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Study of kinetic and adsorption isotherm of ibuprofen on MCM-41 synthesized with rice husk

Olyvia Azzahra Putri Hartono[1], Frida Octavia Purnomo[1*], Dyah Ayuwati Waluyo[1] Tunas Alam[2], Mohammad Jihad Madiabu[3]

[1]Department of Pharmacy, Faculty of Health Sciences and Technology, Binawan University, Jl. Kalibata Raya - Dewi Sartika, No. 25-30, East Jakarta, DKI Jakarta, Indonesia
[2]Department of Pharmacy, STIKes Prima Indonesia, Jl. Raya Babelan, Kebalen, Babelan, Bekasi, Jawa Barat, Indonesia
[3]Department of Analytical Chemistry, Politeknik AKA Bogor, Jl.Pangeran Soegiri, Tanah Baru, Bogor, Indonesia

ABSTRACT

Rice husk is one of the abundant wastes, especially in agricultural countries. Rice husk waste has a silica content of 95.80%, where the silica content can be utilized to make an adsorbent. One of the adsorbents that can be made is Mobil Composition of Matter 41 (MCM-41), a material with a hexagonal structure with a surface area to adsorb ibuprofen. Based on the results of the research that has been done, MCM-41 synthesized with rice husk has the same characterization results as MCM-41 synthesized with commercial materials tetraethyl orthosilicate (TEOS). Fourier Transform Infrared Spectroscopy (FTIR) characterization results show the absorption peak is at wave number 1068.58 cm-1 which shows asymmetric Si-O-Si stretching vibrations and at 799.60 cm^{-1} region is symmetric Si-O-Si stretching vibrations. X-ray diffraction (XRD) characterization results show an hexagonal crystal form at $2\theta = 20^{o}$-30^{o}. Scanning Electron Microscope (SEM) characterization results show particle of 2,664 μm. Based on the results of the research that has been done MCM-41 synthesized from rice husk can adsorb ibuprofen with Langmuir isotherm approach and Pseudo Second Order kinetics, and the maximum adsorbing capacity is 34.48 mg/g.

Keywords: ibuprofen, langmuir isotherms, MCM-41, pseudo second order

Corresponding author:
Frida Octavia Purnomo
Department of Pharmacy, Faculty of Health Sciences and Technology, Binawan University
Jl. Kalibata Raya - Dewi Sartika, No. 25-30, East Jakarta, DKI Jakarta, Indonesia
Email: fridaoctavia@binawan.ac.id

INTRODUCTION

Ibuprofen is a type of Non-Steroidal Anti-Inflammatory Drug (NSAID) that is acidic and has cohesive properties greater than adhesive properties. In these properties, which causes ibuprofen to be difficult to contact with other substances, especially water, so that ibuprofen is also a little difficult to make in certain dosage forms. Absorption of ibuprofen is rapid through the stomach with maximum levels in plasma reached after 1-2 hours, with a half-life in plasma of about 2 hours (Juwita et al., 2015). In general, ibuprofen is adsorbed with polymers, forming a solid dispersion with polymers that can increase solubility in water, for example polyvinyl pyrrolidone (PVP) K30 (Hasanah et al., 2015) .

The application of kinetic models is very useful for understanding the process of reaction rates, changes in the concentration of a reactant. Based on the chemical reaction process, the kinetic model is expressed by the rate of change of the quality constant (k) and the reaction order (n). The value of the constant is a specific parameter that depends on each condition. The value of the reaction order is theoretically unlimited, but the value ranges from 0-2, and is used in kinetic modeling. Kinetic parameters are used as the main benchmark to determine the advantages and disadvantages of a reactant (Priyanto, 2009).

Based on research (Ahda, 2015), MCM-41 can be used to adsorb ibuprofen. By utilizing the large surface area and pore size of MCM-41, it is expected that ibuprofen will enter and spread throughout the pores and surface area of MCM-41. This causes the active site area of ibuprofen to increase, so that the half-life of ibuprofen is longer. MCM-41 has a basic composition including cetyltrimethylammonium bromide (CTAB), tetraethyl orthosilicate (TEOS), NaOH, and HNO_3. TEOS, which is the silica source of the MCM-41 component, can be modified by synthesizing rice husk into MCM-41 itself.

