

Batch Studies of Adsorption of Phenol from Aqueous Solutions Using Rice Husk Ash

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Abstract- The present work involves an investigation of the possible use of rice husk ash (RHA) for phenol removal from aqueous solutions. The study was realized using batch experiments. During the experimental part of this study, the effect of system variables such as burning temperature (400°C and 600°C), burning time (1-4hrs), adsorbent dosage (1, 5 and 10 g^l⁻¹), pH(2-10), temperature (23°C and 40°C), and initial phenol concentration(10-1000mg^l⁻¹) on phenol removal were investigated. In addition, adsorption kinetics and adsorption isotherm studies were also carried out. The results show that RHA600 has less removal efficiency compared to RHA400 at 1,2,3 and 4hrs burning times. The investigation of pH effects indicates that maximum phenol removal can be obtained at pH 4 using RHA400, 1h. It was found that, the sorption capacity increased from 0.97 mg g⁻¹ to 46.19 mg g⁻¹ by increasing the initial phenol concentration from 10 mg^l⁻¹ to 1000 mg^l⁻¹ using RHA400, 1h; while a slight decrease on phenol adsorption capacity was observed as the temperature was increased from 23°C to 40°C. The adsorption isotherms are best fitted by the Freundlich model, while the adsorption kinetics is well described by Elovich model.

Keywords: Phenol removal; Adsorption; Rice Husk; Sorption kinetics; Isotherm; Wastewater Treatment.

I. INTRODUCTION

The presence of hazardous chemicals in the environment continues to be an important concern. The demand for more stringent control and protection of our water resources from pollution has mounted steadily in recent decades [1]. Phenol, a class of organic compounds, has been known as a common and hazardous contaminant in water environment. Phenol constitutes the 11th of the 126 chemicals, which have been designated as priority pollutants by the U.S Environmental Protection Agency (USEPA)[2]. The content of phenols in industrial wastewater is usually in the range of 0.1-6800 mg^l⁻¹[3]. The allowable discharge limit for phenol is 0.1 mg^l⁻¹ and 0.001 mg^l⁻¹ (Standard A) set by the USEPA and the Malaysia Environmental Quality Act (MEQA), 1974 respectively [4]. Human consumption of phenol-contaminated water can cause severe pain leading to damage of the capillaries and ultimately causing death.

Various methods such as microbial degradation, adsorption, chemical oxidation, incineration and solvent extraction have previously been used for removal of phenol from wastewater [5-7]. Among those methods, adsorption process has been identified as the best for removing phenolic compounds from aqueous streams. The efficiency of the adsorption process is mainly determined by the characteristic of the adsorbent such as high surface area,

high adsorption capacity, microporous structure and special surface reactivity. To the present day, activated carbon is the most widely used adsorbent because it has good capacity for adsorption of phenol from wastewater [8-10]. In spite of this, it suffers from a number of disadvantages. Activated carbon is expensive, and the higher the quality the greater the cost [11]. Consequently, there has been a growing interest in developing and implementing various potential adsorbents for the removal of phenol from water, and researchers are always in a hunt for developing more suitable, efficient, cheap and easily accessible types of adsorbents, particularly from the waste materials.

The abundance and availability of agricultural by-products make them good sources of cheap raw materials for natural adsorbents. Rice husk, an agricultural waste, has been reported as a good adsorbent for many metals and basic dyes [12,13]. According to the statistics compiled by the Malaysian Ministry of Agriculture, there are more than 408,000 ton of rice husk produced in Malaysia annually [14].

Rice husk consists of 32.24% cellulose, 21.44% hemicelluloses and 21.34% lignin [14,15]. The cellulose, hemicellulose and lignin forming a very stable matrix structure. The inner surface of rice husk is smooth, and may contain wax and natural fats that provide good shelter for the grain. On the other hand, the presence of these impurities on the inner surface of rice husk also affects the adsorption properties of rice husk, both chemically and physically [16]. Therefore, the removal of these impurities can be expected to improve the adsorption properties of rice husk.

To upgrade or improve the adsorption properties, thermal treatment of rice husk has been established. In practice, the type of ash varies considerably according to the burning temperature and burning time as these parameters affect the porosity and functional groups of the ash produced [17]. Rice husk ash (RHA) is produced from the decomposition of rice husk (RH) by heat treatment and the decomposing rice husk is converted into small particles with large surface area upon extended heating. Different heating conditions produce rice husk ash containing different composition of carbon and silica.

This paper presents a discussion on the comparison of rice husk ash samples and recommends a feasible method to produce rice husk ash as adsorbent for the removal of phenol from artificial wastewater. Factors affecting sorption, such as pH, adsorbent dosage, temperature and initial phenol

concentration were investigated. Kinetic and isotherm studies were also conducted. The effects of surface functional groups that exist on the adsorbent's surface on phenol adsorption were also investigated.

