

Survey of Efficiency Agricultural Waste as Adsorbent for Removal of P-Cresol from Aqueous Solution

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Authors' contributions

This work was carried out in collaboration between both authors. Both authors read and approved the final manuscript.

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ABSTRACT

This study was conducted to evaluate the feasibility of P-Cresol removal from aqueous solutions using canola stalk. The effect of Contact time, Concentration of P-Cresol, biomass dosage and pH on adsorption of P-Cresol by canola biomass were also evaluated. The results showed that the optimum amount of each parameter was as follows: initial concentration = 25 mg/L, pH = 3, contact time = 75 minutes, and adsorbent dosage = 4 g/L. The maximum adsorption efficiency was about 99.1%. The maximum adsorption capacity was calculated to be about 41.6 mg/g of adsorbent. Pseudo first order and Pseudo second-order, intraparticle diffusion models were adopted to evaluate experimental data and thereby elucidate the kinetic adsorption process. Additionally, the Langmuir, Freundlich, Redlich–Peterson and Temkin isotherm models were applied to describe equilibrium adsorption. The increasing of contact time and biomass dosage was led to increase of

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the percentage of P-Cresol adsorption. Conversely, the adsorption percentage decreased as P-Cresol concentration increased. The equilibrium data is best fitted on Langmuir isotherm and the adsorption kinetic model follows pseudo-second model. It is evident from the results that canola biomass have shown good potential for the removal of isomeric phenol from aqueous solution.

Keywords: Adsorption; canola stalk; p-cresol; isotherm; kinetics.

1. INTRODUCTION

Organic compounds is accounted as a very great group of pollutants in the wastewater [1]. Very often small amounts of synthetic organic compounds (including phenol and its derivatives, pesticides, aliphatic and aromatic hydrocarbons, dyes and surfactants among most representatives) are detected in water streams [2,3]. P-Cresol is one of phenol derivatives which a methyl has replaced at the para position relative to hydroxy group [4]. P-Cresol has a wide variety of uses including as disinfectants, fumigants, explosives, in the manufacturing of synthetic resins, in photographic developers, etc [5]. It is a naturally occurring metabolic product that is formed from tyrosine by bacteria under anaerobic conditions [6]. It is highly toxic and even at a very low concentration and it may act as a promoter for stomach tumors [7]. In addition to the corrosiveness effects on eyes, skin and respiratory track, it also may cause adverse effects on the central nervous system, cardiovascular system, lungs, kidney, and liver resulting in CNS depression. Exposure at higher levels may result in lowering in consciousness and death [8,9]. Environmental Protection Agency (EPA) has classified p-cresol as pollutant of group C (possible human carcinogens) [10, 11]. Therefore, it is essential to reduce the p-cresol concentration in effluents to protect our aqueous ecosystem [12]. Several conventional physiochemical techniques are available in the literature to treat wastewater. These include chlorination, ozonation, membrane process, solvent extraction, flocculation, coagulation, etc [13,14]. Most of these methods are face with serious drawbacks such as high cost, tendency of the formation of secondary hazardous byproducts, inadequate purification [15]. Adsorption process is one of the most successful and economically feasible methods for the removal of Phenolic compounds from aqueous solutions [16]. The high cost of activated carbon sometimes makes limited its use [17]. thus, a lot of researchers have attempted to explore the relatively inexpensive alternative materials, which can reveal reasonable adsorptive efficiency

[18-20]. The canola stalk is a lignocellulosic waste widely produced in all over of world due to farming of canola for the production and consumption of vegetable oils [21,22]. Therefore, the canola stalk is easily available and, due to its characteristics, has been used in several studies for pollutant removal [23-25]. The aim of the present study was to investigate the ability of canola for P-Cresol removal from aqueous solution. In addition, the effect of various parameters including contact time, pH, adsorbent dose and initial P-Cresol concentration on removal efficiency was surveyed. Finally, the isotherm and kinetic studies of P-cresol adsorption was performed.

