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## Adsorptive removal of Phenol by using Fly ash and Guava leaves

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### ABSTRACT

This work concerns the studies on batch adsorption of phenol from waste water using adsorbents such as fly ash; guava leaves adsorbent developed from local available tree leaves to assess their suitability to remediate phenol contaminated water.

Factors affecting the adsorption characteristics such as contact time, adsorbent dosage and pH on phenol efficiency were studied and optimum values for maximum uptake were found. It was observed that the percentage removal of phenol increases with increases in adsorbent dosage and reaches an equilibrium value. Also with increase in agitation time, the removal percentage was increased.

Maximum Phenol removal percentage observed at contact time 90min, adsorbent dosage of 20g/l and pH of 7. All the experiments were performed at a temperature of  $34 \pm 1^{\circ}\text{C}$  and at a stirring rate of 120rpm. Among the two adsorbents used, the maximum removal was observed for the Fly Ash. Adsorbents Isotherms (Langmuir and Freundlich) and adsorption kinetics were studied for Fly Ash and Guava leaves.

Keywords: Adsorption, Contact time, Adsorbent Dosage, Phenol, kinetics

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## 1.INTRODUCTION

Phenol is the major pollutant present in effluent from various industries like oil refineries, coke oven v plant, iron and steel industries, petrochemical industries, plastic industries, phenol resin manufacturing unit etc. minute concentration of phenol can cause objectionable taste in potable water and also may taint the flesh of fish. Larger concentration not only kills fish, but also completely destroys all aquatic lives in the stream. Phenol produces inhibitory effects on micro organisms in waste water treatment plant. It is necessary to remove or to reduce the phenol concentration to a safe level. The conventional waste water treatment is meant for removing biodegradable organics but these are ineffective in removing the refractory organics. Various treatment methods used for removal of recalcitrant waste adsorption, Ion Exchange, reverse osmosis, chemical oxidation, precipitation, Distillation, gas stripping, solvent extraction etc among these, is a proven and reliable physicochemical pollution control technology. Extensive studies have shown that the adsorption on fly ash is one of the effective means for removing even trace quantity of numerous bio-resistant organic pollutants from aqueous system.

Fly ash is finely divided mineral residue resulting from powdered coal in power plant. The activated carbon adsorption is highly expensive .Fly ash is cheap and locally available adsorbent and it is found in abundance in India. Fly ash is one such material and can be an alternative to active carbon. Various researchers were conducted on phenol removal utilizing fly ash.

The prime objective of the present work investigation were to conduct the batch sorption tests which include determination of sorption kinetics and isothermal adsorption capacity of the adsorbent and to assess the effective of the PH on the removal of phenol from synthetic waste water. Effect of various parameters like contact time, pH, dose of adsorbent was studied. A

comparison of the adsorption efficiency of these adsorbents materials was done to determine which materials are more suitable for removal of phenol from waste water. The data obtained was fitted to Langmuir, Freundlich and Langergren Isotherm.

## Method of Phenol Removal

- Extraction of solvent
- Chemical precipitation
- Enzymatic separation
- Membrane systems
- Adsorption Process

Chemical Precipitation and reduction process needs another separation technique for the treatment and disposal of high quantities of waste metal residual sludge produced. These techniques use a lot of treated chemicals and the residual phenol. Concentration required in the treated waste water was not achieved because of the precipitation. The application of the membrane systems for the waste water treatment has major problem like membrane scaling, fouling and blocking. Enzymatic separation is high cost and extraction method requires large amount of solvent.

### 1.1.Adsorption isotherms

Overall a small concentration range and particularly for dilute solution, the adsorption isotherms can frequently be described by an empirical expression usually attributed to Freundlich

$$C^* = k [ v ( C_o - C^* ) ]^n \dots\dots\dots(1)$$

Where  $v ( C_o - C^* )$  is the apparent adsorption per unit mass of adsorption and  $k$  and  $n$  constants. Freundlich equation is useful in cases where the actual identity of the solute is not known. In such cases, the concentration of solute can be measured by means of calorimeter or spectrometer.

## 1.2.Adsorption Kinetics

The kinetics of fluoride sorption by fly ash is modeled using the first order rate equation of Lagergren.

$$\log q_e - q_t = \log q_e - k_1 t / 2.3$$

Where  $k_1$  is the Lagergren rate constant of sorption ( $\text{min}^{-1}$ )  $q_e$  and  $q_t$  is the amount of material absorbed (mg/g)

### OBJECTIVES

- To remove phenol from waste water by using adsorbents Fly ash and Guava leaves
- To prepare the natural adsorbents from guava leaves
- To study the effect of various parameters like pH, contact time and adsorbent dosage on adsorption efficiency of both the adsorbents.
- To fit the data by using Langmuir, Freundlich, Lagergren isotherms.

Somnath mukherjee, sunil kumar, Amal k. mishra the adsorption of phenol by carbonious material and presents the results of phenol removal from waste water by different carbonious material as. They conclude that activated carbon showed maximum phenol removal efficiencies approximately 98% followed by, bagasse ash and wood char coal<sup>1</sup>. Mahvi, A.H maleki, A.studied the adsorption of phenol by material and present results of phenol removal from aqueous solution by different adsorbents it conclude that ash is efficient followed by rice husk<sup>2</sup>. Mahadeva M. Swamy et al., in this work it deals with the adsorption of phenol on carbon rich bagasse fly ash and activated carbon. The values of isosteric heat of adsorption varied when the phenol was placed on the surface<sup>3</sup>. Ahmaruzzaman M, Sharma DK., Residual coal is treated with phosphoric acid in removal of phenol from wastewater. Kinetic modeling was done by using Lagergren first order rate expression in the removal of phenol<sup>4</sup>.

