IJISET - International Journal of Innovative Science, Engineering & Technology, Vol. 2 Issue 8, Aug 2015.
www.ijiset.com
ISSN 2348 – 7968

Removal of Phenolic compounds Using Canola Stalks Waste as a new low cost adsorbent

Davoud Balarak[1], Yousef Mahdavi[2], Ali Jogataei[3]

[1-] Department of Environmental Health, Health Promotion Research Center, School of Public Health, Zahedan University of Medical Sciences, Zahedan, Iran

2- MSc Student of Environmental Health engineering, Student Research Committee, Mazandaran University of Medical Sciences, Mazandaran, Iran

3- MSc Student of Environmental Health engineering, Student Research Committee, Qom University of Medical Sciences, Qom, Iran

Abstract: This study was aimed to determine the p-cresol and 2-chlolorphenol removal efficiency using the dried Canola from aqueous solution. The batch experiments were performed to this purpose and the influence of different variables on removal efficiency was investigated. The specific surface area was measured and was found to be $32m^2/g$. The scanning electron microscopy of the adsorbent was performed. The results of this study indicated that the contact times and adsorbent dosage increase the removal efficiency of p-cresol and 2-chlolorphenol however the pH and initial concentration of p-cresol and 2-chlolorphenol decrease the removal efficiency. Equilibrium time was found to be 75 for p-cresol and 90 min for 2-chlolorphenol and maximum Phenolic compounds removal efficiency was obtained in pH=3. The kinetic and isotherm studies indicated that Langmuier isotherm and pseudo-second-order kinetic are able to describe the obtained data. It can be concluded that the dried Canola can be good adsorbent for Phenolic compounds removal from aqueous solution.

Key words: Adsorption, of Phenolic compounds, Canola, adsorption capacity.

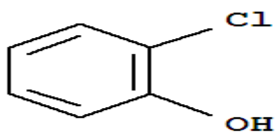
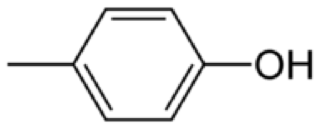
The phenolic compounds are one of important chemicals which are employed in a variety of industries (1). P-cresol (4-Methylphenol) is a polar aromatic and toxic compound which belongs to phenolic compound(2-3). Previous studies has been reported an assortment of application for p-cresol such as disinfectants, fumigants, explosives, in the manufacturing of synthetic resins, in photographic developers (4). Also, p-cresol can be significant threat to water resource due to its high solubility in water(5-6). It is introduced as prior pollutant and can severely affected the central nervous system, cardiovascular system, lungs, kidney, liver and creation of cancer(7).Also Chlorophenols are an important class of aromatic pollutants in industrial wastewaters and belong to a group of common environmental contaminants(8). Thus, the subtraction of phenolic compounds can be supposed as one of important targets to alleviate the hazardous effect of these compounds. A variety of techniques including adsorption, ultrafiltration, reverse osmosis, advanced oxidation, wet oxidation, ion exchange, biological have been suggested to diminish the phenolic compounds from industrial waste(9-10). The adsorption has been found to be the best method to eliminate of these chemical. The activated carbon is employed as a most effective adsorbent but it is not significant option in developing countries due to its high cost associated with use and regeneration(11-12). This problem has been stimulated the researchers to find a low-cost and efficient adsorbent; thus, they were studied on various material such as Azolla(13), Funalia trogii pellets(14), Banana peel(15), Tea waste(16), Saccharomyces Cerevisiae(17), and etc. the Canola is one of agricultural waste and has been used as an inexpensive adsorbent to remove the dyes. The Canola is one plant which is used to produce the oil. High availability and high porosity are the important features of Canola stalks(18-20). In this study, the p-cresol and 2-chlorophenol, was adsorbed onto the dried Canola from aqueous solution and the effect of various parameters (contact time, pH, and temperature, initial concentration of Phenolic compounds and dosage of adsorbent) were studied. Also, the kinetic and isotherm of the p-cresol adsorption was estimated.

