NEW AGRICULTURAL WASTE ADSORBENT FOR THE REMOVAL OF PHENOL AND 4-NITRO PHENOL FROM AQUEOUS SOLUTIONS

Maha Al-Hassani,
Chem. Eng. Dept.
College of Engineering
University of Baghdad, Iraq

Nada S.Ahmedzeki,
Chem. Eng. Dept.
College of Engineering
University of Baghdad, Iraq

Haider Aljendeel
Chem. Eng. Dept.
College of Engineering
University of Baghdad, Iraq

ABSTRACT

The objective of this study is to evaluate the performance of Al-Shamplan (Ceratophyllum demersum L.) in the treatment of industrial waste water polluted with phenolic compounds. This plant is an agricultural waste material that spread in the southern part of Iraq. The effect of contact time, adsorbent dose and initial concentration of phenol and 4-Nitrophenol were investigated in a batch mode. High removal efficiency was obtained for both pollutants, 94.7% for 4-Nitrophenol and 93% for phenol. Results were analyzed to describe the adsorption process using different models for equilibrium isotherm and kinetic study. The experimental data matched the Freundlich isotherm and second order kinetics for both compounds. It was concluded that Al-Shamplan powder could be an alternative for more costly adsorbents to remove phenolic compounds from aqueous media.

Key WORDS: Agricultural waste adsorbent, Adsorption, phenolic compound, Isotherm, Kinetic model

INTRODUCTION

Phenols and their derivatives are disposed to aquatic streams by different industrial processes such as Petrochemical Industries, Petroleum Refineries, Coking, Fertilizer, Batteries, Paper, Pharmaceuticals, as well as phenolic resin industries. (Dhiraj Sud et al., 2008, Celik and Demirbas, 2005).

Phenol and its compounds are considered as very harmful pollutants (Mahvi, 2008) because they are toxic and prejudicial to the human beings and other creatures, even at

low concentrations (Entezari et al., 2003). The use of water included phenol may lead to protein retrogression, smoky colored urine, paralysis of the central nervous system (CNS) and harm to the kidney, liver and pancreas in human bodies (Girod et al., 2009). According to the recommendations of the World Health Organization (WHO), the allowed concentration of phenolic center in drink water is 0.001 mg/L (Banat et al., 2000]. Many economical methods are available for the elimination of phenols from industrial effluents, including physico chemical and biological treatment techniques (solvent extraction, chemical oxidation by ozone, ion exchange by resins, photocatalytic , electrodialysis filtration and adsorption. However, these methods are not preferable due to their high cost of investing, operating and equipping

(Ajmal et al., 2003, Cheuny et al., 2001, Dae and Young, 2005. Over the last few years, the use of adsorption to remove pollutants from aqueous solutions is a powerful technology especially using locally available adsorbents [Mahvi et al 2004]. Agricultural waste materials are cost effective and eco-friendly materials due to their unique chemical composition, availability, low cost, being renewable, and other many factors that attract many researchers toward such studies. This work is a first attempt dealing with the use of (Al-Shamplan) plant as an agricultural waste material and is a unique study on the adsorptive removal of phenolic compounds. The importance of this plant is inspired from local rather than global availability.

Al-Shamplan (Ceratophyllum demersum L.) or Shamlant, which is submersible, perennial jumble in water, dark green with intensive leave and spreads mostly in the southern area Mesan and Basrah governorates in Iraq, particularly the marches as well as in Basra to the south of Al-Kasib. Al-Shamplan plant, also grows in the industrial waste water lake indicating that the plant survives severe conditions and has high resistivity to pollution.

The unique visibility features of this plant have resulted from its fast growth and ability to spread within (10-15) days, even after elimination by different methods. For this reason there is no need to regenerate this biosorbent because of its low cost. The aim of this study is to investigate the feasibility of Al-Shamplan plant in removing phenolic compounds from aqueous solutions considering the effect of important variables.

EXPERIMENTAL WORK

Material and Methods

Al-Shamplan was collected from the marshes in the southern part of Iraq, washed with tap water several times to remove impurities, and then dried in air for many days followed by drying in an oven at 110°C for 24 hours, A coffee blender was used to get a powder sample, finally stored in a desiccator.

