Synthesis of Activated Carbon from Eichhronia Crassipes Plant as Adsorbent for the Removal of Phenol from Aqueous Solution

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Abstract

For a friendly environment, adsorbents are prepared from harmful natural sources to reduce their impact on the environment. The preparation of activated carbon from the Eichhornia crassipes was done by carbonization at 450C°, followed by activation step at 800C°. The adsorption processes were carried out by the batch system to determine the optimal conditions for the removal of phenol from the aqueous solutions. Different parameters were used in batch experiments such as initial concentrations of phenol (in the range from 10-100 ppm), pH (from 2-12), temperature (from 298-328k), AC dosage (from 0.09-0.4g). All the adsorption experiments were carried out in thermostat shaker at 150 rpm. The removal percentage of phenol on activated carbon was 98.2% at optimum conditions of 10 ppm initial phenol concentration, 25 minutes, pH=7, 308 k, 0.2 g of AC dosage). The thermodynamic functions of phenol adsorption were studied at different temperatures. The values of Gibbs free energy (ΔG) at maximum removal with temperatures (298, 308, 318 and 328K) was (-5.17, -6.83, -8.22, -8.48) respectively and enthalpy ΔH, entropy ΔS values was 29.22 and 0.114. The activated carbon extracted from Eichhornia crassipes has a double benefit. first, to remove various organic pollutants such as phenols in aqueous solutions and the second, to create a clean water environment free of this plant found in Iraqi waters.
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Keyword: Adsorption, Phenol, Activated carbon, Eichhornia crassipes.

1.Introduction

At the present time and due to increased human activity, the pollution by organic materials became more prevalent than before. Rivers waters face significant challenges due to the discharge of pollutants to them. Sewage water and agricultural and industrial wastewater all contain organic contaminants. There are many methods can be used to remove organic pollutants in the water solutions (i.e., coagulation, flocculation, reverse osmosis, ozone, sedimentation, advanced oxidation, adsorption, ion exchange) [1]. Adsorption on solid adsorbents is one of the most efficient ways to remove organic pollutants from aqueous solutions [2]. Activated carbon (AC) is a strong adsorbent material with a high surface area containing carbon multi-porous that have high adsorption ability [3]. Activated carbon has wide applications in various areas of life [4]. It is used to remove contaminants because of the high surface area it possesses and because it contains pores of an internal micropore type [5]. Activated carbon has been prepared from several organic materials like (sugarcane, coconut husk, pistachio shell, oil palm, bituminous coal, wood, waste, cassava peel, corncob, agricultural, chicken waste, rice husk)[6]. The adsorption method considered as the most simple method that does not give by-products its materials are stable and constant. which are stable and do not decompose. Cheap and available materials were used for the preparation of activated carbon. It was found that the Eichhornia crassipes plant in the Tigris and Euphrates, where it became a major problem threatening Iraqi waters and its harmful to the environment [7,8]. Adsorption on activated carbon is a very effective way for the removing organic matter, because of the surface area it possesses. The large surface area of activated carbon comes from two methods i.e., physical and chemical method [9]. In the physical method, the raw material is heated in isolation from the air in the range of temperature from 400-500C°. The second method is the chemical activation method, which acid or basic material (e g. KOH, HCl, $ZnCl_2$, H2SO4, H_3PO_4) is added to the carbon

followed by heating to 800C° in isolation from air [10]. The aim of this work was to prepare activated carbon from Eichhornia crassipes plant to be used for the removal of phenol from aqueous solution.

2. Material and method
2.1. Activated carbon (AC) from Eichhornia crassipes.

A sample of the Nile Flower (Eichhornia crassipes) was obtained from a stream branch of the Euphrates River stream in the Husseiniya region of Karbala province in Iraq.

2.1.1. Preparation of Activated Carbon

There are two steps for preparation activated carbon as in below.

A-Carbonization.

