



## **Kinetics and Thermodynamics of 2,4,6-Trichlorophenol Adsorption onto Activated Carbon Derived from Flamboyant Pod Bark**

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**Abstract:** This work was designed to evaluate the suitability of activated carbon produced from flamboyant pod bark (FPBAC) for the removal of trichloro-phenol from polluted water. Various concentrations of trichloro-phenol polluted water was prepared and the adsorption experiments were performed at some selected adsorption factor ranges: agitation, 150 – 250 rpm; contact time, 60 – 120 min; adsorbent dose, 0.15 – 0.25 g; and initial trichloro-phenol concentrations, 100 – 200 mg/L. Results of the study showed that the selected adsorption factors has a significant effect on the adsorption capacity of FPBAC which was found to increase with increase agitation, contact time, adsorbent dosage and initial trichloro-phenol concentrations. The results of thermodynamic studies showed that the process is exothermic and Langmuir isotherm model with correlation coefficient ( $R^2$ ) of 0.919 best described the process. The kinetics of the process was best described by pseudo-second order model with correlation coefficient ( $R^2$ ) value of 0.999.

**Keywords:** *Flamboyant Pod Bark, Trichloro-phenol, Adsorption, Activated carbon,*

### **Introduction**

Water is an essential component of life whose quality plays a major role in human health (Baseri *et al.*, 2013). The increase in demand for clean water which either comes from freshwater or reusing of wastewater is related directly or indirectly to world population increase. Wastewater refers to water that has been adversely affected in quality as a result of human or industrial activities which make it unsafe for usage in its current form (Bansode *et al.*, 2004). Wastewater contains a complex mixture of solids and dissolved components which are normally present in very small concentrations composing of organic compounds (persistent organic pollutant, surfactants and oils), inorganic compounds (heavy metals and soluble ions), suspended solids as well as various gases such as oxygen and hydrogen sulphide (Amuda and Ibrahim, 2006). The continuous discharge of organic pollutants which are not degradable as effluents of manufacturing industries into water bodies poses a big threat to the global community and thus, a serious threat to human survival (Igwe *et al.*, 2003).

For environmental protection from health issues, wastewater containing organic pollutants needs proper methods of treatment that can remove the contaminants effectively (Harvey and Chantawong, 2001). Several methods of wastewater treatment such as adsorption, ion exchange, reverse osmosis, chemical oxidation, precipitation, distillation, solvent extraction and bio-remediation are available for the removal of pollutants. Among the various methods, adsorption has been established to be the most effective method for the removal of colour, odour, organic and inorganic pollutants from wastewater (Krishnaiah *et al.*, 2013).

Adsorption is a process that occurs when a gas or liquid solute accumulates on the surface of a solid or liquid which is known as adsorbent, forming a film of molecules or atoms called adsorbate (Goyal *et al.*, 2004). Adsorption using commercial activated carbon such as coal is expensive due to the non-renewability and high cost of starting materials. These bring about a search for renewable and cheaper agricultural by-products such for production of activated carbon for the removal of various pollutants from wastewater (Tan *et al.*, 2008). Activated carbon produced from high- carbon content agricultural residues possess good adsorbent properties and fast adsorption kinetics which makes it suitable for wastewater treatment and for adsorption of hazardous gases (Sugumaran *et al.*, 2012).

Phenol and its derivatives are toxic, weak acid, organic pollutants which can be found in the effluents of chemical industries such as agro-chemical industries, textile industries, paint industries, pulp and paper industries (Hameed *et al.*, 2009). It can easily permeate into the human skin in vitro

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and is readily absorbed by the gastrointestinal track. It has great adverse effects on human health which makes it a suitable candidate for this study.

Flamboyant tree is a large, deciduous tree with fern-like leaves and pendulous, elongated, woody, compressed pods up to 50 cm long considered as agricultural waste. The pods composed largely of cellulose, hemicelluloses, lignin, tannin and pectin. The ligno-cellulosic component of the flamboyant pods is responsible for its porous and fibrous nature suitable for adsorption purposes (Sugumaran *et al.*, 2012). Its high content of essential functional groups such as hydroxyl, carboxyl and methoxyls required for binding with the pollutants give it high binding ability and good adsorption properties on a wide range of pollutants. Hence, there is need for studies into the significance of adsorption factors on its suitability as adsorbent even in the presence of complex interactions.