Procedure

Different concentrations of phenol and 4-Nitrophenol in aqueous solutions were obtained from a stock of 500 mg/L solution, prepared by dissolving a ready amount of reagent grade of phenol and 4-Nitrophenol (supplied by BDH,99%). Some properties of these components are shown in Table 1. The concentration of phenol and 4-Nitrophenol in aqueous medium was determined by measuring absorbance at wavelength of 270 nm, 320 nm respectively, using UV-Spectrophotometer (Shimadzu uv-160A).

Table 1 Physical properties of adsorbate

Adsorbate	Phenol	4-Nitrophenol
Molecular weight (g/mol)	94.1	139.1
Water solubility (g/ L_{H2O})	93	17
Molecular dimensions, (A^0)	5.76*4.17	6.84*4.17

Batch experiments

Batch adsorption experiments were performed at room temperature. The equilibrium isotherm was determined by mixing 0.5g of Al-Shamplan powder with 25 ml of phenol or 4-Nitrophenol solution in five conical flasks with different concentrations (50-200) ppm. Flasks were shaken in a shaker (type B, Baun Karlkob) at 250 rpm for 3 hrs and at pH 5.5 ensuring a state of equilibrium. Solution and adsorbents were separated by filtering through a filter paper, and then the clear filter was analyzed.

The adsorbed amount at equilibrium, q_e (mg/g) and the removal percentage (R) of phenol and 4-Nitrophenol onto Al-Shamplan was calculated according to the following equation;

$$q_e = \frac{(C_0 - C_e)V}{w} \quad \text{...(1)}$$

$$\%R = \frac{C_0 - C_e}{C_0} * 100 \quad \text{…(2)}$$

Where, C_o and C_e are the initial and equilibrium concentrations of phenol or 4-Nitrophenol (mg/L) respectively, V is the volume of solute (L), w is the weight of adsorbent used(g).

The adsorption kinetic study was investigated by adding 0.5 g of Al-Shamplan powder to 25 ml of phenol or 4-Nitrophenol solution having a concentration of 50 (mg/L). The sample was shaken and analyzed at different time intervals. The effect of adsorbent dosage(0.2-1)g and initial concentration (50-100) (mg/L).

RESULTS AND DISCUSSION

Characterization of the adsorbent

Chemical composition

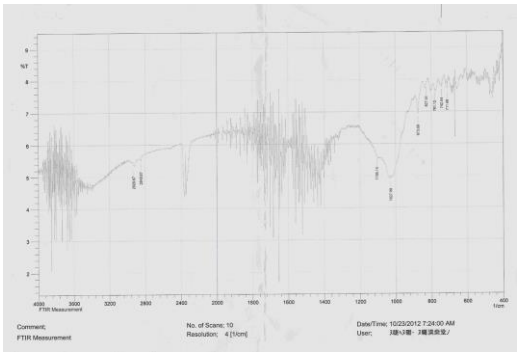
The Al-Shamplan was found to contain 50.5% Carbon, 6.3% Iron, 0.7 % Nitrogen, 0.24% Phosphor and 33 mg/L Calcium.

Surface area:

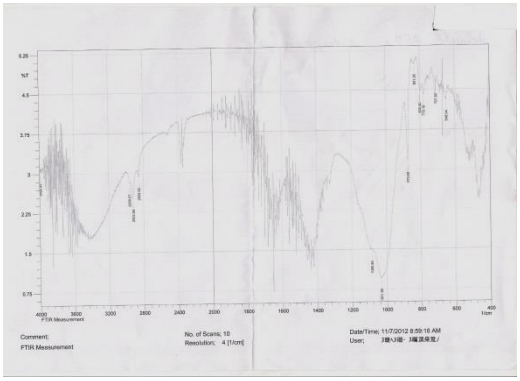
Specific surface area was found to be 3.7m^2/g. This value can be considered low when compared to activated carbon and zeolites however, it reflects porous adsorbent with no cost for preparing or purchasing. It offers a no cost separation process and green technology.