The plant was cut into small pieces, then was washed with tap water to remove the clay and the suspended materials. Next, the small pieces are left exposed to air until dry, then the dry pieces were grinded by an electric mill into small pieces. After that, the small pieces were placed in a crucible, inside a muffle furnace free of air, and isolated atmosphere by inert nitrogen gas at 450C° for 2 hours. To complete the process of carbonization, the obtained carbon was taken and crushed using mortar.

B- Chemical Activation

The carbon was mixed with 0.1 N hydrochloric acid (1.2 ml acid to 1g carbon) and then was placed in the same isolated muffle furnace at 800C° for 2 hours to activate the carbon. The activated carbon took out from the muffler and left for a while until cools and washed with the much from distilled water. Activated carbon was taken and dried by air followed by an electric oven at 105C° for 3 hours. The activated carbon left out until it reaches room temperature to be ready to use for adsorption experiments [11].

2.2. Phenol and Chemicals

Phenol consists of an aromatic ring (aromatic hydrocarbon) and a hydroxyl function of group. It is the simplest molecule of the phenol family it was supplied by Sigma Aldrich with a purity of 99%. These molecules were selected due to its frequency in the residual waters coming from the diverse industries (petrochemical, plastic, pharmaceutical, papermaker, food-processing, etc.). The standard solution of phenol was prepared through dissolve 1g of phenol crystals in 1 liter of double distilled water. The other solution was prepared from standard solution by sequential dilution. The present study worked to removal the contaminates from raw water mainly phenolic compound.

2.3. Characterization Techniques

There are several tests were performed on activated carbon to study the surface morphology and chemical state using Scanning Electron Microscopy (SEM) (model: Inspects 50FEI company), Energy Dispersive X-ray spectroscopy (EDX), and the chemical bonds were detected by Fourier Transform Infrared (FT-IR) SHIMADZU, (IRPRESTIGE 21). Meanwhile, the particle size and surface of the activated carbon were detected by Rasterizer 2000 and N2 Sorption device, respectively.

2.4. Adsorption Studies

Adsorption experiments of phenol on activated carbon were performed by the batch experiments system to obtain optimum conditions for the removal of phenol from aqueous solution. Different initial concentrations of phenol solution were used in the range of 10-100 ppm, pH (2-12), temperature (25-55), and AC dosage (0.09-0.4g). All the adsorption experiments were carried out with thermostat shaker at 150 rpm and 25-minute contact time. The residual phenol concentration was measured by spectrophotometric (Shimadzu.PC1650 Double beam) at wavelength of 270 nm.

2.5. Calculations

The percentage removal of phenol (R%) was measured according to Eq.(1) and a measured amount of phenol adsorbed (Qe mg/g) was from Eq. (2) as in below.

$$\%R = \left(Ci - \frac{Ct}{Ci}\right) * 100 \quad .... Eq.(1)$$
$$Qe = (Ci - Ct) * \frac{V}{m} \quad .... Eq.(2)$$

Ci: initial concentration (ppm). Ct: concentration at any time(ppm). m = Mass of adsorbent (g). V is the volume of phenol solution (ml). The thermodynamic functions can be calculated from equations as in below.

kc= Qe/Ct ....Eq.(3)

ΔG= -RTlnKC ....Eq.(4)

ln KC= ΔS/R-ΔH/RT ....Eq.(5)

KC is the equilibrium partition constant, Qe is the adsorption capacity, C_t is the concentration at the time, and R is the gas constant (8.314 J K /mole), T is the temperature in Kelvin (k), Eq (5) called (vant hofe equation) represent a plot of ln KC vs. 1/T.