II. MATERIAL AND METHODS

A. Preparation of adsorbents

The raw material, rice husk (RH), was obtained from a nearby rice mill. The rice husk was washed thoroughly with distilled water to remove adhering soil and clay, and then dried in air at 105°C in an oven for 24 hrs. Samples of rice husks were converted into an adsorbent-rice husk ash (RHA) by heat-treatment using ceramic crucibles at 400°C (RHA400) and 600°C (RHA600) for 1, 2, 3 and 4hrs in a muffle furnace (Sf 14).

B. Characterization of Adsorbents

The adsorbents were characterized in terms of morphological characteristics using Scanning Electron Microscope (SEM-EDX, model LE01430VP). In addition, the functional groups present on the adsorbent's surface were determined using a Fourier Transform Infrared spectrophotometer (FTIR, model 8400S). The spectra range chosen was from 4000 to 400 cm⁻¹.

C. Batch Adsorption Studies

Batch adsorption experiments were performed using adsorbent dose of 10 g l⁻¹. The sorption study was performed using a mechanical shaker for a period of 24hrs at 195 rpm and ambient temperature. The phenol sample 100 ml of 100 mg l⁻¹ at normal pH was placed in a 250 ml glass Erlenmeyer flask and the flask was attached to the shaker. The suspensions in all sorption assays were filtered to remove any suspended adsorbent. The initial and final concentrations of phenol were determined by HPLC (Agilent 1100 series) equipped with a Diode Array Detector (DAD). The components were separated on a Zorbax SB C18 column (3×250mm). The effect of pH (2-10) was studied by adjusting the pH of phenol solution using dilute HCl and NaOH solutions at ambient temperature. For kinetic studies, the batch experiment was repeated at different periods using 1 g l⁻¹, 5 g l⁻¹ and 10 g l⁻¹ of adsorbents at normal pH and ambient temperature. The effect of initial phenol concentration (10, 50, 100, 300, 500 and 1000 mg l⁻¹) was studied at normal pH and ambient temperature.

The percentage of phenol removal, and equilibrium adsorption uptake, q_e(mg g⁻¹) were calculated using the following relationships:

$$\% \text{ Removal Efficiency} = \frac{(C_0 - C_t)}{C_0} \times 100 \quad (1)$$

$$\text{Amount Adsorbed } (q_e) = \frac{(C_0 - C_e)V}{W} \quad (\text{mg of adsorbate / g of adsorbant}) \quad (2)$$

Where, C₀ and C_e (mg l⁻¹) are the initial and equilibrium liquid-phase concentrations of phenol, respectively, C_t (mg l⁻¹) is the concentration of phenol at time t, V is the volume of the solution (l) and W is the mass of dry adsorbent (g).

For kinetic studies, the adsorption data were analyzed using three different kinetic models namely pseudo first-

order, pseudo second-order and Elovich model (Table 1), while the equilibrium data was analyzed using commonly known adsorption isotherms, the Freundlich model and Langmuir model (Table 2).

In order to evaluate the fitting of the experimental data and the prediction accuracy of the models utilised in the present work, normalized standard deviation is employed, Δq_e (%), which is defined as [18]:

$$\Delta q_e(\%) = 100 * \sqrt{\frac{\sum[(q_{e,\text{exp}} - q_{e,\text{cal}})/q_{e,\text{exp}}]^2}{(N - 1)}} \quad (3)$$

Where N is the number of data points, q_{e,exp} and q_{e,cal} (mg g⁻¹) are the experimental and calculated equilibrium adsorption capacity values, respectively.

TABLE 1
KINETIC EQUATIONS USED IN THIS STUDY

Kinetic models	Equation	Parameters	Ref.
Pseudo-first-order	$\ln(q_e - q_t) = \ln q_e - k_1 t$	q _e (mg g ⁻¹): equilibrium adsorption capacity q _t (mg g ⁻¹): amount of adsorbate adsorbed at time t (min) k ₁ (min ⁻¹): pseudo-first-order rate constant	[18]
Pseudo-second-order	$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t$	k ₂ (g mg ⁻¹ min ⁻¹): pseudo-second-order rate constant	[19]
Elovich	$q_t = \frac{1}{b} \ln(ab) + \frac{1}{b} \ln(t)$	a (mg g ⁻¹ min ⁻¹): initial sorption rate constant b (g mg ⁻¹): desorption constant	[18, 20]

