

Research Article

Effect of Refluxing Time and Kinetics of Synthetic Organic Chemicals Removal in Aqueous Solutions by Carbons Produced from Nipa Palm Fronds

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Abstract

The refluxing time is a significant consideration in synthetic study, because it could reflect the kinetics of the reaction process. Synthetic Organic Chemicals (SOCs) are man-made carbon-based compounds that are not likely to evaporate into the atmosphere and hence they could get into aquatic water bodies through terrestrial runoff or discharge from factories. Therefore, this paper investigated the effect of refluxing time and kinetics of Synthetic Organic Chemicals (SOCs) removal in aqueous solutions by carbonized and surface-modified carbons made from Nipa Palm (*Nypa Fruticans* Wurmb) fronds using chemical oxygen demand (COD) as the measurement parameter. The data showed a rapid reduction of the COD of the SOC's contaminated solutions from 30.19 to 93.46% for PCC, 27.44 to 65.58% for AAC, 41.84 to 98.22% for BAC and 56.71 to 95.16% for CAC between 10 to 60 mins. Optimum reduction was achieved within 20 min of heating the solutions at 150 °C. The rapid COD reduction observed for n-propanol indicates that COD is a rapid, inexpensive means of determining organics in water. Kinetic assessment of the results showed that, pseudo-first order kinetic equation did not provide a very good description of COD reduction of the SOC's in aqueous solution by the Nipa palm derived carbons. However, Nipa palm had been adjudged as a beneficial, eco-friendly and locally available source for the development of activated carbon for elimination of organic pollutants in domestic and industrial wastewaters.

Keywords

Synthetic Organic Chemicals, Chemical Oxygen Demand, Kinetics of Adsorption, Nipa Palm, Carbonized Carbon, Wastewater

1. Introduction

Reflux involves heating the chemical reaction for a specific amount of time, while continually cooling the vapour produced back into liquid form, using a condenser. The vapours produced above the reaction continually undergo condensation, returning to the flask as a condensate. The contact time between the adsorbent and the adsorbate is an important parameter governing the extent of an adsorption process. This is

because it provides information on the sorption kinetics of the adsorbate for a given initial dosage of the adsorbent [1]. Synthetic organic chemicals (SOCs) are organic compounds that originate from domestic, commercial and industrial activities. Many of these compounds have been identified in wastewater and water supplies [2-6]. These compounds have adverse health effects. Also their presence in water may re-

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duce dissolved oxygen concentrations, which is detrimental to aquatic life [5, 7]. Therefore, treatment of wastewater is necessary to correct its characteristics in such a way that its use or final disposal can take place without causing an adverse impact on the ecosystem of the receiving water bodies. Conventional treatment methods for removal of synthetic organic Chemicals from aqueous solution, such as photochemical degradation, biological degradation, coagulation, chemical oxidation and adsorption have been investigated by several workers [8-13]. However, Adsorption is the most successful of these methods due to its simplicity and low cost [14, 15]. Chemical oxygen demand (COD) water analysis is critical in wastewater for determining the amount of organic waste contamination in the water. Waste that is high in organic matter requires treatment to reduce the amount of organic waste before discharging into recipient waters. Chemical Oxygen Demand (COD) is a major parameter used as routine surrogate tests for measuring the load of organic carbon into the environment [16]. Also, COD is a cheap method for the determination of organic load in waste water [17]. Therefore, this paper investigated the effect of refluxing time and kinetics of Synthetic Organic Chemicals (SOCs) removal in aqueous solutions by carbonized and surface-modified carbons made from Nipa Palm (*Nypa Fruticans* Wurmb) fronds using chemical oxygen demand (COD) as the index of measurement.

2. Materials and Methods

Sampling and Processing: The methods used for sample collection, biomass preparation, carbonization of biomass, chemicals required and the instrumentation used has been previously described elsewhere [2, 3].

Activation of Biomass: Three activated carbons and a commercial carbon were prepared. They are (i) Commercial Activated Carbon (CAC) (ii) Physically activated Carbon (PCC), (iii) Acidic reagent activated carbon (AAC) and (iv) Basic reagent activated carbon (BAC). The methodology has been carefully and exhaustively presented in previous works [2, 3].