T. Viraraghavan et al., in this work adsorbents used for analysis were of less cost. The adsorbents found to be adsorbed 46.1%, 41.6% and 42.5%. The entire process was done in batch process. The desirable value of pH found in between 4.0-5.0 in which adsorption of phenol took place with different adsorbents<sup>5</sup>. Konduru R. Ramakrishna et al., The current paper deals with an investigation on four low-cost adsorbents locally available in Saskatchewan, Canada for dye removal. Peat. Bentonite clay and fly ash were utilized for this study and their performance evaluated against that of granular activated carbon. Synthetic dye wastewaters prepared from commercial grade acid, basic and disperse dyes were used in this study and the results showed high removals of acid dyes by fly ash and slag while peat and bentonite exhibited high basic dye removals. The adsorbents which are both effective as well as economical, for colour removal from wastewaters<sup>6</sup>. M.Kermani et al., removal of phenol from aqueous solutions by rice husk Ash and activated carbon. In this rice husk was prepared at three different temperatures. The rice husk ash showed as an efficient adsorbent for the removal of phenol<sup>7</sup>. Sushmita Mishra et al., Potential of leaf litter for phenol adsorption- kinetic study, in this case plant material; shorea robusta was used as adsorbent for the removal of phenol. Equilibrium data were correlated by Freundlich adsorption isotherm. Maximum adsorption was found to be 0.06 mg/g<sup>8</sup>.

### **1.3. Experimental Procedure:**

#### **1.3.1. Calibration chart**

The stock solution of adsorbent was diluted to 100, 200....500 mg/l. the absorbance of phenol was analyzed spectrophotometrically and the calibrated.

### **1.3.2. Adsorption of phenol by using treated Fly ash:**

#### **1.3.2.a. Adsorbent preparation**

Fly ash was washed repeatedly with distilled water to remove dust and soluble impurities. Initially fly ash was kept for drying at room temperature in a shade for 1 day. Then they were sieved using 150-200 mesh. Undersize particles were collected.

#### **1.3.2.b. Preparation of Adsorbate solution**

Stock solution of phenol was prepared by dissolving of 1g of phenol in 1000ml. The solution was diluted as required to obtain standard solutions containing 1000mg/l of phenol.

#### **1.3.2.c. Batch adsorption Experiments**

Batch adsorption experiments were conducted by agitating the standard flasks (250 ml) at room temperature in a mechanical shaker. The effect of time and pH was studied with a phenol concentration of 1000mg/l and an adsorbent dosage of 20g/l. The aqueous solution pH was adjusted in the range of 1-14 by using dilute HCl and NaOH solutions. Experiments were carried out by varying the adsorbents amount by 20g and 100g with a phenol concentration of 1000mg/l. phenol concentrations were analyzed spectrophotometrically. The removal percentage of phenol from synthetic reagent was studied using Fly ash as adsorbent at different parameters like agitation time, adsorbent dosage and pH of the solution.

#### **1.3.2.d. Effect of Agitation time**

The effect of agitation time on phenol removal was observed at phenol concentration of 1000mg/l and adsorbent dosage of 20g. the adsorbent dosage of 1g was added to the 10 different standard flasks containing 50ml of adsorbent solution concentration 1000mg/l. flasks numbered 1, 2, 3, ....., were placed in mechanical shaker at 120rpm for 30min, 60min,....150min

respectively. Final phenol concentration in the absorbate solution was found spectrophotometrically at 500nm and percentage removal of phenol was calculated.

#### **1.3.2.e. Effect of Adsorbent dosage**

The effect of adsorbent dosage on phenol removal was observed at phenol concentration of 1000mg/l at agitation time for 90 minutes. The prepared absorbate solution was diluted to 1000mg/l. Adsorbent solution was transferred into 100ml standard flasks. The adsorbent dosage of 1g, 2g, 3g, 4g, 5g were added to the flasks numbered 1, 2, 3....respectively. Then the flasks were placed in mechanical shaker at room temperature. The speed of the shaker is maintained at 120 rpm and for a contact time of 90 minutes. Then the flask was taken out and liquid in the flasks were filtered by using Whatman filter paper. The filtered solution was analyzed in visible spectrophotometer at a wavelength of 500nm. The percentage removal of phenol was found using standard calibration chart.

#### **1.3.2.f. Effect of pH**

The effect of pH on phenol removal is observed at phenol concentration of 1000mg/l and at agitation time of 90 minutes. The prepared absorbates solution was diluted to 1000mg/l and pH of the solution was found using Ph meter. Then the pH of the solution was made to 2, 4, 7, 9 and 14 by using NaOH and HCl of appropriate concentrations and solution of different pH were taken in different flasks. Then the adsorbent made of 1g was added to the above solutions. These standard flasks were placed in mechanical shaker for 90 minutes. The speed of the shaker was maintained at 120rpm. after the completion of above mentioned agitation time, flasks were taken out and solutions were filtered using Whatman Filter paper. The filtered solution was analyzed using

visible spectrophotometer at a wavelength of 500nm. Similarly the percentage removal of phenol was found using standard calibration.

## **2 .Adosorption of phenol by using treated Guava leaves:**

### **2.1. Adsorbent preparation**

All chemicals used were of analytical reagent grade. Guava leaves were washed repeatedly with diluted water to remove dust and soluble impurities. Initially leaves were kept for drying at room temperature in a shade for 1 day and then in air oven at 80°C till they turn pale yellow. Then they were washed with diluted water to remove free acids and dried at room temperature.

## **2. RESULTS AND DISCUSSION**

Calibration chart shown in figure 4.1 was used as a reference for calculating the phenol concentration. All the experiments were carried out at room temperature  $32 \pm 1^\circ\text{C}$  and speed of the shaker was maintained at 120 rpm and adsorbate solution of concentration 1000rpm.

### **3.1. Effect of Agitation time**

The effect of agitation time on the adsorption of phenol using Fly ash was studied at adsorbent dosage of 20 g/l. The results were tabulated in table 4.1 and the graph is plotted in figure 4.2. from the graph it is observed that percentage removal of phenol increases with increase in agitation time. it is observed that at 90 min the percentage removal is about 70.2%.

### **3.2. Effect of Adsorbent dosage**

The effect of adsorbent dosage on the percentage removal of phenol was studied at 120 rpm and agitation time of 90 minutes using fly ash and the values are tabulated in table 5.2 and the graph is plotted in figure 4.3. it was observed that as the adsorbent dosage increases the percentage

removal increases and after certain dosage it remained almost constant as it reaches equilibrium value. If the adsorbent dosage is 20g/l then the percentage removal was about 70.2%.

### **3.3. Effect of pH**

Effect of solution pH on removal of phenol was studied using fly ash as adsorbent at adsorbent dosage of 20g/l and agitation time 90minutes. The values are tabulated in table 4.3. pH of the solution was adjusted by using HCl and NaOH solution. The effect of pH on the process was presented in figure 4.4. The percentage adsorption of phenol increases with increasing pH. The maximum adsorption taking place in the pH range 1-14, but the pH range is maximum at 7.

