

1 ADSORPTION KINETICS FOR THE REMOVAL OF 2 BIOCHEMICAL OXYGEN DEMAND (BOD) FROM DYE 3 EFFLUENT ONTO POULTRY DROPPINGS 4 ACTIVATED CARBON

7 ABSTRACT

8 **Aims/ objectives:** To prepared activated carbon from poultry droppings using H_3PO_4 as an activating
9 agent and to determine the uptake of BOD from dye effluents at different contact time.

10 **Study design:** Effectiveness of the activated carbon at various contact time.

11 **Place and Duration of Study:** Department of Chemistry Federal University of Technology Minna
12 Niger State Nigeria, between April and November 2010.

13 **Methodology:** Batch adsorption experiments were carried out for the removal of BOD from dye
14 effluent using poultry droppings as an adsorbent. Two step process was adopted with H_3PO_4 as an
15 activating agent and the adsorption was carried out at various contact time. The adsorption kinetic
16 were analysed using pseudo first-order, pseudo second-order, Natarajan and Khalaf first order and
17 Elovich model.

18 **Results:** Poultry droppings ash content was low which is an indication of high carbon yield. As the
19 activation burn off increases the percentage carbon yield decreases because more volatiles are
20 released from the char at higher burn off. The lower conductivity data revealed that the activated
21 carbon contain less leachable waste which is consider impurity and undesirable. Equilibrium contact
22 time was indicated at short contact time, that is 30 minutes for PD/ H_3PO_4 /10 and at long contact time,
23 that is 120 minutes for PD/ H_3PO_4 /5 adsorbent. It was also found that the pseudo second-order kinetic
24 model was the most appropriate model, describing the adsorption kinetics.

25 **Conclusion:** The findings indicated that the poultry droppings could be effectively used as a raw
26 material for the preparation of activated carbon for the removal of BOD from dye effluent and the
27 mechanism of adsorption is intraparticle diffusion control.

28 *Keywords: Adsorption, Contact time, Elovich model, Equilibrium, Kinetics.*

29 **1. INTRODUCTION**

30 Man made activities on water by domestic, industrial, agriculture, shipping, burning of fuels,
31 suspended particulate matter on land by domestic wastes, industrial waste, agricultural chemicals and
32 fertilizers, animal waste have negative influence on natural ecosystems. Activated carbon are widely
33 used in wastewater and drinking water processes. Activated carbon is a form of carbon that has been
34 processed to make it extremely porous and thus to have a very large surface area available for
35 adsorption or chemical reactions. Due to its high degree of porous, just one gram of activated carbon
36 has a surface area in excess of 500 m² as determined typically by nitrogen gas adsorption. Sufficient
37 activation for useful applications may come solely from the high surface area, though further chemical
38 treatment often enhances the adsorbing properties of the material. Activated carbon is manufactured
39 from a wide variety of materials. Poultry droppings, coal, peat, wood, coconut shell, grape seeds, shell
40 of nuts, extracted oil palm fibres have been found to be suitable precursors for producing activated
41 carbon owing to their high carbon and low ash contents (Activated carbon, 2010).

42 Activated carbon is valuable for a variety of applications. Examples include gas purification, water
43 purification, metal extraction, gold recovery, sewage treatment. In addition, activated carbon is useful
44 for the deodorization of closed spaces such as refrigerators, adsorption of ethylene to prevent
45 premature ripening of fruits and vegetables, and decolouration of sugar, juices and liquor etc.
46 Sufficient activation for useful applications may come solely from the high surface area, though further
47 chemical treatment often enhances the adsorption ability of the material (Emmanuel and Badmus,
48 2008).

49 The removal of pollutant using adsorption process provides an alternative means since the raw
50 materials are inexpensive and readily available. The study would provide a cheaper method for
51 control of pollution resulting from the discharge of industrial effluents and could also motivate local
52 industries in sourcing for activated carbon locally which will help to conserve foreign exchange.