TABLE 2
ISOTHERM EQUATION USED IN THIS STUDY

Isotherm models	Equation	Parameters	Ref.
Freundlich	$q_e = K_F C_e^{1/n}$	q _e (mg g ⁻¹): equilibrium adsorption capacity K _F (mg ^{-1/n} l ^{1/n} g ⁻¹): Freundlich constant n: Freundlich constant	[18,20,21]
Langmuir	$q_e = \frac{q_m K_L C_e}{1 + K_L C_e}$	q _m (mg g ⁻¹): the maximum adsorption capacity K _L (l mg ⁻¹): Langmuir adsorption constant, adsorption equilibrium constant	[18,20,21]

III. RESULTS AND DISCUSSIONS

A. Scanning electron microscopy (SEM)

SEM enables direct observation of the changes in the surface microstructures of rice husk due to modification after heat treatment (Figs. 1a-c). Different pore sizes could be observed on the surface of RHA400 compared to the untreated rice husk. According to the micrograph, the pores

were developed from the decomposition of RH structure by heat. On the other hand, the micrograph corresponding to calcination at high temperature (600°C) shows the loss of micropore volume, possibly due to pore collapse (Figs. 1d-e)

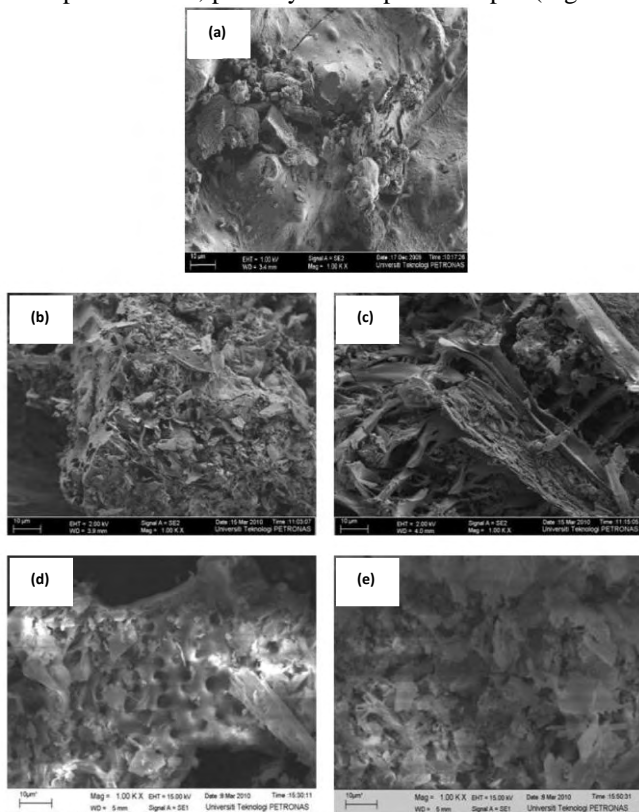


Figure 1. SEM for (a) RH, (b) RHA400,1h, (c) RHA400,4hrs, (d) RHA600,1h and (e) RHA600, 4hrs magnified 1000 times.

B. FTIR spectroscopy of rice husk and rice husk ash

The chemical structure of adsorbent is of vital importance in understanding the adsorption process. The FTIR technique is an important tool to identify the characteristic functional groups, which are instrumental in adsorption of phenol. Table 3 presents the fundamental frequencies of RH and RHA, and their respective possible band frequencies in the FTIR spectrum. The FTIR spectra of rice husk and rice husk ash are shown in Figs. 2(a-b).

As seen in this figures, the adsorption peak around 3404.31 cm^{-1} indicates the existence of free hydroxyl groups. In RH (Fig 2a), the C-H stretching vibration around 2925.81 cm^{-1} indicates the presence of alkane functional group. The peaks around 1641.31-1737.74 cm^{-1} correspond to the C=O stretching that may be attributed to the hemicelluloses and lignin aromatic groups [20]. The C=C stretching vibrations between 1546.80-1652.88 cm^{-1} are indicative of alkenes and aromatic functional groups.

The peaks around 1461.94 cm^{-1} indicate the presence of CH_2 and CH_3 groups while those at 1380 cm^{-1} are indicative of CH_3 [22]. A peak at 1379.01 cm^{-1} band may be attributed to aromatic CH and carboxyl-carbonate structures. The peaks in 1153.35-1300 cm^{-1} correspond to vibration of CO group in lactones. The peaks around 1238.21, 1080.06 and 862.12-476.38 cm^{-1} correspond to CHOH stretching, Si-O-Si stretching and Si-H groups, respectively. The presence of polar groups on the surface is likely to provide considerable

