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Adsorption and Release of Ibuprofen from Silica-Carbon Composites Based on Rice Husks (*Oryza sativa*) and Banana Peels (*Musa acuminata*)

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Abstract

This study combines silica and carbon into a composite as a controlled drug carrier for ibuprofen. The silica source came from rice husks and carbon from the kepok banana peel because both materials are biomass waste, easy to obtain, and abundant. The synthesis of silica-carbon composites was carried out by a pyrolysis process combining the two materials with some variations of mass ratio between rice husk and banana peel charcoal (1:1, 3:1, 5:1), pyrolysis temperature (500, 600, 700 °C), and pyrolysis time (1.5; 2.0; 2.5 hours). The silica-carbon composites were subjected to the ibuprofen adsorption test with the impregnation method at different concentrations (100, 500, 1000, 1500, 2000 mg/L) and the ibuprofen release test using Phosphate Buffer Saline solution. The results showed that the silica-carbon composite sample with the largest adsorption capacity of ibuprofen was $R_5S_7W_{2,5}$, which was synthesized at a mass ratio of 5:1 for rice husks and banana peels, pyrolysis temperature at 700 °C, and pyrolysis time of 150 minutes. The most well-fitted equilibrium model to use is Freundlich (R^2 mean 0.961), with a maximum ibuprofen adsorption capacity of 176.778 mg/g, relative adsorption constant of 19.430 L/g, an adsorption heat constant of 25.768 J/mol, and adsorption energy constant of 0.046 mol^2/kJ^2.

Keywords: Composite; Banana peel; Rice husk; Drug release; Kinetics.
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1. Introduction

Ibuprofen is one of the most commonly used non-steroidal anti-inflammatory drugs (NSAIDs). Ibuprofen is taken orally to relieve inflammation, fever, and pain by inhibiting cyclooxygenase activity, prostaglandin synthesis and physiological pain signals.[1] Ibuprofen is widely used in therapeutic applications such as patent ductus arthrosis, cystic fibrosis, rheumatoid and osteoarthritis, orthostatic hypotension, fever and headache. This medicine is widely used to treat prophylaxis of Alzheimer's disease, Parkinson's disease, dysmenorrhea, toothache, and breast cancer.[2–5] However, ibuprofen, like other NSAIDs, also has a risk of side effects. The main side effects in the digestive tract can cause mild dyspepsia to gastric bleeding.[6] Besides, ibuprofen also has the disadvantage of a short biological half-life (1-3 hours), reducing the duration of the drug's healing effect.[7]

Several solutions to the problem have been done to improve the administration of ibuprofen drugs, one of which is a controlled drug delivery system using carrier particles as drug carriers. Administration with a controlled drug delivery system can minimize ibuprofen's side effects and drawbacks. It happens because a controlled drug can slow down and control the release and spread of the drug.[8] The delivery method with a controlled drug delivery system is advantageous because it can maintain the optimum drug concentration in the blood with a predictable release rate for a long time to reduce the risk of side effects and increase drug therapy effects.[9]

Many materials have been developed as a drug carrier system, one of which is silica. Silica (SiO_2) is in powder form,

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has non-toxic properties, good mechanical and thermal stability and electrical, chemical, and biological compatibility.[10] Mesoporous silica structure has a pore size of 2-50 nm, facilitating ibuprofen's loading with the size of <1 nm.[11] Research from Numpilai *et al*. (2016) has applied silica as a carrier for the anti-inflammatory drug ibuprofen. The study's results indicated that the release of ibuprofen was relatively slow.[12] Also, silica material has often been used as an adsorbent that is easily combined with other compounds so that it is possible to increase its capacity through engineering and modification.[13]

Silica has many advantages and can be used for applications in the medical field. In this study, the silica used as a composite raw material was obtained from local Indonesian natural materials, namely rice husk agricultural waste. Silica material from plants is found in rice husks with a 90-98% content in sodium silica, which is very potential as a raw material for a silica source.[14] Rice husk is categorized as waste and known to have a silica content of 90-98%. This material is very potential to be used and converted to a more valuable material, because the amount is abundant and its utilization has not been maximized.[15]

Silica tends to have pores with a relatively small surface area of only 173 m^2/g.[16] Whereas, if a material will be used as a drug delivery agent, a material is expected to have a high capacity so that there is no need for a lot of repetition in getting the drug into the human body. In order to improve its capacity, silica can be combined with other materials to form a composite.[17] Silica as an inorganic material, can be combined with organic carbon and form a new compound with a higher adsorption ability because the outer surface is more extensive, reaching 400-500 m^2/g.[18] This study used activated carbon from Kepok banana peel as the other material to make a composite.[19] Activated carbon from Kepok banana peel is known to have a high carbon content of 86.64% in hemicellulose, cellulose, and lignin.[20]

The combination of silica and activated carbon as a composite has been carried out by previous researchers. With the coating method, the results obtained are composites that have a fairly high average capacity but the shape, size of the coating, and the distribution of pore sizes are still uneven. The uneven layer and size can affect the adsorption rate.[21] In this research, the method offered was pyrolysis, a chemical decomposition process of organic matter through a heating process without or with only a little amount of oxygen.[22] The rice husks and banana peels were heated by pyrolysis at a temperature of 500-700 ^oC to combine silica molecules and carbon at high temperatures.[23–26]

The development of drug delivery systems has been carried out a lot. However, there has been no specific discussion about the manufacture of drug delivery systems (with ibuprofen as the drug model) based on the pyrolysis results of a mixture of banana peel and rice husk. In this study, we developed a drug delivery system from a composite made from these two wastes and conducted a characterization and analysis of their drug release ability. Furthermore, the adsorption equilibrium model was simulated to determine the type of adsorption on the silica-carbon composite. The equilibrium models applied in this research were the Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich (D-R) isotherm, model.

2. Materials and methods

2.1 Material preparation

Dried rice husks and banana peels were milled, sieved with 60-mesh sieve tray, and then separately pyrolyzed at 400 ^oC into charcoal. After the pyrolysis process, the charcoal was mashed and sieved with a 100-mesh sieve. The two charcoal types were mixed and homogenized with various mass ratios of 5:1, 3:1, and 1:1 (rice husk charcoal: banana peel charcoal). The results of pyrolysis composites were subjected to an adsorption test for the adsorption capacity of ibuprofen. The analysis was carried out by selecting ibuprofen's adsorption capacity on which composite has the highest maximum capacity. The best ratio sample was followed by pyrolysis at the decided temperature variable (500^{o}C, 600^{o}C, and 700 ^{o}C) for 2 hours. The temperature variation is based on the simulation results of the capabilities of the pyrolysis equipment simulated in previous studies. Furthermore, the sample with the best result was continued with the pyrolysis time variable, which was 1.5 and 2.5 h. The choice of added time for 1.5 and 2.5 h is based on the average uniform temperature that is owned by the pyrolysis equipment used.