53 Therefore the study is aiming at investigating the adsorption capacity of poultry dropping as an
54 alternative means of controlling water pollution and the objective is to consider the contact time

55 between the dye effluent and the adsorbent, adsorption kinetics and adsorption mechanism using
56 intraparticle diffusion model.

57 **2. MATERIALS AND METHODS**

58 **2.1 Sample Collection and Preparation.**

59 The poultry droppings used as precursor was collected from Kure farms Chanchaga, Minna and the
60 dye effluent was obtained from local dye industry, Ja'faru Mairiga Street, F-Layout Minna, Niger State,
61 Nigeria. It was stored at room temperature without further purification. The poultry droppings was
62 dried then ground and sieved with a 2 mm mesh size sieve. The less than 2 mm sample were stored
63 in airtight container. Ash content was determined using the method described by AOAC (1990).

64 **2.1.1 Preparation of the adsorbent**

65 Two step activation process was adopted. 5 g of grounded raw sample was weighed into clean and
66 weighed crucibles. The crucibles were introduced into a muffle furnace at 600°C for 5 minutes after
67 which they were poured from the crucible into a bath of ice water. Excess water was drained off. The
68 carbonized sample was washed, using 0.1 M HCl to remove surface ash, followed by hot water wash
69 and further washing with distilled water to remove residual acid. The sample was then sun dried, and
70 further dried in the oven at 100°C for one hour. This process was repeated until a substantial amount
71 of carbonized sample was obtained.

72 Thereafter, 5g of already carbonized sample was mixed with 5cm³ of activating agent (1M H₃PO₄).
73 The sample was allowed to stand for 2 hours, after which it was introduced into a furnace and heated
74 at 800°C for 5 minutes. The activated sample was cooled with ice water, excess water was drained off
75 and the sample dried at room temperature. The above procedure was repeated for different residual
76 time (10 min and 15 min) until substantial amount of activated carbon was obtained. Washing of the
77 activated sample was done with 0.1M HCl to remove surface ash, followed by hot water wash and
78 rinsing with distilled water to remove residual acid. Washing was completed until the pH of 6-7 was
79 obtained, then the sample was dried in oven at 110°C overnight and stored in air tight container
80 (Rahman *et al.*, 2005; Fan *et al.*, 2005; Ahmedna *et al.*, 2000).

81 **2.2 Characteristics of Activated Carbon**

$$\text{Bulk density (g/cm}^3\text{)} = \frac{\text{Weight of dry activated carbon(g)}}{\text{Volume of packed dry material(cm}^3\text{)}} \dots\dots\dots(1)$$

(Apipeeya *et al.*, 2006)

$$\% \text{ Burn off} = \frac{W_0 - W_1}{W_0} \times 100 \dots\dots\dots(2)$$

W_0 = weight of char after pyrolysis, washing and drying.

W_1 = weight of carbon after activation, washing and drying

(Ioannidou and Zabaniotou, 2006).

$$\% \text{ Yield} = \frac{W_1}{W_0} \times 100 \dots\dots\dots(3)$$

W_0 = Original mass of precursor

W_1 = weight of carbon after activation, washing and drying (Apipeeya *et al.*, 2006)

pH was determined using a pH meter and the conductivity was taken using a conductivity meter at room temperature (Okiemen *et al.*, 2004).

2.3 Adsorption Process Using Batch Method

2g of activated carbon was interacted with 40cm³ of industrial dye effluent in a beaker and covered. This was allowed to stand for 30 minutes. It was then filtered using whatman filter paper (No. 42) and the filtrate collected. The process was repeated at pre-set time (60,90,120 and 150 minutes). Each mixture was separately filtered and the filtrate collected. This was continued until 500 cm³ of each filtrate was obtained. This was used for the determination of biochemical oxygen demand (BOD). (Hameed *et al.*, 2007).

2.4 Determination of BOD.

200 cm³ of the filtrate above was measured into a conical flask. 1cm³ each of 2.24M manganese (II) sulphate solution and alkali-iodide-azide solution was added, well shaken and allowed to settle. The clear liquid above the precipitate was decanted. 2 cm³ of conc. H₃PO₄ and some water was added to dissolve the precipitate. The sample was titrated against 0.025M sodium thiosulphate. The titre value was recorded as the initial dissolved oxygen (Initial DO).