FT-IR Spectroscopy

The FT-IR technique was a specific method to identify some important functional groups such as hydroxyl and carboxyl which have effective role in adsorption efficiency [Han, et al., 2010]. The FT-IR spectrums of Al-Shamplan before and after adsorption experiments are shown in Figs. 1 using the same scale on the transmittance axis for all adsorbents. The spectra of adsorbents were measured within a range of 400-4000 cm^{-1} wave number. The FT-IR spectra of adsorbent exhibits a number of absorption peaks, indicating the complex nature of the studied adsorbates. The shift in the absorption peaks observed indicates the presence of a metal binding process taking place on the surface of Al-Shamplan. Changes can be seen in the peaks of the spectrum at less than 873cm^{-1}, which refer to the aromatic bonding C=H that appears in Figs 1.a and b. The other peak can be noticed at 2925 and 2858cm^{-1}, and this refers to the C-H group in the original carbonaceous adsorbent as it appears from the chemical analysis to contain 50% carbon. Normally this group has the spectrum of the range (2850-3000 cm^{-1}) (Rao et al 2008). Also, there is a change in the spectrum near or around 1600 cm^{-1} due to the occlusion of C=C aromatic bonding for both phenol and nitrophenol. A distinct peak is shown for O-H absorption group at 3400 and 3450 cm^{-1} which normally appears at peaks between 3200 and 3550 cm^{-1}. Finally, the N=O group at 1340cm^{-1} is seen and may be attributed to the presence of nitrophenol within the adsorbent pores.



(a)



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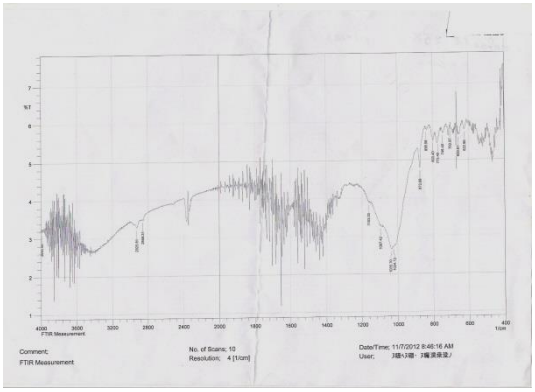


(c)

Fig.1 FT-IR spectrums of Al-Shamplan (a) Before treatment (b) after adsorption of 4-Nitrophenol (c) after adsorption of Phenol

Effect of contact time

Fig. 2 shows the effect of contact time for the adsorption of phenol and 4-Nitrophenol by Al-Shamplan adsorbent. The study of this parameter is necessary to determine the best operational condition in a continuous process of phenolic compound removal (Abdel-Ghani et al.,2009). It is clear from the figures that the removal of both pollutants increases with time and attains equilibrium in about 180 min. It is evident that the removal rate is rapid, due to the availability of active metal binding sites occupancy of these sites. This comportment suggests that in the initial case, sorption took place rapidly on the external surface of the sorbent within the first 50 min, followed by a slower internal diffusion process, which may be the rate determining step. Removal percentage equals 90% and 92% of phenol and 4-Nitrophenol respectively, after 3 hrs, at pH of 5.5, concentration of 50 mg/l and 0.5 g of Al-Shamplan. The uptake of phenol was found to be less than that of 4-nitrophenol, which can be explained by the fact that it has a lower solubility in aqueous solutions according to Table 1. However, there is a possibility that it is due to the different affinities of two phenolic species for the reactive functional groups on Al-Shamplan (Cooney, 1999). It is well known that the aromatic ring is strongly influenced by the nature of the substituent groups, and as more non-polar organic compound is, its affinity for water is lower and its adsorption affinity by the adsorbent is higher (Bada 2007).

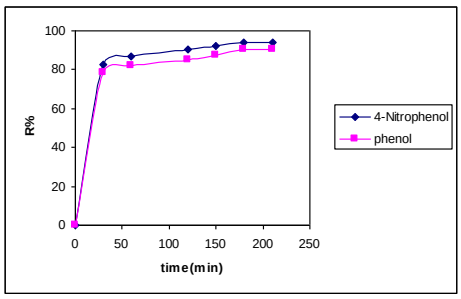


Fig.2 Effect of time on removal efficiency using Al-Shamplan as adsorbent, adsorbent dosage 0.5 g, pH 5.5, rpm 250, concentration of phenol and 4-Nitrophenol 50 ppm.

Effect of pH

The effect of pH on the uptake of phenol and 4-Nitrophenol by Al-Shamplan was investigated by adding 0.5 g of Al-Shamplan powder to 25 ml of a solution of an initial concentration of 50 ppm, and stirred at a constant speed of 250 rpm for 180 min. Initial pH value was 5.5 and 0.1 M of NaOH and HCl solutions were used for pH adjustment. Results are shown in Fig.3. The removal percentage of both phenol and 4-Nitrophenol increased slightly with the increase of pH from 3 to 5.5 and the maximum value of 90% for phenol and of 93.8% for 4-Nitrophenol were obtained, then the removal percentage decreased sharply from pH of 5.5 to pH of 11. It is well known that changes in pH values cause changes in the ionization and surface charge; and as both adsorbates have (-OH) group of negative charge, their affinity to the adsorbent surface will be higher at acidic pH ranges. Consequently, this will directly affect the uptake and ability of the host to accept guest molecules. Similar results were reported by Salame and Bondosz, 2003 and Dhidan 2012.