## Materials and Method

### Materials

Activated carbon produced from flamboyant pod bark (FPBAC) from previous work (Aremu *et al.*, 2017), 2,4,6-trichlorophenol (analytical grade), distilled water, UV-Spectrophotometer (UV-6100A). All glassware used were thoroughly washed with distilled water, and oven dried before use.

### Preparation of simulated water

50 mg/L of 2,4,6-trichlorophenol was prepared by dissolving 50 mg of 2,4,6 trichlorophenol in distilled water in appropriate standard volumetric flask. The procedure was repeated for preparation of 100, 150, 200 and 250 mg/L of 2,4,6-trichlorophenol (Alade *et al.*, 2012).

### Adsorption studies

Batch adsorption studies were carried out to evaluate the adsorption performance of the prepared adsorbent from the flamboyant bark pod. This was done by adding various dosage of the prepared activated carbon (FPBAC) to 25 ml each of the prepared different initial concentrations (50 mg/L, 100 mg/L, 150 mg/L, 200 mg/L and 250 mg/L) of 2,4,6-trichlorophenol in 100 mL conical flasks. Adsorption was allowed to proceed at different agitation rates and various time intervals. Samples were taken at pre-set time intervals and their concentrations determined. Filter paper was used to filter the content of each sample and the filtrate analyzed for residual 2,4,6-trichlorophenol using UV-Spectrophotometer (UV-6100A) at wavelength of 296 nm.

The percentage removal was evaluated using equation 1.1:

$$\text{Removal (\%)} = \frac{C_0 - C_t}{C_0} \quad 1.1$$

where,  $C_0$  and  $C_t$  are the liquid-phase 2,4,6-trichlorophenol concentrations at zero time and at any time  $t$ , respectively.

The adsorption capacity of the adsorbent (FPB) was evaluated using equation 1.2:

$$\text{Adsorption capacity} = \frac{(C_0 - C_t) V}{M} \quad 1.2$$

where,

$C_0$  (mg/L) is the initial concentration of 2,4,6-trichlorophenol in contact with adsorbent,

$C_t$  (mg/L) is the final concentration of 2,4,6-trichlorophenol after the batch adsorption procedure at any time  $t$ ,

$M$  (g) is the mass of adsorbent used and

$V$  is the volume of the aqueous solution in liter (L).

### Adsorption experiments

The adsorption experiments were performed at some selected factor ranges: agitation, 150 – 250 rpm, contact time, 60 – 120 min, adsorbent dose, 0.15 – 0.25 g and initial trichloro-phenol concentration, 100 – 200 mg/L. The selected ranges fall between the ranges used by previous researchers (Tan *et al.*, 2008). Equations of adsorption capacity was employed to determine the optimum conditions for adsorption. After establishing the optimum conditions (agitation, contact time, adsorbent dosage and initial concentration) for adsorption, one-factor-at-a-time (OFAT) was employed to study the effects of adsorption factors at the optimum conditions.

### Effects of Agitation

The effects of agitation on the removal of 2,4,6-trichlorophenol (TCP) was investigated by varying the rate of agitation of the contents of the conical flask in the rotary shaker from 100 - 250 rpm.

*Effects of Contact time*

The effects of contact time on the removal of 2,4,6-trichlorophenol (TCP) was investigated by varying the contact time of the FPBAC with TCP in the prepared water from 30 - 180 min.

*Effects of initial concentration of 2,4,6-trichlorophenol (TCP)*

The effects of the initial concentration of TCP on the adsorption capacity of the prepared FPBAC was investigated by varying the initial concentrations of 2,4,6-trichlorophenol in simulated water from 50 – 250 mg/L in contact with definite amount of the FPBAC in the conical flasks.

*Effects of Temperature*

The effects of temperature on the adsorption of 2,4,6-trichlorophenol (TCP) by FPBAC was investigated by varying the temperature of the thermostat incubator shaker from 30 – 60 °C.