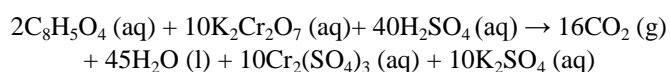
2.1. Determination of Effect of Reflux Time

2.0 g each of PCC, AAC, and BAC of 106 μm mesh particle size and CAC were weighed and put in various 250 ml conical flasks. 50 ml of 100 mgL^{-1} DMABA, kerosene and n-propanol solution were added to the flasks to form a suspension. The pH values of these suspensions were adjusted to 6.0. The flasks were labelled for refluxing time interval of 10, 20, 30, 40, 50, 60 minutes. The mixtures were transferred to 150 ml plastic sample containers and shaken for 60 min at 150 rpm. At the end of 60min, the suspensions were allowed to stand on the bench for 30 min to separate and then filtered using Whatman No 40 filter paper. The clear supernatant SOC solutions were analyzed for final COD (COD_f).

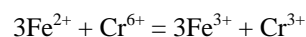
2.2. Determination of Chemical Oxygen Demand (COD)

COD measurement is a two-step process which are digestion and determination [5, 18]. The chemistry of these two processes is explained below.

Digestion Step: organic matter is oxidized by dichromate ions in sulphuric acid to CO_2 and H_2O . During heating the dichromate ions ($\text{Cr}_2\text{O}_7^{2-}$) form orange-colored solutions.



Determination Step: During titration FAS reacts with dichromate solution to form green coloured chromic ion solution. After end point, FAS reacts with indicator to form brown colour solution



To determine the COD of each sample, 10 ml 0.125 M standard potassium dichromate solution was added to 20 ml of the different solutions and the clear solutions in a 250 ml round-bottom flask. 1.0 g silver sulphate and 40 ml conc. sulphuric acid were added in small portions with careful swirling until the silver sulphate was completely dissolved. A few glass beads were added to serve as anti-bumping aid, and the flask was connected to reflux condenser. The mixture was heated gently for 10 minutes or more as the case may be, after which the content of the flask was cooled. 50 ml distilled water was flushed through the condenser, and the cooling was completed under running tap water. 2 drops of indicator solution were added and the resulting mixture titrated with standardized 0.025 M ferrous ammonium sulphate (FAS) solution until there was a change in colour from yellow-green via blue-green to reddish brown. 20 ml distilled water as blank and 20 ml clear solution from batch adsorption studies were also taken through the same process and their COD values determined.

Computation of COD: The COD values of the respective solutions and blank were estimated as presented in eqn 1.

$$\text{COD as mg O}_2/\text{L} = \frac{(A-B) \times m \times 8000}{V_s} \quad (1)$$

Where A = ml of ferrous ammonium sulphate (FAS) used for blank; B = ml of FAS used for sample; M = molarity of FAS; V_s = Volume of sample used (ml) and 8000 = milliequivalent weight of oxygen $\times 1000$ ml/L.

Calculation of% COD Reduction: Calculation of percent reduction of chemical oxygen demand (COD) in the solutions after being contacted with the carbons was estimated by Eqn 2.

$$\% \text{ COD Removed} = \frac{\text{COD}_i - \text{COD}_f}{\text{COD}_i} \times 100 \quad (2)$$

Where COD_i = COD concentration of initial SOC working solutions before interactions with the carbons (mg/L) and COD_f = COD concentration of SOC working solutions after interactions with the carbons (mg/L).

2.3. Kinetic Model Parameters for Batch Removal

The time dependent experimental data in this study was analysed using first and second - order pseudo kinetic models.

Pseudo – First Order Kinetics: The linearized form of the pseudo – first order equation presented in eqn 2.

$$\log (q_e - q_t) = \log q_e - (k_1/2.303)t \quad (3)$$

Where q_e is the amount of SOCs adsorbed by PCC, AAC, BAC and CAC at equilibrium (mg/g), q_t is the amount of SOCs adsorbed at time t (mg/g), k_1 is the first order rate constant (min^{-1}) and t is time in (min).

The straight line plot of $\log (q_e - q_t)$ against time t , should give a linear relationship from which the pseudo-first order rate constant (k_1) and equilibrium sorption capacity (q_e), can be calculated from the slope and intercept respectively.