Identification of the sample in this study with RxSyWz, R is a ratio variable whereas "x" is a type of ratio variable which can be the number "1" indicating the ratio 1:1, "3" indicating the ratio 3:1, and "5" indicating 5:1. S is a pyrolysis temperature variable while "y" is a type of temperature variation, which can be the number "5", which indicates a temperature of 500 ^{o}C, and "6", which indicates a temperature of 600 ^{o}C, "7", which indicates a temperature of 700 ^{o}C. W is a time variable while "z" is a type of pyrolysis time variable which can be a number "1,5", which indicates a time of 1.5 hours; "2,0" indicates a time of 2 hours; "2,5" which indicates a time of 2.5 hours.

2.2 Material characterization

Characterization of functional groups was carried out on rice husk charcoal, banana peel charcoal, silica-carbon composites, ibuprofen, and silica-carbon composites impregnated by ibuprofen using Fourier Transform Infrared Spectroscopy (FTIR: Perkin Elmer Frontier type). FTIR spectra were obtained with wave numbers 400-4000 cm^{-1}. The crystal structure's characterization was carried out using X-ray diffraction (XRD) on the material of rice husk charcoal, banana peel charcoal, and silica-carbon composite, which aims to determine the changes in the crystal structure before and after the pyrolysis process. The diffraction calculations in the 10-80 at 2θ were obtained with Cu-Kα radiation at ambient temperature. The Surface morphology was characterized using a Scanning Electron Microscope (SEM: PanAlytical type

Xpert 3 Power) on rice husk charcoal, banana peel charcoal, and silica-carbon composites. It aims to determine the pores' surface morphology changes in the sample before and after the pyrolysis process.

2.3 Ibuprofen adsorption

The process of loading ibuprofen on the silica-carbon composite material was prepared by the impregnation method. One hundred grams of the composite were suspended in 25 mL ibuprofen solution with different concentrations (100, 500, 1000, 1500, 2000 mg/L) for two days in closed containers and atmospheric environment by a batch process. Variations were made to obtain the maximum capacity of the composite adsorbent. Preparation of solutions with several concentrations was done by dilution method. The adsorbed liquid was filtered with a whatman filter paper to separate the liquid containing the ibuprofen concentration from the composite solid impregnated with ibuprofen. The liquid was tested for absorbance with a UV-Vis Spectrophotometer (Genesys 10uv: Thermo scientific) at a wavelength of 265 nm, while the composite solid was dried at 90 °C for 2 hours using an oven.

2.4 Isotherm of adsorption

The performance of composite can be determined by the adsorption isotherm data generated from laboratory experiments. The isotherm equilibrium data model can predict the adsorption performance and optimize the adsorption mechanism and adsorbent capacity. Langmuir, Freundlich, Temkin, and Dubbinin-Raduschevic (D-R) commonly used isothermic equilibrium models. The calculation of the equilibrium model uses the Solver program in Microsoft Excel 2013 with a non-linear method.

Langmuir isotherm can be represented by Equation 1, as follow:

$$q_e = \frac{Q_m K_L C_e}{(1+K_L C_e)} \quad (1)$$

Where q_e is the equilibrium of the substance absorbed in the adsorbent (mg/g), C_e is the solute concentration at equilibrium (mg/L). Q_m is the maximum adsorption capacity of the substance in a single layer (mg/g), while K_L is the Langmuir constant (L/mol).

Freundlich isotherm can be represented by Equation 2, as follow:

$$q_e = K_F C_e^{1/n} \quad (2)$$

Where q_e is the equilibrium of the substance absorbed in the adsorbent (mg/g), C_e is the solute concentration at equilibrium (mg/L). K_F is the Freundlich Constant, while n is a heterogeneity factor.

Temkin Isotherm can be represented by Equation 3, as follow:

$$q_e = B_T \ln(K_T) + B_T \ln(C_e) \quad (3)$$

Where q_e is the equilibrium of the substance absorbed in the adsorbent (mg/g), C_e is the solute concentration at equilibrium (mg/L). B_T is the adsorption heat constant (J/mol), and K_T is the equilibrium binding constant (L/mol).

Isotherm D-R can be represented by Equations 4 and 5, as follow:

$$q_e = (Ce)\exp(-K_{ad}\varepsilon^2) \quad (4)$$

Where q_e is the equilibrium of the substance absorbed in the adsorbent (mg/g), C_e is the solute concentration at equilibrium (mg/L), and K_{ad} is the constant D-R isotherm (mol2/ kj2)

Where ε can be known by the equation as follow:

$$\varepsilon = RT \ln\left(1 + \frac{1}{C_e}\right) \quad (5)$$

Where ε is the Polanyi potential, R is the gas constant (8.314 J/mol.K), and T is the absolute temperature (K).

2.5 Ibuprofen release test

The process of releasing ibuprofen from the composites was carried out in a Phosphate Buffer Saline (PBS) solution. A total of 50 g of the dried composite was immersed in 25 mL PBS solution while stirring at 150 rpm. The amount of ibuprofen to be released was determined by taking a solution of 3 mL at each predetermined time interval for 24 hours. The concentration of ibuprofen was obtained by calculating its absorbance at a wavelength of 265 nm on a UV-Vis Spectrophotometer (Genesys 10uv: Thermo scientific).

3. Results and discussion

3.1 Characterization

The sample characterization carried out in this study included functional group analysis using FTIR, crystal structure analysis using XRD, and internal morphological analysis using SEM on silica-carbon composite materials.

3.1.1 Analysis of functional group before and after pyrolysis using FTIR

Functional groups were analyzed before and after the pyrolysis process for banana peel charcoal, rice husk charcoal, and silica-carbon composites.

Figure 1(a) shows the banana peel charcoal spectra, which have a peak point of 1056 cm^{-1}, indicating C-O bonds are present as ether compounds. Another peak point at 1630 cm^{-1} showed a C=C stretching vibration belonging to an aromatic alkene compound, while the peak points of 2865 cm^{-1} and 2930 cm^{-1} were also found in the charcoal spectra of banana peels, both of which indicated the presence of C-H group strapping vibrations from alkane group. Broad peaks in the banana peel charcoal spectra were also found at 3000-3700 cm^{-1}, with peak points at 3424 cm^{-1} and 3741 cm^{-1}, indicating the presence of O-H bonds stretching vibration from the hydroxyl group. It is the same as Tahir *et al*. about the functional groups of banana peels pyrolyzed at a temperature of 300-600 °C.[27]

Figure 1(b) shows the rice husk charcoal spectra. The rice husk charcoal spectrum showed a peak at 1097.09 cm^{-1}, indicating the presence of Si-O-Si asymmetrical stretching vibration. At the same time, the peaks of 468 cm^{-1} and 800 cm^{-1} indicate that the sample has stretching vibration and bending of the siloxy (Si-O) group. Broad peaks occur in the range 3400-3900 cm^{-1} with a peak point at 3738 cm^{-1}, which indicates an O-H bond stretching vibration from the hydroxyl group. The peak point of 3566 cm^{-1} shows the OH group stretching vibration of the hydroxyl group. The spectrum is the same as previous research by Daffalla *et al.*, (2013) who studied the hydrolyzed rice husks at 400 ºC.[28] Both rice husk charcoal and banana peels were mixed with a 5: 1 ratio and pyrolyzed at 700 ºC to produce a silica-carbon composite.