### **3.4. Adsorbent: Treated guava leaves**

All the experiments were carried out at room temperature  $32 \pm 1^\circ\text{C}$  and speed of the shaker was maintained at 120 rpm and adsorbate solution of concentration 1000rpm.

#### **3.4.1. Effect of agitation Time**

The effect of agitation time on the adsorption of phenol using Guava leaves was studied at adsorbent dosage of 20g/l. The results are tabulated in table 4.4 and the graph is shown in figure 4.5. It was observed that percentage removal of phenol increases with the increase in agitation time. It was observed that at 90 minutes the percentage removal was about 67.0636%.

#### **3.4.2. Effect of adsorbent dosage**

The effect of adsorbent dosage on the percentage removal of phenol was studied at 120rpm and agitation time of 90 minutes using Fly ash and the values are tabulated in table 4.5 and are plotted in figure 4.6. From the figure 4.6 it was observed that as the adsorbent dosage increases the percentage removal increases and after certain dosage it remained almost constant as it

reaches equilibrium value. If the adsorbent dosage was 20g/l then the percentage removal was about 67.036%.

### 3.4.3. Effect of pH

Effect of solution pH on removal of phenol was studied using Guava leaves as adsorbent at adsorbent dosage of 20g/l and agitation time of 90minutes. The values were tabulated in table 4.6. pH of the solution was adjusted by using HCl and NaOH solutions. The effect of pH on the process was plotted in figure 4.7, the percentage adsorption of phenol increases with increasing pH. The maximum adsorption taking place in the pH range 1-14, but the pH range was maximum at 7.

### 3.5. Adsorption Isotherm for Fly ash

Freundlich isotherm was studied using Fly ash as adsorbent using concentration data. The Freundlich equation is expressed linearly as

$$\log q_e = \log k_f + 1/n \log C_e \dots\dots\dots(2)$$

where  $k_f$  and  $n$  are constants. The values of  $q_e$  and  $C_e$  were calculated from the concentration data. A graph was drawn between  $\log q_e$  and  $\log C_e$  in figure 4.8, a best fit straight line was drawn. From the graph shown in figure 4.8,  $k_f = 0.0386$ ,  $n = 0.599$ . From the graph it is observed that Freundlich isotherm won't fit the data.

#### 3.5.1. Langmuir isotherm for fly ash

Equation can be expressed in linear form in terms of concentration as

$$C_e / q_e = 1/q_m b + (1/q_m) (C_e) \dots\dots\dots(3)$$

where  $q_e = (C_o - C_e / m)v$

The value of  $C_e / q_e$  and  $C_e$  were calculated from the experimental values conducted at different initial absorbate concentration and tabulated in table 4.8. A graph was drawn between  $C_e / q_e$  and  $C_e$  and the best fit straight line was drawn as shown in figure 4.9 intercept and the slope of the line was repeated. From the graph 4.9  $q_m = 30.30$  ,  $b = -0.0582$  were observed. The data was reasonably fit using Langmuir Isotherm.

### 3.5.2. Adsorption Kinetics for Fly ash

The kinetics of phenol sorption by fly ash has been modelled using first order rate equation of Lagergren.

$$\log (q_e - q_t) = \log q_e - K_1 t S \dots \dots \dots (4)$$

Where  $K_1$  is the Lagergren rate constant of sorption( $\text{min}^{-1}$ )  $q_e$  and  $q_t$  are the amounts of metal sorbed at equilibrium and at a particular time respectively per gram of adsorbent ( $\text{mg g}^{-1}$ ). From the graph shown in figure 4.10  $q_e = 36.66 \text{ mg/g}$  and  $K_1 = 0.016 \text{ min}$  were observed. As the data points are best fitted as shown in figure 4.10 adsorption of phenol using Fly ash was explained by first order kinetics.

### 3.5.3. Adsorption isotherm for guava leaves

Freundlich isotherm was studied using guava leaves as adsorbent using concentration data.

$$\log q_e = \log k_f + 1/n \log C_e \dots \dots \dots (5)$$

Where  $K_f$  and  $n$  are constants.

The values of  $q_e$  and  $C_e$  were calculated from concentration data. A graph was drawn between  $\log q_e$  vs  $\log C_e$  a best fit straight line was drawn. From the graph shown in figure 4.11,  $k_f = 0.0386$ ,  $n = 0.599$ .

#### 3.5.4. Langmuir Isotherm

Equation can be expressed in linear form in terms of concentrations as

$$C_e/q_e = 1/q_m b + (1/q_m) (C_e) \dots \dots \dots (6)$$

Where  $q_e = (C_o - C_e/m) \times v$

$C_o$  = initial synthetic metal solution

$C_e$  = equilibrium concentration of the adsorbate solution

$m$  = mass of the adsorbent (g)

The value of  $C_e/q_e$  and  $C_e$  were tabulated from the experimental values conducted at different initial adsorbate concentrations and tabulated in table 4.11. From the graph  $q_m = 25.64$ ,  $b = -0.039$  were observed. The data seems to be reasonably fit using Langmuir isotherm.

#### 3.5.5. Adsorption kinetics for guava leaves

The kinetics of phenol sorption by guava leaves has been modified using the first order rate equation of Lagergren.

$$\ln (q_e - q_t) = \ln q_e - k_1 t \dots \dots \dots (7)$$

From the graph shown in figure 4.13  $q_e = 35.1$  mg/g and  $k_1 = 0.016$  min were observed. As the data points are best fitted as shown in figure 4.13 adsorption of phenol using Guava leaves was explained by first order kinetics.

#### 3.6. Comparison of percentage removal of phenol using different adsorbents

The effect of agitation time using four adsorbents was computed in the graph shown in figure 4.14. From the graph shown in figure 4.16 it was observed that Fly ash and Guava leaves are showing the same effect on removing the phenol at different pH levels of adsorbate solution. The

variation in the sorption capacity between the various adsorbents could be related to the nature and concentration of surface groups responsible for interaction with the metal ions.