**Adsorption Thermodynamics studies**

Temperature is an important parameter that can affect the adsorption capacity of activated carbon (FPBAC) and transport/kinetics of 2,4,6-trichlorophenol. The data obtained from the adsorption experiments was used to calculate the thermodynamic parameters. The main three thermodynamics parameters of the process; enthalpy ( $\Delta H^\circ$ ), entropy ( $\Delta S^\circ$ ) and free energy ( $\Delta G^\circ$ ) were calculated using the equations;

$$\Delta G = -\Delta RT \ln k_c \dots\dots\dots 1.5$$

$$\Delta G = \Delta H - T\Delta S \dots\dots\dots 1.6$$

Combining equation (1.5) and (1.6) gives equation 1.7:

$$\ln k_c = \frac{\Delta H}{R} \left(\frac{1}{T}\right) + \frac{\Delta S}{R} \dots\dots\dots 1.7$$

$$\text{and } k_c = \frac{q_e}{C_e} \dots\dots\dots 1.8$$

where, R is the gas constant (8.314 J/mol K), T is the temperature,  $k_c$  is the equilibrium constant,  $q_e$  is the concentration of the adsorbed 2,4,6-trichlorophenol (mg/L) and  $C_e$  is the concentration of remaining 2,4,6-trichlorophenol in solution (mg/L) (Aksu, 2002).

**Adsorption Isotherm studies**

The adsorption isotherm shows the movement of molecules between the solid phase and the liquid phase when the process reached an equilibrium state. In finding a suitable model that can describe this process, data obtained from the adsorption experiments were fitted to four different isothermic models namely Langmuir, Freundlich, Temkin and Dubinin-Radushkevich isotherms. The applicability of the model was evaluated by comparing the correlation coefficients,  $R^2$  values (Dada *et al.*, 2012).

*Langmuir Isotherm*

The Langmuir isotherm assumes monolayer adsorption sites of uniform energies of adsorption with no trans-migration of adsorbate in the surface planes (Dada *et al.*, 2012). The linear form of Langmuir equation is given in equation 1.9:

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{1}{q_m} C_e \dots\dots\dots 1.9$$

*Freundlich Isotherm*

Freundlich isotherm proposed that adsorption occurs on heterogeneous surfaces while interaction occurs between adsorbate molecules (Tan *et al.*, 2008). The linear form of Freundlich isotherm is shown in equation 1.9:

$$\ln q_e = \frac{1}{n} \ln C_e + \ln k_f \dots\dots\dots 1.9$$

*Temkin Isotherm*

Temkin isotherm model predicts the energy distribution pattern in the adsorbent layers and also the interaction between adsorbate. The linear plot of Temkin equation is given by equation 1.10:

$$q_e = B \ln A + B \ln C_e \dots\dots\dots 1.10$$

*Dubinin–Radushkevich model*

The Dubinin-Radushkevich (D-R) equation has been effectively used to describe adsorption by microporous solid (Nemr *et al.*, 2009), to understand the adsorption type and to distinguish between the physical and chemical adsorption.

The linearized Dubinin-Radushkevich equation is expressed by equation 1.11:

$$\ln q_e = \ln q_m - \beta \epsilon^2 \dots\dots\dots 1.11$$

## Adsorption Kinetics Studies

### *Pseudo First Order Model*

According to Ho (2006), pseudo-first order model is based on solid capacity. The linear form of first order model is expressed by equation 1.12;

$$\ln(q_e - q_t) = \ln q_t - k_1 t \quad \dots\dots\dots 1.12$$

### *Pseudo second order model*

The linear form of Pseudo-second order model is expressed by equation 1.13:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad \dots\dots\dots 1.13$$

## Results and Discussion

The results of the studies showed that adsorption factors (agitation, contact time, adsorbent dosage and initial trichloro-phenol concentrations) has a significant effect on the adsorption capacity of FPBAC. Generally, adsorption capacity increases with increase agitation, contact time, adsorbent dosage and initial concentration of the adsorbate. According to Alam *et al.* (2007) an increase in agitation with contact time would enhance mass transfer of the adsorbate to the surface of the adsorbents. The maximum adsorption capacity of 37.64 mg/g was obtained at agitation of 200 rpm, contact time of 90 min, 0.20 g of adsorbent dosage and 150 mg/L of initial concentration of the adsorbate while the minimum adsorption capacity of 6.80 mg/g was obtained at agitation of 200 rpm, contact time of 90 min, 0.10 g of adsorbent dosage and 50 mg/L of initial concentration of the adsorbate.

