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Effect of Chitosan Addition on Porosity of Rice Husk Calcium Silicate Used for Free Fatty Acid Adsorbent from Palm Oil

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ABSTRACT

Studies have been done on developing calcium silicate pores derived from rice husk with and without including chitosan as a template. The chitosan solution was supplemented with sodium silicate derived from rice husk, and then $CaCl_2$ was added. The calcium silicate was calcinated at 900ºC for 4 h. The calcium silicate obtained was characterized using X-ray diffraction (XRD), fourier transform infrared (FT-IR), and Brunauer-Emmett-Teller (BET) analysis. The XRD analysis showed the presence of diffraction peaks corresponding to Wollastonite ($CaSiO_3$) crystals, Wollastonite 2m ($CaSiO_3$), Alite (Ca_3SiO_5), and larnite (Ca_2SiO_4). Next, the FT-IR spectrum exhibits Si-O-Si, Si-O-Ca, and Si-OH functional groups, suggesting the presence of calcium silicate material. The BET study of calcium silicate using chitosan templates revealed a pore size of 3.88 nm, a pore volume of 0.1577 cc/g, and a surface area of 81.1519 m^2/g. The BET analysis results obtained differed from those of the BET analysis of calcium silicate conducted without using chitosan templates, which were 2.06 nm, 0.069 cc/g, and 13.531 m2/g, respectively. The developed mesoporous calcium silicate was employed as an adsorbent to decrease the concentrations of free fatty acids in palm oil. The acid-base titration method is used to evaluate quantities of free fatty acids. The results obtained from the computation of free fatty acid levels indicate a decline in the concentration of free fatty acids in palm oil. The concentration of free fatty acids derived from palm oil was 1.23%. Therefore, mesoporous calcium silicate-free fatty acids at a concentration of 1.06% resulted in an 86.1% reduction.

Keywords: Calcium Silicate, Chitosan, Crude Palm Oil, Free Fatty Acid, Rice Husk

ABSTRAK

Telah dilakukan penelitian untuk mengembangkan kalsium silikat berpori yang berasal dari sekam padi dengan dan tanpa menyertakan kitosan sebagai template. Larutan kitosan ditambah dengan natrium silikat yang berasal dari sekam padi, kemudian ditambahkan $CaCl_2$. Kalsium silikat dikalsinasi pada suhu 900ºC selama 4 jam. Kalsium silikat yang diperoleh dikarakterisasi menggunakan difraksi sinar-X (XRD), fourier transform inframerah (FT-IR), dan analisis Brunauer-Emmett-Teller (BET). Analisis XRD menunjukkan adanya puncak difraksi yang berhubungan dengan kristal Wollastonit ($CaSiO_3$), Wollastonit 2m ($CaSiO_3$), Alite (Ca_3SiO_5), dan larnit (Ca_2SiO_4). Selanjutnya, spektrum FT-IR menunjukkan gugus fungsi Si-O-Si, Si-O-Ca, dan Si-OH, yang menunjukkan adanya bahan kalsium silikat. Studi BET kalsium silikat menggunakan templat kitosan menunjukkan ukuran pori 3,88 nm, volume pori 0,1577 cc/g, dan luas permukaan 81,1519 m^2/g. Hasil analisis BET yang diperoleh berbeda dengan analisis BET kalsium silikat yang dilakukan tanpa menggunakan templat kitosan, yaitu masing-masing sebesar 2,06 nm, 0,069 cc/g, dan 13,531 m^2/g. Kalsium silikat mesopori digunakan sebagai adsorben untuk menurunkan konsentrasi asam lemak bebas dalam minyak sawit. Metode titrasi asam basa digunakan untuk mengevaluasi jumlah asam lemak bebas. Hasil yang diperoleh dari perhitungan kadar asam lemak bebas menunjukkan adanya penurunan konsentrasi asam lemak bebas pada minyak sawit. Konsentrasi asam lemak bebas yang berasal dari minyak sawit sebesar 1,23%. Memperkenalkan asam lemak bebas kalsium silikat mesopori pada konsentrasi 1,06% menghasilkan pengurangan sebesar 86,1%.

