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Adsorption of Flagyl on Prepared Ash from Rice Husk

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Abstract

In recent years, it has been evident that searching for alternative methods with low-price and eco-friendly features that produce high-quality adsorbents is in high demand. In the present work, rice husk from Iraqi rice named (Amber) had been used as the primary source to produce rice husk ash (RHA) for the removal of the antibiotic metronidazole (Flagyl) from water. After good drying of rice husk, RHA was obtained at 600 °C using an electric oven. RHA has been investigated using X-ray diffraction (XRD), porosity, and surface area. The adsorption data of the experimental work were optimized to assess Langmuir and Freundlich's constants. The thermodynamic parameters is likely a change in (Gipp's energy ($\Delta G°$), enthalpy ($\Delta H°$), and entropy ($\Delta S°$)). The impacts of increasing temperature on adsorption capacity were investigated using variant temperatures (25, 30, 45,65), and the results revealed that the dynamic adsorption data could be represented by the pseudo-second-order kinetics model. The observed values for the heat of adsorption as well as the free energy were revealed that the adsorption process of Flagyl on RHA favors high temperatures.

Keywords: Amber rice, Flagyl, rice husk, adsorption

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1- Introduction

Data obtained from recent studies have recognized that drug manufacturing discharges and hospital effluents into the environment bypass the toxic concentrations [1, 2]. Numerous methods have been attempted to specify the types of active pharmaceutical ingredients (APIs) that constitute the highest levels of environmental hazards [3, 4]. One of the essential medications with daily uses during hospitalization is antibiotics [5].

Studies have shown that the release of antibiotics into the waterier ambiance could contribute to the origination of bacteria with antibiotic-resistant genes [6, 7]. Metronidazole is a well-known antibiotic related to the metronidazole class, used to treat infections caused by anaerobic bacteria, and protozoa are among the highest use antibiotics [8].

Metronidazole is accused of resulting in phytotoxicity [9, 10]. Recently, several methods have been applied to aid in removing or at least minimizing the hazardous effects of such drugs using more economical materials [11, 12].

RHA has gained noticeable attention due to its effectiveness and being economically acceptable. [13, 14] As a byproduct, RHA has plentiful uses, such as reinforcing materials, fertilizers, fillers, covering and protecting materials, building material constituents, and as a source of silica owing to its high silica content after combustion (approximately 95% silica), as shown in **Table 1** [15].

Table 1. uses of RHA [15]

The form	Commercial uses
As ash	Fertilizer, filler, oil absorbent, covering agent in steel production, adsorbent
As a source of silica	Refractory, cement production
As silica chemicals	Soap, catalysts, silicon, semi-conductor, etc
The thermal yield of rice husk combustion	Activated carbon, xylose

The draining and rivers of vast quantities of impurities wastes (such as plastics, textile, pharmaceutical industries, etc.) into water has been considered the most critical worldwide problem [16].

Globally, adsorption is one of the different methods experimented with to eliminate medical from wastewater and found to be very effective in terms of simplicity and economically low cost [17, 18].

Adsorption is physicochemical processing in which variant types of molecules will depose on a surface [19, 20]. Variant carbons have been obtained from different agricultural and wood wastes, which are widely available and considered environmentally friendly [21-28].

The current study was designed to inspect the preparation and characterization of RHA from Amber rice waste in addition to evaluating the ability of RHA to adsorbed Flagyl (metronidazole) from wastewater.

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2- Experimental Work

2.1. Preparation and Investigation of RHA Characterization

The raw materials, the Iraqi rice husk named (Amber) at the beginning were washed several times with (deionized water) distilled water for removing dust, dirt, and other impurities then it all have been removed, but before that, it will be weighted and then drying process had been applied for 6 hours using the dryer at 100 °C to ensure that any humidity has been removed, dried rice husk was separated to take the weight and placed in diluted acid for 24 hours, After that the drying process was returned to ensure that any humidity has been removed, the resultant had been applied in a furnace at 600 °C for 6 hours to convert a solid RHA. The RHA properties have been checked by XRD, porosity, and BET surface area.
The prepared RHA was investigated using XRD in the Ministry of Industry, and Minerals, the Chemical and Petrochemical Center. Crystallinity was examined while the pores volume of the RHA, as well as the surface area, were determined using Brunauer–Emmett–Teller (BET) method. The later method was performed using surface area apparatus analyzer-Q surf series.

