



Effect of Ficus Leaf Modification and Coal to Removal P-Nitro Phenol from Aqueous Solutions

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Abstract

In present study two types of adsorbents surfaces were used to removal P-nitrophenol (P-NP) from aqueous solutions, the first adsorbent surface was prepared from the leaves and branches of the ficus tree after chemically treatment to convert into the activation carbon ficus (ACF), and the second one was a commercial coal used as adsorbent surface for comparisons, The FTIR technique was used to analysed adsorbent materials, moreover the UV-visible was used to analysis the concentration of P-NP in aqueous solutions under effect of different conditions such as acidity, contact time, initial concentrations and temperature of solution. Three adsorption kinetic models were studied, pseudo first-order, pseudo second order and diffusion rate of intra particles for the adsorption system, the results shown that the adsorption process follows the pseudo-second-order model. The adsorption isotherm parameters for the Freundlich, Langmuir and Temkin models were investigated; the obtained data showed that the Langmuir model was optimum on surface of coal, while the adsorption of P-NP on the surface ACF most fitted with a Temkin model. Thermodynamically adsorption systems on the both surfaces adsorbents were exothermic, spontaneous and random.

Keywords: Activation Carbon, Ficus, P-nitro phenol, adsorption isotherm systems, thermodynamic

1. Introduction

The aquatic environment that must be preserved is exposed to many risks, including exposure to organic pollutants such as para-nitro-phenol.

Water treatment and purification is one of the important and directly related issues to human health, and there are many methods used in this field, including sedimentation, ozonation, and adsorption with effective carbon.

The expulsion of organic materials from various sources (agricultural and household industries and the remnants of some industries such as the canning industry, fish and aquaculture production) to the bodies is one of the important factors in polluting water with pollutants. Nurse fees, they usually work to deplete oxygen from these water bodies when they are thrown in large quantities. Bacteria in the water work in changing these organic compounds into inorganic compounds, and the water is suitable, physically or

chemically, and makes the water usable directly or directly and negatively affects living organisms and affects Significant impact on society, the family and the individual.

Water is a vital requirement for humans and other living organisms, and water may be a major reason for ending life on earth if it is polluted. Water pollution is divided into two main types, the first is natural pollution and appears in a change in water temperature, an increase in salinity or an increase in suspended substances and oil spills with waste water. The other type is chemical pollution and its various forms, such as pollution with agricultural pesticides and other pollutants

A watercourse is considered polluted when the composition or condition of its water changes directly or indirectly as a result of human action, and thus its water becomes less suitable for uses in its natural state. Water pollution is also every change in the natural

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characteristics of water by adding foreign substances that cause it to disturb it or give it an odor, color or taste. Microbes may be a source of pollution. Which makes it a source of harassment or harm to the legitimate uses of life. Polluted water contains substances foreign to its natural component, which may be dissolved or suspended solids, dissolved organic or inorganic materials, or fine materials such as bacteria, algae or parasites, which leads to a change in its natural properties. Or chemical or biological, which makes the water unsuitable for drinking or domestic consumption, as well as not suitable for use in agriculture or industry [1].

Phenols are considered one of the most important organic solvents widely used in the fields of life and industry in particular. The main source of liquid waste is organic solvents, polluting the environment, surface water and groundwater in particular [2], the high toxicity present in the organic solvent P-NP, several studies induce a reduction in the concentration of P-NP from aqueous solutions by different adsorbents surfaces such as studied was P-NP adsorption on the carbon surface produce from Alhagi [1], thermal decomposition for a surface pine sawdust biochar [2], magnetic adsorbent [3], Carbon nanotubes their compounds [4], activated carbon fiber [5]. It also studied the removal of phenols compounds on the adsorbent surface char ash [6], the surface of adsorbent was taken from activated carbon commercial (ACS) containing carbon atoms with different structural and engineering properties and studied effect on removing P-NP from aqueous solutions [7], the increasing adsorption capacity of P-NP studied by activated carbon, which was thermally treated with N₂ [8].

This study focuses on the investigation of an efficient removal of P-NP from aqueous solutions using adsorption method with a new technique for the synthesis of adsorbent surface (activated carbon) from plant, this method should be considering a simple, novel and inexpensive in comparison with experimental results obtained at same conditions using commercial coal as adsorbent surface.

