International Journal of ChemTech Research
CODEN(USA): IJCRGG ISSN : 0974-4290
Vol.1, No.4, pp 859-864, Oct-Dec 2009

Activated Charcoal: Preparation, characterization and Applications : A review article

A. Mohammad-Khah and R. Ansari*
Department of Chemistry, Faculty of Science, University of Guilan, Rasht, Iran
P.O.Box: 41335-1914, Tel/Fax +98, +131- 3233262

Corres.author email (1)*: ransari@guilan.ac.ir
e-mail (2) :Mohammadkhah@guilan.ac.ir

Introduction

Adsorption is a widely used as an effective physical method of separation in order to elimination or lowering the concentration of wide range of dissolved pollutants (organics, inorganic) in an effluent. It is big news that activated carbon (AC) is a well known adsorbent that can be used efficiently for removal of a broad spectrum of pollutants from air, soil and liquids. Adsorbents are usually porous solids, and adsorption occurs mainly on the pore walls inside particles. Examples are activated carbon (adsorbs mainly organics), silicagel and activated alumina (adsorb moisture), zeolites and molecular sieves and synthetic resins. Among them, AC is more efficient adsorbent for elimination of many pollutants (organic, inorganic, and biological) of concern in water and wastewater treatment.

In the recent years, it has been increasingly used for the preventation of environmental pollution and antipollution laws have increased the sales of AC for control of air and water pollution. AC is a broad-spectrum agent that effectively removes toxic and bio-refractive substances such as insecticides, herbicides, chlorinated hydrocarbons, heavy metal ions, and phenols, typically present in many water supplies [1-10]. AC in fact is a microcrystalline, non-graphitic form of carbon with porous structure that has been processed to develop its internal porosity. AC has a high degree of porosity, an extensive surface area, and a high degree of surface reactivity. Its large specific surface area of (500 - 2000 m^2/g) is in fact the most important physical property of AC which allows the physical adsorption of gases or vapors and dissolved or dispersed substances from liquids. It has large number of very fine pores (microspores) gives the AC a large inner surface, which is the basis of its remarkable adsorption properties. Consequently, they are effective adsorbents for many pollutant compounds (organic, inorganic, microbial and biological) of concern in water and wastewater treatment.

Applications of Activated Carbon (AC)

Applications of AC are enormous. Its important use is for gasoline vapor emission control canisters in automobile. AC can act as a filter material in air cleaning filters for removal of gases and vapors in the industrial environment. Especially impregnated grades are used in cigarette filters to adsorb some of the harmful components of tobacco, and as the catalyst or carrier of catalytically active substances. Heavy metal ions such as mercury, lead and cadmium in drinking waters are very dangerous even in trace amount, and adsorption method for removing these ions can be essential for water and waste water contaminated by heavy poisonous metal ions. For example lead, cadmium, mercuric ions all are very toxic and carcinogenic. Lead is also a cumulative metabolic poison, acting as a mutagen when adsorbed in excessive amounts. These ions can not be removed from water with classic physical or chemical treatments completely. Activated carbon can be used for removal of poisonous heavy metal ions from aqueous solutions. Adsorption in this case is due to the surface complex formation between the metal ions and the acidic surface function group of AC. Adsorption is due to the surface complex formation between the metal ions and the acidic surface function group of AC. The removal efficiency is influenced by various factors, such as solution concentration, solution pH, ionic strength, nature of adsorbate, adsorbent modification procedure, Physical properties (surface area, porosity), and the chemical nature of AC [11-20].

