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Effect of activation temperature on ability of activated carbon on removal of reactive yellow 145 dye from simulated industrial textile wastewaters

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ABSTRACT

The present work describes synthesis of new type of activated carbon (AC) from Iraqi berhy date palm seeds (IPDPS). This type of AC was synthesized using chemical activation method with using $ZnCl_2$ as a chemical activator. The activation process was performed at four different temperatures, 400, 500, 600, and 700 ºC. The synthesized AC was investigated using scanning electron microscopy (SEM), and Fourier transform infrared spectroscopy (FTIR). Different physical properties were undertaken such as ash content, adsorption uptake capacity, humidity, point zero charges of the AC were calculated. The activity of this material was investigated via following removal of reactive yellow dye 145 (RYD145) from simulated industrial textile wastewaters. Removal of this dye in this study was conducted by applying different reaction conditions. These conditions were the amount of the used AC, effect of pH on dye removal a, effect of temperature, and duration of time on the adsorption process. Adsorption isotherm for dye adsorption was investigated and from the obtained results it was follows Freundlich model. The activity of the synthesized AC on dye removal was compared with the non- activated carbon (NAC) that is synthesized from same date palm seeds (IPDPS. The obtained results showed that, the activity of dye removal was effected by activation temperatures of as well as the AC was more efficient in dye removal in comparison with NAC under the same conditions. Also the activity of dye removal was increased with increasing of activation temperatures as follows: AC600 > AC500>AC400>AC700.

Keywords: Activated carbons, simulated industrial wastewaters, Textile dyes removal, Iraqi berhydate palm seeds

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INTRODUCTION

Recently, high levels of pollution was reported through our environment this including pollution of air, water, and soil. Pollution with dyes and pigments seems to be most reported one. Among different types of polluted dyes, textile dyes seem to be most studied dyes. Over all the word, textile industries use wide range of these dyes such as natural and synthetic dyes, vast majority of textile dyes that are used in dyeing processes have rigid complex structures with different chromophoric groups that imparted deep colors for these dyes .Polluted effects of these dyes occurs after elution textile wastewaters from dyeing processing units into the nearest ambient environment. These eluted textile wastewaters normally have deep colors with potentially high concentrations of polluted dyes. Most of these dyes are polluted and probably toxic towards environment especially for soil and available water streams including rivers, lakes, and sometimes to the pipes of drinking waters.

Besides that, some of these can produce some carcinogenic and toxic by-products under normal atmospheric [1,2]. These textile dyes have complex structure, so that these dyes can resist normal treatment processes that have a high resistance for decomposition under normal ambient environmental conditions of air and sunlight. Therefore, it not worth to used normal treatment methods such as physical , chemical and biological treatment methods. These methods can be used to do so but there are some drawbacks for this approach such as [3,4],high cost, low removal efficiency, and sometimes these methods can produce sludge and/ or some toxic and carcinogenic by-products [5,6]. Due to these above drawbacks of traditional methods of dye removal from industrial wastewaters, adsorption

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processes seem to be the most suitable candidate method that can exhibit high removal efficiency, relatively low cost , easily processing and reproducibility of the used adsorbent for further usage [7].

Currently, many types of adsorbents are used to do this aim and among these types of adsorbents, activated carbons (ACs) can be used effectively in textile dye removal with high efficiency and low costly also this type of adsorbent can be recycled using simple activation methods which means that it can be used for another times with reasonable efficiency [8]. However, the current commercially used AC has relatively high cost, which means that use of ACs in dye removal is not suitable for mass applications. So that the key point here is the synthesis of relatively low cost types of ACs. These types of ACs can be synthesized from widely available raw materials [9]. In this way, synthesis of AC from available agricultural raw materials can be an promising alternative way towards synthesis of low cost ACs which can be used for mass applications [10-15].

The present study, describes the synthesis of AC from Iraqi berhy date palm seeds, synthesis of AC was conducted using physiochemical activation method with using $ZnCl_2$ as an chemical activator. The activation process was performed at four different temperatures 400, 500, 600 and 700 ºC. The activity of this type of AC was investigated via following removal of RY145 from the simulated industrial textile waste waters.

EXPERIMENTAL SECTION

Used textile dye model:-Simulated model of textile dye that was used in this study was reactive yellow dye -145, its molecular formula (MF: $C_{28}H_{20}ClN_9Na_4O_{16}S_5$), molecular weight of this dye MW: 1026.26g/mol, λmax for this dye in aqueous solution was 416 nm. This dye was provided by Fluka Company 98% and it was used as provided without further purification processes.

Synthesis of the activated carbon:-In this study ACs was derived from Iraqi berhy dates palm seeds, these materials are agricultural raw materials and are available widely in Iraq. Iraqi berhy date palm seeds (IBDPS) were firstly collected, washed with hot distilled water for several times to remove dust and any other wastes with these seeds. The obtained samples were dried at 110 ºC for two hours and mixed with the desired activator in appropriate ratio with $ZnCl_2$. Then these samples were filtered off and dried at 110 ºC for two hours. Then these samples were heated at 400, 500, 600 and 700 ºC under nitrogen flush in a graphite furnace for one hour for each case. Then these samples were cooled to room temperature under inert atmospheric conditions. The obtained samples were washed with distilled water to reduce pH values for the obtained samples at around 7. The final products were dried at 110 ºC for two hours to give final activated carbon. Non-activated carbon was synthesized using same IBDPS with following the same method that was used in the synthesis of AC without using a chemical activator. The obtained ACs that were activated at different temperatures will refer to as follows: AC400, AC500, AC600, and AC700.

