

Efficient Removal of Phenol and 2,4,6-TCP Trichlorophenol by Okra Leaves; Optimization, Kinetic and Equilibrium Studies

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ABSTRACT

Phenolics are regarded as high priority organic pollutants, their possible negative effects on human health and other organisms even at extremely low levels of concentration compel researchers to work out ways to remove phenolics. The current research explores the nature of phenol and 2, 4, 6- Ttrichlorophenol (2, 4, 6-TCP) elimination on to low cost and economical adsorbent Okra leaves. The effects of various parameters like pH, initial solution concentration, and amount of adsorbent, agitation time on the adsorption of phenol and 2,4,6-TCP were studied using batch adsorption. The results showed that, the percentage adsorption rose with pH and time and went down with the initial solution concentration. At neutral pH, phenol and 2, 4, 6-TCP adsorption reach the equilibrium after 180 and 60 minutes respectively. The kinetic model that was observed to be followed in the adsorption process was the pseudo-second-order kinetic model. The Langmuir, Freundlich and Dubinin-Radushkevich models were used to analyze equilibrium data through non-linear regression equations. The freundlich calculated isotherm adsorption capacity of Phenol and 2,4,6-TCP are 323 and 616 mg g⁻¹ respectively at 25 Oliver C.

Keywords: Phenol Removal; Okra Leaves; Batch adsorption; Kinetic Models

1. Introduction

With the presence of non-biodegradable and volatile organic compounds formed because of the industrial effluents, water resources were quickly polluted. One of the most widespread organic water pollutants is the phenol; which is released through the industrial effluents, e.g. gasoline, rubber profiling, coal tar, plastics disinfectant, pharmaceuticals, chemical spills and agricultural run-off. Phenol is not only toxic at low concentration but also its presence in the natural water may further cause the formation of substituted compounds during the disinfection and oxidative process [2]. Even small amounts of phenol consumed by human beings have a negative effect as they lead to vomiting, nausea, coma, paralysis, greenish or smoky colored urine and even death due to respiratory failure or cardiac arrests. The adsorption of phenol by the skin when exposed to a large area of the skin can also lead to fatal poisoning, [3]. The World Health Organization (WHO) recommendation states that the maximum concentration of phenolic contents in drinking water is 0.002 mg L⁻¹ and the Environmental Protection Agency (EPA) established the concentration of phenols in wastewater less than 1 mg L⁻¹ [4]. Different physical and chemical processes have been researched in the removal of phenolic compound in wastewater but adsorption method is mostly recommended because it is very easy to adsorb [5]. Even though adsorption onto activated carbon has been one of the most popular and efficient methods of treating high strength and low volume of phenolic wastewaters. However, because of the high price of activated carbon and the vigorous regeneration of the used activated carbons makes it non economical adsorbent as opposed to the cheaply available, ecofriendly naturally occurring alternatives [6].

Among the studied that are aimed at removing phenolic compound like organic resins, clay materials, silicon, zeolite, agricultural wastes [7-11], many researchers are interested in the development of alternative low-cost adsorbents in the removal of organic pollutants in the aqueous solution. The agricultural wastes which are not utilized pose great disposal issues hence recycling of agricultural wastes is one of the key areas of study. The agricultural sector produces a lot of agricultural wastes that is utilized as domestic fuel in most countries of the world. Recycling this cheap, effective and massive agricultural wastes as an inexpensive

alternative adsorbent interests researchers a lot [12]. In current research agricultural waste okra leaves were used as a removal agent of toxic phenol as an adsorbent in a bid to come up with economical and friendly environment adsorbent. The vegetable plant Okra (*Abelmoschus esculentus* L. Moench, this was formerly *Hibiscus esculentus* L.) of the family Malvaceae. Okra plant is extensively planted in various regions around the world. Typically used as crop either as human food or as industrial fibre. The okra seed pods are very rich in nutrients, as well as, protein, vitamins carbohydrates and minerals, and the bulk of okra leaves consists of insoluble fiber [13]. In literature, okra is reported to be used in treatment of industrial textile waste water 2. Experimental Procedure

