



Evaluation of Adsorption Isotherms and Industrial Wastewater Treatment for COD Reduction Using Acid Impregnated Peanut Shell Activated Carbon

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Author's contribution

The sole author designed, analyzed and interpreted and prepared the manuscript.

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ABSTRACT

Phosphoric acid impregnated activated carbon has been successfully prepared from readily available agro waste (peanut shell) and applied in the treatment of industrial wastewater for reduction of organic matter contributing to the chemical oxygen demand (COD). Functional groups identification and surface morphology characterization of the activated carbon have been performed by Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). Effects of pH (2.1-10.2), initial concentration (50-320) mgL⁻¹, contact time (10-70) min and adsorbent dose (0.1-0.7) g on the percentage reduction of the COD have been studied and evaluated. Four selected adsorption isotherm models have been tested and their linear regression correlation coefficient (R²) values were in the order of Langmuir>Freundlich>Temkin> Dubinin Radushkevich (0.9433>0.9383>0.8967>0.8415) respectively.

Keywords: Activated carbon; peanut shell; adsorption isotherm; COD; wastewater.

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1. INTRODUCTION

Contamination of surface waters (i.e. oceans, rivers and lakes) by industrial activities remains a major source of water pollution particularly in developing countries. This could be attributed to none compliance to the minimum permissible discharge limit (MPDL) of specific pollutants in industrial wastewaters or none existence of such regulations. In countries where those standards exist enforcement remains a major drawback. Industrial activities could pollute water in different ways depending on the industry and the type of pollutant it generates. Water pollutants fall into two major categories, they can be biodegradable or non-biodegradables [1-4]; The biodegradable ones could be discharged from the industries such as dyeing, pharmaceutical, pesticides, or insecticides manufacturing industries etc. whereas those from the latter category are generated from fertilizer manufacturing companies[5], electroplating activities [6,7] and tanneries [8]. There are several quality parameters and indicators employed for the assessment of water quality and its pollution level. These parameters include total suspended solids (TSS), biological oxygen demand (BOD), and chemical oxygen demand (COD).

COD is a measure of the amount of organic pollutants present in a given water or wastewater sample. It is usually measured in milligrams per liter of water. There are different COD contributing sources which include food and drink processing industries, antifreeze and from the manufacturing of emulsified oil [9]. Various countries set their MPDLs for COD in municipal and industrial wastewaters. For instance, the Swiss government sets its minimum permissible level of COD in wastewater as 200-1000 mg L⁻¹ [10], while the North Carolina's benchmark as reported by USEPA was pinned at 120 mg O₂ L⁻¹ [11]. There are various methods for the reduction of COD in water treatment plants, including reverse osmosis, Advanced Organic Destruction Cell (ODC), UV and ozone treatment technologies [12]; Most of these methods are expensive and require frequent maintenance. The inherent disadvantages associated with these methods for reduction of COD in water treatment plants created a vacuum that could be filled by continued searching through research of cheaper and efficient alternatives.

Activated carbons remain the most ubiquitous and highly effective adsorbents that are produced from various agro waste precursors

such as periwinkle [13], rice husk [14], water melon rinds [15] mango leaves [16], sawdust [17,18] doum seed [19] and coconut shell [20]. The non-selective property of the activated carbons enables it to combine the ability of removing biodegradable organics, toxic and harmful agents in addition to their potentials in simultaneous removal of colour and odour [21].

Application of activated carbons for adsorption studies and reduction of COD in wastewaters has been ongoing for decades. For instance, Sharma et al [22] performed characterization and adsorption investigation on the activated carbon prepared from *Cocos nucifera L.* and achieved almost a hundred percent removal of the targeted chemical species. In another study, granular activated carbons were produced from coal and wood and employed in the removal of COD from wastewater [23]. The wood activated carbon used in this study showed a COD removal capacity of 23.8 L kg⁻¹ after more than 60hr of treatment. Nekoo et al [24] conducted a study on the potential of activated carbons in wastewater treatment for the removal of COD in oil refinery effluent. In another report [25], activated carbon of periwinkle shell was tested for COD removal in industrial wastewater and its results were relatively low when compared to those of a commercially procured activated carbon. Bo et al [26] treated wastewater from resin manufacturing industry by biologically activated carbon achieving significant COD removal after several adsorption cycles. In a related study [27] effect of activated carbons on COD and BOD removal using dissolved air floatation unit was investigated and attained excellent COD removal with increase in the initial concentration of feedstock wastewater.

