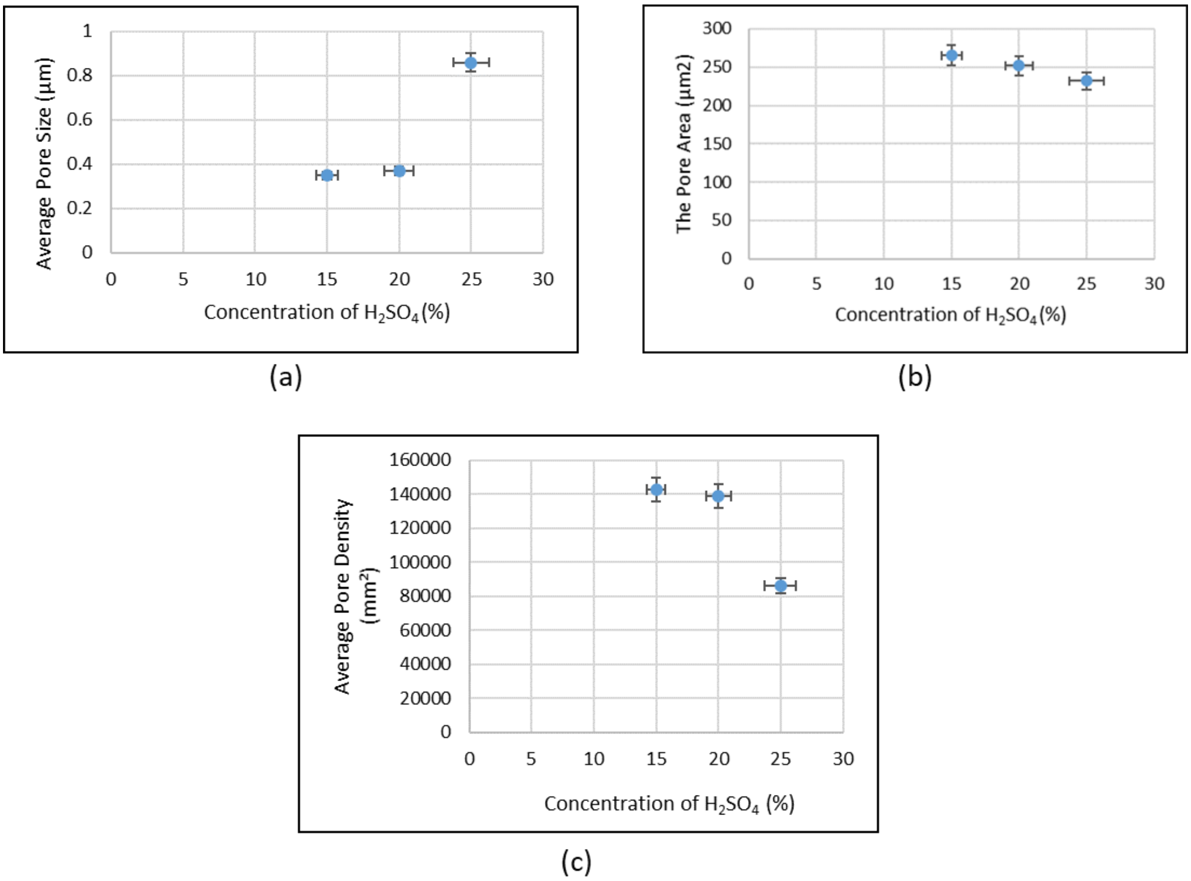
The porosity of porous nata de soya membranes with varying concentrations of sulfuric acid shows interesting variations at different concentrations. At a concentration of 15%, the porosity reaches 51%, attributed to significant cellulose fiber breakdown without excessive structural damage. SEM 3D images at this concentration reveal a more open network of fibers with uniformly formed large pores, indicating high porosity levels [30].