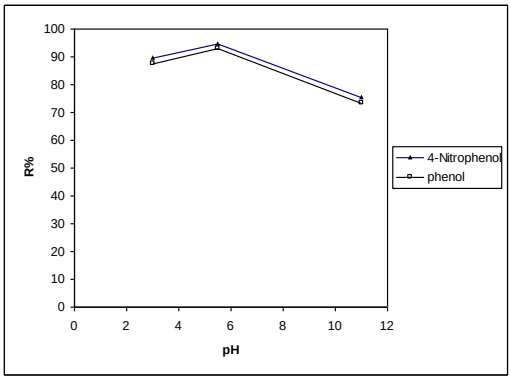


Fig.3 Effect of initial pH on the removal percentage of phenol and 4-Nitrophenol, adsorbent dosage 0.5 g, rpm 250, concentration of phenol and 4-Nitrophenol 50 mg/L.

Adsorbent dosage

The influence of the adsorbent dosage on phenol and 4-Nitrophenol removal was studied using (0.25, 0.5, 0.75, and1g), of Al-Shamplan powder in a 25 ml solution of 50 ppm as an initial feed concentration, is shown in Fig.4. It was observed that the removal percentage of phenolic compound increases proportionally with the adsorbent dosage. Increasing the amount of the adsorbent, offers more adsorption sites available and ready for higher uptake. Similar results are obtained by Ghaedi et al 2013 and , A maximum of 94.7 % and 93% of phenol and 4-Nitrophenol are removed respectively.

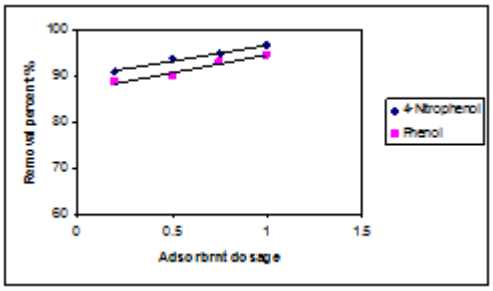


Fig.4 Effect of adsorbent dosage on removal percentage for 4-Nitrophenol and Phenol , contact time 3 hr, pH 5.5, rpm 250, conc. of phenol or 4-Nitrophenol 50 ppm

Effect of initial adsorbate concentrations

The effect of different initial concentrations of (50,100,150 and 200) mg/L with 0.5 g of adsorbent was investigated. This effect the removal percentage is shown in Fig.5. It can be seen that increasing the initial feed concentration causes the removal percentage to decrease. This can be attributed to surface saturation so that there are no available adsorption sites for extra ions at a constant amount of the adsorbent (Taghizadeh et al. 2013 and Ghaedi et al (a)). Also, the solute molecules interactions are greatly enhanced, decreasing their diffusion and hence, their adsorption (Liu et al 2004).

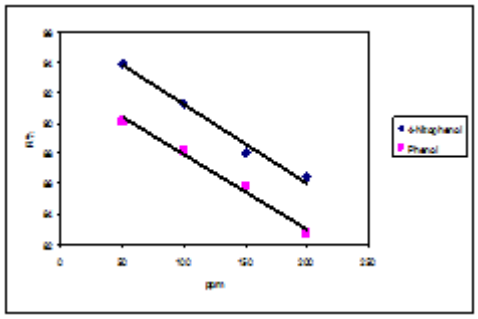


Fig.5 Removal of phenol and 4-Nitrophenol using different concentrations of adsorbate

Adsorption kinetic model

Kinetics adsorption data were investigated using the pseudo first order model (PFOM) and pseudo second order model (PSOM) [Anirudhan et al., 2009]. The linear form of PFOM equation is :

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad \ldots (3)$$

Where q_e, q_t is the amount of phenol and 4-Nitrophenol adsorbed at equilibrium(mg.g-1) and at time t (min) respectively, k_1 is the rate constant (min-1) which can be calculated from the plot of $\ln(q_e - q_t)$ versus time for different concentration of phenol and 4-Nitrophenol as shown in Fig.6(a).