This research aimed at preparing activated carbon from readily available agro waste (peanut shell) for reduction of COD in industrial wastewater from pesticides and insecticides manufacturing industry. The research findings would significantly motivate renewed interest in monitoring the level of water pollution resulting from unmonitored activities of industries. It will as well provide a means for using locally generated waste in reclaiming polluted water from such industries.

2. EXPERIMENTAL

2.1 Preparation of Activated Carbon

Peanut shells were obtained from a shelling unit at Dawanau in Kano metropolis; Unwanted

materials were sorted out followed by washing with double distilled water and then dried at 105°C. The dried sample was impregnated in 250 mL of 5.0M H₃PO₄ for 24hr. After the impregnation, the sample was dried at 105°C and then placed in a reactor at 450°C under inert atmosphere of nitrogen (N₂) at a flow rate of 175 ml/min resulting in the formation of carbonized material. The activated carbon was washed several times with distilled water until the pH of the discarded water was 6.8. It was then dried in an oven 105°C for 8 hr and kept for the batch experiment test.

2.2 Batch Adsorption Experiment

The adsorption experiments were carried out using batch adsorption technique at 298 K, effect of initial concentration of the adsorbate (50, 100, 150, 200, 250, 300 and 320 mgL⁻¹); Contact time (10, 20, 30, 40, 50, 60 and 70 min), adsorbent dose (0.1, 0.2, 0.3, 0.4, 0.5 and 0.7 g) and pH (2.1-10.2) on the percentage reduction of COD in wastewater were studied. The mixtures were subjected to constant agitation at approximately 250 rpm. The data for the evaluation of the adsorption study were acquired by transferring appropriate amount of the adsorbent into a 250ml flask containing 100ml of the adsorbate with the characteristic values of COD, BOD, TSS and amount of dissolved oxygen as shown in Table 1; The pH was adjusted using either 0.1 M HNO₃ or 0.1 M NaOH. After attainment of equilibrium the agitated mixture was filtered using Whatman filter paper No. 44 and aliquot representative was taken and analyzed using DR 2800 spectrophotometer for COD. Percentage COD removal and equilibrium adsorption capacities, q_e (mg/g) were determined using equations (1) and (2) respectively.

$$\% \text{ COD Removal} = \frac{(C_o - C_e)}{C_o} \times 10^2 \quad (1)$$

$$q_e = (C_o - C_e) \frac{V}{M} \quad (2)$$

C_o, C_e, V and M are initial and equilibrium concentrations (mgL⁻¹) of the adsorbate, volume (L) and weight of the adsorbent (g) respectively.

2.3 Characterization of Activated Carbon

2.3.1 FTIR spectral analysis

The FTIR spectra of the peanut shell activated carbon and non-activated carbon were obtained

using FTIR spectrometer (Cary 630, Agilent). The analysis was conducted at the spectral range of 4000-650 cm⁻¹ and resolution of 8 cm⁻¹. The background and samples were scanned 32 times while the apodization was in Happ-Genzel mode.

Table 1. Characteristics of the raw wastewater before the adsorption study

Parameter	Level
^a COD	320 mgL ⁻¹
^b BOD	36 mgL ⁻¹
^c TSS	102 mgL ⁻¹
pH	5.6

^aChemical oxygen demand, ^bBiological oxygen demand, ^cTotal suspended solid

2.3.2 Scanning electron microscopic analysis

The surface morphology characterization was performed on the activated carbon before and after the adsorption study using Phenom ProX desktop scanning electron microscope at 10 kV and about 400 x magnifications.