The synthesis of MCM-41 with controlled size and porous structure has been carried out at laboratory scale. MCM-41 is the first material analyzed by Mobil Corporation. It features hexagonal mesoporous arrays that can be used for various applications in biosensors, drug loading and other fields that require large surface areas and porous materials. The hexagonal sized pores in MCM-41 are an advantage to this material as it has the ability to perform drug adsorption processes (Amalia et al., 2020).

Adsorption is a state in which a substance (molecule or ion) is adsorbed on the surface of an adsorbent. The mechanism of adsorption is described as the process by which molecules initially present in solution physically attach to the surface of an adsorbent. Molecules are adsorbed when the adhesive force between the adsorbate and adsorbent is greater than the cohesive force of each molecule. The adsorption process occurs at the surface. The more molecules that hit the surface of the sorbent, the more molecules are adsorbed (Wijayanti & Kurniawati, 2019).

In agricultural countries, rice husk waste is very abundant. The current utilization of rice husk is mostly only used as fertilizer. Rice husk waste is one of the wastes that has the largest silica content. The dry weight of rice husk that has undergone complete combustion contains 87%-97% silica. In addition to the abundance of rice husk waste, silica can be obtained from rice husk very simply and at relatively low cost by alkaline extraction (Handayani et al., 2014)

MATERIALS AND METHOD

Materials

The tools used are thermometer, reagent bottle, measuring pipette, beaker, erlenmeyer, spatula, weighing bottle, separatory funnel, oven, magnetic stirrer, bulb, and analytical balance. Other instrumentation equipment are centrifuge (OREGON LC-04R), X-Ray Fluoresence (XRF) (Panalytical Epsilon 3 XLE), X-ray diffraction (XRD) (XPERT PRO PANalytical PW3040/60), Scanning Electron Microscope (SEM) (Hitachi SU-3500), Fourier Transform Infrared Spectroscopy (FTIR) (UATR PerkinElmer Frontier C90704 Spectrum IR Version 10.6.1), and spetrofotometri UV-Vis (THERMO SCIENTIFIC Genesys 50).

The materials used in this study were rice husk, ibuprofen (IOL Chemical), isopropyl alcohol

(Merck), CTAB (Cetyltrimethylammonium bromide) (Merck), Sodium Hydroxide (NaOH) (Merck), Sulfuric Acid (H_2SO_4) (Technical), Nitric Acid (HNO_3) (Technical), and distilled water.

Methods

Preparation of rice husk MCM-41 synthesis

Silica extraction from rice husk was carried out by mixing 80 grams of rice husk with 600 mL of 2 M HNO_3 solution in a beaker and then allowed to stand at room temperature for 24 hours. Then the rice husk was washed with distilled water until pH 7. The washed rice husk was then put into the melting furnace and burned in a muffle furnace at 600°C for 6 hours. As a result of this combustion, rice husk ash was obtained. XRF characterization was then conducted to see the silica content of the rice husk. XRF analysis is carried out to quickly determine the elemental composition of a material/sample (Amalia et al., 2020).

Synthesis of MCM-41 with rice husk

The synthesis of MCM-41 begins with the preparation of Natrium Silicate (Na_2SiO_3) solution based on research (Usgodaarachchi et al., 2021) which has been modified, carried out by dissolving 2 g of rice husk ash with 100 ml of 3.5 M NaOH, then stirring vigorously with a magnetic stirrer for 5 hours at 80°C. The resulting solution that has been obtained is then filtered with Whatman filter paper (No. 1) until there is a colorless thick solution, which is determined as a Na_2SiO_3 stock solution.

The formation of silica MCM-41 was then made by sol-gel technique. The contents of solution A mixing 2.4 g CTAB dissolved in a solution of 25 mL isopropyl alcohol and 25 mL distilled water. Na_2SiO_3 stock solution was added dropwise into solution A, with vigorous stirring using a magnetic stirrer at 60°C until froth was obtained in the solution. Then concentrated H_2SO_4 drop by drop was added to solution A which already appeared foam until it reached pH 4. Stirring with a magnetic stirrer is done to form a gel, then stored for 12 hours at room temperature. Silica was then rinsed with distilled water, then filtered. The silica residue was then dried at 80°C for 12 hours. The results from the oven were then crushed to be calcined at 550°C for 4 hours. (Usgodaarachchi et al., 2021).