Uptake adsorption capacity of the synthesized activated carbon:-Adsorption ability of the synthesized ACs samples was investigated using a suspension of 0.1 g of each AC type in aqueous solution of methylene blue (MB), (100 mL, 20 ppm). The obtained mixture was shacked under normal atmospheric conditions for overnight at room temperature. Then this mixture was centrifuged for sometimes to separate particles of AC fine particles . The absorbance of the obtained supernatant liquid was measured at 665 nm using UV-visible spectrophotometer. The uptake adsorption capacity of AC was investigated using a suitable calibration curve of standard solutions of MB to find the amount of the MB dye that was adsorbed on the AC. Then the uptake adsorption capacity of the synthesized ACs types was calculated by comparing these concentrations with the initial concentration of MB (20 ppm) [16].

Ash contents of the synthesized activated carbon**:-**Inorganic materials (ash content)of ACs types was investigated using a precise weighting of a required amount of carefully dried AC in a finely dried crucible. This sample burned under air conditions in a furnace at 1000 ºC for one hour under. The weight percentage of these inorganic materials for ACs types was estimated by comparing the remaining amount of material in the crucible was the initial weight of the AC. Ash content can be calculated via comparing this weigh and the initial weight of the initially taken ACs[17].Humidity of the synthesized activated carbon:-The percentage of the humidity of synthesized AC samples was investigated via subjecting (0.10 g) of dried AC under normal air conditions at the lab for a period of overnight. Then the obtained samples are weighted carefully and the percentage of humidity for each sample was calculated by comparing the weights for each sample at these two different state[18].

Fourier transform infrared spectroscopy:-The surface of AC samples was studied with FTIR spectroscopy using Perkin Elminer Spectrophotometer. FTIR analysis was recorded in the range of 450 to 4000 $cm^{-1}$with a resolution power of 1 cm^{-1} for each scan for all measured samples.

The point zero charge of activated carbons:-Point zero charges of the synthesized ACs samples were investigated by applying potentiometric titration method[19]. This method involves using 100 mL of 0.03 M KNO_3 as a blank solution and to this solution (1mL of 1M) of NaOH was added. The obtained mixture then was titrated against standard solution of nitric acid (0.10M). Also there was another mixture of 100 mL of KNO_3 with 0.10 g of ACs samples were stirred under ambient air conditions for overnight. For these two mixtures, 1.0 mL of NaOH was added and it was then titrated carefully with HNO_3.The resulting titration results were plotted as a volume of the acid against pH of the mixture and the recorded intersection point was taken to be equal to the PZC of the AC. The obtained results are presented in Table 4.

Adsorption studies:-In the present study, All adsorption studies were undertaken under normal atmospheric conditions at 15- 30 C under shaking in shaker water bath. Adsorption processes were carried out using an initial dye concentration of equal to (30 ppm, 100 mL). The used AC was loaded at different masses in a volume of 100 mL of the dye and these masses were as follows: 0.01, 0.05, 0.10, 0.15g. For each experiment, 2 mL of the reaction mixture were withdrawn periodically at each ten minutes and for duration of adsorption of one hour for all runs. Then these samples were centrifuged for several times to ensure separation of all fine particles of AC and the absorbance of the obtained supernatant liquid was measured at a wavelength of 416 nm. The obtained absorbance was recorded using Spectrophotometer Shimadzu 1650 PC-UV-visible. The percentage of dye removal (R%) was calculated using the following relationship [20-22]:

$$R\,\% = \frac{C_i - C_f}{C_i} \times 100 \qquad (1)$$

Whereas, Ci is the initial dye concentration which was 30 ppm, and C_f is the final concentration after one hour of adsorption, and the term q, refers to the concentration of the dye that were adsorbed on AC in (mg/g). The capacity of adsorption at a given time (qt) can be obtained as follows:

$$q_t = \frac{(C_i - C_t) \times v}{m} \qquad (2)$$

From above relation, C_t represents the concentration of the dye as a function of adsorption time, the term (v) refers to the volume of solution and (m) is the mass of the used AC in each case. Effect of contact time and adsorbent loading dosage:-Study the effects of duration of reaction time and adsorbent masses loading on dye removal under same applied conditions was investigated in this part. To do that, a series of experiment were undertaken using RY145 (30 ppm, 100 mL). These experiments were conducted using different contact times ranged from zero to sixty minute of reaction time. The effect of mass loading of the used AC was performed via using different masses of AC. In each mass loading same experimental conditions were applied for all set of adsorption experiment.

Effect of pH of mixture on dye removal:-The effect of pHs of the reaction mixture on the efficiency of dye removal from industrial wastewaters was investigated, and to do that, a series of experiments were conducted. Different pHs values of reaction mixture were performed , these pHs values were as follows 3, 7, and 9. These pHs values of mixture was obtained by genteelly adding of small droplets of 0.1N HCl for acidic media, and NaOH for basic media to the initial mixture of simulated industrial wastewaters of dye solutions. These pHs values of reaction mixture were recorded using pH meter .