3. RESULTS AND DISCUSSION

3.1 FTIR Analysis

The FTIR spectra of the activated and non-activated peanut shell are presented in Figure 1. The broad band observed in the spectrum of the raw peanut shell (green) at 3304 cm⁻¹ is characteristic of O-H stretching which could be attributed to the alcohols and phenolics present in the peanut shell [28]. The same band has been observed in the spectrum of the activated peanut shell (black) which is weak upon comparison with that of non-activated shell. A weak peak has been observed at 2883 cm⁻¹ characteristic of C-H attributed to asymmetric stretching peculiar to aliphatic and aromatic compounds. A relatively intense peak has been observed in both activated and non-activated peanut shell spectra at 1603 cm⁻¹, this is characteristic of C=O of ketones, aldehydes and esters. Aromatic C-C stretching has been observed at 1510 cm⁻¹ which disappeared in the activated shell spectrum. 1421cm⁻¹ represents asymmetric bending vibration of aromatic methyl group [29]. The peak at 1260 cm⁻¹ is characteristic of C-O stretching vibration [30]. A rocking vibration [31] characteristic of CH₃ has been observed at 1023 cm⁻¹.

3.2 SEM Analysis

The surface of the activated carbon appears rough and porous before the adsorption study as seen in the SEM micrograph in Fig. 2a; This

facilitated the high adsorption achieved. It became filled or smooth following the adsorption study as depicted in Fig. 2b. This could be attributed to the adsorption of organic molecules contributing to the COD in the wastewater.