Material and method

The Canola stalks were supplied from Agricultural university of Tabriz, Iran. The Canola stalks were washed and then dried in the in the oven at 105 ℃ for 5 hours followed by were treated with 0.1 M HCl for a period of 5 h. the prepared biomass was rinsed with distilled water and finally was sun dried. The resultant biomass was crushed and sieved to obtain the particle size of 1-2 mm and then was stored to use in next experiments. The adsorbent was analyzed for determination of the specific surface area by a Gemini2357 surface area analyzer of Micromeritics Instrument Corporation, USA. In addition, the morphology of adsorbent before and after use was estimated by a Philips XL30 scanning electron microscope (SEM), Amsterdam, Netherlands .

The analytical grade of p-cresol and 2-chlolorophenol was purchased from Merck .Co and was used in this study. All desired experimental solution was diluted from the stock solution (1000 mg/L) of p-cresol and 2-chlolorophenol. The characteristics and chemical structure of Phenolic compounds was presented in Table 1 .

Table1: the charactristics of 2-Chlorophenol and p-cresol

C.I. name	Molecular weight	λ_{max} (nm)	Molecular formula	chemical structure
2-Chlorophenol	128.56 g/mol	274	C_6H_5ClO	
p-cresol	108.13 g/mol	280	$CH_3C_6H_4(OH)$	

In this study, the dried canola stalks was utilized as an inexpensive adsorbent to remove the p-cresol in batch experiments. Some variables and parameters which can be effective on p-cresol removal efficiency were assessed. The parameters are included contact time (10-180 min), pH (3-11), p-cresol and 2-chlorophenol concentration (10-200 mg/L) and adsorbent dosage (0.5-8 gr/L). All experiments were carried out in 250 ml flask Erlenmeyer. The 100 mL of p-cresol solution was mixed with certain amount adsorbent. The pH was regulated by 0.1 N HCl or 0.1 N NaOH solutions. The flasks containing samples were shaked for certain contact time at 120-125 rpm and 30 °C by an electrically thermostated reciprocating shaker, LSI-3016R, Daihan LabTech Co, Ltd, Korea. The samples were centrifuged at 3600 rpm for 10 min. finally. The residues concentration of p-cresol and 2-chlorophenol in samples was measured Japan at the maximum wavelength of 280 and 274 nm by DR-2800 spectrophotometer, Hitachi, Tokyo, Japan.

The removal efficiency (R) and adsorption capacity (q_e) is calculated by following equation:

$$q_e = \frac{(C_e - C_0)V}{M} \quad (1)$$

$$R = \frac{C_e - C_0}{C_0} \times 100 \quad (2)$$

Where qe is the adsorption capacity (mg/g), C0 and Ce are the initial and the equilibrium concentrations of dye solution (mg/L), respectively. V is the volume of the dye solution (L), and M is the mass of the adsorbent(21-22).

Result and discussion:

The effect of contact time: This parameter has been introduced as effective parameter on adsorption process. The Fig 1 depicts the effect of contact time on p-cresol and 2-cholorophenol removal efficiency. This Fig indicated that the increasing of contact time is led to increase of removal efficiency. The p-cresol removal efficiency increases from 33.7 to 94.5 percent by increasing of contact time from 10 to 180 min, respectively. Also at this time 2-chlorophenol removal efficiency increases from 29.9 to 82.7. The contact time 75 and 90 min for -cresol and 2-cholorophenol was considered as equilibrium for further experiments. This can be explained by the greater accessibility of adsorption surface site which it provides greater adsorption chance for p-cresol removal(23, 24). Similar results have been observed in other studies which are conducted to remove the phenolic by other adsorbents (25-26).

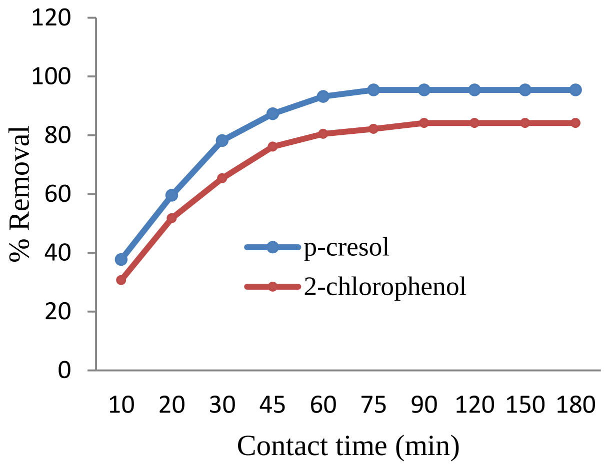


Fig 1: The effect of contact time on p-cresol and 2-cholorophenol removal efficiency by Canola