Kata Kunci : Asam Lemak Bebas, Kalsium Silikat, Minyak Kelapa Sawit, Kitosan, Sekam Padi

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1. Introduction

Rice husk is a hard layer that encloses the grain cariopsis, consisting of interlocked two hemispheres called lemma and palea. Rice husk consists of 34-44% cellulose, 23-30% lignin, 13-39% ash, and 8-15% water [1]. Rice husk is utilized as a source of silica due to its high silica content (86.90-97.30%) [2].

Calcium silicate, known by the chemical formula $CaSiO_3$, has a melting point of 1540° C, a density of 2.91 g/cm^3, and a hardness of 4-7 Mohs. Based on the mass calculation of calcium silicate ($CaSiO_3$), it has a theoretical composition of CaO 48.28% and SiO_2 51.72% [3].

The use of $CaSiO_3$ as an adsorbent has been widely reported, including for removing aflatoxin B1 from aqueous solutions and removing endotoxin from water (Zeng, 2014) [4]. Mazmur (2016) reported using calcium silicate as an adsorbent for free fatty acids (FFA) from palm oil has an 81.47% ability, whereas $CaSiO_3$ has better adsorption power than other silicate salts [5]. Siahaan (2018) reported glycerol as a template calcium silicate for free fatty acid adsorbent from crude palm oil has an ability of 80% [6].

One important factor affecting the adsorption process is the type of adsorbent. Adsorbents are currently of interest to researchers are porous materials. The larger the pore size of the adsorbent, the greater the adsorption power of the adsorbate. According to IUPAC, porous materials are divided into three classes: microporous materials with pore diameter sizes of less than 2 nm, mesopores with pore diameter sizes of 2 to 50 nm, and macropores with diameter sizes of more than 50 nm. Mesoporous materials have been extensively studied for their outstanding performance in drug manufacturing, catalysis, healthcare, and especially adsorption [7].

Calcium silicate has high pore size, surface area, particle size, and pore volume. The pore size of calcium silicate can be modified by adding carbon-containing templates. The template is used as a mold (auxiliary and directional) in the formation of pores, where the primary colloidal particles will fill the gaps between the template arrangement so that when the template is removed from the calcium silicate particles, hollow particles will be formed [8]. This template can be easily removed by calcination or heating at high temperatures [9].

Aritonang (2017) synthesized calcium silicate from volcanic ash silica with the addition of polyethylene glycol as a template. Calcium silicate was obtained from the reaction between $CaCl_2$ and silica from volcanic ash, then mixed with polyethylene glycol, and the mixture was calcined at 900°C for 4 h. The data displayed that the calcium silicate pores formed were getting smaller, from 10.1 nm to 8.8 nm [10]. Chitosan is a biocompatible hydrophilic polysaccharide that is found in abundance in crustaceans. Chitosan is insoluble in water and some organic solvents but soluble in dilute acids/minerals at pH < 6.5 [11].

Therefore, the effect of chitosan addition on the porosity of calcium silicate from rice husk is more effective in reducing free fatty acid levels in palm oil. In this study, sodium silicate was slowly added to the chitosan template. Then, the mixture obtained was added to the $CaCl_2$ solution. The resulting calcium silicate was dried in an oven at 90^{o}C for 24 h and then calcined at 900^{o}C to remove chitosan.

2. Materials and Methods

2.1 Equipment

The tools used in this research include glassware, furnace control, oven, hotplate stirrer, digital balance, universal indicator, filter paper, porcelain cup, thermometer, Fourier Transform Infrared (FT-IR), X-ray Diffraction (XRD), Brunauer-Emmett-Teller (BET), and adsorption instrument.

2.2 Material

The materials used in this study include rice husk, NaOH, HCl, $CaCl_2$, n-hexane, isopropyl alcohol, chitosan, phenolphthalein indicator, crude palm oil, and aquadest.