RESULTS AND DISCUSSION

Uptake adsorption capacity of the synthesized activated carbon:-Adsorption capacity of the prepared AC was performed by the following of adsorption of methylene blue dye from the aqueous solution. The obtained results of this study are presented in Table 1 as (mg/g). These results showed that, the synthesized AC materials showed high external surface areas due to high adsorption capacity which confirms that these materials have a high porosity in their structures[23,24]. Also it can be seen that adsorption capacity was increased with increase temperature of the activating process from 400 to 600 ºC. This probably due to formation of more porous within structure of AC due to evolve some of gases and other inorganic materials with elevation in temperature. At higher temperature (700 ºC), there is reduction in adsorption capacity this probably due to occur of sintering process with elevation of temperature which leads to reduce surface area. For a comparison adsorption uptake capacity for non-activated carbon (NAC) was also undertaken besides AC under the same conditions. These results are summarized in Table 1.

Table 1

Sample of AC	NAC	AC400	AC500	AC600	AC700
Uptake capacity (mg/g)	178.4	184.2	192.2	199.4	180.5

Ash contents of the synthesized activated carbon:-Ash content of the synthesized AC for both activated at four different temperatures and non-activated carbon was performed. The presence of ash content in AC results from the existence of non-carbon materials (inorganic materials) in porous structure of these materials. These inorganic materials can be found as constituents' materials in the matrix composition of AC. It was found that presence of these residual materials in the porous structure of AC can affect negatively on the adsorption capacity of AC and therefore presence of these residual materials AC can reduce its total activity as adsorbents. In general ash content for NAC was higher than that of AC, and the ration of ash content was decreased with increase of activation temperature. This probably due to remove of inorganic materials from AC matrix with increase of activation temperature [25]. The results of ash contents for both AC and NAC are summarized in Table 2.

Table 2-Ash content percentages for the synthesized ACTable 1- Adsorption uptake capacity for the synthesized AC

Sample of AC	NAC	AC 400	AC500	AC600	AC700
Ash (%)	0.31	0.18	0.12	0.08	0.05

Humidity of the synthesized activated carbon:-Humidity in AC is related to the ability of AC to adsorb available humidity into the inner porous of the AC. The obtained results are summarized in Table 3,from the obtained results in this study it was found humidity of AC was more than that for NAC under the same ambient humidity conditions. This probably due to the presence of high porosity in AC in comparison with that of NAC due to remove of inorganic and other volatile materials from AC which provide high porosity which enable AC to uptake more humidity. This probably arise from difference in the porous structure for AC and NAC and due to high level of residual inorganic materials that may present within the porous structure of NAC sample[26,27].Also this process increase with elevation of activation temperature up to 600 ºC. At higher temperature (700 ºC), there is a reduction in humidity of AC, this probably arises from sintering processes at higher activation temperature.

Table 3-Percentage of moister content for the synthesized AC

Sample	N AC	AC400	AC500	AC600	AC700
Humidity%	24	36	40	44	32

FTIR spectroscopies of AC samples:-Functional groups of the ACs and NAC were studied using FTIR spectroscopy. Generally, FTIR spectra for both ACs and NAC samples are similar to large extent. FTIR spectra of both ACs and NAC samples showed three main peaks from 1380 to 1690 cm^{-1}.The broad peak that appears around 1690-1715 cm^{-1} can be assigned to the stretching vibration modes for C=O bonds that are present of the both AC and NAC surfaces. The other band that appeared around 1430 cm^{-1} can be related to the vibration mode of C-C bonds of the ACs surfaces [28]. The broad band that appears around 1590 cm^{-1} can be related tothe vibrations modes of aromatic rings of the surface of both ACs and NAC samples[29]. In addition to that, both of AC and NAC showed absorption band around 3000 cm^{-1} which confirms peak confirms the presence of the unsaturated alkynes C=C bond on the AC surface. The broad bands around 3300- 3650 $cm^{-1}$is related to vibration of OH groups[29]. The FTIR spectra of both AC and NAC samples are summarized in Figure 1.