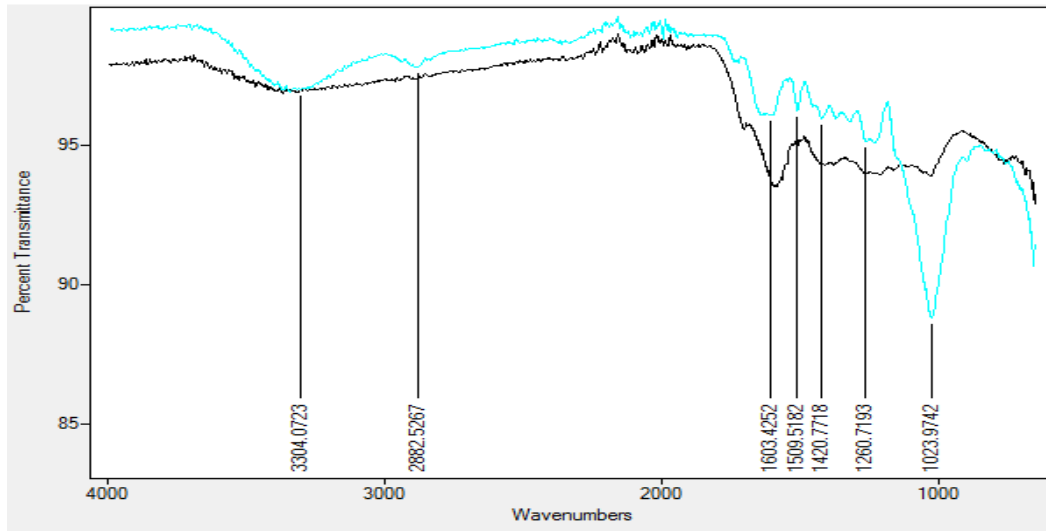
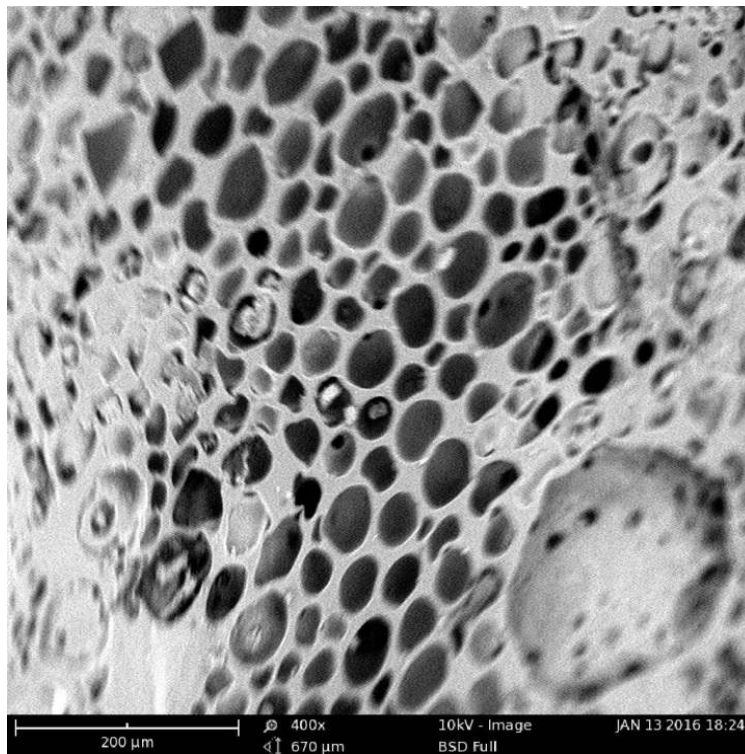
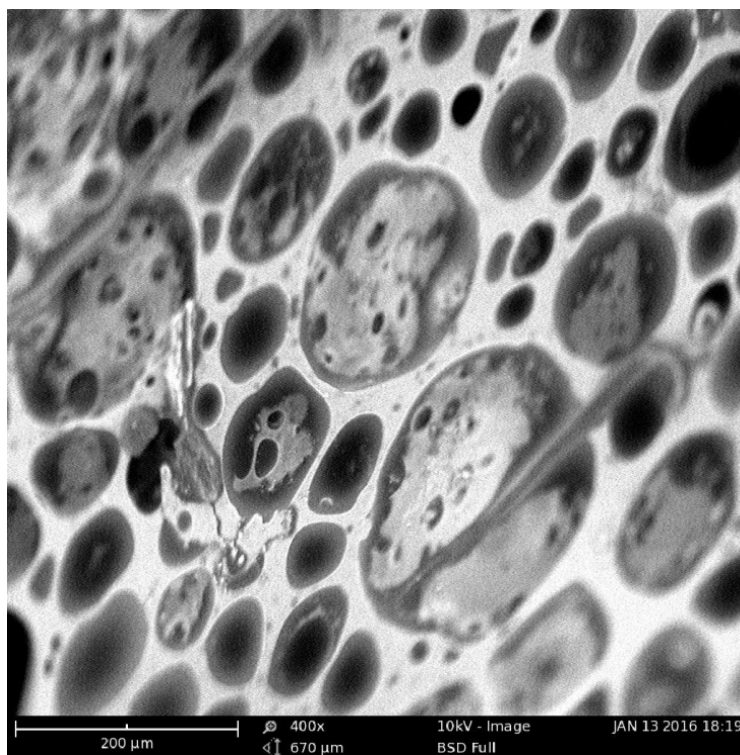


Fig. 1. Fourier transform infrared spectra of raw peanut shell (green) before activation and after activation (black)



A



B

Fig. 2. SEM micrographs of phosphoric acid impregnated Peanut shell activated carbon (a) before adsorption (b) after adsorption

3.3 Effect of Initial Concentration

Variation of percentage reduction of COD with adsorbate initial concentration was studied using the activated carbon. The raw wastewater was analyzed spectrophotometrically for COD level and then subjected to serial dilution to obtain appropriate concentrations for the study. It can be observed from Fig. 3(a) that the percentage reduction of the COD decreases with increase in concentration of the adsorbate. This could be due to the gradual occupation of the available sites which become filled as the concentration increases. Hence, the low percentage reduction observed at high concentration as shown in Fig. 3(a). The same observations were reported by Meena et al. and Mengistie et al. [32,33].

