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Loading and Activating a Carbon Surface and Applied for Congo Red Adsorption, Kinetic Study

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Abstract. this paper contains preparation of Active carbon surface (AC) from pro so millet grain husks and Loading and activating by Iron oxide and hydrogen peroxide sequentially to obtain surface (ACIPE). The changes of previous processes on Active carbon surface were diagnosed by Fourier transform infrared spectroscopy (FTIR) and Scanning electron microscopy (SEM). These surfaces (AC and ACIPE) were using as adsorbent for removing of congo red dye from aqueous solutions under certain conditions through batch system .More than one kinetic model was applied to congo red dye adsorption process and it was found that the most kinetic model applied to it is a model (pseudo second order model). In addition to the adsorption efficiency of the surface after activation and loading, it was much better than the main surface where the adsorption efficiency of the first and second surface was equal (27%, 85%) sequentially at higher temperatures.

Keyword: Active carbon; waste plant; Adsorption; Conge red.

1.Introduction

Industrial waste dumped in water is a serious environmental issue in the world, especially developing countries. [1-3]. The presence of many organic and inorganic pollutants in water areas by different industries stimulated the world's attention because of its harmful effects on environment human health the and [4.5].Dyes are one of the most dangerous pollutants, which are produced in large quantities in the form of liquid waste from several different industries such as textiles, paper, rubber, leather, cosmetics and food industries. The unacceptable treatment of these colored wastes of water has negative effects on living organisms in nature in addition to harmful health effects on humans [6,7]. Therefore, it is important to remove the dyes from the waste before releasing it into the environment [8]. Among these dyes is the Congo red dye (CR), an anion dye, (4aminonaphthalene-1-sulphonic acid) is a benzidine-based anionic diazo dye, which is generated by some industries, They are toxic to many organisms and suspect they are carcinogenic and mutagenic [6]. Several physical, chemical and microbial methods have been used to remove CR from colored water [7,9]. Adsorption technology is preferred among several techniques, as it is simple, easy and efficient, and it uses environmental friendly adsorbents— such as activated carbon [10], montmorillonite [11], rice husk [12], bentonite [13],

Activated carbon has a high surface area and this qualifies it to adsorb many pollutants, including organic pollutants, but it is also characterized by its high cost and inability to recover it [2,10]. Use Aloe Vera leaf shells as a cheap absorbent material to produce activated

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carbon to absorb the CR dye from industrial wastewater. Particle sizes of 300 to 500 micrometer were carbonized in an oven at $550 \degree C$ [14].

One of the recent studies included the synthesis of activated carbon by collecting leaves and trunks of the Bambusa plant. Then they were thermally and chemically treated with nitric and phosphoric acids. The properties of the resulting carbon surface have been diagnosed with several techniques (FTIR, EDX, SEM and BET). The adsorption of the methylene blue dye on the activated carbon surface has been studied due to its relatively large surface area and a high degree of porosity.[15] The aim of this study was to prepare new surface (activated carbon) from Millet crusts, loaded with oxidized iron particles and use as adsorbent surfaces of Congo red dye.

2.Experiments part

2.1. Materials and methods

2.1.1. Congo red dye

Congo red dye the sodium salt of 3,3'-(biphenyl-4,4'-diyldidiazene-2,1-diyl)bis(4-aminonaphthalene-1-sulfonate) (chemical formula : $C_{32}H_{22}N_6Na_2O_6S_2$,M.wt: 696.66 g/mol) was obtained from Merck (Darmstadt, Germany) , Used in this study as an adsorbent material without any purification. Use distilled water twice to prepare all required solutions and reagents.. chemical structure of Congo red dye was shown as 'Figure 1', Congo red [3, 9- bis dimethylaminophenazo thionium chloride], $\lambda_{max} = 498$ nm.



Figure 1. chemical structure of Dye

2.2. Preparation of activated carbon

2.2a. Iron loading on carbon surface

Millet husks were collected from a famous Iraqi market for birds (algazel market) in Baghdad, Iraq, where the crusts were washed several times from the liquefied water and distilled water to remove the dust and foreign bodies, then the crusts were burned after being wrapped with silicon sheets in a burning oven (Furnaces oven Vindon Scientific LTD, OLDHAM, England) at a temperature (500° C) for an (3) hour, Then the grinding process was done with a home mill,A certain amount of the resulting carbon surface was suspended in (400) ml of water under the influence of ultrasound and continuous stirring for a period of (90) minutes interspersed with the gradual addition of (0.5) gram of iron oxide, and then the filtering and drying process was completed in an oven (Daihan Labtech Oven , LDO-60e, south korea) at a temperature (80°C) Then grinding., the result was a carbon surface with the symbol (AC).

2.2b. Activation of loaded crbon surface

Amount of the modified carbon surface was taken with iron particales and added to (300) ml of hydrogen peroxide with constant stirring and dispersion by ultrasound for one hour and then the carbon surface It was washed with distilled water many times and then filtered and dried at a temperature (80°C) after grinding and sieving at size of 150 µm to produce the modified carbon surface, symbol (ACIPE).