Whereas in Fig. 1(c), which is the spectrum curve of the silica-carbon composite, you can see the characteristic clusters that are owned by banana peel charcoal and rice husk charcoal. The presence of peaks in the wavenumber area of 3424 cm^{-1} and peaks of 1097.09 cm^{-1} indicates that there are O-H groups from banana charcoal and Si-O-Si groups from rice husk charcoal. This indicates that the composite synthesis has been successfully carried out.

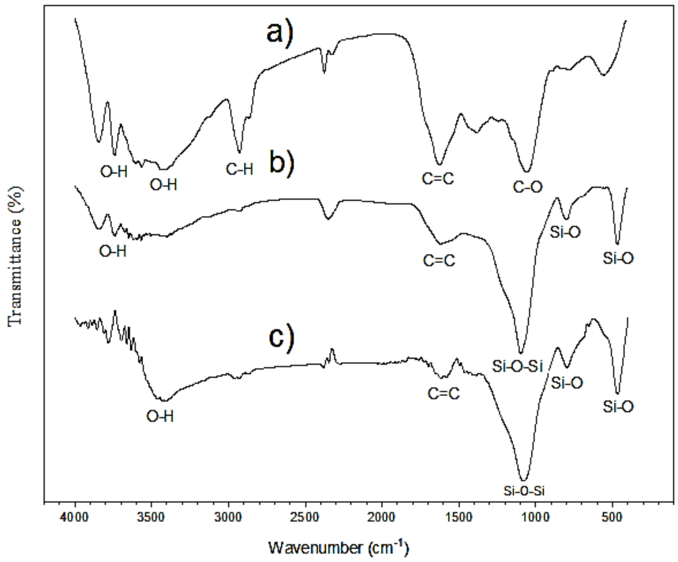

Fig. 1 Results of FTIR spectra are a) banana peel charcoal, b) rice husk charcoal, and c) silica-carbon composite.

3.1.2 Analysis of functional groups before and after adsorption

Analysis of all samples' functional groups was also carried out before and after the adsorption test.

Figure 2(c) shows the ibuprofen spectra, which peak at 1720 cm^{-1}, indicating a COOH group from the carboxylate group. Besides, broad peaks were found at wavenumbers 2400-3500 cm^{-1} with peak points at 2923 and 2956 cm^{-1}, indicating the C–H group stretching vibration of the alkyl group ibuprofen sample. It is the same as the ibuprofen spectra that previous studies have carried out.[29] Furthermore, the characterization of adsorbed ibuprofen in silica-carbon composites can be observed in Fig. 2(b). The resulting spectra show the same characteristics as the carbon silica composite in Fig. 2(a), but in Fig. 2(b), a peak point of 2925.81 cm^{-1} is formed, which indicates the presence of the C-H group stretching vibration from the alkyl group as well as the characteristics of the ibuprofen functional group. It shows that ibuprofen can be adsorbed on the pores of the silica-carbon composite.[30]

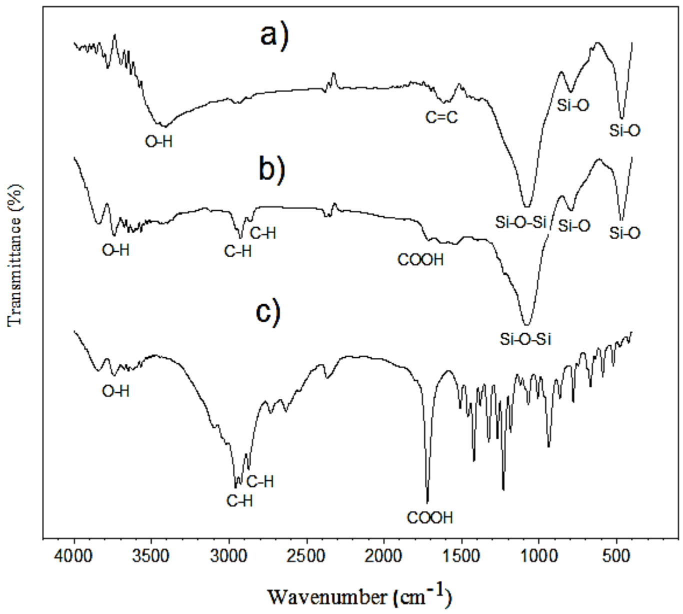

Fig. 2 Results of FTIR spectra a) silica-carbon composite, b) adsorbed ibuprofen from silica-carbon composite and c) ibuprofen.

Also, the peak position of the wavenumber 3406 cm^{-1} was not found, and the peak point appeared at 3741 cm^{-1}. It indicates a hydrogen bond formation between the carboxylate group on the ibuprofen molecule and the hydroxyl group on the surface of the composite (Fig. 3). The hydroxyl group in the composite comes from the content of activated carbon derived from banana peel charcoal. It follows research from Guedidi *et al.*, which makes ibuprofen in activated carbon where the formation of hydrogen bonds between activated carbon and ibuprofen occurred.[19] The reduction in peak points at 467 cm^{-1} and 795 cm^{-1} also shows hydrogen bonds between carboxylate groups on the ibuprofen and SiO_2 molecules on

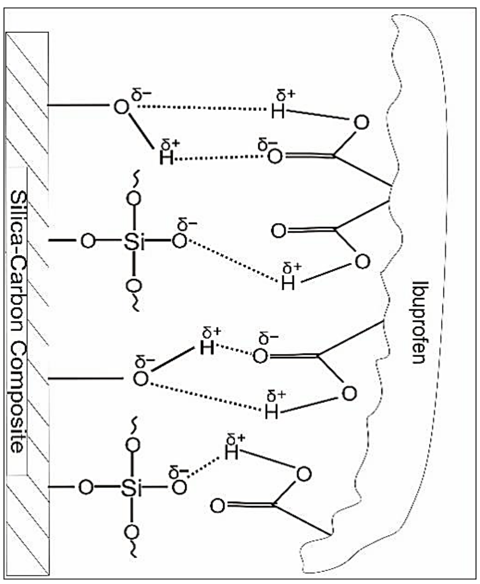

Fig. 3 The hydrogen bond between the ibuprofen functional group and the silica-carbon composite.

the composite surface (Fig. 3). The content of SiO_2 is in the silica in the composite, which comes from rice husk charcoal. It is the same as previous studies that synthesized ibuprofen drug carriers from silica made from rice husks, where the formation of a hydrogen bond between ibuprofen and silica occurred.[12]

3.1.3 Analysis of crystal structure using XRD

Analysis using X-Ray Diffraction (XRD) was aimed to determine the differences in the crystal structure of banana peel charcoal, rice husk charcoal, and silica-carbon composites.