#### 4. Conclusion:

Fly ash exhibited highest phenol removal efficiency 70.2% at pH of 7, initial phenol concentration of 1000mg/l, and contact time of 90 minutes, adsorbent dosage of 20 g/l and temperature of  $34 \pm 1^\circ\text{C}$ . Guava bio sorbent exhibited highest phenol removal efficiency 65.5% at pH of 7, initial phenol concentration of 1000mg/l, contact time of 90minutes, adsorbent dosage of 20g/l .the optimum sorbent dosage was found to be 20 g/l for both Fly ash and Guava leaves. Enhanced adsorption was obtained at 90 minutes. It could be seen that bio sorbent prepared from treated leaves of Guava are less efficient in removing phenol from waste water as compared with Fly ash. The Langmuir adsorption isotherm was found to be more suitable than Freundlich isotherm. As the adsorbents used in this work are natural, they are economically cheap and ecofriendly.

Agitation Time (min)	% Removal of Phenol
30	27.8536
60	60.79
90	70.2004
120	71.7688
150	73.3372

Table 4.1: Effect of Agitation Time for Fly ash

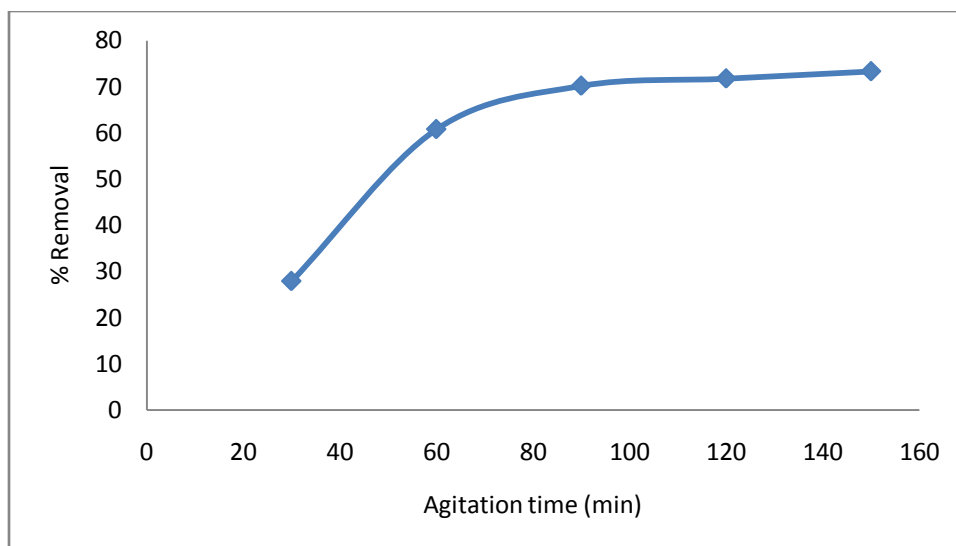


Figure 4.2: Effect of agitation time on percentage removal of phenol at adsorbent dosage of 1g for Fly ash.

Agitation Dosage (g/l)	% Removal of Phenol
20	70.2004
40	73.3372
60	74.9056
80	76.474
100	78.0424

Table 4.2: Effect of Adsorption on Fly Ash Dosage

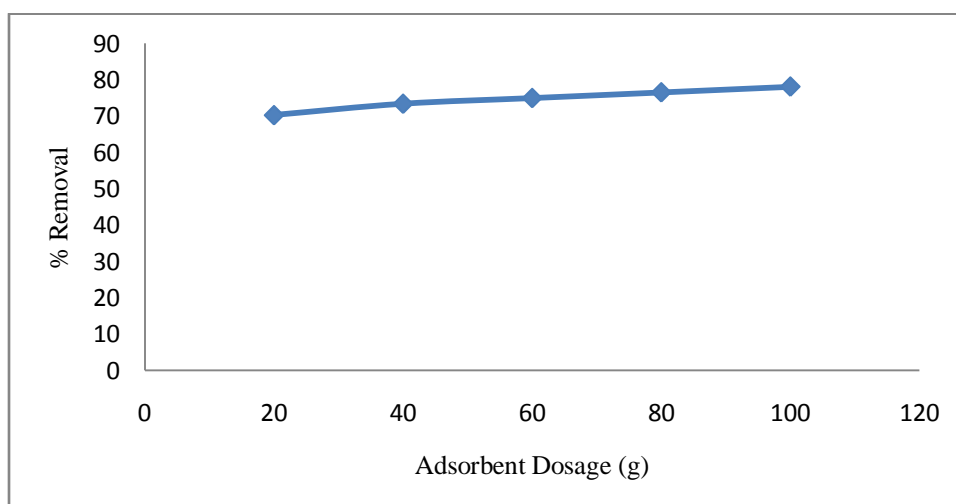


Figure 4.3: Effect of Adsorbent dosage on percentage removal of phenol at 120 rpm agitation time 90min for Fly ash.

pH	% Removal of Phenol
1.09	43.5376
4.25	59.2216
7.04	70.2004
9.05	71.7688
12.05	73.3372

Table4.3: Effect of pH on adsorption efficiency Fly ash

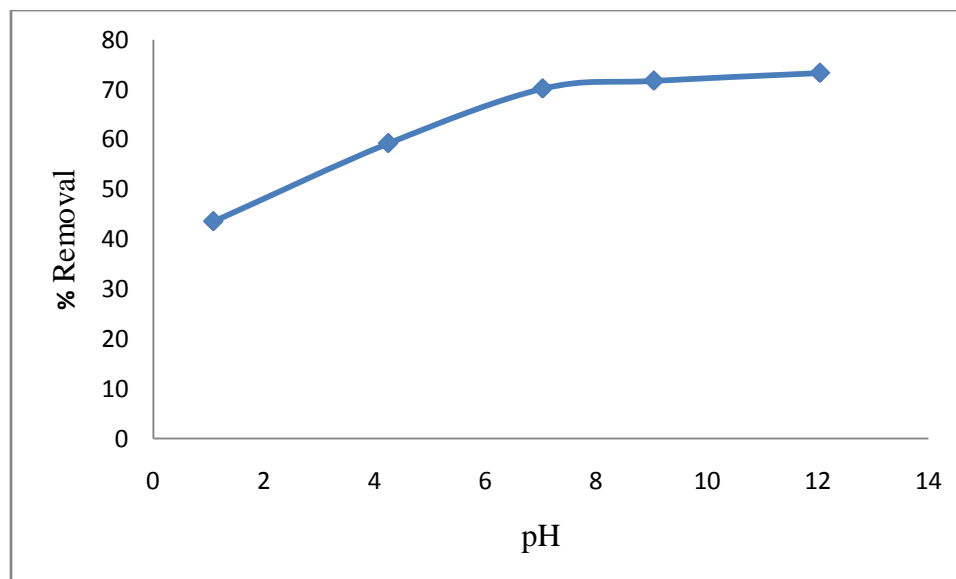


Figure 4.4: Effect of pH on percentage removal of phenol at adsorbent dosage of 20g/l of agitation of 90 min for Fly ash.

