PRODUCTION AND CHARACTERIZATION OF GRANULAR ACTIVATED CARBON FROM ACTIVATED SLUDGE

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Abstract - In this study, activated sludge was used as a precursor to prepare activated carbon using sulfuric acid as a chemical activation agent. The effect of preparation conditions on the produced activated carbon characteristics as an adsorbent was investigated. The results indicate that the produced activated carbon has a highly porous structure and a specific surface area of 580 m^2/g. The FT-IR analysis depicts the presence of a variety of functional groups which explain its improved adsorption behavior against pesticides. The XRD analysis reveals that the produced activated carbon has low content of inorganic constituents compared with the precursor. The adsorption isotherm data were fitted to three adsorption isotherm models and found to closely fit the BET model with R^2 equal 0.948 at pH 3, indicating a multilayer of pesticide adsorption. The maximum loading capacity of the produced activated carbon was 110 mg pesticides/g adsorbent and was obtained at this pH value**.** This maximum loading was found experimentally to steeply decrease as the solution pH increases. The obtained results show that activated sludge is a promising low cost precursor for the production of activated carbon.
Keywords: Activated carbon; Activated biomass; Sulphuric acid activation; Carbon activation; Activated carbon characterization; Spectroscopic analysis.

INTRODUCTION

Activated carbons are carbonaceous materials that can be distinguished from elemental carbon by the oxidation of the carbon atoms found on the outer and inner surfaces (Mattson and Mark 1971). These materials are characterized by their extraordinary large specific surface areas, well-developed porosity and tunable surface-containing functional groups (Baker et al. 1992, Zongxuan et al., 2003). For these reasons, activated carbons are widely used as adsorbents for the removal of organic chemicals and metal ions of environmental or economic concern from air, gases, potable water and wastewater (El-Hendawy 2003).

The surface oxygen functional groups can be easily introduced to the carbon by different activation methods including dry and wet oxidizing agents. Dry oxidation methods involve the reaction with hot oxidizing gas such as steam and CO_2 at temperatures above 700°C (Smisek and Cerney 1970). Wet oxidation methods involve the reaction between the carbon surface and solutions of oxidizing agents such as phosphoric acid H_3PO_4, nitric acid HNO_3, hydrogen peroxide H_2O_2, zinc chloride $ZnCl_2$, potassium permanganate $KMnO_4$, ammonium persulphate $(NH_4)_2S_2O_8$, potassium hydroxide KOH, etc. From the above oxidizing agents, phosphoric acid and zinc chloride are usually used for the activation of lignocellulosic materials,

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which have not been carbonized before (Puziy et al., 2002). On the other hand, potassium hydroxide is usually used to activate coal or chars precursors. It has been reported that zinc chloride produces activated carbon with higher specific area than that produced by using phosphoric acid (Okada et al., 2003). However, phosphoric acid activation is widely preferred over zinc chloride because $ZnCl_2$ has bad environmental impact and the activated carbon produced when using it can not be used in the food and pharmaceutical industries (Srinivasakannan and Abu Baker 2006).

Activated carbon usually increases the cost of the treatment process. Its economical drawback has stimulated the interest to utilize cheaper raw materials for the production of activated carbon (Rengaraj et al., 2002). Consequently, a wide variety of agricultural by-products and wastes has been investigated as cellulosic precursors for the production of activated carbon in addition to hard wood and bituminous coal. These precursors include: coconut shell and wood (Laine et al., 1989), Olive stones (Rodrigues-Reinoso et al., 2001, Lafi 2001, Elsheikh et al., 2003), sugarcane bagasse (Ahmedna et al., 2000), pecan shells (Shawabkeh et al., 1998), palm seed (Rengaraj et al., 2002), apple pulp (Garcia et al., 2003), rubber seeds (Rengarag et al., 1996) and molasses (Legrouri et al., 2005). Furthermore, more interest has been devoted to utilize some wastes of carbonaceous materials such as paper mill sludge (Khalili et al., 2000), old newspaper (Okada et al., 2003) and waste tires (Rozada et al., 2005).