2. MATERIALS AND METHODS

2.1 The Characteristics of P-Cresol

The used P-Cresol (4-methylphenol) was the analytical grade which were purchased from Sigma-Aldrich CO (purity 99%). It's molecular weight is 108.14 mg/mol and chemical formula is $\text{CH}_3\text{C}_6\text{H}_4\text{OH}$. The stock solution (1000 mg/L) was prepared and then desired concentration of P-Cresol solution was prepared by dilution of stock solution. A calibration plot (absorbance versus concentration of p-cresol) was drawn and used for estimating the concentration of unknown p-cresol concentration. The plot was linear ($R^2=0.992$) between 0 and 20 mg/L p-cresol solution. The test samples drawn from experiments with higher concentrations of p-cresol were adequately diluted and then the absorbance was determined. Samples were taken from the reactor at predetermined intervals for analysis.

2.2 Adsorbent Characterization

The specific surface area of dried Canola was determined by the BET-N₂ method using an ASAP 2000 apparatus based on nitrogen adsorption-desorption isotherms at 77K. The surface images of dried Canola before and after adsorption process were captured by a Philips XL30 scanning electron microscope (SEM).

2.3 Preparation Procedure of Adsorbent

In this study, the Canola was used as low cost natural or agricultural wastes for P-Cresol removal from aqueous solutions. The stalks of Canola(C) were collected from research farm of Tabriz agricultural school. The stalks were washed several times with water to remove the contaminant then dried in the oven at 105°C for 5 h. The prepared biomass was then treated with 0.1M HCL for 2 h followed by the washing with distilled water and subsequently was oven dried at 105°C for 5 h. After drying, adsorbent were sieved to obtain particle size of 18 mesh prior to being used for adsorption studies [26].

2.4 Batch Adsorption Experiments

In present study, the batch adsorption system was employed and the most effective factors for adsorption process including contact time (10-150 min), pH (3-11), adsorbent dose (0.5-8 g L⁻¹) and initial concentration of P-Cresol (10- 200 mg/L) were assessed.

The optimum pH was determined by varying the pH in range of 3-11 and keeping constant of other variables (contact time, adsorbent dose and initial P-Cresol concentration). The experiments were performed in 200 milliliter beaker with a constant concentration of P-Cresol. Then this mixture was shaken with a shaker device of enforce model with 180 rpm and the room temperature of 20 – 25°C. In the next step, the optimum adsorbent dose was estimated by keeping constant of contact time and initial P-Cresol concentration and also the obtained optimum pH. After determination of the optimum pH and adsorbent dose, the various concentrations of the P-Cresol in the specified times of contact was investigated. The effect of solution pH on the retention of p-cresol on the studied adsorbents was investigated by adding 0.1 M NaOH or HCl (for pH adjustment) to the stock solution containing the aromatic compound. The concentration of p-cresol in influent and effluent was measured using DR5000 UV/Vis spectrophotometer (HACH, USA) at 280 nm wavelength [8]. All the adsorption measurements were carried out at room temperature in a stirred batch system thermostatically controlled with an external circulating bath. Before these experiments, kinetics studies were performed to determine the equilibration time of the systems.

All the adsorption measurements were done in triplicate. The amount adsorbed was determined by the following equation [27]:

$$q_t = (C_0 - C_t)W/V$$

Where q_t is the amount (mg g⁻¹) adsorbed at time t , C_0 is the initial concentration (mg/L), C_t is the concentration at time t (mg/L), and V is the volume (L) of the adsorbate solution and W is the weight (g) of the adsorbent.

3. RESULTS AND DISCUSSION

Scanning electron microscopy (SEM) images were used to analyze the surface structure of Canola (Fig. 1). Fig. 1(a) clearly shows the pore textural structure of dried Canola before use. However, the Fig. 1(b) indicated that the clear pore textural structure is not observed on the surface of dried Canola after use which it could be due to either agglomeration on the surface or the invasion of P-Cresol into the pores of dried Canola. It was found that the adsorbent has Heterogeneous surface structure with deep pores. The specific surface area of modified Canola was determined in the size of 32 m²/g. The similar results were obtained for sorption of Acid blue 113 and Cr⁺⁶ from aqueous solutions onto Canola, also [22,23].

