



## **Isotherm, kinetics and thermodynamics of phenol adsorption onto rice husk activated carbon**

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### **Abstract**

This research was aimed at the study of isotherm, kinetics and thermodynamics of phenol adsorption onto rice husk activated carbon. The rice husk activated carbon was produced by carbonization followed by activation with phosphoric acid and batch experiment was conducted to obtain the relevant experimental data for analysis. Langmuir and Freundlich models were employed for the isotherm analysis while pseudo-second order rate equation was used for the kinetics study in addition to the thermodynamic model analysis. The adsorption process was found to be inconsistent with the assumption of Langmuir monolayer but conformed to Freundlich assumption of multilayer and physical adsorption. The experimental data followed pseudo-second order kinetic model and the adsorption process was found to be exothermic, feasible and better favoured at lower temperatures.

### **Keywords**

Agricultural wastes; Adsorption capacity; Model equations; Data analysis; Equilibrium; Isotherms; Kinetics; Thermodynamics; Wastewater

## **Introduction**

Rice husk, a potential source of activated carbon, is locally available in Nigeria but underutilized thereby constituting solid waste menace in the environment. Incidentally, large volume of wastewater containing high concentrations of toxic contaminants such as phenol and other contaminants are continuously being generated as a result of increased industrial activities. This has led to the continuous search for simple, cleaner and cost-effective treatment approach for wastewater before discharge into the environment [1]. Agricultural waste materials including rice husk, when suitably modified could serve as activated carbon for efficient adsorption of wastewater contaminants. For a successful adsorption design, information on the isotherm, kinetics and thermodynamics of the process is necessary.

Isotherm analysis provides knowledge of important information that can be deduced from the estimated parameters such as adsorption pattern and surface properties as well as identifying an equation that accurately represent the experimental data which could be used for design purpose [3, 4]. Application of isotherm models requires conducting batch adsorption experiments with agitation at constant temperature and volume of solution and either varying the mass of the adsorbent or the concentration of the solute for a predetermined time sufficient enough to attain equilibrium [4]. Adsorption kinetics gives information about the solute uptake rate that determines the residence time required for the completion of the adsorption process and the kinetic parameters are helpful for predicting rate of adsorption while also providing important information for design and modelling [5, 6]. Thermodynamic parameters provide important information such as feasibility and spontaneity of the adsorption process as well as nature (endothermic or exothermic) of the process [7].

According to Somasundaram *et al.*, [8], there is little study on the use of phosphoric acid for the production of activated carbon from rice husk.

Therefore the objective of this research was to study the isotherm, kinetics and thermodynamics of phenol adsorption onto rice husk activated carbon produced by carbonization followed by activation with phosphoric acid.

## **Material and method**

### ***Production of activated carbon***

Rice husk was the natural precursor used in the preparation of the adsorbent. It was collected from the rice mill of National Cereal Research Institute, Badeggi. It was washed with distilled water to remove dirt and surface impurity, then oven-dried at 100°C for 24h [9]. For the thermal pre-treatment, it was placed on a ceramic flat surface, charged into a furnace and heated to a temperature of 300°C at a heating rate of 20-25°C/min and residence time of 1h. The carbonized rice husk (the charred residue) was collected and cooled to room temperature. The procedure above was repeated for 400°C and 500°C. The thermal pre-treated rice husks samples were labelled RH300, RH400 and RH500. In the chemical pre-treatment process, each of the carbonized rice husk was activated with 1M H<sub>3</sub>PO<sub>4</sub> for 3h at impregnation ratio of 2:1 (volume *ml* of acid/mass *g* of rice husk) and later oven-dried overnight at 200°C to ensure proper drying [10, 11]. Each of the sample was then removed from the oven, cooled for 2h and then washed with distilled water to bring the pH to 7.0 and again oven-dried overnight at 100°C [10].

### ***Batch experiment***

In the Batch Adsorption, 2g of the pre-treated adsorbent was added to 100ml of different phenol initial concentration (10, 20, 30, 40 and 50mg/l) in 250ml conical flask. The mixture in the flask was placed on magnetic stirrer at 150 rpm [12, 13] for 90min at ambient temperature. Using a 5ml syringe, samples were withdrawn at different time intervals (10, 20, 30, 60 and 90min) and centrifuged at 3000 rpm for 20min. The supernatant solution was collected from the centrifuge by decantation and filtered using a micro filter attached to a 5ml syringe. The micro filter was backwashed after each use with de-ionized water using a 50ml syringe. The procedure above was repeated for 3 and 4g of pre-treated adsorbents. The procedure above was also carried out at temperatures of 30, 40 and 50°C, under the optimum operating condition developed from preliminary study that is pre-treatment temperature of 441.46°C, initial concentration of 40.61mg/l and adsorbent dosage of 4g [40]. The analysis of phenol in each sample filtrate was carried out using UV spectrophotometer set at wavelength of 270nm absorbance. Prior to analysis, a technical calibration curve was prepared and it was linear over the concentration range used. Therefore, un-adsorbed phenol concentration in the filtrate was determined by interpolation using the calibration curve.