Recently, activated sludge has been produced as a result of wastewater treatment activities and has emerged as an interesting option for the production of activated carbon (Jeyaseelan and Lu 1996, Tay et al., 2001). The results reported in these studies indicate that chemical activation of the sewage sludge with $ZnCl_2$ and H_2SO_4 produced activated carbon of high adsorption capacity comparable with that of commercial activated carbon. In addition, the choice of a cheap precursor for the production of activated carbon means both considerable savings in the production cost and a way of making use of a waste material, thus reducing its disposal problem (Rozada et al., 2003).

The sewage sludge used in the previous investigations is characterized by its carbonaceous nature and its high content of volatile compound. However, this sludge seems to contain appreciable quantities of inorganic impurities as it comes from urban treatment plants. Therefore, H_2SO_4 seems to be suitable as a chemical activation agent because it is able to dissolve the majority of inorganic impurities found in the sludge. For these reasons, this investigation is directed to the use H_2SO_4 as a chemical activation agent for the production of activated carbon. A relatively pure biomass that is obtained from a wastewater treatment unit of a dairy factory was used as the precursor for the activated carbon prepared in this study. It was reported that the ash content of this biomass is less than 15% (Al-Qodah 2006). The product properties were characterized using some spectroscopic techniques. Furthermore, the adsorption performance of the produced activated carbon was tested with some organic pesticides usually found in industrial wastewater.

MATERIALS AND METHODS

Materials

Samples of activated sludge were collected from a local dairy wastewater treatment unit, separated from water, dried in an oven at 105°C for 24 h, grinded to pass 45 µm screens and stored in a closed vessel for further usage. Sulfuric and nitric acids, and sodium hydroxide of analytical grade were brought from Scharlau chemical company, Spain. Pesticide containing 25 wt% Triadimenol (Vydan) was kindly donated by Veterinary & Agricultural Products Manufacturing Company (VAPCO), Jordan. Its structure is shown in Figure 1.

Deionized water was prepared using Milli Q system (Millipore, France). All Chemicals were analytical grade reagent and the glassware were Pyrex washed with soap, rinsed with nitric acid and then washed with deionized water.

Activation

The activation procedure was carried out in a 2 L beaker where a sample of 200 g of dried sludge was mixed thoroughly with 200 mL of concentrated sulfuric acid. The mixture was heated to 200°C with continuous agitation for 1h. During activation water was evaporated and the mixture became slurry and started to solidify. At this moment agitation was stopped while heating continued until the produced material became a carbon-like material. Trace amount of distilled water was injected into the carbonized material using 10 mL syringe to promote activation. Then, the resulting carbon was allowed to cool to room temperature; washed with distilled water; soaked in dilute sodium hydroxide solution for 30 min; washed a second time with distilled water; dried; and stored in a closed container for characterization.
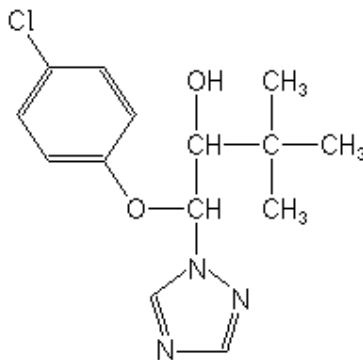
Figure 1: Chemical Structure of triadimenol

Characterization of the Activated Carbon

a) Physical Properties

The apparent density of the material was obtained by weighting five grams of the produced activated carbon and transferring it into a 10 mL graduated cylinder. The cylinder was tamping with a rubber pad while activated carbon was being added until the entire original sample was transferred to the cylinder. Tamping was continued for 5 minutes until there was no further settling produced. The volume was recorded and the apparent density was calculated on the dry basis:

$$\text{Apparent density} = \frac{\text{weight of the sample (g)}}{\text{volume of the sample (l)}} \quad (1)$$

The iodine number is used to measure the porosity of the activated carbon by adsorption of iodine from solution. Each 1.0 mg of iodine adsorbed is ideally considered to represent 1.0 m^2 of activated carbon internal surface area.

Ash content was measured by burning the produced activated carbon in a muffle furnace at 973 K. One gram of dry carbon was transferred into a crucible and then placed in the furnace for four hours. The difference between the original and final weight of the carbon represents the ash content per gram.