However, when the concentration of sulfuric acid increases to 20%, the porosity decreases to 48%. This is due to more intense damage to the cellulose fibers. Sulfuric acid at this concentration causes deeper degradation, leading to narrowing of pores or even closure of previously open pores. SEM 3D images at this stage show pores beginning to narrow and some areas appearing denser, reflecting the decrease in porosity [31]. At a concentration of 25%, further reduction in porosity to 47%

occurs due to more severe structural damage. The high concentration of sulfuric acid causes excessive decomposition of cellulose fibers, resulting in many pores being closed or even collapsed. SEM 3D images at this concentration reveal a denser structure with few open pores and many fibers appearing degraded or clumped. The decrease in porosity at higher concentrations indicates that while sulfuric acid is effective in opening up the structure at low concentrations, excessively high concentrations can damage membrane integrity, reducing the number of open pores and overall porosity [32].

3.2. Analysis of Pore Size, Area, and Density

The pore density of nata de soya membranes was calculated using **Equation 1**. Meanwhile, pore size and area were analyzed using Microsoft Excel 2007. The results can be seen in **Figure 4**.



(a)

(b)

(c)

Figure 4. (a) The effect of sulfuric acid variation on the average pore size of nata de soya membranes (b) The effect of sulfuric acid variation on the pore area of nata de soya membranes (c) The effect of sulfuric acid variation on the average pore density of nata de soya membranes

From the graph above, it can be seen that the average pore size undergoes significant changes. At a concentration of 15%, the average pore size is 0.35 µm, which increases to 0.37 µm at a concentration of 20%, but then jumps to 0.86 µm at a concentration of 25%. This change indicates that although the number of pores decreases with increasing sulfuric acid concentration, the remaining pore size tends to increase, possibly due to merging or expansion of existing pores. The membrane area decreases with increasing sulfuric acid concentration. At a concentration of 15%, the average area is 265 μm^2. When the concentration is increased to 20%, the area decreases to 252 μm^2, then further decreases to 232 μm^2 at a concentration of 25%. The decrease in area at higher concentrations may be due to a reduction in the total number of effective pores despite the increase in pore size. This suggests the possibility of damage to the membrane structure, reducing the available surface area for the filtration process. The pore density per square millimeter (mm^2) shows a significant decrease from 142.900 at a concentration of 15% to 139.000 at a concentration of 20%, and 86.200 at a concentration of 25%. This indicates that although there are fluctuations in pore density, the general trend shows that increasing sulfuric acid concentration reduces pore density, resulting in a denser and less permeable membrane.

The decrease in pore density with increasing sulfuric acid concentration can be attributed to several factors. Firstly, higher concentrations of sulfuric acid lead to more aggressive chemical dissolution of the membrane material. This results in the dissolution of the membrane matrix, including its pores, significantly reducing pore density. Additionally, the increased acidity due to the higher sulfuric acid concentration can also cause structural damage to the membrane. This includes degradation of the membrane matrix or collapse of pore structures, directly reducing the overall number of pores per unit area. Furthermore, the strong acid environment can lead to the formation of by-products or deposits that partially block or cover existing pores.

Consequently, the number of accessible pores decreases, further reducing pore density. In some cases, increased acidity may also promote the aggregation or merging of small pores into larger ones. Although this increases the average pore size, it overall reduces the total number of pores per unit area. As a result, the decrease in pore density with increasing sulfuric acid concentration indicates the disruptive effect of a strong acid environment on the membrane structure, resulting in fewer and larger pores per unit area.

3.3. Analysis of Permeability Result

The permeability of nata de soya membranes was calculated using Darcy's law (**Equation 2**). Darcy's law relates the flow rate (Q) to permeability (k), cross-sectional area (A), pressure difference (ΔP), and membrane thickness (ΔL). The flow rate (Q) is calculated as the volume of water dripped divided by the time taken for the flow (Q=V/t). The cross-sectional area (A) of the effective membrane area involved in the filtration process is calculated using geometric formulas, specifically $\frac{1}{4}\pi d^2$ where d represents the diameter of the tap. ΔL is the difference in thickness of the sample before and after immersion in sulfuric acid (m). The dynamic viscosity of the fluid is 1.002 mPa·s.