200 cm³ of the filtrate above was measured into an incubation bottle. 1cm³ each of 2.24M manganese (II) sulphate solution and alkali-iodide-azide solution was added, well shaken and incubated for 5 days at 20°C. The clear liquid above the precipitate was decanted. 2 cm³ of conc. H₃PO₄ and some water was added to dissolve the precipitate. The sample was titrated against 0.025M sodium thiosulphate. The titre value was recorded as the final dissolved oxygen (Final DO) (Udo and Ogunwale,1986).

$$\text{BOD (mg/L)} = \text{Final DO} - \text{Initial DO}$$

The BOD was determined for the dye effluent before interaction with the adsorbent.

$$\% \text{ BOD removed} = \frac{\text{BOD}_i - \text{BOD}_f}{\text{BOD}_i} \dots\dots\dots(4)$$

where

BOD_i(mg/L)=BOD concentration of dye effluent before interaction with the adsorbent.

BOD_f(mg/L) = BOD concentration of dye effluent after interaction with the adsorbent.

2.5 Determination of the Adsorption Capacity.

$$q_t = \frac{(C_i - C_t)V}{W} \dots\dots\dots(5)$$

Where

q_t (mg/g) = adsorption capacity at time t.

C_i (mg/L)= BOD_i.

C_t (mg/L) = BOD_f .

V (L) = volume of the dye waste water .

W (g) = weight of the activated carbon . (Hameead, 2009).

2.6 Adsorption Kinetics

kinetics of adsorption was investigated using the following models.

Pseudo first-order (Lagergren, 1898).

$$\text{Log } (q_e - q_t) = \text{Log } q_e - \frac{K_1}{2.303} t \quad \text{.....(6)}$$

Pseudo second-order (Ho *et al.*, 2000).

$$t/q_t = 1/k_2 q_e^2 + t/q_e \quad \text{----- (7)}$$

Natarajan and Khalaf first order (Raji *et al.*, 1986).

$$\text{Log } (C_i/C_t) = K_1/2.303 t \quad \text{----- (8)}$$

Elovich model equation: (Chien and Clayton, 1980; Sparks 1986)

$$q_t = 1/\beta \ln(\alpha \beta) + 1/\beta \ln(t) \quad \text{----- (9)}$$

Where

q_e (mg/g) = amount of BOD adsorbed at equilibrium.

K_1 (min^{-1}) = Adsorption rate constant for first-order.

K_2 ($\text{g.mg}^{-1}\text{min}^{-1}$) = Adsorption rate constant for second-order.

α ($\text{mg.g}^{-1}\text{min}^{-1}$) = Initial adsorption rate.

β (g.mg^{-1}) = desorption constant during any experiment.

2.7 Test of Kinetics Model

The applicability of pseudo first- order and pseudo second- order kinetic models are verified through the sum of error squares (SSE, %).

$$\% \text{ SSE} = \frac{\sqrt{\sum (q_{e(\text{exp})} - q_{e(\text{cal})})^2}}{N} \quad \text{.....(10)}$$

$q_{e(\text{exp})}$ (mg/g) = adsorption capacity at equilibrium experimental.

$q_{e(\text{cal})}$ (mg/g) = adsorption capacity at equilibrium calculated.

N= number of data point (Hameed *et al.*, 2007)

Intraparticle diffusion was used to described the mechanism of the adsorption process using the following equation.

$$q_t = K_p t^{1/2} + C \text{-----(11)}$$

where C= Intercept.

