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Email: tapas-info@tapasinstitute.org, tapasinstitute@yahoo.com

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TRANSPORT MODEL FOR THE ADSORPTION OF CHEMICAL OXYGEN DEMAND FROM INDUSTRIAL TEXTILE EFFLUENT: A KINETIC STUDY

F. W. Abdulrahman¹, L. G. Hassan¹, S. Idris², A. U. Itodo³ and M. J. Ladan¹

Department of Chemistry, Usmanu Danfodiyo University, Sokoto, Nigeria

Department of Chemistry, Federal University of Technology PMB 65 Minna, Nigeria

Department of Chemistry, Kebbi State University of Science and Technology, Aliero, Nigeria

Corresponding author: S. Idris; E-mail:suleman_drs@yahoo.co.uk

ABSTRACT: Shea nut shell, an abundant and low cost agricultural waste product was used to prepare activated charcoal by means of two step process using H₃PO₄ and ZnCl₂ as an activating agents. The percentage activation burn off increased with increasing activation dwell time, while the percentage yield decreased. The highest chemical oxygen demand (COD) removal from industrial effluent were reported to be 59.99, 56.64, 51.62, 59.97, 53.32 and 59.99% for the series of adsorbents indicated as SS/H₃PO₄/5, SS/H₃PO₄/10, SS/H₃PO₄/15, SS/ZnCl₂/5, SS/ZnCl₂/10 and SS/ZnCl₂/15 respectively and thus implies that the salt catalyzed shea nut shell adsorbent were more effective than the corresponding acid modified biosorbent. Pseudo-first-order and Pseudo-second-order model were selected to study the adsorption kinetic. The removal of COD could be best described by the Psoudo-second-order model and the result also revealed that the activated charcoal produced from shea nut shell was very effective in removing COD from industrial effluent.

Keywords: Activated carbon, chemical oxygen demand, kinetic model, dye, adsorbent

INTRODUCTION

One method of treating wastewater is through the use of activated carbon. Numerous adsorbents including agricultural by-products have been considered for the adsorption on dyes. The use of activated carbons however, has been widely favored because of their high adsorption of dyes. The textile dye colour effluent has mainly the complex nature and most of them are toxic to aquatic life, carcinogenic and can cause some health problems. The removal of color effluent from textile dyes is one of the main problems because of the difficulty in treating such wastewaters by using traditional treatment methods. Activated carbons are widely used as adsorbents in pharmaceutical and food industries due to their highly porous structure and large adsorption capacity. Evaluation of waste biomass is getting increased attention in all over the world as it is renewable, widely available, cheap, and environmental friendly. Pyrolysis (Carbonization) has been proposed as one of several optional technologies for disposing and recycling waste products. By-products of both plants and animal origin were therefore pyrolyzed at temperature between 250°C- 800°C in closed containers, usually a Muffle furnace to yield active carbon (Yoshiyuki and Yutaka, 2003). Chemical oxygen demand (COD) measurements are commonly made on samples of wastewaters or a natural waters contaminated by domestic or industrial wastes. COD is a vital test for assessing

requirement of the effluent and is used for monitoring and control of discharges and for assessing treatment plant performance.

The aims of the work was to prepared activated charcoal from shea nut shell and use it for chemical oxygen demand adsorption from industrial effluent.

the quality of effluents and wastewaters prior to discharge. The COD test predicts the oxygen

MATERIALS AND METHODS

Sample collection and sample treatment: The shea nut shell was obtained from Kataeregi, Katcha local government area of Niger State. The industrial dye effluent was procured from Chelko textile Limited, Kaduna. It was stored at room temperature without further purification. The chemicals used were manufactured by M & B, BDH Chemicals and Shermond Sussex of England. The shea nut shell was pretreated as described by Omonhenles *et al.*, (2006). The sample was grind and sievied with a 2 mm mesh size sieve. The less than 2 mm samples were stored in airtight container. Proximate analysis and physicochemical variables were estimated, according to the standard method by AOAC, (1990), to obtain the ash, moisture content and pH.

Activation (two step process): Activation, involving the two steps activation scheme was adopted. Firstly, 5g of blended raw sample was weighed into six different clean and pre-weighed

crucibles. They were introduced into a Muffle furnace at 600°C for 5 minutes after which they were poured from the crucible into a bath of ice block. The excess water was drained off. The carbonized sample was washed, using 0.1M HCl to remove surface ash, followed by hot water wash and further washing with distilled water to remove residual acid. The sample was then sun dried, and further dried in the oven at 100°C for one hour. This process was repeated until a substantial amount of carbonized sample was obtained.