2.1 The materials employed will consist of instruments and chemical reagents.

HP LC (Hitachi Model 655) liquid chromatography with ZORB Eclipse XDB-C-18, (4.6× 150 mm, 5 µm) column were used in the determination of the concentration of Phenol 2,4,6-TCP in aqueous solutions. The UV detector wavelength was set to 270 nm. The digital (Origin 5 star) pH Meter was used to measure the pH. The batch adsorption experiments were carried out using an automatic thermostated shaker (Irmeco GmbH Model- 1-4000 Germany). IR spectra were recorded using the FT-IR spectrometer (Thermo Nicolet, USA) with the help of the diamond accessory. All the chemicals employed were of fine grade or analytical grade. A stock standard solution (1000 mL⁻¹) of phenol and 2, 4, 6- TCP (Merk Germany) was prepared by dissolving 0.1g of the stock standards solution into 100 mL of deionized water and working solution was obtained by diluting the stock standards solution. Phenol and 2, 4, 6- TCP solutions had their pH adjusted with hydrochloric acid HCl, Sodium hydroxide NaOH, Acetic acid CH₃COOH and Sodium acetate CH₃COONa.

2.2 The preparation of the adsorbent material will be carried out in

Okra leaves were taken to the fields, washed thoroughly with tap water to remove the dust and dried in the air. Additional dry leaves were ground and washed in deionized water and subjected to 1M HCl and 1M NaOH each after 30 minutes. Lastly the leaves were rinsed with deionized water until the effluent was neutral. Leaves that were washed with acid and base were dried at 80 °C in an oven over 18 hours. Sieving of various mesh sizes was done. In the present study, the size of the

particle was 85-100.

2.3 The batch adsorption of 2,4,6-TCP was studied on phenol

The behavior of phenol and 2,4, 6-TCP adsorption on the okra leaves were examined using batch equilibrium experiments. The 100 ml of capped glass bottles contained a fixed volume of phenol and 2,4,6-TCP solution of different initial concentration (10-1000 mL⁻¹) and was incubated with 0.1g of adsorbent in an incubated shaker with 150 rpm at pH 7 and temperature (25-45 °C) within 1 hour. Once the equilibrium had been reached, phenol and 2,4,6-TCP samples were filtered using a 0.45 µm filter paper and concentration of phenol and 2,4,6-TCP were measured by HPLC.

2.4 HPLC Analysis

The phenol and 2,4,6-TCP were analyzed by HPLC using a mobile phase of methanol:water (70:30) and methanol: 0.05 M formic acid (70:30), respectively. It was detected at 270 nm. Phenol had retention times of 1.8 and 2,4,6-TCP 6.6 minutes respectively.

Percentage sorption of phenol and 2,4,6-TCP and quantity of phenol and 2,4,6-TCP adsorbed on okra leaves are estimated by using the following equations.

$$\% \text{ Removal} = (C_0 - C_e) / C_0 \times 100 \quad (1)$$

C₀ is the initial phenol, TCP concentration and C_e is the equilibrium concentration (µg L⁻¹) of phenol and 2,4,6-TCP. To reduce the error, duplicate experiments were performed.

3. Result and Discussion

3.1 Characterization of the adsorbent was done using

The presence of functional groups was explored using Fourier-Transfer Infra-Red (FT-IR) analyzer. To determine the changes in the peaks, the data was taken prior to and after adsorption. FTIR spectrum indicate that there is a broad band at 3273.37 cm⁻¹ and two peaks at 2918.78 and 2850.61 cm⁻¹ this means that they are the O-H and N-H stretching. Other than that a strong peak at 1602.78 is attributed to C=O bond stretching. These peaks verify the presence of amino and carboxylic acid groups in adsorbent. In the meantime a shift of the maxima in N-H and C=O bonds following adsorption indicates that both phenol molecules are bound to these groups. fig.1

The adsorption process on okra leaves was optimized.