K_p = Intraparticle diffusion rate constant ($\text{mg.g}^{-1}\text{min}^{-1/2}$) (Crank, 1933)

3.RESULTS AND DISCUSSION

3.1 Characteristic of the Activated Carbon

The percentage ash content (3.65 ± 0.01) of the precursor is low which is an indication of high carbon yield. The pH of the adsorbent is within the range of $6.3 \pm 0.03 - 6.7 \pm 0.11$ which 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). Percentage activation burn off increase with increase activation dwell time as the percentage yield decreases. It was evident that at longer dwell time, more volatiles are released from the char, thereby, resulting into higher burn off and corresponding lower yield. Carbons with an adequate bulk density also help to improve the filtration rate by forming an even cake on the filter surface. Bulk density of the sample is within the range of $0.51 \pm 0.11 - 0.52 \pm 0.02 \text{ g/cm}^3$ which revealed better adsorption for the adsorbent. The sample conductivity values shows that the adsorbents has low leachable waste which is consider impurity and undesirable (Table 1).

Table 1: Characteristics of the activated carbon

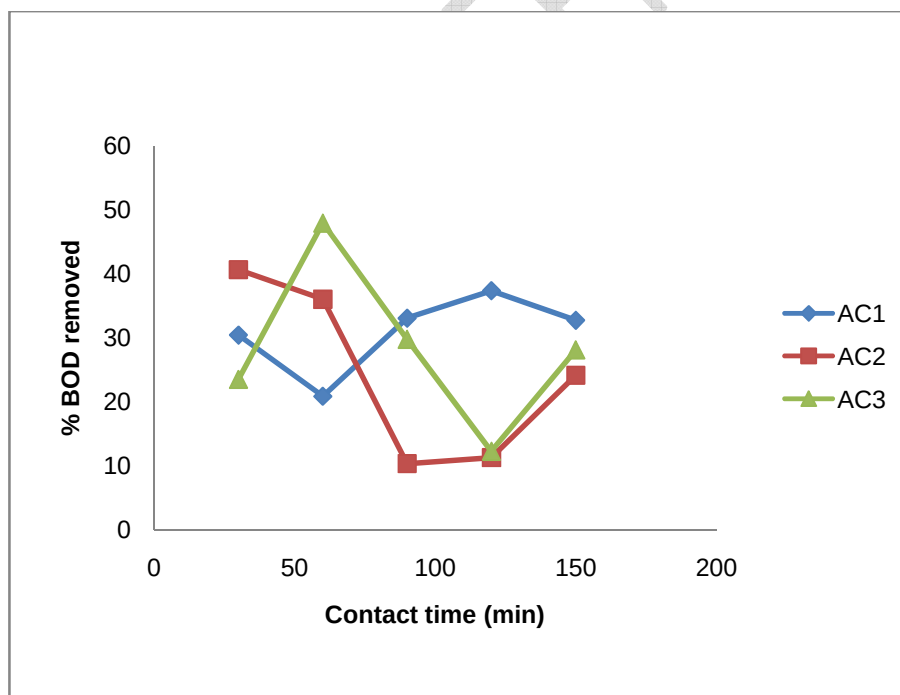
Parameter	PD/H ₃ PO ₄ /5	PD/H ₃ PO ₄ /10	PD/H ₃ PO ₄ /15
Activation burn off(%)	14.12 ± 0.01	20.01 ± 0.11	28.15 ± 0.01
Yield (%)	86.12 ± 0.01	80.05 ± 0.03	72.23 ± 0.02
Bulk density (g/cm^3)	0.52 ± 0.01	0.52 ± 0.02	0.51 ± 0.11
pH	6.5 ± 0.01	6.3 ± 0.03	6.7 ± 0.11

183	Conductivity ($\mu\text{S/cm}$)	1.6 ± 0.02	1.5 ± 0.11	1.9 ± 0.01
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185 3.2 Effect of Contact Times