2.3 Research procedure

2.3.1 Extraction of Silica from Rice Husk Rice

Rice husk was as much as 200 g, washed with distilled water, dried, and then soaked in 2 L of 10% NaOH while heated for 6 hours and filtered. The filtrate was added 11% HCl until pH = 7, and a brown gel formed. Then, it was allowed to stand for one night at room temperature and filtered. The silica gel was washed with hot distilled water until it turned white. Then, it was dried in the oven at 110^{o}C and pulverized to powder form.

2.3.2 Preparation of Sodium Solution Silicate

4.5 g of silica powder was dissolved in 70 mL of 10% NaOH. Then, it was stirred while heated at 110ºC with a hotplate stirrer for 3 hours until a sodium silicate solution was formed.

2.3.3 Preparation of Calcium Silicate

Sodium silicate solution was added to $CaCl_2$ solution while stirring and heated for 4 hours at 110ºC using a hotplate stirrer until a white solid was formed. Then it was aged for 24 h at 90ºC, then calcined at 900ºC for 4 h. Next, the solid was washed with distilled water and dried at 110ºC in the oven. FT-IR, XRD, and BET characterized the white solid obtained.

2.3.4 Preparation of Calcium Silicate with Chitosan Template

A solution of sodium silicate was introduced into a solution of calcium chloride while being agitated and then subjected to heating for 4 hours at a temperature of 110ºC using a hotplate stirrer until a solid substance of white coloration was produced. Subsequently, the substance underwent a 24-hour aging process at 90ºC, followed by a 4-h calcination at 900ºC. Subsequently, the solid was rinsed with distilled water and subjected to desiccation at 110ºC within the confines of an oven. The white solid was analyzed using FT-IR, XRD, and BET to determine its properties.

2.3.5 Adsorption of FFA from CPO

2 g of CPO and 20 mL of n-hexane were combined in a 250 mL Erlenmeyer flask and swirled until a uniform mixture was obtained. 2 g of calcium silicate adsorbent was added to the mixture, followed by stirring and allowing it to stand for 30 minutes. Subsequently, the mixture was filtered. The resulting filtrate was transferred into a 20 ml volumetric flask and then distributed in equal amounts of 5 ml each into three 250 ml Erlenmeyer flasks. Subsequently, 5 mL of isopropyl alcohol was added to each flask, and the mixture was agitated until it became uniform. Phenolphthalein indicator (3 drops) was added to the mixture, followed by NaOH (0.1 N) titration to determine the free fatty acid level.

3. Results and Discussion

3.1 Preparation of Calcium Silicate

The preparation of calcium silicate uses raw materials, namely silica from rice husks and $CaCl_2$ as a source of Ca metal. Silica from rice husk was obtained by heating 200 g of rice husk in 2 L of 10% NaOH for 6 h. This was done to react the silica from the rice husk with NaOH. To the filtrate, 11% HCl until pH = 7 to form silica gel. The silica gel was taken and washed using hot distilled water to remove lignin and cellulose and then dried in an oven at 110ºC. The obtained silica extraction results are about 9.9 g. Next, the extracted silica was taken 4.5 g and then dissolved in 70 mL of 10% NaOH to form a sodium silicate solution. The mixed-with $CaCl_2$ solution obtained by dissolving 11.025 g $CaCl_2$ in 100 mL of distilled water. Then, this mixture was stirred while heated at 110ºC until it formed a white precipitate of calcium silicate ($CaSiO_3$). The solid formed was then allowed to stand for one night, filtered and washed using hot distilled water, then dried in an oven at 110ºC. $CaSiO_3$ solids of 8.7 g were obtained. Then, the $CaSiO_3$ solid was calcined at 900ºC for 4 hours. The result was obtained in the form of a coarse white solid. Then, it was characterized by FT-IR, XRD, and BET.

3.1.1 FTIR Spectrum of Calcium Silicate

Calcium silicate obtained was analyzed using FT-IR to determine the presence of functional groups. The FT-IR spectra of the calcium silicate obtained are shown in Figure 1.