Thereafter, 5g of already carbonized sample was mixed with $5 \, \mathrm{cm}^3$ of each activating agent (1M $\mathrm{H_3P0_4}$ and 1M $\mathrm{ZnCl_2}$). The sample was allowed to stand for 2 hours, after which it was introduced into a furnace and heated at $800^{\circ}\mathrm{C}$ for 5 minutes. The activated sample was cooled with ice – cold water, excess water was drained off and the sample dried at room temperature. The above procedure was repeated for different residual time (10 min and 15 min) until substantial amount of activated carbon was obtained. A modified version of washing method was adopted, until pH of sample solution fell within 6-7 (Rahman *et al.*, 2005; Fan *et al.*, 2005).

The bulk density, activation burn off and % yield were calculated as in equation 1, 2 and 3.

Bulk density $(gcm^{-3}) = weight of dry activated carbon <math>(g)$

Volume of packed dry material (cm³)- ----- -1 (Rao et al., 2003). % Burn off= X 100 Wo Wo Weight of char after pyrolysis, washing and drying W_1 Weight of carbon after activation, washing and drying. (Ioannidou and Zabaniotu, 2006). Yield 100 W_0 W_0 Original mass of precursor W_1 Weight of carbon after activation, washing and drying. (Yulu et al., 2001).

Batch equilibrium experiment: Two gram of activated carbon was interacted with 40cm³ of industrial dye wastewater in a 50 cm³ erlenmeyer flask and this was allowed to stand for 30 minutes. It was then filtered using Whatman Filter Paper (No.42). The filtrate was collected for further analysis. The process was repeated at pre-set time (60, 90, 120 and 150 minutes). This was continued until 50cm³ of each filtrate was obtained and was used for the determination of chemical oxygen demand (COD) (Hameed *et al.*, 2006).

Determination of Chemical Oxygen Demand (COD): COD was determined using method by Udo and Ogunwale, 1986. 50cm^3 of the filtrate was measured into 250cm^3 conical flask. 10cm^3 of 0.00833M potassium dichromate ($K_2\text{Cr}_2\text{O}_7$) solution was added. 1g of HgSO₄, 80cm^3 of mercury sulphate acid solution and a few beads was also added. The mixture was heat under reflux condenser for 10 minutes. It was allowed to cool, the condenser was rinsed with 50cm^3 distilled water and the flask cool under running tap. Two drops of Ferroin indicator solution was added and titrated with 0.025M ferrous ammonium sulphate solution till the color changes from blue green to red-brown. The titer value was recorded for the sample. The blank was determined by using distilled water with the above procedure.

COD= The difference between the sample and blank titre values.......4 (Udo and Ogunwale, 1986). NOTE: 1cm³ of titre value = 4.0mg/l of COD (Udo and Ogunwale, 1986).

Batch kinetic studies: The most common models used to fit the kinetic adsorption experiments are Lagergren's pseudo – first-order model and pseudo – second-order model (Hameed, 2008). The pseudo –first-order kinetic model is as simplified in equation 5.

Log
$$(q_e-q_t) = log \ q_e - \frac{K_1 t}{2.303}$$

Where:

 K_1 = Pseudo – first – order rate constant (Min⁻¹), $q_{e \text{ and }} q_t$ are the amount of COD adsorbed at equilibrium and at time, t respectively and are described as adsorption capacities of sorbent per until gram of sorbate (Hameed, 2008).

Data were also fitted into the pseudo-second -order kinetic model given as equation 6.

 K_2 = Pseudo – second – order rate Constant (g.mg⁻¹ min⁻¹). A fairly good value of correlation coefficient, R^2 was obtained for both models and validity test was conducted using equation 7.