Characterization of MCM-41

Characterization of MCM-41 was done by XRD and FTIR testing. XRD is used to identify crystallized or amorphous materials and measure the crystalline compounds formed (Wirawan et al., 2018). SEM was performed to see the morphological structure of the MCM-41 surface (Prasetyo et al., 2013). FTIR is performed to identify compounds, detect functional groups, and analyze the analyzed mixtures and samples (Sari & Fajri, 2018).

Preparation of ibuprofen calibration curve

The preparation of the ibuprofen calibration curve begins with the preparation of a parent solution, namely 1000 ppm ibuprofen in 100 ml isopropyl alcohol. Furthermore, a dilution of the standard solution with a concentration of 300 ppm - 700 ppm in 25 ml of isopropyl alcohol was made. After making the solution, the wavelength was determined, using a concentration of 400 ppm. After obtaining the wavelength, absorbance measurements can be made in spectrophotometry UV-Vis.

Adsorption isotherm of ibuprofen on MCM-41

The maximum adsorption capacity of MCM-41 towards ibuprofen is determined through adsorption isotherms, for that it is necessary to first determine the weight of the adsorbent and the optimum contact time of ibuprofen adsorbed with MCM-41 adsorbent. Based on the modified research (Apriyanti et al., 2018), the determination was carried out by making ibuprofen solutions with concentrations of 400 ppm, 500 ppm, and 600 ppm in isopropyl alcohol mixed with 0.05 g of MCM-41. The mixing results were then stirred using a magnetic stirrer for 30 minutes at room temperature, then measured the absorbance using a Spectrophotometry UV-Vis. To determine the adsorption pattern of ibuprofen solution on the surface of MCM-41 adsorbent, it was determined through

Langmuir and Freundlich adsorption isotherm models. Adsorption isotherms were used to determine the adsorption mechanism of MCM-41 on ibuprofen. Liquid solid phase adsorption belongs to the Freundlich and Langmuir isotherm types. Determination of the use of a suitable adsorption isotherm model for MCM-41 on ibuprofen can be known by looking at the correlation coefficient (R2) which is close to the value of 1 (Nafi'ah, 2016).

This adsorption isotherm process occurs when a single layer is formed, based on the assumption that particle capacity is independent of position or adjacent positions. The Langmuir isotherm was presented in equation (1).

$$W = \frac{a.b.Ce}{1 + b.Ce} \quad \text{.....(1)}$$

The equation (1) can be linearly reduced to equation (2):

$$\frac{Ce}{W} = \frac{1}{a}Ce + \frac{1}{a.b} \text{.....(2)}$$

Description:
W = Adsorption effectiveness (mg/g)
B = Langmuir constant (mg/L)
Ce = Equilibrium concentration (residual concentration)
a = Maximum adsorption capacity or power

This adsorption isotherm process occurs in many layers and shows weak bonding. Adsorption under these conditions is called physical adsorption. This adsorption model shows that multiple layers can form due to the uniform surface of the adsorbent. The Freundlich Isotherm equation can be formulated as equation (3).

$$W = KCe^{\frac{1}{n}} \text{.....(3)}$$

The linear form of the above equation can be changed by taking its logarithmic form (4):

$$Log\, W = Log\, K + \frac{1}{n} Log\, Ce \quad \text{.....(4)}$$

Description:
W = Adsorption effectiveness (mg/g)
K = Maximum adsorption capacity or power (mg/g)
N = Adsorption constant
Ce = Solute concentration in solution after adsorption equilibrium is reached (mg/L)

Kinetics of ibuprofen on MCM-41

The adsorption kinetics of MCM-41 with ibuprofen was evaluated based on the Pseudo First Order (PFO) and Pseudo Second Order (PSO) reaction equations. The treatment to determine the adsorption kinetics was carried out on a solution of ibuprofen with a concentration of 500 ppm in isopropyl alcohol mixed with 0.05 g of MCM-41, then observed through Spectrophotometry UV-Vis with a variation of observation time of 30 minutes, for 2 hours (Nafi'ah, 2016).

