

Adsorption of acid black 1 by using activated carbon prepared from scrap tires: kinetic and equilibrium studies

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The effects of initial dye concentration, pH, contact time and the adsorbent dosage were studied. The chemical composition and solid structure of the activated carbon were analyzed using scanning electron microscope (SEM) imaging coupled with energy dispersive spectrometry (EDS). The surface area was measured using Brunauer-Emmett-Teller (BET) isotherm. Experimental adsorption isotherms were well fitted by Langmuir model and maximum adsorption capacity (Q_m) for AB1 was found to be 14.51 mg g⁻¹. The kinetic studies showed that the adsorption of AB1 complies with the pseudo-second-order kinetic model ($R^2 > 0.9999$).

Keywords: Adsorption, Activated Carbon, Scrap tires.

Introduction

Almost 250,000 tons per year of scrap tires are produced in Iran;¹ but only 21% is recycled². The typical compounds are shown in Table 1. Stored scrap tires cause environmental problems such as: the accumulation of insects and flies; ignition of such tires and illegal dumping³. There are many opportunities for producing new products, such as activated carbon using scrap tires as a cheap and abundant resource. The amount of fixed carbon (almost 25% of mass), and a small amount of ash (<5%) in tire plastic is an advantage for producing activated carbon⁴. Also, pyro-oil and pyro-gas produced by pyrolysing of scrap tires is a recyclable source of energy⁵. Value added activated carbon from scrap tires can be used in industries to improve air quality and also for wastewater treatment. Producing activated carbon from scrap tires has an economic benefit and an environmental one. Activated carbon can be used to treat dye wastewater in the textile industries and other industries which use dyes and dye pigments⁶. Most of

the dyes are not biodegradable due to their complex ring structure^{7, 8, 9} consequently treating dye wastewater is difficult¹⁰. The presence of dye in the effluent prevents sunlight penetration into the water and as a result interferes with photosynthetic processes in shallow water resources^{11, 12}. Besides some of the dyes or their metabolites have toxin, mutation and cancerous characteristics. Some studies have reported respiratory and skin disorders due to dyes¹³. Acid dyes are considered hazardous in textile industries¹⁴. The general aim of this study is to produce powdered activated carbon from scrap tires by pyrolyzing, define its features and study its ability to remove Acid Black1 (AB1) from aqueous solutions as an Azo dye. This study also consists of determining the isotherm and adsorption kinetics, the effect of initial dye concentration, adsorbent dosage, contact time, and pH in the function of eliminating this pollutant with the produced activated carbon from scrap tires.

Materials and methods

Instruments

A digital balance with precision of ± 0.0001 g (model Sartorius ED124S) was used for weighing the materials.

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Table 1—Elements and compound of a scrap tire

Compound in a scrap tires		Elements in scrap tires	
Compound	Weigh percentage	Compound	Weigh percentage
(SBR)	60 - 65	Carbon	85.16
carbon Black	29- 31	Hydrogen	7.27
Zinc oxide	1.9 – 3.3	Oxygen	0.54
Sulfur	1.1 2.1	Nitrogen	.38
Covering oil	2	Sulfur	2.3
Supplementary materials	0.7	Ash	4.36

Carbonization of the scrap tire sample was carried out by a muffle furnace (model Exiton). For the pH measuring of dye solutions a digital pH-meter (model Sartorius Professional Meter PP-50) was used. Stirring the dye solutions was done by a magnetic induction mixer (Oxitorp IS 12) within a WTW- TS 606/2-i incubator. A 301 Sigma Centrifuge was used for samples centrifuging. The dye concentrations were measured by spectrophotometry (Pharmaspec Shimadzo UV-1700) at the maximum wave length of 622 nanometer. The structure and characteristics of the Acid Black1 are shown in Table 2.