3.4 Effect of Contact Time

The result obtained for studying percentage removal with respect to contact time as seen in Fig. 3b clearly shows direct relation between the COD reduction and contact time. An optimum reduction of about 87.95% was achieved at 70 min.

3.5 Effect of Adsorbent Dose

The influence of the amounts adsorbent on the COD removal has been studied by varying its mass (0.1-0.7g), it could be seen from Fig. 4a that increase in the amount of the adsorbent facilitated a rapid increase in the percentage removal of the COD. This is due to the availability of more sites which aided the adsorption; hence the optimum removal was achieved at 0.7 g of the adsorbent.

3.6 Effect of pH

pH plays a significant role during adsorption processes, this also applies to the reduction of COD from wastewater as shown in Fig. 4b. The percentage removal increases gradually with increase in pH until it reaches optimum beyond which it decreases; it is concluded that the COD contributing molecules possess high affinity for the adsorbent at the optimum pH (6.6). Another possibility is that, the contributing molecules for the COD value remain in monomeric form at the optimum pH and tend to form dimers or

tetramers at high pH values. This makes it difficult for such high 'mer' molecules to occupy the available sites on the adsorbent.

with the data acquired as shown by the parameters in Table 2 for the models studied.

3.7 Adsorption Isotherms

The adsorption parameters were evaluated by studying the adsorption patterns of Langmuir, Freundlich, Dubinin- Radushkevich and Temkin isotherms. The Langmuir isotherm was best fitted

3.7.1 Langmuir isotherm

Equilibrium study for the removal of COD was performed on the peanut shell activated carbon by Langmuir adsorption model. According to this isotherm first introduced by Irving in 1916 [34], the adsorption sites are uniformly equal and no

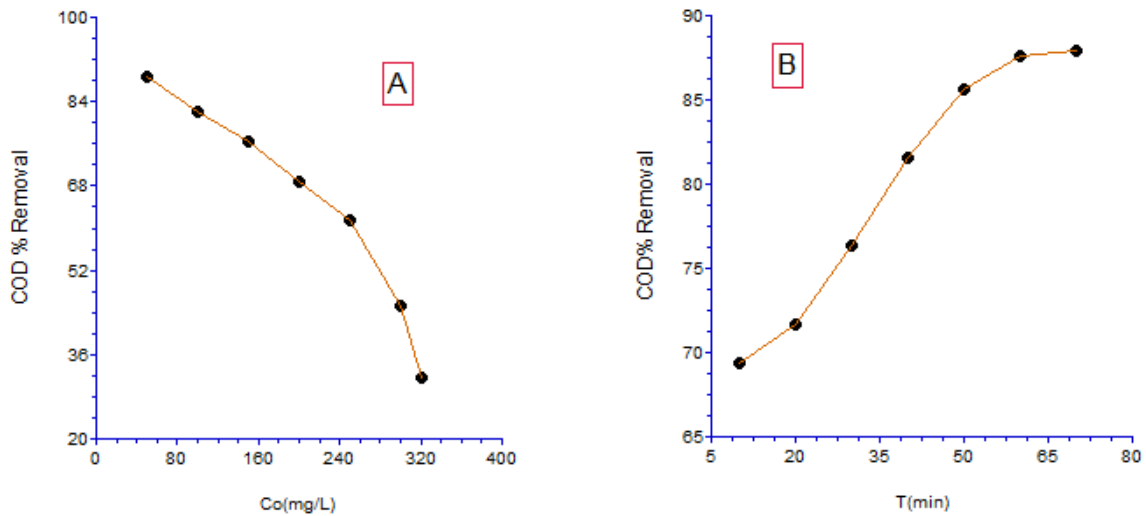


Fig. 3. Variation of percentage removal of COD with (a) adsorbate initial concentration (b) contact time