Pseudo – Second order Kinetics: The linearized form of the kinetic rate expression for a pseudo second-order model as expressed by Ho and Co-workers [19] was applied to the experimental data using eqn 4

$$\frac{t}{q_t} = \frac{1}{h_o} + \frac{1}{q_e} \quad (4)$$

Where q_e is the amount of SOCs adsorbed by PCC, AAC, BAC and CAC at equilibrium (mg/g), q_t is the amount of SOCs adsorbed at time t (mg/g), k_1 is the first order rate constant (min^{-1}) and t is time in (min). The parameters h_o and k_2 are determined experimentally from the slope and intercept of a plot of $\frac{t}{q_t}$ against t .

2.4. Analytical Precision and Quality Control

Care was taken during sample collection and preservation. Glassware was properly cleaned and reagents were of analytical grade. Reagent blanks, method blanks and working standards/solutions were prepared freshly. For quality control purpose a commercial activated carbon (CAC) with the trade name *Aqua Nucha* was purchased and used as a control. *Aqua Nucha* was supplied by Rovet Scientific Ltd, Benin City.

2.5. Statistical Analysis of Experimental Data

The reliability of experimental results was obtained by carrying out triplicate analysis and calculation of the mean, standard deviations, standard errors and analysis of variance (ANOVA).

3. Results and Discussion

3.1. Effect of Refluxing Time on COD Reduction

COD is a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant. The COD determination uses a strong chemical oxidant (potassium dichromate), acid, and heat to oxidize organic carbon to carbon dioxide and water. During the course of digestion, these reagents oxidize organic carbon compounds to CO_2 and H_2O . The refluxing time is essential in the consideration of the optimum COD reduction of the organic contaminant. Lower temperature increases adsorption capacity except in the case of viscous liquids. The effect of reflux time on the COD reduction of DMABA, kerosene and n-propanol was investigated at fixed initial SOCs concentrations and fixed refluxing temperature of 150°C . The consequence of refluxing time on the COD reduction of DMABA by PCC, AAC, BAC and the CAC is presented in figure 1. The COD reduction characteristics for DMABA indicate that the decomposition time is independent on the type of adsorbent and maximum reduction was achieved within 10 to 15 min of digestion and equilibrium attained in 20 min. The equilibrium time indicate the completeness of digestion. The rapid digestion time indicates that easy-to-digest samples can be analyzed with confidence in a short period of time.

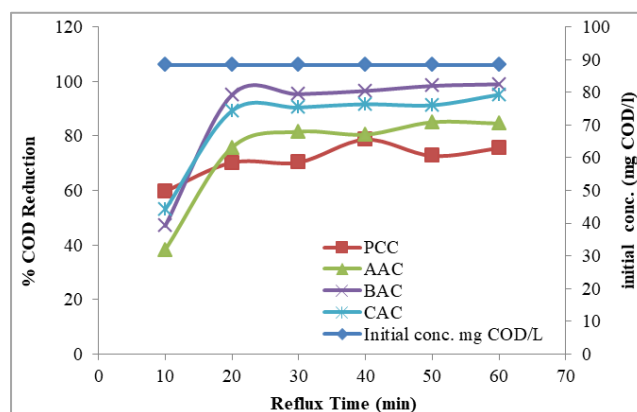


Figure 1. Effect of refluxing time on COD reduction of DMABA solution by PCC, AAC, BAC and CAC at 30°C .

The COD reduction of kerosene at a fixed pH of 6.8 and recommended temperature of 150°C by PCC, AAC, BAC and CAC was further studied for refluxing times varying from 10 to 60 min and is presented in figure 2. The data clearly demonstrate that COD reductions for kerosene solutions increased from 10 to 50 min with equilibrium occurring at 45 min. This extended digestion period for kerosene as compared to DMABA could be as a result of the high viscosity of kerosene and also the presence of more resistant

organic compounds in kerosene. Also iso-propyl alcohol was used as solvent for kerosene, therefore the extended refluxing time could be the time required to complete the digestion kerosene on one hand and iso-propyl alcohol on the other hand.