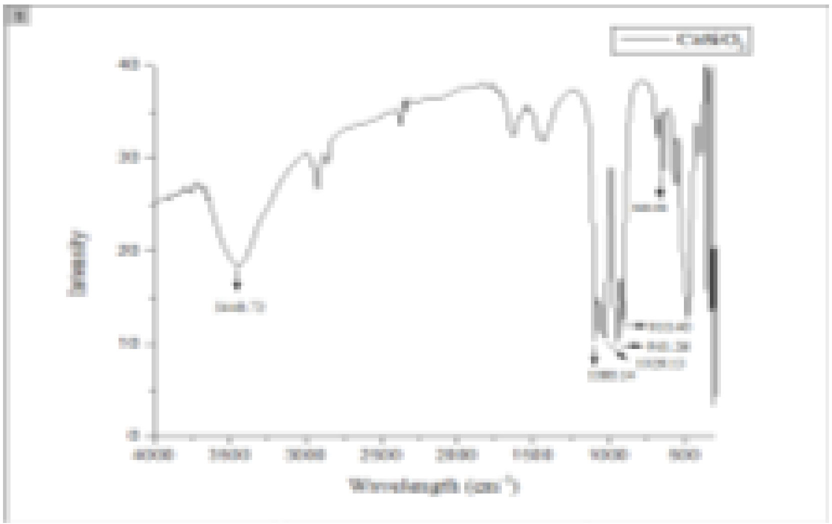

Figure 1. FT-IR Spectrum of Calcium Silicate

The wave number 3448.72 cm^{-1} signifies the existence of the Si-OH group, whereas the wave numbers 1026.13 cm^{-1} and 1080.14 cm^{-1} show the presence of an asymmetric Si-O-Si group. The wave numbers 478.35 cm^{-1} and 648.08 cm^{-1} suggest the existence of symmetry Si-O-Si groups, whereas the absorption peaks at wave numbers 910.4 cm^{-1} and 941.26 cm^{-1} show the presence of Si-O-Ca groups [12].

3.1.2 X-ray Diffraction (XRD) of Calcium Silicate

X-ray diffraction (XRD) method was used to analyze the crystal composition in a sample. The diffraction pattern obtained from calcium silicate is shown in Figure 2.

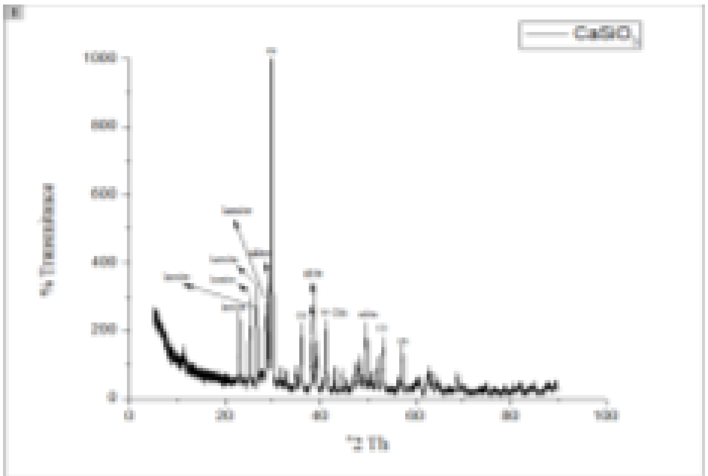

Figure 2. XRD diffractogram of calcium silicate

The XRD diffractogram shown in Table 1 at 2θ angles shows several different peaks; namely, there are 48 readable peaks and 14 peaks with quite sharp intensity. The list of these peaks indicates the presence of wollastonite -2m ($CaSiO_3$-2M) at 41.17º wollastonite ($CaSiO_3$) at 29.91º, 36.10º, 57. 29º, 53.10º; alite (Ca_3SiO_5) at 29.81º, 38.18º, 38.93º, 49.61º; larnite (Ca_2SiO_4) at 22.96º, 25.12º, 26.72º, 28.67º, 29.28º.

3.1.3 BET (Burnaur-Emmet-Teller) Analysis of Calcium Silicate

Nitrogen adsorption-desorption isotherm graph is the result of BET characterization of calcium silicate. Nitrogen adsorption-desorption isotherm is performed to measure the surface area, pore volume and pore size of calcium silicate material. The curve of adsorption-desorption isotherm graph with BJH method can be seen in Figure 3.