The results of the characterization of rice husk charcoal and silica-carbon composites can be observed in Fig. 4. It showed the XRD diffraction results of banana peel charcoal with the highest diffraction peak at 2θ = 20.59 followed by several smaller peaks at 2θ=16.88; 2θ=14.79; and 2θ=24.55. The diffraction peaks correspond to the semi-crystalline structure of the carbon compound. The diffraction peaks area extends from 2θ=10 to 2θ=60, indicating the presence of carbon in the amorphous phase. The diffraction results are similar to previous studies that tested the crystal structure of banana peels using the XRD instrument. Fig. 4(b) shows the XRD diffraction results for rice husk charcoal, which has a broad peak with the peak point occurring at 3 points, namely 2θ=21.69; 2θ=22.33; and 2θ=23.08. It indicates the presence of SiO_2. The diffraction is similar to previous studies, which showed that pyrolyzed rice husks at 400 °C formed an amorphous silica phase.[31] After both materials were mixed (5:1, rice husk charcoal: banana peel charcoal in the mass ratio) and pyrolysis (700 °C for 2.5 hours), few changes occurred, as shown in Fig. 4(c), there is peak diffraction at the point 2θ=21,87, which indicates that the formation of SiO_2 in the cristobalite phase has begun. Minor peaks were also found at 2θ=28.24 and 2θ = 77.36, which indicates that the quartz-phase SiO_2 formation began. It is the same as previous research, which synthesized silica-carbon composites from the pyrolysis process at a temperature of 600-800 °C.[32] With this data, the pyrolysis treatment and the synthesis of composites can increase the crystallinity of the material. A higher degree of crystallinity would result in a stiffer, harder, and less ductile behavior. Moreover, the surface area and porosity of the material will also decrease. From the results obtained and previous studies, it is known that when the temperature is 700 °C, these crystal peaks are just starting to appear.

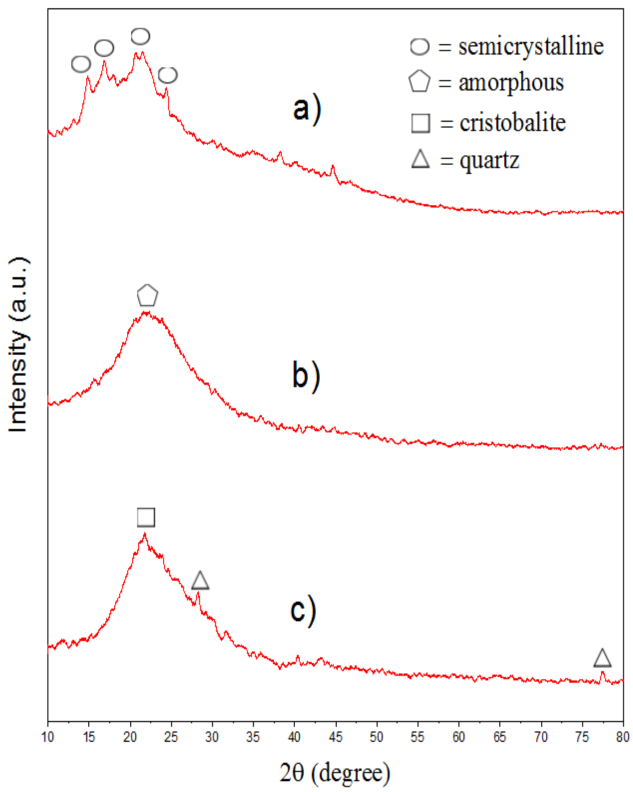

Fig. 4 Results of XRD Diffraction are (a) banana peel charcoal, (b) rice husk charcoal, and (c) silica-carbon composite.

3.1.4 Analysis of surface morphology using SEM

A scanning electron microscope (SEM) analysis was performed to determine the surface morphology of rice husk charcoal, banana peel charcoal, and silica-carbon composite. The operating conditions used in analyzing banana peel charcoal, rice husk charcoal, and silica-carbon composites were at a voltage of 5 kV with a magnification of 5000 times the results shown in Fig. 5.

Figure 5 shows the surface morphology between the samples before and after pyrolysis at a magnification of 5000 times (10 μm). The surface morphology of banana peel charcoal can be seen in Fig. 5(a). It can be seen that the banana peel charcoal still has very few pores on the surface, and most of the surface has not yet formed pores. Fig. 5(b) shows the surface of rice husk charcoal with a few pores. The elongated morphology has common fractures with a slightly smooth surface. It differs from the block shape of a banana peel charcoal with an irregular, bumpy, and rougher surface. After mixing with banana peel charcoal (5:1, rice husk charcoal: banana peel charcoal in the mass ratio) and pyrolysis (700 °C for 2.5 hours), some changes occur, as seen in Fig. 5(c). It can be seen that the surface morphology of the silica-carbon composite has more pores than before it was pyrolyzed. This increase in pore number dan its uniformity means that the surface area of the material also increases and will have an impact on the improved adsorption capacity and properties of the material. The composite surface morphology shows a mixture of two materials: silica from rice husks, a slightly smooth porous surface, and carbon from banana peel charcoal, which has a slightly rough porous surface.

3.2 Adsorption equilibrium model

The isotherm equilibrium data model is essential for predicting and comparing the adsorption performance, optimizing the adsorption mechanism, the adsorbent capacity, and the valuable model of the adsorption system. Adsorption isotherm also represents the relationship between the adsorbate in the surrounding phase and adsorbate adsorbed on

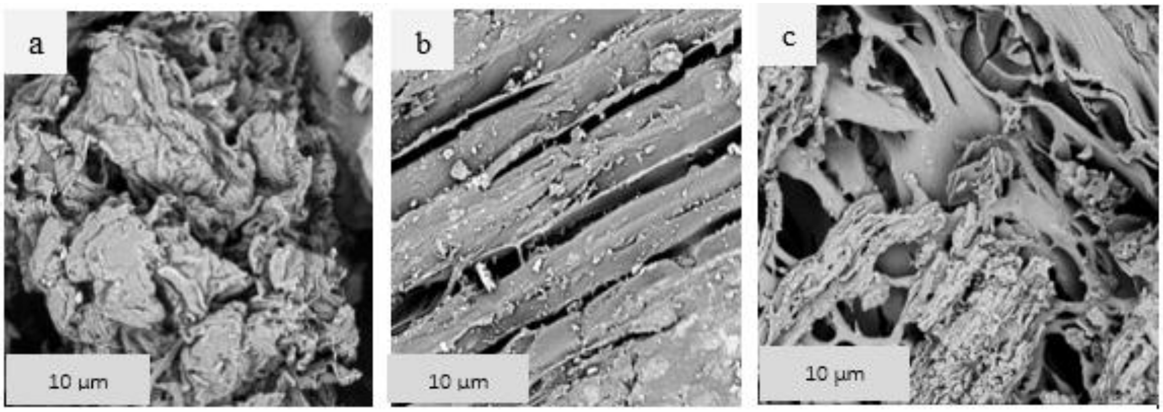

Fig. 5 Results of morphological images with SEM a) banana peel charcoal, b) rice husk charcoal, and c) silica-carbon composites.

the surface of the adsorbent at equilibrium and constant temperature. In addition, it is also significant to study dealing with adsorption techniques. The adsorption equilibrium model was obtained from processing data on variations in the amount of ibuprofen (100, 500, 1000, 1500, and 2000 mg/L). The data were then processed using the adsorption equilibrium model, Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich (D-R) model. The calculation of the equilibrium model used the Solver program in Microsoft Excel 2013 with a non-linear method.