Preparation of tire derived activated carbon

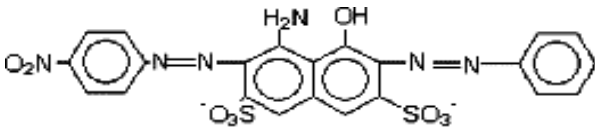
The car scrap tire was ground and sieved to a size of 0.2 to 0.3 cm and rinsed several times with water. The pyrolysis of the scrap tires was carried out by an adjustable electrical furnace at 700°C for 2 hr in the presence of KOH as a chemical reagent. Chemical activation with KOH in the ratio of chemical reagent to tire (W/W) is equal to 4 prior to pyrolysis. Pyrolyzed tire samples washed with 250 ml of 0.5N HCl solution at 85°C for 30 min then filtered. The final products were washed using distilled water several times until the water-carbon mixture showed pH>6.9; The products were dried in a vacuum at 110°C for 24 hr overnight and cooled to room temperature to obtain final carbon and then mesh wrap by standard ASTM sieves with a 100 mesh (149 µm).

Activated carbon characterization

The total pore volume, average pore size and specific surface area, the Barrett-Joyner-Halenda (BJH) and Brunauer-Emmett-Teller (BET) isotherms were determined by nitrogen adsorption/desorption using Belsorb software (Ver.5) (BEL, Japan, Inc). In this

Table 2—The structural formula and characteristic for Acid Black1

Dye rate (%)	MW	$I_{\max}$ (nm)	Chemical formula	Azo dye
85	616.50	622	$C_{22}H_{14}N_6Na_2O_9S_2$	AB1



Acid Black 1

relation W is the average pore diameter of the sample, V the total pore volume and S is the obtained specific surface area by BET-N₂. The distribution size of the pores was measured using the Barret, Joyner and Halenda (BJH) model. Scanning electron microscope (SEM) imaging coupled with energy dispersive spectrometry (EDS) (kV 20.0 Takeoff Angle 25.0° and Elapsed Livetime 10.0) of Majlesi University of Material Engineering was also utilized. The surface chemistry at specific surface locations (as visualized on the SEM screen) was determined using energy dispersive spectrometry (EDS, Model Noran 5502 Series II).

Adsorption experiments

The adsorption tests were performed in a batch system and the effective parameters on the adsorption process included the contact time (5, 10, 15, 30, 45, 60 and 120 minutes), pH of dye solution (3, 5, 7, 9 and 11), adsorbent dosage (0.2, 0.4, 0.6, 0.8 and 1 g) and initial dye concentration (50 to 400 mgL⁻¹) were studied in different stages. The speed of stirring was 200 rpm; the temperature 25±1°C and the size of adsorbent particles were mesh 100. The volume of test solutions was 0.250Li; and the adsorbent particles were separated after the dye removing process by centrifugation. The tests were performed by making other variables constant. To study the isotherm model fitting, Langmuir, Freundlich, Dubinin-Radushkevich and Temkin models were used. Four kinetic models name by pseudo-first order, pseudo-second order, modified pseudo-first order and intra particle diffusion models were assessed and the most suitable model was selected by considering the regression coefficient (R²). In order to determine pH_{ZPC}, the equilibrium technique in a batch system was applied¹⁵. This technique is based on estimating H⁺ and OH⁻ which are the ions determining the electric potentials. 0.1 g of

Table 3—Scrap tire derived activated carbon characteristics

Specifications	Value
pHZPC	6.87
BET Surface area(m ² gr ⁻¹)	185.0
BJH Surface area(m ² gr ⁻¹)	146.4
Total Pore volume(cm ³ gr ⁻¹)	0.58
The size of used powder activated carbon (μm)	<149
Pore size (nm)	52.46
Maximum frequency pore size distribution	
BJH normalize (nm)	86.28

powder carbon was mixed in the incubator machine with 20ml of KNO₃ solution in a sealed container for 24 hours. Primary pH was measured using the 0.1 M KOH and HNO₃ in the range of pH 2 to 12. The final pH of each container was measured using pH meter and their amounts were drawn in the primary pH. The percent of removed dye by activated carbon was calculated from the following equation^{16,17}:

$$\text{Removed dye(\%)} = [(C_{\text{æ\%}} - C) / C_{\text{æ\%}}] \times 100 \quad (1)$$

Where $C_{\text{æ\%}}$ is the initial dye concentration before addition of the sorbent and C is the dye concentration after adsorption with produced activated carbon and using equation 2, the carbon yield after pyrolysing was calculated.

$$\text{Carbon Yield\%} = W_{\text{ac}} / W_{\text{ti}} \times 100 \quad (2)$$

In the equation W_{ti} is the weight of used scrap tire for pyrolysing and W_{ac} was final produced activated carbon.