The use of Darcy's law in calculating the permeability of nata de soya membrane is crucial as it provides a comprehensive framework for understanding how fluid flow and membrane properties interact with each other. By understanding this law, we can optimize the design and performance of nata de soya membrane in various applications, including filtration processes and water purification. The research results can be seen in **Figure 5**.

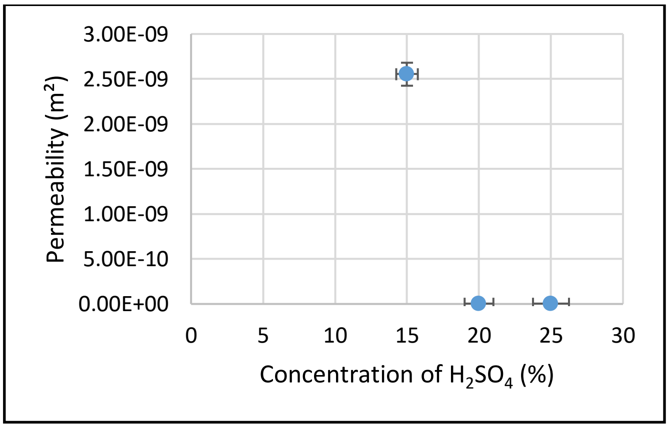


Figure 5. The effect of sulfuric acid variation on the permeability of nata de soya membranes

At a concentration of 15% with a soaking time of 30 minutes, the permeability value recorded is 2.55287×10^{-9} m². This value indicates that the membrane has a fairly high ability to allow fluid to pass through. However, when the sulfuric acid concentration is increased to 20%, the membrane permeability drastically decreases to 2.55287×10^{-12} m². This decrease indicates that the increase in sulfuric acid concentration results in a reduction in the membrane's ability to allow fluid to pass through. At a concentration of 25%, the membrane permeability further decreases to 2.12739×10^{-12} m². The decrease in permeability values with increasing sulfuric acid concentration can be explained by changes in the pore structure of the membrane. Sulfuric acid can cause shrinkage or partial closure of the pores in the membrane, reducing the number of pathways available for fluid flow. This is consistent with the findings of Gao et al. [26] which showed that acid modification of cellulose acetate membranes can reduce permeability due to a reduction in pore size and an increase in material density.

The decrease in permeability value of nata de soya membrane along with the increase in sulfuric acid concentration can be attributed to several factors. Firstly, higher concentrations of sulfuric acid tend to cause structural damage to the membrane. Jun et al. [33] found that the increase in sulfuric acid concentration led to a decrease in membrane elasticity and loss of structural integrity, directly reducing its permeability. Additionally, a strong acidic environment can trigger the formation of deposits or by-products within the membrane pores, which can block or obstruct fluid flow. This is supported by research by J. Wei and X. Wu [34] which found that the increase in sulfuric acid concentration resulted in an increase in the amount of trapped deposits within the membrane pores, consequently reducing its permeability. Furthermore, increased acidity can also enhance the agglomeration or merging of small pores into larger ones, which can further reduce the total number of pores and lead to decreased permeability. As a result, the increase in sulfuric acid concentration during the production of nata de soya membrane can result in decreased permeability values due to structural damage, deposition formation, and pore agglomeration. These studies underscore the importance of understanding the mechanisms of structural changes in membranes in response to specific environmental conditions to enhance membrane performance in filtration applications.