Moisture content was also obtained by weighing 10 grams of the carbon and placed in an oven at 105°C for 3 h. Then the carbon was cooled in the absence of humidity and reweighed again. The difference between the initial and final mass of the carbon represents the water content in the sample.

b) Fourier Transform Infrared Spectroscopy

FTIR analysis was made using IPRrestige-21, FTIR-84005, SHIMADZU Corporation (Kyoto, Japan). Sample of 0.1 g was mixed with 1 g of KBr,

spectroscopy grade (Merk, Darmstadt, Germany), in a mortar. Part of this mix was introduced in a cell connected to a piston of a hydraulic pump giving a compression pressure of 15 kPa / cm^2. The mix was converted to a solid disc which was placed in an oven at 105°C for 4 hours to prevent any interference with any existing water vapor or carbon dioxide molecules. Then it was transferred to the FTIR analyzer and a corresponding chromatogram was obtained showing the wave lengths of the different functional groups in the sample which were identified by comparing these values with those in the library.

c) X-Ray Diffraction Measurements

X-ray diffraction spectroscopy (XRD) analyses were carried out with PANalytical X-ray, Philips Analytical. A dried sample of the produced material is grinded using an agate mortar and pestle and tested at 40kV and 40mA. The spectra were analyzed using PC-APD diffraction software.

d) Surface Morphology and Surface Area Measurement

The surface morphology was studied using electron scanning micrographs and then recorded without sample coating by JOEL JSM-5600LV Scanning electron microscope. The surface area was estimated by agitating 1.5 g of the activated carbon sample in 100 ml of diluted hydrochloric acid at a pH = 3. Then a 30 g of sodium chloride was added while stirring the suspension and then the volume was made up to 150 ml with deionized water. The solution was titrated with 0.10 N NaOH to raise the pH from 4 to 9 and the volume V recorded.

Batch Asorption of Pesticide

Equilibrium isotherms for pesticide were conducted in a set of 250-ml Erlenmeyer flasks by

mixing different concentrations of pesticide with 0.1 g of the produced carbon and allowing to equilibrate in an isothermal shaker ($22\pm1^{o}C$) for 24 h. After equilibration, the solution was separated from the solid by filtration. The final concentration was then measured using αS2 Heλios UV-Vis Spectrophotometer at 219 nm. Similar procedures were used at different values of the solution pH and others were performed for blank samples.

THEORETICAL

The mathematical interpretation of the adsorption isotherms is studied using the three popular models; Langmuir, Freundlich and Brunauer, Emmett and Teller (BET) models. Langmuir model is valid for single-layer adsorption, which assumes maximum adsorption corresponding to a saturated monolayer of pesticide molecules on the surface of the carbon where the energy of adsorption is considered to be constant. The mathematical expression for the Langmuir model in terms of pesticide concentration in solution, $Ce(mg/L)$ in equilibrium with that on the solid surface, $qe(mg/g)$ is given by:

$$q_e = \frac{QbC_e}{1+bC_e} \qquad (2)$$

where $Q(mg/g)$ is the maximum amount of adsorbate per unit mass of activated carbon required to form a complete monolayer, and $b(L/mg)$ is the Langmuir constant related to the affinity of binding sites.

Freundlich model is used to incorporate the effect of heterogeneous surface energy in which the energy term, b, in the Langmuir model varies as a function of surface coverage due to heat of adsorption. The Freundlich equation takes the form

$$q_e = K_F C_e^{\beta} \qquad (3)$$

where K_F and β are constants.

On the other hand, BET model assumes that a number of layers of pesticide molecules form at the surface of adsorbent and that the Langmuir equation is applied to each layer of adsorption thus:

$$q_e = \frac{Qk_1 C_e}{(1-k_m C_e)[1+(k_1-k_m)C_e]} \qquad (4)$$

Where k_1 and k_m are the equilibrium constants for the first and subsuent layers, respectively.

These models were applied to fit the experimental data and the corresponding parameters were obtained.

RESULTS AND DISCUSSION

Characterization of the Produced Carbon

The physical properties of the carbon produced from activated sludge obtained using ASTM tests (D 2854-96, D 2866-94, D28867-95, and D 3838-80) are presented in Table 1. These results were compared with those obtained from NORIT SA5. The produced carbon has higher ash content, bulk density and moisture content while it has a lower pH and a close value of iodine number than Norit's.