Simple metal ions are not usually good adsorbates because of their good solvation in aqueous solutions. The better solvation may lead to poor absorbability of metal ions. However it is possible to increase the rate of adsorption with impregnation of AC with suitable chelating agents or modification of the adsorbents. Since AC is a hydrophobic adsorbent, so it seems to be not a good candidate for adsorption of simple inorganic ions from polar aqueous solution; however, it is possible to improve the rate of adsorption of heavy metal ions by pretreatment of AC with some suitable chemical reagents. Sorption improvement is based on simple chemical reactions that are common in chemistry such as acid-base or neutralization, complex formation, redox, precipitation, hydrolysis and catalytic reactions. For example if AC is impregnated by molecule, ions and chelating agents that can combine with transition metal ions to form precipitation, complex or chelates, they can improve adsorption of metal cations significantly. The adsorption of mercury ion is also increased in acidic solutions and also when treated with surface sulphurized AC. AC impregnated with organic compounds with active groups like –SH, -NH can provide more effective adsorption and elimination of heavy metals from the effluents.

As a general rule of thumb is that similar materials tend to associate, AC generally, most effectively adsorbs large and least soluble organic molecules from aqueous solutions. The smaller organic molecules can also be adsorbed if they fit into the smaller pores. Activated carbon has many applications for the purification of products of the chemical, food and pharmaceutical industries. It is widely used to remove color and other impurities such as those causing foaming or retarding crystallization from sugar, in electroplating bath to removing organic impurities and to recover valuable substances from gas and liquid streams. Highly colored waste streams such as result from dying operations can be cleaned-up by activated carbon. Its first use came into prominence through its use as an adsorbent for certain poisonous gases in gas masks in World War 1.

The major use of activated carbon is in solution purification and for the removal of taste, color, odors and other objectionable impurities from liquids, water supplies and vegetable and animal oils. In the recent years, it has been increasingly used for the preventation of environmental pollution and antipollution laws have increased the sales of AC for control of air and water pollution. The amount of material that can be adsorbed by AC is surprisingly large, amounting frequently to form a quarter to an equal weight of such vapors as gasoline, benzene, and carbon tetrachloride. It has been reported that coal carbons are 99% effective for dye removal and could accept dye loadings as 0.40 kg dye per kg of AC. In order to make it economical, fixed bed systems containing granular carbon could be used in tertiary treatment of wastewater. Although there are many chemical, physical, and biological methods of treating water contaminated with industrial and municipal wastes to produce safe and palatable drinking water, none of them have the potential of AC treatment.

Preparation of Active Carbon

Any carboneous materials (animal, plant, or mineral origin) with high concentration of carbon can be simply changed into activated carbon (using both chemical or gas activation methods), The most common raw materials are wood, charcoal, nut shells, fruit pits, brown and bituminous coals, lignite, peat, bone and paper mill waste (lignin), synthetic polymers like PVC, are used for manufacturing of activated carbon. Activated carbon obtained from hard wood is preferable for adsorption because charcoal obtained from soft wood, such as pinewood, is very unstable and readily crumbles. It has been reported that the best grades of AC are obtained from the coconut shell and apricot pits. Activated carbons are commonly prepared by two basic processes: (i) Physical or gas activation method, and (ii) Chemical activation. The choice of activation method is also depending upon the starting material and whether a low or high density, powdered or granular carbon is desired. In gas activation method the raw material with less than 25% moisture, is carbonized first at 400 - 500 ^oC to eliminate the bulk of the volatile matter and then the carbon is subjected to oxidizing gases usually carbon dioxide or steam at 800-1000 ^oC or and with air at law temperature, for selective oxidation [20-24]. The oxidation is preceded usually by a primary carbonization of raw material. The pyrolysis of wood starts at temperature about 225 ^oC.

Carbon is oxidized by atmospheric oxygen is oxidized to CO_2, so the air should be excluded or very controlled during carbonizing and activating. Steam and carbon dioxide act as mild oxidizing agents at 800-1000 ^oC as follow:

$C_x(H_2O)_y \rightarrow C(s) + y\,H_2O$ Carbonization

$C(s) + 2H_2O \rightarrow CO_2 + 2\,H_2$ Steam activation (ΔH = +75 KJ)

$C(s) + CO_2 \rightarrow 2\,CO$ Activation by CO_2 (ΔH = +159 KJ)

The activation of charcoal consists in thermal treatment at high temperatures (800-1000^oC), as a result, these incomplete combustion products burn up and volatilize. Then the surface of the carbon is tremendously increased by the removal of hydrocarbon or tars (Figure 1).