The maximum adsorption capacity of 37.64 mg/g obtained for FPBAC investigated in this study compared adequately well with 40 mg/g obtained for microporous ZnCl<sub>2</sub> activated coir pith carbon in the work of Subha and Namasivayam (2008) and well above 22.2 mg/g obtained for activated carbon derived from oil palm empty fruit bunches (Alam *et al.*, 2007).

### Effects of adsorption parameters

#### *Effects of Agitation*

It was observed that increase in the agitation rate from 100 – 250 rpm increases the percentage removal of TCP from 91.36 – 95.18 % (Fig.1a). This is because increase in agitation rate will enhance the mass transfer rate of trichloro-phenol to the surface of activated carbon. Similar result of 92 – 96 % was reported in the adsorption of phenol using activated carbon from treated coconut stalk by Girish and Swaroop (2014).

#### *Effects of Contact time*

It can be seen from Figure 1b that the rate of TCP adsorption was higher at the beginning of the process but later remained constant when equilibrium was reached at 120 min. This may be due to availability of large surface sites of the activated carbon at the beginning of the process in contrast to the equilibrium time when surface sites of the adsorbents has been exhausted.

#### *Effects of Adsorbent dosage*

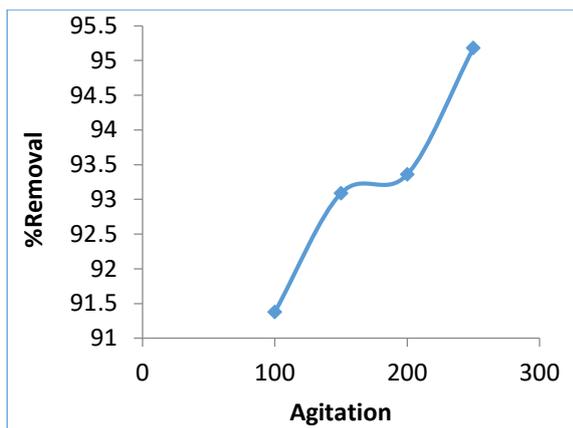
It was shown in Figure 1c that TCP removal increased from 92.64 – 93.64 % on increasing the adsorbent dosage from 0.1 – 0.3 g. This is due to the enhancement of the sorption surface and increase in the number of the adsorption sites. This result is similar to 90 – 92 % removal reported in the adsorption of phenol using treated coconut stalk by Girish and Swaroop (2014).

#### *Effects of Initial concentration of 2,4,6-trichlorophenol (TCP)*

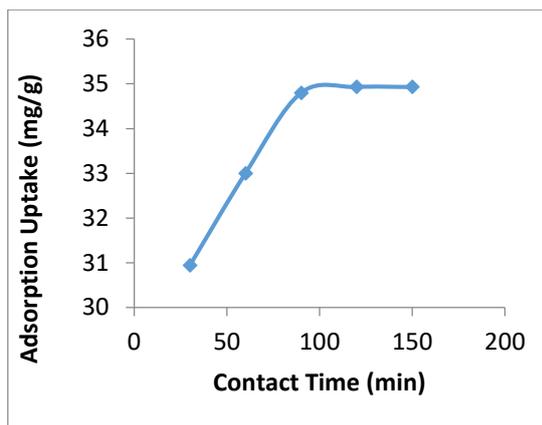
It is shown in Figure 1d that as the initial concentration of TCP increases from 50 – 250 mg/g, the percentage removal increase from 80.9 – 97.97 % due to strong attraction between the activated carbon and TCP. Similar results of 95 – 97 % were reported in the adsorption of TCP using ZnCl<sub>2</sub> activated coir pith carbon by Subha and Namasivayam (2008).