3.2.1 Langmuir isotherm

Langmuir isotherm is used for adsorption based on a single layer (monolayer). Langmuir isotherm type is an adsorption process that chemically combines in an adsorption layer. The Langmuir modelling value is satisfactory if the value of the R_L is between 0 and 1. The R_L value can be obtained by Equation 6.[33]

$$R_L = {}^{1}/_{(1 + K_L C_o)} \tag{6}$$

The result of the Langmuir isotherm calculation is seen in Table 1 and Fig. 6. Based on the calculation of the Langmuir isotherm in Table 1, and the resulting Q_m value is the maximum adsorption capacity of ibuprofen. The K_L value is the Langmuir equilibrium constant, and R^2 is the correlation value between the experimental and equilibrium model correlation data. The R^2 value close to one shows that the correlation data is getting closer to the experimental data.[33] Data Table 1 also shows the R_L value between 0 to 1, which shows that Langmuir modelling in the ibuprofen adsorption process for all composite samples is considered appropriate for adsorption purposes.[34]

Comparing the adsorption capacity of the material ratio variables (banana peel charcoal and rice husks charcoal) can be seen in Table 1. The adsorption capacity value shows that the $R_5S_7W_{2,0}$ sample (5:1 ratio) has a Q_m value of 172.292 mg/g higher than the Q_m value of $R_3S_7W_{2.0}$ (3:1 ratio) of 168.591 mg/g and $R_1S_7W_{2,0}$ (1:1 ratio) of 164.848 mg/g. It shows that the greater the composition of rice husk charcoal to banana peel charcoal, the more significant the adsorption capacity. The main composition of rice husk charcoal is silica, which has a pore surface area of 173 m^2/g. In contrast, the main content of banana peels is carbon, which has a smaller surface area than silica, which is only 81.38 m^2/g.[35] The more significant charcoal composition will increase the pore surface area of the composite. The larger pore surface area can increase the adsorption capacity, so in this study, the 5:1 ($R_5S_7W_{2.0}$) composition had the optimal amount of adsorbed ibuprofen. However, combining the two materials was

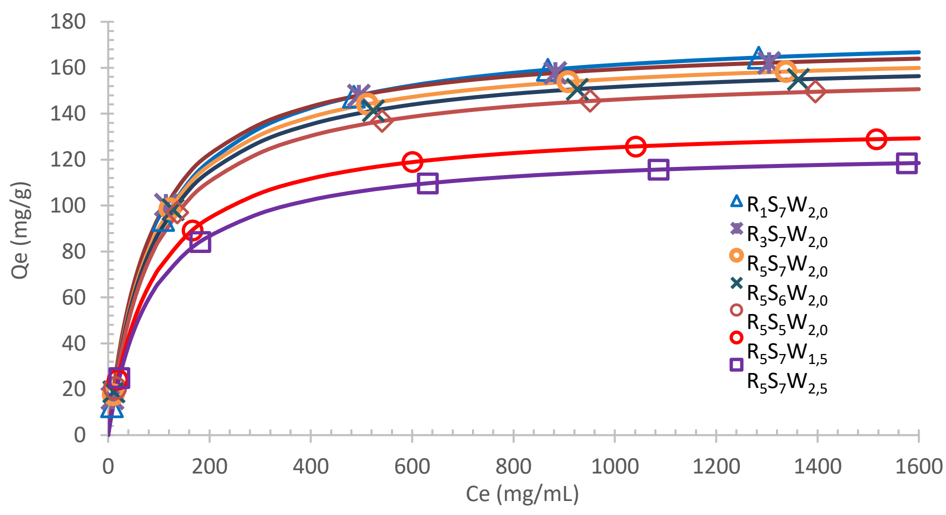

Fig. 6 Comparison of Langmuir isotherms of the ibuprofen adsorption process on composites.

insufficient to increase the adsorption capacity. It can be seen from the adsorption capacity of each component of activated carbon and silica. Guedidi *et al*. showed that activated carbon chemically-physically activated (pyrolyzed at 700 ºC) and adsorbed by ibuprofen showed an adsorption capacity of 146.6-19.7 mg/g, while in silica material, the adsorption capacity of ibuprofen amounting to 152.654-196.574 mg/g. The combination of silica and carbon materials does not increase the number of active sites for ibuprofen, so the adsorption capacity is relatively minor.

Table 1. Langmuir isotherm parameter of ibuprofen adsorption on composite samples.

Parameter	Sample		
	$R_1S_7W_{2.0}$	$R_3S_7W_{2.0}$	$R_5S_7W_{2.0}$
Q_m (mg/g)	164.848	168.591	172.292
K_L (L/mg)	0.011	0.011	0.012
R^2	0.950	0.952	0.951
R_L	0.042-0.466	0.042-0.465	0.039-0.449

Parameter	Sample			
	$R_5S_6W_{2.0}$	$R_5S_5W_{2.0}$	$R_5S_7W_{1.5}$	$R_5S_7W_{2.5}$
Q_m (mg/g)	136.390	125.055	158.899	176.778
K_L (L/mg)	0.011	0.011	0.011	0.010
R^2	0.945	0.945	0.948	0.953
R_L	0.042-0.469	0.042-0.470	0.042-0.467	0.046-0.491

After getting the best ratio variable 5:1 (rice husk charcoal: banana peel), then the pyrolysis temperature was varied at 500 ºC ($R_5S_5W_{2.0}$), 600 ºC ($R_5S_6W_{2.0}$), 700 ºC ($R_5S_7W_{2.0}$). Sample $R_5S_7W_{2,0}$ had a Q_m value of 172.292 mg/g higher than $R_5S_6W_{2,0}$ (136,390 mg/g) and $R_5S_7W_{2,0}$ (125.055 mg/g). It shows that the higher the pyrolysis temperature, the greater the adsorbed ibuprofen. The higher the pyrolysis temperature, the more organic bonds are released from the silica-carbon composite while increasing the volume and surface area of open pores in the composite so that the adsorption capacity increases. Therefore, in this study, the optimal pyrolysis temperature is 700 ºC ($R_5S_7W_{2.0}$) because it has the largest adsorption capacity.

After getting the best pyrolysis temperature variable of 700 ºC, then the pyrolysis time was varied by 1.5 hs ($R_5S_7W_{1.5}$), two h ($R_5S_7W_{2.0}$), and 2.5 h ($R_5S_7W_{2.5}$). Sample $R_5S_7W_{2.5}$ showed the highest Q_m value of 176.778 mg/g, followed by $R_5S_7W_{2.0}$ (172.292 mg/g) and $R_5S_7W_{1.5}$ (158.899 mg/g). It indicates that the length of pyrolysis time affects the ability of the composites to adsorb ibuprofen. The longer the pyrolysis time, the more the physical activation process can increase the volume and surface area of the pores on the composite surface.[36] an adsorbent with the longest pyrolysis time was 2.5 h ($R_5S_7W_{2.5}$) had the largest adsorption capacity of ibuprofen per 1 g of the composite.