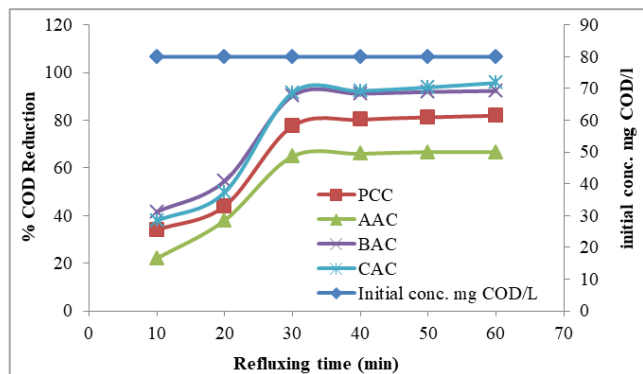


Figure 2. Effect of refluxing time on COD reduction of kerosene solution by PCC, AAC, BAC and CAC at 30 °C.

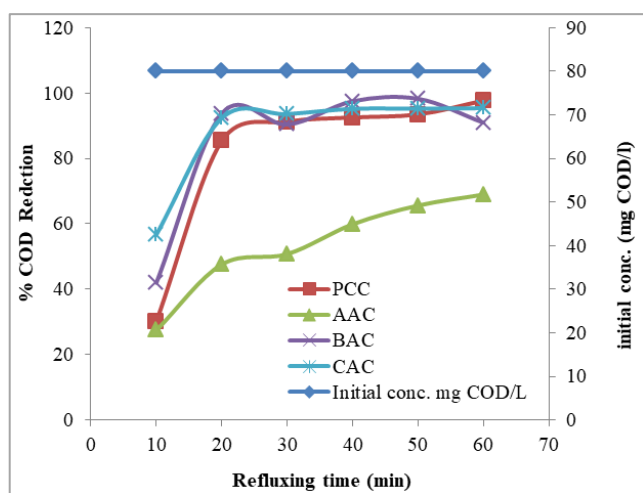


Figure 3. Effect of refluxing time on COD reduction of n-propanol solution by PCC, AAC, BAC and CAC at 30 °C.

The COD reduction of n-propanol solution using PCC, AAC, BAC and CAC is presented in figure 3. The data showed a rapid reduction of COD from 30.19 to 93.46% for PCC, 27.44 to 65.58% for AAC, 41.84 to 98.22% for BAC and 56.71 to 95.16% for CAC between 10 to 60 mins. Optimum reduction was achieved within 20 min of heating the solutions at 150 °C. The rapid COD reduction observed for n-propanol indicates that COD is a rapid, inexpensive means of determining organics in water.

3.2. Kinetic Adsorption Models for Batch Experimental Data

Kinetics describes the solute uptake rate that controls the

residence time of sorbate uptake at the solid-solution interface. Kinetic studies are important in determining the time needed to reach equilibrium and examinations into the rates of adsorption can be used to develop models and an understanding of solutes on adsorbent surface. Kinetics is so important that, it can also be used to predict the rate of COD reduction of organic contaminants in water and wastewater which could be used in the design of appropriate sorption treatment plants. Sorption kinetic models that have been used widely are the Lagergren pseudo-first order rate equation [20-22] and recently the pseudo-second order kinetic expression developed by Ho and co-workers [23].

The study of kinetics in wastewater treatment is significant as it provides available insights into the reaction pathways and mechanisms of the sorption reactions. In an attempt to understand the sorption kinetic processes, various kinetic rate expression models were applied to the batch experimental data in this study. In this study, the pseudo-first order and pseudo-second order models were used to investigate the sorption kinetics of the experimental data. This is because these two kinetic models are the most versatile and most frequently used in research. Pseudo-reaction is a reaction in which one of the reactant species is present in large excess such that the rate of the reaction appears to be independent of the concentration of the that reactant species. This is due to the little changes in concentration of the excess species when compared with its concentration.

Thus making it appear virtually constant in concentration at the end of the reaction. In such reactions, the rate of reactions will appear to be dependent on the concentration of species that are much smaller in amount. The species present in smaller concentration is the limiting species. Thus if the rate of reaction is dependent on the concentration of the limiting species raised to the power one, the reaction is said to be pseudo-first order reaction; but if the rate of reaction is dependent on the concentration of the limiting species raised to the power two, then the reaction is called pseudo-second order reaction.