3.2.1 Effect of pH

The removal of adsorbates by adsorbents is very pH sensitive. To this end, the experiments were conducted in pH (1-11) to note the impact of pH in the removal of phenol and 2,4,6-TCP on Okra leaves. To this end, the model solutions of phenol (20 mg L⁻¹) and 2,4,6-TCP (20 mg L⁻¹) were adjusted to pH 1-11. In this model solutions 10 mL of each phenol and 2,4,6-TCP were added into 100 mL capped glass bottles and were interacted with 100 mg of adsorbent and shaken in incubated shaker with 150 rpm in 1 h. As illustrated in the Figure 2 initially % removal of phenol and 2,4,6-TCP rises with the rise of pH of solution. The percent removal at pH 7-9 at pH 5-7 at pH 7 and 5 respectively. Beyond this point of pH % reduction in both phenol and 2,4,6-TCP. The pK_a values of phenol and 2,4,6-TCP are 9.9 and 5.6 respectively. Typically, pH < pK_a phenolic compounds will be in molecular form and at pH > pK_a phenolic compounds will be in anionic form and at pH = pK_a, both molecular and anionic form will be 50% respectively. In addition to this the natural/agricultural adsorbents have various functional groups of varying chemical natures which may also be influenced by pH. As a result, the surface groups charges of adsorbent at very low PH may not support the adsorption of the phenols and 2,4,6-TCP and with higher PH but less than the pK_a values the molecular form of the two phenols and the surface groups are more attracted and hence the adsorption. At very high PH further the negative sites of adsorbent are more and phenolate ion concentration is also more which leads to repulsion forces and consequent decrease in the percentage removal at high PH [14]. To continue experiments the pH is set to pH 7 and pH 5 of phenol and 2,4,6-TCP respectively.

3.2.2 Impact of quantity of adsorbents

To investigate the influence of adsorbent amount 10 mL of model solutions of phenol (20 mg L⁻¹) and 2,4,6-TCP (20 mg L⁻¹) at pH 7 and 2 respectively in contact with different amounts of adsorbent 10-300 mg in 100 mL capped flasks at 150 rpm. It was noted that the higher the amount of adsorbent used in adsorption the higher the percentage of removal of both phenol and 2,4,6-TCP as depicted in figure 3. The fact that the amount of adsorption increases with the amount is

primarily because of the rise in surface area of the adsorbent as well as the rise in adsorption sites [15]. However, further addition of amount beyond 100 mg, the slight decrease of the percent removal of phenol and 2,4,6-TCP can be observed which could be due to the fact that addition of amount beyond 100 mg could result in adsorbent particles attaching with each other. Thus, 100 mg of adsorbent amount were chosen as a further study.

3.2.3 Shaking speed effect

The shaking speed also contribute significantly to adsorption process hence, the influence of shaking speed was researched. In this case, the 10 mL of model solution of phenol (20 mg L⁻¹) at pH 7 and 2,4,6-TCP (20 mg L⁻¹) at pH 5 were mixed with 100 mg of adsorbent in 100 mL capped glass bottles and stirred at 50-250 rpm in 1 h. As in figure 4. The percentage of removal of phenol and 2,4,6-TCP increased up to 200 rpm and then slightly reduced. Thus 200 rpm shaking rate was chosen in case of additional experiments.

3.2.4 Effect of time

The experiments were conducted in order to find out the effects of time on adsorption of phenol and 2, 4, 6- TCP onto okra leaves. In this 10 mL of phenol (20 mgL⁻¹) and 10 mL 2, 4,6-TCP solution (20 mg L⁻¹) at the optimum pH, the amount of adsorbent and the speed of shaking were in contact with various time intervals, 1 mint to 12 h. The percentage removal of phenol and 2, 4, 6-TCP at first rises with time due to the presence of the adsorption sites. The equilibrium in the adsorption of phenol onto Okra leaves was achieved after 180 min and the equilibrium time in the adsorption of 2,4,6-TCP was achieved after 60 min (Figure 5) and therefore, the adsorption experiments were conducted with a contact time of 180 and 60 min of phenol and 2,4,6-TCP respectively.

3.2.5 Effect of concentration

The influence of concentration on adsorption of phenol and 2,4, 6- TCP were studied at three temperatures i.e. 298, 308 and 318 K. In this model, the solutions of phenol and 2, 4, 6-TCP (1-500 mg L⁻¹), were in contact with adsorbent at constant values of other parameters. It was determined that the constant percentage removal of phenol was observed up to of 30 mg L⁻¹ and thereafter, it declined with the further

addition of concentration. In the case of 2,4,6-TCP, the percentage removal reached a peak of 100 mg L⁻¹ and then reduced as the concentration was further added. But the quantity of phenol and 2, 4, 6-TCP adsorbed on the surface increased with the initial concentration. This trend was similar at each of the three chosen temperatures (Figure 6 and 7).

3.3 Kinetics of adsorption

The Lagerren pseudo-first-order equation, pseudo second-order and Morris-Weber equations were used to study the kinetics of phenol and 2, 4, 6-TCP adsorption onto okra leaves. Correlation coefficient (R²) values were used to show how well experimental data and model-predicted values were similar to each other. The value of R² (R², values close or equal to 1) is relatively large, which means that the model is able to describe the kinetic.