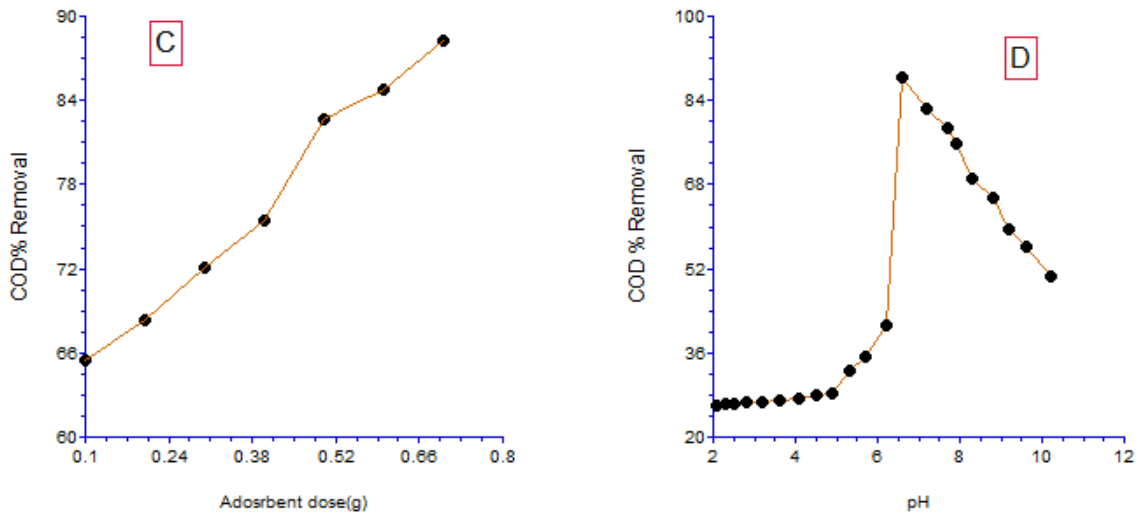


Fig. 4. Variation of percentage removal of COD with (a) adsorbate dose (b) pH

attraction occurs between the adsorbed molecules, hence monolayers of the molecules are deposited on the activated carbon. The linearized form of this model [35] is presented in equation (3) and was used in determination of its parameters in accordance with Fig. 5(a).

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{q_m K_L} \cdot \frac{1}{C_e} \quad (3)$$

Where q_e represents the amount adsorbed in milligram per gram of the adsorbent, and C_e (mg/L) is the concentration of the adsorbate at equilibrium. K_L and q_m represent Langmuir adsorption constant and the maximum amount of adsorbate that can be adsorbed on the activated carbon. The values of K_L and q_m for this study were determined from the slope and intercept as shown in Figure 5a. One of the essential features of this model is in its ability to determine R_L as shown in equation (4) which is a dimensionless parameter known as equilibrium parameter or separation factor [36].

$$R_L = \frac{1}{1 + (1 + K_L C_o)} \quad (4)$$

The adsorption process can be described as unfavourable if $R_L > 1$, linear if $R_L = 1$, favourable if $0 < R_L < 1$ and irreversible if $R_L = 0$. The value of R_L obtained in this study as presented in Table 2 is greater than 0 but less than 1 suggesting that the Langmuir model is favourable in this study. The maximum monolayer coverage capacity (q_m) obtained was 158.73 mg/l. This result is in agreement with the values obtained in relatively similar studies as reported by El-Dars et al. and Nekoo et al. [37,24].

3.7.2 Freundlich isotherm

This model based its argument on the fact that the adsorption sites are heterogeneous in nature; its linearized form is as shown in equation (5); where K_f and n represent adsorbent capacity and sorption intensity of the adsorbent [38], while q_e and C_e maintain their meaning as in Langmuir model. The parameters for the Freundlich model were determined from the slope and intercept values as shown in Fig. 5(b).

$$\ln q_e = \ln K_f + \frac{1}{n} \cdot \ln C_e \quad (5)$$

The partition between the heterogeneous phases is said to be independent of the concentration at $n = 1$. If n is between one and ten, it indicates a

favourable sorption [39]. However, if $1/n$ is below one then it signifies normal adsorption but if it is above one it indicates cooperative adsorption [40]. The values of n and $1/n$ obtained from this study as presented in Table 2 indicate that the adsorption of the molecules responsible for COD onto the peanut activated carbon is both favourable and normal. Similar results were obtained for COD removal using bio-based activated carbons by Syafalni, Emmanuel et al and Ghodale et al [41-43].