From the three variables, the ratio of rice husk and banana peel charcoal, pyrolysis temperature, and time, the highest performance was obtained by $R_5S_7W_{2.5}$ with a Q_m of 17.778 mg/g with an R^2 value of 0.951, which was the closest to one compared to the other. It indicates that the $R_5S_7W_{2.5}$ sample has the best correlation data with the experimental data than other adsorbent samples.

3.2.2 Freundlich isotherm

Freundlich isotherm is used for adsorption, which is based on multilayer with heterogeneous surface types. There is no separation of molecules on the surface after adsorption in this system, and no chemical adsorption occurs. The adsorbate molecule binding is not at a specific site, so there is no stoichiometric limiting factor. Freundlich's model only occurs in physical adsorption because there is no exchange of molecular configurations during adsorption. Adsorption Freundlich model fulfils if the constant (K_f) value is between 1 and 20.The calculation results of Freundlich's isotherm are shown in Table 2 and Fig. 7.

Based on the results of the calculation of the Freundlich isotherm in Table 2, the resulting K_f value is the Freundlich constant indicating the relative adsorption of the adsorbent, and n shows the relative distribution of energy and the heterogeneity of the adsorbate site. R^2 is the correlation value between the experimental and equilibrium model correlation data. The R^2 value close to one shows that the correlation data are getting closer to the experimental data.[37] All K_f values in Table 2 are between 1-20, which indicates that Freundlich's isotherm modelling in the ibuprofen adsorption process on the adsorbent is considered appropriate for adsorption purposes.

Table 2. Freundlich isotherm parameter of ibuprofen adsorption on composite samples.

Parameter	Sample		
	$R_1S_7W_{2.0}$	$R_3S_7W_{2.0}$	$R_5S_7W_{2.0}$
K_f (L/g)	17.366	18.785	17.366
n	3.232	3.214	3.232
R^2	0.963	0.970	0.949

Parameter	Sample			
	$R_5S_6W_{2.0}$	$R_5S_5W_{2.0}$	$R_5S_7W_{1.5}$	$R_5S_7W_{2.5}$
K_f (L/g)	15.701	15.717	16.773	19.430
n	3.725	3.506	3.263	3.219
R^2	0.949	0.949	0.959	0.973

The comparison results of Freundlich's isotherm calculations in Tables 2 and 4.7 show that the greater the ratio of rice husk charcoal to banana peel charcoal, the pyrolysis temperature and pyrolysis time increased the K_f value. It can be seen that the most considerable K_f value of 19.430 occurs in the adsorbent $R_5S_7W_{2,5}$, where the adsorbent has the most significant ratio of husk charcoal (5:1), the highest pyrolysis temperature (700 ºC), and the longest pyrolysis time (2.5 h). The adsorbent, which has a ratio of rice husk charcoal less than 5:1, pyrolysis temperature below 700 ºC, and pyrolysis time less than 2.5 h, has a K_f value of less than 19.430 (L/g). It indicates that the relative adsorption capacity of $R_5S_7W_{2.5}$

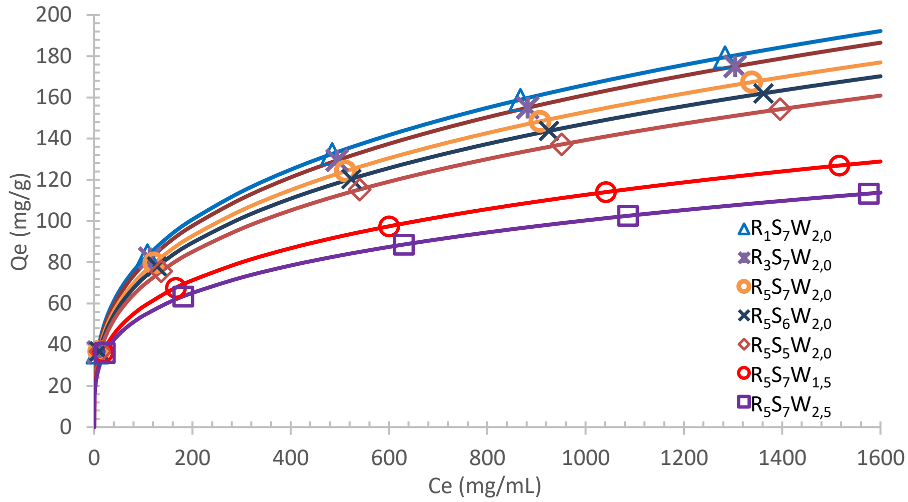

Fig. 7 Comparison of Freundlich isotherms of the ibuprofen adsorption process on composites.

adsorbents is the largest compared to other adsorbents. Also, $R_5S_7W_{2.5}$ has an R^2 value of 0.973, which is the closest to one compared to other adsorbents. It indicates that the correlation fitting data on the $R_5S_7W_{2.5}$ adsorbent is the closest to the experimental data.

3.2.3 Temkin isotherm

The Temkin isotherm is an equilibrium model that considers the interaction between adsorbent and adsorbate regardless of small and large concentration values. This model assumes that the adsorption heat of all the molecules in the layer decreases the more the adsorbent surface is covered. A uniform distribution of bond energy characterizes adsorption.[38] The calculation results of the Temkin isotherm are shown in Table 3 and Fig. 8.

Based on the Temkin Table 3 isotherm calculation, the B_T value is the adsorption heat constant, and K_T shows the binder constant at equilibrium. R^2 is the correlation value between the experimental and equilibrium model correlation data. The R^2 value close to one shows that the correlation data is getting closer to the experimental data.[39]

The comparison results of Temkin isotherm calculations in Tables 3 and Fig. 7 show that the greater the ratio of rice husk charcoal to banana peel charcoal, the pyrolysis temperature and pyrolysis time increased the B_T value. The most considerable B_T value of 25.768 J/mol occurs in the $R_5S_7W_{2.5}$

Table 3. Temkin isotherm parameter of ibuprofen adsorption on composite samples.

Parameter	Sample		
	$R_1S_7W_{2.0}$	$R_3S_7W_{2.0}$	$R_5S_7W_{2.0}$
B_T (J/mol)	22.623	22.623	22.623
K_T (L/mg)	0.278	0.278	0.278
R^2	0.905	0.905	0.905

Parameter	Sample			
	$R_5S_6W_{2.0}$	$R_5S_5W_{2.0}$	$R_5S_7W_{1.5}$	$R_5S_7W_{2.5}$
B_T (J/mol)	21.438	21.438	21.438	21.438
K_T (L/mg)	0.352	0.352	0.352	0.352
R^2	0.909	0.909	0.909	0.909