3 Results and Discussion
3.1.The particle Size of Adsorbents

The particles size for AC was measured using Rasterizer 2000 is one of the physical methods to know the nature of the particles size. The test samples were prepared by mixing one spoon from the powder within less than ten milliliters of distilled water and adding some materials for helping maintain the homogeneity of the mixture for a longer period. After adding the powder to the water they are mixed by the spoon to overcome clusters before entering the ultrasonic mixing where it is kept less than 10 minutes after that, a number of droplets are taken and added to the test apparatus provided that the absorption rate is not greater than 20% where the window of software showed that and the absorption capacity every closer to 20 % as the accuracy increases. The material is then analyzed by the program to obtain the results of the particle size. The particles size to the activated carbon was measured, and the particles size values were between 1.26 – 632.4 µ m as in Fig.(1).
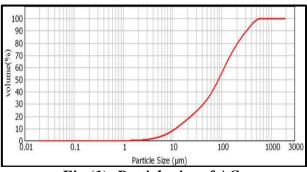

Fig.(1): Particle size of AC.

3.2. The Surface Area of the Adsorbent

The surface area and porosity of carbons are prominent factors in determining their adsorption capacities [12]. The textural properties of solids are conventionally determined from the adsorption of nitrogen at 77 K and the adsorption data are usually analyzed by the application of the BET equation [13]. The surface area of activated carbon was measured by nitrogen adsorption-desorption (N2 Sorption device) at equilibrium temperature 200 Cᵒ. The measure of AC shows the total surface area value was 360.5 m2/g. This result shows the surface area increases with the present large number of small particle and shows that the increased surface area of activated carbon was compatible with the small particle size values in Fig.(2).

3.3. Scanning Electron Microscopy

The magnified images of activated carbon were obtaining using the scanning electron microscopy (SEM). The image shows the particles sizes of activated carbon in the form of small pieces irregular shape containing rough layers in which cavities that increase from the surface area [14] as in Fig.(2).
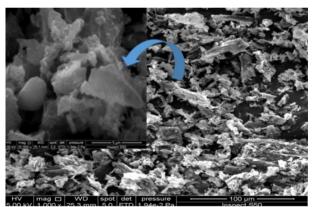

Fig.(2): The image of the activated carbon by Scanning Electron Microscope(SEM).

3.4. Energy dispersive x-ray spectroscopy (EDX).

Energy dispersive X-Ray analyzer (EDX).an examination that gives quantitative information about the elements of the material, the elements are estimated by X-rays resulting from Exposing the atom to pack of medium - power electrons. When the excited atoms relax, they emit rays in the form of photons representing atoms themselves, and these called X-rays[15].
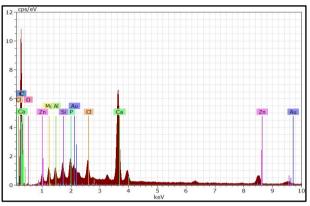
Fig.(3): EDX of the activated carbon.

There are many elements appear from EDX test of the AC adsorbent as in Fig.(3), the main elements percentage of activated carbon was recorded as in Table (1). The highest elements percentage was 66.2% to the carbon element in the AC.

Table (1)
Elements of activated carbon.

EDX of activated carbon	
Elements	ratio%
C	66.26
Sb	13.07
Ca	6.07
W	3.87
Pt	2.22
Zn	1.45
Mg	1.44
Cl	1.38
P	1.29
Si	1.16

3.5. Spectrum Analysis by Fourier Transform Infrared (FT-IR)

Fourier Transform Infrared (FT-IR) is an effective analytical method for detecting functional groups and characterizing covalent bonding information. The FTIR analysis appears many spectra of the AC before and after adsorption with phenol as in Fig.(4) and the values in Table (2). The FT-IR analysis of phenol shows characteristic absorption band at 3309, 3087, 1595, 1471, 1364, 1220, 1068, 750 and 687 cm^{-1} which can be attributed to O-H stretching, Ar. C–H stretching, C=C stretching, C–H bending, C–O asymmetrical and symmetrical stretching and out of plane bending for mono substituted benzene ring