3.7.3 Dubinin radushkevich

The data acquired were fitted into the Dubinin Radushkevich (DR) equation as shown in equation (6), where β and ϵ represent the activity coefficient related to mean adsorption energy (mol^2/KJ^2) and Polanyi potential energy (mol/J^2) [44] respectively. The following parameters β and q_m were determined as $2 \times 10^{-4} \text{ mol}^2/\text{KJ}^2$ and 75.5 mg/g from the slope and the intercept of the graph shown in Fig. 5(c). The mean free energy (E) can be determined from equation (7) once the activity coefficient β is known. Its value distinguishes the nature of adsorption process involved. If E is between 8 and 16 KJ/mol the process is described as chemical adsorption or adsorption by ion-exchange, however, if it is lower than 8 KJ/mol it is described as physical adsorption [45]. The mean free energy obtained in this study was 0.05 KJ/mol signifying that physical adsorption was involved. Similar results were obtained by Itodo et al and Liu et al [46,47].

$$\ln q_e = \ln q_m - \beta \epsilon^2 \quad (6)$$

$$E = \left(\frac{1}{\sqrt{-2\beta}} \right) \quad (7)$$

3.7.4 Temkin isotherm

Temkin isotherm states that the heat of adsorption of the molecules involved in the adsorption process decreases linearly with coverage, this according to the model is attributed to the adsorbent-adsorbate interaction. Equation (8) describes one form of Temkin model, where A_T and B_T represents Temkin constant (Lmg^{-1}) and sorption heat in joule per mole respectively. The sorption heat obtained in this study was 38.124 Jmol^{-1} and the correlation coefficient (R^2) was 0.8967. It can be observed that the R^2 obtained from Fig. 5(d) signifies that this model does not fit well with the data as

compared with values obtained for the Langmuir and Freundlich models studied. Similar results were reported by Moradi et al and Kaman et al [48, 49] in their studies for COD removal using

carbon nanotubes and coconut shell activated carbon.