2.2. Adsorption of Flagyl

A 500 mg/L (ppm) stock solution of Flagyl was made by dissolving the calculated quantities of Flagyl in deionized water. The study was established using 500 ml conical flasks by mixing a previously weighed amount of Flagyl with the 1-gram of RHA. An agitation has been applied to the solution at 300 rpm with a stirrer at a pre-specified temperature for every run (25, 30, 40, and 50 °C). After shaking, the mixtures were centrifuged and filtrated. The time interval for reading was (5, 15,25,35,45,60,75,90, and 420 minutes). The Flagyl concentration was measured at 336 nm as the absorbance wavelength by UV-VIS apparatus Recording Spectrophotometer (UV-160A) presented in the Chemical Engineering Department, College of Engineering, University of Baghdad.
The adsorption capacity, which refers to the quantity of the adsorbed Flagyl per weight of the adsorbent (rice ash) at equilibrium (qe, mg/g) was computed using Equation (1). The Flagyl removal trend was determined by Equation (2).

$$q_e = \left(C_o - C_e\right) \times \frac{V}{m} \tag{1}$$

$$\text{Flagyl removal, \%} = \frac{C_o - C_t}{C_o} \times 100\% \tag{2}$$

Where q_e refers to the capacity of equilibrium adsorption of the adsorbent (in mg/g) while C_o , C_t and C_e in (mg/L) represent the initial, at any time, and the final concentrations of Flagyl, respectively. V represents the volume of adsorption, which has the unit of L, and m that has the unit (g) refers to the weight of RHA consumed.

2.3. Kinetics Models and Adsorption Isotherms

The concept of adsorption isotherms demonstrates the tendency of interaction that occurs between the adsorbates and adsorbents. The currently used method of two-parameter isotherms was chosen to illustrate the precise amount of the adsorbed Flagyl on the prepared RHA. The adsorption models that were used were Langmuir (Equation (3)) [29] and Freundlich (Equation (4)) [30].

$$q_e = \frac{q_{\max} K_L C_e}{1 + K_L C_e} \tag{3}$$

$$q_e = K_F C_e^{\frac{1}{n}} \tag{4}$$

Where q_e refers to the adsorption capacity (in mg) of the adsorbate per each gram of adsorbent, C_e is the concentration of Flagyl at equilibrium in (mg/L), q_{max} is the maximum capacity of adsorption to produce a superficial monolayer on the rice ash surface in (mg/g). K_L presents Langmuir coefficient associated with the degree of affinity between the adsorbate and adsorbent in (L/mg), while K_F refers to the Freundlich coefficient, and n refers to the total numbers of multilayers produced.

Two models [31], which are pseudo-first-order (Equation 5), and pseudo-second-order (Equation 6) were performed to show the variation in the capacity of adsorption with time.

$$ln\left(q_e - q_t\right) = lnq_e - k_1 t \tag{1}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e} + \frac{t}{q_e} \tag{2}$$

Where q_e , and q_t (in mg/g) refer to the capacity of adsorption at equilibrium and at any time (t), respectively. The adsorption rates constant k_1 in (1/min) is used for pseudo-first-order, while the adsorption rate constants k_2 in (g/mg. min), and k_3 in (mg/(g.min$^{0.5}$)) are used for pseudo-second order reaction.

