EVALUATION OF ACTIVATED CARBON PRODUCED FROM NUTSHELL OF TERMINALIA CATAPPA IN DYE WASTEWATER REMEDIATION.

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ABSTRACT:
Activated carbons were produced from terminalia catappa nutshell using H3PO4 as activating agent. The activated carbons were activated with the activating agent for 5, 10 and 15 minutes and characterised. The results shows the ranges of % yield, % burn off, bulk density (g/cm^3) and pH of activated carbons from nutshell to be 17.67±0.04 to 28.94±0.07, 71.06±0.02 to 82.33±0.05, 0.49±0.03 to 0.57±0.01 and 6.31±0.02 to 6.64±0.01 respectively, while ranges for Conductivity (µS/cm), % Moisture content and % ash content are 0.68±0.03 to 1.51±0.01, 4.20±0.03 to 7.03±0.04 and 6.50±0.03 to 13.50±0.04 respectively. Each of these adsorbents was interacted with dye wastewater for 30, 60, 90, 120 and 150 minutes and their BOD and COD determined before and after interaction with dye wastewater. The highest percentage BOD and COD of the dye wastewater reduction were 75.81% and 94.44 % respectively. Analysis of the results showed that the adsorption kinetics followed pseudo second order kinetics with R^2 values > 0.93 and close agreement between the $qe_{(cal)}$ and $qe_{(exp.)}$. Hence, the activated carbons produced can find application in various wastewater remediations.

Keywords: Activated carbon, BOD, COD, terminalia catappa, adsorption, isotherm.

1.1 Introduction

In water treatment plants, pre-chlorination, post chlorination, sedimentation, fluoridation, alum coagulation with hydraulic flocculation, dual anthracite and sand filtration are all used for disinfection and reduction of organic and metallic substances from water. Activated carbon (AC) has been widely used worldwide as an effective filtration or adsorption material for removing biological and chemical contaminants from drinking water, (Hapazari *et al.*, 2011). In most developing countries, the AC is imported at high cost, limiting the quantities of safe drinking water available to the people (Misihairabgwi *et al.*, 2007).

In Nigeria and other developing countries, Acs are imported in enormous quantities at a very high cost to meet their requirement, whereas large quantity of agricultural residues, which can be used for the production of Ac to meet local demands and even for exportation, are generated annually. These agricultural residues can be converted into Ac adsorbents, which can be used for water and waste water treatments, to remove hazardous organic compounds or those that impart odour or taste, cleanup off gases containing volatile organic compounds, decolourisation, solvent recovery and purification, treatment of industrial waste, surface and groundwater redemption, food processing, pharmaceutical and environmental remediation, amongst other uses. Alot of income can be generated from production of AC with the price of about US $450.00/ton, (Olafadehan *et al.*, 2012). The high cost of AC has simulated interest in examining the feasibility of using cheaper raw materials for its production. Agricultural wastes/byproducts are considered important precursors for production of AC not only because of their low cost but because they are renewable. Besides, it is a way of recycling agricultural wastes which could otherwise constitute environmental pollution, especially as it relates to solid waste management (Akpen *et al.*, 2011).

Water pollution due to dyeing industry is another issue of great concern since large quantity of effluent is discharged into the water bodies. The dye effluent is highly toxic in nature as it contains high suspended solid, chemical oxygen demand (COD), dye and chemicals along with high concentration of heavy metals like Cu, Cd, Zn Ni and Pb (David *et al*., 2014).

The discharge of dyes from paper and pulp, textile and tanning industries releases colour into the water. A combination of several dye classes in one mixture could have an effect on the rate of reduction and degree of decolourization, making the reduction process challenging. The primary reason for this is the presence of diversified functional groups in different classes of dyes (Gupta *et al.*, 2015). Colour in the water affects the nature of water, inhibit sunlight penetration and reduces photosynthetic action. Some of the dyes that cause rapid depletion of dissolved oxygen are toxic and carcinogenic. Thus, the use of dye contaminated water without any treatment may cause adverse effect on human health, domestic animals, wildlife and on the environment. Therefore, it is necessary to remove colour from the industrial effluent before it is discharged (Dada *et al*., 2012). AC is the easiest, cheaper and promising answer to achieving water pollution remediation.

Literature Review

1.2.1 Comparison of ACs Activated with Different Activating Agents

Jibril *et al*. (2008) prepared Ac from palm stems (*Phoenix dactylifera*) at Nizwa region. They were carbonized at different temperatures (400–600 °C) to investigate the effects of their impregnation with aqueous solution of either H_3PO_4 (85 wt %) or KOH (3 wt %). In the presence of either H_3PO_4 or KOH, the layer of fibrous cellular structure was destroyed at all temperatures with corresponding generation of porous surfaces. Generally, H_3PO_4 treated precursor yielded larger number of more regular pores. Surface area of 1100 m^2/g was obtained from the sample carbonized at 500°C. AC was prepared from birch by using various activation procedures:

a) treatment with H_3PO_4 and pyrolysis at 600°C in inert atmosphere,
b) the same as in (a) treatment with H_3PO_4 and pyrolysis at 600°C in inert atmosphere followed by steam activation at the same temperature and
c) treatment with H_3PO_4 and direct pyrolysis in a steam of water vapour at 700°C.