### ***Determination of adsorption capacities***

Adsorption capacity  $q_t$  at any given time  $t$  for each sample withdrawn was determined using the equation [14, 15]:

$$q_t = (C_0 - C_t)V/m \quad (1)$$

Adsorption capacity  $q_e$  at equilibrium for each batch experiment was determined using the equation [16, 17]:

$$q_e = (C_0 - C_e)V/m \quad (2)$$

where:  $q_e$  = adsorption capacity at equilibrium (mg/g),  $q_t$  = adsorption capacity at time  $t$  (mg/g),  $C_0$  = initial concentration of solute (mg/L),  $C_e$  = equilibrium concentration of solute (mg/L),  $C_t$  = concentration of solute at time  $t$  (mg/L),  $V$  = volume of solution (L),  $m$  = mass of activated carbon used (g).

### ***Adsorption equilibrium isotherm study***

For the equilibrium isotherm assessment, the isotherm models below were used to analyze the experimental data.

#### *Langmuir isotherm*

The linearized form of Langmuir isotherm model given below was used by plotting  $C_e/q_e$  against  $C_e$ . Equilibrium isotherm parameters for the model were obtained from the slope and intercept of the plot [18, 19]:

$$C_e/q_e = 1/bQ_m + C_e/Q_m \quad (3)$$

#### *Freundlinch isotherm*

The linearized form of Freundlinch isotherm model given below was used by plotting  $\log q_e$  against  $\log C_e$ . Equilibrium isotherm parameters for the model were obtained from the slope and intercept of the plot [20, 21]:

$$\log q_e = \log K_f + 1/n \log C_e \quad (4)$$

where:  $q_e$  = adsorption capacity at equilibrium (mg/g),  $C_e$  = equilibrium concentration of solute (mg/L),  $Q_m$  = maximum monolayer adsorption capacity (mg/g),  $b$  = Langmuir isotherm constant ( $\text{dm}^3/\text{mg}$ ),  $K_f$  = Freundlich isotherm constant (mg/g),  $n$  = adsorption intensity.

The Langmuir isotherm parameter that is maximum monolayer adsorption capacity, is a measure of the maximum amount of solute a unit adsorbent coverage can accommodate, assuming only a monolayer is formed while the Freundlich isotherm parameter that is

adsorption intensity, is a measure of surface heterogeneity and adsorption pattern.

### ***Adsorption kinetics***

The linearized form of pseudo-second-order kinetic model given below was used by plotting  $t/q_t$  against  $t$ . Kinetic parameters for the model was obtained from the slope and intercept of the plot [22, 23]:

$$t/q_t = 1/k_2q_e^2 + (1/q_e)t \quad (5)$$

where:  $q_t$  = adsorption capacity at time  $t$  (mg/g),  $q_e$  = adsorption capacity at equilibrium (mg/g),  $t$  = time (min),  $k_2$  = kinetic rate constant ( $\text{g mg}^{-1} \text{min}^{-1}$ )

### ***Adsorption thermodynamics***

The thermodynamic model given below was used by plotting  $\ln k_c$  against  $1/T$ . Thermodynamic parameters for the model were obtained from the slope and intercept of the plot [24, 25]:

$$\ln k_c = \Delta S^0/R - \Delta H^0/RT \quad (6)$$

and the Gibbs free energy was determined from the equation [26, 27]:

$$\Delta G^0 = \Delta H^0 - T\Delta S^0 \quad (6)$$

where:  $k_c$  = thermodynamic equilibrium constant (but  $K_c = q_e/C_e$ ),  $q_e$  = adsorption capacity at equilibrium (mg/g),  $C_e$  = equilibrium concentration of solute (mg/L),  $T$  = absolute temperature (K),  $R$  = gas constant (8.314 J/mol K),  $\Delta G^0$  = change in Gibbs free energy (kJ/mol),  $\Delta H^0$  = change in enthalpy (kJ/mol),  $\Delta S^0$  = change in entropy (kJ/mol).