SSE (%) =
$$\sqrt{\sum(q_{e \text{ (exp)}} - q_{e \text{ (cal)}})^2}$$
 -----7

Where N

Number of data point (Hameed, et al., 2006)

RESULTS AND DISCUSSION

The percentage ash and moisture content for the shea nut shell was 6.67 ± 2.89 and 9.67 ± 0.58 . The percentage ash content was low indicating of high carbon yield. The pH of the activated carbon produced using H₃PO₄ and ZnCl₂ as an activating agent fall within the range of 6.74 ± 0.01 to 6.82 ± 0.01 and 6.30 ± -0.02 to 6.47 ± 0.01 respectively. A pH range of 6.30-6.82 is an indication of removal of residual chemical through the series of washing, this rendering the carbon fit to contact consumables, including drinking water. This could also be linked to the low conductivity values (Table 1). This is in accordance with Ahmedna *et al.* (1997), that activated carbons produced from precursors with low ash content have been found to have low pH (less than 7.0).

Table 1: The result of pH and conductivity of activated carbon after washing and removal of chemicals

Chemicals		
Samples	pН	Conductivity (µs/cm)
SS/H ₃ P0 ₄ /5	6.74± 0.01	7.3 ± 1.53
SS/H ₃ PO ₄ /10	6.71± 0.01	11.33 ± 1.15
SS/H ₃ P0 ₄ /15	6.82± 0.01	14.33 ± 1.53
SS/ZnCl ₂ /5	6.30± 0.02	10.67 ± 1.53
SS/ZnCl ₂ /10	6.40± 0.01	5.67 ± 0.58
SS/Zncl ₂ /15	6.47± 0.01	7.33 ± 1.15

A/B/C: Sample (A) modified with chemical (B) at activation time (C)

The percentage activation burn off increase with increase activation contact time regardless of the nature of the activating agent while the percentage yield decrease with increasing activation burn off. The expected low yield for precursors with higher percentage burn off is similar to values presented by Martinez *et al.* (2006) and Itodo *et al.* (2008). It was evident that at longer dwell time, more volatiles are released from the char, thereby, resulting to a higher burn off and a corresponding lower yield (Martinez *et al.*, 2006). The bulk density (g/cm³) increase with decrease percentage yield of activated carbon which is an indication that at lower percentage yield the hardness (attrition) of activated carbon is high with a resultant high tendency for regeneration (Table 2).

Table 2: The result of activation burn off, % yield and bulk density of activated carbon

Samples	Activation burn off (%)	%Yield	Bulk Density (g/cm³)
SS/H ₃ P0 ₄ /5	32.67± 3.06	67.33± 3.06	0.54± 0.01
SS/H ₃ PO ₄ /10	35.33± 2.31	64.00± 2.00	0.56 ± 0.02
SS/H ₃ PO ₄ /15	37.33± 1.15	62.67± 1.15	0.57 ± 0.01
SS/ZnCl ₂ /5	22.87± 2.53	77.13± 2.53	0.49± 0.01
SS/ZnCl ₂ /10	31.93± 2.61	68.07± 2.61	0.51 ± 0.01
SS/ZnCl ₂ /15	35.33± 3.06	64.67± 3.06	0.54 ± 0.01

A/B/C: Sample (A) modified with chemical (B) at activation time (C)

The highest COD uptake by H_3PO_4 catalyzed activated carbon at 5 minutes dwell time (SS/ H_3PO_4 /5) was obtained at longer interaction time (150 minutes), that is, 59.99% removed (Table 3). Analysis also revealed that activated carbon produced by H_3PO_4 catalyzed at contact time of 10 and 15 minutes (SS/ H_3PO_4 /10) and (SS/ H_3PO_4 /15) show the highest percentage removal of COD at shorter interaction time (30 minutes), that is, 56.64% and 51.62% uptake (table 4 and 5). On the contrary, the highest percentage adsorbed of COD for ZnCl₂ catalyzed activated carbon at 10 and 15 minutes dwell time (SS/ZnCl₂/10) and (SS/ZnCl₂/15) was obtained at longer interaction time (120 minutes), that is, 53.32% and 59.99% removed (Table 7 and 8). Analysis also showed that the activated carbon produced, using ZnCl₂ as an activating against at 5 minutes dwell time (SS/ZnCl₂/5) have the highest percentage uptake of COD at short interaction time (30 minutes), that is 59.97% adsorbed (Table 6) and figure 1. From these analyses, it was observed that the nature of activating agent and activation interaction time affects the removal of COD from industrial wastewater. Namasivayam and Kadirvelu (1994) reported similar result for the removal of COD from dyeing wastewater using carbonized coirpith.