186 The result shows that for PD/H₃PO₄/5 the adsorption rate was rapid for the first 90 minutes, reach
 187 equilibrium at 120 minutes, that is 37.29% removed thereafter decrease at 150 minutes while for
 188 PD/H₃PO₄/15 the equilibrium time was attained at faster rate of 60 minutes, that is 47.85% adsorbed
 189 then desorption occur at 90 minutes. For PD/H₃PO₄/10 the adsorption rate was higher at the initial
 190 contact time of 30 minutes then decrease between 60-150 minutes contact time respectively (Figure
 191 1). The initial faster rate may be due to the availability of the uncovered surface area of the
 192 adsorbents, since the adsorption depend on the surface area of the adsorbents. The BOD adsorption
 193 takes place at the more reactive sites. As these sites are progressively filled the more difficult the
 194 sorption becomes, as the sorption process tends to be more unfavourable. This is general
 195 characteristic of adsorption (Johnson, 1990; Appel and Lena, 2002; Abdus-Salam and Adekola,
 196 2005; Qadeer and Akhtar, 2005).



197

198 **Figure 1: Effect of contact time on BOD removed by poultry dropping adsorbent.**

199 **AC1 = PD/H₃PO₄/5 AC2= PD/H₃PO₄/10 AC3 =PD/H₃PO₄/15**

3.3 Adsorption Kinetic

Kinetic models like pseudo first-order, pseudo second-order, Natarajan and Khalaf first order and Elovich model were tested for the adsorption process. Sample with correlation coefficient (R^2) values close or equal to 1 is accepted for a given model or with R^2 values within the range $0.5 \leq R^2 \leq 1$ successfully describe the adsorption kinetics. Pseudo second-order showed higher R^2 values than the other kinetic model tested (Table 2- Table 5) with the accepted value greater than 0.980 (Figure 2). The result indicated that pseudo second-order fitted the BOD adsorption onto poultry dropping activated carbon. Badmus *et al.*(2007) reported a pseudo second-order kinetic adsorption model.

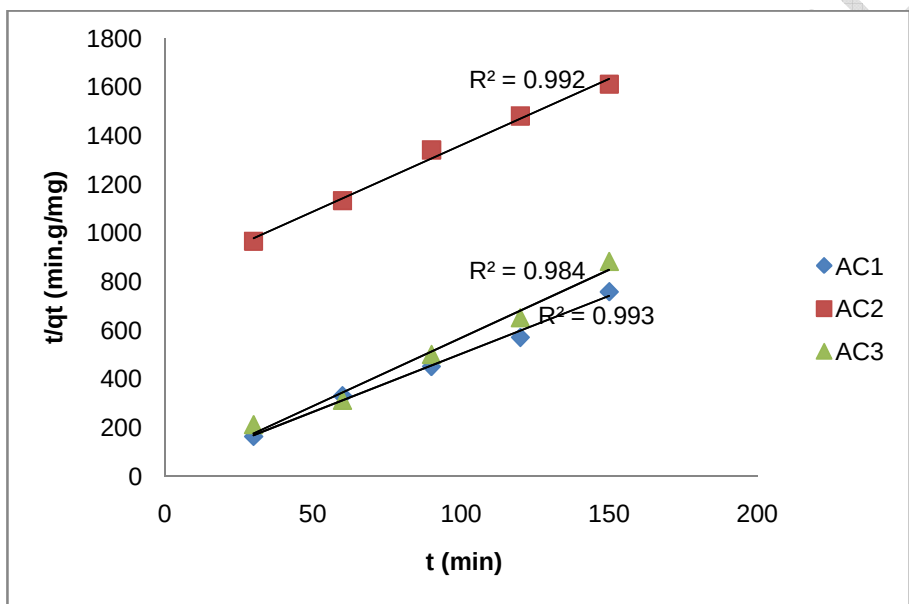


Figure 2. Pseudo second-order model for BOD adsorption onto poultry dropping activated Carbon

Table 2: Kinetic parameters of the pseudo first-order for BOD adsorption.

Sample	R^2	$K_1(\text{min}^{-1})$	$q_{e(\text{exp})}(\text{mg/g})$	$q_{e(\text{cal})}(\text{mg/g})$	% SSE
PD/H ₃ PO ₄ /5	0.025	-0.005	0.226	0.050	0.0787
PD/H ₃ PO ₄ /10	0.114	0.009	0.246	0.368	0.0546
PD/H ₃ PO ₄ /15	0.116	0.005	0.290	0.379	0.0398

Table 3: Kinetic parameters of the pseudo second-order for BOD adsorption.