3.3.1 Lagergrens pseudo-first-order equation The pseudo-first-order kinetic model has been widely used to evaluate the adsorption kinetics. The model liner form of the equation is:

$$\log (q_e - q_t) = \log (q_e - \frac{k_f}{2.303} t) \quad (2)$$

Where K_f is pseudo-first order rate constant, (min⁻¹), q_e (mg g⁻¹) the amount of adsorbate at equilibrium and q_t (mg g⁻¹) the amount of adsorbate at time t.

3.3.2 Pseudo-second-order equation The pseudo-second-order equation based on equilibrium

adsorption and the linear form of the equation is:

$$\frac{t}{q_t} = \frac{1}{k_s q_e^2} + \frac{1}{q_e} t \quad (3)$$

Where K_s is pseudo-second-order rate constant (g mg⁻¹ min⁻¹), q_e (mg g⁻¹) the amount of adsorbate at equilibrium and q_t (mg g⁻¹) the amount of adsorbate at time t.

3.3.3 Weber-Morris equation

The linear form of the equation is

$$q_t = k_{ip} t^{1/2} + c \quad (4)$$

In which, the rate constant of intra-particle diffusion is denoted as K_{ip} ($\text{mg g}^{-1} \text{min}^{-1/2}$) which is a fixed value, C is the intercept of the curve.

Lagerren pseudo-first-order equation, pseudo second-order, Morris-Weber (Eq. 2-4) were used to analyze the kinetics of phenol and 2,4,6-TCP adsorption on okra leaves respectively. Correlation coefficient (R^2) values were used to express the conformity of the experimental data and the values predicted by the models. Table 1 was a summary of the parameters of calculation of equations of the kinetic models. Though the q_e experimental and q_e determined using the pseudo first and second order model are quite similar to each other but based on the R^2 values it is evident that the pseudo-second order had a better fit to the kinetic data. A different model of description of kinetics process i.e. intra particle diffusion model was also tested. The extrapolation of the straight parts of the plots to the y-axis which is the measure of the thickness of boundary layer can provide the intercept. The straight line deviation of the origin shows that the pore diffusion is not the only rate-limiting step [16].

Tabl 1 kinetic parameters of phenol and 2,4,6-TCP onto Okra leaves

Adsorbate	Pseudo first order			Pseudo second order			Intra-particle diffusion		$q_e \text{ Exp}$ mgg^{-1}
	K_f min^{-1}	R^2	q_e mgg^{-1}	K_s $\text{gmg}^{-1} \text{min}^{-1}$	R^2	q_e mgg^{-1}	K_{ip} $\text{mg g}^{-1} \text{min}^{-1/2}$	R^2	
Phenol	-0.0145	0.97	18.88	0.0001	0.99	20.88	0.017	0.94	19.92
2,4,6-TCP	-0.027	0.98	42.34	0.0024	0.99	41.84	0.530	0.99	40.66

3.4 Equilibrium adsorption models

To check the efficiency of the prepared adsorbent, the adsorption phenol to adsorbent was conducted at three temp viz 25, 35, 45 C o to observe the equilibrium studies that were done at different initial concentration of phenol and 2, 4, 6-TCP. The data was explored using three various isotherms, the Langmuir, Freundlich and Dubinin-Radushkevich D-R isotherms.

3.4.1 Langumir isotherm

Langumir isotherm describe the homogeneous surface of the adsorbent. The linear form of the Langmuir equation is

$$\frac{C_e}{C_{ads}} = \frac{1}{Q_b} + \frac{C_e}{Q} \quad (5)$$

Where C_e (mgL^{-1}) is the equilibrium molar concentration of the phenol, C_{ads} (mgg^{-1})

is the amount of phenol adsorbed, Q (mLg^{-1}) is the maximum adsorption capacity and K_L (Lg^{-1}) is the constant for the binding energy of adsorbate.