PFO modeling describes the adsorption mechanism with a fast rate with a short time span

(Priyanto, 2009). The PFO model can be calculated by the equation (5):

$$In\ (q_e - q_t) = In\ qe - k_t t \quad \text{.....(5)}$$

Description:
q_e and q_t = Amount of ibuprofen absorbed at equilibrium at a given time (mg/g)
k_1 = Pseudo first order rate constant (min^{-1})
t =Time (min)

PSO modeling describes the adsorption mechanism on a relatively long time span with a slow process (Priyanto, 2009). The PSO model can be calculated with the equation (6):

$$\frac{t}{qt} = \frac{t}{k_2 qe^2} + \frac{t}{qe} \quad \text{.....(6)}$$

Description:
q_e and q_t = Amount of ibuprofen absorbed at equilibrium at a given time (mg/g)
K_2 = Pseudo second order rate constant (min^{-1})
t = Time (min)

Data Analysis

Data analysis was carried out on the results of XRD characterization using Origin software. In the SEM characterization results, data analysis was carried out using Images-J, Origin, and Microsoft Excel software. Microsoft Excel was also used to analyze the results of adsorption kinetics and isotherms by looking at linearity (R^2).

RESULT AND DISCUSSION

XRD Characterization Results of MCM-41

In this study, XRD characterization was carried out to determine the composite results of MCM-41 material synthesized with rice husk. The composite results of this characterization can be seen at the 2θ angle read, measurements were taken from the range 2θ = 0°-100° with the diffractogram results shown in Figure 1. The peak of the diffractogram of the read characterization results is 2θ = 25.4993°, which proves that the MCM-41 material forms amorphous crystals with an increasingly regular crystal arrangement. The increasingly regular form of crystal arrangement is caused by the loss of CTAB surfactant. In addition, the loss of CTAB surfactant in the calcination process of MCM-41 synthesis also supports this material to have smaller unit cells (Hasanah et al., 2018).

The results of XRD characterization in the form of diffraction diffractogram peaks show amorphous and hexagonal material curve profiles. The amorphous curve profile is mentioned in research (Izzati et al., 2013) where the amorphous material curve shows a wide peak curve, as well as a hexagonal curve profile. Furthermore, it is mentioned in research (Asral et al., 2022) where the hexagonal structure curve shows long 2θ small peaks, as in Figure 1 with a range of 2θ = 20°-30° showing a wide curve peak. The number of rows of peaks from the characterization results depends on the number of atoms or ions contained in the unit cell of the material. The diffraction pattern of each crystalline solid is very distinctive and depends on the crystal lattice, unit parameters, and X-ray wavelength used.

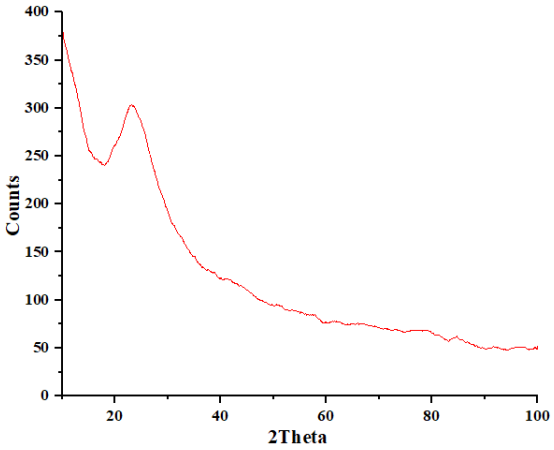


Figure 1. XRD characterization results of MCM-41

The amorphous hexagonal crystal shape in the XRD characterization results shows that the results of MCM-41 synthesis with rice husk are in accordance with the results of MCM-41 synthesis with commercial materials. Hexagonal crystals are characteristic of the shape of MCM-41, then amorphous crystals show that MCM-41 synthesized with rice husks has adsorption ability (Rahayu et al., 2020) .