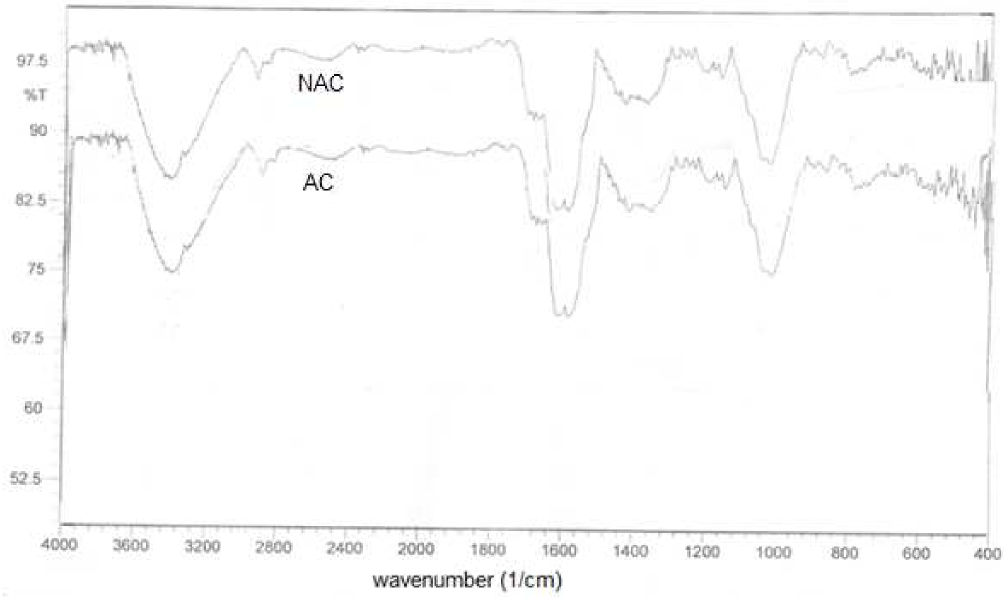

Figure 1- FTIR spectra for ACs and NAC samples that synthesized from IPDPS

The point zero charges of the activated carbon :-The point zero charges of the activated carbon (PZC) of both ACs and NAC were investigated using potentiometric method. The obtained results are shown in Table 4. From the obtained results, the PZCs values of both AC and NAC showed in almost basic value (8.10- 8.75). This indicates that these materials are showed an alkaline pH values. The PZC values of both AC and NAC are presented in Table 4.

Table 4-The point zero charges for the synthesized AC and NAC

Sample	N AC	AC400	AC500	AC600	AC700
pH of AC	8.208.30		8.40	8.60	8.75

Effect of AC loading and contact time on dye removal:- The effect of reaction time and loaded mass of the used ACs and NAC samples on the removal of RY145 from simulated industrial wastewaters were investigated via following dye removal under using different masses of the used AC under other constant reaction conditions. Also the effect of contact time on dye removal was undertaken under applying the same conditions . The obtained results are summarized in Figure 2. From these results it can be seen that, there is a increment in the efficiency of dye removal upon adsorption on AC and NAC. For all experiment batches, a shaking process for a time duration of one hour at 25 ºC under normal air conditions was applied to achieve a adsorption equilibration for all doses of the used AC [30,31]. The obtained results showed a progress development in dye removal over AC and NAC with development of adsorption time. This enhancement in dye removal with time for this case is probably due to increase of uptake adsorption capacities of the used ACs as a function of time. Comparing the ability of each type of AC in dye removal under the same applied conditions, it can be seen that, AC was more efficient than NAC in dye removal as it shown in Figures 2 and 3. This is probably arises from difference in porous structure of AC which makes it has high humidity percentage and low ash contents in comparison with NAC. In addition to that, AC has higher uptake adsorption capacity in comparison with that for NAC. These results are summarized Figures 2 and 3.

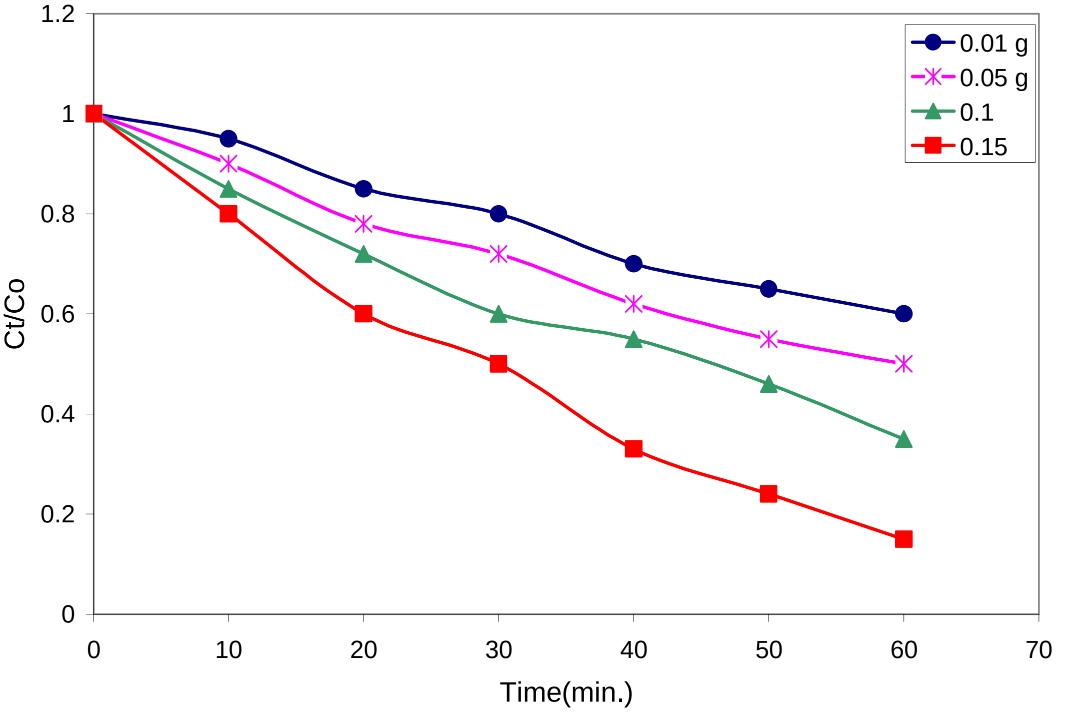

Figure 2- Effect of contact time and dosage of AC600 on the removal of RY145 dye from simulated wastewaters