## **Results and discussion**

### ***Adsorption isotherm***

#### ***Langmuir isotherm***

It was observed in Table 1 that for RH300, maximum monolayer adsorption capacity of 0.112mg/g, 0.198mg/g and 0.398mg/g were obtained for 2g, 3g and 4g adsorbent dosage and this are less than the corresponding experimental adsorption capacity. For RH400, the table shows that maximum monolayer adsorption capacity of 0.042mg/g, 0.375mg/g and 0.03mg/g were obtained for 2g, 3g and 4g adsorbent dosage and this are also less than the

corresponding experimental adsorption capacity. It was also observed in the table that for RH500, maximum monolayer adsorption capacity of 0.177mg/g, 0.140mg/g and 0.197mg/g were obtained for 2g, 3g and 4g adsorbent dosage and this are also less than the corresponding experimental adsorption capacity. Generally, the maximum monolayer adsorption capacity of the experimental data deduced from the Langmuir isotherm model was less than the experimental adsorption capacity indicating that the maximum amount of phenol monolayer coverage on a unit of rice husk activated carbon was exceeded by the experimental value; hence the adsorption process is inconsistent with the Langmuir isotherm assumption.

**Table 1.** Langmuir adsorption isotherm

m (g)	C <sub>o</sub> (mg/l)	RH300			RH400				RH500				
		q <sub>e, exp</sub> (mg/g)	q <sub>m</sub> (mg/g)	R <sup>2</sup>	q <sub>e, cal</sub> (mg/g)	q <sub>e, exp</sub> (mg/g)	q <sub>m</sub> (mg/g)	R <sup>2</sup>	q <sub>e, cal</sub> (mg/g)	q <sub>e, exp</sub> (mg/g)	q <sub>m</sub> (mg/g)	R <sup>2</sup>	q <sub>e, cal</sub> (mg/g)
2	10	0.282	0.112	0.945	0.298	0.339	0.042	0.891	0.376	0.391	0.177	0.932	2.764
2	20	0.741	0.112		0.716	0.833	0.042		0.591	0.904	0.177		0.889
2	30	1.235	0.112		0.864	1.326	0.042		1.488	1.396	0.177		1.629
2	40	1.719	0.112		1.694	1.825	0.042		2.326	1.895	0.177		1.780
2	50	2.208	0.112		4.576	2.354	0.042		0.187	2.393	0.177		2.173
3	10	0.207	0.198	0.749	0.247	0.253	0.375	0.857	0.585	0.281	0.140	0.751	0.332
3	20	0.518	0.198		0.377	0.585	0.375		0.608	0.608	0.140		0.501
3	30	0.826	0.198		0.649	0.905	0.375		0.964	0.939	0.140		0.640
3	40	1.129	0.198		1.783	1.237	0.375		1.008	1.263	0.140		2.098
3	50	1.548	0.198		0.216	1.558	0.375		1.726	1.605	0.140		0.640
4	10	0.180	0.398	0.950	0.997	0.207	0.030	0.978	0.222	0.214	0.197	0.927	0.227
4	20	0.447	0.398		0.460	0.454	0.030		0.429	0.456	0.197		0.385
4	30	0.689	0.398		0.643	0.703	0.030		0.752	0.697	0.197		0.790
4	40	0.930	0.398		0.997	0.952	0.030		1.445	0.945	0.197		0.941
4	50	1.177	0.398		1.142	1.191	0.030		0.176	0.391	0.197		168.71

Therefore, this trend suggests the existence of multilayer adsorption phenomenon meaning that phenol molecules deposit on not only the free surface of the rice husk activated carbon but also on already adsorbed ones, that is sorption sites accommodate more than one phenol molecules. However, review of related literature revealed that this observation or inference has not been reported.

*Freundlich isotherm*

It was observed in Table 2 that for RH300, adsorption intensity of 7.09, 3.47 and 2.79 were obtained for 2g, 3g and 4g adsorbent dosage respectively. For RH400, the table shows that adsorption intensity of 17.7, 3.51 and 14.39 were obtained for 2g, 3g and 4g adsorbent

dosage respectively. It was also observed in the table that for RH500, adsorption intensity of 8.62, 5.23 and 3.18 were obtained for 2g, 3g and 4g adsorbent dosage respectively.