#### *Effects of Temperature*

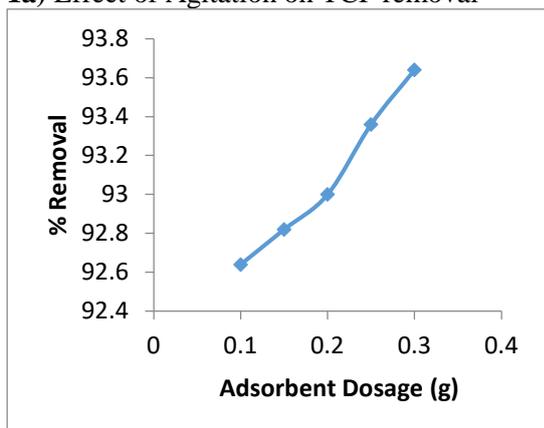
It can be seen from Figure 1e that as the temperature increases from 30 - 60 °C, adsorption of TCP increases from 34.93 – 35.33 mg/g which suggested that temperature is a significant factor in the adsorption process. The process was observed to be exothermic in nature. In the work of Subha and Namasivayam (2008), similar results of 35 mg/g was reported in the adsorption of TCP using ZnCl<sub>2</sub> activated coir pith carbon.



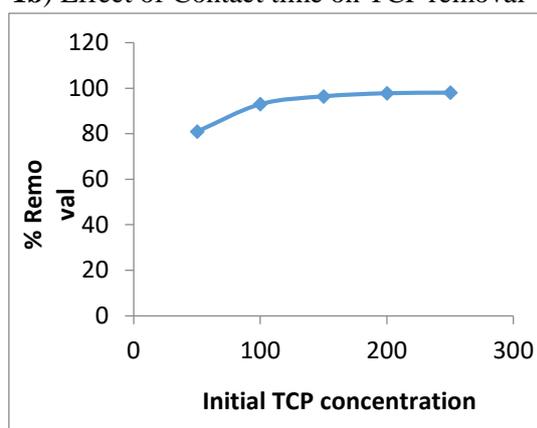
**1a)** Effect of Agitation on TCP removal



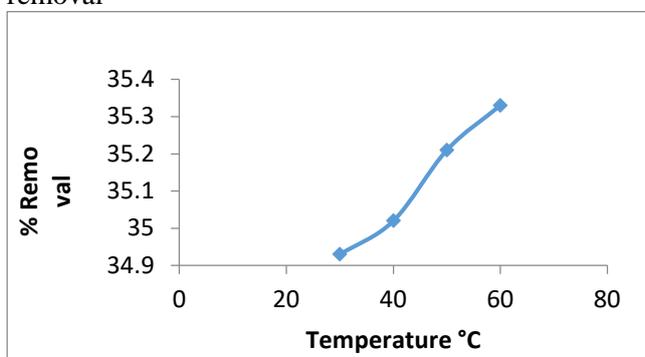
**1b)** Effect of Contact time on TCP removal



**1c)** Effect of Adsorbent Dosage on TCP removal



**1d)** Effect of Initial TCP conc. on TCP removal



**1e)** Effect of Temperature on TCP removal

**Figure 1(a – e).** Effect of Adsorption factors on TCP removal

### Adsorption Isotherm studies

#### *Freundlich Isotherm*

From Figure 2a, Freundlich constants  $k_f$  of 16.91 was obtained from the intercept,  $n$  value of 0.54 was obtained from the slope and correlation coefficient  $R^2$  of 0.912 was obtained from the plot.  $k_f$  and  $n$  is the indicator of adsorption capacity and adsorption intensity respectively.

#### *Temkin Isotherm*

From Figure 2b, Temkin constant  $A$  of 2.93 was obtained from the intercept, Temkin constant  $B$  of 90.26 was obtained from the slope, heat of adsorption  $b$  of 0.03 and correlation coefficient  $R^2$  of 0.804 was obtained from the plot.