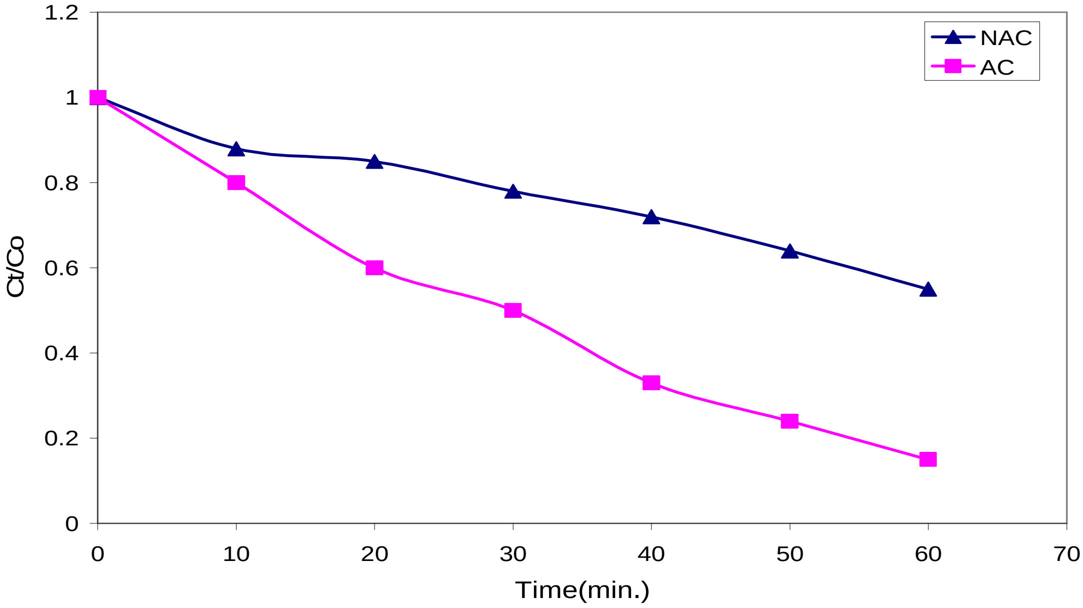

Figure 3- Comparison of activity of AC600 and NAC on RY145 removal under the same conditions

Effect of activation temperature on dye removal:-In order to investigate the effect of activation temperature on dye removal, a series of experiments were performed using same masses of activated carbon samples (AC400, AC500, AC600 and AC700) under the same reaction conditions. The obtained results are summarized in Figure 4. From these results it was found that there was enhancement in the efficiency of dye removal with increase in activation temperature of the used ACs in the order (AC600> AC500>AC400 >AC700). It is believed that increasing in activation energy from 400 to 600 ºC can lead to increase porosity of AC materials due to remove volatile and inorganic residual constituents that may present with the matrix of AC. As a consequence of that enhancement in ability of adsorption would be expected to occur. At higher activation temperature (700 ºC), sintering processes can occur which results in reduction in effective surface area of AC which leads to reduce its ability towards adsorption of dye at this activation temperature[31].Removal efficiency of RY 145 over ACs at different activation temperature are summarized in Figure 5.

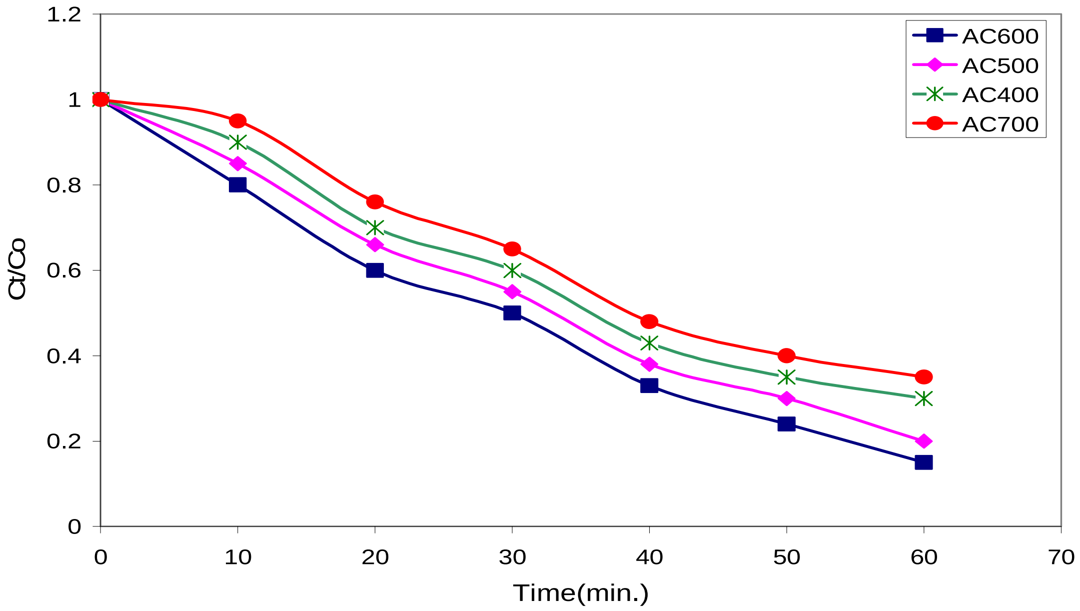

Figure 4- Effect of activation temperature of AC on dye removal

Effect of pH of reaction mixture on efficiency of dye removal: -The pH value of reaction mixture can effect on the charge of the surface and this consequently this can effect on adsorption/desorption processes on the surface. This depends on the net charge of adsorbed species on the surface and at the same on the charge of the surface of the adsorbent. Variation of pH of reaction mixture was conducted by adding controlled amount of acid and base into the reaction mixture and the reaction was performed at three pHs values 5,7, and 9. The results of pH effect on dye removal over ACs are summarized in Figure 5.