3.1 Effect of Contact Time and Initial Concentration on P-Cresol Adsorption

Fig. 2 depicts the effect of contact time on the removal of P-Cresol at various initial concentrations (25–200 mg/L). The saturation curves rise sharply in the initial stages illustrate that there are plenty of readily accessible sites. Eventually, a plateau is reached in all curves indicating that the adsorbent is saturated at this level. It can be seen from Fig. 2 that the contact time needed for P-Cresol solutions with initial concentrations of 25–200 mg/L to reach equilibrium was 75 min. It was also seen that an increase in initial P-Cresol concentration resulted in increased P-Cresol uptake. The removal curves are single, smooth and continuous, indicating the formation of monolayer coverage of the P-Cresol molecules onto the outer surface of the adsorbent. The adsorption capacity at equilibrium (q_e) increased from 6.19 to 35.6 mg/g with an increase in the initial P-Cresol concentrations from 25 to 200 mg/L. Three consecutive mass transport steps are associated with the adsorption of solute from solution by

porous adsorbent [28]. First, the adsorbate migrates through the solution, i.e., film diffusion, followed by solute movement from particle surface into interior site by pore diffusion and finally the adsorbate is adsorbed into the active sites at the interior of the adsorbent particle. This phenomenon takes relatively long contact time [29,30].

3.2 Effect of pH

The pH of the solution plays an important role in the adsorption capacity. The effect of initial pH of P-Cresol solution on the percentage removal was studied by varying the initial pH under constant process parameters (4 g/L of adsorbent

at initial P-Cresol concentration 20 mg/L). The results are shown in Fig. 3. The uptake of P-Cresol increased with decreasing initial pH and it was greatest at pH 3. The pH primarily affects the degree of ionization of the P-Cresol and the surface properties of Canola. At low pH values, the functional groups of chitin would be protonated and result in a stronger attraction for negatively charged ions in the adsorption medium. P-Cresol being weakly acidic would be partially ionized in solution [8]. These ions will be negatively charged and will be directly attracted due to electrostatic forces by the protonated amino groups of the chitin. As the pH increased, the overall surface charge of the Canola became negative and adsorption decreased [26,31].