The experimental amount adsorbed q_t (mg/g) at time t was calculated by the following equation

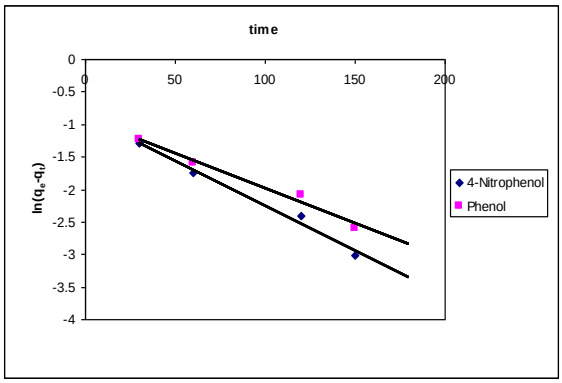
$$q_t = \frac{V(C_0 - C_t)}{m_s} \quad \ldots (4)$$

The pseudo second order model (PSOM) is given by the following equation

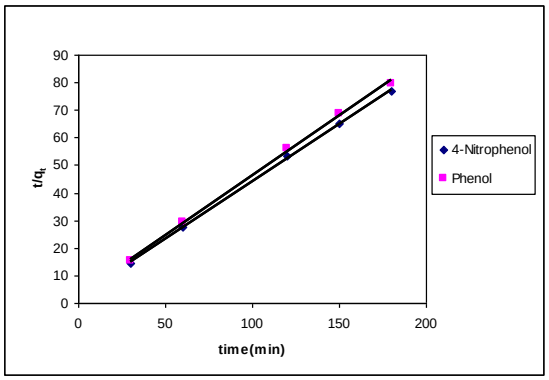
$$\frac{t}{q_t} = \frac{1}{k_2 q_e^{\,2}} + \frac{t}{q_e} \quad \ldots (5)$$

Where k_2 (g/ (mg.min))is the rate constant of PSOM.

Plots of $\frac{t}{q_t}$ against t is presented in Fig.6 (b).



(a)



(b)

Fig.6 (a) PFOM (b) PSOM plots for phenol and 4-Nitrophenol

The matched constants for two models are tabulated in Table (2) for phenol and 4-Nitrophenol. Table 2 indicates that the PSOM provides the best match as judged by its correlation coefficient. Similar behavior has been observed in the adsorption of phenol compound on beet pulp (Dursun et al., 2005).

Table 2. Rate constants and correlation coefficients for pseudo first and second order kinetic models

Kinetic model	Parameters	4-Nitro- -phenol	Phenol
PFOM	k1(min-1)	0.0138	0.0108
	q_e (mg/g)	0.417	0.405
	R12	0.9878	0.9812
PSOM	k2 (mg.g-1. min-1)	0.07	0.062
	q_e (mg/g)	2.4	2.3
	R12	0.999	0.998

Equilibrium isotherms

Langmuir and Freundlich models were used to fit the experimental data obtained by this study. The equilibrium isotherm displays a nonlinear dependence on the equilibrium concentration. The obtained adsorption data were analyzed using Freundlich and Langmuir and according to the following equations

$$q_e = kC_e^{\frac{1}{n}}$$ … (6) Freundlich

$$q_e = \frac{q_m b C_e}{1 + bC_e}$$ … (7) Langmuir

Equations 6 and 7 can be written in linear form as given below respectively

$$\ln q_e = \ln k_f + \frac{1}{n}\ln C_e$$ … (8)

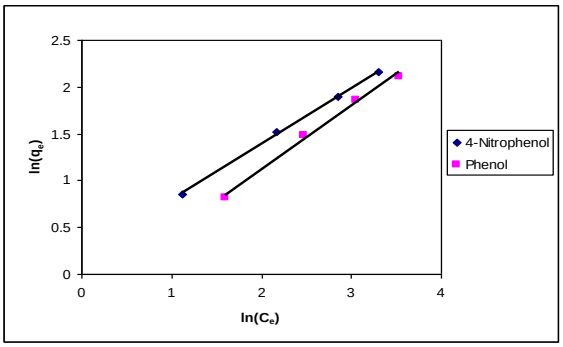
$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{q_m b}$$ … (9)

Where q_e and C_e are the equilibrium concentrations of phenol and 4-nitrophenol in adsorbed and liquid phase in mg/g and mg/L respectively , k_f and n are the Freundlich constants which can be calculated from the slope and intercepted of the linear plot of $\ln q_e$ vs. .ln C_e . q_e and b are Langmuir constants which can be calculated from the intercept and slope of the linear plot q_e vs. C_e . It is known that Langmuir model assumes that adsorption occurs in a homogenous mono layer with no interaction between molecules while Freundlich model assumes multilayer adsorption and the heterogeneity of the surface.