The essential characteristic of Langmuir isotherm can be expressed in terms of dimensionless constant called separation factor or equilibrium parameter R_L which is defined by equation:

$$R_L = \frac{1}{1 + (bC_e)} \quad (6)$$

The value of R_L describe adsorption type as linear when $R_L = 1$, favorable when $0 < R_L < 1$, unfavorable when $R_L > 1$ and irreversible when $R_L = 0$, [17]. The R_L values have been calculated for the adsorption of phenol on okra leaves was less than unity (0.001-0.920, 0.02-0.940, 0.09-0.950) at 25, 35, 45 $^{\circ}\text{C}$ respectively, showing the favorable nature of adsorption.

3.4.2 Freundlich Adsorption Isotherm Freundlich is an empirical equation and assume the heterogeneous surface of the adsorbent. Freundlich Equation can be written in following linear form:

$$\log c_{ads} = \log k_f + \frac{1}{n} \log c_e \quad (7)$$

Where K_f is Freundlich constants related to adsorption capacity and exponent $1/n$ is heterogeneity factor [18]. C_{ads} and C_e are described previously.

3.4.3 Dubinin-Radushkevich D-R adsorption Isotherm D-R isotherm assumes no homogeneous surface of the adsorbent D-R isotherm plotted by following equation:

$$\ln c_{ads} = \ln K_{D-R} - \beta \varepsilon^2 \quad (8)$$

$$\varepsilon = RT \ln \left(1 + \frac{1}{c_e} \right) \quad (9)$$

$$E = \frac{1}{\sqrt{-2\beta}} \quad (10)$$

Where, the concentration of the solution and on the surface are at equilibrium C_e (mg L^{-1}) and C_{ads} (mgg^{-1}) respectively. The constants of the equations are K_D-R and e which is the maximum adsorption capacity and Polanyi potential respectively and can be calculated using the equation 9. Using equation 10 β slope of the curve can be used to determine energy of adsorption (Eq 10) and therefore nature of the adsorption. T = temperature and R = general gas constant. Magnitude of the E calculate can show the nature of the adsorption process i.e. whether $E > 8-16 \text{ kJ mol}^{-1}$ adsorption can be considered as chemisorption and $E < 8 \text{ kJ mol}^{-1}$ physisorption may be the one that adsorbs adsorbate [18]. Linear graph between $\ln C_{ads}$ vs e can be plotted.

Phenol and 2,4,6-TCP adsorption was studied on adsorbent at three temperatures viz 298, 308, 318 K to find out the efficiency of prepared adsorbent, the equilibrium studies were conducted at different initial concentration of phenol and 2,4,6-TCP. The data was explored using three various isotherms i.e. Langmuir, Freundlich and Dubinin-Radushkevich D-R isotherms. The various parameters derived based on the isotherm models of phenol and 2,4,6-TCP are tabulated in table 2 and 3 respectively.

Table 2: Isotherms parameters of phenol onto Okra leaves at different temperatures

Temperature k	Langmuir				Freundlich			D-R		
	Q mgg^{-1}	R_L	b L g^{-1}	R^2	K_F mgg^{-1}	1/n	R^2	K_{D-R} mgg^{-1}	E kJ mol^{-1}	R^2
298	85.77	0.001-0.920	8.1×10^4	0.998	323	0.26	0.993	94.00	10.24	0.986
305	67.64	0.020-0.940	3.2×10^4	0.995	297	0.26	0.992	73.21	9.53	0.985
315	43.30	0.09-0.950	9.6×10^4	0.995	190	0.23	0.991	72.43	9.51	0.974

Table 3: Isotherms parameters of 2,4,6-TCP onto Okra leaves at different temperatures

Temperature k	Langmuir				Freundlich			D-R		
	Q mgg ⁻¹	R _L	b L g ⁻¹	R ²	K _F mgg ⁻¹	1/n	R ²	K _{D-R} mgg ⁻¹	E kJ mol ⁻¹	R ²
298	138.8	0.92	1.05×10 ⁴	0.992	616	0.21	0.991	184	11.47	0.98
305	120.2	0.092	2.20×10 ⁴	0.992	597	0.23	0.991	175	10.16	0.98
315	109.4	0.090	4.61×10 ⁴	0.991	580	0.27	0.982	162	10.07	0.97

3.5 Thermodynamics study

The adsorption nature of the phenol and 2,4,6-TCP was evaluated by thermodynamic parameters. The thermodynamic constants, enthalpy change ΔH , entropy change ΔS and free energy change ΔG were calculated from equation 1.5-1.7.

$$kc = \frac{Fe}{1-Fe} \quad (11)$$

$$\ln kc = -\frac{\Delta H}{RT} + \frac{\Delta S}{R} \quad (12)$$

$$\Delta G = -RT \ln K \quad (13)$$

Where T is the gas constant, R, is the temperature in kelvin and K, is the equilibrium constant. The values of ΔH were determined as -33.8, -40.2 kJ mol⁻¹ of phenol and 2,4,6-TCP respectively are an indication of exothermic nature of adsorption onto okra leaves where is -1.64, -2.30 kJ mol⁻¹ of phenol and 2,4,6-TCP respectively and S 0.10, 0.