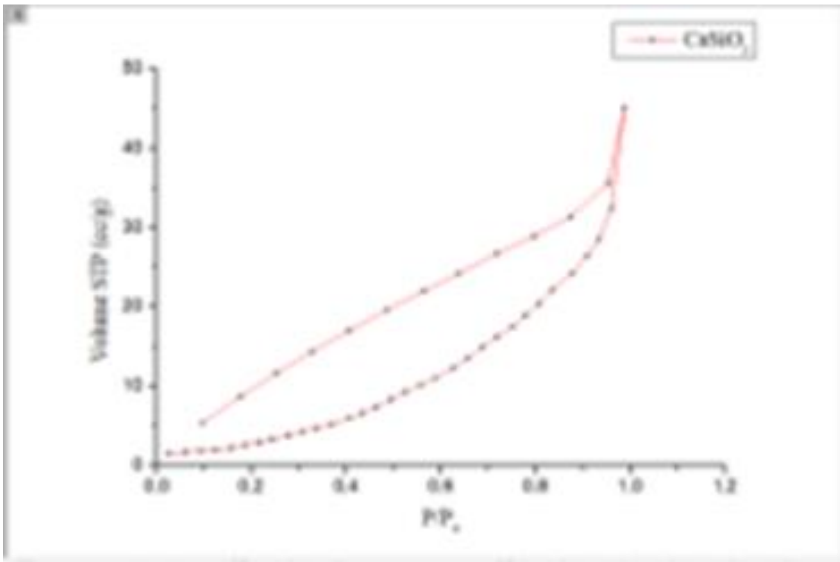

Figure 3. Calcium silicate nitrogen adsorption-desorption isotherm chart

Figure 3 displays the type V adsorption isotherm, as classified by IUPAC. The nitrogen adsorption-desorption isotherm graph of calcium silicate shows the production of several layers due to capillary condensation at relative pressures (P/P0) ranging from 0.2 to 0.8 atm. Capillary condensation is the result of a pressure difference that occurs during the processes of adsorption and desorption. Type V is used to classify porous materials exposed to nitrogen gas and belong to the mesoporous category (Quercia, 2013). The pore size distribution was calculated using the Barret-Joyner-Halenda (BJH) method, and the results can be seen in Figure 4.

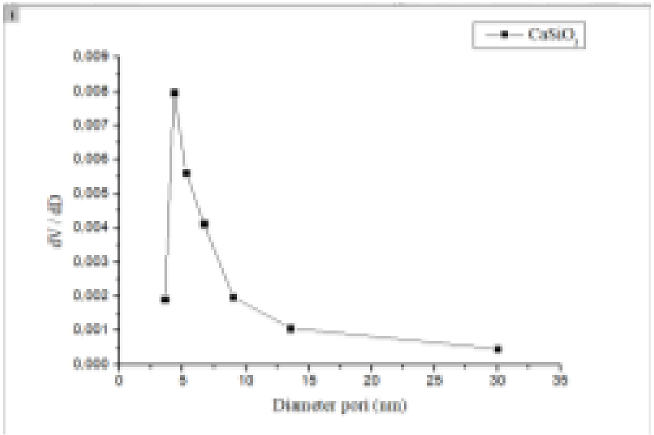

Figure 4. Calcium silicate pore size distribution chart

Based on Figure 4, the pore size distribution graph of calcium silicate is the most dominant pore with a diameter of 4.04 nm, which is classified into mesopore size. The results of desorption adsorption analysis for calcium silicate can be seen in Table 1.