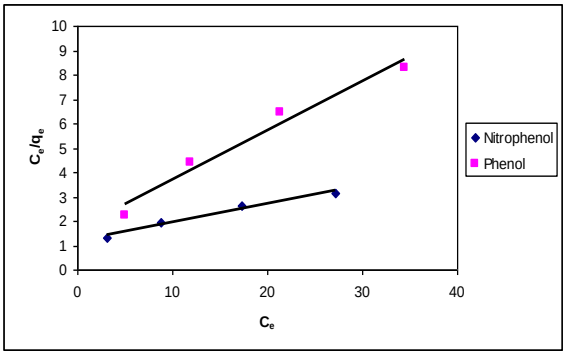
Freundlich and Langmuir isotherms are shown graphically in Figs 7(a) and (b). The matched constants for these models are tabulated in Table (3). This table indicates that Freundlich model provides the best fit as judged by its correlation coefficient for phenol and nitrphenol. The n value of Freundlich model between 1 and 10 indicates good adsorption efficiency (Treybal, 1980). This demonstrates that the developed adsorbent has good adsorption efficiency for phenol and nitrphenol removal. Calculations of the separation factor (R) were also made using the following equation:

R=1/1+bCo -----(10)

This factor equals 0.147 and 0.238 for phenol and nitro-phenol respectively, indicating favorable adsorption for both adsorbates with superiority of nitro-phenol over phenol. Comparison with some previous studies are shown in Table.4.



(a)



(b)

Fig.7 Adsorption isotherm of phenol and nitrphenol at pH (5.5) by using (a) Freundlich model (b)Langmuir

Table (3) Parameter of equilibrium isotherms

Kinetic model	Parameters	4-Nitro-phenol	Phenol
Langmuir	q_m (mg/g)	13.23	5.01
	b L/mg	0.064	0.116
	R^2	0.9796	0.9686
Freundlich	$k_f \frac{mg}{g}(\frac{L}{mg})^{\frac{1}{n}}$	1.223	0.79
	n	1.68	1.48
	R^2	0.999	0.993

Table.4 Some previous studies

4-nitophenol/Adsorbent	**Freundlich**		**Reference**
	K_f	**n**	
Phanerochaete Biomass	0.187	1.21	Wu J and Yu HQ 2006
South African coal fly ash	0.115	1.267	Potgieter et al 2009
Kaolin	0.107	1.221	Ahmedzeki et al 2013
Zeolite	0.229	1.178	
Coated sand	0.728	37.53	Asrar Al-Obaidy 2013
Phenol / Adsorbent			
Amberlite XAD-4	4.985	2.181	Lia et al. 2013
TiO2-powdered activated carbon PAC,75%P25 PPG	0.062	1.668	Tu et al 2013
TiO2-powdered activated carbon PAC, 25%P25 PPG	0.841	2.976	
Coated sand	4.265	37.07	Asrar Al-Obaidy 2013

CONCLUSIONS

It was concluded that Al-Shamplan natural plant which is locally available, could be used as an effective adsorbent for the removal of phenol and 4-Nitrophenol from waste water. The adsorption of phenol and 4-Nitrophenol on Al-Shamplan is found to be dependent on contact time**,** initial concentration and adsorbent dose. It was observed that the removal process increases with contact time and attains equilibrium at about 180 minutes. The study of kinetics of adsorption revealed that the pseudo-second order mode provides better correlation than pseudo first order model. Moreover, Freundlich model was found to fit the present experimental data with high correlation coefficient. Finally Al-Shamplan plant is not only considered as a successful and low cost adsorbent, but also as an agriculture waste plant which lives naturally and has high rate of growth that would be beneficial to remove it from river down streams and may be utilized in a useful industrial process of dual profit.

ACKNOWLEDGMENTS: The authors express their grateful appreciation to the Ministry of Science and Technology, College of Agriculture and the Petroleum Research and Development Center (PRDC)for implementing the FTIR, chemical analysis and surface area measurement.

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