2. EXPERIMENTAL SECTION.

2.1 Chemicals and reagents

P-nitro phenol were obtained from Merck Chemicals Company, Commercial Coal brought from BDH, England, Ficus was collected from cultivated areas (Iraq), Sodium hydroxide (0.1N) NaOH (99.5%) and Sulfuric acid H₂SO₄ 97% were brought from BDH, England, Hydrochloric acid (0.1N) HCl (35-

36%) . All solutions were prepared using deionized water under atmospheric pressure.

2.2 Instrumentation

UV-Visible Spectrophotometer (Cary 300 UV-Vis), Shimadzu with 10mm quartz cell measurement of absorbance. Fourier Transform-Infrared (FTIR) Spectroscopy (Bruker Tensor 27 instrument), Germany. PH meter (HANA 211 microprocessor) instrument), Romania used for pH measurement. Shaking water bath (BS-11) has a temperature range from ambient +5°C to 100°C, Korea. Electronic balance - Adventurer for four Digit, Switzerland. Air drying oven, Wgll-125BE, China.

2.3 Procedure

2.3.1. Preparation of activated carbon as a surface adsorbent by Ficus acidification method.

In this study a novel method we developed for preparing an activated carbon Ficus (ACF) surface to be was using in adsorption process for removal of P-NP from aqueous solutions, Ficus was collected from agricultural areas, the leaves separated from the stems, washed, dried in an oven at 95°C for 6h. It is chopped with mortar, after that plant was charring by added some drops of H₂SO₄, then placed in the oven at 120°C for 24 hours followed by washing many times with deionized water to remove the acid content, finally dry, grind with mortar and using as adsorbent.

2.3.2 The titration curve of P-NP solution

For determine the calibration curve, prepared stock solution 500 mg/L of p-nitro phenol also a series of dilute solutions were prepared (3, 5, 7,10, 20, 30 mg/L), the absorbance were found using a spectrophotometer at a wavelength 348 nm, showed the figure(1a,1b) titration curve, wavelength used respectively. The amount of adsorbent and removal ratios at a given time calculated using equations (1&2) [9, 10]

$$q_e = \frac{V(C_i - C_t)}{m} \quad 1$$

$$\% \text{Removal} = \frac{(C_i - C_t)}{C_i} \times 100 \quad 2$$

Where: q_e Adsorbed P-NP (mg P-NP/g biosorbent) at time, V is the volume of solution (L), C_i is initial concentration of P-NP solution before adsorption (ppm), C_t concentration at time after adsorption (ppm), m weight of adsorbent surfaces (g) for ficus or coal, %R Removal percentage.