Sample	R^2	$K_2(\text{g mg}^{-1}\text{min}^{-1})$	$q_{e(\text{exp})}(\text{mg/g})$	$q_{e(\text{cal})}(\text{mg/g})$	% SSE
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219	PD/H ₃ PO ₄ /5	0.993	0.168	0.226	0.241	0.0067
220	PD/H ₃ PO ₄ /10	0.992	0.576	0.246	0.238	0.0036
221	PD/H ₃ PO ₄ /15	0.984	-0.592	0.290	0.276	0.0063

222

223 **Table 4: Kinetic parameters of the Elovich model for BOD adsorption.**

224	Sample	R ²	β(g/mg)	α(mg/g/min)
225	PD/H ₃ PO ₄ /5	0.223	37.04	0.3112
226	PD/H ₃ PO ₄ /10	0.488	-9.90	-0.0003
227	PD/H ₃ PO ₄ /15	0.050	-37.04	-5.63X10 ⁻⁷

228 **Table 5: Kinetic parameters of the Natarajan and Khalaf first order for BOD adsorption.**

229	Sample	R ²	K ₁ (min ⁻¹)
230	PD/H ₃ PO ₄ /5	0.304	0.000
231	PD/H ₃ PO ₄ /10	0.411	-0.002
232	PD/H ₃ PO ₄ /15	0.107	0.000

233

234 3.4 Test of Kinetic Models

235 Applicability of pseudo first-order and pseudo second-order were tested using percentage sum of
 236 error square (% SSE). The higher the value of R² and the lower the values of % SSE, the better will
 237 be the goodness of the fit. Also sample with least % SSE is accepted for a given model. The lower
 238 values of % SSE of pseudo second-order model than that of pseudo first-order support the suitability
 239 of pseudo second-order kinetic model to explain the adsorption onto carbonized poultry droppings
 240 (Table 2 and Table 3). Similar result was reported by Senthilkumar *et al.* (2010).

241 3.5 Intraparticle Diffusion Model

242 Adsorption is a multi-step process involving transport of the solute molecules from the aqueous phase
 243 to the surface of the solid particulate, followed by diffusion of the solute molecules into the pore
 244 interiors. If the value of C is zero, then the rate of adsorption is controlled by intraparticle diffusion for
 245 the adsorption process (Hameed, 2009). As seen from Table 6, if the values of C are approximate to
 246 a whole number it give zero indicating the adsorption is intraparticle diffusion dependent. Similar
 247 result was obtained by Ofomaja (2006).

Table 6: Intraparticle diffusion parameters for BOD adsorption.

Sample	R ²	K _p (mg/g/min ^{1/2})	C
PD/H ₃ PO ₄ /5	0.165	0.001	0.319
PD/H ₃ PO ₄ /10	0.069	-0.001	0.284
PD/H ₃ PO ₄ /15	0.018	0.000	0.121

4. CONCLUSION

Poultry droppings has been successfully used to produce high quality activated carbon because of high carbon content. Batch adsorption method was adopted at the different contact time. The highest BOD removal was 47.85% at the contact of 60 minutes. Adsorption kinetic data were analysed using the pseudo first-order, pseudo second-order, Natarajan and khalaf first order and Elovich model. The results revealed that the process can be best described by a pseudo second-order model. The advantage of using this model is that the adsorption capacity and the rate constant of pseudo second-order can be predicted from the equation. The use of poultry droppings and other related waste as an adsorbent should be encourage to keep our environment clean.

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333 waste.

334 **APPENDIX**

335 PD/H₃PO₄/5, PD/H₃PO₄/10 and PD/H₃PO₄/15 are poultry droppings activated using H₃PO₄ as an
336 activating agent at the residual time of 5, 10 and 15 minutes respectively.

337 Residual time is the time the activated carbon spend in the furnace at 800°C to increase the porosity
338 and better adsorption site for the activated carbon.

339