#### *Langmuir Isotherm*

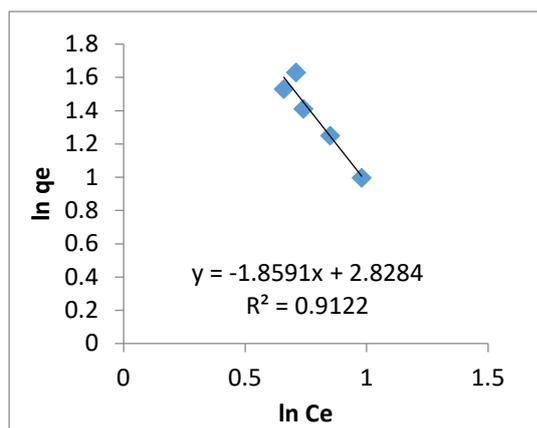
From Figure 2c, maximum adsorption capacity ( $q_m$ ) at monolayer coverage of 5.62 mg/g was obtained from the slope. The constant  $k_L$  value of 0.23 L/mg related to free energy of adsorption was obtained from the intercept giving correlation coefficient  $R^2$  of 0.919.

*Dubinin–Radushkevich model*

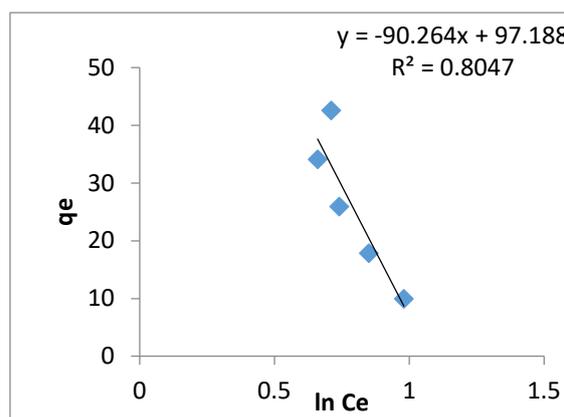
From Figure 2d, free energy of sorption per mole of 2,4,6-trichlorophenol,  $\beta$  of 3.048 KJ<sup>2</sup>/mol<sup>2</sup> was obtained from the slope, maximum adsorption capacity  $q_m$  of 2.40 mg/g was obtained from the intercept and correlation coefficient  $R^2$  of 0.846 was obtained from the plot.

**Table 1.** Isotherm constant and Correlation Coefficients for adsorption of TCP onto FPBAC

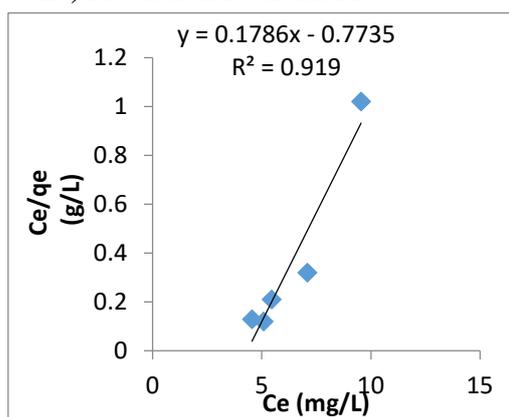
Models				
<b>Langmuir</b>	$q_m$ (mg/g)	$K_L$ (L/mg)	$R^2$	
	5.62	0.23	0.919	
<b>Freundlich</b>	$n$	$K_f$	$1/n$	$R^2$
	0.54	16.91	1.859	0.912
<b>Temkin</b>	<b>A</b>	<b>B</b>	<b>b</b>	$R^2$
	2.93	90.26	0.03	0.804
<b>Dubinin-Radushkevich</b>	<b>B (mol<sup>2</sup>/KJ<sup>2</sup>)</b>	$q_0$ (mg/g)	<b>E (KJ/mol)</b>	$R^2$
	3.05	2.40	0.41	0.85