3.3. Pseudo – First Order Kinetics

The time-dependent experimental COD reduction data in this study was analyzed using the pseudo-first order rate equation by Lagergren [24] and used by Adowei and co [25]. Using that equation, plots of $\log (q_e - q_t)$ against time were made to confirm the rate model of the COD reduction capabilities of the Nipa palm derived carbons. These plots were used to estimate the pseudo-first order rate constant (k_1) and equilibrium sorption capacity (q_e) from the slope and intercept respectively.

The Lagergren pseudo-first order plot for DMABA by PCC, AAC, BAC and CAC is presented in figures 4, 5, 6 respectively.

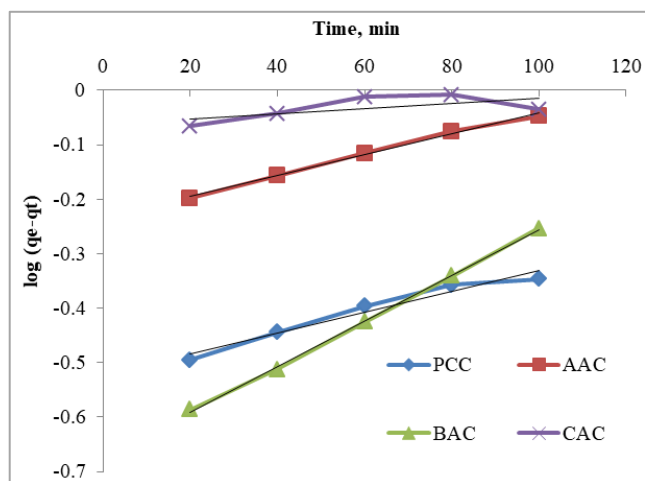


Figure 4. Lagergren Pseudo-first order sorption kinetic plots for COD reduction of DMABA by PCC, AAC, BAC and CAC.

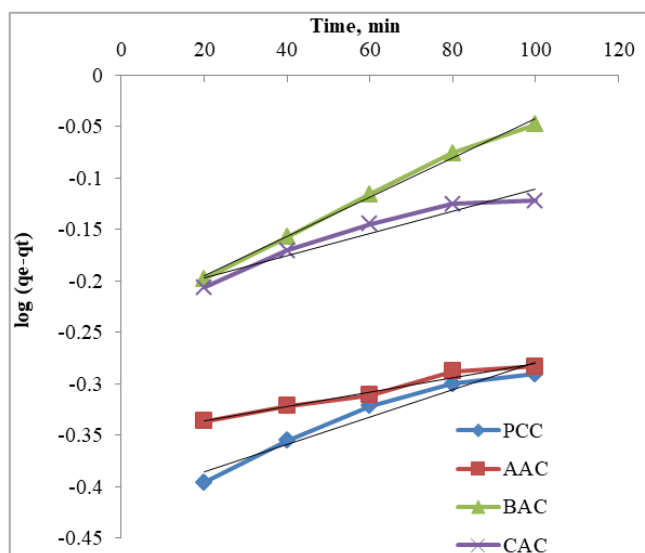


Figure 5. Lagergren Pseudo-first order sorption kinetic plots for COD reduction of kerosene solution by PCC, AAC, BAC and CAC.

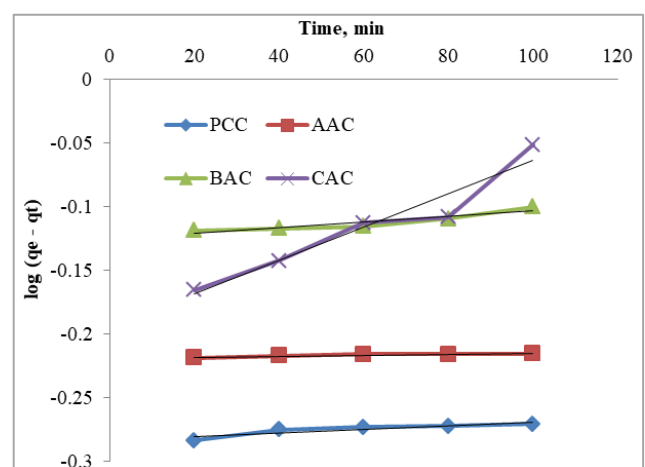


Figure 6. Lagergren Pseudo-first order sorption kinetic plots for COD reduction of n-propanol solution by PCC, AAC, BAC and CAC.