The effect of pH : The pH has identified as a critical parameter in the adsorption process. The effect of p-cresol and 2-cholorophenol removal efficiency by Canola was evaluated in various values of pH from 3 to 11 and the results are shown in Fig 2. These results reveal that the increasing of pH decreases the Phenolic compounds removal efficiency. As it can be seen in this Fig, Maximum p-cresol and 2-cholorophenol removal efficiency was detected in pH=3.. This inverse relationship between pH and phenolic removal efficiency has been obtained in various studies which conducted by other researchers (27-28). The Anbia et al conducted a study to evaluate the phenolic compound removal efficiency in adsorption process. In their study, the maximum phenols removal was obtained in pH=3 which it was exactly in accordance with the results of our study. Also, they expressed that this event can be due this fact that the adsorbent surface is positively charged in acidic pH which it is led to better interaction between the positive surface of adsorbent and the negatively charge of phenols molecules (29).

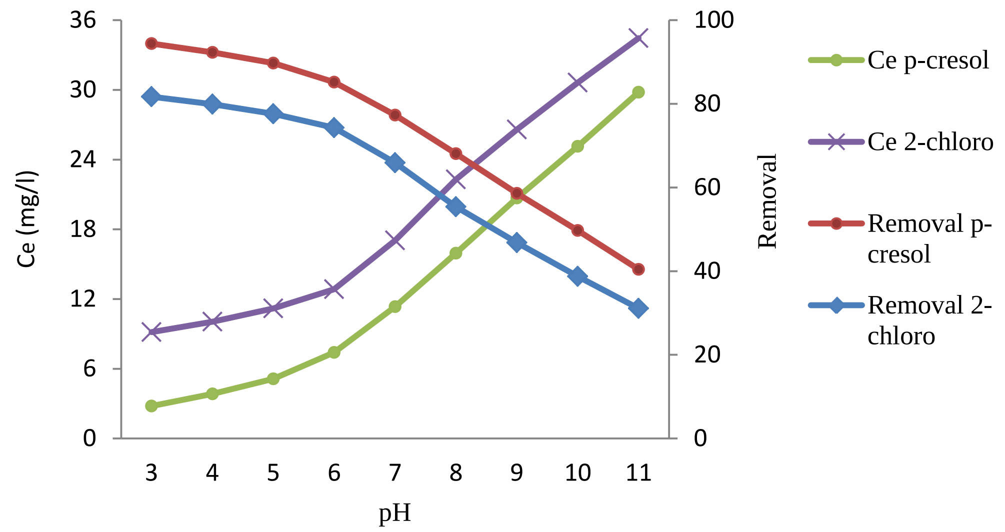


Fig 2: The effect of pH on p-cresol and 2-cholorophenol removal efficiency by Canola

The effect of initial concentration of p-cresol: The effect of initial concentration of p-cresol and 2-chlorophenol on adsorption efficiency was evaluated by varying of this parameter between 10 to 200 mg/L. the Fig 3 shows the results obtained from this study. As it is observed, the p-cresol and 2-chlorophenol removal efficiency is decreased by increasing of initial concentration. The p-cresol removal efficiency was 97.7% in p-cresol concentration of 10 mg/L but it was decreased to 72.35% in concentration of 200 mg/L. The 2-chlorophenol removal efficiency was 82.7 % in initial concentration of 10 mg/L but it was decreased to 59.1% in concentration of 200 mg/L. these results are consistent with the results of other studies on phenolic adsorption process (30-31). Also, Zazouli et al has been surveyed the ability of dried Azolla and Lemna minor for p-cresol removal and they found that the increasing of initial concentration of p-cresol is caused to diminish the removal efficiency. they has been explained that it will be due to that there is large amount of adsorbent site in lesser concentration of p-cresol which it improves the removal efficiency in these concentration(32).