Figure 1: Gradual activation process of carbon during thermal treatment

The Poor absorbability of normal charcoal can be due to its small and very limited surface area due to the filling its pores largely with resins and products of incomplete combustion which are formed during preparation of charcoal. Activation process sweeps the tarry materials, opens the pores, develop porosity, and increase surface area significantly. Gas-phase, vapor-adsorbent carbons are granule form usually prepared from physical or gas activation method. These carbons are hard granules, relatively dust-free pellets having pores diameter less than 3 nm. These carbons are made from hard, dense starting materials such as nutshells and fruit pits. These are carbonized first, and then crushed to a powder, form it into granule with a tar or pitch binder then activate with steam or flue gas at 800-1000 ^oC. In chemical method, both carbonization and activation processes are carried out simultaneously. The raw material generally used is sawdust or peat, which is mixed with the chemical agent in proper ratio, dried, and carbonized at temperatures up to 800 ^oC in the absence of the air (under vacuum or inert gas), after carbonization has been completed, the residual impregnating agent is removed by leaching with water. Inorganic chemicals such as zinc chloride, phosphoric acid and alkali metal hydroxide to degrade or dehydrate the organic molecules during carbonization that prevent deposition of hydrocarbon on the carbon surface. Calcination or carbonization is carried out in a furnace specially designed (internally heated rotary kilns are most commonly used) for the purpose to permit removal of the entire adsorbed hydrocarbon and some of the carbon, so increase the surface area.

A highly active decolorizing carbon is obtained by a rapid process in high yield at a relatively low reaction temperature (600-700 ^oC) with zinc chloride activation. However, the cost of recovery of the activating chemicals is high. In the case of sawdust, the activating agent most commonly used is H_3PO_4 (350-500 ^oC). Because of pollution problem of $ZnCl_2$, this method has become most popular recently. Some raw materials such as bones contain inorganic salts that impart some degree of activity to the carbon when is simply heated or heated in an inert atmosphere. Commercial grades of activated carbon are assignated as either gas-phase or liquid phase adsorbents. Liquid-phase carbons are generally in the form of powdered or granular is characterized as having larger pores (3 nm in diameter and larger) because of need for rapid diffusion in the liquid decolorizing activated carbon are usually employed as powdered.

Powdered activated carbon has an extremely high ratio of area to volume, and since adsorption is a surface phenomenon, so this increases its effectiveness, but also makes it slow to settle and difficult to remove once added. Many decolorizing carbons are prepared by chemical activation.

Decolorizing carbons are coal and lignite based granules, or light, fluffy powders derive from low density starting materials such as sawdust or peat. Approximately 60% of the activated carbon manufactured for liquid phase applications is used in powdered form. Regeneration of the used granule form of activated carbons can be achieved by washing with organic solvents, mineral acids, caustic soda, steam, or dry heat under vacuum but powdered form is very difficult. High-density starting materials such as coconut shells are used for preparation of AC for gas phase applications.

The relative capacity of different carbons is best assessed by trial upon the water to be treated. The pore structure limits the size of molecules that can be adsorbed, and the surface area limit the amount of material that can be adsorbed, assuming suitable molecular size. For screening purposes many manufacturers test their products upon standard solutions to yield iodine, molasses, phenol and methylene blue numbers. The most commonly applied gas adsorption tests are carbon tetrachloride and benzene activity tests. Concentration of organic contaminants can affect the adsorption process. A given AC filter may be more effective than another type of AC filter at low contaminant concentrations, but may be less effective than the other filter at high concentrations. The filter manufacturer should be consulted to determine how the filter will perform for specific chemicals at different levels of contamination. The process of adsorption is also influenced by the length of time that the AC is in contact with the contaminant in the water and amount of adsorbent.