TABLE 3
PEAK IDENTIFICATION OF FTIR SPECTRA OF RAW AND MODIFIED RICE HUSK

Wavenumbers (cm^{-1})	Functional group
3404.31	-OH and Si-OH
2925.81	C-H stretching of alkanes
1641.31-1737.74	C=O stretching of aromatic groups
1546.8-1652.88	C=C stretching of alkenes and aromatic
1461.94	CH_2 and CH_3
1379.01	Aromatic CH and carboxyl-carbonate
1238.21	CHOH stretching of alcohol group
1153.35-1300	CO group in lactones
1080-1090	Si-O-Si
935.41	C-C
469-800	Si-H

cation exchange capacity to the adsorbent [22]. As can be inferred from the FTIR analysis (Figs. 2a-b), heating rice husk at 400°C and 600°C for 1h of burning time resulted in a loss of C-H stretching band (2925.81 cm^{-1}), C-C (935.41 cm^{-1}), C-O and C-O-C (1153.35 cm^{-1} , 1115 cm^{-1}) and C-O-H (899 cm^{-1}). These are replaced by the primary functional groups of C=O (1654.81 cm^{-1} , 1658.67 cm^{-1} , 1656.74 cm^{-1} , 1656.74 cm^{-1}) and silica functional groups namely Si-O-Si (1099.35 cm^{-1}), Si-H (800.4 cm^{-1}) and Si-OH (3200- 3700 cm^{-1}) [23].

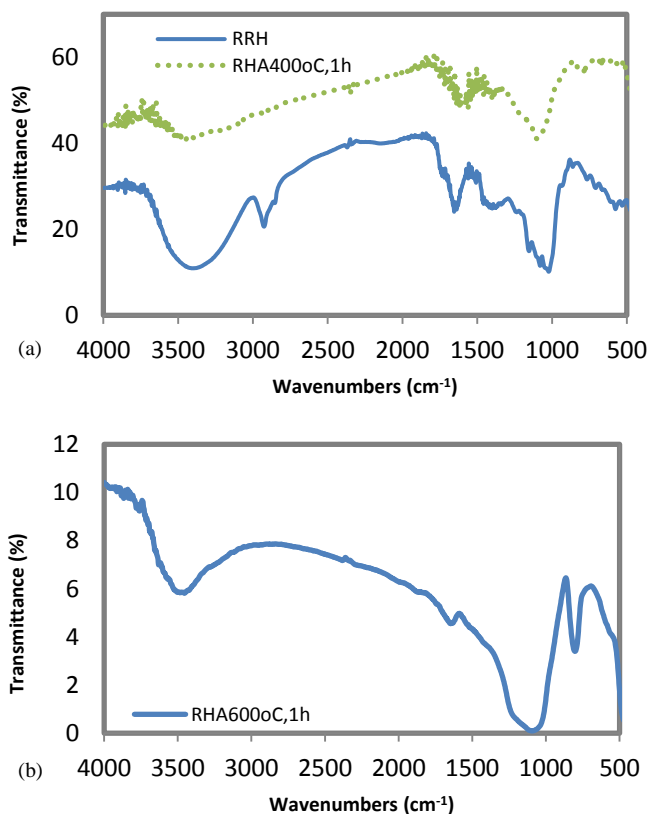


Figure 2. FTIR spectra of (a) RH and RHA400,1h and (b) RH A600, 1h.

C. Elemental analysis

Elemental analysis provided the complete elemental composition of both adsorbents as shown in Table 4. The presence of silica functional groups, as interpreted from the.

TABLE 4
ELEMENT ANALYSIS OF ADSORBENT

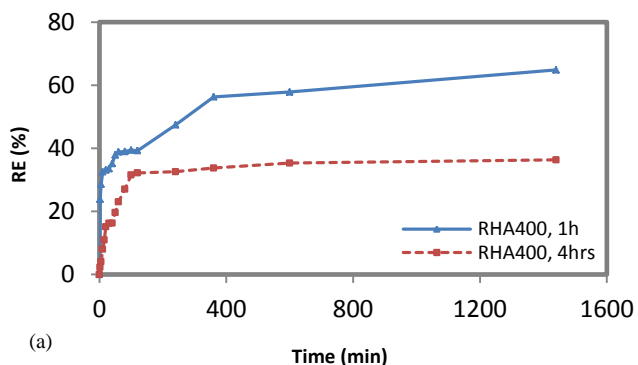
Adsorbent	Carbon %	Silica %	Oxygen %
Rice Husk (RH)	49.15	11.96	38.89
RHA 400 C, 1h	38.7	18.84	41.57
RHA 400 C, 2hrs	53.67	8.64	34.2
RHA 400 C, 3hrs	34.18	24.21	41.42
RHA400 C, 4hrs	25.14	27.89	43.92
RHA 600 C, 1h	n.d.	41.77	53.76
RHA 600 C, 2hrs	n.d.	15.1	54.21
RHA 600 C, 3hrs	n.d.	38.48	55.96
RHA 600 C, 4hrs	n.d.	48.15	47.87