$$q_e = B_T \ln A_T + B_T \ln C_e \quad (8)$$

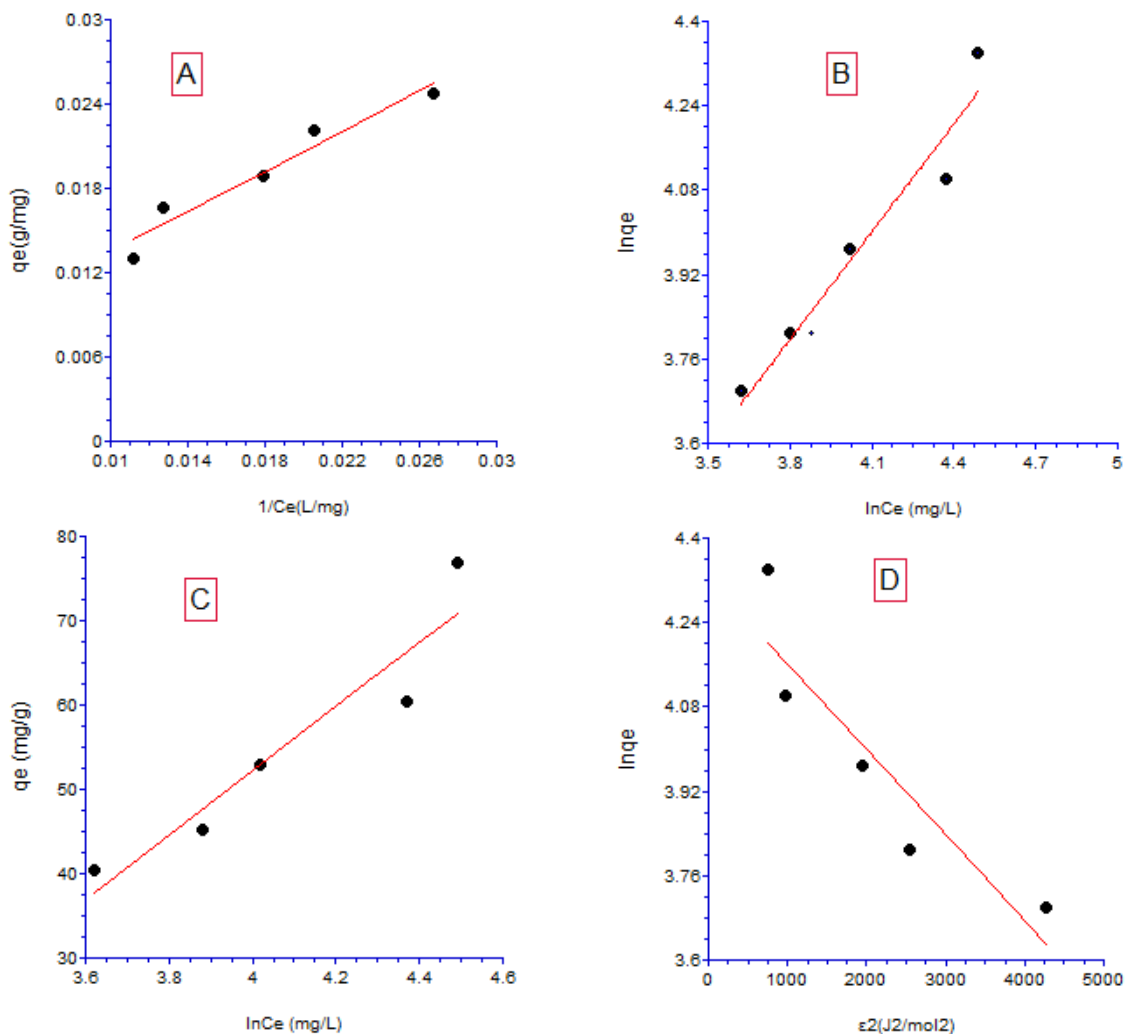


Fig. 5. Langmuir (a), Freundlich (b), Temkin (c) and Dubinin Radushkevich plots for COD reduction using phosphoric acid impregnated peanut shell activated carbon. Treatment time: 70 min, pH:6.6, temperature: 298K and agitation speed: 250rpm

Table 2. Adsorption isotherm parameters for the reduction of COD in wastewater using phosphoric acid impregnated peanut shell activated carbon as adsorbent

Model	q_m (mg/L)	K_L (Lmg ⁻¹)	R_L	R^2
Langmuir Isotherm	158.73	8.8×10^{-3}	2.62×10^{-1}	0.9433
Freundlich Isotherm	K_F (mg/g)	$1/n$	n	R^2
	3.34	0.68	1.47	0.9383
Temkin Isotherm	A_T (Lmg ⁻¹)	B_T (Jmol ⁻¹)	-	R^2
	0.072	38.124	-	0.8967
Dubinin Radushkevich	q_m (mg/g)	β (mol ² /KJ ²)	E (KJ/mol)	R^2
	75.5	2×10^{-4}	0.05	0.8415

4. CONCLUSION

In this study, acid impregnated peanut shell activated carbon was successfully prepared and applied in the treatment of industrial wastewater for reduction of chemical oxygen demand. Optimum adsorption was achieved at pH 6.6 beyond which the adsorption decreased sharply. Percentage reduction in COD value was found to be increasing with increase in contact time and adsorbent dose, and decreased with increase in initial concentration of the adsorbate. Among the adsorption models studied, Langmuir isotherm fitted well with the acquired data having the highest regression coefficient value (R^2) while Dubinin Radushkevich model was least fitted.

COMPETING INTERESTS

Author has declared that no competing interests exist.

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