4. REMOVAL OF PHENOL 2,4,6-TCP FROM REAL WATER SAMPLE

The resulting adsorbent was used in the purification of phenol and 2,4,6-tcp polluted water in various industrial discharges in Sindh Pakistan. Table 4 presents the results.

Table 4: % Removal of Phenol and 2,4,6-TCP from wastewater samples

Sample	Analyte	Concentration (mg L ⁻¹)	Removal (%)	RSD
S1	Phenol	3.52	95	1.6
S2	Phenol	5.34	94	1.5
S3	2,4,6-TCP	2.5	97	1.3
S4	2,4,6-TCP	5.2	98	1.7

5. Conclusion. Okra leaves are agricultural waste material that can be used as an economically best adsorbent for effective removal of phenol 2,4,6-TCP from water. Pseudo second order model described well the mechanism of adsorption and freundlich isotherm describe the multilayer adsorption of phenol 2,4,6-TCP onto okra leaves.

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Figure 1: FTIR spectrum of Okra leaves

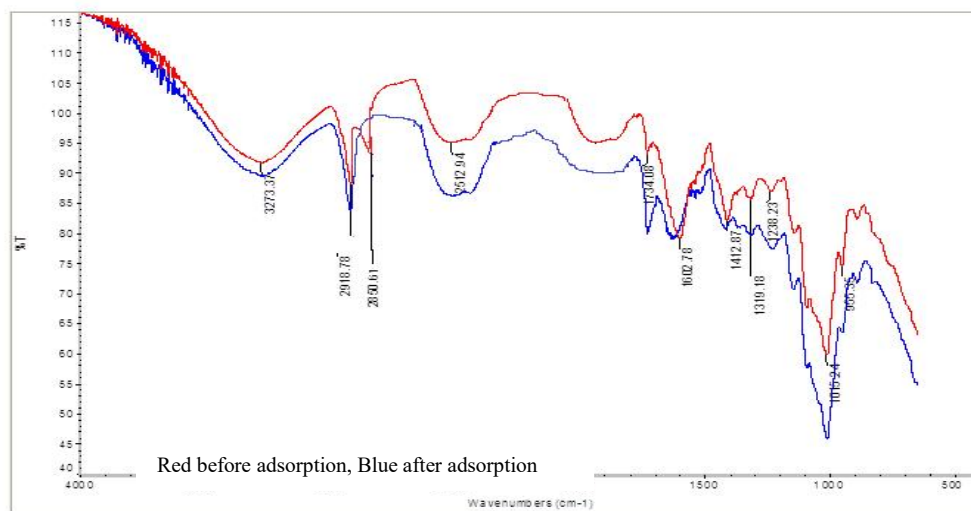


Figure 2: Effect of pH on % removal of phenol and 2,4,6-TCP onto Okra leaves

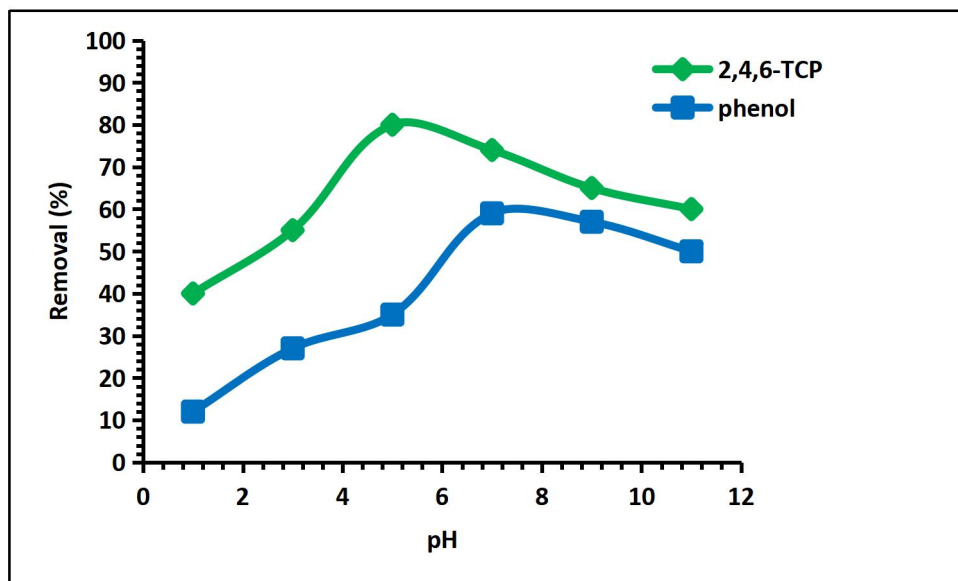


Figure 3: Effect of adsorbent amount on % removal of phenol and 2,4,6-TCP onto Okra leaves

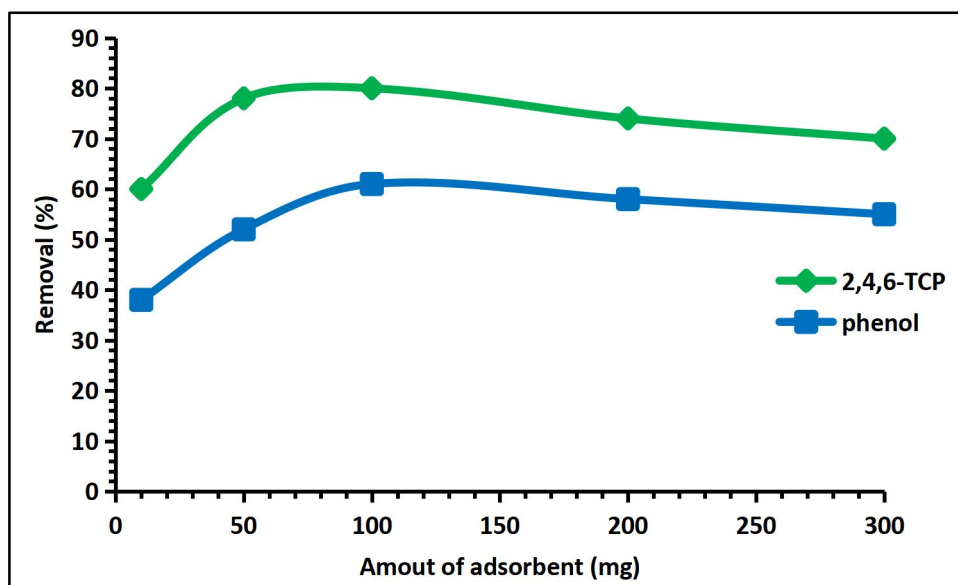


Figure 4 : Effect of shaking speed on % removal of phenol and 2,4,6-TCP onto Okra leaves