Results and discussion

The structural characteristics of the produced active carbon

In this study the activated carbon production output was 32 to 36.5%. The results regarding the analysis of the produced active carbon are shown in figures 1, 2 and table 3.

Table 3 shows the BET surface area of 185 m²gr⁻¹ which, although low compared to some literature values¹⁸, is of a comparable order of magnitude with other literature data¹⁹; in the next phase of our project we will attempt to produce higher surface area carbons now that we have demonstrated the feasibility of Producing activated carbons from Iranian waste tires.

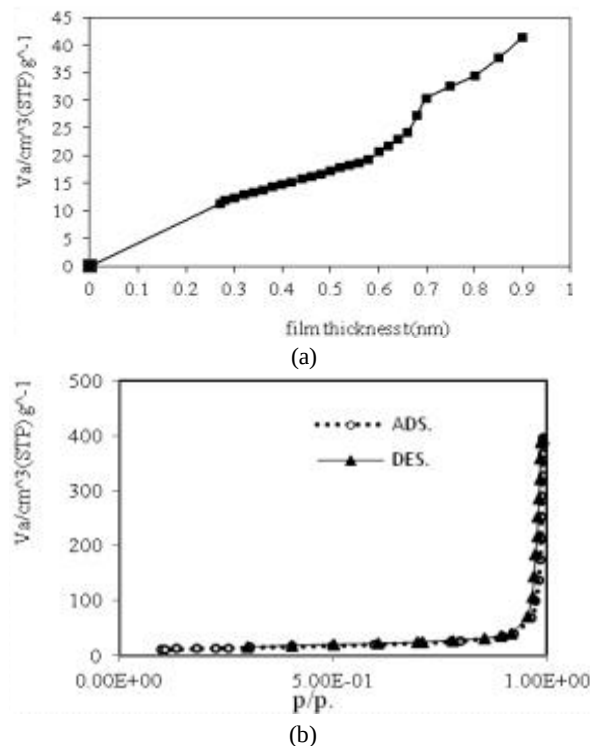


Figure 1— (a) Pore size distribution, (b) adsorption-desorption analysis for produced activated carbon

The carbon to oxygen ratio of the active carbon sample was 7.54 units. With an increase in temperature the excess carbon is burnt and therefore a higher mass of initial material is decomposed as CO. Also, this decreases the amount of oxygen in the produced active carbon's structure. Therefore, for producing non-polar carbonaceous material, the product is prepared at higher temperatures in order to eliminate the existing oxygen in the structure. As shown in the SEM graph, the surface of the activated carbon has pores with various sizes. The existence of such pores on the surface of the activated carbon facilitates the accessibility to internal pores. A slight increase in the nitrogen adsorption amount with an increase in relative pressure (Fig. 1) depicts the existence of mesopores in the activated carbon structure. As shown in figure 1, with an increase in relative pressure, the amount of nitrogen adsorption increases suddenly which can be due to the destruction of some micropores which are transformed to macro and meso types when pressure is exerted on them. The nitrogen desorption curve is in the form of a residue ring indicating mesoporosity. This happens when materials have the maximum number of pores in the meso range, because evaporation through pores is different from their condensation in the pores. Figure 1a shows the pore