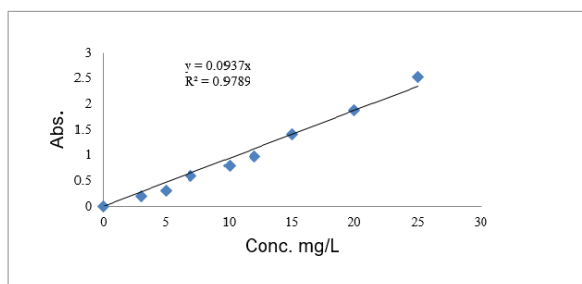


Figure 1a.: titration curve for para nitrophenol

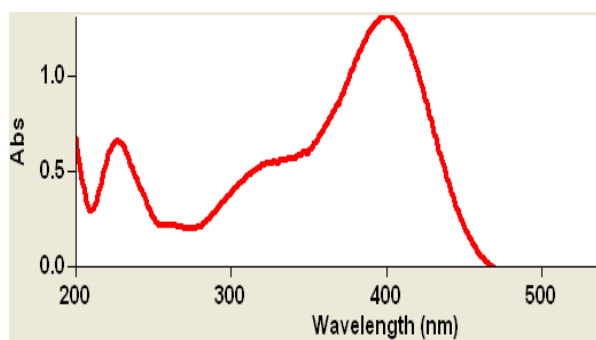


Figure 1b.: Uv-vis spectrophotometry curve of P-NP

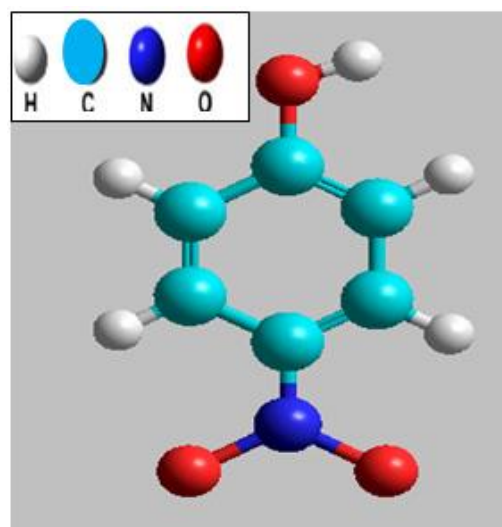


Figure 1c : Chemical Structure of Para-Nitro-Phenol

Table (1) Molecule properties of P-nitro phenol

Parameters	E HOMO	-10.1786 eV
	ELUMO	-1.070602 eV
	ΔE gap	9.107998 eV
	(Ionization potential) IE	10.1786 eV
	(Electron affinity)EA	1.070602 eV
	(chemical Hardness) η	4.55399 eV
	(Global electrophilicity index) ω	3.473452 eV
	(Eelectronegativity) χ	5.624601 eV
	(Dipole moment) μ	5.565 Debye
	(chemical softness) S	0.219587 eV ⁻¹
	(polarizability) σ	12.79
Single point energy :		-1609.4407 kcal/ mol
Thermodynamic calculation	Free energy	-42159.6 kcal/mol
	Enthalpy	-31.912592 kcal/mol
	Entropy	154.3138 kcal/mol. k

Table (2) The bond lengths of the molecule PNP

Description bond length	Bond length (A0)	Description Angle (deg.)	Angle (deg.)
C4-O7	1.3596	C4=C2-C1	119.394
O7-H15	0.9498	C5-C3=C1	119.833
C2-H12	1.096	O9-N8-O10	120.955
C4-C2	1.404	C1-C3-N8	120.1
C4=C6	1.4034	C15-O7-H4	108.477
C1=C2	1.384	C12=C2-H1	120.281
C3-N8	1.4922		

RESULTS AND DISCUSSION

3.1 Fourier Transform Infrared Analysis

An FTIR spectrum used apparent Figure (3a, 3b) and Table (3) measure spectra for functional groups on the adsorbent solid after and before adsorption of process [6] was use of wave number range 600-4000 cm^{-1} .

show the FTIR spectra the activation groups on the surfaces solid for coal and ACF, if the appears absorption band at a range (2685-2500) cm^{-1} represents the stretching vibration for alkyl groups (C-H) and is attributed bonding C-H of aliphatic compounds, The wave number 1750-1650 cm^{-1} gives an indication of the presence of the C=O carboxylic groups. Band at range 1550-650 cm^{-1} due to stretch O-H, S = O [1, 2, 25, 28].

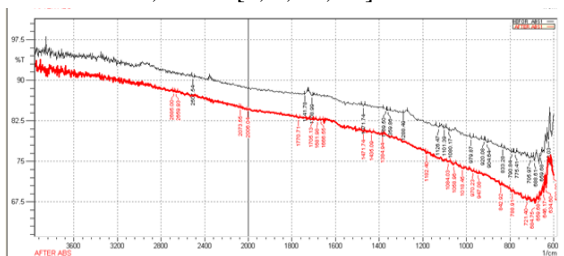


Figure 3a: FTIR spectrum adsorbent surface before and after adsorption of commercial coal

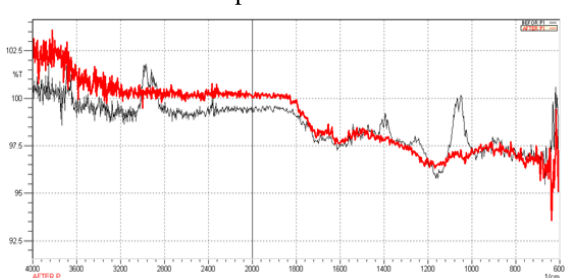


Figure 3(b): FTIR spectrum of adsorbent surface before and after adsorption (ACF)

Table (3) Fourier transforms infrared (FTIR) band assignments of adsorbents surfaces