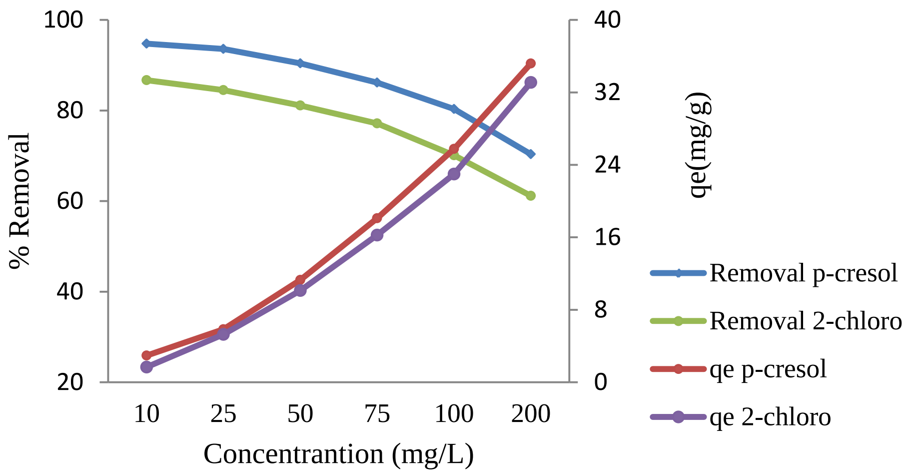


Fig 3: The effect of C_0 on p-cresol and 2-cholorophenol removal efficiency by Canola

The effect of adsorbent dose : The adsorbent dosage is another efficient variable which effects on adsorption process. This effect was studied by varying the adsorbent dosage in range of 0.5-8 g/L. the results of this study are present in Fig 4. The results reveal the significant effect of adsorbent dosage on Phenolic compounds removal efficiency. This Fig shows the p-cresol removal efficiency increase from 42.4% to 96.1% by increasing the adsorbent dosage from 0.5 to 0.8 g/L, respectively. Also for 2-chlorophenols with the same amount of adsorbent dosage removal efficiency increase from 31.9% to 82.7%. These results are in accordance with results of several other studies(33-34). Zhou et al who conducted a study to evaluate ability of natural biosorbent for adsorption of bisphenol were observed that the percentage of bisphenol removal is developed by increasing of adsorbent dosage which they implied that it can be due to greater surface area and increasing amount of available binding sites for BPA by increasing of adsorbent dosage. In addition, they were found that the adsorption capacity decreased by increasing of adsorbent dosage which it maybe because of the reduction in the amount of BPA adsorbed onto a unit mass of a sorbent with increasing sorbent mass concentration(35).

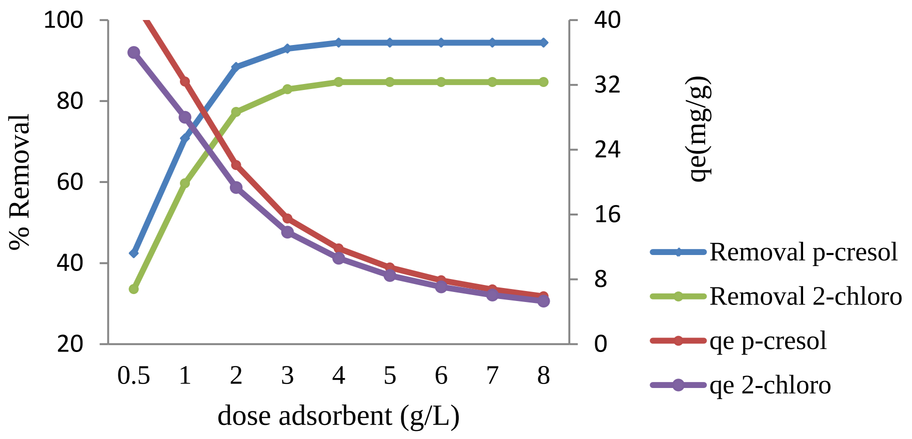


Fig 4: The effect of Adsorbent dosage on p-cresol and 2-cholorophenol removal efficiency by Canola

The isotherm studies : The experimental data obtained from the adsorption of p-cresol onto the dried canola was analyzed by isotherm models. The obtained data from this isothermic study helps to successful design of adsorption system. Four well-known isotherm models including Langmuir, Freundlich, Temkin and Dubinin–Radushkevich models were applied and their applicability for p-cresol adsorption was investigated. The equation of these models is presented in table 2.