**Table 2.** Freundlich adsorption isotherm

m (g)	C <sub>0</sub> (mg/l)	RH300			RH400			RH500					
		q <sub>e,exp</sub> (mg/g)	1/n	R <sup>2</sup>	q <sub>e,cal</sub> (mg/g)	q <sub>e,exp</sub> (mg/g)	1/n	R <sup>2</sup>	q <sub>e,cal</sub> (mg/g)	q <sub>e,exp</sub> (mg/g)	1/n	R <sup>2</sup>	q <sub>e,cal</sub> (mg/g)
2	10	0.282	7.09	0.97	0.271	0.339	17.7	0.97	0.365	0.391	8.621	0.889	2.432
2	20	0.741	7.09		0.928	0.833	17.7		0.710	0.904	8.621		0.866
2	30	1.235	7.09		1.096	1.326	17.7		1.472	1.396	8.621		1.706
2	40	1.719	7.09		1.652	1.825	17.7		1.759	1.895	8.621		1.833
2	50	2.208	7.09		2.192	2.354	17.7		0.065	2.393	8.621		2.114
3	10	0.207	3.47	0.95	0.242	0.253	3.51	0.93	0.565	0.281	5.227	0.911	0.328
3	20	0.518	3.47		0.431	0.585	3.51		0.594	0.608	5.227		0.569
3	30	0.826	3.47		0.740	0.905	3.51		1.013	0.939	5.227		0.734
3	40	1.129	3.47		1.294	1.237	3.51		1.058	1.263	5.227		1.475
3	50	1.548	3.47		0.195	1.558	3.51		1.610	1.605	5.227		0.734
4	10	0.180	2.78	0.98	1.005	0.207	14.4	0.997	0.202	0.214	3.179	0.979	0.220
4	20	0.447	2.78		0.451	0.454	14.4		0.471	0.456	3.179		0.430
4	30	0.689	2.78		0.670	0.703	14.4		0.706	0.697	3.179		0.787
4	40	0.930	2.78		1.005	0.952	14.4		0.920	0.945	3.179		0.873
4	50	1.177	2.78		1.114	1.191	14.4		17.517	0.391	3.179		1.592

m = mass of activated carbon used (g), C<sub>0</sub> = initial concentration of solute (mg/L), q<sub>e,exp</sub> = experimental adsorption capacity at equilibrium (mg/g), q<sub>e,cal</sub> = theoretical adsorption capacity at equilibrium (mg/g), q<sub>m</sub> = maximum monolayer adsorption capacity (mg/g), n = adsorption intensity, R<sup>2</sup> = correlation co-efficient.

Generally the adsorption intensity was found to be greater than one, indicating co-operative [28] or physical [4] adsorption. Similar trends of 1/n value greater than one were also reported [29-34]. This is supportive of the earlier suggestion of a multilayer physical adsorption process. The greater the value of 1/n, the less heterogeneous is the surface of the activated carbon [35] but has high affinity between solute and adsorbent [36].

For the correlation co-efficient (R<sup>2</sup>), Table 1 shows that Langmuir isotherm has correlation co-efficient in the range of 0.749-0.95 while that of Freundlich isotherm as noted in Table 2 range between 0.889-0.997. In addition, a careful comparison of the correlation co-efficient of the isotherms in Table 1 and 2 at the same carbonization temperature and adsorbent dosage revealed that with the exception of RH500 carbonization temperature against 2g adsorbent dosage, Freundlich isotherm has higher correlation co-efficient than Langmuir isotherm in all the equilibrium isotherm analysis. This relative goodness of fit of the experimental data to Freundlich isotherm further supports the existence of a multilayer physical adsorption process.

**Adsorption kinetics**

Pseudo-second order kinetics parameters are presented in Table 3. It was observed in Table 3 that for RH300, theoretical equilibrium adsorption capacity of 2.263mg/g, 1.54mg/g and 1.18mg/g were obtained for 2g, 3g and 4g adsorbent dosage and this reasonably predicts the corresponding experimental equilibrium adsorption capacity. For RH400, the table shows that theoretical equilibrium adsorption capacity of 2.34mg/g, 1.57mg/g and 1.2mg/g were obtained for 2g, 3g and 4g adsorbent dosage and this also reasonably predicts the corresponding experimental equilibrium adsorption capacity. It was also observed in the table that for RH500, theoretical equilibrium adsorption capacity of 2.45mg/g, 1.59mg/g and 1.19mg/g were obtained for 2g, 3g and 4g adsorbent dosage and this reasonably predicts the corresponding experimental equilibrium adsorption capacity. Generally, it was observed that adsorption experimental data correlate very well with pseudo-second order rate model with correlation co-efficient of 0.9 to 1. Similar observation was also reported by El Shafey, [37].