Other important factors that determine the adsorption properties of AC are the pore size distribution and the type of functional groups on the surface. All activated carbons posses not only carbon, but also small amounts of chemically bonded oxygen and hydrogen in the form of various functional groups that usually gives acidic character to solid carbon, plus mineral matters which is usually indicated as ash or residue after ignition. The ash (inorganic) has catalytic effects during carbonization, activation, and adsorption processes. The ash content, ash composition, nature of the surface chemistry and pH of the carbon are the most important chemical properties of activated carbon. The presence of H, O, and other atoms in activated carbon has important effects on adsorption. For example, for removing polar organic compounds from water, the carbons associated with polar oxygen surface structures should preferable, where oxygen-free carbons should be used for removing nonpolar organic compounds. Similarly, when the organic compounds have a large size, carbons with a greater percentage of large pores should be used.

Although AC has been mostly recognized for its application for removal of organic pollutants from polar solutions like water, but we have found in our Lab. Research that it can be used for sorption of many inorganics efficiently. Most of these sorptions are very pH dependent. Chromium hexavalent ion (a well known carcinogenic agent) can be adsorbed by AC only under acidic conditions and under alkaline conditions ($pH > 8$) not any adsorption of these substances was observed [5].

Mechanism of adsorption

When a gas or vapor is brought into contact with a solid, part of it is taken up by the solid. The molecules disappear from the gas or solution either enter the inside of the solid (termed absorption), or remain on the outside attached to the surface (termed adsorption). When the phenomena occur simultaneously, the process is termed sorption. The solid that takes up the molecule is called adsorbent, and the gas, vapor or solute taken up on the surface is called adsorbate. Many organic and inorganic contaminants are removed from either gaseous or liquid solutions by adsorption on the very porous medium (solid phase) with large internal surfaces. Adsorption is a reversible reaction; at a given solute concentration. Adsorbates can attach themselves onto surfaces in two ways. In physisorption (physical adsorption), there is a weak van der waals attraction of the adsorbates to the surface. During the process of physisorption, the chemical identity of the adsorbate remains intact. Physisorption is a spontaneous process ($\Delta G < 0$), since ΔS is negative, so ΔH be exothermic. In chemisorption (chemical adsorption), the adsorbates stick to the solid by the formation of a chemical bond with the surface. This interaction is much stronger than physisorption and in general, chemisorption has more stringent requirements for the compatibility of adsorbate and surface than physisorption [25-28].

The steps of adsorption can be summarized: solute diffuses near the solid surface, diffuses into the pores of particle, then to the pore wall and adsorbs to the pore wall surface. Adsorption of dissolved contaminants is a complex phenomenon caused by several mechanisms, including London- van der Waals forces, Coulomb forces, hydrogen bonding, ligand exchange, chemisorption, dipole- dipole forces, and hydrophobic forces. For example, hydrocarbons most commonly exhibit adsorption through the process of hydrophobic bonding. Thus adsorbents are characterized first by surface properties such as surface area, pore size distribution of micropores and polarity (hydrophilicity, hydrophobicity). The gas adsorption itself, however, can be used to determine the accessible surface area of most adsorbents. When the surfaces of the sorbent matrix are less polar than the water molecule, as is generally the case, there is a strong tendency for the nonpolar contaminant molecules (e.g. oil or hydrocarbons) to partition from the solution and adsorb to the adsorbent. This phenomenon referred as hydrophobic bonding, is a very important factor controlling the fate of many organic pollutants in water and soils. A large specific surface area is preferable for providing large adsorption capacity. Especially materials such as zeolite and carbon molecular sieves can be specifically engineered with precise pore size distribution tuned for a particular separation.