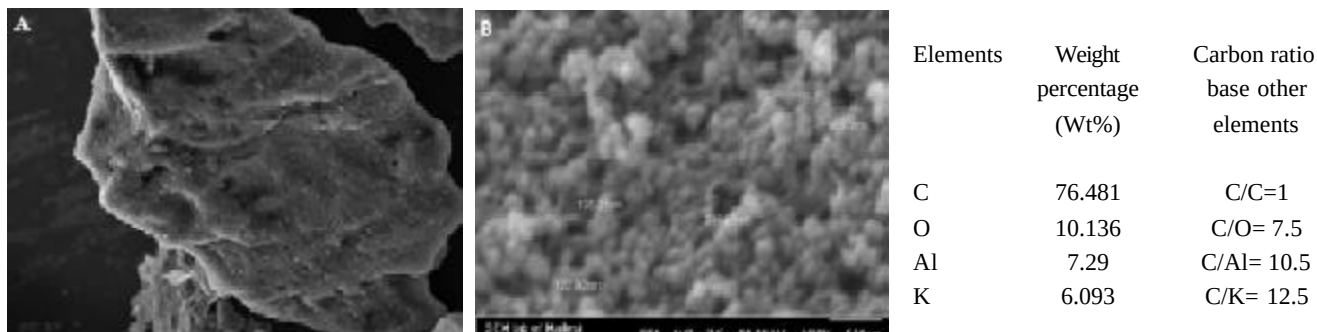


Figure 2—SEM (20 Kv, x60K) and EDS (20 Kv, 2400 x) of produced activated carbon

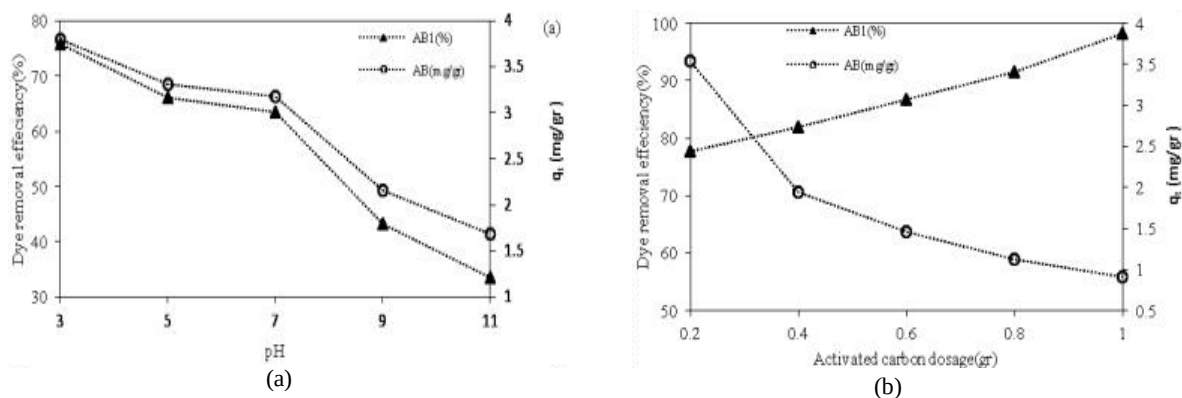


Figure 3—Effect of solution pH and activated carbon loading on percentage removal Acid Black1 dye: (a) Effect of pH (C=50ppm, time=60min and activated carbon=0.2gr); (b) Effect of activated dosage loading (C=50ppm, time=60min and activated carbon=0.2-1gr).

distribution in the 25-90 nanometer range, which confirms the existence of mesopores. It is important to know the chemical characteristics of the activated carbon surface, because the elements present on the surface of the activated carbon influence the characteristics and sorption type.

The existing potassium from the EDS analysis is related to adsorbed potassium from KOH, which existed on the carbon sample although it was washed. This reduces the adsorption efficiency of the produced activated carbon. Carbon that has been chemically activated has more special surface area than carbon that has been activated physically, because chemical factors create more pores in the carbon. The results obtained from EDS analysis showed that the dominant element in the structure of this adsorbent is carbon. The comparison of results obtained from our study is consistent with the findings of other studies. Teng and *et al.*⁴, Sharma and coworkers²⁰ and Galvagno *et al.*⁵ reported that carbon is the main element in the produced active carbon from scrap tires.