[16]. While the spectroscopic identification of activated carbon shows bands at 3524, 2973, 1451, 1245, 1150 and 869 cm-1 for O–H stretching, C–H asymmetrical stretching, C–H bending, C–O asymmetrical stretching, C–O symmetrical stretching and C–C stretching vibration [17]. All the above spectroscopic data were disappeared when the phenol was mixed with activated carbon this indicating complete the adsorption and bound of all active sites with phenol molecules.
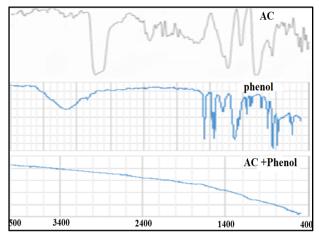

Fig.(4): FTIR spectra of the AC, phenol, and (AC +phenol).

Table (2)
FTIR values of the AC, phenol and (AC +phenol).

Activated carbon		phenol		(AC+ phenol)	
Bonds cm^{-1}	Functional group	Bonds cm^{-1}	Functional group	Bonds cm^{-1}	Functional group
3524	O – H ν	3309	O – H ν		
2973	C – H asym. ν	3087	C – H Arm. ν		
1451	C – H δ	1595	C = C ν		
1245	C – O asym. ν	1471 1364	C – H δ		
1150	C – O sym. ν	1220	C – O ν		
869	C – C ν	1068	C – O δ		
		750 & 687	γ Momo subs. Benzene ring		

4. Determination of the Optimum Conditions of Phenol Adsorption on AC
4.1. Effect of Contact Time

The adsorption of phenol was carried out in a batch system with thermostat of shaker 150 rpm at10ppm initial phenol concentration, 0.2g of AC at fixed pH=7. The phenol solution was adjusted by 0.1M of NaOH or HCl solution. Different contact time were studied (i.e. 1,3,5,10,15,25 and 30 minutes) with the number replicates was 3. Fig.(5) shows the maximum percentage of removal of phenol was 98.2% at 25 minutes. At the beginning of the reaction, the removal percentage increased, due to the number of active sites on the activated carbon was large and gradually the reaction becomes slowly because the number of active sites was decreased with increasing the contact time until reached maximum removal [18]. The 25 minutes was the optimal contact time.
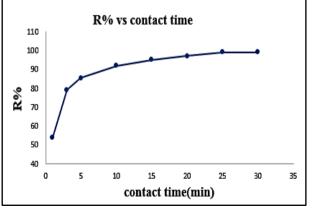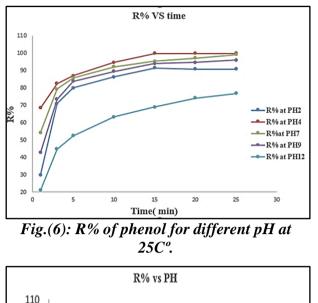

Fig.(5): R% of phenol on (AC) with the contact time.

4.2. Effect of pH on Adsorption Process

The effect of pH on adsorption of phenol on the activated carbon was studied at initial phenol concentration of 10 ppm with 0.2 g of AC as an adsorbent with different pH values (2, 4,7, 9 and 12) at fixed (temperature, contact time) respectively. The adsorption process was carried out in a batch system with thermostat of shaker150 rpm. Fig.(6) shows the percentage removal (R%) of phenol increased at pH value (from 2 to 7), after the pH=7 the percentage removal was decreased sharply to 75.7% at pH=12. The pka value of phenol is 9.89 thus, phenol has a negative charge in case of pH>7. When the pH > pH PZC = 9.7, the surface of activated carbon has a negative charge, in this case, the electrostatic repulsion increase that led decrease in the adsorption process.