FTIR characterization results of MCM-41

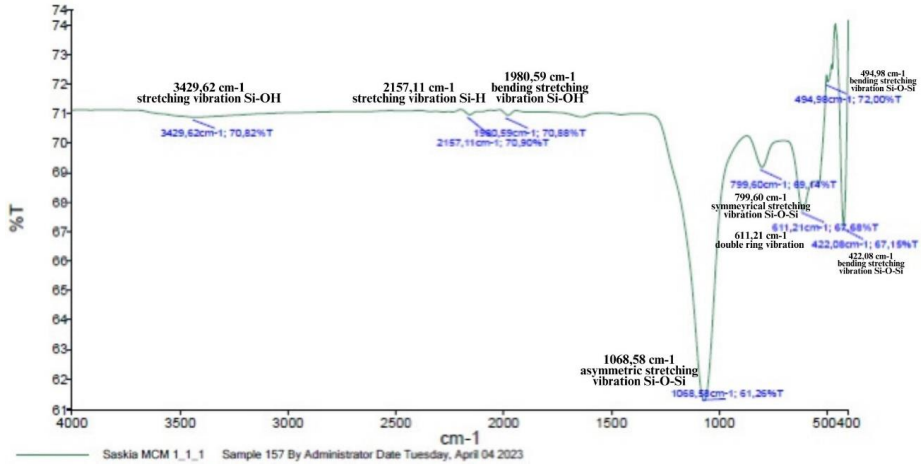


Figure 2. FTIR characterization results of MCM-41

In this study, FTIR characterization was carried out on MCM-41 material synthesized with rice husk, the absorption wave number in this characterization is 400 - 4000 cm^{-1} and the spectrum of the sample results is shown in Figure 2. FTIR results show typical absorption peaks for MCM-41 which has silica content, namely silanol (Si-OH) and siloxane (Si-O-Si). Some typical absorption spectra in silica materials are at 3429.62 cm^{-1} (Table 1) indicating the presence of asymmetric OH stretching vibrations from Si-OH (Rumiyanti et al., 2021). In the 2157.11 cm^{-1} region there is an absorption of Si-H stretching vibrations (Putrinesia et al., 2017). The bending stretching vibration of the Si-OH group of water is seen at 1980.59 cm^{-1} (Rumiyanti et al., 2021). The asymmetric Si-O stretching vibration of Si-O-Si is characterized by a wide and sharp peak at wave number 1068.58 cm^{-1} The same vibration also occurs in the 799.60 cm^{-1} region which indicates the symmetrical Si-O-Si stretching vibration (Hasanah et al., 2018).

Table 1. Vibration result of MCM-41

Wave Numbers (cm^{-1})	Indications
3429.62	Si-OH stretching vibrations
2157.11	Si-H bending vibration
1980.59	Si-OH bending stretching vibrations
1068.58	Si-O-Si asymmetric bending vibration
799.60	Si-O-Si symmetric bending vibration
611.21	Double ring vibration
494.98	Si-O-Si bending stretching vibration
422.08	Si-O-Si bending stretching vibration

In general, based on the FTIR results, the synthesized MCM-41 material with rice husk has a functional group match with MCM-41 synthesized with commercial materials. The results of the absorption peak in MCM-41 material are found at wave number 1068.58 cm^{-1} which shows asymmetric Si-O-Si stretching vibrations and in the 799.60 cm^{-1} region is a symmetrical Si-O-Si stretching vibration.

SEM characterization results of MCM-41

SEM characterization provides results in the form of three-dimensional images. The sample used for characterization in this study is MCM-41 synthesized from rice husk, with the results shown in Figure 3. The results of the characterization in Figure 3 are images of the morphological structure of the sample with a magnification of 1000x and voltage at 10 kV. Magnification and voltage used in characterization affect the clarity of the results.

In Figure 3 there are 2 views of the results seen from the same magnification and voltage, but from different detectors. The thing that distinguishes the image is the condition of the electrons that are not visible in Figure 3 (a) on the backscattered electron (BSE) detector, while in Figure 3 (b) the SE (secondary electron) detector the electrons are very clear. In research (Sujatno et al., 2017) explained the difference in appearance is due to the interaction of most of the electron beam managed to come back out, the electrons are referred to as BSE, a small part of the electrons enter the material then transfer most of the energy to the atomic electrons so that they bounce off the surface of the material, namely secondary electron (SE).

The results of SEM characterization on MCM-41 material synthesized with rice husk are hexagonal crystal morphology shape, where these results show the morphological shape that is in accordance with MCM-41 synthesized with commercial materials. The results of this characterization are then analyzed into digital data using Image-J digital data processing software, origin, and Microsoft excel to obtain particle distribution and size. Based on the results of the origin data analysis in Figure 3 (c), it can be obtained that the particle distribution of MCM-41 is well distributed, because it has R-Square (COD) = 0.99032. The average particle diameter size is analyzed using Microsoft excel, which can be seen that the average particle diameter size is 2.664 μm (Table 2).