By maximizing the correlation coefficient (R^2), all the observational values of the isothermal and kinetics models were calculated numerically. (Equation (7)) [32].

$$R^2 = \frac{\sum_{i=1}^{n}\left(q_{cal} - \overline{q_{\exp}}\right)^2}{\sum_{i=1}^{n}\left(q_{cal} - \overline{q_{\exp}}\right)^2 + \sum_{i=1}^{n}\left(q_{cal} - q_{\exp}\right)^2} \tag{3}$$

In Equation (7), q_{exp} and q_{cal} (in mg/g) refer to the experimental value and computed value (from the model) of the adsorption capacity, respectively. (i) refers to the current number of the experiment, and n refers to the overall numbers of the performed experiments.

2.4. Characterization of Thermodynamic Analysis

The thermodynamic trend of Flagyl adsorption on the prepared rice husk surface was inspected to calculate thermodynamic parameters during adsorption. The alterations in Gibbs free energy ($\Delta G°$enthalpy ($\Delta H°$) and entropy ($\Delta S°$) had been computed by conventional thermodynamic equations (Equation (8) and Equation (9)) using the distribution coefficient (K_d) for determination of adsorption of Flagyl on the surface of the RHA (Equation 10) [23, 33].

$$\Delta G = \Delta H - T\Delta S \tag{4}$$

$$\Delta G = -RT\ln\left(K_d\right) \tag{5}$$

$$K_d = \frac{q_e}{C_e}\left(\frac{m}{V}\right) \tag{6}$$

In Equation (9), R implies the gas constant (which is equal to 8.314 J/mol K), while T (K) refers to the absolute temperature used during the process of adsorption.

3- Results and discussion

3.1. Characterization of RHA

Analysis of the RHA using X-ray diffractograms was presented in Figure 1. The main peak at angles (2θ = 22° refers mainly to silica. However, the measured surface area and pore volume were equal to 148.53 m^2/g and 0.2540 cm^3/g, respectively.

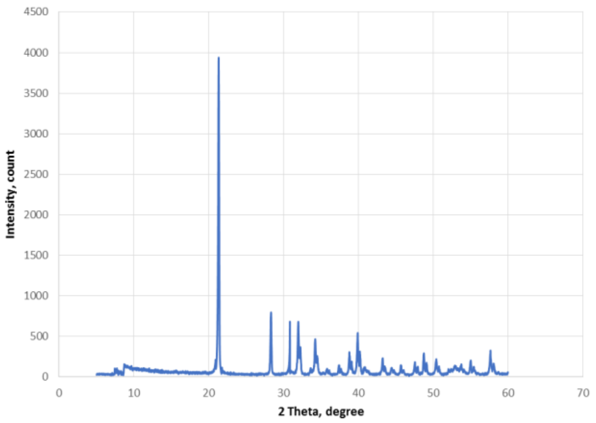

Fig. 1. X-ray diffractogram model of the RHA

3.2. The Pattern of Adsorption of Flagyl on The RHA

a. Equilibrium and adsorption isotherms

The Flagyl removal versus different initial concentrations of Flagyl was represented in **Table 2**. The Flagyl (organics) removal increased with the decreasing of initial concentration.

The increase in the removal values of Flagyl molecules accompanied by the decrease in the initial concentration could be explained by the presence of competition of Flagyl molecules on the most predominant sites of adsorption, which are the pores and the surface of the prepared adsorbents, and this competition decreases when lower Flagyl concentrations were used [34].

Table 2. Flagyl removal versus initial concentrations

C_o, ppm	503	389	308	207	72	51
Removal%	57.85	60.67	66.23	69.57	73.61	76.47

Fig. 2 showed the equilibrium adsorption capacity of RHA increased from 39 to 291 mg/g with an increment in the equilibrium concentrations from 12 ppm (obtained at a lower initial concentration of Flagyl) to 212 ppm.

These data were used to solve and obtain via the nonlinear regression analysis, the Langmuir and Freundlich isotherms, and solution results were summarized in **Table 3**.