The FT-IR spectroscopic study of the produced carbon is shown in Figure 2. The sample showed four major absorption bands at 2900-3500 cm^{-1}, 1300-1750 cm^{-1}, 1000-1250 cm^{-1} and 450-750 cm^{-1}. A wide band with two maximum peaks can be noticed at 2930 and 3450 cm^{-1}. The band at 3450 cm-1 is due to the absorption of water molecules as result of an O-H stretching mode of hydroxyl groups and adsorbed water, while the band at 2930 is attributed to C-H interaction with the surface of the carbon. However, it must be indicated that the bands in the range of 3200-3650 cm^{-1} have also been attributed to the hydrogen-bonded OH group of alcohols and phenols (Yang and Lua 2003, Puziy et al., 2003). In the region 1300-1750 cm-1, amides can be distinguished on surface of the activated carbon which has two peaks at 1640 and 1450 cm-1. These functional groups were obtained during the activation process as a result of the presence of ammonia and primary amines that usually exist in the sludge. Moreover, the band at 1500 cm^{-1} may be attributed to the aromatic carbon–carbon stretching vibration. The two peaks at 1125-1150 cm^{-1} yield the fingerprint of this carbon. The sharp absorption band at 1125 cm^{-1} is ascribed to either Si-O (Misra et al., 2005) or C-O stretching in alcohol, ether or hydroxyl groups (Park et al., 1997, Attia et al., 2006). The band at 1150 cm^{-1} can also be associated with ether C-O symmetric and asymmetric stretching vibration (-C-O-C- ring) (Lapuente et al., 1998). This band could also be attributed to the antisymmetrical Si-O-Si stretching mode as a result of existing alumina and silica containing minerals within the sludge samples (Calzaferri and Imhof 1996).

**Table 1: Physical properties of the produced activated carbon
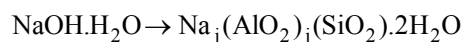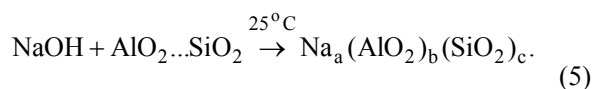compared with the commercial carbon obtained from Norit.**

Material	Produced AC	Norit SA5 AC
Ash content wt%	12	6
Density (g/L)	1051	340
pH	5.5	Alkaline
Moisture wt%	10	10
Iodine number	660	650
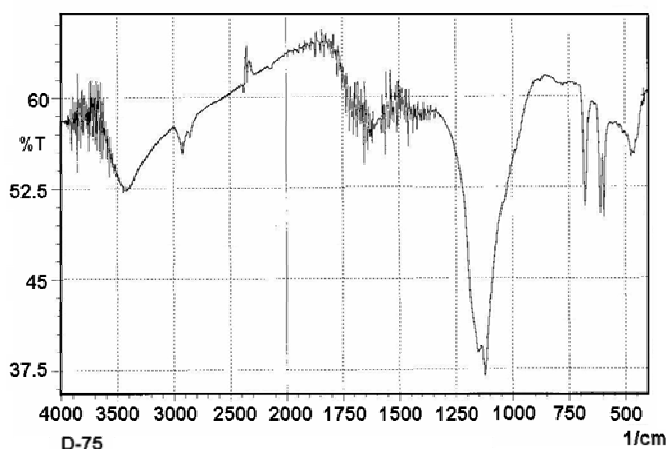
Figure 2: FT-IR spectrum for activated carbon from sludge

The region 450-750 cm^{-1} show two bands in the 480 and 485 cm^{-1} which are associated with the in-plane and out-of-plane aromatic ring deformation vibrations (Socrates 1994). Peaks at 598 and 680 cm^{-1} are assigned to the out-of-plane C-H bending mode. These spectra were also suggested to be due to alkaline groups of cyclic ketons and their derivatives added during activation (Park et al., 1997, Guo and Lua 1999).