2.3. Sorption Experiment of Congo red (CR)

Adsorption of the Congo red dye was carried out with an initial concentration (55 mg/L) and its factors were studied in terms of the adsorbent dose, contact time and the temperature in the batch system, which includes direct contact between the dye solution with the weight of the

surfaces(AC,ACIPE) studied in round flask (50 ml) placed in shaker water bath (Labtech ,South korea) at a thermal range (288, 298, 308, 318) K and at A period of time (5-120)min, then separated of adsorbent surface for the dye solution by a centrifuge (Hettich EBA-20,Germany) and then the absorbance of the dye solution was measured by the UV-vis spectrophotometer (Shimadzu 1800,Japan) the amount of adsorbed dye (mg/g) is calculated through the following equation:[16] $q_t=V(C_0-C_t)/m$ (1)

where q_t = the amount of solute adsorbed from the solution at time. V = Volume of the adsorbate (L), C_0 = the concentration before congo red adsorption, C_t = the concentration after Congo red adsorption and m = the weight of the adsorbent(g).

3.Result and discussion

3.1. Characterization of AC and ACIPE surfaces

3.1.1.FTIR analysis

'Figures 2, 3' shown the FTIR spectra of (AC) and (ACIPE) surfaces respectively, We noticed from the 'Figures 2,3' the presence of peaks for (ACIPE) surface after the loading and activation process more than the (AC) surface before loading and activation, which indicates the reorganization of surface oxides after being exposed to heat in high temperatures in addition to its treatment with iron oxides and hydrogen peroxide, and this leads to changes in the peaks locations, intensity and shape in addition to disappearance And the emergence of new peaks, which indicates the success of the process of loading and activation to cause changes in the carbon surfaces studied. Where we note the presence of a weak and wide peak that is within the range (3300-3000) cm⁻¹ stretch vibration returns to the (OH) group in addition to the vibration of aliphatic compounds already present in the cellulosic tissue of the carbon surface (AC), Also found at (1699 cm⁻¹) return to the (C=O) group and also the presence of a peak at (1576 cm⁻¹) returns to the bend vibration of the (NH) group for primary amine compounds with a peak at (1367cm⁻¹) returns to the vibration of the (C-O) group For alcoholic and carboxylic compounds as well, these compounds contain a peak at(1211cm⁻¹) which is due to the bend vibration of (OH) group in addition to the vibration of the group CH at frequencies within the area of (1100-550cm⁻¹) as well as the presence of weak peaks at frequencies less than (500cm⁻¹) which are due to the bend patterns of the aromatic compounds in the cellulosic tissue. These peaks give a less sharp and more homogeneous spectrum, which gives a less active characteristic in terms of functional groups, While we observe a spectrum 'Figure 2' that includes sharper and more pronounced peaks, especially those that appear at (3160 cm^{-1}) and which are due to the vibration of (OH) group of the carbon surface (ACIPE) after treatment with hydrogen peroxide and iron oxide this gives a less homogeneous spectrum which gives a more effective characteristic in terms of totals Effective functionality compared to the first surface.[17,18]



Figure 2. FTIR spectrum of AC surface



Figure 3. FTIR spectra of ACIPE surface

3.1.2.FESEM analysis

The FESEM images exhibited a description of the morphology of the surfaces of the studied materials with specific enlarging dimensions and forces (100 nm) from the figure. The surface morphologies of (AC) and (ACIPE) surfaces were observed, the FESEM image 'Figure 4 ' of the first surface shown surface particles in the form of individual longitudinal particles that are more than (200nm) intermittently interfering with each other to give Irregular clusters, this in the end gives heterogeneous surfaces, not at the level of their surfaces or at the end, which may cause a kind of obstruction in the reception of any of the materials to be loaded to it, and thus this affects his efficiency when using it in a specific field, as the 'Figure 5' (FESEM) includes On the second surface, surfaces are more flat, in addition to large cavities

On the whole, it given more uniform, homogenous and porous scarves, which greatly improves its surface properties.[19]



Figure 4. SEM micrographs of (AC)surface



Figure 5. SEM micrographs of (ACIPE)surface

3.2. Study of (CR) adsorption

3.2.1.Effect of adsorbent dose

Absorption dose is an important parameter that strongly influences the adsorption process by affecting the adsorption capacity. Therefore, the influence of adsorbent dose on (CR) dye adsorption by (AC,ACIPE) surfaces were investigated in the range of (0.01-0.05) g. We noticed from the 'Figure 6' that the greatest adsorbing amount of dye for both surfaces was at weight (0.01 g) and then after that either they are relatively stable or decrease with the increase in the weight of the adsorbed surface and this was due to more than the reason that the surfaces may have reached the level of saturation and there are no places for more adsorbed dye particles, which pushes the escape from surface to solution [20]



Figure 6. Effect of adsorbents dose on adsorption of (CR) dye

3.2.2.Effect of contact time on (CR) dye adsorption

'Figure 7 ' shown the time effect on initial concentration (55 mg/L) adsorption of (CR) dye by adsorbents (AC,ACIPE). 'Figure 7 ' shown an increase in the amount of adsorbed dye with increasing time. This increase was rapid during (10), and then the adsorption speed of the dye on both surfaces slows down to a constant amount of adsorption at the time (45 min) and (60 min) of the surfaces (AC) and (ACIPE) sequentially. This time represents The equilibriminum time for adsorption of the dye on both surfaces. This time and beyond represents the saturation area of the two surfaces with this dye. [21,22]