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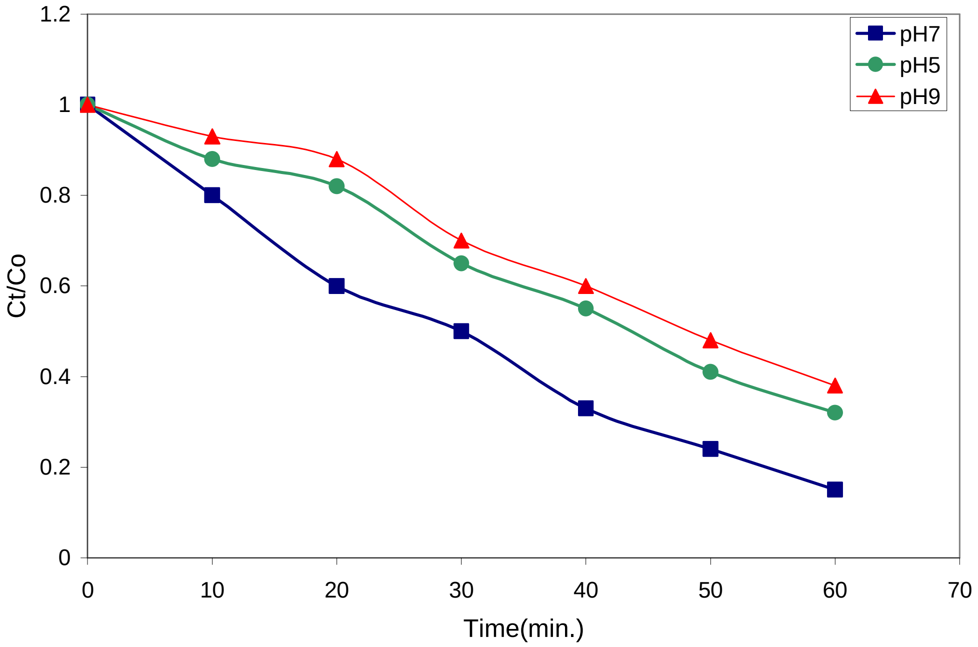

Figure 5- The effect of pH of reaction mixture on the removal of RY145dye over AC600

From above results, efficiency of RY145 dye removal over AC was varied according to pH value of reaction mixture when applying same other reaction conditions such as amount of the used ACs materials, dye concentration, volume of the solution, rate of stirring, and reaction temperature . From these results, it was found that the best removal efficiency for RY145 dye was observed at neutral media at around pH=7 which gives removal efficiency (around 85%). The lowest removal efficiency of this dye over the used AC was observed at basic media around (pH=9). This probably arises from the low adsorption ability of this dye on the surface at acidic and basic pH values as this dye has both acidic and basic groups in its structure. The presence of these groups can lead to generate arepulsion between adsorbed species (dye molecules) and the surface which reduces the efficiency of dye removal under these conditions [32,33].

Adsorption isotherms:-Adsorption isotherms for adsorption of RY145 over AC600 at 20 ºC were investigated via applying Langmuir and Freundlich isotherm models. Langmuir adsorption isotherm model is based on formation of homogeneous monolayer of the adsorbed dye molecules. For this type of isotherm, all the adsorption positions on the surface of AC are considered to be energetically equivalent to each other and there isn't any interaction between any adsorbed adjacent molecules. Freundlich adsorption model is applied for physical adsorption in this case multilayers adsorption is supposed to be occurred. Both of Langmuir and Freundlich adsorption isotherms can be explained mathematically the following relations [34-37]:

$$1/q_e = 1/q_m + 1/K_L q_m C_e \qquad (Langmuir) \qquad (3)$$

$$\log q_e = \log K_F + 1/n \log C_e \qquad (Freundlich) \qquad (4)$$

In this context, q_e refers to amount of adsorbed dye molecules on AC surface in (mg/g); q_m is refers to capacity of monolayer adsorption of the used dye in (mg/g); K_L is refers Langmuir adsorption constant in (L/mg); C_e is refers to dye concentration at equilibrium case in (mg/L); both K_f and 1/n are referred to the constants of the Freundlich adsorption isotherm. Generally, maximum adsorption capacity for AC can be estimated using Langmuir equation as mentioned above. This was performed by applying reaction conditions which include reaction temperature of temp. 20 ºC, pH 7.0, and the initial concentration of dispersed dye 30 ppm. The doses of the used AC ranged from 0.01, 0.05, 0.10 and 0.15 g. The results of adsorption isotherm constants and R^2 are summarized in Table 1 and these results are presented in Figures 6 and 7 as $1/q_e$ against $1/C_e$. From the obtained results it clear that these results were more fitted with Freundlich isotherm, which arises from high value correlation coefficients for these results. This means that the adsorption process is agree with physical adsorption and this is very promising aspect towards use AC as adsorbent to remove textile dyes from their solution. This because the used AC can be recycled easily by simple methods such as washing and thermal activation to remove adsorbed dye molecules. Then used AC can be used for further applications.