n.d.: not detectable

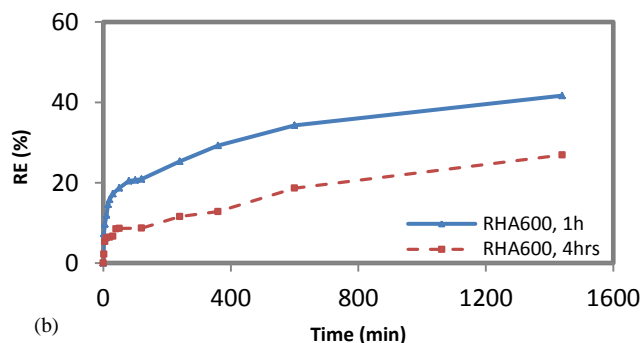
FTIR spectra of RHA400 and RHA600, is again confirmed by the increase in silica composition from 11.96% in RH to 18.84% in RHA400, 1h and 41.77% in RHA600, 1h

D. Effect of Burning Time

The elemental composition of RHA is given in Table 4. Heating of rice husk at different temperatures produced ash containing different content of carbon and silica. The rice husk heated at 400°C contains a higher amount of silica compared to carbon while the RH heated at 600°C contains high amorphous silica and no carbon was detectable. This observation is in accordance with [17]. Figs. 3(a-b) show that adsorption efficiency decreased by increasing the burning time of RHA400°C and RHA600°C from 1h to 4hrs. The preliminary test showed that RHA600 had less removal efficiency compared to RHA400 at different burning times. Regarding the relationship between phenol adsorption and carbon content shown in Table 4, the finding suggests that carbon has a significant effect in removal efficiency of phenol. Based on this finding, RHA400, 1h was selected for further investigations.



(a)



(b)

Figure 3. Effect of burning time (a) RHA400 and (b) RHA600 (adsorbent dose=10g^l⁻¹).

E. Effect of pH

pH of solution plays a vital role in phenol adsorption. The number of chemically active sites is changed by varying the pH. Fig. 4 depicts the effect of pH on removal efficiency of phenol by RHA400, 1h. The effect of pH was studied over the pH range of 2-10 with 10g l⁻¹ adsorbent dose 100mg l⁻¹ initial phenol concentration, 48hrs of agitation time and at ambient temperature. From Fig. 4, it is clear that a lower pH value is favorable for higher sorption of phenol and the maximum uptake of phenol takes place at pH 4. At pH 6 and above, the removal efficiency seems to remain constant. A decrease in pH probably results in a reduction of the negative charges at the surface of RHA400,1h. Because at lower pH the hydration shell exhibited by RHA400,1h can be positively charged by the hydronium ion in the solution. Also, in the higher pH range phenol forms salts, which readily ionize leaving negative charge on the phenolic group. At the same time the presence of OH⁻ ions on the adsorbent prevents the uptake of phenolate ions. Similar behavior has been reported by [15].

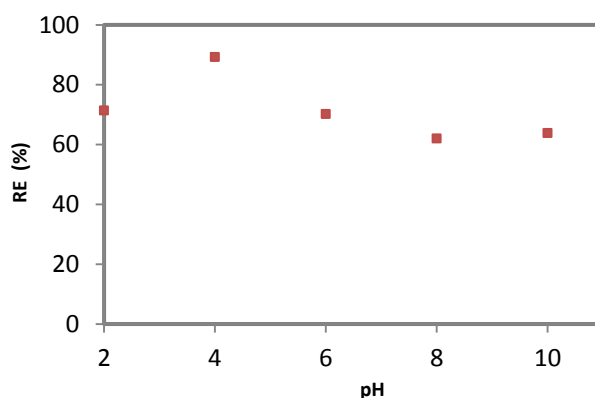


Figure 4. Effect of pH on phenol adsorption by RHA400, 1h (adsorbent dose=10g^l⁻¹).

F. Effect of initial concentration

The effect of concentration on phenol removal was investigated by varying the initial phenol concentration while other experimental parameters were kept the same; 10g l⁻¹ adsorbent at normal pH, 24hrs of agitation time and ambient temperature. Six phenol solutions with phenol concentrations of (10, 50, 100, 300, 500 and 1000 mg l⁻¹) were prepared. Fig. 5 shows that the removal efficiency (%) decreases as the initial concentration of phenol increases. At lower concentrations, all sorbate ions present in the adsorption medium could interact with the binding sites therefore higher adsorption yields were obtained. On the other hand, the sorption capacity increases with increasing phenol concentration. Using RHA400,1h, when the initial phenol concentration was increased from 10 mg l⁻¹ to 1000mg l⁻¹, the sorption capacity increased from 0.97 mg g⁻¹ to 46.19 mg g⁻¹, because the initial sorbate concentration provided an important driving force to overcome all mass transfer resistance.