**Table 3.** Pseudo-Second-Order Kinetics Data ( $C_o=50\text{mg/l}$ )

m (g)	t (min)	RH300			RH400				RH500				
		$q_e$ (mg/g)	k	$R^2$	$q_{t,cal}$ (mg/g)	$q_e$ (mg/g)	k	$R^2$	$q_{t,cal}$ (mg/g)	$q_e$ (mg/g)	k	$R^2$	$q_{t,cal}$ (mg/g)
2	10	2.262	8.278	0.999	2.235	2.342	28.216	0.999	2.334	2.451	9.249	0.999	2.425
2	20	2.262	8.278		2.249	2.342	28.216		2.338	2.451	9.249		2.438
2	30	2.262	8.278		2.253	2.342	28.216		2.339	2.451	9.249		2.442
2	60	2.262	8.278		2.258	2.342	28.216		2.341	2.451	9.249		2.447
2	90	2.262	8.278		2.259	2.342	28.216		2.341	2.451	9.249		2.448
3	10	1.541	15.87	1.000	1.531	1.567	16.327	1.000	1.558	1.590	7.395	0.999	1.569
3	20	1.541	15.87		1.536	1.567	16.327		1.563	1.590	7.395		1.579
3	30	1.541	15.87		1.538	1.567	16.327		1.564	1.590	7.395		1.583
3	60	1.541	15.87		1.539	1.567	16.327		1.566	1.590	7.395		1.586
3	90	1.541	15.87		1.540	1.567	16.327		1.566	1.590	7.395		1.587
4	10	1.176	59.00	1.000	1.174	1.199	8.047	0.999	1.184	1.186	24.713	0.999	1.181
4	20	1.176	59.00		1.175	1.199	8.047		1.192	1.186	24.713		1.184
4	30	1.176	59.00		1.176	1.199	8.047		1.194	1.186	24.713		1.185
4	60	1.176	59.00		1.176	1.199	8.047		1.197	1.186	24.713		1.185
4	90	1.176	59.00		1.176	1.199	8.047		1.197	1.186	24.713		1.186

$m$ = mass of activated carbon used (g),  $C_o$  = initial concentration of solute (mg/L),  $t$  = time (min),  $q_e$  = adsorption capacity at equilibrium (mg/g),  $q_{t,cal}$  = theoretical adsorption capacity at time  $t$  (mg/g),  $k$  = kinetic rate constant ( $\text{g mg}^{-1} \text{min}^{-1}$ ),  $R^2$  = correlation co-efficient.

**Adsorption thermodynamics**

From Table 4, it was observed that the change in enthalpy of the adsorption process has negative value of -6.229kJ/mol and this indicates that the adsorption process is exothermic. Khalir *et al.*, [38] reported that low negative values of change in enthalpy is an



indication that physical adsorption is among the mechanism of the adsorption process.

**Table 4.** Adsorption Thermodynamics Data

T (k)	Ink (g/l)	1/T (k <sup>-1</sup> )	$\Delta G^{\circ}$ (kJ/mol)	$\Delta S^{\circ}$ (kJ/mol)	$\Delta H^{\circ}$ (kJ/mol)
303	-0.867	0.00330	2.216	-0.028	-6.229
313	-0.983	0.00319	2.495	-0.028	-6.229
323	-1.019	0.00310	2.773	-0.028	-6.229

T = absolute temperature (K), k = thermodynamic equilibrium constant (g/L),  
 $\Delta G^{\circ}$  = change in Gibbs free energy (kJ/mol),  $\Delta H^{\circ}$  = change in enthalpy (kJ/mol),  
 $\Delta S^{\circ}$  = change in entropy (kJ/mol)

Therefore, the low negative values observed further show that the adsorption is a physical process. The change in entropy was also observed to have negative value of -0.028kJ/mol and this implies a decrease in disorder at the sorbent/solution interface [39]. The Gibbs free energy was observed to have positive value and this value increased from 2.216 to 2.773kJ/mol as the temperature was increased from 30 to 50°C. Though this indicates that the process is not spontaneous, the weak positive values indicate that the process is feasible and the weaker values at lower temperatures imply that adsorption is favored at lower temperatures.

### Conclusions

The adsorption of phenol onto rice husk activated carbon was found to be inconsistent with the assumption of Langmuir monolayer (maximum monolayer adsorption capacity obtained from Langmuir isotherm was exceeded by the experimental adsorption capacity) but conformed to Freudlinch assumption of multilayer and physical adsorption. Therefore, adsorption process is multilayer and physical. The experimental data followed pseudo-second order kinetic model and the adsorption process was found to be exothermic. Though the adsorption was found not to be spontaneous, it was noted to be feasible and better favoured at lower temperatures.

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