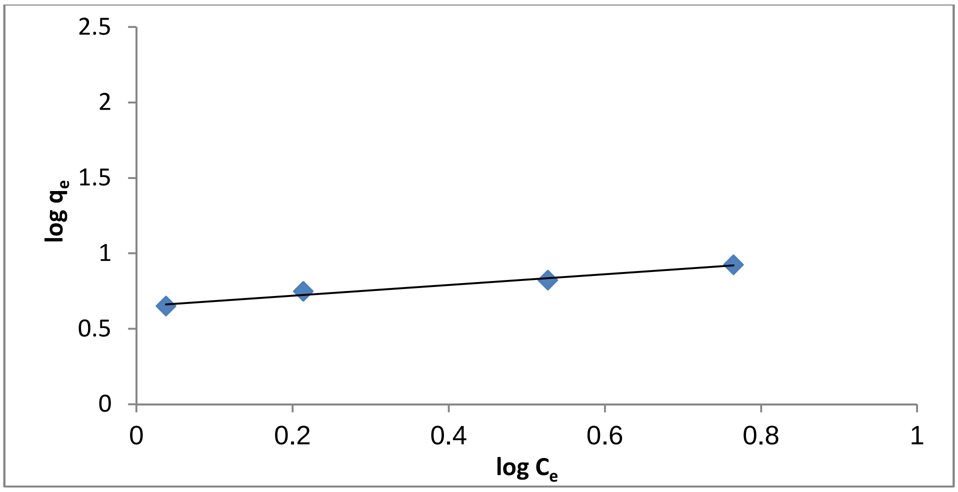

Figure 6- The linear Freundlich adsorption isotherms for RY145 (30 ppm) adsorption over AC600 at 20 ºC

Table 5-The Langmuir and Freundlich isotherm constants

Langmuir	R^2	q_m	K_L
	0.9790	8.976	0.145
Freundlich	R^2	K_F	n
	0.9810	3.884	2.915

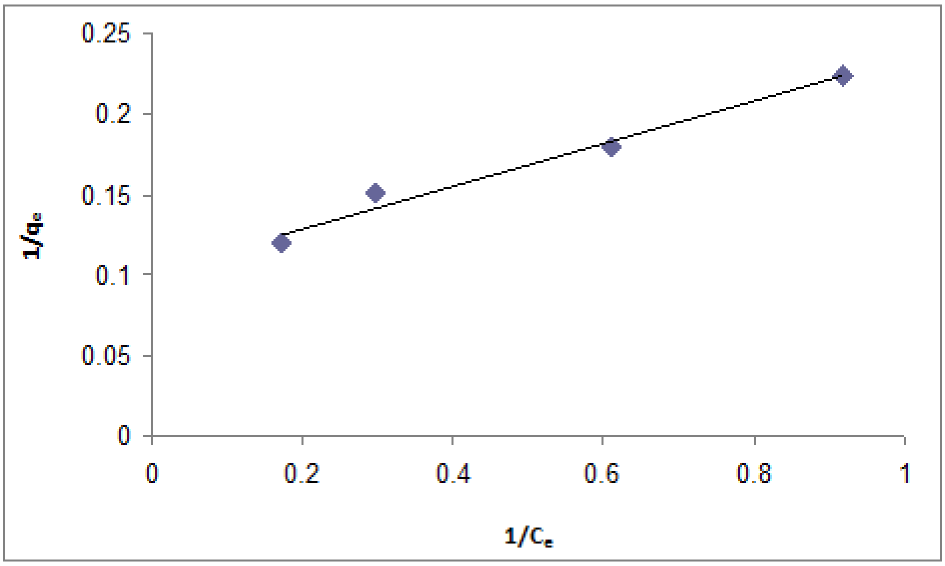

Figure 7- The linear Langmuir adsorption isotherms for RY145 (30 ppm)adsorption over AC600 at 20 °C

Adsorption thermodynamics:-Thermodynamic parameters for adsorption of RY145 over AC600 were investigated in this study. This involves calculation of each of Gibbs free energy change (ΔG°), enthalpy change (ΔH°), and entropy change ΔS° for the adsorption processes. These parameters were estimated by applying the following relationships [38-41] :

$$\Delta G^{\circ} = -RT\ln K_{ad} \qquad (6)$$

$$\ln K_{ad} = -\frac{\Delta H^{\circ}}{RT} + \frac{\Delta S^{\circ}}{R} \qquad (7)$$

From above equations, R is the general gas constant (8.314J $mol^{-1}K^{-1}$) and T is the absolute temperature in Kelvin. The thermodynamic parameters are summarized in Table 6.

Table 6- The thermodynamic parameters for adsorption of RY145on AC600

T/K	ΔG°/ k J mol^{-1}
288.15	-2.10
293.15	-3.95
298.15	-5.72
303.15	-6.95
ΔH° / kJ.mol^{-1}	58.14
ΔS^{o}/ kJ.mol^{-1}.K^{-1}	0.23

The negative values of Gibbs free energy means that adsorption of RY145 dye on the used AC was a spontaneously occurs. Generally, it can be seen that, there is a decrease in the values ofΔG° with increase in reaction temperature which indicates that adsorption processes become more efficient with elevation in temperatures[42]. The positive values of ΔH° confirm that the adsorption of RY145 dye on AC600.is an endothermic process in its nature. Figure 8 shows plot of Ln K_{ad} against 1/T for the estimation of thermodynamic parameters for adsorption of this dye over AC600.