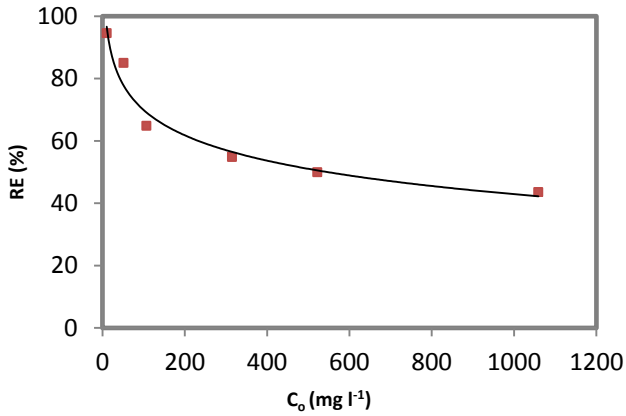


Figure 5. Effect of initial phenol concentration on the removal of phenol (adsorbent dose=10gl⁻¹) on RHA400, 1h.

G. Kinetic Study for the Removal of Phenol

Kinetic studies were conducted to determine the rate of phenol adsorption on the surface of adsorbent and to determine the equilibrium time for phenol uptake by RHA400, 1h. It was found that after 240 min the concentration reached the steady state (Fig.6).

In this study, the experimental equilibrium data of phenol adsorption onto RHA400,1h was first analyzed by the pseudo-first-order, pseudo-second-order and Elovich kinetic models (Table 1). The results of experimental data fitted with pseudo-first-order, pseudo-second-order and Elovich models are presented in Table 5. The accuracy of the models is given by the coefficient of determination (R^2) and normalized standard deviation, Δq_e (%). It can be seen in Table 5 that the coefficient of determination (R^2 values) for the pseudo-first order kinetic model lies in the range of 0.7130-0.87. Furthermore, the experimental values (q_e) do not agree with the calculated ones, which indicate that the pseudo-first-order equation cannot provide an accurate fit of the experimental data. On the other hand, a better fit of the experimental data is obtained with the pseudo-second-order kinetic model as can be observed in Figs. 7(a-c). The coefficient of determination (R^2 values) for the pseudo-second-order kinetic model is in the range of 0.9876-0.9986. However, a comparison between the pseudo-second-order kinetic and Elovich model shows that the Elovich model gives a better fit between experimental and calculated data (Figs. 7(a-c)), which indicates that the kinetics of phenol adsorption follows the Elovich model.

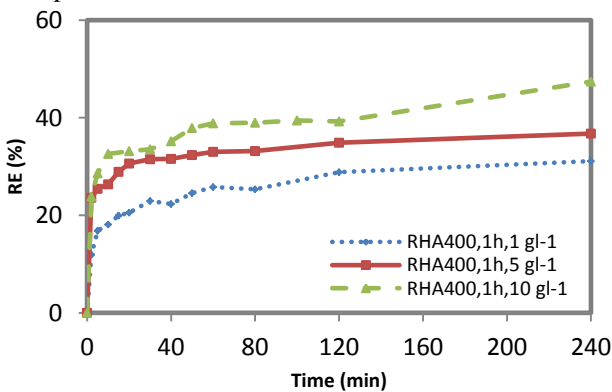
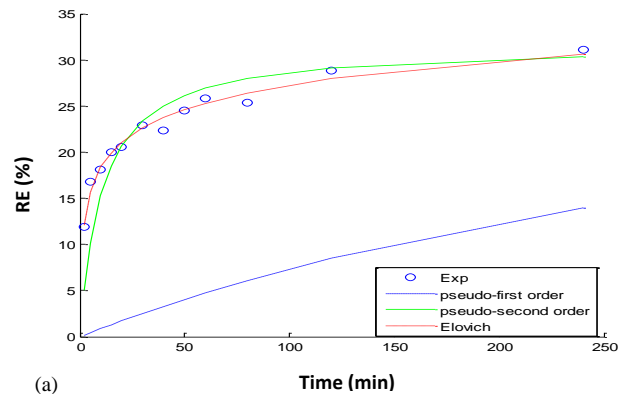