Agitation Time (min)	% Removal of Phenol
30	27.8536
60	59.2216
90	67.0636
120	68.632
150	70.2004

Table 4.4: Effect of agitation time of Guava leaves

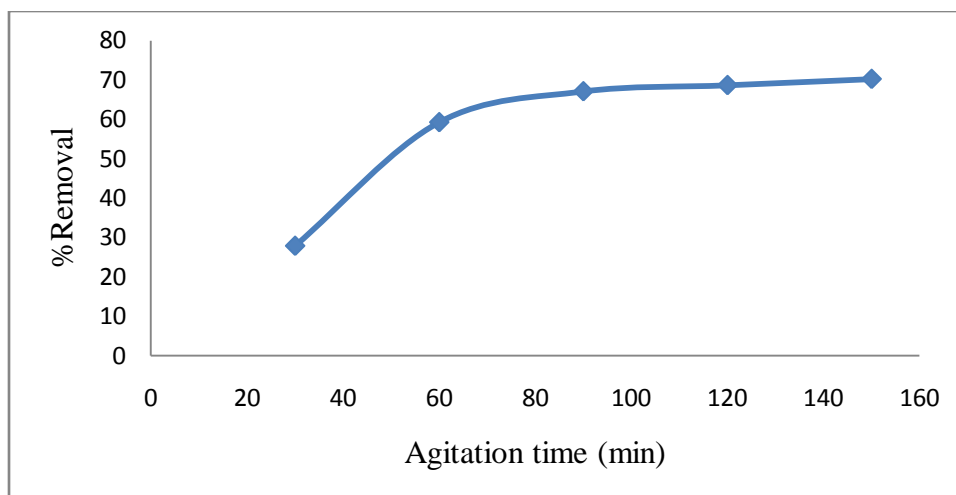


Figure 4.5: Effect of agitation time on percentage removal of phenol at adsorbent dosage of 1g Guava leaves

Adsorbent Dosage(g)	% Removal of Phenol
1	67.0636
2	70.2004
3	71.7688
4	74.9056
5	76.474

Table4.5: Effect of Adorbent Dosage of Guava Leaves

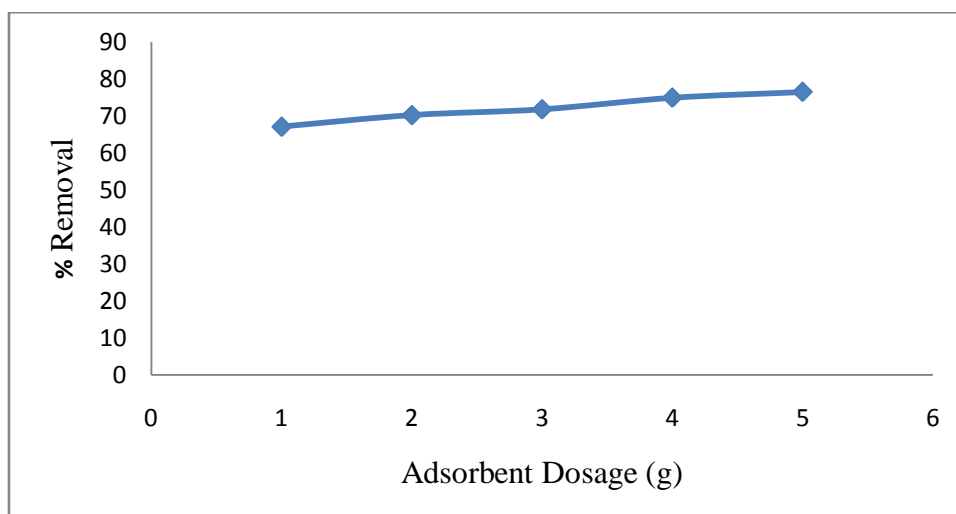
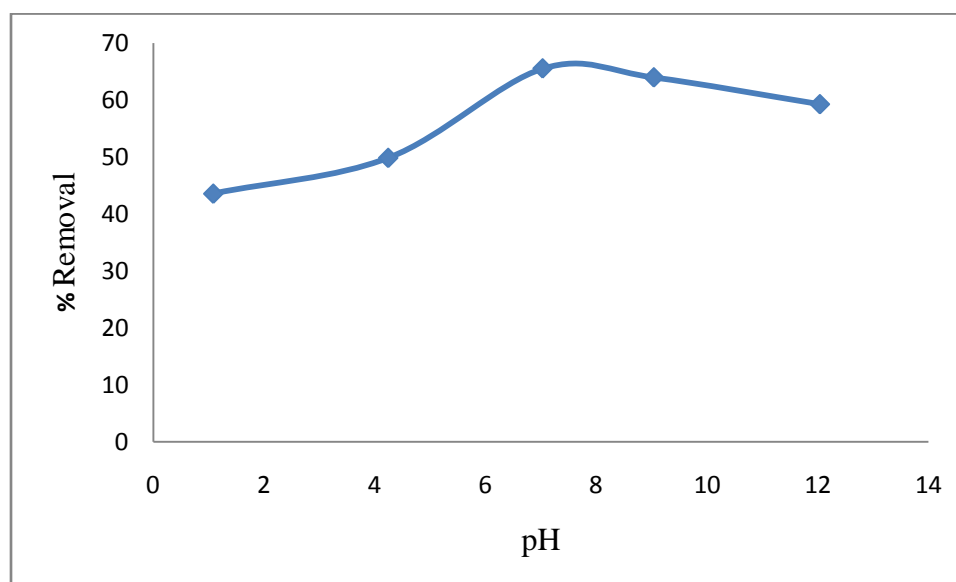


Figure4.6: Effect of Adosorbent dosage on percentage removal of phenol at 120rpm and agitation time 90 min for Guava leaves.