3.4. Discussion of Findings and Implications

This study demonstrates that the addition of sulfuric acid significantly influences the porosity and permeability properties of nata de soya membranes. At low concentrations (15%), the membranes exhibit higher porosity and permeability, which are suitable for applications requiring good fluid flow. However, at higher concentrations (20% and 25%), the membranes become denser with lower porosity and permeability, which may be more suitable for applications requiring stricter filtration or reduction of smaller particles.

The implications of this research are highly significant for industries utilizing membranes for various applications, including fluid separation, air filtration, and water purification. According to I. Gudavadze and E.-L. Florin [8], bacteria typically range in size from 0.2 to 2 μm. In porous nata de soya membranes, pore sizes of 0.35 μm and 0.37 μm at concentrations of 15% and 20%, respectively, are larger than the typical size range of bacteria, which generally varies from 0.2 to 2 μm. Therefore, membranes at these concentrations can still be effective in filtering some bacteria. However, because these pore sizes are larger than 0.2 μm, smaller bacteria may potentially pass through. On the other hand, at a concentration of 25%, with a pore size reaching 0.86 μm, this membrane becomes less effective in filtering smaller bacteria because the pores are too large, allowing these bacteria to pass through.

Hutabarat et al. [35] reported that *E. coli* bacteria can cause diarrhea, sepsis, and other diseases if consumed by humans. It is commonly found in wastewater, rivers, and wells. Based on the report by Bisping and Amtsberg (1988), the size of *E. coli* bacteria varies with widths around 1.1-1.5 μm and lengths approximately 2-6 μm. Porous nata de soya membranes with pore sizes of 0.35 μm at 15% concentration and 0.37 μm at 20% concentration have different implications for filtering *E. coli* bacteria. At 15% and 20% concentrations, the membrane pore sizes (0.35 μm and 0.37 μm) are smaller than the width of *E. coli* bacteria (1.1-1.5 μm). This suggests that membranes at these concentrations may effectively filter most *E. coli* bacteria, as their pores are smaller than the dimensions of these bacteria. However, there is a possibility that some larger *E. coli* bacteria or those with unusual shapes may still pass through these pores.

In contrast, at 25% concentration with a pore size of 0.86 μm, the membrane pores are larger than the width of *E. coli* bacteria. Therefore, membranes at this concentration may be less effective in filtering *E. coli* bacteria, as many bacteria can pass through these larger pores without hindrance. Overall, the pore size of nata de soya membranes at lower concentrations (15% and 20%) tends to be more suitable for effective filtration of *E. coli* bacteria, while higher concentrations (25%) may be more appropriate for applications requiring retention of particles larger than the size of these bacteria. The conclusion is that the porous membrane from nata de soya is effective for microfiltration of bacteria and water treatment, both for drinking water and wastewater. Understanding how chemical modifications such as sulfuric acid addition affect membrane properties enables researchers and practitioners to design membranes with desired characteristics for specific applications. This research also opens up opportunities for further exploration of other chemical combinations that can be used to modify membrane properties. Additionally, further research can be conducted to understand the detailed mechanisms behind changes in pore structure and surface properties due to sulfuric acid treatment, as well as to develop more effective optimization methods.

4. Conclusion

This study examines the effect of sulfuric acid addition on the porosity and permeability of nata de soya membranes. Porosity testing using a Scanning Electron Microscope (SEM) showed that increasing the sulfuric acid concentration from 15% to 25% caused a decrease in porosity from 51% to 47%. The pore size increased from 0.35 µm at 15% to 0.86 µm at 25%. Both pore area and density also decreased with the increasing concentration of sulfuric acid, indicating a reduction in membrane effectiveness in filtration processes. Additionally, membrane permeability dropped significantly from 2.55287×10^{-9} m² at 15% to 2.12739×10^{-12} m² at 25%, indicating that sulfuric acid caused pore closure and structural damage to the membrane, reducing its ability to pass fluids. The implications of this research suggest that porous nata de soya membranes are effective for microfiltration of bacteria (such as *E. coli*) and water treatment, both for drinking water and wastewater.

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