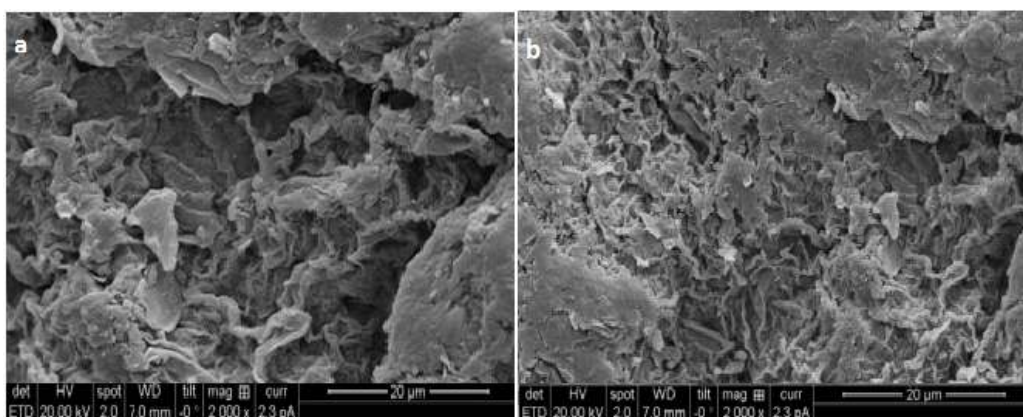


Fig. 1. The SEM image of dried Canola: (a) before use; (b) after use

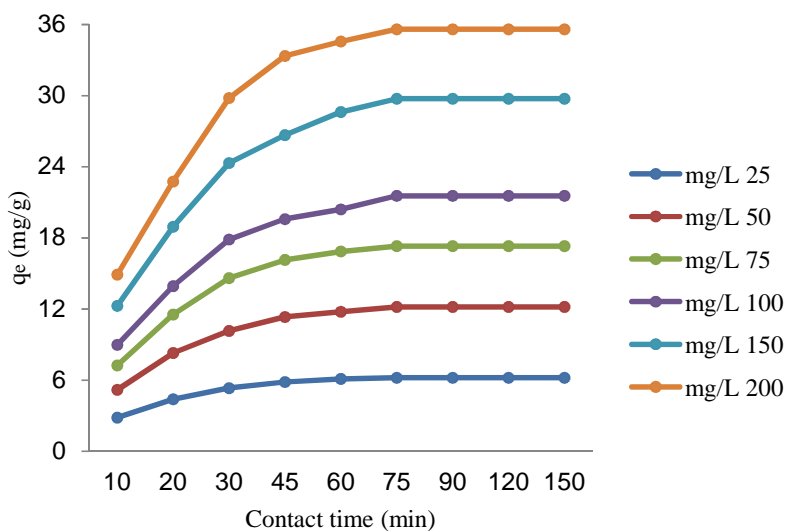


Fig. 2. Effect of contact time and initial P-Cresol concentration on removal efficiency (pH = 3, adsorbent dose 4 g/L, Temp: 25°C)

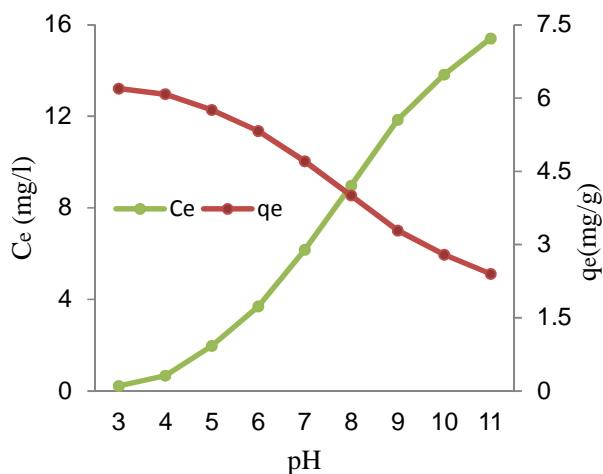


Fig. 3. Effect of pH on P-Cresol removal efficiency ($C_0 = 25$ mg/L, adsorbent dose of 4 g/L, Time = 75 min and Temp: 25°C)

3.3 Effect of Adsorbent Dose

The dependency of adsorption of the P-Cresol on the concentration of dried Canola was investigated by varying the quantity of the adsorbent from 0.5 to 8 g/L in 50 mg/L solution of the P-Cresol while keeping constant of other parameters (contact time, pH, initial P-Cresol concentration and temperature). As shown in Fig. 4, adsorption capacity decreased from 41.6 to 6.11 mg/g for an increase in adsorbent mass from 0.5 g to 8 g, whereas the percentage removal increased from 41.6 % to 97.5% with the same increase in adsorbent mass. This observation is usually attributed to the increase in the adsorbent pore surface areas and availability of more adsorption sites with increasing mass of adsorbent [32,33].

3.4 Adsorption Isotherms

Adsorption isotherm is basically important to describe how solutes interact with adsorbents, and is critical in optimizing the use of adsorbents. The Langmuir, Freundlich, Temkin and Dubinin–Radushkevich isotherm models were used to describe the relationship between the amount of P-Cresol adsorbed and its equilibrium concentration in solutions. The Langmuir isotherm is valid for monolayer adsorption on a surface containing a finite number of identical sites. The model assumes uniform adsorption on the surface and no transmigration in the plane of the surface. The linear form of the Langmuir isotherm can be represented by the following equation [34]:

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} \times \frac{C_e}{q_m}$$

Where C_e (mg/L) is the equilibrium concentration of the adsorbate, q_e (mg/g) is the amount of adsorbate adsorbed per unit mass of adsorbent, q_0 and b are Langmuir constants related to adsorption capacity and rate of adsorption, respectively. The Langmuir plots for P-Cresol adsorption onto Canola biomass are obtained in Fig. 5, and the parameters are shown in Table 1. The correlation coefficients of the all isotherms models, used in this study, are higher than 0.995 at the different concentration, but it can be seen that the Langmuir isotherm can best described the equilibrium data. The applicability of the model suggests monolayer coverage of the adsorbate at the outer surface of the adsorbent is significant. The essential characteristics of the Langmuir isotherm can be expressed in terms of a dimensionless separation factor (R_L) which is defined by [35]:

$$R_L = \frac{1}{1 + bC_0}$$

Where b is the Langmuir constant and C_0 is the highest P-Cresol concentration (mg/L). The value of R_L indicates the type of the isotherm to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$). The value of R_L was found to be 0.32-0.64 and this again confirmed that the Langmuir isotherm model is more favorable for adsorption of P-Cresol onto the activated carbon under the conditions used in this study.