X-ray diffractograms for both the activated sludge and activated carbon are shown in Figures 3 and 4. The XRD spectra of the activated sludge illustrated the presence of different alumino-silicate minerals. Zeolite X-Y was observed at 2θ = 29.4° with relative intensity of 158 cps, followed by faujasite detection at 2θ = 26.5°. Other peaks where located at 2θ = 32.9, 35.9 and 39.4° for mullite, hematite and quartz, respectively. While the rest of the peaks for sodalite, analcime and sodium silicates were located at 2θ = 43.1, 47.5 and 48.5°, respectively. When this sludge is treated with acids the majority of those peaks disappear due to leaching out the corresponding minerals during activation and washing with water. However, minor peaks were observed at 2θ = 32 and 41.5 zeolite X and mullite,

respectively. These minerals might be formed after washing and neutralizing the produced carbon with sodium hydroxide which could react with the remaining aluminosilicates in the yielding zeolite according to the following equation (Shawabkeh et al., 2004):

$$NaOH + AlO_2...SiO_2 \xrightarrow{25^{\circ}C} Na_a(AlO_2)_b(SiO_2)_c.$$

(5)

$$NaOH.H_2O \rightarrow Na_j(AlO_2)_j(SiO_2).2H_2O$$

The surface area of the produced carbon can be precisely estimated using the physical adsorption of nitrogen at 77K. However, Sears' method was applied to give a rapid and estimated value of the surface area according to the equation (Sears, 1976):

$$S \ (m2/g) = 32 \ V - 25 \tag{6}$$

where V is the volume of sodium hydroxide required to raise the pH of the sample from 4 to 9. This volume was measured in replicate and found 28.9 ml and the corresponding surface area is 900 m^2/g.
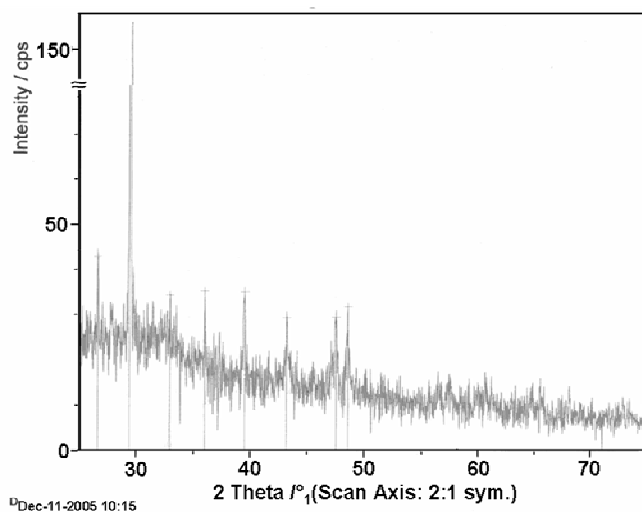
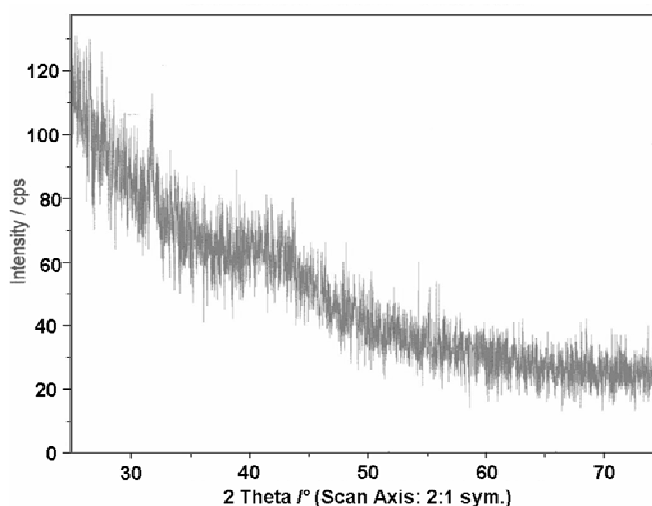**Figure 3:** X-ray diffractogram for sludge sample

Figure 4: X-ray diffractogram for activated carbon from sludge

Adsorption of Pesticide by Activated Carbon

a) Adsorption Isotherm

Adsorption isotherm for Triadimenol pesticide using the produced activated carbon is illustrated in Fig. 5. It is clear that an S-shaped curve appeared which indicates the formation of a multilayer of pesticides molecules on the surface of the carbon. This behavior could be attributed to the deposition of Triadimenol on the surface of the produced carbon.