pH	% Removal of Phenol
1.09	43.5376
4.25	49.8112
7.04	65.4952
9.05	63.9268
12.05	59.2216

Table 4.6: Effect of pH on Adsorption efficiency of Guava Leaves



Figur 4.7: Effect of pH on percentage removal of phenol at Adsorbent dosage of 20g/l of agitation of 90min for Guava leaves.

logqe	% Removal of Phenol
2.425	1.5643
2.342	0.3695
2.4742	0.3934

Table4.7: Freundlich isotherm for Fly ash

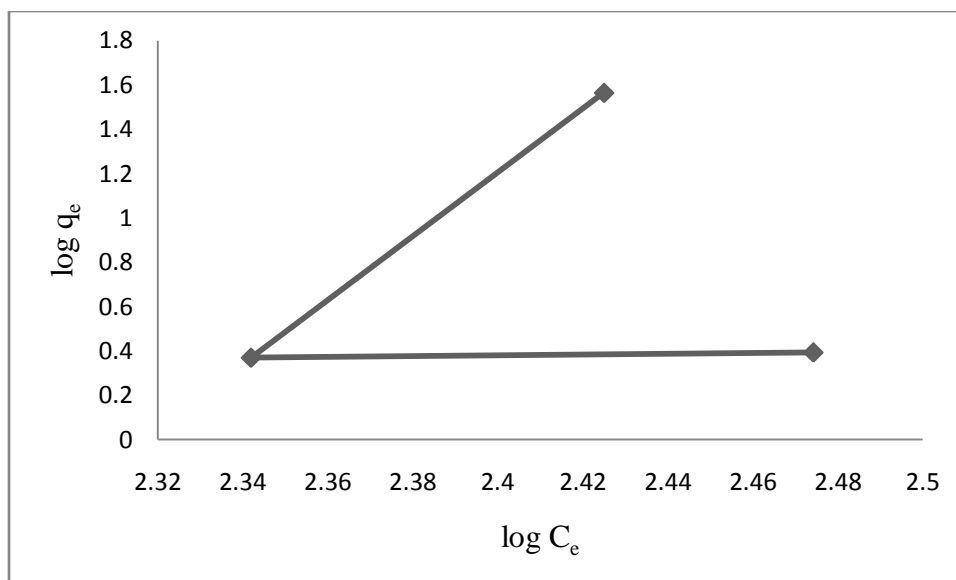


Figure 4.8: Freundlich isotherm for Fly ash

$C_e/q_e$	% Removal of Phenol
7.27	266.63
5.62	219.576
8.255	297.996

Table 4.8: Langmuir isotherm for fly ash

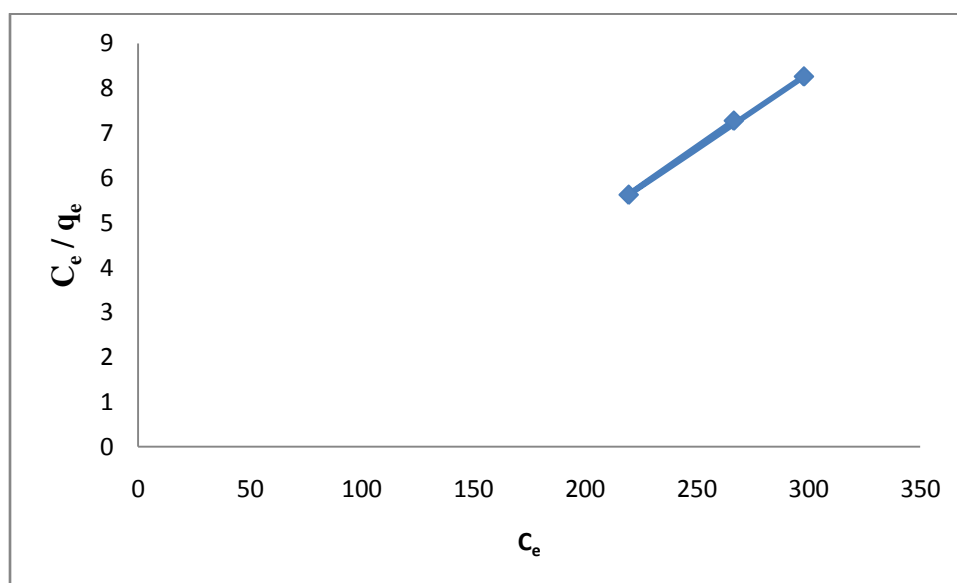


Figure 4.9: Langmuir isotherm for Fly ash

Time ( min)	$\ln ( q_e - q_t )$
30	1.3568
60	0.7975
90	0.19545
120	0.10314

Table 4.9: Adsorption Kinetics of Fly ash