Adsorption processes can be operated on either a batch or continuous- flow basis. In batch processes the sorbent and wastewater are mixed together in a suitable reaction vessel until the concentration of solute has been reduced to the desired level. Separation of the spent sorbent leaves an effluent suitable for discharge. The sorbent can be regenerated and reused. Most continuous– flow adsorption systems are operated as single column or multiple fix-bed adsorption columns in series. Furthermore, they may be operated in either the upflow and packed or expanded) or downflow mode. In downflow systems, the adsorbent can serve for adsorption and for filtration of suspended solids (make adsorption less efficient). Adsorption columns behave in much the same way as ion exchange columns during operation. Most commercial applications use AC in a fixed bed. Fluid passes down through the bed and components adsorb onto the solid. Continuous –flow adsorption systems are capable of treating large volume of wastewater and are widely used for both municipal and industrial applications.

The use of upflow or down flow columns in series are used to optimize adsorbent usage and operating costs. The feasibility of using adsorption for a given waste treatment application must be determined by laboratory and pilot plant tests. In almost all cases the initial tests consist of laboratory scale, constant temperature (preferably at the value anticipated for the wastewater during treatment), constant pH (should match with the value anticipated for the actual wastewater), batch adsorption studies.

Adsorption isotherms

The performance of an adsorbent for adsorption of a specific adsorbate and also valuable information (e.g. the theoretical capacity of the sorbent exhaustion) can be obtained from plotting adsorption isotherm. The relationship between the concentration of chemical adsorbed (S) and the concentration remaining in the solution C at equilibrium is referred as the *adsorption isotherm* because the experiments are performed at constant temperature. Adsorption isotherms generally exhibit one of three characteristic shapes, depending on the sorption mechanism. These isotherms are referred as the linear isotherm, the Freundlich isotherm and the Langmuir isotherm.

Adsorption isotherm will describe the equilibrium distribution of solute between the solid and liquid phases. The results are usually expressed as a plot of the concentration of chemical adsorbed (mg g^{-1}) versus the concentration remaining in solution (mg L^{-1}). Adsorption isotherms are generated by contacting a fixed quantity of contamination (e.g. 100 mL) with different amounts of adsorbent (e.g. 5 flasks) for a fixed length of time in order to remove impurities. Three types of equilibrium-controlled sorption isotherms are considered in transport models: linear, Freundlich and Langmuir [26, 29-32]. The linear sorption isotherm assumes that the sorbed concentration is directly proportional to the dissolved concentration. Freundlich equation is one of the most popular empirical equations which is used to express the mathematic relationship between the quantity of impurity remaining in solution versus the quantity adsorbed:

$$X/m = KC_e^{1/n} \qquad (1)$$

Where X/m is the amount of impurity (mg) adsorbed per gram of the adsorbent, m is the mass of adsorbent (g), x/m value represents the adsorptive capacity of an adsorbent. C_e stands for equilibrium concentration or final conc. (g L^{-1}) or concentration of adsorbate left in solution, k and n are empirical constants. According to this equation, with increasing weight of adsorbent, x/m is decreased and C_e is decreased since more sorption is occurred and less impurity is left. According to Fruendlich equation, Plotting x/m vs. C_e, a straight line is obtained on a log-log paper. The slope of this line (1/n) provides adsorption intensity. The intercept of such a line will occur at a value of C=1.0 mg/L, such that log C= 0, then K = X/m. In Fruendlich adsorption system, the value of n is determined experimentally from the slope of the line which is equal to 1/n. For a good adsorbent it is between 2 and 10. Values near 1.0 are valid for many organic contaminants but lower values (0.4- 0.6) have to be used e.g. for heavy metals and phosphorus. In the special case that n=1, Freundlich isotherm is the same as the linear isotherm.