Surfaces adsorbents	Assignment	Wave number (cm^{-1})	Wave number (cm^{-1})
Plant modified (ACF)	C = O stretching	1750	
	C = C stretching	1650	
	C – O stretching	1150	
	C – O, S = O	1050	
	C – H bending	650	
Coal	C – H stretching	2685	2507
	N = C =	2073	2006
	S stretching	1705	1741
	C = O stretching	1471	1471
	O – H, C	1435-	1369-
	– H bending	1384	1359
	Aromatic rings	684-	688-659
	C = C bending	659	

3.2 Parameters affecting the adsorption process for P-NP

3.2.1 The Effect of pH

The pH effect of the strongly on the sites effective present in both the adsorbate material P-NP and the adsorbent surfaces of (ACF) and coal that affect on the efficiency of adsorption processes, also affect on the ionization of the adsorbate material, it is known that heterogeneous surfaces contain active sites with a positive or negative charge, as that the increase and decrease of the PH values lead to get attraction or repulsion of electrostatic force between the ions of adsorbent/ adsorbate [11]. It can be seen Figure (4), that the percentage of removal decreases with the increase of the acidic function, When it is valuable PH 3, the removal efficiency ratio are high, due to the increase in the electrostatic attraction force between for the molecular P-NP and the adsorbent surface that negative charge, causing an increase in the electrostatic attraction force between the P-NP for aqueous solution and the (ACF) also coal, while that pH 9 have been electrostatic repulsion force between for the molecule P-NP and the adsorbent surfaces therefore the capacity adsorption is weak [12].

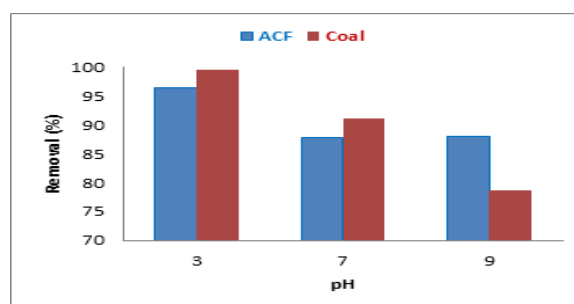


Figure 4.: Effect of pH for percentage of removal P-NP from aqueous solutions on surfaces adsorbents (ACF) and coal.

3.2.2 The Effect of contact time

Contact time means the time required to reach equilibrium between the aqueous solution of P-NP and the adsorbents surfaces [13]. The apparent figure (5) was percentage of removal against the contact time for the aqueous solution of P-NP and the adsorbents surfaces at 25°C, pH 3, at initial concentration 10 mg/L and dosage 6.7g/L.

It was found from the experimental values at the equilibrium time of removal P-NP from aqueous solutions with the ACF surface is 40 minute, while to be the equilibrium time of the P-NP adsorption with the surface coal is 10 minutes.

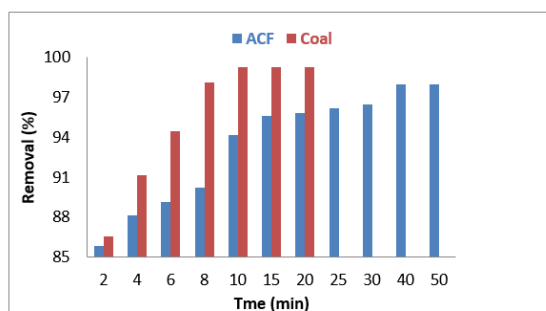


Figure 5.: Effect of contact time for percentage of removal P-NP from aqueous solutions on surfaces adsorbents (ACF) and coal.

3.2.3 Effect of initial concentration for P-NP

Apparent of Fig. (6) The removal P-NP from aqueous solutions on the biosorbents surfaces ACF and coal at initial concentrations (10, 20, 30, 40, 50) mg/L, pH = 3, and the adsorption dosage of 6.7 g/L in equilibrium time at the temperature of 298.15K. The adsorption process increases with the initial concentration of P-NP solution increases were the adsorption capacity is highly dependent on initial concentration of the P-NP due to the lower the driving force between for the molecules adsorbate P-NP on the adsorbent surface there is a tendency in the P-NP molecule that transfer from the solid phase to the liquid phase [14], that best result of the initial concentration for P-NP solution with solid surface of ACF range (10 to 20) mg/L while surface coal be initial concentration 10mg/L because increased interaction between adsorbate P-NP solution and adsorbent solid, also observed a decrease in the efficiency adsorption at high initial concentrations due to the solid surface area adsorbent is a low or insufficient to absorption the adsorbate molecule P-NP [15].