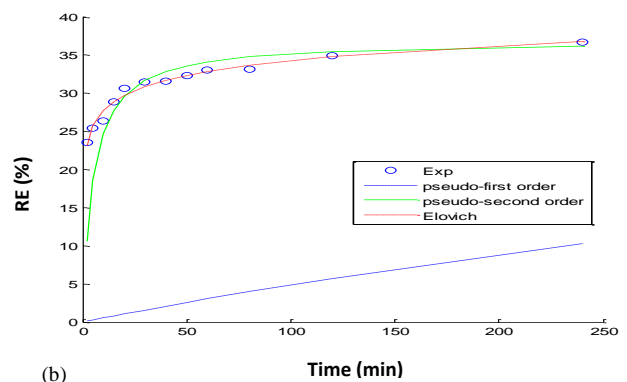
Figure 6. Effect of contact time(t) on percent removal (RE %) of phenol RHA400, 1h (adsorbent dose = 1 g l⁻¹, 5g l⁻¹ and 10 g l⁻¹)

TABLE 5
PSEUDO-FIRST-ORDER, PSEUDO-SECOND-ORDER AND ELOVICH MODEL, CONSTANT AND CORRELATION COEFFICIENT FOR ADSORPTION OF PHENOL BY RHA400,1H (ADSORBENT DOSE = 1 GL⁻¹, 5 GL⁻¹ AND 10 GL⁻¹)

Model	Parameters	RHA400,1h		
		1gl ⁻¹	5 gl ⁻¹	10 gl ⁻¹
Pseudo -first order	$q_{e,exp}(mg\ g^{-1})$	46.7648	12.1285	6.9201
	$q_{e,cal}(mg\ g^{-1})$	26.2562	5.9151	3.6789
	$K_1\ (min^{-1})$	0.0037	0.0020	0.003
	R^2	0.8204	0.7130	0.8700
	$\Delta q_e\ (%)$	13.2227	15.4464	14.1220
Pseudo -second order	$q_{e,cal}\ (mg\ g^{-1})$	35.0235	8.1678	4.9980
	$K_2(g\ mg^{-1}\ min^{-1})$	0.0027	0.0246	0.0196
	R^2	0.9942	0.9986	0.9876
	$\Delta q_e\ (%)$	7.5701	9.8463	8.3745
Elovich	$q_{e,cal}\ (mg\ g^{-1})$	37.7610	8.7099	5.1118
	$a(mg\ g^{-1}\ min^{-1})$	49.3279	1.04×10^3	66.9969
	$b\ (g\ mg^{-1})$	0.2342	1.5850	2.2309
	R^2	0.9805	0.9780	0.9352
	$\Delta q_e\ (%)$	5.8051	8.4986	7.8789



(a)



(b)

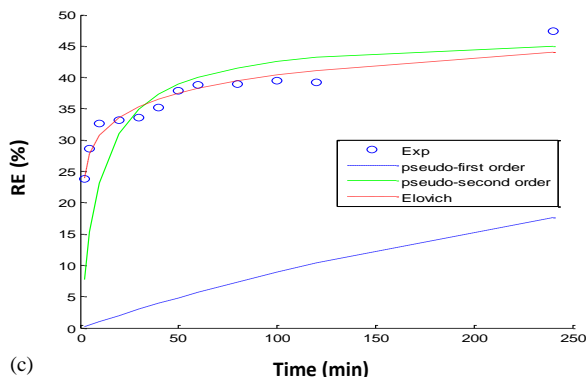


Figure 7. Comparison of experimental and calculated RE values (%) of RHA400, 1h at adsorbent dose (a) 1 g l^{-1} , (b) 5 g l^{-1} and (c) 10 g l^{-1}

H. Adsorption equilibrium study

The equilibrium isotherm is fundamental in describing the interaction behaviors between the solutes and adsorbents. To determine the equilibrium isotherms, the initial phenol concentration was varied from 10 to 1000 mg l^{-1} , while the adsorbent concentration was kept constant (10 g l^{-1}). The study was conducted at 23°C and 40°C . The isotherm constant and correlation coefficient are tabulated in Table 6. As shown in Figs. 8(a-b), it was found that a slight decrease could be observed on phenol adsorption capacity as temperature increased from 23°C to 40°C , which shows that the equilibrium amount of adsorbed phenol could be affected by temperature. Based on the R^2 values, the sorption data are best fitted with Freundlich model than the Langmuir model for both temperatures.