Effects of pH

Adsorption experiments were performed at different dye solution pH values (3, 5, 7, 9, and 11) and fixing the other process variables. Experiments in different pH showed the dye removal percentage changes with pH (figure 3a). With an increase in pH the amount of adsorbed dye per unit mass of adsorbent mass decreased from 3.79 to 1.68 mggr⁻¹. The pH affects adsorption by influencing the dye structure and activated carbon surface charge¹⁶; in this study the pH_{zpc} was 6.87. The pH effects the reaction between the anions of the existing dye and the surface charge of the activated carbon. When the pH of the solution is less than the pH_{zpc}, dye anions are attracted to the adsorbent with a positive surface charge and therefore, higher amounts of dye are adsorbed¹⁷.

When the pH of the solution is higher than the pH_{zpc}, the carbon surface has more negative charge and so decreases the adsorption of AB1 anions. During the adsorption experiments, the solution pH slightly increased may be due to the transfer of OH⁻ from the adsorbent's surface into the solution. In other words, the results of

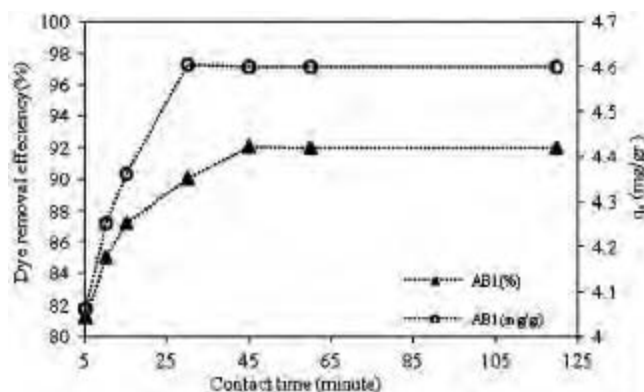


Figure 4—Effect of contact time ($C=50\text{ppm}$, $\text{time}=5\text{--}120\text{min}$ and activated carbon= 1gr).

this stage show the strong reaction power between dye and the elements present at the activated carbon structure, and also the H^+ and OH^- ions can influence adsorption capacity. The adsorption amount in higher pH than pH_{zpc} has reached its minimum amount because the surface of the adsorbent has negative charge which causes mutual repulsion between AB1 ions and the adsorbent surface¹⁷. Hameed *et al.*²¹ reported that with an increase in pH (from 2 to 13) the adsorption amount of AG25 dye reduced following an increase in the hydroxyl anions which changes the surface charge of the adsorbent, which is consistent with the results of our study but Ong and colleagues²² reported that the biosorption of reactive dyes on rice bran increased as pH increased from 2 to 10; in low pH dye adsorption is prevented and the adsorbent surface is surrounded by the carboxyl groups. The results of Sivarajasekar *et al.* studied on assessing the biosorption of Azo dye are consistent with our study²³. In general, based on the findings of present study and also other studies, it can be concluded that pH has a considerable and different effect on the removal of pollutants by adsorption, based on the type of pollutant and adsorbent^{22, 24}.

Effect of Dye Concentration and Contact Time

With an increase in the initial dye concentration from 50 to 400 mg/l, the amount of dye adsorption increased from 2.47 to 13.3 mgg⁻¹. This is because at higher concentrations, resistance to adsorption mass transfer increases; acid dyes have lower adsorption because they have higher solubility. This issue itself depends on the dye sulfonation degree²³. Sulfonation makes dyes negatively charged and hydrophilic. In other words, the high solubility of acid dyes was an important factor which leads to low adsorption. The effect of contact time

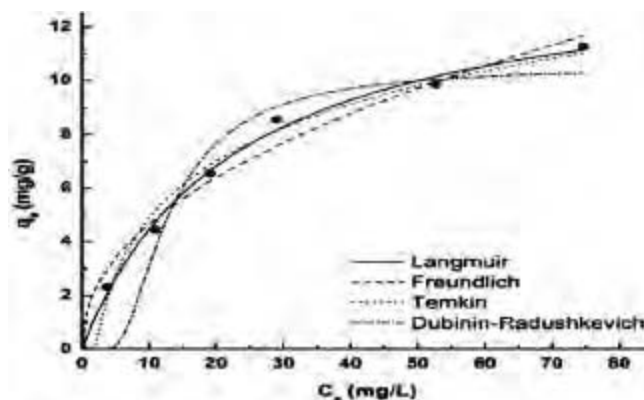


Figure 5—Isotherm plots for adsorption of AB1 dye onto produced activated carbon (Freundlich, Langmuir, Temkin and Dubinin-Radushkevich plot).