The results obtained indicated that the surface area and the porosity of the Acs were strongly dependent on the treatment after impregnation with H_3PO_4. Ac prepared by impregnation with H_3PO_4 followed by steam pyrolysis (steam activation) had highly developed porous structure and the largest surface area among all the prepared carbons (iodine number 1280 mg/g and BET surface area 1360 m^2/g) (Budinova *et al*., 2006). Gimba and Turoti (2006) investigated the adsorption efficiency of $FeCl_3$, $CaCl_2$ and K_2CO_3 Acs produced from previously carbonized coconut shell on some adsorbates obtained from an industrial effluent and wastewaters as well as the colour of molasses. The results showed that the Acs used in this study are capable of reducing the level of colour present in molasses and amount of oil/grease from a textile effluent. 92 % and 97.4 % of molasses and oil/grease respectively can be adsorbed onto 500 µm particle size of $ZnCl_2$ – Ac at room temperature. Out of these ACs, $FeCl_3$-AC is the most effective in adsorbing the chemical oxygen demand, biochemical oxygen demand, dissolved suspended and total solids present in a sample of a textile effluent.

1.2.2 Adsorption Kinetic Studies

In order to evaluate the kinetic parameters, pseudo-first order and pseudo-second-order models were used to analyze the adsorption kinetics (Demirbas *et-al.*, 2004). Adsorption kinetics governs the solute uptake rate, measures the adsorption efficiency of the adsorbent, and determine its applicability for explaining the experimental data (Ghasemian and Palizban, 2016). The applicability of pseudo first-order and pseudo second-order kinetic models can be verified through the sum of error squares (SSE, %) given by:

$$\% \text{ SSE} = \sqrt{\sum(qe_{(exp)} - qe_{(cal)})^2/N} \quad \ldots\ldots \quad (1)$$

$qe_{(exp)}$ = adsorption capacity at equilibrium experimental (mg/g).
$qe_{(cal)}$ = adsorption capacity at equilibrium calculated (mg/g).
N = number of data point (Abechi *et al*., 2011)

2.0 Materials and methods

2.1 Sample collection and pre-treatment

The matured and sun dried *Terminalia catappa* fruits were collected by hand picking from the trees at Usmanu Danfodiyo University Sokoto, Sokoto State, Nigeria. The fruits obtained were washed, dried and crushed to remove the nuts. The deseeded fruits were washed thoroughly with water and dried in an oven and further crushed to separate the pericarp from the shells. The shells were then powdered with mortar and pestle and sieved with 2.0mm mesh. The less than 2.0mm sample were kept in air tight container.

Dye wastewater (DWW) sample was collected from local dyes at marina in Sokoto metropolis. The bottles for sample collection were thoroughly cleaned with hydrochloric acid, washed with tap water to render free of acid, washed with distilled water twice, again rinsed with the water sample to be collected and then filled up the bottle with the sample leaving only a small air gap at the top (Ram, Pravin and Deepali, 2011). The experiments were conducted in chemistry laboratory, Usman Danfodiyo University Sokoto.

2.2 Production of Activated Carbon

2.2.1 Carbonization (pyrolysis)

Cleaned and pre-weighed crucibles were used to weigh 5 g of the sample (crushed nutshell). This was then placed in a muffle furnace and the temperature allowed to rise to 600 ^{o}C for 5 minutes after which it was poured from the crucible into bath of ice block. The excess water was drain off. The carbonized sample was washed with 0.1M HCl to remove surface ash. This was further washed with hot water and distilled water to remove residual acid, sun dried and dried in oven at 100°C for 1 hour. These processes were repeated until a substantial amount was obtained (Turoti *et al*., 2007).

2.2.2 Activation of the Carbonized Sample

The activation was achieved by mixing 5 g of the carbonized sample with 5cm^3 of the activating agent (1 M H_3PO_4). This was then allowed to stand for 2 hours, after which it was introduced into a furnace and heated at 800℃ for 5 minutes. The activated sample was cooled with ice-cold water, the excess water was drained off and the sample dried in an oven at 110 ^{o}C overnight. The above procedure was repeated for different dwelling time (10 min. and 15 min.) until substantial amount of each of the Acs were obtained. The activated samples were then washed with 0.1 M

HCl to remove surface ash, followed by hot water wash and rinsing with distilled water to remove residual acid. Washing was completed when the pH of the supernatant fall within 6-7, then the sample was dried in an oven at 110℃ overnight and stored in air tight container (Musah, 2011).

2.3 Physico-chemical parameters of the Acs (AC)

Determination of Moisture and ash contents, bulk density, activation burn off, % yield of AC, pH and conductivity measurements were carried out according to the standard method (Aziza, *et al.*, 2008).

2.4 Batch adsorption studies

In the adsorption studies; 2 g of each of the Ac were mixed with 40 cm^3 of each of the wastewater samples in a beaker and covered. These were allowed to stand for 30 minutes and filtered, using Whatman filter paper (No. 42).This process was repeated at pre-set times (60, 90, 120 and 150 minutes). Each mixture was separately filtered and the filtrates collected. This was continued until 500 cm^3 of each of the filtrates was obtained. These were used for the determination of BOD and COD (Hameed, Din and Ahmad, 2006). The BOD and COD of the wastewater samples were also determined before interacting with the Ac. The BOD and COD ex periments were conducted on the sampling dates (Hur *et al.*, 2010).

2.5 Determination of BOD and COD of Dye wastewater (DWW)

The BOD and COD of dye wastewater (DWW) sample were determined according to the procedures adopted by Idris *et al.*, (2013) and Olafadehan and Jinadu, (2012).

2.6 Determination of Adsorption Capacity

The adsorption capacity at time (qt) was determined using the following formular:

$$q_t = \frac{(Ci - Cf)V}{W} \quad \ldots\ldots\ldots\

Bulk density	0.56±0.02	0.49±0.03	0.57±0.01
pH	6.53±0.01	6.31±0.02	6.64±0.01
Conductivity	0.68±0.0	1.51±0.01	0.68±0.04
% Moisture	5.50±0.06	7.03±0.04	4.20±0.03
% Ash content	6.50±0.03	13.00±0.06	13.50±0.04

AC_1, AC_2, and AC_3 are ACs activated for 5, 10 and 15 minutes respectively.