TABLE 6

ISOTHERMS CONSTANT FOR PHENOL ADSORBED ON RHA400, 1H						
T($^\circ\text{C}$)	Langmuir model			Freundlich model		
	q_{max}	K_L	R^2	K_F	n	R^2
23	12.6824	0.1462	0.9804	1.2761	1.8561	0.9885
40	11.2780	0.1491	0.9752	1.2622	2.0148	0.9934

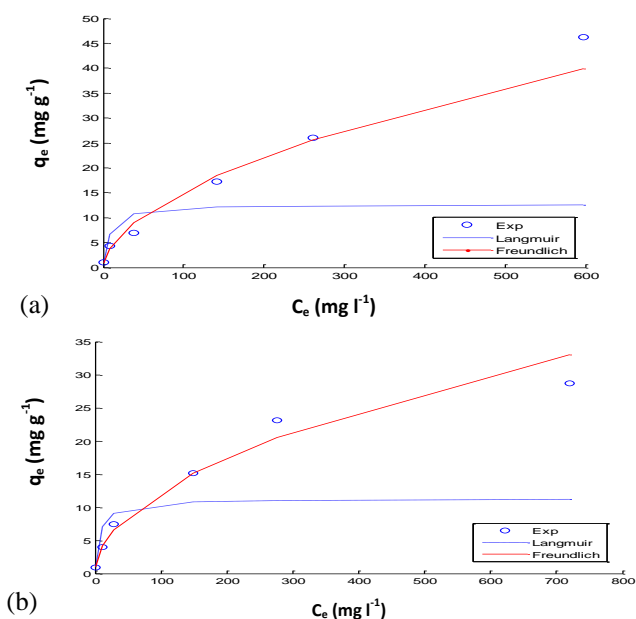


Figure 8. Comparison of experimental and predicted adsorption isotherms of phenol by RHA400, 1h according to Langmuir and Freundlich equations (a) 23°C and (b) 40°C .

I. Characterization of Adsorbent after Adsorption Experiments using FTIR

The effects of surface functional group on phenol adsorption were analyzed by observing the shifting of the FTIR peaks after the adsorption experiment. As shown in Fig. 9, it is observed that some of the FTIR peaks of RHA400, 1h after the adsorption experiment have shifted particularly at lower and higher wave numbers. The identified peaks for RHA400, 1h both before and after the sorption experiment are tabulated in Table 7.

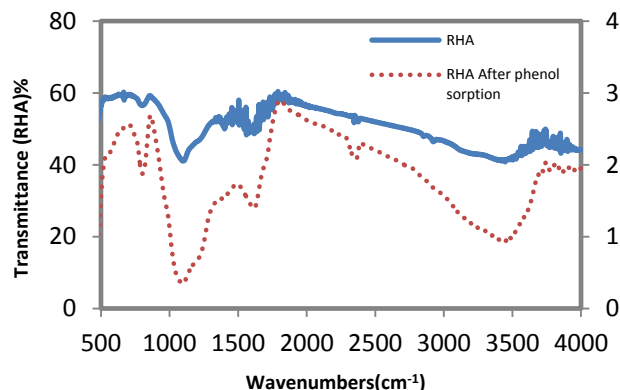


Figure 9. FTIR spectra of RHA400, 1h before and after phenol sorption

TABLE 7

FTIR SPECTRA OF RHA400, 1H BEFORE AND AFTER PHENOL SORPTION

Adsorbent	Si-OH,-OH	C=C	C=O	Si-O-Si	Si-H
RHA before phenol sorption	3425.34	1600.81	1659.3	1099.35	678.90
RHA after phenol sorption	3382.91	1612.38	1655.3	1093.56	677.81

IV. CONCLUSION

The present study has shown the feasibility of rice husk ash (RHA) as adsorbent for removal of phenol from aqueous solution. The experimental results show that adsorption capacity of the sorbent is affected by burning temperature and burning time of rice husk, pH, adsorbent dose and initial phenol concentration. The investigation shows that RHA400 is a promising adsorbent for removing phenol from aqueous solution compared to RHA600. This finding suggests that carbon has a significant role in the removal efficiency of phenol. Using RHA400, 1h as adsorbent, the maximum uptake of phenol took place at pH 4. It was found that, the sorption capacity of RHA400, 1h increased from 0.97 mg g^{-1} to 46.19 mg g^{-1} when the initial phenol concentration was increased from 10 mg l^{-1} to 1000 mg l^{-1} . On the other hand, a slight decrease on phenol adsorption capacity was observed as the temperature was increased from 23°C to 40°C . A comparison among two isotherm models (Langmuir and Freundlich models) shows that the sorption isotherms are better represented by Freundlich model. The kinetics of phenol adsorption is found to follow the Elovich model. FTIR analysis shows that the $-\text{OH}$, $\text{C}\equiv\text{C}$, $\text{C}=\text{O}$, Si-O-Si , Si-OH and $-\text{Si-H}$ groups contribute to the adsorption of phenol onto the surface of adsorbent.

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