Pseudo-first order rate parameters obtained from the plots and presented in Table 1 showed that the rate constants (k_1) for COD reduction of DMABA are 4.38×10^{-3} , 1.15×10^{-3} , 9.66×10^{-3} and $4.38 \times 10^{-3} \text{ min}^{-1}$ for PCC, AAC, BAC and CAC; that of COD reduction of kerosene are 1.15×10^{-3} , 2.30×10^{-3} , 4.61×10^{-3} , $7.10 \times 10^{-3} \text{ min}^{-1}$ and for PCC, AAC, BAC and CAC, while COD reduction of n-propanol solution by PCC, AAC, BAC and the CAC gave 2.53×10^{-3} , 2.99×10^{-3} , 1.61×10^{-3} and $4.38 \times 10^{-3} \text{ min}^{-1}$ respectively.

The results showed that the pseudo-first order rate constants, k_1 , for COD reduction of DMABA in solution by all the carbons investigated were higher than n-propanol and kerosene. The values for the coefficient of determinations, r^2 , obtained from the Lagergren pseudo-first order kinetic equation plots are 0.854, 0.416, 0.822 0.854 for DMABA by PCC, AAC, BAC and CAC; 0.829, 0.784, 0.870, 0.931 for kerosene by PCC, AAC, BAC and CAC and 0.946, 0.925, 0.974 and 0.995 for n-propanol by PCC, AAC, BAC and CAC. The results showed that the coefficient of determination for COD reduction of n-propanol in aqueous solution by all the carbons investigated were higher than DMABA and kerosene. The non-linearity of the plots (figures 4, 5, 6) showed that the pseudo-first order equation was not adequate in describing the reactions between the three SOCs on the Nipa palm derived carbons.

3.4. Pseudo – Second Order Kinetics

Since there was no fit between the predicted curve and the experimental data points for the pseudo-first order kinetics, the pseudo-second order rate kinetic model developed by Ho and co-workers [19]. Plots of $\frac{t}{qt}$ against t were made for DMABA (Figure 7), kerosene (Figure 8) and n-propanol (Figure 9) to obtain pseudo-second order equation parameters which are presented in Table 1.

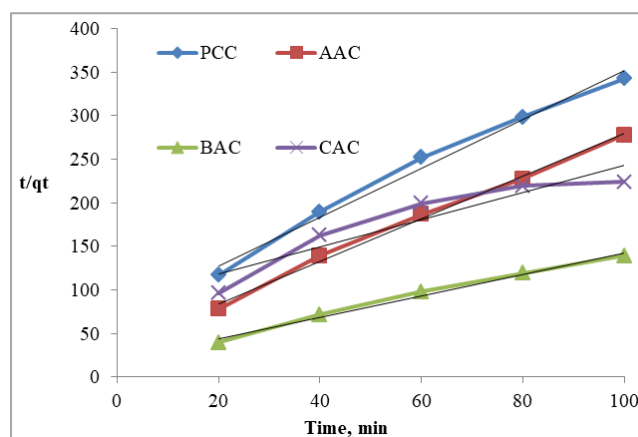


Figure 7. Pseudo-second order sorption kinetics plots of COD reduction of DMABA solution by PCC, AAC, BAC and CAC.

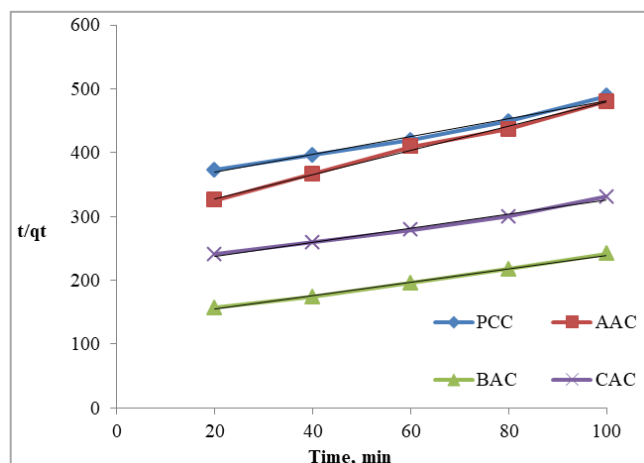


Figure 8. Pseudo-second order sorption kinetics plots of COD reduction of kerosene solution by PCC, AAC, BAC and CAC.