Table 2: The equations of used isotherm model in this study(36,37)

Isotherm model	equation
Langmuier	$\frac{C_e}{q_e} = \frac{1}{q_m . K} + \frac{C_e}{q_m}$
Freundlich	$log\ q_e = log\ k_F + \frac{1}{n}\ log\ C_e$
Tekmin	$q_e = B \ln(k_t) + B \ln(C_e)$
Dubinin–Radushkevich	$q = q_0 \exp(-B\varepsilon^2)$

The calculated constants and parameter from each model is presented in table 3. The coefficient correlation (R^2) obtained from the applied model is utilized to determine the best model to describe the experimental data. it can be observed the values of R^2 for Langmuier isotherm model was obtained to be 0.989 which it is greater that R^2 values of other models which is used in this study. This indicates that the Langmuier isotherm can better described the experimental data. These results can be confirmed by the results of other studies(38).

Table 3:The adsorption isotherms constants for the for adsorption of p-cresol and 2-chlorophenol onto dried Canola

	Langmuir model			Freundlich model			Temkin model			Dubinin–Radushkevich			
	R_L	K_L	R^2	n	K_F	R^2	B	A	R^2	K	q_m	E	R^2
p-cresol	0.541	0.021	0.989	3.15	1.84	0.821	19.25	0.44	0.945	0.941	18.65	9.142	0.784
2-chloro	0.692	0.015	0.994	2.44	1.35	0.796	14.66	0.73	0.927	0.863	14.27	7.941	0.736

The kinetic studies: There widely use kinetic model e.g. the pseudo-first-order, pseudo-second-order and intra particle diffusion models were used to find the better model to describe the experimental data. The kinetic models can predict the adsorption behavior. These models are presented in table 4.

The constant and parameters of the kinetic models for adsorption of p-cresol and 2-chlolorophenol onto dried Canola is brought in Fig 5. The comparing of the R^2 related to each model reveals that the R^2 value of pseudo-second order kinetic model in all concentration is greater that other models. This means that the experimental data are best fitted on pseudo-second-order model. There are many studies to verify the results of present study (41, 42).

Table 4: The equation of the used kinetic models in this study (39-40)

Kinetic models	equation
Pseudo-first-order model	$log(q_e - q_t) = log(q_e) - \frac{k_1}{2.303} t$
Pseudo-second-order model	$\frac{t}{q_t} = \frac{1}{k_2 . q^2} + \frac{1}{q_e} t$
Intra particle diffusion model	$q_t = k_{dif}\, t^{0.5}$

Table 5:The adsorption kinetic model constants e for adsorption of p-cresol and 2-chlorophenol

Pseudo second-order model				Pseudo first-order model			Intraparticle diffusion		
C_o	k_2	R^2	q	K_1	R^2	q	K_{dif}	C	R^2
p-cresol									
50	0.0074	0.994	12.17	0.056	0.848	9.85	0.575	4.17	0.835

100	0.0097	0.997	23.95	0.041	0.839	18.75	0.849	6.89	0.846
200	0.014	0.998	35.19	0.012	0.872	28.73	0.991	9.16	0.829
2-chlorophenol									
50	0.0045	0.996	10.89	0.091	0.798	8.11	0.142	2.79	0.894
100	0.0066	0.998	21.72	0.059	0.812	14.23	0.227	4.06	0.881
200	0.0084	0.999	31.18	0.019	0.834	24.96	0.531	5.73	0.916

Conclusion: This study was to evaluate the ability of dried Canola for p-cresol and 2-chlolorophenol removal which it shows this low-cost adsorbent was successfully able to remove the Phenolic compounds. The effect of various parameters including contact time, pH, adsorbent dosage and initial concentration of Phenolic compounds was investigated and concluded that they are most effective variables on Phenolic compounds removal efficiency. The increasing of contact time and adsorbent dosage has positive effect and was led to increasing of p-cresol and 2-chlolorophenol removal efficiency. The contact time equal with 75 min and 90 min was found to consider as equilibrium time for p-cresol and 2-chlolorophenol respectively. The maximum removal efficiency was obtained in pH=3.. The isotherm and kinetic studies shows that Langmuier isotherm model and pseudo-second-order kinetic can be best models to describe the experimental data. Finally, it can be concluded that the dried Canola can be considered as cost-effective adsorbent to remove the phenolic compound.

Acknowledgements: Hereby, authors are grateful from Student Research Committee of Gom University of Medical Sciences, Iran due to supporting this research

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