Table 3.2: Pseudo first order kinetic studies experimental constants for the reduction of DWW BOD by using the ACs.

Adsorbent	R^2	$K_1(min^{-1})$	qe(cal.)(mg/g)	qe (exp.)(mg/g)	%SSE
AC_1	0.80	0.0235	19.83	33.60	2.75
AC_2	0.59	0.0161	14.97	32.80	3.57
AC_3	0.68	0.0161	16.99	35.10	3.62

Table 3.3: Pseudo first order kinetic studies experimental constants for the reduction of DWW COD by using the ACs.

Adsorbent	R^2	$K_1(min^{-1})$	qe(cal.)(mg/g)	qe (exp.)(mg/g)	%SSE
AC_1	0.47	0.0100	4.05	62.40	11.67
AC_2	0.93	0.0138	6.43	68.00	12.31
AC_3	0.01	-0.0012	0.80	66.40	13.12

Table 3.4: Pseudo second order kinetic studies experimental constants for the reduction of DW BOD by using the ACs.

Adsorbent	R^2	$K_2(min^{-1})$	qe(cal.)(mg/g)	qe (exp.)(mg/g)	%SSE
	0.97	0.0008	42.55	33.60	1.79

AC1a					
AC1b	0.96	0.0015	35.09	32.80	0.46
AC1c	0.96	0.0014	37.74	35.10	0.53

Table 3.5: Pseudo second order kinetic studies experimental constants for the reduction of DW COD by using the ACs.

Adsorbent	R^2	$K_2(min^{-1})$	qe(cal.)(mg/g)	qe (exp.)(mg/g)	%SSE
AC1a	1.00	0.0054	63.69	62.40	0.061
AC1b	0.97	0.0039	69.44	68.00	1.089
AC1c	1.00	0.0340	66.67	66.40	0.533

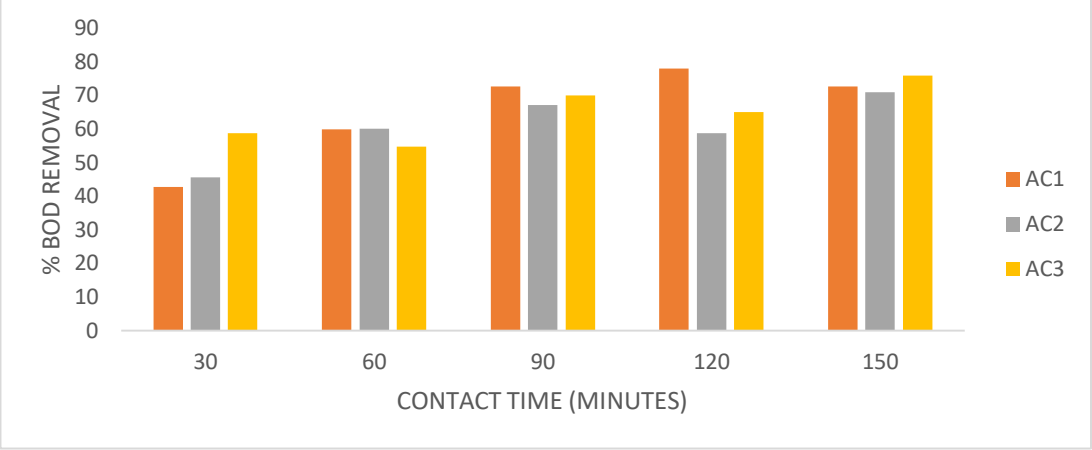

Figure 1. % BOD reduction from DWW by derived the adsorbents

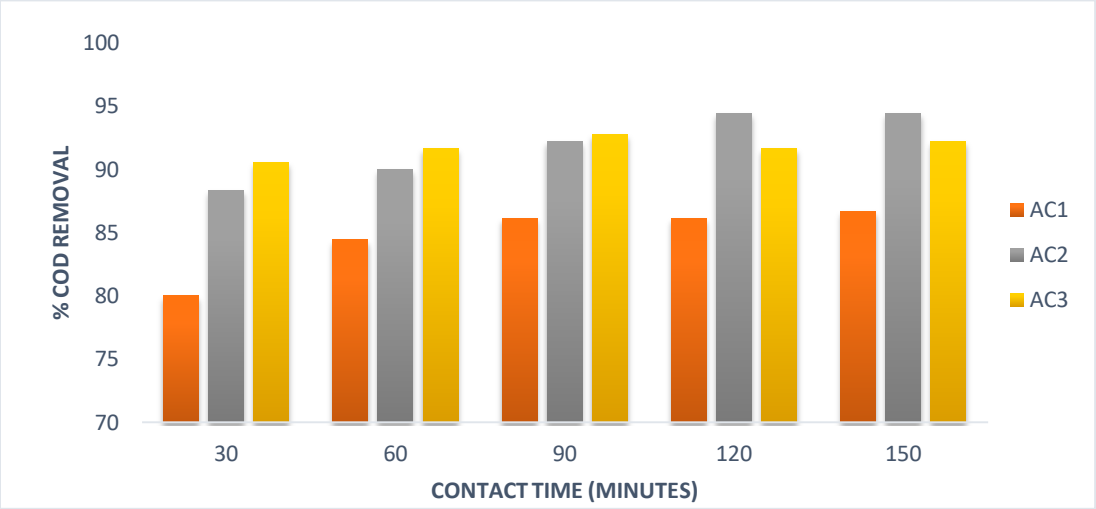

Figure 2: % COD reduction from DWW by the derived adsorbents.