The resultant data revealed that the manufactured rice husk possesses a maximum capacity of adsorption with the amount of Flagyl equal to 588.24 mg for each one gram of the produced rice husk.

The elevated value of $R^{2,}$ which was (0.9940), indicated that the Langmuir model best suits the data of the adsorption equilibrium and the Flagyl adsorbed on the RH surface as a monolayer.

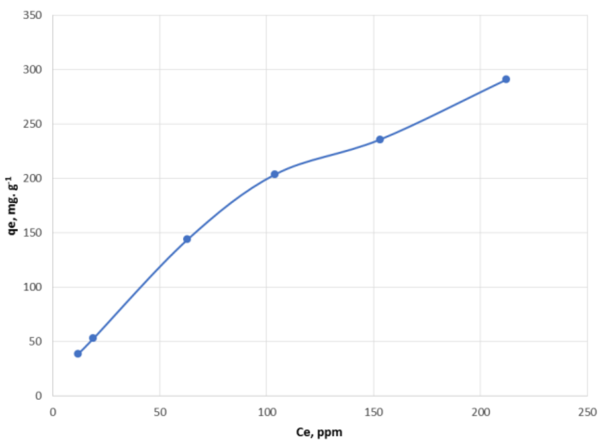

Fig. 2. Flagyl concentration equilibrium effect on the equilibrium capacity of the prepared rice husk

Table 3. Adsorption models constants and the correlation coefficients (R^2) for the Flagyl removal pattern on the papered rice husk

Isotherm model	parameters	Value of Parameter	R^2
Langmuir	q_{max}, mg/g	588.24	0.9940
	K_L, l/mg	0.0060	
Freundlich	K_F, $mg^{1-n}\ l^n/g$	1.59	0.9577
	n, -	8.88	

As shown in Figure 3, the removal of Flagyl was raised obviously in the first hour of the experiment because the pores of the prepared rice husk were empty at the beginning. The significant difference in concentrations led to the rapid transfer of Flagyl molecules from liquid bulk to the rice husk surface. In the second period (up to 180 minutes), the removal increased gradually because of the decreasing of Flagyl concentration that caused a decrease in the transfer driving force, and the pores were filled with more Flagyl that adsorbed on the RHA surface, and then competition increasing between Flagyl molecules to find an empty site.

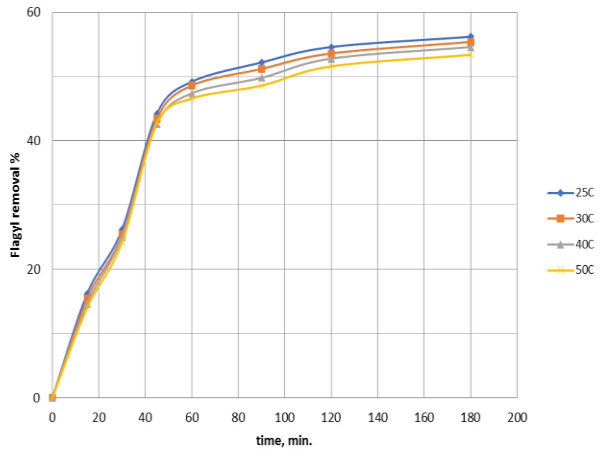

Fig. 3. Relationship between time and the pattern of Flagyl removal via adsorption process on RHA

The kinetic analysis obtained from the adsorption of the Flagyl on the rice husk was summarized in **Table 4**.

The resulted values of the R^2 indicated that the pseudo-second-order model (Equation 6) was well expressed in the data of the experiment.

The R^2 data for the pseudo-second-order model ranged from 0.9533 to 0.9658, which demonstrated a significant and precise description of the resultant data of the experiment with the pseudo-second-order model.