However, at lower pH (2 and 4). When the pH < pHPZC = 9.7 the H+ ion will competing with the phenol onto adsorption sites on the surface of AC that leading decrease in the adsorption process [19] Fig.(7) shows the maximum percentage removal at pH=7 and pH=4 was (98.2%,98.8%) respectively. In this study, the removal of organic pollutants from water solutions, which can be applied to drinking water treatment plants. Therefore, according to the standard of drinking water, the pH values are required (from 6.5 to 8.5) [20]. So consider the pH=7 value is ideal for use, and it is close to the highest percentage removal value at pH=4.
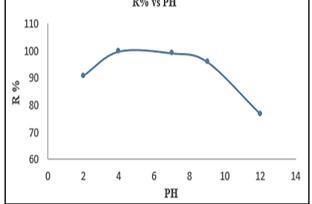
Fig.(6): R% of phenol for different pH at 25Cᵒ.

Fig.(7): Maximum R% for 25min at 25Cᵒ.

4.3. Effect of Temperature on Adsorption Process

The effect of temperature on adsorption of phenol with activated carbon was carried out in a batch system with thermostat shaker, at initial phenol concentration at 10 ppm with constant contact time at 25 min and pH=7. Different temperatures were used to estimate the effect of temperature on the adsorption process (i.e.298, 308, 318 and 328 k). The result in Figure8 show the percentage of removal increased with an increase in temperature. The suggested reason is that when the temperature increases, the kinetic energy of the phenol molecules increases, giving a stronger impetus to the entry of these molecules into the small pores in the activated carbon structure, this interpretation was according to previous studies in terms of the effect the temperatures on the phenol adsorption using activated carbon [21,22]. Several studies have also shown that phenol adsorption on the surface of activated carbon decreases with increasing temperature because

of break the bonds at the high temperatures [23]. The maximum removal efficiency of phenol was 98.8% at 328K and this result nearby to removal efficiency 98.2% at 308k. Therefore, the temperature at 308 k value is considered optimal to use.
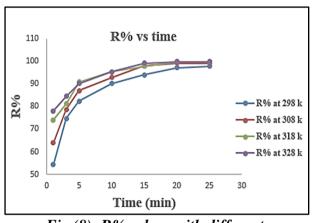

Fig.(8): R% values with different temperature.

4.4. Effect of Initial Concentrations on the Adsorption Process

The effect of a different initial concentration of phenol on the adsorption process with 0.2g of activated carbon was studied at 10, 20, 40, 60, 80, and 100 ppm with a fixed temperature of 308K, contact time of 25min and pH=7. The adsorption process was carried out in a batch system with thermostat of shaker 150 rpm. Fig.((9 shows the percentage of removal of phenol at different initial concentration. The results detect a decrease in removal percentage with increasing of initial concentration of phenol solution. This could be ascribed that in an early stage the number of vacant sites and a functional groups on the activated carbon was large and available for adsorption of phenol molecules and an increase of phenol uptake dependent on the mass driving force[24]. This force has little resistance and allows to the solutes to pass through pores on the adsorbent and gradually the number of vacant sites decreases with increasing concentration of phenol solution, thus led decrease in the R% after contact time (25min). The lower R% was 76.2% at an initial concentration of phenol 100ppm, and the maximum R% was 98.2% at initial phenol concentration of 10ppm therefore, this value as the optimum value in this study.
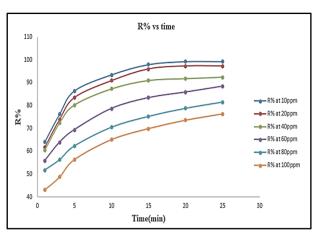
Fig.(9): R% at a different initial concentration of phenol solution.