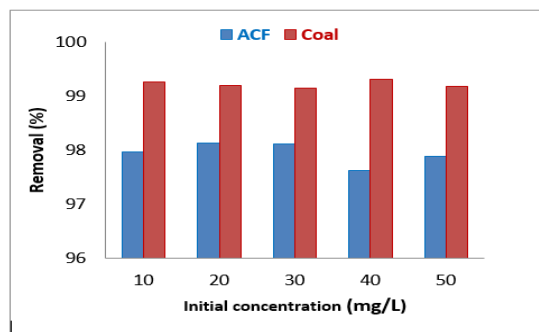


Figure 6.: Effect of initial concentration to removal P-NP from aqueous solutions

3.2.4 The Effect of temperature

The temperatures affect on adsorption process also the nature both of adsorbate of substance and surfaces adsorbent, the decreases percentage of removal P-NP from aqueous solutions on surfaces of adsorbents with increasing temperature [9], as showed in the figure (7) discussed data that P-NP has better adsorption at low temperature, also adsorption process are exothermic unless occurs of absorption process or diffusion within pores on the adsorbent surfaces [17], which that affects the interactions the between the adsorbents surfaces and the particles of the adsorbate, the temperatures of high causes increase the kinetic energy of molecules P-NP adsorbate on the surfaces adsorbents, leading to their separation of molecules from the adsorbents surfaces and their return to the solution[11,16].

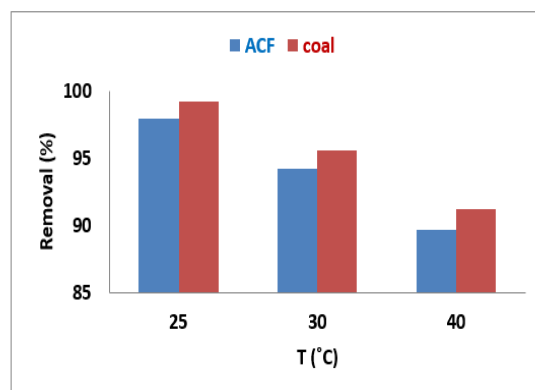


Figure 7.: Effect of temperature on removal P-NP from aqueous solutions

3.3 Adsorption Kinetic

The kinetic adsorption process is the rate of change in the amount for adsorbate material on the surfaces of the adsorbents over time, until reaching a state of equilibrium. The kinetic adsorption systems explain of the mechanics for course of adsorption reactions, and those systems are pseudo-first order reaction, the pseudo-second order reaction and the intraparticle diffusion reaction. The linear relationship of the pseudo first order lagergren equation (3) [18].

$$\ln(q_e - q_t) = \ln q_e - K_1 t \quad (3)$$

Where q_t , q_e (mg/g) are the values of the quantity of adsorbate P-NP per mass of a biosorbent at time t and at time $= \infty$, respectively. k_1 (min^{-1}) the adsorption rate constant of the pseudo first order, can be found the value q_e , k_1 from slope and the intercept when taking an plot $\ln(q_e - q_t)$ opposite t , as show in Fig. (8a). the pseudo second order equation can be expressed as (4) [19].

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e} \quad 4$$

Where k_2 ($\text{g} \cdot \text{mg}^{-1} \cdot \text{min}^{-1}$) the adsorption rate constant of the pseudo second order, and that the both are (q_e , t) can be found for slope and intercept respectively, by diagram between $\frac{t}{q_t}$ and time [20], as show in Fig. (8b). the values of the correlation coefficient (R^2) for the pseudo first order of the kinetic equilibrium model are small also, the values of q_e and k_1 are negative. This makes the kinetic adsorption do not follow the law first of the pseudo adsorption velocity rate, While the values of R^2 are higher for the pseudo-second order, the kinetic model follows the rate of the pseudo-second order. The diffusion rate intra particles are determined by the following mathematical formula (5) [9].

$$q_t = K_p t^{1/2} + C \quad 5$$

Where K_p is the diffusion rate constant intra particles, $t^{1/2}$ the half-life time of the adsorption process, C is a constant that is the thickness of the layer and find from the intercept. The adsorption process does not follow the law of diffusion rate inside the particles due to the fact that the linear relationship between q_t versus $t^{1/2}$ of the adsorption process for P-NP on both adsorbents surfaces does not pass the origin [15] that show in Figure (8c), the record of results in Table (4).