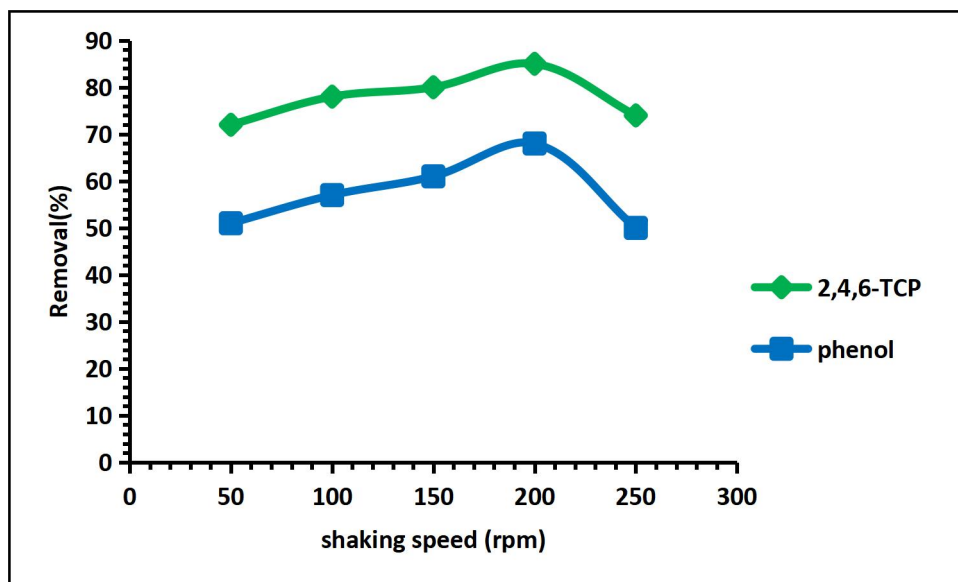


Figure 5: Effect of time on % removal of phenol and 2,4,6-TCP onto Okra leaves

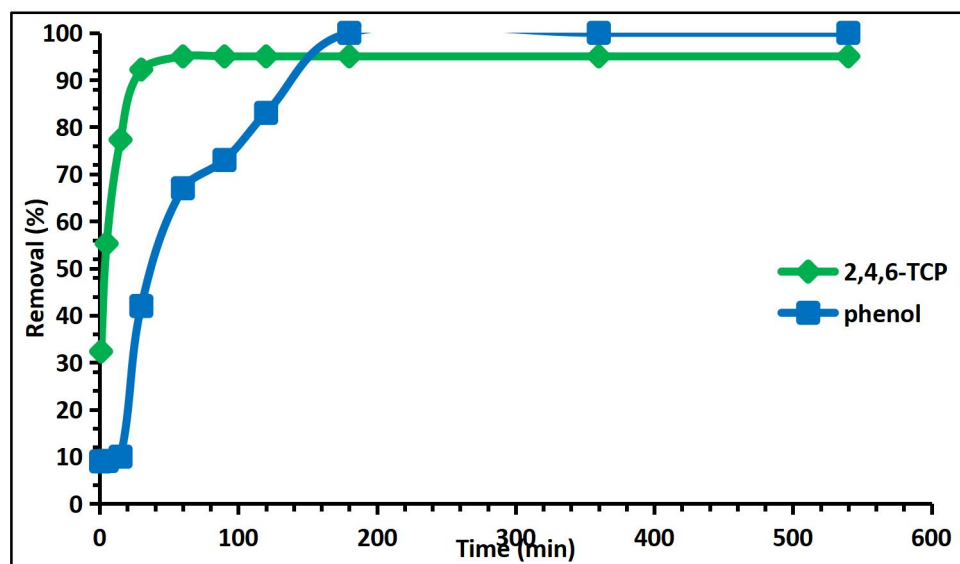


Figure 6: Effect of concentration on % removal of phenol onto Okra leaves

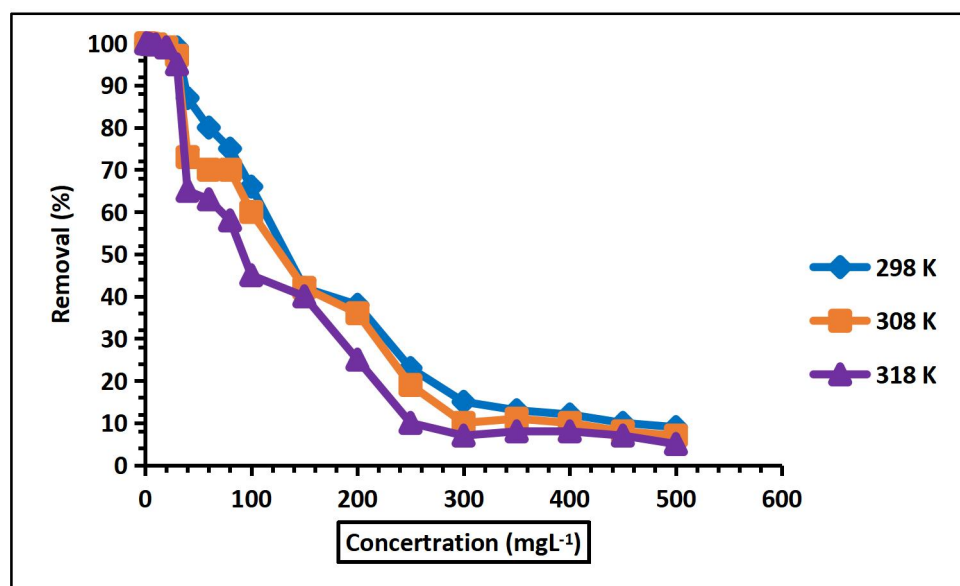


Figure 7: Effect of concentration on % removal of phenol onto Okra leaves