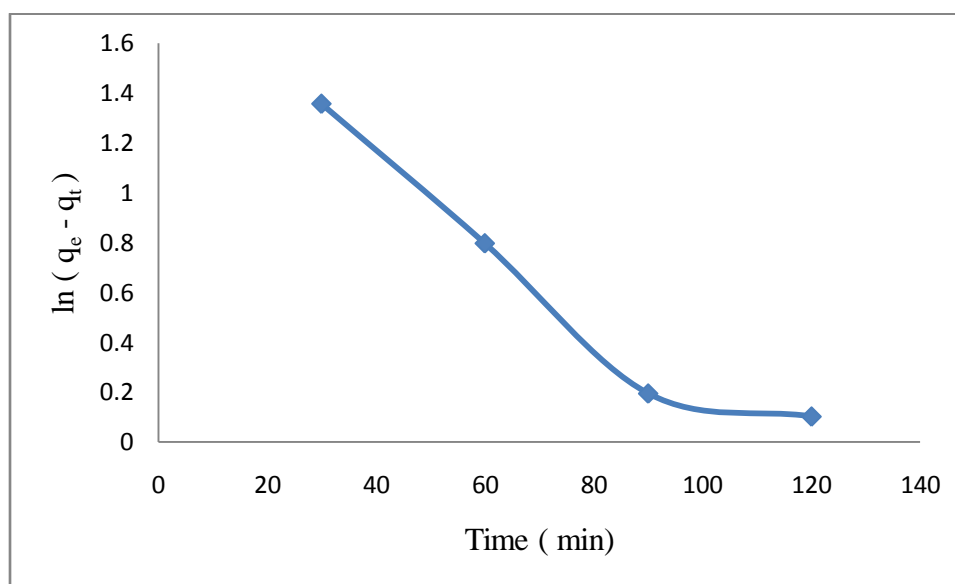


Figure 4.10: Adsorption Kinetics for Fly ash

$\log q_e$	$\log C_e$
2.474	1.545
2.3715	0.8835
2.5378	1.515

Table 4.10: Freundlich isotherm for guava leaves

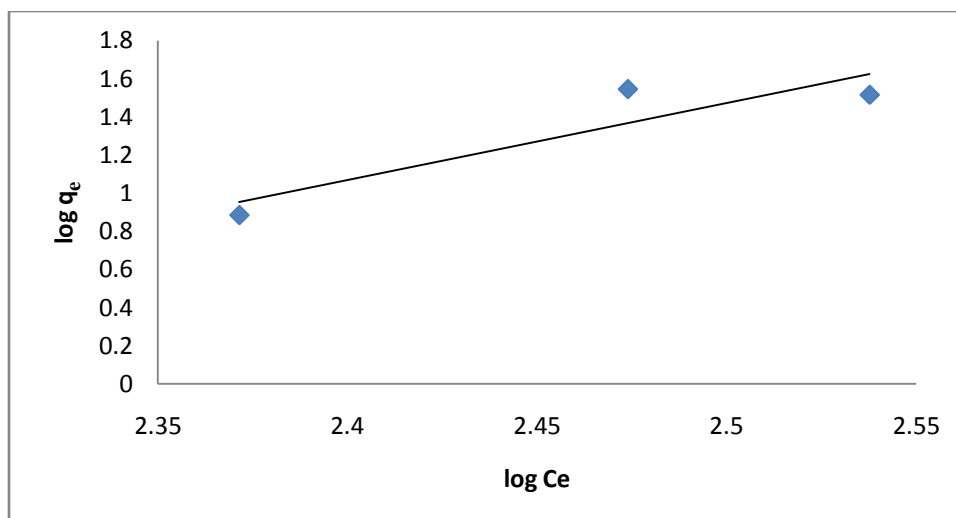


Figure 4.11. Freundlich isotherm for guava leaves

$C_e / q_e$	$C_e$
297.996	8.489
235.26	30.7633
345.048	10.5367

Table 4.11: Langmuir isotherm for Guava Leaves

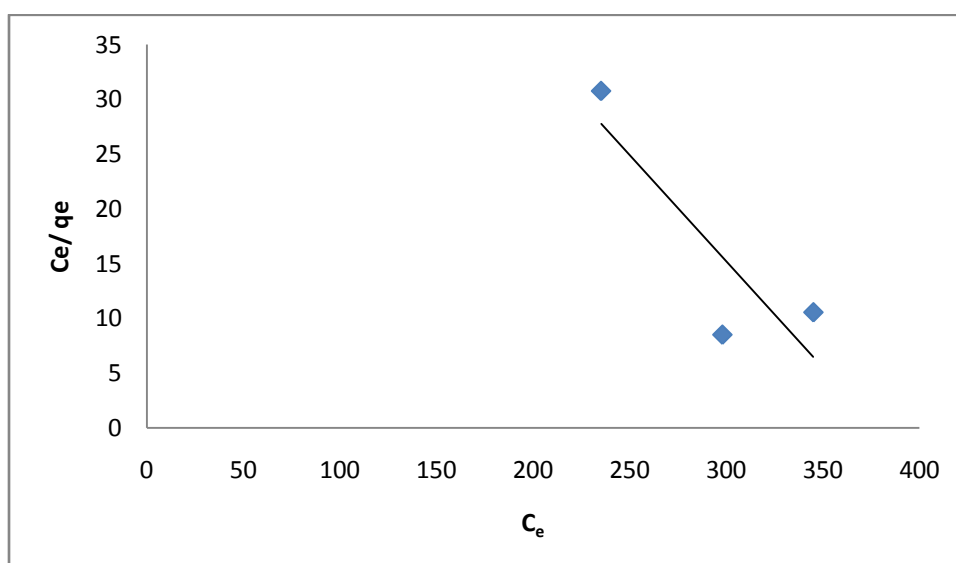


Figure 4.12: Langmuir isotherm for guava leaves

Time ( min)	$\ln (q_e - q_t)$
30	1.3258
60	0.7395
90	0.19545
120	-0.10557

Table 4.12: adsorption kinetics for Guava Leaves

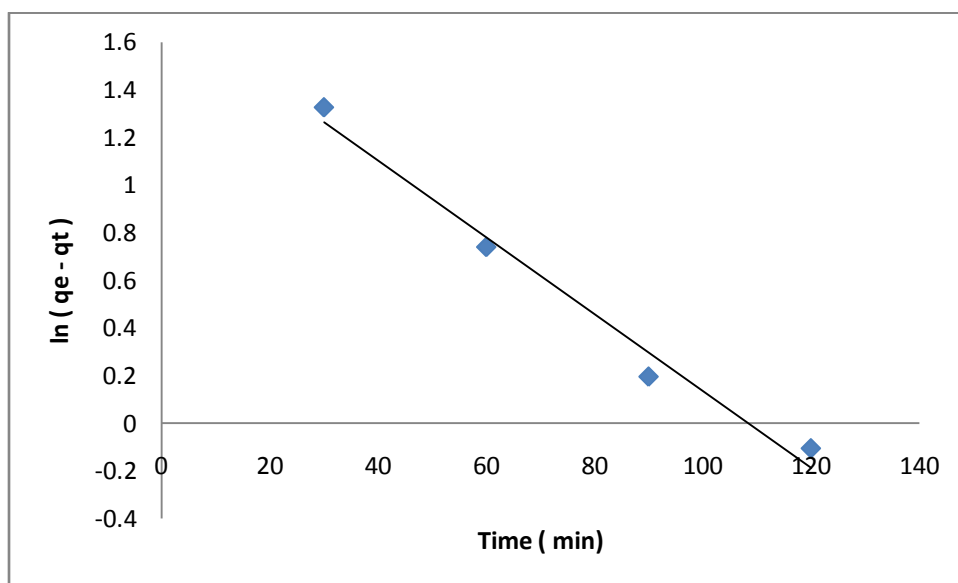


Figure 4.13: Adsorption kinetics for Guava Leaves

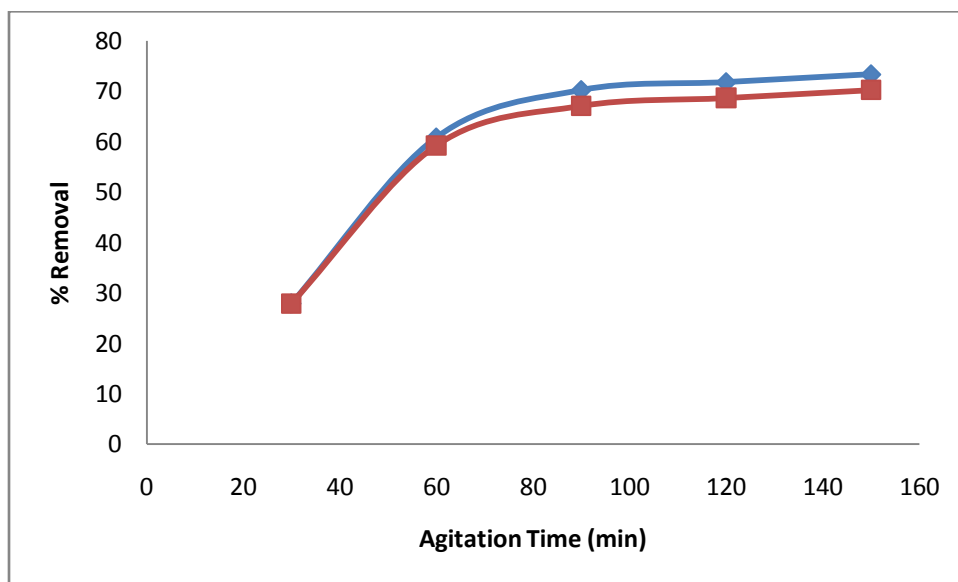


Figure4.14: Effect of agitation time on percentage removal of phenol with two adsorbents