Ibuprofen adsorption isotherm results with MCM-41

Quantitative determination of analyte concentrations in samples using chemical instruments can generally be done through calibration curves with acceptable linearity. A calibration curve is a straight-line (linear) graph showing the relationship between the concentration of the working solution, including blank values, and the proportional response of the instrument used (Wardhani & Nurbayanti, 2019).

The calibration curve was made on ibuprofen compounds that produce maximum absorbance at a maximum wavelength of 263.8 nm. Measurement of the wavelength of ibuprofen from isopropanol at 263.8 nm was then used as the basis for the appropriate wavelength to produce a calibration curve. The calibration curve for the relationship between the effect of ibuprofen concentration on absorbance showed linearity with a value of $R^2 = 0.9922$. These results indicate that concentration and absorbance

have a correlation. The linear regression equation follows the formula y = 0.0013 x - 0.0841. The equation has a slope of 1.434 and an intercept of 0.155 as shown in Figure 4

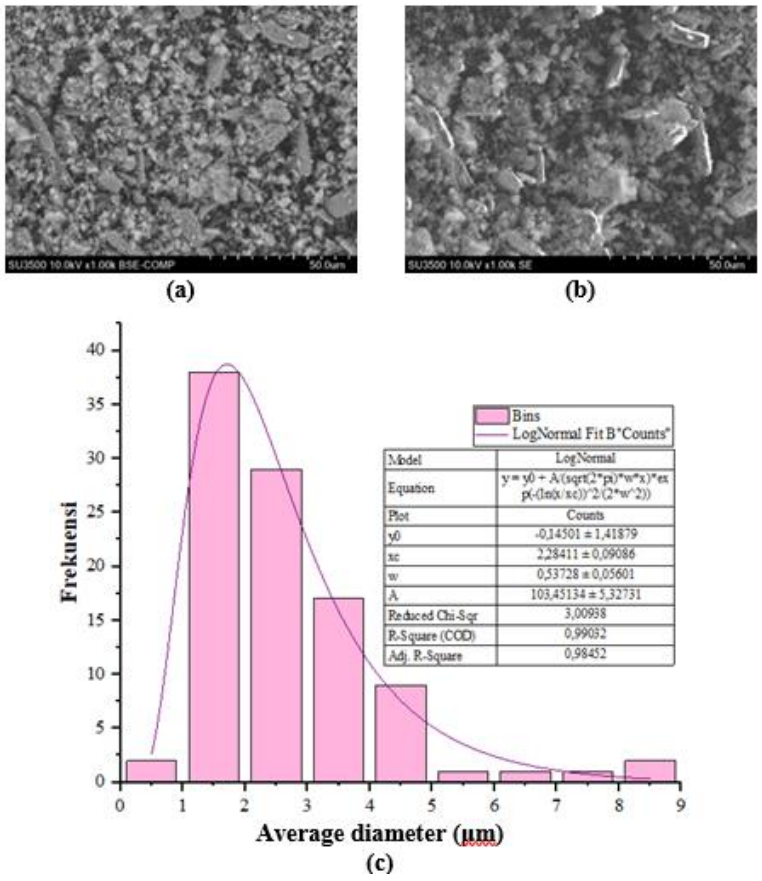


Figure 3. SEM characterization results of MCM-41 (a) detector BSE, (b) detector SE, (c) digital data analysis