between the adsorbent and AB1 is shown in figure 4. Samples were taken from the solution at different time intervals and the AB1 removal amount increased gradually up to 120 minutes when it remained constant. In Arioli *et al.* study, the adsorption of acid and basic dyes on activated carbon reached equilibrium at 50 minutes²⁴. Also, in Ho *et al.* study, the Acid Blue25 dye adsorption on wood reached equilibrium at 250 minutes²⁵. The difference of our findings from the findings of other studies may be due to the pore structure of adsorbate and initial pollutants concentration, on the other hand dye molecular size and its initial concentration as well as the pore size of activated carbon is key parameters for adsorption.

Isotherm study

Choice of the best fit isotherm was done by linear regression analyses and a comparison of their correlation coefficient (R^2). Equilibrium studies which express the adsorbent capacity for the dye are explained by adsorption isotherms. The Freundlich and Langmuir isotherms are the oldest and simplest isotherms and were compared with the linear models of the Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich isotherm models. The general form of their linear equations is presented in table 4^{6,7,23,25}. In the Freundlich isotherm equation (no. 4) q_e is the equilibrium dye concentration on adsorbent (mgg⁻¹), C_e is the equilibrium concentrations of dye (mg/l⁻¹), k and n are constants that describe the capacity and the intensity for the adsorption, respectively. For Langmuir model (no. 5) q_{max} and b are the maximum adsorption capacity (mgg⁻¹) and b is constant in the model (mg/l⁻¹), respectively. The linear form of Temkin isotherm (no. 6) is $q_e = RT/b(\ln K_t + \ln C_e)$.

Table 4 - Kinetic and isotherm model equations used in this study

Kinetic equations			Linear form of isotherm equations	
	$\ln \left(1 - \frac{q_t}{q_e} \right) = -k_1 t$		Freundlich(4)	$\log q_e = \log k + \frac{1}{n} \log c_e$
Pseudo-first order(8)				$\frac{c_e}{q_e} = \frac{1}{q_m K} + \frac{1}{q_m} c_e$
Pseudo-second order(9)	$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t$		Langmuir(5)	
Modified pseudo-first order(10)	$\ln \left(1 - \frac{q_t}{q_e} \right) + \frac{q_t}{q_e} = -k_m t$		Temkin(6)	$q_e = \frac{RT}{b} \ln [(K_f) C_e]$
Intraparticle diffusion model(11)	$q_t = k_i t^{0.5}$		Dubinin-Radushkevich(7)	$q_e = q_D \exp(-B_D [RT \ln(1+1/C_e)]^2)$

Table 5 - Kinetic and isotherm results study

(a) The constants of used kinetic models for AB1 adsorption onto the activated carbon

C_0	Pseudo-first order			Pseudo-second order			Modified pseudo-first order			Intraparticle diffusion model	
	R^2	q_e	$k_1(1/\text{min})$	R^2	q_e	$k_2(\text{g/mg min})$	R^2	q_e	$k_m(1/\text{min})$	$(\text{mg g}^{-1} \text{ min}^{-0.5}) K_i$	R^2
50ppm	0.8046	2.4	0.00258	0.9999	2.4	0.306	0.8094	2.4	0.0246	0.0419	0.8067

(b) The isotherm study results for AB1 adsorption onto the activated carbon

	Dubinin-Radushkevich(D-R)		Temkin		Langmuir		Freundlich	
	q_D	b_D	b_T	A	q_m	K_a	n	K_f
	10.5	0.055	797	0.494	14.5	0.044	2.19	1.62
Standard Error	0.856	0.017	48.72	0.076	0.59	0.004	0.217	0.276
R^2	0.9072		0.9876		0.9950		0.9789	

In this equation T ($^{\circ}\text{K}$) is the absolute temperature, R is the fixed gas coefficient under standard conditions ($8.314 \text{ K, kJmol}^{-1}$), and KT is the equilibrium constant (mgL^{-1}). Also, bT is the adsorption energy variable.