Freundlich model is an empirical equation assuming heterogeneous adsorptive energies on the adsorbent surface, which can be written as [36,37]:

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e$$

Where q_e is the equilibrium P-Cresol concentration on the adsorbent (mg g^{-1}); C_e , the equilibrium P-Cresol concentration in solution (mg L^{-1}); and K_f is the Freundlich constant. In this function, it is assumed that the sorbent has a surface with a non-uniform distribution of sorption heat. This equation was primarily proposed on a purely empirical basis for adsorption phenomena occurring on gas–solid interfaces, although it can be theoretically derived for an adsorption model in which the heat of adsorption varies exponentially with surface coverage. The slope of plot $1/n$ ranging 0 and 1, is a measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero.

Tempkin considered the effect of some indirect sorbate/adsorbate interactions on the adsorption isotherm. This isotherm assumes that; the heat of adsorption of all the molecules in a layer decreases linearly with surface coverage of adsorbent due to sorbate–adsorbate interactions. This adsorption is characterized by a uniform distribution of binding energies. The linear form of the Tempkin isotherm equation is represented by the following equation [38]:

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

Where $B = RT/b$, T is the absolute temperature in K, R the universal gas constant ($8.314 \text{ K}^{-1} \text{ mol}^{-1}$), A the equilibrium binding constant and the constant B is related to the heat of adsorption.

Another isotherm which was used for the prediction of adsorption behavior is the Redlich–Peterson model. Redlich–Peterson equation included three adjustable parameters into an empirical isotherm. This equation is widely used as a compromise between Langmuir and Freundlich systems. The equation for this model is [39]:

$$\ln \left[\frac{k_R C_e}{q_e} - 1 \right] = \ln aR + \beta \ln C_e$$

Where K_R and β are the constants of R–P model. The above equation is reduced to the Langmuir isotherm when the value of β is equal to 1 but it is reduced to a Freundlich isotherm in β value of much bigger than 1.

Table 1 lists the parameters of the isotherm models along with the regression coefficients (R^2). The R^2 values in Table 1 reveal that the Langmuir isotherm fits the experimental results better than other model, implying that the adsorption of P-Cresol onto Canola biomass follows the mechanism of monolayer adsorption (chemisorption) on a homogenous surface. Although the Langmuir and Redlich-Peterson equations have similar correlation coefficients, the Langmuir is a simpler equation and therefore easier to use. Consequently, it was selected to predict q_e values in the present work.

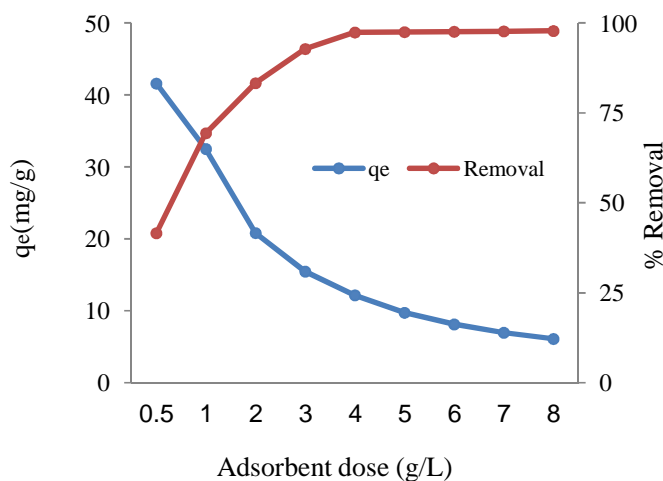


Fig. 4. Effect of biomass dose on P-Cresol biosorption ($C_0 = 50 \text{ mg/L}$, $\text{pH}=3$, Contact time=75 min and Temp: 25°C)