Table 4. Adsorption constants and correlation coefficients (R^2) for the reduction of COD on the papered rice husk

Adsorption kinetic model	Model rate constant	Model parameter value at temperature			
		25 °C	30 °C	40 °C	50 °C
Pseudo-first order	k_1, (1/min)	0.0205	0.0168	0.0143	0.0122
	q_e, (mg/g)	208.0961016	193.0791098	191.464038	185.6754023
	R^2	0.9397	0.8999	0.878	0.8344
Pseudo-second order	k_2, (g/mg.min)	0.025431426	0.02417962	0.022988506	0.023144453
	q_e, (mg/g)	357.1428571	357.1428571	357.1428571	344.8275862
	R^2	0.9658	0.9614	0.9596	0.9533

Table 5 showed that the thermodynamic process of Flagyl removal had a positive value of ΔH (equal 1827.3 J/mol) which means that the Flagyl adsorption increases as the temperature of the experiment decrease.

Additionally, the increasing values of ΔG with the temperatures imply that the adsorption favored a reduction in temperatures.

Furthermore, the resultant observations of negative values ΔG with the positive value of ΔS (which is equal to 8.70 J/ mol K) refer to the spontaneous adsorption of Flagyl molecules on the prepared RHA.

Table 5. Adsorption thermodynamic parameters and correlation coefficients (R^2) for the reduction of COD on the papered rice husk

Temperature, °C (K)	K_d	Thermodynamic parameters			R^2
		ΔG, J/mol	ΔH, J/mol	ΔS, J/mol K	
25	1.358	-759.1	1827.3	8.70	0.9944
30	1.381	-813.1			
40	1.415	-904.2			
50	1.439	-977.4			

4- Conclusion

In the recent few decades, there has been a big deal of attention to produce adsorbents characterized by high adsorption capacity while minimizing the cost of the production and impurities associated with the production process. The present work inspected the efficiency of RHA as an adsorbent to remove metronidazole (Flagyl) molecules. The current study results revealed that the percent of removal of Flagyl was inversely proportional to their initial concentrations, i.e., the percent of removal of Flagyl increased as the initial concentration was decreased. Additionally, RHA equilibrium adsorption capacity was directly proportional to the equilibrium concentration. Moreover, the removal of Flagyl molecules was significantly rapid in the first hour of the adsorption process. The adsorption process occurred due to the emptiness of the RHA surface, leading to a less competition effect than the second period of the process when the concentration of Flagyl was gradually increased. Furthermore, the thermodynamic analysis results indicated the inverse relationship between the temperature of the process and the adsorption capacity, with the positive value of ΔH (1827.3 J/mol).

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امتزاز الفلاجيل على سطح الرماد المحضر من قشور الرز

حيدر عباس و عمارصالح عباس

جامعة بغداد/كلية الهندسة

الخلاصة

مؤخرا أصبح من الواضح البحث عن الطرق الرخيصة والصديقة للبيئة في عمل البحوث والسعي للحصول على أحسن النتائج ومن ضمن المهمة الامتصاص باستخدام مواد متوفرة و رخيصة. في هذا البحث تم استخدام قشور الرز في صناعة رماد قشور الرز بمواصفات عالية لامتصاص المادة الفلاجيل بعد تجفيف قشور الرز يتم عملية الحرق باستخدام الفرن بدرجة حرارة 600 درجة مئوية. بعد الحصول على الرماد تم اجراء فحوص لمعرفة قابلية المادة للامتصاص وتكوين فكرة عن خواصها مثل فحص حيود الاشعة السينية ومعرفة الفراغات البينية والمساحة السطحية للعينات. وتم استخدام طرق المعايرة للنتائج لمنحنيات لونكماير وفراندلج ومعرفة احسن النتائج حيث كانت الافضل باستخدام معادلات الدرجة الثانية و عندما تكون درجات الحرارة عالية حيث استخدمت درجات الحرارة (25,30,45,65) و تم حساب الانثالبية و الانتروبية و طاقة كبس ورسم العلاقات لتوضيح فعالية المادة في الامتصاص.

الكلمات الدالة: رز العنبر، فلاجيل، قشر الرز، الامتزاز