Table 3: The % COD removal by SS/H₃PO₄/5 adsorbent

Time (min)	CODi	COD _f	% Removal	OTHER DESIGNATION OF THE PERSON OF THE PERSO
(min)	(mg/l)	(mg/l)	= <u>COD_i-COD_f</u> x 100 COD _i	
30	60.01	28.01	53.32	
60	60.01	29.02	51.64	
90	60.01	26.02	56.64	
120	60.01	25.01	58.32	
150	60.01	24.01	59.99	

Table 4: The % COD removal by SS/ H₃PO₄/10 adsorbent

Time	CODi	COD _f	% Removal	
(min)	(mg/l)	(mg/l)	= COD _i -COD _f x 100	
	20 (102 (200)		CODi	
30	60.01	26.02	56.64	
60	60.01	28.02	53.31	
90	60.01	27.01	54.99	
120	60.01	30.01	49.99	
150	60.01	29.03	51.62	

Table 5: The % COD removal by SS/ H₃PO₄/15 adsorbent

	% Removal	COD _f	CODi	Time
		CODF	1.00.00	
	= COD _i -COD _f x 100	(mg/l)	(mg/l)	(min)
	CODi	v		
	51.62	29.03	60.01	30
	26.66	44.01	60.01	60
	46.64	32.02	60.01	90
	19.99	48.01	60.01	120
300	33.31	40.02	60.01	150

Table 6: The % COD removal by SS/ZnCl₂/5 adsorbent

Time	CODi	COD _f	% Removal		
(min)	(mg/l)	(mg/l)	$= COD_i - COD_f \times 100$		
			CODi	1	17
30	60.01	24.02	59.97		
60	60.01	28.03	53.29		
90	60.01	29.01	51.66		
120	60.01	31.02	48.31		
150	60.01	30.01	49.99		

Table 7: The % COD removal by SS/ ZnCl₂/10 adsorbent

Table /:	The % COD Tellion	al by 55/ 211012/1	U adsorbent
Time	CODi	COD _f	% Removal
(min)	(mg/l)	(mg/l)	= COD _i -COD _f x 100
			CODi
30	60.01	36.01	39.99
60	60.01	32.03	46.63
90	60.01	40.02	33.31
120	60.01	28.01	53.32
150	60.01	29.01	51.66

Typical Kinetic data were generated from experimental data. These data were separately treated as table 9 and 10. To evaluate the adsorption kinetics of industrial effluent, the Lagergren's pseudofirst-order and pseudo-second-order kinetics models were used to fit the experimental data. Sample with higher correlation coefficients (R^2) is accepted for a given model while sample with values for the calculated and experimental q_e ($q_{e(cal)}$ and $q_{e(cap)}$) nearness to each other is also accepted for a given model. The first-order rate constant (K_1) and $q_{e(cap)}$ determined from the model with the corresponding correlation coefficients (R^2) were presented in table 9. The values for the correlation coefficients (R^2) for the pseudo-First-order model were low and a wide range of

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variations were recorded for the calculated and experimental q_e data. This indicates that the adsorption of COD on the adsorbent does not follow Pseudo-First-order kinetics.

Table 8: The % COD removal by SS/ZnCl₂/15 adsorbent

Time	CODi	COD _f	% Removal
(min)	(mg/l)	(mg/l)	$= \frac{\text{COD}_{i} \cdot \text{COD}_{f}}{\text{COD}_{i}} \times 100$
30	60.01	40.03	33.29
60	60.01	36.01	39.99
90	60.01	24.02	59.97
120	60.01	24.01	59.99
150	60.01	30.02	49.98

Where: COD_f = Chemical Oxygen demand of industrial effluent after interaction.

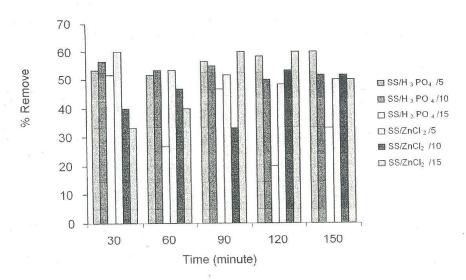


Fig. I: The percentage of chemical oxygen demand removed

Table 9: Pseudo first order parameter of Chemical Oxygen Demand estimation by H₃PO₄ and