Table 1. The adsorption isotherms constants for the removal P-Cresol onto Canola biomass

Dose (g/l)	q_e exp (mg/g)	Langmuir model			Freundlich model			Temkin model			Redlich–peterson model			
		q_m	K_L	R^2	n	K_F	R^2	B	A	R^2	k_R	aR	β	R^2
0.5	46.22	41.81	0.0045	0.997	3.71	9.14	0.876	29.12	0.245	0.741	14.4	0.172	0.98	0.984
0.1	34.71	32.17	0.0067	0.999	3.22	12.45	0.849	21.45	0.384	0.796	17.7	0.346	0.95	0.976
0.2	20.82	19.74	0.0079	0.998	2.64	14.66	0.854	17.24	0.514	0.823	21.2	0.593	0.96	0.965
0.4	12.17	12.45	0.0094	0.996	1.95	16.73	0.832	11.69	0.694	0.804	23.4	0.845	0.99	0.989

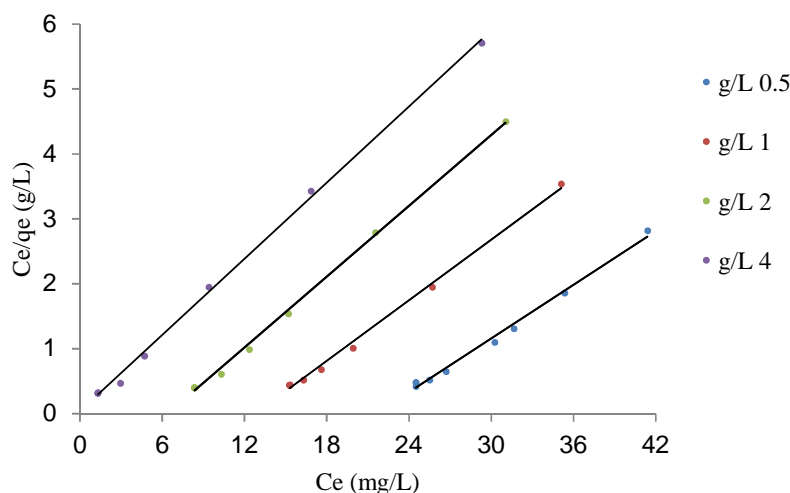


Fig. 5. Langmuir plots for the P-Cresol adsorption onto Canola biomass

3.5 Adsorption Kinetics

In order to examine the mechanism and rate-controlling step in the overall adsorption process, three kinetic models, pseudo-first-order, pseudo-second-order and intra-particle diffusion, are adopted to investigate the adsorption process.

The pseudo-first-order equation can be expressed as the following equation [40,41]:

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1}{2.303} t$$

Where q_e and q_t (mg/g) are the P-Cresol adsorption capacity at equilibrium and at time t (min), respectively, and k_1 (min^{-1}) is the rate constant of the pseudo-first-order.

The parameters k_1 and q_e could be calculated from the slope and intercept of the plots of $\log(q_e - q_t)$ versus t and are given in Table 2. The values of the correlation coefficient R^2 obtained at all the studied concentrations are low and in range of 0.741–0.862. Furthermore, the experimental values of q_e, exp (mg/g) are far from the calculated q_e, cal (mg/g). This suggests that the pseudo-first-order kinetic model is not suitable to describe the adsorption process.

On other hand, the pseudo-second-order kinetic model can be expressed in linear form as follows [42]:

$$\frac{t}{q_t} = \frac{1}{k_2 \cdot q_e^2} + \frac{t}{q_e}$$

where k_2 (g/mg min) is the rate constant of the pseudo-second-order. The q_e and k_2 values can be obtained from the slope and intercept of plots of t/q_t versus t which are illustrated in Fig. 6. The good linear plots of t/q_t versus t at different concentrations with the correlation coefficients (R^2) higher than 0.997 suggest that adsorption of P-Cresol onto canola biomass predominantly follows the pseudo-second-order kinetic model. Besides, the calculated data (q_e, cal) is well agreed with the experimental data (q_e, exp). The parameters are listed in Table 2. The same conclusion was found in the literature on the adsorption of P-Cresol by *lemna minor* and *azolla filiculoides* biomass.