Table 1 Calcium silicate pore characterization data

Parameter	**Nilai**
Surface area	13.531 m^2/g
Pore volume	0.069 cc/g
Pore size	2.06 nm

3.2 Preparation of Calcium Silicate with Chitosan Template

The preparation of calcium silicate with chitosan template was carried out by adding 2 g of chitosan to the sodium silicate solution, then adding $CaCl_2$ solution while stirring and heating using a hotplate stirrer at 110ºC until a white solid was formed, then allowed to stand (aging) for one night and then filtered. Then, the white solid was dried in the oven at 110ºC and then calcined at 900ºC for 4 hours. The white solid was washed with hot distilled water, and white solids with refined grains were obtained. Then, it was characterized by FT-IR, XRD, and BET.

3.2.1 FTIR Spectrum of Calcium Silicate with Chitosan Template

Calcium silicate obtained was analyzed using FT-IR to determine the presence of functional groups. The FT-IR results obtained are shown in Figure 5 below :

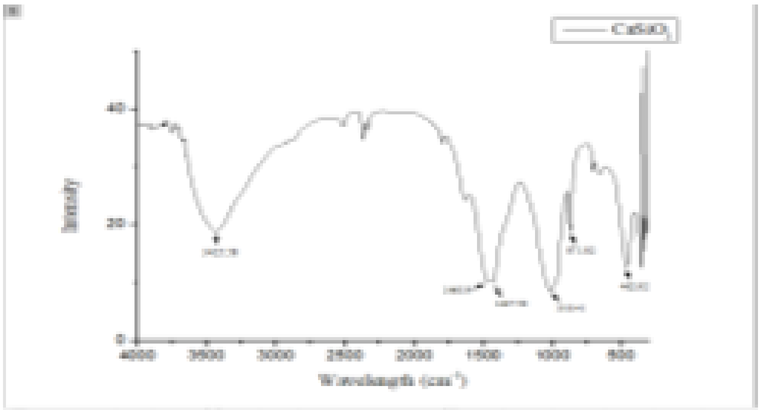

Figure 5. FT-IR spectrum of calcium silicate with chitosan template

The FT-IR spectra depicted in Figure 5 elucidate that the chitosan has ceased to exist after being subjected to calcination at 900ºC. The absence of $-NH_2$ absorption in the region of (750 - 800) cm^{-1} and -OH absorption in the range of (2800 - 3300) cm^{-1} indicates this. The absorption at wave number 3425.58 cm-1 indicates the presence of Si-OH groups. The absorption peaks at wave numbers 1427.32 cm^{-1} and 1465.9 cm^{-1} indicate the presence of asymmetric Si-O-Si groups. The absorption peaks at wave numbers 462.92 cm^{-1} and 871.82 cm^{-1} indicate the presence of symmetry Si-O-Si groups. Lastly, the absorption peak at wave number 1018.41 cm^{-1} indicates the presence of Si-O-Ca groups [12].

3.2.2 X-ray Diffraction (XRD) of Calcium Silicate with Chitosan Template

X-ray diffraction (XRD) method is used to analyze the crystal composition in a sample. The diffraction pattern obtained from calcium silicate is shown in Figure 6.

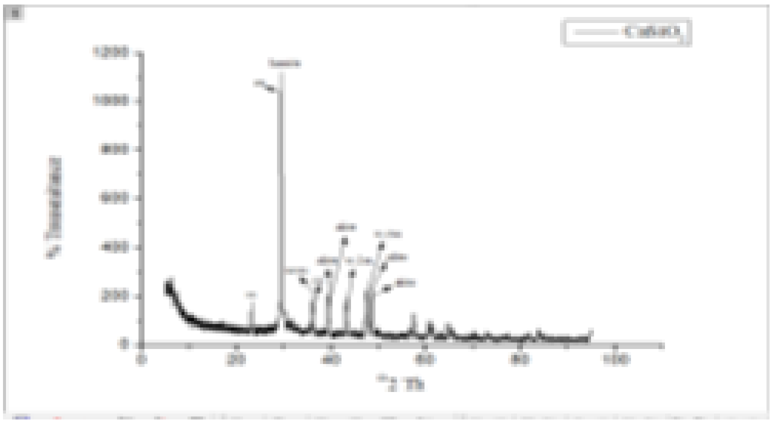

Figure 6. XRD diffractogram of calcium silicate templated with chitosan

The XRD diffractogram shown in Table 2 at 2θ angles shows 28 readable peaks, and there are 11 peaks whose intensity is quite sharp. The list of these peaks indicates the presence of wollastonite -2m ($CaSiO_3$-2M) at 43.109º, 43.387º; wollastonite ($CaSiO_3$) at 23.253º, 29.369º, 36.18º; alite (Ca3SiO5) at 39.359º, 39.648º, 47.469º, 48.441º; larnite (Ca2SiO_4) at 29.54º, 35.962º.