Figure 7. Effect of contact time on the adsorption of (CR) dye on both adsorbents

3.2.3. Effect of temperature on (CR) dye adsorption

⁶Figures 8, 9 ⁷ Explained the effect of temperature on the dye adsorption rate (CR) on adsorbents (AC, ACIPE), researched at four different temperatures (288, 298,308 and 318) K, using the initial concentration of (55 mg/L). It was observed that the removal percentage of (CR) increases with increasing temperature for both adsorbents studied. An increase in temperature increases the rate of diffusion of the adsorbate molecules across the external boundabotry layer and within the internal pores of the adsorbent particles, due to a decrease in the viscosity of the solution. The obtained results revealed that an increase in temperature from 288 to 318 K increased the (AC) surface adsorption efficiency from 16%.1 to 27% while at same range temperatures K increased the (ACIPE) surface adsorption process was of a endothermic nature. This may be due to the movement of the particles, which generally increases with increasing temperature, which facilitates the formation of mono layers.[23,24]



Figure 8. Effect of temperature on the adsorption efficiency of (CR) dye on (AC) surface



Figure 9. Effect of temperature on the adsorption efficiency of (CR) dye on (ACIPE) surface

3.2.4.Kinetic study

A study was conducted Kinetics of the (CR) dye adsorption process on both surfaces (AC,ACIPE) from the application of the most famous kinetic models (pseudo first order [25] and pseudo second order [26]) which were recorded in Tables 1,2 where they were represented graphically to obtain 'Figures 10a,b' for the pseudo first order for (AC,ACIPE) surfaces and 'Figures 11a,b' for the pseudo second order for (AC,ACIPE) surfaces and from these figures are calculated coefficients of the two orders and included in the Tables 1,2.



Figure 10.the graphical representation of first order of (CR) dye adsorption on a)AC ,b) ACIPE surfaces at different temperatures



Figure 11. the graphical representation of second order of (CR) dye adsorption on a) AC ,b) ACIPE surfaces at different temperatures

Table 1 .Kinetic parameters for adsorption of (CR) dye onto AC surface based on Pseudo (first and second) order equations

Model kinetic		Parameter	Temperature / K			
			288	298	308	318
Pseudo-first	$ln(q_e \text{-} q_t) = lnq_e \text{-} k_1 t$	$k_1(\min^{-1})$	0.0553	0.0455	0.0227	0.0254
order		q _e (mg/g)	12.9461	4.8235	3.2713	9.2202

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		\mathbf{R}^2	0.8282	0.9448	0.8217	0.8897
Pseudo- second order	$t/q_t = 1/k_2 q_e^2 + (1/q_e)t$	k ₂ (g/mg.min)	0.0020	0.0237	0.0295	0.0063
		$q_e(mg/g)$	11.976	9.727	10.964	15.723
		R^2	0.805	0.997	0.998	0.967

Table 2 .Kinetic parameters for adsorption of (CR) dye onto ACIPE surface based on Pseudo (first and second) orders equations

Model kinetic		Parameter	Temperature / K				
			288	298	308	318	
Pseudo-first order	$ln(q_e \text{-} q_t) = lnq_e \text{-} k_1 t$	$k_1(\min^{-1})$	0.0207	0.0429	0.0318	0.0165	
		q _e (mg/g)	7.3419	3.2828	4.9170	2.8688	
		\mathbf{R}^2	0.9844	0.9714	0.676	0.7561	
Pseudo- second order	$t/q_t = 1/k_2 q_e^2 + (1/q_e)t$	k ₂ (g/mg.min)	0.0121	0.0432	0.0260	0.0374	
		$q_e(mg/g)$	36.231	36.231	46.082	46.948	
		\mathbf{R}^2	0.998	0.999	0.999	0.999	

Where $q_{t(exp)}$ (mg.g⁻¹) and $q_{e(theo)}$ (mg.g⁻¹) were the adsorption capacity of dye at time t and at equilibrium, k_1 (min⁻¹) and k_2 (g.mg⁻¹.min⁻¹) are the rate constants of pseudo-first order and pseudo-second order models respectively.

From the Tables 1,2 shown the applicability of the pseudo second order equation more than the pseudo first order to the (CR) dye adsorption process for both adsorbents because the values of (R^2) for the second order were higher than their values for the first order and on the other hand the values of (q_e) (practical and theoretical) for the second rank are closer more than the The first order.[27]

4.Conclusion

In this study we concluded that the process of activating and modifying the carbon surface (AC) with iron particles and hydrogen peroxide was introduced through diagnostic tools (FESEM,FTIR) in addition to this process led to improving the adsorption efficiency of the ACIPE surface and was (85 %) while it was for the (AC) surface (27 %) and the nature of both processes was endothermic. adsorption (CR) dye follows a pseudo-second kinetics, which follows the mechanism of monolayer chemical adsorption.

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