The two above models cannot identify the diffusion mechanism during the adsorption process, so the experimental data was tested by the intra-particle diffusion model, which can be expressed by following equation [43]:

$$q_t = k t^{0.5} + C$$

Where q_t (mg/g) is the amount of P-Cresol adsorbed at time t (min), k ($\text{mg/g min}^{1/2}$) is the intra-particle diffusion rate constant and C is the intercept.

The adsorption of a solute from solution by porous adsorbents is essentially related to three consecutive steps. The first step is the external surface adsorption or the instantaneous

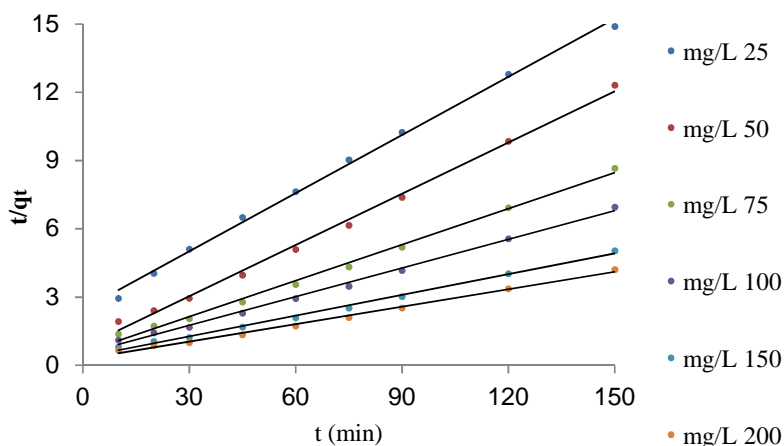


Fig. 6. Pseudo-second-order kinetic plots for P-Cresol adsorption onto Canola biomass at different concentrations

Table 2. Adsorption kinetic constants P-Cresol adsorption onto canola biomass

C_o (mg/L)	q_e exp (mg/g)	Pseudo-first order			Pseudo-second order			Intraparticle diffusion		
		K_1	q_e	R^2	K_2	q_e	R^2	K	C	R^2
25	6.196	0.446	3.251	0.782	0.0914	5.994	0.997	2.14	1.212	0.956
50	12.17	0.149	7.144	0.741	0.0689	11.49	0.998	3.84	3.176	0.967
75	17.30	0.0929	12.45	0.862	0.0341	16.42	0.998	4.45	2.459	0.972
100	21.55	0.0551	15.64	0.793	0.0172	20.85	0.999	5.98	4.193	0.981
150	29.73	0.0468	21.19	0.821	0.0035	29.11	0.999	6.29	5.648	0.945
200	35.61	0.0119	26.38	0.842	0.0056	36.24	0.998	7.36	6.198	0.963

adsorption. The second step is the gradual adsorption stage where intra-particle diffusion is rate-limiting. The third step is the final equilibrium stage where intra-particle diffusion started to slow down due to the extremely low adsorbate concentrations left in the solutions. One or more of these three steps control the adsorption rate. The parameters are listed in Table 2 and show a double straightline nature. Initially, it is postulated that P-Cresol is transported to the external surface of canola biomass through film diffusion within a very short time. The first linear part could be due to the entry of P-Cresol molecules into the canola biomass particle by intra-particle diffusion. The second linear part represents the final equilibrium stage. As given in Table 2, it is obvious that values of k increase from 2.14 to 7.36 mg/g min^{1/2} when the initial P-Cresol concentrations increase from 25 to 200 mg/L.

4. CONCLUSIONS

The results obtained from batch equilibrium experiments indicated that the studied parameters including initial solution pH,

biosorbent dosage, contact time and initial P-Cresol concentration was significantly able to effect on the biosorption of P-Cresol by Canola biomass. Low pH and initial P-Cresol concentration increased the biosorption of P-Cresol. With increase in biosorbent dosage and Contact time, the removal efficiency is also enhanced. The results indicated that the Langmuir model provided the best correlation of the experimental data. Pseudo-second-order model was best applicable to the sorption data. The study showed that Canola biomass can be used successfully to remove the pollutant from effluents.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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