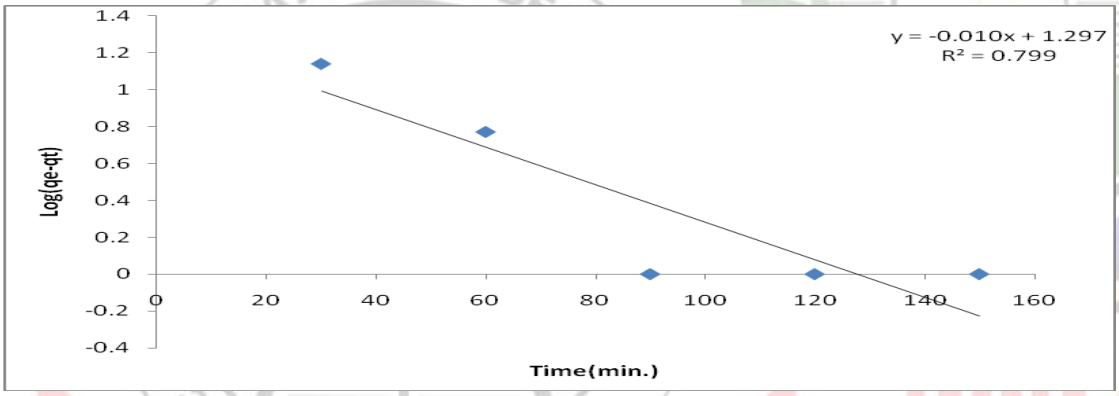

Fig. 3 Pseudo first order Kinetic plot for BOD reduction from DW using ACI

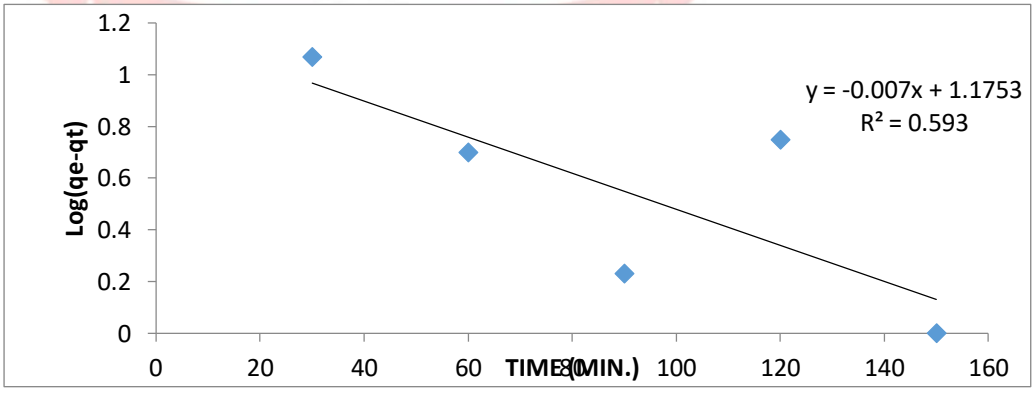

Fig. 4 Pseudo first order Kinetic plot for BOD reduction from DW using AC2

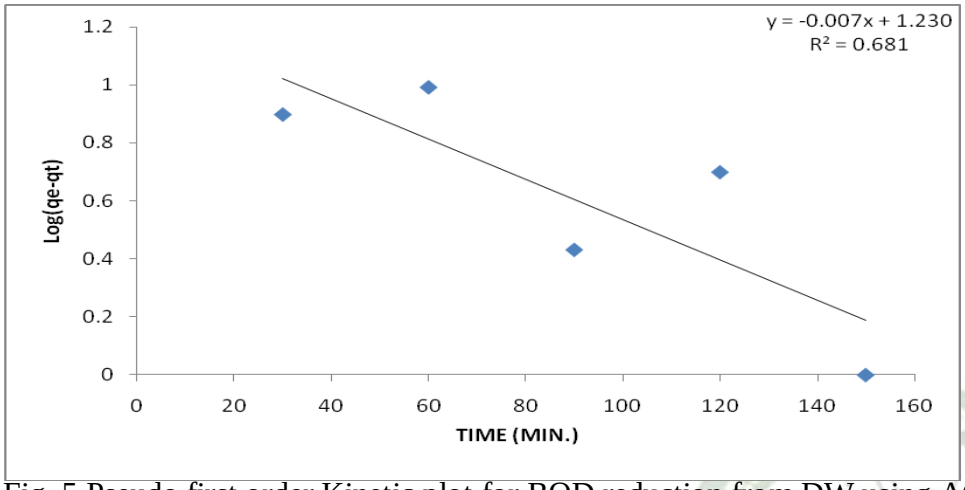

Fig. 5 Pseudo first order Kinetic plot for BOD reduction from DW using AC3

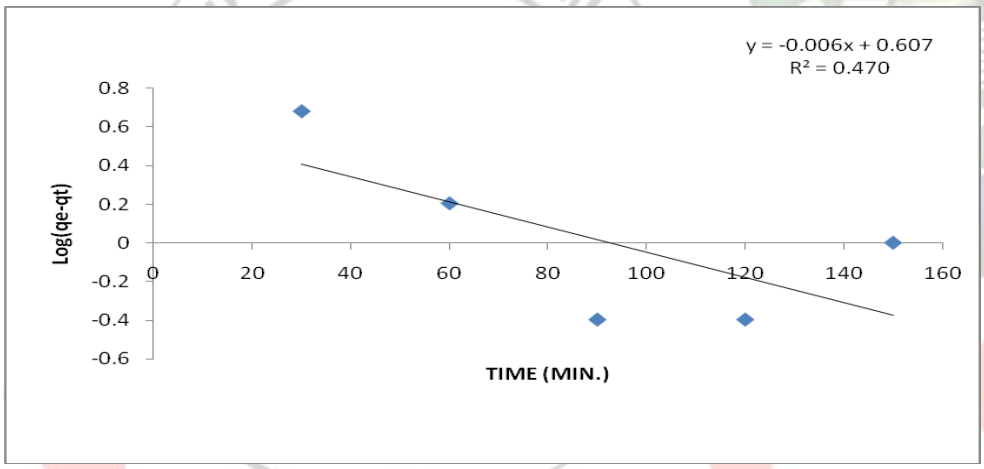

Fig. 6 Pseudo first order Kinetic plot for COD reduction from DW using ACI

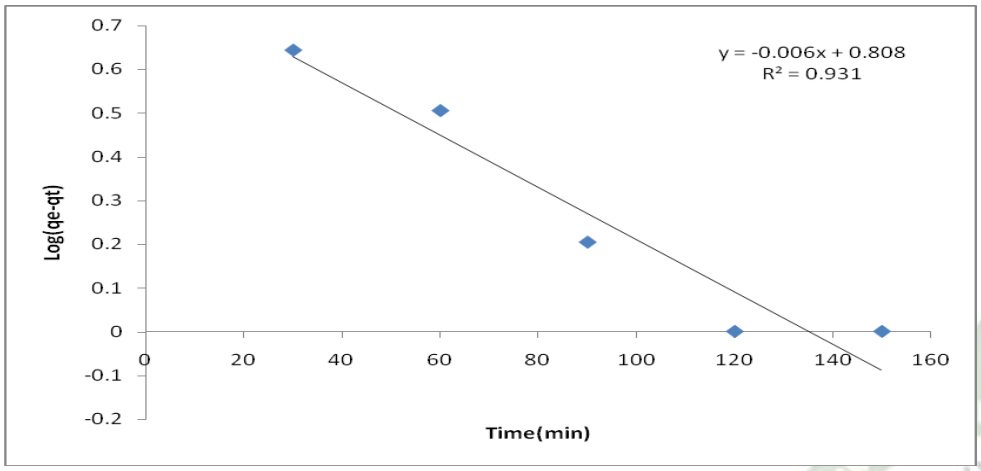

Fig. 7 Pseudo first order Kinetic plot for COD reduction from DW using AC2

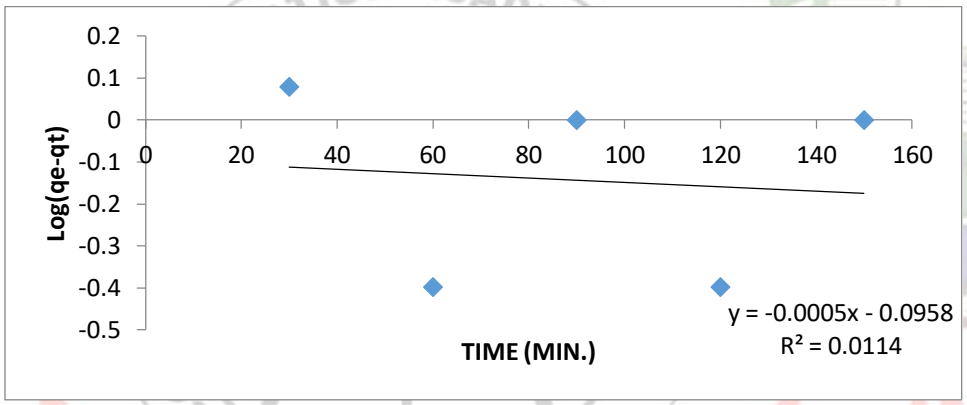

Fig. 8 Pseudo first order Kinetic plot for COD reduction from DW using AC3

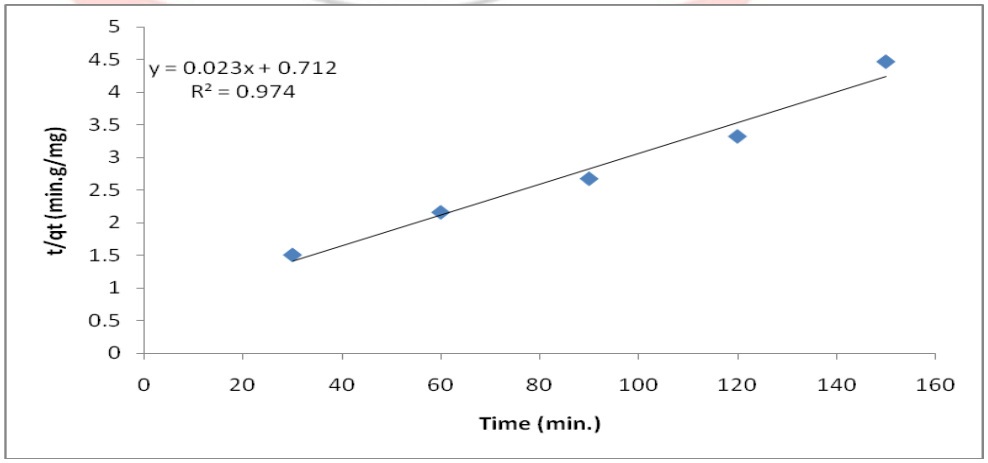

Fig.9 Pseudo second order Kinetic plot for BOD reduction from DW using AC1

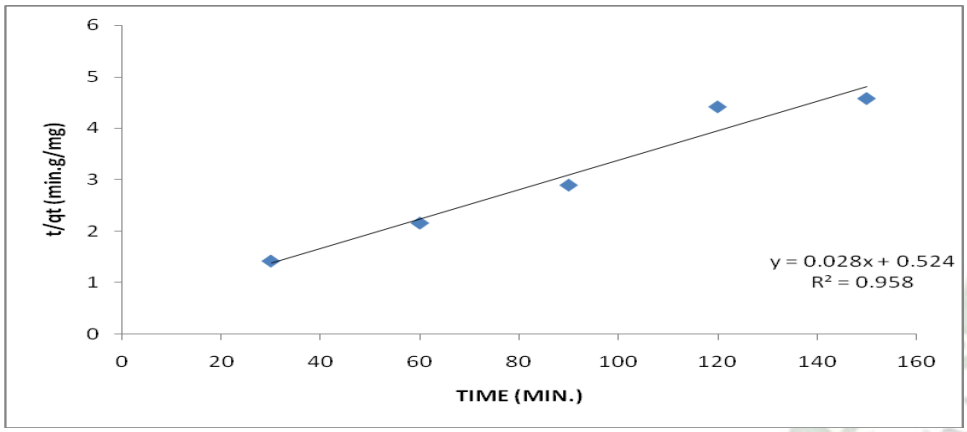

Fig. 10 Pseudo second order Kinetic plot for BOD reduction from DW using AC2

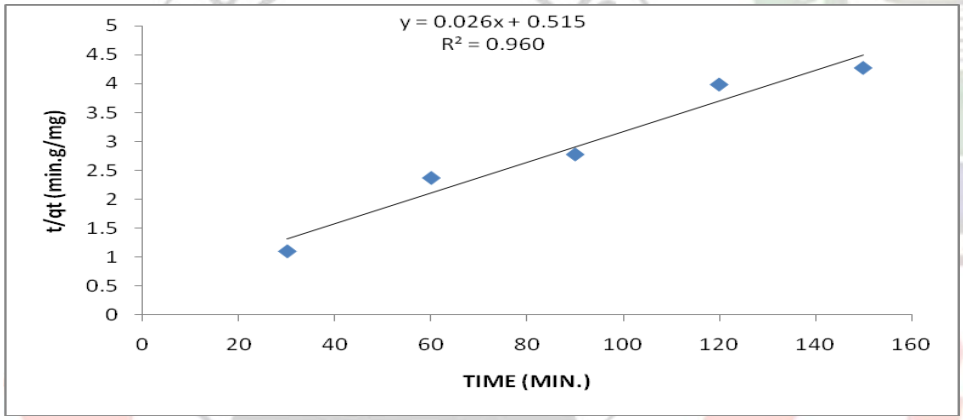

Fig. 11 Pseudo second order kinetic plot for BOD reduction from DW using AC3

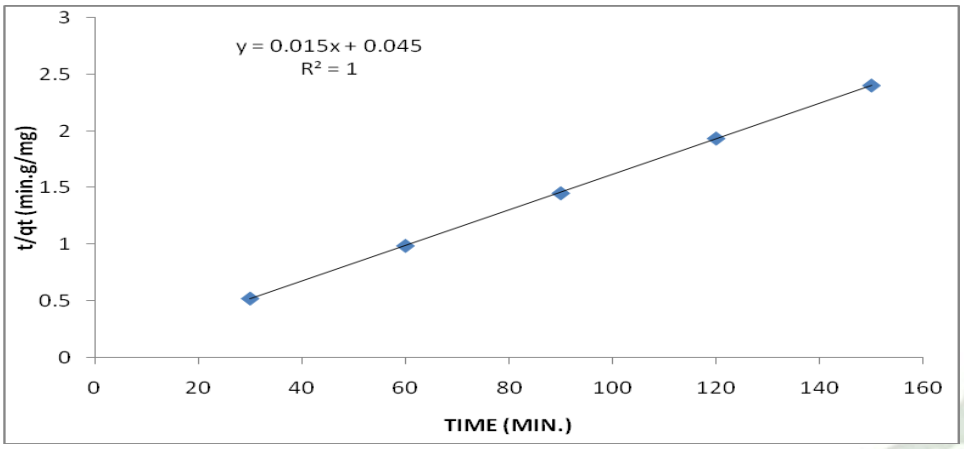

Fig. 12 Pseudo second order Kinetic plot for COD reduction from DW using AC1

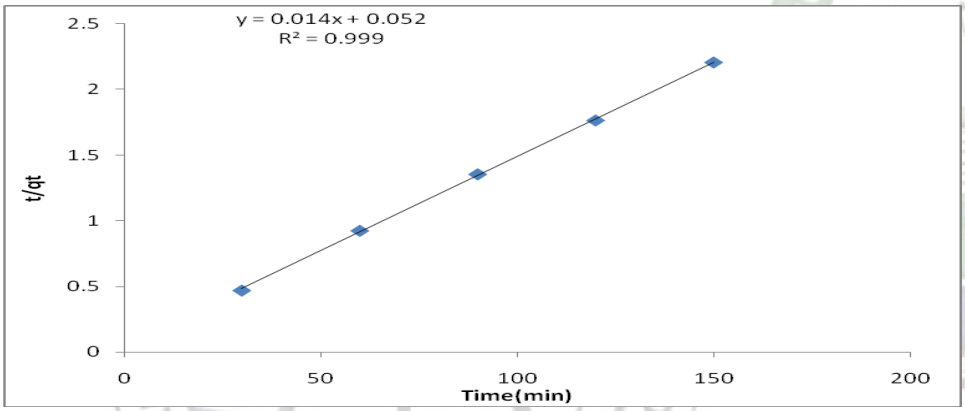

Fig. 13 Pseudo second order kinetic plot for COD reduction from DW using AC2

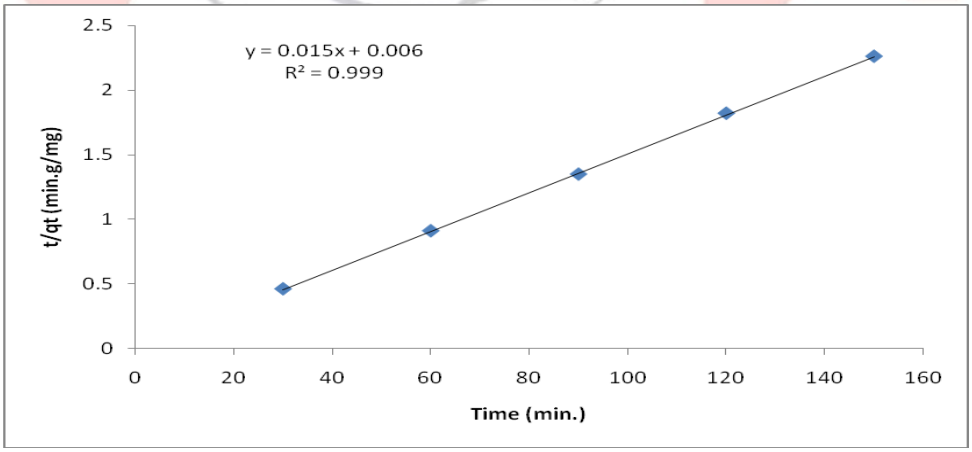

Fig.14 Pseudo second order kinetic for COD reduction from DW using AC3