The molecular structure of triadimenol has both ionic and organic qualities. The ionic nature could play a large role in retaining the species on the surface of the carbon by electrostatic attraction in one hand. On the other hand, the organic part of the triadimenol has hydrophobic moieties. If these groups approach closely to the surface oxygen atoms in carbon surface, van der Waals interaction becomes very strong and dominates the influence of the hydrophobic binding sites of the carbon (Lagaly 2001).
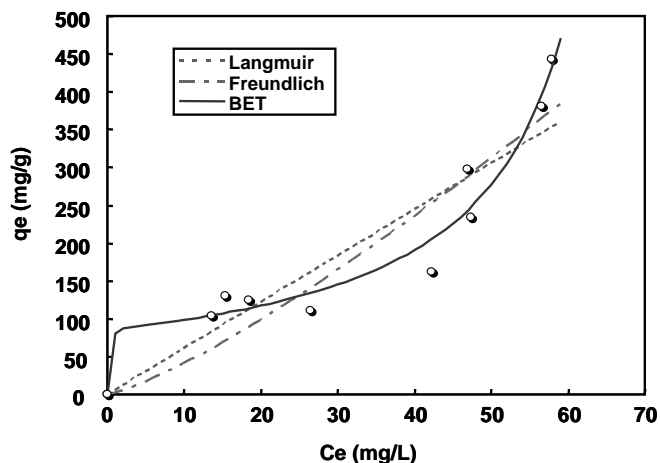
Figure 5: Adsorption isotherm and models of triadimenol

Table 2: Adsorption isotherm model parameter at different pH values..

pH \ MODEL	LANGMUIR	FREUNDLICH	BET
3	Q = 111.8 mg/g B = 24.16 L/mg R^2 = 0.795	K_F = 2.284 β = 1.257 R^2 = 0.861	Q = 110.0 mg/g k_1 = 0.138 L/mg k_m = 0.0128 L/mg R^2 = 0.948
5	Q = 66.43 mg/g b = 0.014 L/mg R^2 = 0.555	K_F = 0.00288 β = 2.582 R^2 = 0.875	Does not fit
9	Q = 48.85 mg/g B = 0.031 L/mg R^2 = 0.610	K_F = 3.517×10^{-5} β = 3.455 R^2 = 0.726	Does not fit

The isotherm data were fitted to different sorption isotherm models namely Langmuir, Freundlich and BET. The corresponding parameters are shown in Table 2. The monolayer capacity s ranging from 48.85 to 111.80 mg/g obtained by Langmuir model which is developed to represent the physical adsorption of adsorbates to cover monolayer on the surface of adsorbent. However, this model did not fit the data adequately especially when the solute concentration exceeded 40 mg/L where the second layer started to occur. Also Freundlich model has a poor fit to the experimental data at such high level of concentration. This isotherm does not predict any saturation of the pesticide by the carbon surface. Nevertheless, it gives an indication about the surface heterogeneity where non-uniform surface may display various levels of energetic heterogeneity.

Apart from the above models, BET shows a good fit to the experimental data at pH 3 with R^2 = 0.948 and $\sum (\text{error})^2 = 6710$. The monolayer of adsorption is predicted by this model with a value of 110.0 mg/g which is in good agreement with the experimental data and that obtained by Langmuir model (111.8

mg/g). Other isotherms obtained at different pH values of 5 and 9 did not fit by BET model adequately.