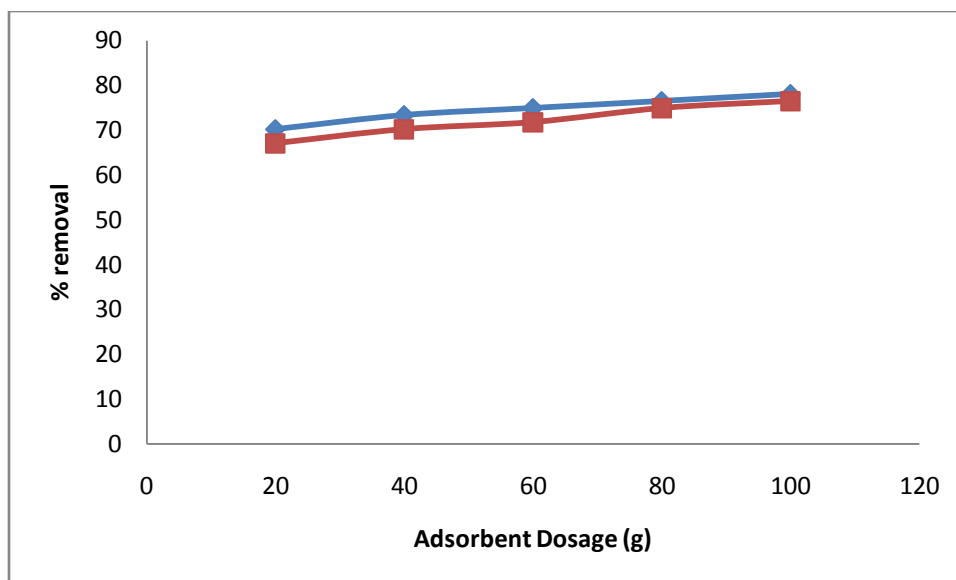


Figure 4.15: Effect of Adsorbent Dosage on percent removal of phenol with two adsorbents

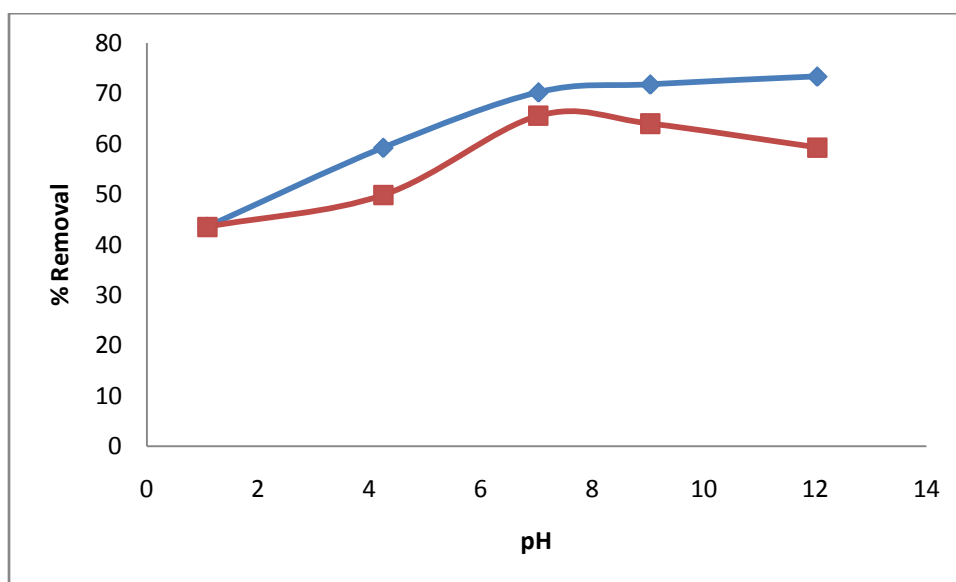


Figure 4.16: Effect of pH of two adsorbents.

## Nomenclature

$^{\circ}\text{C}$  – Degree Centigrade

$C_e$  - The equilibrium adsorbate concentration in solution, mg/l

$C_o$  – Initial concentration of solute, mg/l

K- Rate constant of adsorption, in Langmuir isothermal

k- A constant in Langergren isotherm,  $\text{min}^{-1}$

$k_f$  – Freundlich constants

q – Amount of phenol adsorbed at time t , mg/g

$q_e$  – Amount of phenol adsorbed at equilibrium, mg/l

t – Time, min

m – mass of adsorbent (g)

b – A constant related to energy

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