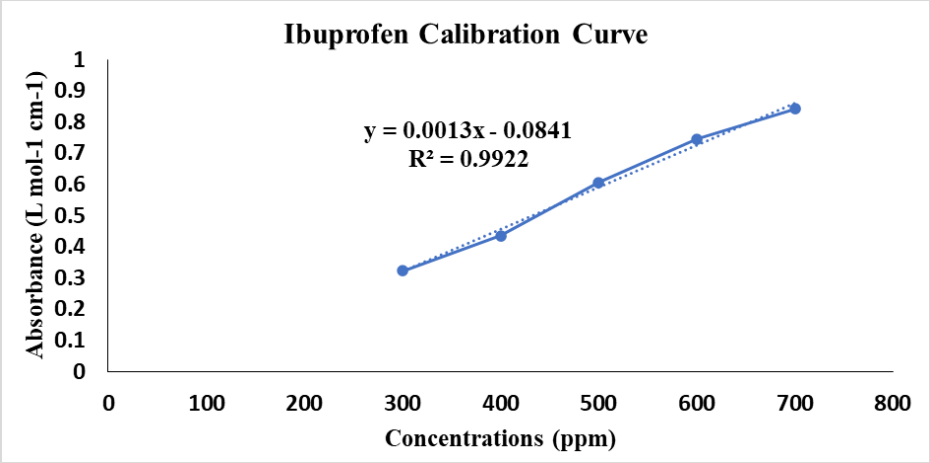


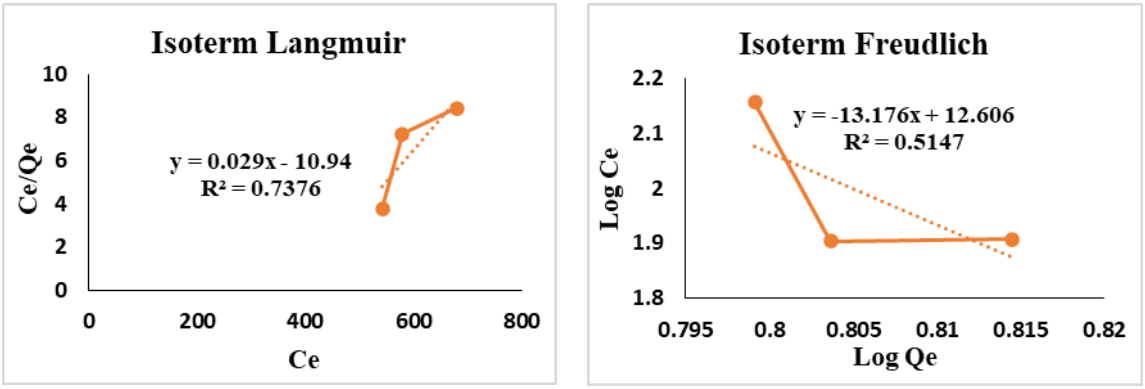
Figure 4. Ibuprofen calibration curve

Table 2. Average particle diameter calculation results

Sample	Average Diameter (µm)
MCM-41from Risk Husk	2.664

Ibuprofen adsorption isotherm results with MCM-41

Based on the calibration curve, the concentration range that can be used for adsorption isotherm testing is in the range of 300 ppm - 700 ppm. In this study, the ibuprofen concentrations used were 400 ppm, 500 ppm, and 600 ppm, while the amount of MCM-41 used was 0.05g. Adsorption isotherm measurements were carried out on UV-Vis spectrophotometry, the results of which can be seen in Figure 5 (a) and (b).



(a) **(b)**

Figure 5. Adsorption isotherm curve (a) Langmuir isotherm, (b) Freundlich isotherm

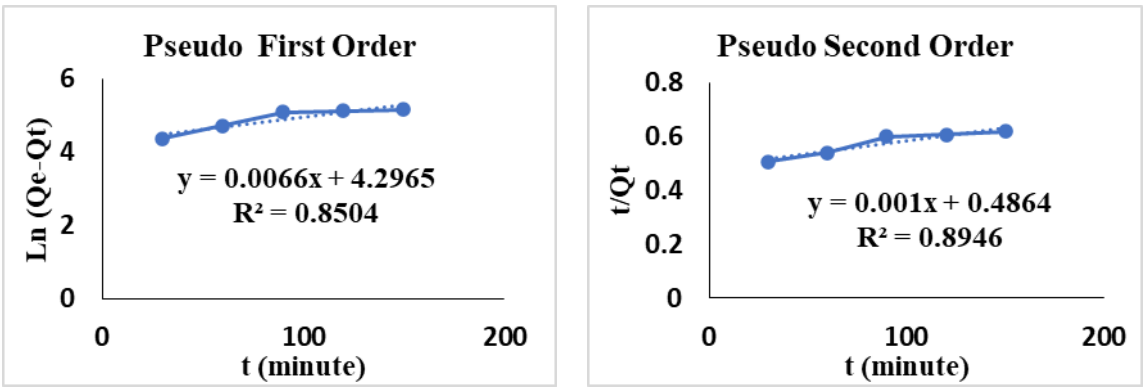
Based on the adsorption isotherm results in Figure 5 (a) and (b), it can be seen that the highest correlation coefficient (R^2) value is the Langmuir isotherm. This indicates that the adsorption of ibuprofen by MCM-41 has a Langmuir isotherm approach, with a maximum adsorption capacity of 34.48 mg/g. In previous research, which discussed the adsorption of tapioca liquid waste by MCM-41, the maximum capacity of absorption was 15.923 mg/g (Darmansyah et al., 2016). So that the adsorption of ibuprofen by MCM-41 synthesized from rice husk is better than the adsorption of tapioca liquid waste by MCM-41.