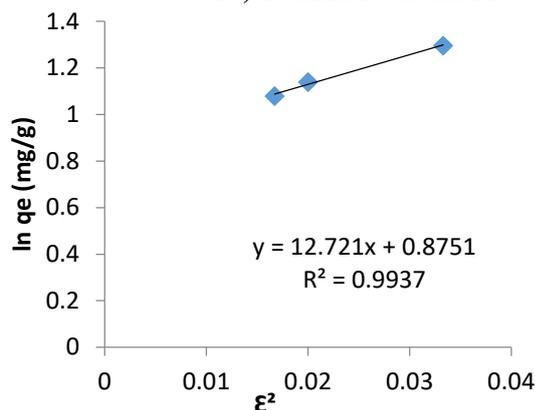
**2a)** Freundlich Isotherm Plot



**2b)** Temkin Isotherm Plot



**2c)** Langmuir Isotherm Plot



**2d)** Dubinin–Radushkevich plot

**Figure 2.** Isotherm plots for adsorption of TCP onto FPBAC

From the results of correlation coefficients showed on Table 1, Langmuir isotherm model with the highest value of correlation coefficient ( $R^2$ ) of 0.919 was best fitted and suitable for the adsorption of 2,4,6-trichlorophenol onto flamboyant pods bark activated carbon (FPBAC).

**Adsorption Kinetics Studies**

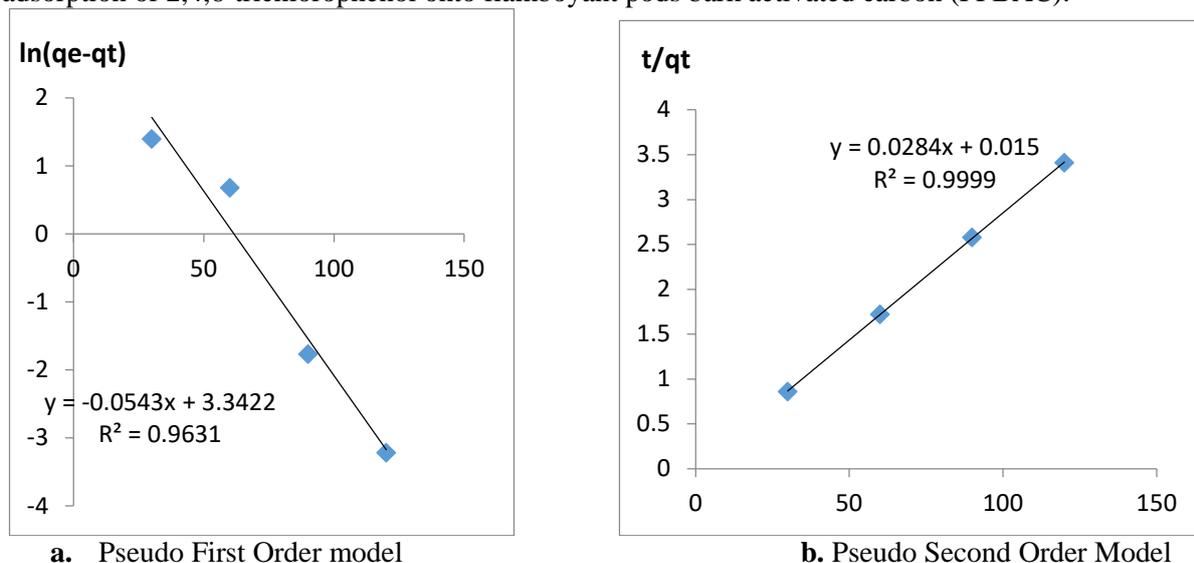
*Pseudo first order model*

From Figure 3a, pseudo first order rate constant  $k_1$  of 0.054 (1/min) was obtained from the slope, adsorption capacity at time  $t$  ( $q_t$ ) of 28.28 mg/g was obtained from the intercept and correlation coefficient  $R^2$  of 0.963 was obtained from the plot. The value of correlation coefficient obtained falls within the range obtained for the removal of TCP from water and petroleum refinery industry effluents by surfactant-modified bentonite (Anirudhan and Ramachandran, 2014).

*Pseudo second order model*

From Figure 3b, adsorption capacity value of 35.71 mg/g which was obtained from the slope, pseudo second rate constant  $K_2$  of 0.052  $\text{gmg}^{-1} \text{min}^{-1}$  obtained from the intercept and correlation coefficient  $R^2$  of 0.999 was obtained from the plot.