3.2.0 Discussion

3.2.1 Physicochemical Parameters of Derived Adsorbent

Tables 3.1 shows the % yields,% burn offs, bulk densities, conductivities, % moisture and ash co ntents of the Acs. The % yields of the Acs activated for 5, 10 and 15 minutes activation times (AC1, AC2 and AC3), are 28.94±0.07%, 22.41±0.03 and 17.67±0.04% respectively while the corresponding % burn offs are 71.06±0.02, 77.59±0.03 and 82.33±0.05 respectively. The % burn offs increase with increase in activation time while the % yield decrease with increase in % burn offs. At longer activation time, more volatile substances are released which leads to increase in % burn offs with corresponding decrease in % yield. From the above results, the % yield is low as a result of double thermal decomposition resulting from initial pyrolysis followed by activation (Itodo 2008). The bulk densities of the Acs activated for 5, 10 and 15 minutes are 0.56±0.02, 0.49±0.03 and 0.57±0.01 (g/cm^3) respectively. These values are higher than the bulk density of 0.42 (g/cm^3) of the Ac prepared from Thevetia Peruviana wood activated with phosphoric acid (Raffiea *et al.,* 2012) and are lower than the bulk density of 0.62g/cm^3 for the Ac prepared from palm kernel (Abechi *et al.,* 2013). The bulk density is an important characteristic of carbon and is invariably related to the starting material. It is an important parameter of powdered solids. The American Water