7nCi2 modified SS-Carbon

SS-Carbon				
R ²	K ₁	q _e (cal)	$q_e(exp)$	%SSE
0.13	-0.01	0.03	0.72	0.3086
0.29	-0.02	0.01	0.62	0.2728
0.40	-0.01	0.07	0.39	0.1431
0.000	-0.01	0.02	0.6	0.2594
	-0.01	0.06	0.62	0.2504
Standill and		0.08	0.59	0.2281
	R ² 0.13	R ² K ₁ 0.13 -0.01 0.29 -0.02 0.40 -0.01 0.12 -0.01 0.09 -0.01	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	R² K₁ qe(cal) qe(exp) 0.13 -0.01 0.03 0.72 0.29 -0.02 0.01 0.62 0.40 -0.01 0.07 0.39 0.12 -0.01 0.02 0.6 0.09 -0.01 0.06 0.62

SS = Shea nut shell

The experimental kinetic data were further analyzed using the pseudo-second-order model. The $q_{e(cal)}$ and the $q_{e(exp)}$ values along with correlation coefficients (R^2) for the Pseudo-second-order model were presented in Table 10. The values of correlation coefficient (R^2) were high $(R^2>0.75)$ for data generating using second-order model, the best fit generally follows the trend: $SS/H_3PO_4/10$, $SS/ZnCl_2/5>SS/ZnCl_2/15>SS/ZnCl_2/10>SS/H_3PO_4/15$. Based on nearness calculated and experimental $(q_{e(cal)})$ and $q_{e(exp)}$, values for the $q_{e(cal)}$ were closer to the $q_{e(exp)}$, and

the best fit generally follows the trend; $SS/ZnCl_2/10$, $SS/H_3PO_4/5 > SS/H_3PO_4/10 > SS/ZnCl_2/5 > SS/ZnCl_2/15 > SS/H_3PO_4/15$. It was thus, concluded that the pseudo-second-order kinetic model provided a good correlation for the adsorption of COD on the adsorbent compared to the pseudo-first model. This is in agreement with the work by Hamced (2008), who reported pseudo-second-order kinetics for adsorption of methylene blue on papaya seed activated carbon.

Table 10: Pseudo second order parameter of Chemical Oxygen Demand estimation by H₃PO₄ and ZnCl₂ modified SS - Carbon

and Znciz	modified 35 - Cal	DOII			
Sample	R ²	K ₂	q _e (cal)	q _e (exp)	%SSE
SS/H ₃ PO ₄ /5	0.99	0.33	0.72	0.72	0.0000
SS/H ₃ PO ₄ /10	0.99	-0.77	0.61	0.62	0.0045
SS/H ₃ PO ₄ /15	0.79	-0.55	0.33	0.39	0.0268
SS/ZnCl ₂ /5	0.99	-0.54	0.58	0.6	0.0089
SS/ZnCl ₂ /10	0.89	0.17	0.62	0.62	0.0000
SS/ZnCl ₂ /15	0.94	0.14	0.69	0.59	0.0447

SS = Shea nut shell

Statistical sum of error (SSE, %) is another parameter used to test the applicability of both kinetic models. Sample with least percentage statistical sum of error (SSE, %) is accepted for a given kinetic model. Comparing table 9 and table 10, the percentage statistical sum of error on table 10 are low compared to that of Table 9. It can also be concluded that the COD adsorption by the activated carbon can best, be described by the second-order-kinetic model. The general trend for best fit in terms of % statistical sum of error (SSE, %) is as follows: $SS/H_3PO_4/5$, $SS/ZnCl_2/10 > SS/H_3PO_4/10 > SS/ZnCl_2/5 > SS/H_3PO_4/15 > SS/ZnCl_2/15$ in Table 10 respectively.

CONCLUSION

The adsorption of COD from industrial dye wastewater using shea nut shell as low-cost adsorbent was investigated using batch process. The adsorption kinetics can best, be described by pseudo-second-order kinetic. Considering the R^2 , this parameter fits well for $SS/H_3PO_4/5$, $SS/H_3PO_4/5$, $SS/H_3PO_4/5$, $SS/H_3PO_4/5$, $SS/H_3PO_4/5$, based on $Q_{e(exp)}$ nearness, the parameter $SS/ZnCl_2$ /10, $SS/H_3PO_4/5$, than $SS/H_3PO_4/5$. For percentage statistical sum of error (% SSE), $SS/H_3PO_4/5$, $SS/J_3PO_4/5$

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