The results of the pseudo-second order kinetics as presented in Table 1. The equilibrium COD reduction capacities (mg/g) for sorption of DMABA; kerosene and n-propanol by the carbons are 13.94 (PCC), 2.80 (AAC), 51.77 (BAC), 53.48 (CAC); 7.05 (PCC), 5.28 (AAC), 9.51 (BAC), 9.02 (CAC) and 3.16 (PCC), 4.34 (AAC), 9.90 (BAC), 7.17 (CAC) respectively. The behaviour indicates that kerosene has the lowest on all the Nipa palm derived carbon and also on the commercial carbon.

The results as presented in Table 2 showed that the pseudo-second order rate constants, k_2 , were found to be 9.19×10^{-3} g/mg min (PCC), 26.0×10^{-3} g/mg min (AAC), 0.302×10^{-3} g/mg min (BAC), 0.224×10^{-3} g/mg min (CAC) for DMABA, 9.19×10^{-3} g/mg min (PCC), 26.0×10^{-3} g/mg min (AAC), 0.302×10^{-3} g/mg min (BAC), 0.224×10^{-3} g/mg min (CAC) for kerosene and 9.19×10^{-3} g/mg min (PCC), 26.0×10^{-3} g/mg min (AAC), 0.302×10^{-3} g/mg min (BAC), 0.224×10^{-3} g/mg min (CAC) for n-propanol respectively.

The coefficient of determination, r^2 , for the pseudo-second

order rate kinetic model for all the SOC's on the COD reduction by the Nipa palm derived carbons and the commercial carbon are greater than 0.995. This is an indication that the pseudo-second order rate law is an excellent criterion in describing the COD reduction behaviour of the carbons. Judging from the r^2 values (Table 3), the accuracy of the model of the COD reduction kinetic of the three SOC's is better described by a pseudo-second order chemical reaction; but much better for DMABA than kerosene and propanol. This is because, although DMABA is considered to be organic compound, it ionizes in solution to form positive ion. The structure of the carbons contain lactones and its surface in contact with water generates negatively charged species at the pH level studied, so it is likely that a chemical reaction may be taking place, which appears to be the main rate determining factor in the COD reduction process. In the adsorption of DMABA onto the carbons, the cationic dye carry charge opposite to that of the adsorbent, aiding the adsorption process.

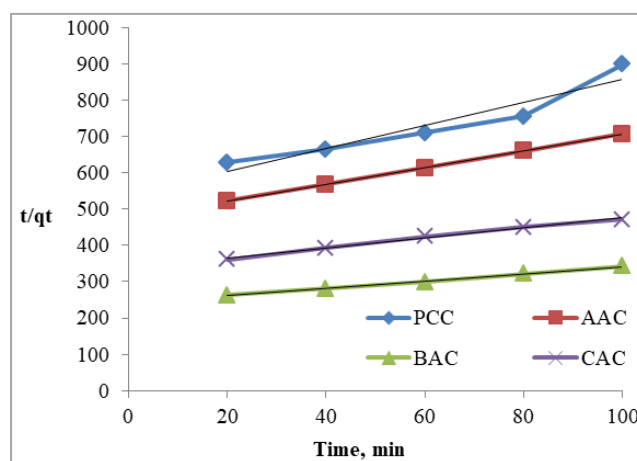


Figure 9. Pseudo-second order sorption kinetics plots of COD reduction of n-propanol solution by PCC, AAC, BAC and CAC.

Table 1. COD reduction capacity at equilibrium (mg/g) for pseudo-first and - second order rate models for COD reduction of SOC's in solutions by Nipa palm derived carbons and commercial activated carbon.