Langmuir isotherm is an adsorption process that occurs in a chemisorption monolayer. Chemisorption is the adsorption or attachment of molecules, ions or atoms to the surface to prevent the penetration of water molecules (Hasran et al., 2021). According to research (Nurhasni et al., 2014), on the Langmuir isotherm the adsorbent surface has homogeneous active sites that are proportional to the surface area. Each active site can only adsorb one adsorbate molecule so that adsorption will only be limited to the formation of a single layer (monolayer), while the Freundlich isotherm is a process that occurs by physisorption of many layers.

In this study ibuprofen is the adsorbate, and MCM-41 is the adsorbent. Based on the Langmuir isotherm approach, Ibuprofen is adsorbed with MCM-41 irreversibly where the absorbance increases, following the amount of adsorbent addition. The addition of MCM-41 as an adsorbent for ibuprofen, can form ibuprofen to be more stable absorption. In addition, the addition of MCM-41 to ibuprofen can also increase the surface area of ibuprofen (Jamburidze et al., 2019). The large particle surface area causes the particle contact surface with water to be large so that the solubility of particles in water increases (Ferdiansyah et al., 2018).

Results of ibuprofen adsorption kinetics with MCM-41

In this study, the concentration of ibuprofen used was 500 ppm, with the amount of MCM-41 addition used being 0.05 g. Measurement of adsorption kinetics was carried out on UV-Vis spectrophotometry, the results of which can be seen in Figure 6 (a) and (b).



(a) **(b)**

Figure 6. adsorption kinetics curves (a) pseudo first order, (b) pseudo second order

Based on the results of adsorption kinetics in Figures 6 (a) and (b), it can be seen that the highest correlation coefficient (R^2) value is in Pseudo Second Order. This indicates that the kinetics of ibuprofen adsorption by MCM-41 has an approach to the Pseudo Second Order. In previous studies, which discussed the adsorption of methylene green with MCM-41 also showed the same approach, namely Pseudo Second Order (Alardhi et al., 2020).

The Pseudo Second Order adsorbs kinetics model can be used to predict the speed of transfer from the adsorbate solution to the designed adsorbent (Kurniawati et al., 2016). The Pseudo Second Order kinetics model is modeling based on the ability of absorption in the solid phase with the chemisorption mechanism being the controlling factor of adsorption speed (Nuansa & Istyanti, 2013). The Pseudo Second Order kinetics model depends on the adsorption capacity of each solid phase. If it is assumed that the adsorption capacity is proportional to the number of active sites on the adsorbent (Kurniawati et al., 2016).

Based on the Pseudo Second Order approach, ibuprofen adsorption with MCM-41 has a long contact time, following the amount of adsorbent addition (Revellame et al., 2020). This is related to the Langmuir isotherm approach, where the absorbance of ibuprofen also increases in Pseudo Second Order kinetics. The Pseudo Second Order kinetics model is a suitable kinetics model for ibuprofen because the adsorption process can be applied to the process at any time (Revellame et al., 2020).

CONCLUSION

Rice husk that has been activated with HNO_3 has a high silica content so that it can be utilized for the synthesis of MCM-41 which can be used for ibuprofen adsorption. Based on the results of FTIR characterization, MCM-41 material synthesized with rice husk has an absorption peak at wave number 1068.58 cm^{-1} which shows asymmetric Si-O-Si stretching vibrations and in the 799.60 cm^{-1} region is symmetrical Si-O-Si stretching vibrations. In XRD characterization, it is proven that the crystal form of MCM-41 is amorphous hexagonal which has been proven in the analysis where in the $2\theta = 20^o$-30^o area the shape of the curve widens and other small peaks. In the SEM analysis that has been through digital data processing, it is mentioned that the particles are well distributed, with an average particle size of 2.664. Ibuprofen adsorption kinetics model with MCM-41 synthesized with rice husk is pseudo second order model. The adsorption isotherm of Ibuprofen with MCM-41 synthesized with rice husk is Langmuir isotherm, with the maximum capacity of Ibuprofen adsorption with MCM-41 of 34.48 mg/g.

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