The model with the highest correlation coefficient value is considered to best describe the process. Therefore, pseudo second order kinetic model with ( $R^2$ ) of 0.999 was best kinetic model for the adsorption of 2,4,6-trichlorophenol onto flamboyant pods bark activated carbon (FPBAC).



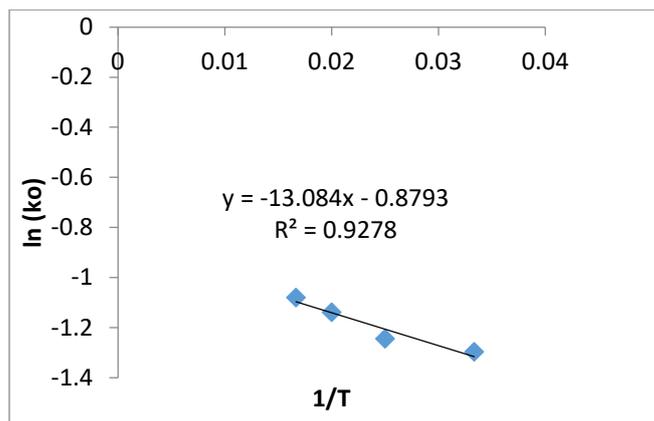
**Figure 3.** Plots of Kinetic Models for Adsorption of TCP onto FPBAC

**Table 2.** Comparison of values for pseudo first and pseudo second order kinetic models

	$q_e$ (mg/g)	$K$ (1/min)	$R^2$
Pseudo 1 <sup>st</sup> Order	28.28	$K_1=0.054$	0.963
Pseudo 2 <sup>nd</sup> Order	35.71	$K_2=0.052$	0.999

**Adsorption Thermodynamics studies**

From Figure 3, the change in enthalpy ( $\Delta H$ ) of -108.75 J/mol was obtained from the slope, the change in entropy ( $\Delta S$ ) of -7.31 J/molK obtained from the intercept and correlation coefficient ( $R^2$ ) of 0.927 was obtained from the Vont Hull plot. It is evident from Table 3 that  $\Delta G$  is positive in the whole range of temperatures while the value of  $\Delta H$  and  $\Delta S$  are negative. The positive values of  $\Delta G$  indicates the presence of an energy barrier in the adsorption process while the negative value of enthalpy ( $\Delta H$ ) indicates the exothermic nature of the process. The increase of adsorption capacity of FPBAC with increase in temperature can be explained from the exothermic nature of the process. The negative value of  $\Delta S$  implies that there was decrease in randomness at solid/solution interface during adsorption of 2,4,6-trichlorophenol onto flamboyant pods bark activated carbon (FPBAC).



**Figure 4:** Vont Hull plot Adsorption Thermodynamic

**Table 3:** Thermodynamic parameters for TCP adsorption onto FPBAC at different temperatures

Thermodynamic parameters	Value
$\Delta H$ (J/mol)	-108.75
$\Delta S$ (J/molK)	-7.31
$\Delta G$ (J/mol), 303° K	2106.18
$\Delta G$ (J/mol), 313° K	2179.28
$\Delta G$ (J/mol), 323° K	2252.38
$\Delta G$ (J/mol), 333° K	2325.48

### Conclusion

The study showed that activated carbon developed from flamboyant (*Delonix regia*) pods bark (FPBAC) served as an effective adsorbent for the removal of 2,4,6-trichlorophenol from aqueous solution. The adsorption factors (agitation, contact time, adsorbent dosage and initial concentration) has a significant effect on the adsorption capacity. It was observed that adsorption capacity increases with increase agitation, contact time, adsorbent dosage and initial trichloro-phenol concentration. The maximum adsorption capacity of 37.64 mg/g was obtained at agitation of 200 rpm, contact time of 90 min, 0.20 g of adsorbent dosage and 150 mg/L of initial concentration of 2,4,6-trichlorophenol (TCP). The results of thermodynamic studies showed that the adsorption process is an exothermic one whose isotherm was best described by Langmuir model while its kinetics conforms to pseudo-second order kinetic model.

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