3.4 Adsorption isotherm

Adsorptions isotherms have been studied of removal P-NP from aqueous solutions on of the adsorbents surfaces at equilibrium. The study was the Langmuir model the nature of the interactions between the molecules P-NP and the adsorbent surface producer a homogeneous monolayer on the surface in the solution, the following mathematical relationship is to find the Langmuir equation (6) [18].

$$\frac{1}{q_e} = \frac{1}{b q_o C_e} + \frac{1}{q_o} \quad 6$$

Where q_o the maximum adsorption capacity, b equilibrium constant for Langmuir it describes the energy of adsorption, when plotting between $\frac{1}{q_e}$ versus

Table 4: represents the parameters kinetic reaction of the pseudo- first order, the pseudo second order and the rate of diffusion intraparticle

Surfaces adsorbents	Pseudo-first-order			Pseudo-second-order			Intraparticle diffusion		
	K_1 (min^{-1})	q_e ($\text{mg} \cdot \text{g}^{-1}$)	R^2	K_2 ($\text{g} \cdot \text{mg}^{-1} \cdot \text{min}^{-1}$)	q_e ($\text{mg} \cdot \text{g}^{-1}$)	R^2	K_p ($\text{mg} \cdot \text{g}^{-1} \cdot \text{min}^{-1/2}$)	C	R^2
ACF	0.079	-1.703	0.8988	0.3849	0.6738	0.9999	0.0327	1.2702	0.8602
Coal	0.412	-0.622	0.907	0.231	0.658	0.9998	0.062	1.252	0.784

$\frac{1}{C_e}$ take up the slope and the intercept obtain on constants Langmuir (b , q_o), the figure (9a) show of the Langmuir model, the R_L is separation factor describe the behaviour of the adsorption processes which can be obtain from the following relationship (7) [12, 20].

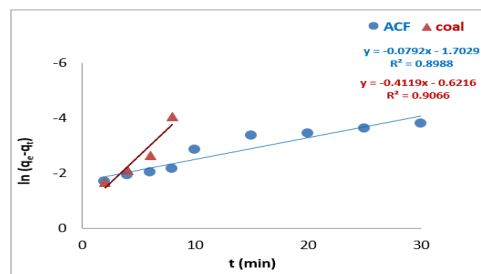


Figure 8a. the kinetic adsorption of the pseudo first order for P-NP of aqueous solutions on the surfaces of the ACF and coal

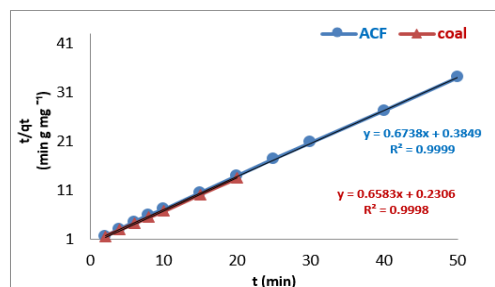


Figure 8b. the kinetic adsorption of the pseudo second order for P-NP of aqueous solutions on the surfaces of the ACF and coal

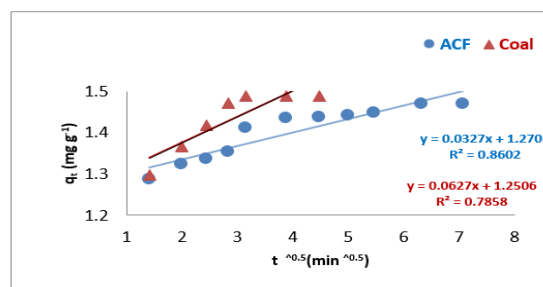


Figure 8c. the kinetic adsorption of the diffusion rate intra particles for P-NP of aqueous solutions on the surfaces of the ACF and coal

$$R_L = \frac{1}{1 + b C_e}$$

Where the values (dimensionless) $1 < R_L < 0$, It follows that the Langmuir isotherm is suitable. Describes of the Freundlich isotherms of the heterogeneous multilayer adsorption process and is not limited to only a monolayer, Freundlich equation can be expressed by taking log-linear relationship the following (8) [21, 22].