4.5. Effect of Adsorbents Dosage on the Adsorption Process

Effect of adsorbents dosage on the adsorption process of phenol using the activated carbon was studied in a batch system with thermostat of shaker 150 rpm. The initial phenol concentration was 10 ppm, with different dosage of activated carbon (i.e. 0.09, 0.1, 0.2, 0.3, and 0.4g) and fixed temperature of 308 K, contact time 25 mint, pH=7. Fig.(10) shows the maximum removal% of phenol was 98.8% at 0.4g of AC, however, the percentage removal was 98.2% at (0.2 and 0.3g) and these values consider the best economic result. Generally, from batch experiments, the adsorption process increase gradually with increasing the dosage of adsorbent because the surface area increases with the increasing amount of AC dosage. Farther more, the number of active sites on AC and the ion transfer sites increase[25]. These factors led to increasing adsorption process and removal percentage. The results show the 0.2g value is considered as the optimal.
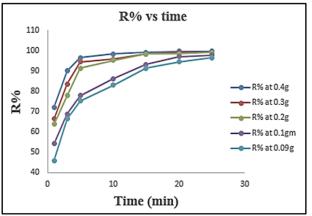

Fig.(10): R% values for different (AC) dosage at25 min.

4.6. Thermodynamic Studies of phenol Adsorption

The thermodynamic parameters were studied during the adsorption experiments of phenol on activated carbon with different temperatures and at pH=7. The thermodynamic parameters can be calculated from maximum values at 208K using equations 3, 4 and 5. When ΔG values were negative means the reaction was spontaneous. Other parameters such as (ΔH and ΔS) was calculated using the values in Table (3) with Eq 5 (Van, t Hoff equation). Fig.(11) shows the slope and intercept from the plot of ln k versus 1/T. the value of ΔG in between 0 and -20kj/mole that means the reaction is electrostatic interaction (physical adsorption), while the value of ΔG is more negative ranging from -80 to -400 kJ/mole that means the adsorption involves charge sharing (chemisorption) [26]. The values of ΔH and ΔS shows positive values, therefore, the reaction is endothermic and has (disorder), and the interaction between phenol and active sites on the surface of AC show the adsorption-type was the electrostatic (physical adsorption).

Table (3)
Thermodynamics Parameters for Adsorption of phenol on AC at pH=7.

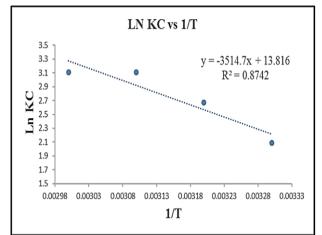| Ct ppm	Qe Mg/g	KC	Ln KC	T (Cº)	T (K)	1/T(K)	ΔG KJ/mol	ΔH KJ/mol	ΔS KJ/mol
0.3	2.42	8.066	2.08	25	298	0.0033	-5.17		
0.17	2.45	14.411	2.66	35	308	0.0032	-6.83	29.22	0.114
0.11	2.47	22.454	3.11	45	318	0.0031	-8.22		
0.11	2.47	22.454	3.11	55	328	0.003	-8.48		

Fig.(11): The Vanʾ t Hoff Plot for Adsorption of phenol on AC at pH=7.

Conclusion

The Eichhornia crassipes plant use for the preparation activated carbon at two steps. The first step is carbonization at 450C° and the second step is the activation at 800C°. The adsorption processes were carried out by the batch experiments system to determine the optimal conditions for the removal of phenol from the aqueous solutions. Different parameters were used in batch experiments such as initial concentrations of phenol (from 10-100 ppm), pH (from 2-12), temperature (from 25-55 K), AC dosage (from 0.09-0.4g). The conditions of improvement were determined by removal of phenol from aqueous solutions by activated carbon and the highest removal percentage was 98.2% under these conditions (10 ppm initial conc.,25 minutes, pH=7, 308k,0.2g of AC dosage). The thermodynamic functions of adsorption reactions were calculated at different temperatures. The results showed that the adsorption process was spontaneous due to the negative value of the free energy (ΔG) and the reaction was endothermic and it has kinetic energy (randomness) due to the positive values of (ΔH, ΔS). Eichhornia crassipes possesses a double benefit and proved high efficiency to phenol removal from aqueous solutions and at the same time, harmful plants in the aquatic environment can be eliminated.

Acknowledgment

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