3.2.3 BET (Burnaur-Emmet-Teller) Analysis of Calcium Silicate with Chitosan Template

The nitrogen adsorption-desorption isotherm graph results from BET characterization of Calcium Silicate with chitosan as a template. Adsorption - desorption nitrogen isotherm is performed to organize the pore size distribution of calcium silicate material with chitosan template. The curve of the adsorption-desorption isotherm graph with the BJH method can be seen in Figure 7.

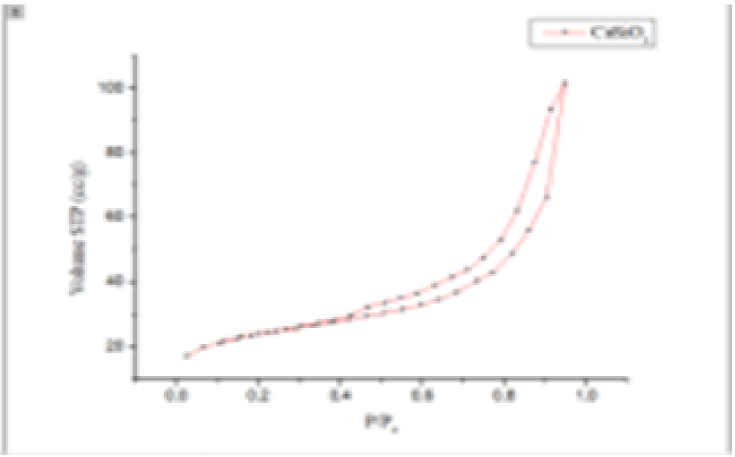

Figure 7. Nitrogen adsorption-desorption isotherm graph of calcium silicate templated with chitosan

Figure 7 displays the V-type adsorption isotherm, as classified by the IUPAC. Calcium silicate's nitrogen adsorption-desorption isotherm graph shows multilayer development through capillary condensation at relative pressures (P/Po) ranging from 0.5 to 0.9 atm. Capillary condensation is a phenomenon that occurs when there is a pressure differential between the processes of adsorption and desorption. The designation of Type V indicates that the porous material falls into mesoporous materials when exposed to nitrogen gas (Quercia, 2013). In order to determine the pore size distribution, the Barret-Joyner-Halenda (BJH) method was used and can be seen in Figure 8.

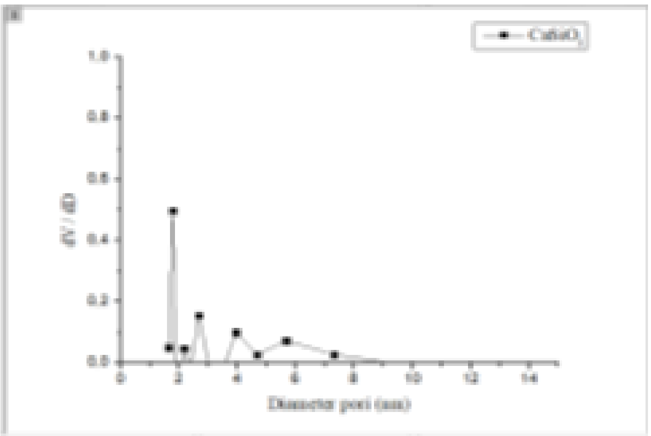

Figure 8. Pore size distribution graph of chitosan-templated calcium silicate

Based on Figure 8, the pore size distribution graph of calcium silicate is the most dominant pore with a diameter of 1.82 nm, classified into micropore size. The results of desorption adsorption analysis for calcium silicate can be seen in Table 3 below:

Table 3 Pore characterization data of calcium silicate with the addition of chitosan template

Parameter	**Value**
Surface area	81.1519 m^2/g
Pore volume	0.1577 cc/g
Pore size	3.88 nm

3.3 Adsorption of Free Fatty Acids with Calcium Silicate Adsorbent

The free fatty acid content reduction in palm oil with calcium silicate adsorbent was conducted. The palm oil used in this study was obtained from PT Perkebunan Nusantara III Medan between 2011 and 2013 and has been stored for a long time in the Inorganic Chemistry Laboratory of FMIPA USU. Before the free fatty acid content of palm oil was adsorbed, the Alkalimetric titration method determined the free fatty acid content contained in the palm oil.

Table 4. Results of free fatty acid content analysis

Treatment	Titration Volume (mL)	FFA Level (%)	FFA Rate (%)	Standard Deviation
1	0.9	1.15	1.23	0.05
2	1.0	1.28		
3	1.0	1.28		

Calculation of Standard Deviation : (Cahya, R.A., 2014)

$$S = \sqrt{\sum \frac{(v - v_{\text{rate}})^2}{N-1}}$$

Description:

S = Standard Deviation
V = Value of % FFA
V_{rate} = Rate value of % FFA
N = Number of Analysis

$$S_1 = \sqrt{\sum \frac{(1.15 - 1.23)^2}{3-1}} = \sqrt{0.0032} = 0.06$$

$$S_2 = \sqrt{\sum \frac{(1.28 - 1.23)^2}{3-1}} = \sqrt{0.00125} = 0.04$$

$$S_3 = \sqrt{\sum \frac{(1.28 - 1.23)^2}{3-1}} = \sqrt{0.00125} = 0.04$$

$S_{\text{rate}} = 0.05$

Based on Table 4, palm oil's free fatty acid content is 1.23%. Palm oil's free fatty acid content was reduced by adding 2 g of calcium silicate adsorbent to 2 g of palm oil in 20 mL of n-hexane, stirring until homogeneous, and filtering. The filtrate was added 5 mL isopropyl alcohol. The remaining free fatty acid content was determined by alkalimetric titration according to AOCS.

Table 6 Results of free fatty acid content analysis after addition of calcium silicate adsorbent

Treatment	Titration Volume (mL)	FFA Level (%)	FFA Rate (%)	Standard Deviation
1	0.8	1.02	1.06	0.04
2	0.9	1.15		
3	0.8	1.02		

Calculation of Standard Deviation :

$$S = \sqrt{\sum \frac{(v - v_{rate})^2}{N-1}}$$

$$S_1 = \sqrt{\sum \frac{(1.02 - 1.06)^2}{3-1}} = \sqrt{0.0008} = 0.03$$

$$S_2 = \sqrt{\sum \frac{(1.15 - 1.06)^2}{3-1}} = \sqrt{0.00405} = 0.06$$

$$S_3 = \sqrt{\sum \frac{(1.02 - 1.06)^2}{3-1}} = \sqrt{0.0008} = 0.03$$

$S_{rate} = 0{,}04$

Based on the results, the analysis reveals that palm oil's free fatty acid content has reduced. The initial palm oil had a free fatty acid number of 1.23%. After introducing calcium silicate adsorbent, the free fatty acid concentration decreased by 86.1% to 1.06%. The surface area and pore size of the calcium silicate determine the adsorption capacity of mesoporous calcium silicate for free fatty acids. The significant surface area is a result of the substantial pore size.

4. Conclusion

In conclusion, the use of chitosan as a template produces mesoporous calcium silicate with a pore size of 3.88 nm, surface area of 81.1519 m^2/g and pore volume of 0.1577 cc/g analyzed by nitrogen gas desorption adsorption. Mesoporous calcium silicate can reduce palm oil-free fatty acids, namely the initial free fatty acid of 1.23%. After adding calcium silicate adsorbent, the free fatty acid becomes 1.06%, decreasing by 86.11%.

5. Acknowledgements

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6. Conflict of Interest

Authors declare no conflicts of interest

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