$$\text{Log } q_e = \text{Log } K + \frac{1}{n} \text{Log } C_e \quad 8$$

K is adsorption capacity constant, n a constant related to the intensity of the adsorption process for P-NP on both adsorbents surfaces and calculated by the graph $\text{Log } q_e$ versus $\text{Log } C_e$ that through slope and intercept, respectively. The figure (9b) show of the Freundlich model. Teimkin isotherms give an idea of the nature of interactions between the adsorbent surface/adsorbate material, when increasing the surface coverage resulting from the interactions between the liquid-solid phases due to a linear decrease the heat of adsorption on over all the particles in the layer [26, 27], the mathematical relationship of the Teimkin equation (9, 10, 11) [1, 16].

$$q_e = B \ln(K_T C_e) \quad 9$$

$$q_e = B \ln K_T + B \ln C_e \quad 10$$

$$B = \frac{RT}{b} \quad 11$$

Where K_T equilibrium constant explains maximum binding energy (L/g), B isotherm Temkin constant, b is heat of adsorption (J/mol), R gas constant 8.314(J.mol⁻¹.K⁻¹), T temperature of adsorption (K), it is found B, KT from the slope and intercept respectively by the graph q_e vs. $\ln C_e$, showed of the Temkin model the figure (9c), and recorded parameters for Langmuir- Freundlich -Temkin models in the Table (5). The results showed that the isothermal Langmuir model is suitable for the adsorption of P-NP on the adsorbent surface of the coal, due to the value of the correlation coefficient R² that follows the Langmuir model 0.9718 also the RL value is 0.1216 less than 1 and more than 0 [21, 25], As for the absorption of P-NP on adsorbent surface (ACF), it follows the Temkin model where the value of R² = 0.9753 appears [27, 28]. Appear values of (n) greater than 1, this indicates that the P-NP of adsorption process is a favorable physical process [21, 27], adsorption energy (b) is positive for all surfaces in the liquid phase, indicating that adsorption is exothermic [21, 25, 27].

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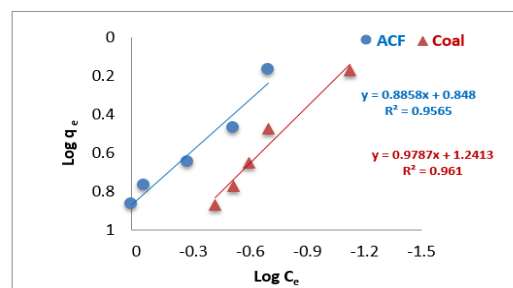


Figure 9a. The Freundlich isotherm for P-NP adsorption at pH 3 and temperature 25°C

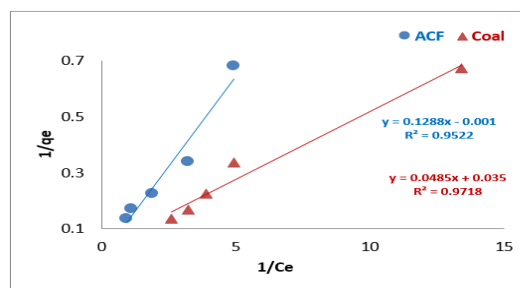


Figure 9b. The Langmuir isotherm for P-NP adsorption at pH 3 and temperature 25°C.

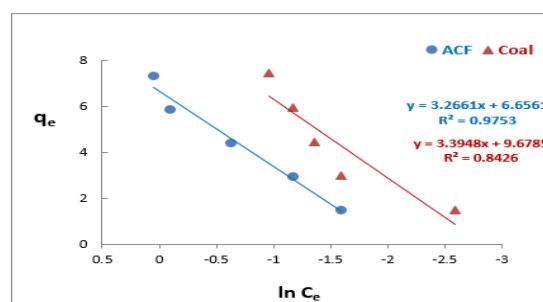


Figure 9c. The Temkin isotherm for P-NP adsorption at pH 3 and temperature 25°C

3.5 Thermodynamics of adsorption

The effect of solution temperature for removing P-NP from its aqueous solutions studied by two types of adsorbents surfaces (ACF and coal) at temperatures (298.15, 303.15, 313.15) K, the results were recorded in Table (6), Gibbs free energy (ΔG°) was calculated by the equation (12) [26].