The adsorptive capacity of an adsorbent is defined for any equilibrium effluent concentration. The ultimate capacity of an adsorbent is defined as the amount of adsorbate adsorbed when the adsorbent is in equilibrium with the maximum (influent) solute concentration. From a logarithmic plot of the data, it is possible to determine the sorbent' capacity to adsorb the particular impurities at a specified equilibrium concentration, making it possible to determine whether or not the desired degree of adsorbate is plausible with the particular sorption system. The Freundlich equation was linearized as follows:

$$\log(X/m) = \log k + 1/n \log C_e \qquad (2)$$

Plotting values of x/m vs. C on log-log paper, the isotherm is obtained. The intercept of such line will occur at a value of C=1.0 mg L^{-1}, 1/n= Slope of the line (showing the variation of adsorption with concentration), log K is the intercept, showing the adsorption capacity from solution of unit concentration. An isotherm plot can be analysed to determine the performance of a given adsorbent. In general, sorbents with steep isotherms are more efficient than flat isotherm sorbents. For column applications more adsorptive capacity is obtained for sorbents having steep isotherm adsorption and more adsorptive capacity is obtained for flat isotherm adsorptions at lower equilibrium concentrations. The objective in wastewater treatment is to exhaust the sorbent as completely as possible. The amount of adsorption at equilibrium was calculated using the following equation:

$$\text{Adsorption capacity } (q_e) = (C_o - Ce)V/ W \qquad (3)$$

Where C_o is the initial adsorbate concentration (mg L^{-1}), C_e is the final or equilibrium concentration (mg L^{-1}) of the adsorbate, V is the volume of the adsorbate solution (L) and W is the weight of the sawdust (g). The rate of sorption should be time and area (bare surface) dependent. Langmuir isotherm can be used as a model to describe the adsorption isotherm. The Langmuir equation is given as:

$$C_e / q_e = 1/Q_o \times b + C_e / Q_o \qquad (4)$$

where C_e is the concentration of adsorbate solution at equilibrium (mg L^{-1}), q_e is the amounts of adsorbate adsorbed per mass of adsorbent (mg g^{-1}), b is the equilibrium constant related to the sorption energy between the adsorbate and adsorbent (L mg^{-1}) and Q_o is limiting amount of adsorbate, the monolayer or maximum capacity that can be taken up per mass of adsorbent.

Langmuir derived a relationship for X and C based on some reasonable assumptions. These are: a uniform surface, a single layer of adsorbed material, and constant temperature. Langmuir isotherm assumes that the number of adsorption sites is fixed and that adsorption is reversible.

In the Langmuir model adsorption increases linearly with increasing solute concentration at low concentration values and approaches a constant value at high concentrations. The adsorbed concentration approaches a constant value because there are limited numbers of adsorption sites in the adsorbent. If the number of adsorption sites is large relative to the number of contaminant molecules it is possible to use the Freundlich isotherm. It has been reported that the effect of isotherm shape with a view to predict if an adsorption system is "favourable" or "unfavourable". The essential features of a Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor or equilibrium parameter, R_L, which is expressed by the following equation:

$$R_{L=} 1/1 + bC_o \qquad (5)$$

Where *C*o is the initial concentration of adsorbate (mg L^{-1}) and *b* is the Langmuir adsorption equilibrium constant (mL mg^{-1}). Based on the effect of separation factor on isotherm shape, the R_L values are in the range of $0 < R_L < 1$.

General Conclusions

Adsorption process is a powerful technique that can be used for efficient removal or uptake of toxic materials from gas and liquid phases. Activated charcoal is one of the most important adsorbent that can be employed for these purposes. The use of AC is perhaps the best broad-spectrum control technology available at present moment. It is also quite possible to increase the amount of adorption of inorganics by impregnating the activated carbon with suitable chemicals. The selection of impregnating material should be so that it encourages the adsorption via usual chemical reactions (e.g. neutralization, redox, hydrolysis, precipitation and catalytic reactions). Among the many factors affecting sorption or removal of toxic materials from aqueous solutions, the pH effect is the most prominent especially in the case of inorganics, weak organic acid and bases which their dissociation is highly pH dependent.

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