Carbons	Pseudo-First Order Kinetic model				Pseudo-Second Order Kinetic model			
	q_e , mg/g				q_e , mg/g			
	PCC	AAC	BAC	CAC	PCC	AAC	BAC	CAC
DMABA	5.32	2.10	6.10	7.90	6.41	2.80	8.77	9.48
Kerosene	2.59	1.47	2.40	4.10	4.60	1.92	3.94	5.95
n-propanol	3.67	2.85	4.56	5.32	4.16	3.33	6.97	7.17

q_e = COD reduction capacity at equilibrium mg/g

Table 2. Rate constants for pseudo-first and - second order rate models for COD reduction of SOC_s in solutions by Nipa palm derived carbons and commercial activated carbon.

Carbons	DMABA			Kero			N-P		
	$k_2 \times 10^{-3}$	h_0	$k_1 \times 10^{-3}$	$k_2 \times 10^{-3}$	h_0	$k_1 \times 10^{-3}$	$k_2 \times 10^{-3}$	h_0	$k_1 \times 10^{-3}$
PCC	9.19	0.357	4.38	7.05	0.295	1.15	9.21	0.184	2.53
AAC	6.0	0.411	1.15	5.28	0.893	2.30	5.57	0.210	2.99
BAC	3.02	0.809	9.66	9.51	0.414	4.61	2.20	0.414	1.61
CAC	2.24	0.641	4.38	9.02	0.862	2.99	2.89	0.297	4.38

k_1 = Pseudo-first order kinetic rate constant (min^{-1}); k_2 = Pseudo-second order rate constant ($\text{mg g}^{-1} \text{min}^{-1}$); h_0 = Ho's constant related to initial COD reduction rate ($\text{mg g}^{-1} \text{min}^{-1}$)

Table 3. Coefficient of determinations (r^2) for pseudo-first and - second order rate models for COD reduction of SOC_s in solutions by Nipa palm derived carbons and commercial activated carbon.

Carbons	Pseudo-First Order Kinetic model				Pseudo-Second Order Kinetic model			
	r^2				r^2			
	PCC	AAC	BAC	CAC	PCC	AAC	BAC	CAC
DMABA	0.854	0.416	0.822	0.854	0.997	0.995	0.999	0.997
Kerosene	0.829	0.784	0.870	0.873	0.985	0.996	0.997	0.998
n-propanol	0.846	0.825	0.874	0.895	0.903	0.999	0.998	0.995

r^2 = coefficient of determination

Physical adsorption and chemisorption may be indistinguishable in certain situations and in some cases agree of both types of bonding can be present, as with covalent bonds between two atoms having some degree of ionic character and vice versa. The mechanism of adsorption can also be described as chemisorption, involving valence forces through sharing or the exchange of electrons between adsorbent and adsorbate as covalent forces. Allen and others [26] confirmed the application of pseudo first-and second-order equations for the description of the adsorption kinetics of three basic dyes onto kudzu, finding the pseudo second-order equation to be more suitable and stating that the chemical reaction was important and significant in the rate-controlling step.

4. Conclusions

Reflux involves heating the chemical reaction for a specific amount of time, while continually cooling the vapour produced back into liquid form, using a condenser. Data obtained reveals that, within 30 minutes of refluxing, maximum breakdown of SOC_s has been achieved. Kinetic assessment of the results showed that the pseudo-second order kinetic equation gave

coefficients of determination were < 0.95 for the three SOC_s. The significance of this observation is that, the COD reduction is dependent on the concentration of the SOC_s present in solution. Amongst the Nipa palm carbons, BAC is adjudged to have a better efficiency than PCC and AAC. These removal potentials are significantly comparable to the commercial activated used to validate the carbons. The feasibility was further assessed by using separation factor; and the data obtained revealed that the interaction between the Nipa palm derived carbons and the various SOC_s investigated are favourable and the predominant mechanism is physisorption. The outcome of this investigation has revealed that carbons developed from Nipa palm leaves are an attractive option for organic contaminants removal from aqueous solution.

Abbreviations

AAC: Acidic Reagent Activated Carbon
 ANOVA: Analysis of Variance
 BAC: Basic Reagent Activated Carbon
 CAC: Commercial Activated Carbon
 COD: Chemical Oxygen Demand

DMABA: 2-(N, N-Dimethyl-4-aminophenyl)-azo-benzene carboxylic Acid

FAS: Ferrous Ammonium Sulphate

PCC: Physically Activated Carbon

SOCs: Synthetic Organic Chemicals

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Conflicts of Interest

The author declares no conflict of interest.

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