$$\Delta G^\circ = -nRT \ln K_d \quad 12$$

Where $K_d = \frac{q_e}{C_e}$ is the distribution coefficient for adsorption (g/L) or equilibrium constant, R=8.314(J.mol⁻¹.K⁻¹) gas constant, T absolute temperature (K), The heat of adsorption (ΔH°) is obtained from the slope of the graph $\ln K_d$ versus $\frac{1}{T}$, as in Fig.(10), (ΔS°) can be obtained from the Gibbs equilibrium equation (13) [28].

Table 5: Adsorption isotherm parameters for the removal P-NP from aqueous solutions by adsorbents surfaces (ACF and coal)

Freundlich	biosorbents surfaces		n	k (mg/g)(mg/L)	R ²
	ACF		1.129	7.046	0.9565
Coal		1.022	17.34	0.961	
Langmuir	ACF	q _o (mg/g)	b (L/g)	R _L	R ²
		-1012.59	-0.008	1.1811	0.9522
Coal	Coal	q _o (mg/g)	b (L/g)	R _L	R ²
		28.57	0.72	0.1216	0.9718
Temkin	ACF	K _t (L/g)	b (J/mol)	R ²	
		7.67	758.60	0.9753	
Coal	Coal	17.30	729.81	0.8426	

Table 6: thermodynamic functions of P-NP adsorption on two different surfaces (ACF, and coal) at different temperatures

Parameter					
ΔG° (KJ/mol)			ΔH° (J/mol)		ΔS° (J/mol.K)
adsorbents surfaces	298.15 K	303.15 K	313.15 K		
ACF	-4.909	-2.261	-0.681	-8.3847	16.439
Coal	-7.417	-2.992	-1.164	-12.203	24.836

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad 13$$

It was found that the enthalpy values are negative, this indicates that the adsorption process of adsorbents surfaces is an exothermic process, As well as process spontaneous to be $\Delta G^\circ < 0$ and spontaneity increases with increasing temperatures, we observe that the entropy values are positive and mean that the increases system of randomness for adsorbate molecule on the adsorbents surfaces [28].

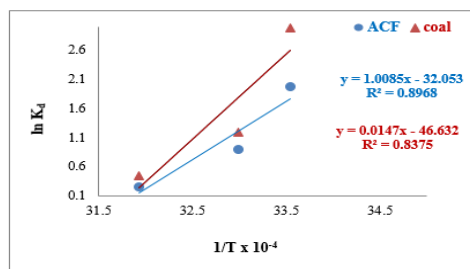


Figure 10. The adsorption isotherm of the P-NP at different temperatures

3. CONCLUSION

The aim of this study is the possibility of using the adsorbent surface ficus modified of plant (ACF) instead of coal to remove the organic compound (P-NP) from aqueous solutions, the plant surface was prepared by acidification and converted to activated carbon. The FTIR properties of a bio-adsorbent material are shown, before and after bio-adsorption of both surfaces, and the functional groups indicating their bio-adsorbent material are identified, from a

discussion of the results that apparent the following conclusions:

- The equilibrium of time for P-NP was 40 min of (ACF) adsorbent and 10 min for charcoal adsorbent with 0.1g dose at 25°C and 10ppm initial concentration P-NP.
- The best result for removal P-NP on adsorbent surface of (ACF) was about 97.97% at low temperature, pH 3, contact time 40 minute, which was better than removal P-NP on surface of coal about 99.25% at same conditions.
- The study kinetics of the bio-adsorbent on the ACF and the coal surfaces were most fitted with pseudo-second order model.
- The results showed that Langmuir isotherm model fits well with experimental data of adsorption of P-NP on the coal surface and the Temkin model on ACF surface.
- The values of the thermodynamic parameters indicated that the adsorption of P-NP on both adsorbents surfaces is an exothermic, occurs spontaneously and randomly.
- The results proven that the ACF particles can be used as adsorbents surfaces, so it is effective in removing pollutants, low cost material and environmentally friendly.

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