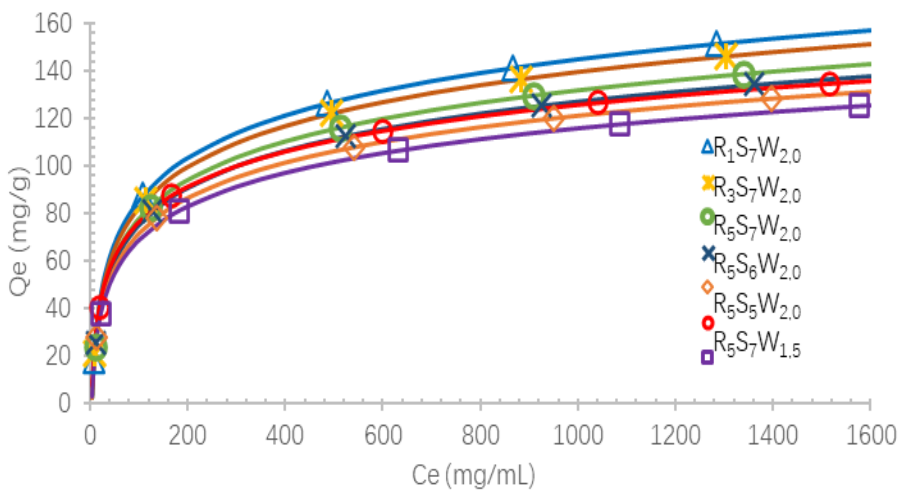

Fig. 8 Comparison of Temkin isotherms of the ibuprofen adsorption process on composites.

adsorbent, which has the most significant ratio of husk charcoal (5:1), the highest pyrolysis temperature (700 ºC), and the longest pyrolysis time (2.5 h) while the adsorbent which has a ratio of rice husk charcoal less than 5:1, pyrolysis temperature is below 700 ºC. Pyrolysis time is less than 2.5 h and has a B_T value of less than 25.768 J/mol. It indicates that ibuprofen's adsorption heat constant on the $R_5S_7W_{2.5}$ adsorbent is the largest compared to other adsorbents. Also, $R_5S_7W_{2.5}$ has an R^2 value of 0.922, which is the closest to one compared to other adsorbents. It indicates that the correlation fitting data on the $R_5S_7W_{2.5}$ adsorbent is the closest to the experimental data.

3.2.4 Dubinin-Radushkevich (D-R) isotherm

The Dubinin-Radushkevich isotherm is generally applied to express the adsorption mechanism through a heterogeneous distribution of causal energy to the surface. This model is a semi-empirical equation in which the adsorption follows a pore-filling mechanism instead of a layer-by-layer form. It assumes a multilayer adsorption process involving Van Der Waal forces, which can be applied to the physical adsorption process. The results of D-R isotherm calculations are shown in Table 4 and Fig. 9.

Table 4. D-R isotherm parameter of ibuprofen adsorption on composite samples.

Parameter	Sample		
	$R_1S_7W_{2.0}$	$R_3S_7W_{2.0}$	$R_5S_7W_{2.0}$
Q_m (mg/g)	22.623	22.623	22.623
б (mol^2/kJ^2)	0.278	0.278	0.278
R^2	0.905	0.905	0.905

Parameter	Sample			
	$R_5S_6W_{2.0}$	$R_5S_5W_{2.0}$	$R_5S_7W_{1.5}$	$R_5S_7W_{2.5}$
Q_m (mg/g)	21.438	21.438	21.438	21.438
б (mol^2/kJ^2)	0.352	0.352	0.352	0.352
R^2	0.909	0.909	0.909	0.909

Based on D-R Table 4 isotherm calculations, Q_m resulting value is the maximum adsorption capacity, and б shows the adsorption energy constant. R^2 is the correlation value between the experimental and equilibrium model correlation data. R^2 value close to one shows that the correlation data is getting closer to the experimental data.

The comparison results of D-R isotherm calculations in Tables 4 and Fig. 9 show that the greater the ratio of rice husk charcoal to banana peel charcoal, the pyrolysis temperature and pyrolysis time decreased the Q_m value. The most considerable value of Q_m of 141.437 mg/g occurs in the $R_5S_7W_{2.5}$, where the adsorbent has the most significant ratio of husk charcoal (5:1). Pyrolysis time is less than 2.5 h and has a Q_m value of less than 141.437 mg/g. It indicates that the maximum adsorption capacity of the adsorbent $R_5S_7W_{2.5}$ is the largest compared to other adsorbents. However, the R^2 value is inversely proportional to Q_m. The higher the ratio of rice husk charcoal to banana peel charcoal, the pyrolysis temperature and pyrolysis time decreases the R^2 value. The R^2 value closest to one occurs in the $R_5S_5W_{2.0}$ adsorbent of 0.922, while the smallest is the $R_5S_7W_{2.5}$ adsorbent of 0.755. It indicates that the best adsorbent on the D-R isotherm is $R_5S_5W_{2.0}$ with a Q_m of 106.099 mg/g because the modelling data is the closest to experimental data.

3.2.5 Equilibrium approach model on ibuprofen adsorption on silica-carbon composites

A comparison of the regression calculations results for the four equilibrium models is shown in Table 5. It can be seen that the average R^2 value of all adsorbent samples in each type of equilibrium model analysis. The comparison of the results shows that the highest average R^2 occurs in the Freundlich isotherm model of 0.961. It indicates that the adsorption process of ibuprofen on the adsorbent sample occurs through physical mechanisms on heterogeneous surfaces and occurs multilayer. The best type of adsorbent sample occurred at a ratio of 5:1 (rice husk charcoal: banana peel charcoal), pyrolysis temperature of 700 ºC, and pyrolysis time of 2,5 hours, which had an ibuprofen adsorption capacity of 176.778 mg/g, relative adsorption constant of 19.430 L/g, adsorption heat constant of 25.768 J/mole, and adsorption energy constant

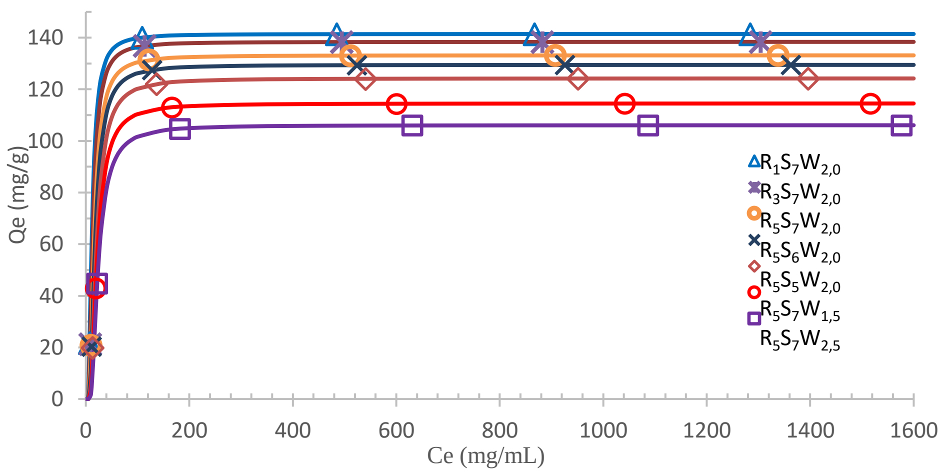

Fig. 9 Comparison of D-R isotherms of the ibuprofen adsorption process on composites.

Table 5. R^2 Value comparison of the equilibrium model.