The linear Dubinin-Radushkevich (D-R) isotherm equation used for the adsorption of AB1 onto the activated carbon (equation 7). In the equation q_e is amount of solute adsorbed per unit weight of material (mgg^{-1}), q_s is D-R, maximum adsorption capacity (mgg^{-1}), β ; a constant for activity ($\text{mol}^2\text{J}^{-2}$) and e is the Polanyi potential. The results of the isotherm studies

are shown in figure 5. The calculated parameters for the isotherm equations are presented in table 5b.

The results of this study showed that the dye adsorption is best correlated by the Langmuir isotherm model indicating homogeneous process with each adsorbate molecule has equal adsorption activation energy⁶. When the adsorption process follows the type L isotherm of the Langmuir model, it shows that the ion substrate adsorption (cation or anion) is confronted with weak resistance of the solution molecules²⁵. The findings of our study are consistent with Arivoli and co-workers

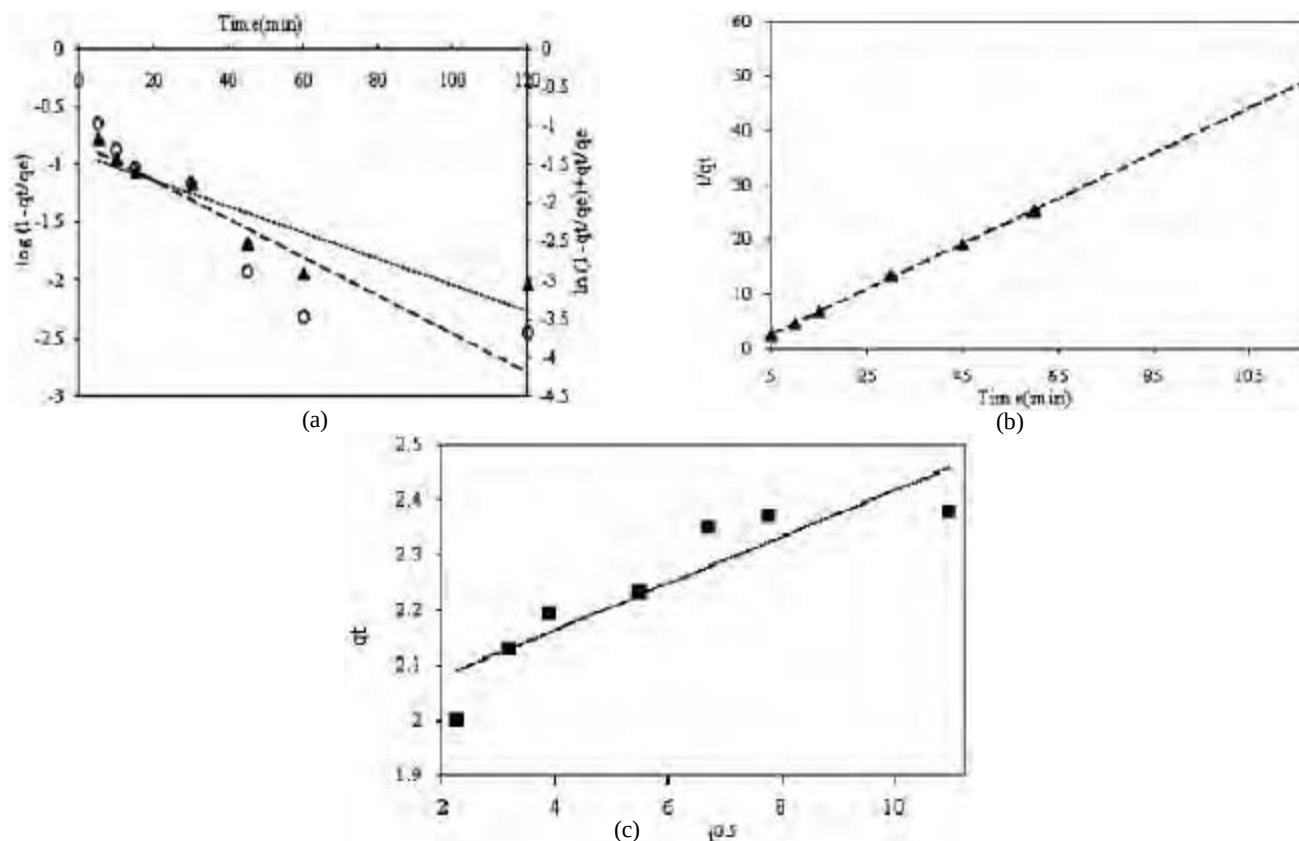


Figure 6—Kinetic plots for adsorption of AB1 dye onto produced activated carbon: (a) Pseudo First order and modified Pseudo First order kinetic plot; (b) Pseudo second order kinetic plot; (c). Intra-particle diffusion model plot.

study. In this study, the maximum adsorption capacity for Acid Black1 was 14.51 mgg^{-1} and the Langmuir constant (K_{ads}) was 0.044 mg l^{-1} for it. The parameters related to the Freundlich isotherm the slope, n ; an index for evaluating the adsorption intensity, is relatively low ($n < 1$).

Kinetic studies

In this study, the adsorption kinetics of the AB1 dye with the produced activated carbon from scrap tires was assessed in order to understand the behaviour of this adsorbent by four models of pseudo- first and second-order, modified pseudo first order and the intra-particle diffusion models²⁵. The kinetic equations of the models have been used presented in table 4. In the kinetic models, k_1 and k_2 are the rate constants for the pseudo-first-order (no. 8) and the pseudo-second-order model (no. 9), respectively and k_i ($\text{mgg}^{-1}\text{min}^{-0.5}$) is the intra-particle diffusion (no. 11) coefficient which is calculated by the equation slope of the drawn line for the data. q_e and q_t are the amount of adsorbed material based on mgg^{-1} at equilibrium and the square root of time, t respectively, and k is the speed based on min^{-1} . The