b) Effect of pH

Fig. 6 shows the effect of pH on adsorption of pesticide onto the produced activated carbon. It is evident from Fig. 6 that the adsorption capacity increases significantly as pH decreases. The monolayer adsorption capacity at pH 3 is ten times more than that at pH 9. The increase in solution acidity can affect the mobility of pesticide ions in the surface and subsurface of the carbon. Moreover, Triadimenol pesticide is a polar compound which easily hydrolyzes at high pH values. The non-hydrolyzed molecule is more easily adsorbed on the surface of the activated carbon due to its higher hydrophobicity than the hydrolyzed molecule (Yang et al., 2005). Also, effect of pH might suggest a possible mechanism of chemical reaction between the reactive groups (–OH) of pesticide and the surface of the activated carbon according:
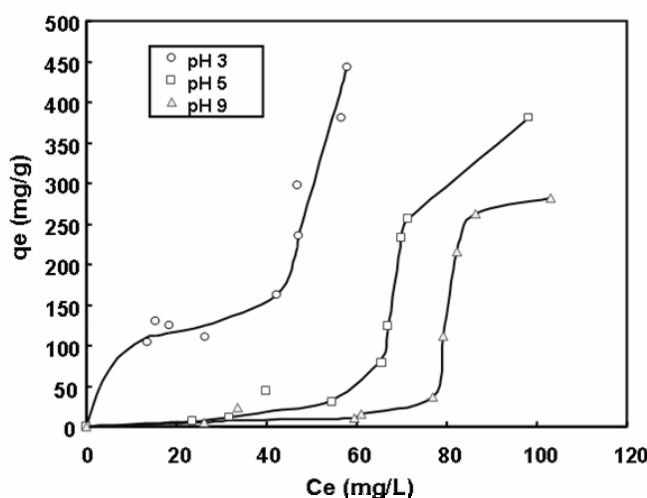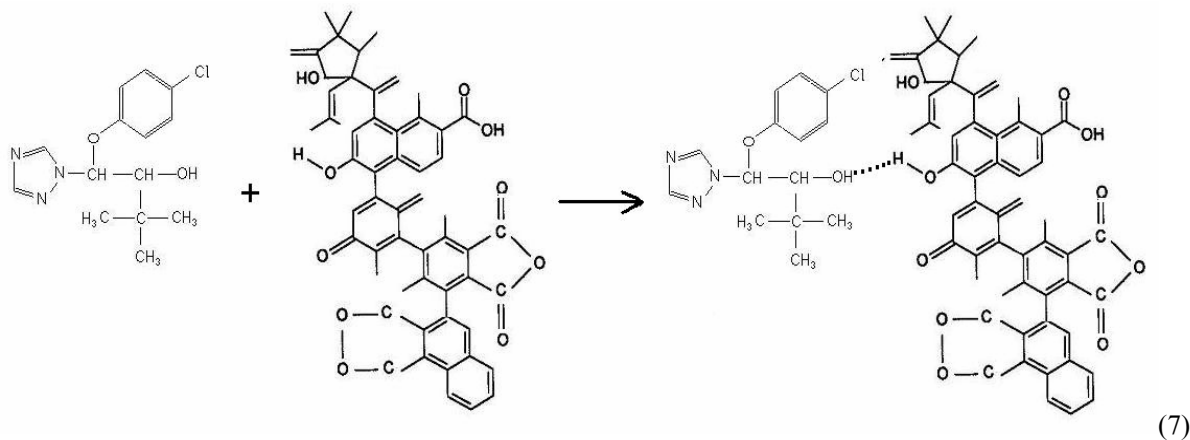
$$(7)$$

Figure 6: Effect of pH on adsorption of triadimenol by activated carbon

Consequently, a covalent bond between the pesticide and the surface of the carbon is formed. Kiriakopoulos et al. indicated that this phenomenon usually occur due to possible changes in the solid surface charge and ionization of the molecule groups or atoms, altering therefore the mechanisms and the extent of adsorption (Kyriakopoulosa and Anagnostopoulos 2005, Rosen 1989). At low pH the pesticide molecule possesses charged sites which could be attracted by the carbon surface. Moreover, the ionization of nitrogen atoms and/or NH groups in pesticide molecules is likely to occur, leading to the adsorption of more pesticide molecules on the surface of the carbon.

CONCLUSIONS

Activated carbon was produced from activated sludge using sulphuric acid as a chemical activation agent. The properties of the produced activated carbon such as surface area, chemical functional groups and chemical composition reveals that it had an improved adsorption behavior comparable to those of high performance adsorbents. The adsorption behavior of the produced activated carbon was tested with pesticides in aqueous solution. The adsorption isotherm data obtained at pH 3 were closely fitted to the BET adsorption model indicating multilayer pesticide adsorption. The estimated maximum adsorption capacity was 110 mg/g and this value was sharply reduced by increasing the pH of the solution.

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