Work Association has set a lower limit on bulk density at 0.25 g/ml for Ac to be of practical use (Veena *et al.,* 2012) cited by (Shalna and Yogamoorthi, 2015). The bulk density values of the Acs satisfies this condition. The range of pH values for the Acs is 6.31±0.02 - 6.64±0.01. This rhymed with the pH of 6.30 for rice husk Ac activated with phosphoric acid (Abbas *et al.,* 2013). This range is above the pH values of 5.44 for maize cob Ac (Emmanuel *et al.,* 2013) and 5.80±0.71 for acid activated coconut shell Ac, Amuda and Ibrahim, (2006), and less than the pH of 6.8 for Thevetia Peruviana impregnated with phosphoric acid (Raffiea *et al.,* 2012). It has been reported by Ahmedna *et al.,* (2000) that for most applications, carbon pH of 6-8 is acceptable.

The % ash content of the ACs activated for 5, 10 and 15 minutes are 6.5±0.03, 13.00±0.06 and 13.50±0.04 respectively. % ash contents of 2.94 % for Lapsi seed stone Ac and 34.80 % for Jatropha pericarp Ac activated with sodium chloride which are both outside the range were reported by (Shrestha *et al.,*2012) and (Okeola *et al.,* 2012) respectively. The % moisture contents of the ACs activated for 5, 10 and 15 minutes are 5.50±0.06, 7.03±0.04 and 4.50±0.03 respectively. The moisture contents of 1.90% for used tea dust activated with phosphoric acid (Shalna and Yogamoorthi, 2015) and 0.27 % for phosphoric acid activated Lapsi seed stone carbon (Shrestha *et al.,* 2012) are lower than the observed values. The % moisture contents of 5.21 for banana empty fruits bunch Ac (Sugumaran *et al.,* 2012) and 5.60 for kenaf core fibre activated with phosphoric acid (Shamsuddin *et al.,* 2015) were within the range of the produced

Acs while the moisture contents of 13.20% for Thevetia Peruviana Ac activated with phosphoric acid (Raffiea *et al.*, 2012) is higher than the observed values for the produced Acs. Even though moisture content of the carbon has no effect on its adsorptive power, it dilutes the carbon and necessitates the use of additional weight carbon during treatment process (Raffiea *et al.*, 2012). The conductivity test reveals the presence of impurities (ions) on the AC. The conductivity values of the ACs activated for 5, 10 and 15 minutes are 0.68±0.03, 1.51±0.01 and 0.68±0.04 respectively. The conductivity value of 51.00±0.6 (µS/cm) for coconut shell AC (Amuda and Ibrahim, 2006) and that of *Delonix regia* fruit pod which is 353.66±3.51 (µS/cm) (Sugumaran *et al.*, 2012) are higher than the conductivity value of the produced ACs. The conductivity of 1.5±0.11mS for poultry droppings Ac activated with phosphoric acid for 10 minutes (Idris *et al.*, 2013) is similar to that of the produced Ac activated for 10 minutes. The low conductivity values of the produced adsorbents showed that they contain low leachable waste, which are undesirable.