kinetic experimental results are shown in table 5a and figure 6.

In figure 6(a), two plots are shown, the upper dotted line is based on using the best fit q_e value from the equilibrium isotherm and the lower value (better fitting dashed line) is based on the best fit value of q_e using the first order kinetic equation (8). The results of assessing the best fit AB1 adsorption kinetics showed that the dye kinetics followed the pseudo second order type⁶. The second order kinetics shows that chemical sorption is the rate controlling phase and controls the adsorption processes. Various studies show that most adsorption systems have a good compatibility with this system at different pollutant concentrations. The q_{a} amount resulting from kinetic calculations for AB1 is close to the amount of q_e obtained from experiments. This indicates the accuracy of the applied tests.

Conclusion

Characteristics, abundance, availability and the low-cost raw material of scrap tire derived activated carbon illustrates that scrap tire can be used as an adsorbent for the removal of pollutants from the environment. The

pHzpc of this sorbent can provide suitable conditions for adsorption of acidic dyes from acidic aqueous solutions. The AB1 anion dye removal percentage increases with an increase in contact time because of the accessible increasing adsorption rates. The amount of AB1 anion dye removal reduced with an increase in initial dye concentration because the adsorbent surface is saturated in higher concentrations. Considering the correlation coefficient (R^2), we can conclude that equilibrium and kinetic data of the adsorption process have better compatibility with the Langmuir isotherm and the pseudo second order kinetic model. In general, the results of our study show that the produced activated carbon from scrap tires can be economically beneficial and can have better compatibility with the environment. This method could be used as a recycling method for scrap tires to produce a cheap new adsorbent with the capability which can be used in water purifying industries. Therefore, powdered active carbon can be used for removing acid dyes in textile industries.

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