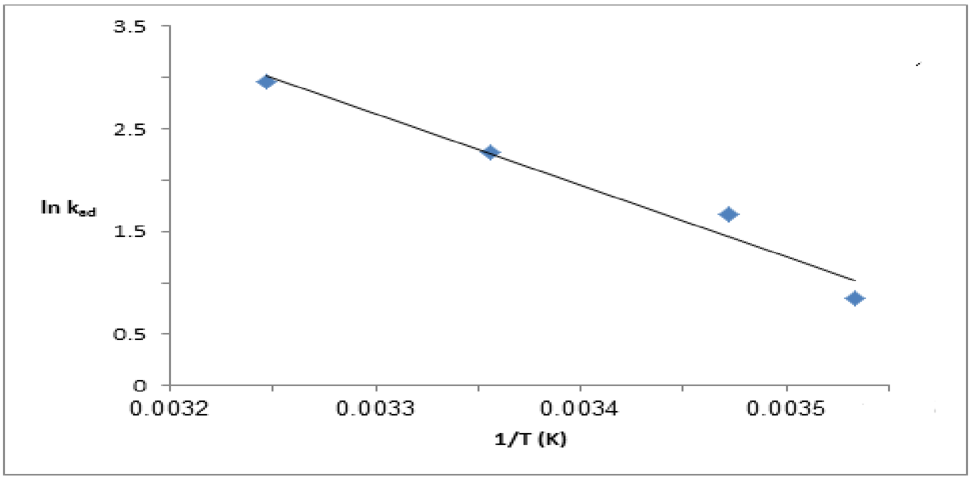

Figure8- The plot of Ln K_{ad} versus 1/T for the estimation of thermodynamic parameters

Recycle of the used activated carbon**:-** Reuse of the AC that was previously used in t removal of RY145 dye from e simulated textile wastewater was conducted using solvents to wash AC for several times with a thermal treatment. The pre-used AC600 was washed with distilled water and heated at250 ºC to remove the sticking molecules of dye. Then the obtained material was dried carefully in oven for overnight. The resultant AC was used in removal of RY145 using the same reaction conditions that were used initially and the obtained results are summarized in Figure 9. From these results it was found that the efficiency of dye removal was still efficient after three times in spite it was reduced gradually after each successive use. Generally, decrease in the efficiency of dye removal for the re-cyclized AC600 can be attributed to the reduction in the active sites on the surface of the used AC after each use. Besides that, in spite of washing and thermal activation processes that were applied after each use there is a probability of remaining of some sticking species that may remain adsorbed on the pores of the AC surface. These observations can lead to reduce the ability of adsorption and consequently reduce the efficiency of dye removal for further usages [42-44]. The obtained results for recycle of the used activated carbon are summarized in Figure 9.

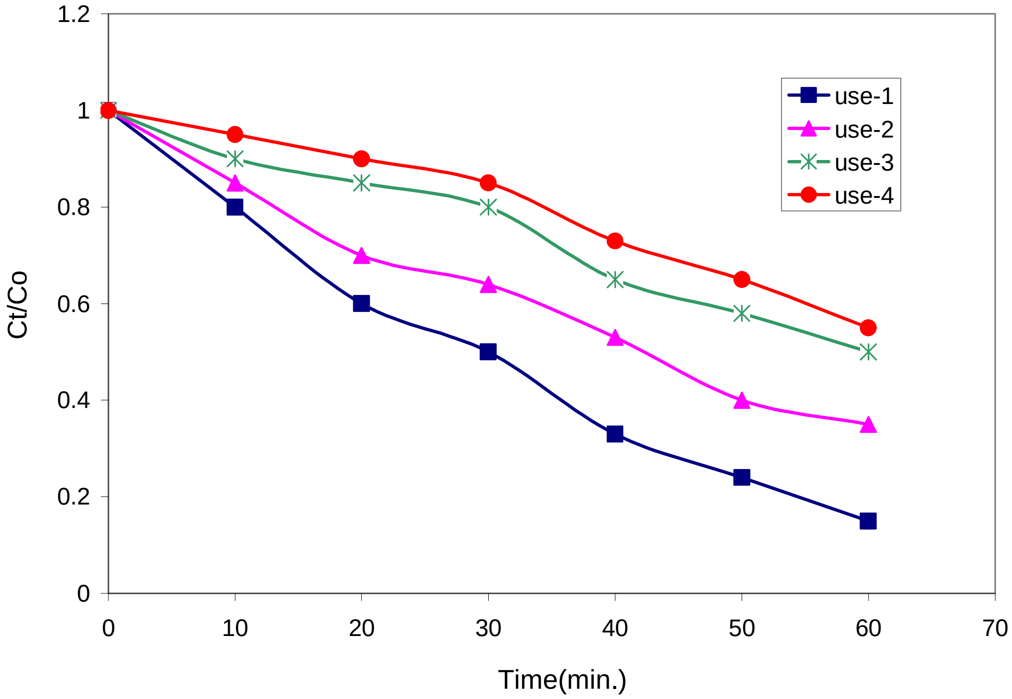

Figure9-Effect of recycling usages of the used AC600on the efficiency of RY145 dye removal

CONCLUSION

In this study activated carbon was synthesized from Iraqi berhy date palm seeds using chemical activation method and the activation processes were performed at three different temperatures 400, 500, 600 and 700 ºC. Adsorption activity of the synthesized AC was investigated by following the removal of RY145 dye from simulated industrial wastewaters. The synthesized ACs showed high uptake adsorption capacity which makes this material as a good candidate adsorbent in comparison with NAC. In addition to that, the activity of AC was increased with increase in activation temperature.

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