3.2.2 BOD and COD Reduction in Dye Wastewater (DWW)

The highest % reduction of DWW BOD by the AC activated for 5 minutes was achieved at 120 minutes interaction time, that is, 77.97% (Figure 1). The AC activated for 10 minutes have the highest % DWW BOD reductions at 150 minutes interaction time, which is 70.84 % while the Ac activated for 15 minutes has its optimum % DWW BOD reduction at 15 minutes interaction time, that is, 75.81%. Comparatively, 47.85 % BOD was reduced by Ac prepared from poultry droppings at 60 minutes contact time in dye effluent (Idris *et al.*, 2013) which is lower than the observed values.

The optimum reduction of DWW COD by the Ac activated for 5 minutes was 86.67 % at 150 minutes interaction time while 94.44 % was reduced by the Ac activated for 10 minutes. The Ac activated for 15 minutes has its highest COD reduction at 90 minutes interaction time, which was 92.78 % (Figure 2). Comparatively, 65.15 % of COD was reduced by carbonized poultry waste at both 120 and 150 minutes contact time in DWW (Idris *et-al.*, 2012) which is lower than the values obtained

in this work while higher % COD reduction of 99.18 % was achieved in coffee processing plant wastewater by using Avocado peel carbon at optimum operating conditions (Devi *et al.*, 2008).

3.2.4 Kinetic Studies of BOD and COD of DWW

Pseudo first and second order models were employed to evaluate the adsorption kinetic of BOD and COD of the DWW. The accepted kinetic model for a given adsorption is characterized by three common validity tests.

i. A good and high correlation coefficient (R^2) indicates the applicability and reliability of a given model.
ii. A close agreement between the calculated and the experimental qe values.

iii. The accepted model must have the least sum of error squares (SSE) (Itodo and Itodo, 2010).
From pseudo first order equation, log (qe-qt) against t was plotted (Fig. 3 - 8). The first order rate constant (K_1), calculated adsorption capacity at equilibrium ($qe_{(cal)}$) and the correlation coefficients (R^2) were determined from the model for each of the three different samples of activated carbons (AC1, AC2 and AC3).

The pseudo second order model was also used to plot graphs of t/qt against t (Fig. 9-14) from which pseudo second order rate constants (K_2), $qe_{(cal)}$ and the corresponding correlation coefficients of the different samples of the Acs used were determined. The values of these parameters were presented in tables 3.7 for DWW BOD and 3.8 for DWW COD.

In the pseudo first order parameters, there are wide variations between the $qe_{(cal)}$ and the $qe_{(exp)}$ values (tables 3.2-3.3). The correlation coefficients (R^2) values range between 0.01-0.93 while the % SSE have very low range of 2.75-13.12. The range of 0.87-28.97 was reported for the adsorption of methylene blue dye on termite soil (Russiarani and Balakrisharan, 2015) which was lower than the range of 40.412-42.118 for sheanut shell biosorbent for textile wastewater treatment (Itodo and Itodo, 2010).

For the pseudo second order model, there are close agreement between the qe(cal) and the $q_{e(exp)}$ values (tables 3.4-3.5). The values of the correlation coefficients (R^2) are very high with a range of 0.96-1.00 while the values of the % statistical sum of error squares (% SSE) have a range of 0.061-1.79 (Tables 3.4-3.5).

4.0 Conclusion

The results of this research shows that the Ac from nutshell of *Terminalia Catappa activated* with $H_3PO_{4(aq)}$ is an effective adsorbent for the adsorption of BOD and COD (organic pollutants) in DWW. This implies that it is an essential commodity which can be used to generate wealth. This Ac can equally be used as an alternative for the expensive commercial Ac in waste water treatment.

The results of physico-chemical parameters of the Acs (tables 3.1) revealed that both Acs have high % burn offs (71.o6±0.02 to 82.33±0.05) with corresponding low % yield (17.67±0.04 to 28.94±0.07) which was due to the two steps method of production of the Ac. Low conductivity values (0.68±0.03 to 1.51±0.01) were obtained for the Acs which indicated that the adsorbate reduction was by physical adsorption in each case. The % ash content of the Acs was in the range of 6.50±0.03 to 13.00±0.06 while the % moisture content was found to be in the range of 4.20±0.03 to 7.03±0.04.

The capacities of the Acs in the adsorption of BOD and COD (organic pollutant) in DWW showed appreciable % reductions. The highest value of the maximum % DWW BOD reduction by the Ac was 77.97 % which was shown by the Ac activated for 5 minutes (AC1) at interaction time of 120 minutes. The highest maximum % DWW COD reduction by the Ac was 94.44 % which was shown by the Ac activated for 10 minutes (AC2) at interaction time of 150 minutes.

The kinetic modeling of DWW BOD and COD adsorption kinetic followed pseudo second order kinetic model with correlation coefficient (R^2) value of 0.69 and % sum of error squares (% SSE) of 2.75. The close agreement of the $qe_{(cal)}$ and the $qe_{(exp)}$ values further favoured the second order adsorption kinetic model.

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