R^2 Value	Langmuir Isotherm	Freundlich Isotherm	Temkin Isotherm	D-R Isotherm
R^2 Mean	0.949	0.961	0.909	0.809

of 0.046 mol^2/kJ^2.

3.3 Ibuprofen release test from silica-carbon composites

The silica-carbon composite that has passed the adsorption process is then carried out by the ibuprofen drug release test using a 25 mL Phosphate Buffer Saline (PBS) solution. The comparison of the release test for each adsorbent is shown in Fig. 10. In the adsorbent ratio of rice husk charcoal and banana peel ratio, the sample $R_1S_7W_{2.0}$ (ratio 1:1) showed the fastest release than others, where the release of ibuprofen began to reach equilibrium on 16 h as much as 97.183%. In contrast, the $R_3S_7W_{2.0}$ sample (ratio 3:1) at the same time was released 95.236%, which began to reach equilibrium at 18 h. Sample $R_5S_7W_{2.0}$ (ratio 5:1) simultaneously, only 91.354% were released, which began to reach equilibrium on 20 h. It shows that the greater the rice husk charcoal content can reduce the diffusion rate of ibuprofen. Rice husk charcoal has abundant silica, which means that the silica's surface area is greater than the carbon in banana peel charcoal. A larger surface area has a larger volume. The larger volume affects the more significant amount of release to slow the diffusion rate of the release of ibuprofen from the pores on the composite surface to the PBS solution. Therefore, sample $R_5S_7W_{2.0}$ has the best control over the release of ibuprofen compared to other ratio variable samples.

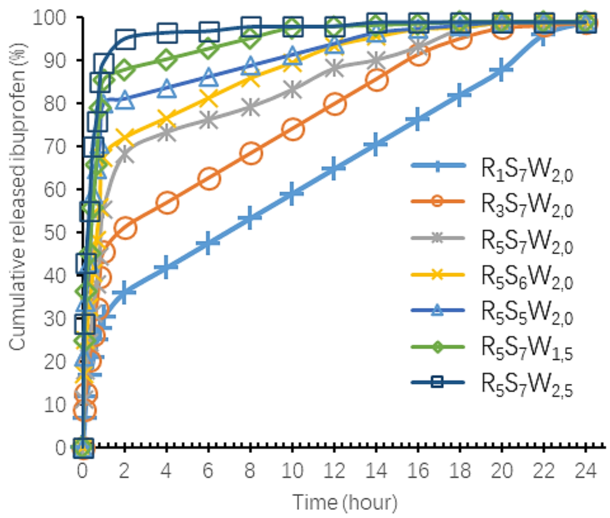

Fig. 10 Dissolution profile of ibuprofen from different composite samples.

In the pyrolysis temperature variable, sample $R_5S_5W_{2,0}$ (temperature 500 °C) showed the fastest release of the others, where the release of ibuprofen began to reach equilibrium on eighth as much as 97.795%. Sample $R_5S_6W_{2.0}$ (temperature 600 °C) at the same time just released as much as 95.043%, which started to reach equilibrium in 10 h, and in the sample $R_5S_7W_{2.0}$ (temperature 700 °C) at the same time, only 53.199% were released, which began to reach equilibrium on 20 h. It shows that the greater the pyrolysis temperature can reduce the diffusion rate of the release of ibuprofen. In the study of Jia *et al*., samples pyrolyzed at a temperature of 700 °C had a larger surface area than samples that were pyrolyzed at a temperature of 500 and 600 °C. The adsorbent, which has a large surface area, also has a large storage capacity.[40] The large storage capacity affects the longer the ibuprofen is released to reduce the diffusion rate of ibuprofen from the pores on the composite surface to the PBS solution. Therefore, the pyrolysis temperature at the most in the sample $R_5S_7W_{2.0}$ had the most controlled ibuprofen release.

The same form of release is shown by research conducted by Lamprecht, et.al (2004). In the initial phase, some ibuprofen will be released rapidly, then gradually reach a constant level until all are released within 24 hours. In the adsorbent pyrolysis time variable, the sample $R_5S_7W_{1.5}$ (time of 1.5 h) showed the fastest release of the others, where the release of ibuprofen began to reach equilibrium in 16 h as much as 97.231%. Sample $R_5S_7W_{2.0}$ (pyrolysis time of 2 h) at the same time, 91.354% of which started to reach equilibrium in 20 h and in the sample $R_5S_7W_{2.5}$ (pyrolysis time of 2.5 h) at the same time only 76.150% were released which began to reach equilibrium in 24 h (98.716%). It shows that the greater the pyrolysis time can reduce the diffusion rate of ibuprofen release. The longer pyrolysis time affects the longer the reaction so that the formation of pores will increase and the volume of the adsorption capacity will increase. More adsorption capacity can slow the diffusion rate of ibuprofen release. Therefore, the most controlled release of ibuprofen was achieved with pyrolysis time on sample $R_5S_7W_{2.5}$.

Based on the three variables, the order of release starting from the best of all adsorbents is $R_5S_7W_{2.5} > R_5S_7W_{2.0} > R_3S_7W_{2.0} > R_1S_7W_{2.0} > R_5S_7W_{1.5} > R_5S_6W_{2.0} > R_5S_5W_{2.0}$. This sequence that the best control release occurs in the $R_5S_7W_{2.5}$ sample obtained with a ratio of 5:1 (rice husk charcoal and banana peel charcoal), pyrolysis temperature 700 °C, and pyrolysis time 2.5 h. The sample has the lowest release control occurs at the ratio of 1:1 (rice husk charcoal and banana peel charcoal), the pyrolysis temperature is 500 °C, and the pyrolysis time is 1.5 hours. The equilibrium release of ibuprofen in sample $R_5S_7W_{2.5}$ occurred at 24 h (98.716%), which means that the release control was able to slow down the drug release rate four times slower than conventional ibuprofen drugs, which only have a half-life time of about 3 h.[41]

4. Conclusions

Based on the ibuprofen adsorption test results, the best ratio of rice husk and banana peel charcoal is 5:1, the best pyrolysis temperature is 700 °C, and the best pyrolysis time is 2.5 h. The higher the ratio of rice husk charcoal to banana peel charcoal, pyrolysis temperature, and pyrolysis time, the greater the adsorption capacity of ibuprofen. Freundlich's model is more appropriate for modelling in this study because the R^2 value is greater than the Langmuir, Temkin, and D-R models, which means the adsorption process of ibuprofen on the adsorbent sample occurs through physical mechanisms on heterogeneous surfaces and occurs multilayer. The best type of adsorbent sample for the ibuprofen adsorption process is $R_5S_7W_{2.5}$, which has an ibuprofen adsorption capacity of 176.778 mg/g, relative adsorption constant of 19.430 L/g, an adsorption heat constant of 25.768 J/mol, and adsorption energy constant of 0.046 mol^2/kJ^2. The best type of adsorbent sample for the ibuprofen release process is $R_5S_7W_{2.5}$, which can control the release of ibuprofen by 98.716% in 24 h or four times slower than conventional ibuprofen drugs.

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Conflict of Interest
There is no conflict of interest.

Supporting Information
Not applicable.

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