



CHAPTER II

LITURATURE REVIEW

2.1 Activated Carbon

Activated carbon is produced from a variety of carbon-containing raw materials, such as coal, wood, and coconut shell (Mc Laughlin, 1995). These carbon-containing materials are exposed to a series of three controlled temperature regimes. The first temperature regime, dehydration or so called devolatilization, occurs at 100-200°C, where moisture and low-molecular-weight volatile compounds from the raw materials are removed. The second regime is carbonized at 250-500°C, where the raw materials decompose into graphitic backbone and emit as gas compounds. The final regime is activation at 800-1,000°C, where steam is reacted with the graphitic backbone to create the internal pores of the activated carbon. The structure of activated carbon consists of elementary microcrystallites of graphite, but these microcrystallites are stacked together in random orientation and it is the spaces between the crystals which form the micropores. The obtained product is activated carbon with approximately 1,000 m²/g of internal surface area. The surface of activated carbon is essentially nonpolar although a slight polarity may arise from surface oxidation (Ruthven, 1984). As a result, carbon adsorbent tends to be hydrophobic and organophilic, which preferentially adsorbs organic compounds from both vapor and aqueous stream.

Activated carbon is available in different forms such as powder, fiber, cloth and granule forms. Among these forms, granular activated carbon is a key element in a number of control technologies to capture and recover volatile organic compounds (VOCs) from air and water. It possesses a phenomenally high surface area per unit weight, an intricate pore structure, and primarily hydrophobic surface. For gas phase applications, activated carbon normally used is microporous adsorbent since their higher capacity and less diffusion mass transfer limitation (Mc Laughlin, 1995). A phenomenon called capillary condensation allows certain carbons to hold gas-phase VOCs at a capacity of 30% or more of its weight.

2.2 Applications of Activated Carbon

2.2.1 General Application

Activated carbon has been used in many applications such as (Mc Laughlin, 1995)

a) Ground water remediation: to remove chlorinated hydrocarbons and aromatic compounds including benzene, toluene, ethylbenzene, and xylene (BTEX). It can also be used for the removal of VOCs from air stream resulting from *in-situ* removal technique such as soil vapor extraction.

b) Industrial wastewater treatment: to adsorb hazardous wastes and regulated compounds such as nonbiodegradable organic compounds, adsorbable organic halogen (AOX), toxicity, color, inhibitory compounds for biological treatment systems, aromatic compounds, chlorinated/halogenated organic compounds, and pesticides. In addition, it also can be used in protection of industrial waste treatment facilities, which are liable to damage from the oxidation by free chlorine, such as ion exchange resin and membrane units, such as reverse osmosis by removing free chlorine and reducing concentration of organic compounds that may foul these processes.

c) Removal of VOCs: to remove VOCs from air and other gases with very high removal efficiencies, typically greater than 99% at contact times as low as 0.1 second such that the most stringent air quality regulations can be exceeded. Moreover, the technology is reliable, simple and has been proven over many years. Activated carbon is used for VOCs abatement in many applications for examples, ground water remediation, soil vapor extraction and air sparking, chemical and pharmaceutical industries, coating and printing industries, degreasing and cleaning, solvent recovery, tank venting, and ventilation and conditioning system.

d) Chemical and pharmaceutical purification: to decolorize and purify a wide range of organic and inorganic compounds including amines, hydrochloric and other mineral acids, amino acids, glycols, and hydrocarbons.

e) Landfill leachate treatment: activated carbon in combination with biological pretreatment is the leading technology for the treatment of landfill

leachate for the reduction of chemical oxygen demand (COD), adsorbate organic halogens (AOXs), and other toxic substances.

2.2.2 Activated Carbon in Gas Phase Application

Applications of activated carbon in gas phase have been investigated by a number of researchers. Cal *et al.* (1997) examined the use of activated carbon cloths (AAC) to remove low concentrations of acetaldehyde, acetone, benzene, and methyl ethyl ketone (MEK) from air. Adsorption isotherms were measured for VOC adsorbate concentration in the 10-10,000 ppmv range and for water vapor from 0-95% relative humidity. Suyadal *et al.* (2000) investigated the adsorption of TCE vapor in a laboratory scale packed bed adsorber by using GAC at constant pressure (101.3 kPa) to obtain TCE breakthrough curves. The deactivation model was tested to obtain adsorption rate constants and first-order deactivation rate constants. Kim *et al.* (2001) conducted adsorption and steam regeneration of n-hexane, MEK, and toluene on activated carbon fiber (ACF) for single, binary, and ternary systems and were compared with those on GAC. An ACF showed not only larger adsorption capacity but also faster steam regeneration than GAC bed. Wu and Pendleton (2001) investigated interrelationship between the adsorbent surface chemistry and relative hydrophobic nature and the solution ionic strength and solution hydrophobicity during dilute anionic surfactant solution adsorption using activated carbon as an adsorbent. The study demonstrated that both the surface chemistry and the solution properties have a significant influence on the adsorption process. Yun *et al.* (1998) studied the adsorption equilibrium isotherms of dichloromethane, TCE and 1,1,1 trichloroethane on ACF at 289, 323, and 348 K. The Dubin-Astrakolov isotherm equation was found to provide an excellent fit to the experimental data.

2.2.3 Dynamics of Adsorption of Activated Carbon in Packed Bed Column

Adsorption is one of separation processes, which are in widespread industrial use, particularly in the petroleum refining and petrochemical industries.

The underlying physical and chemical principles on which such processes are based are reasonably well understood.

Brunauer *et al.* divided the isotherm for physical adsorption into 5 classes (Ruthven, 1984). The isotherms for true microporous adsorbents, the pore size of which is not very much greater than the molecular diameter of the sorbate molecule, are normally type I. That is because there is a definite saturation limit corresponding to complete filling of the micropores. Adsorption isotherm types II and III are generally observed in adsorbents, in which there is a wide range of pore sizes. In such the system, there is a continuous progression with increasing loading from monolayer to multilayer adsorption and then the capillary condensation. An isotherm of type IV suggests the formation of two surface layers either on plane surface or on the wall of the pore very much wider than the molecular diameter of the sorbate. In addition, there are other adsorption isotherms proposed over the years such as Harkins-jura, Dubinin-Redushkevich-Kaganer, and deactivation model (Suyadal *et al.*, 2000).

2.3 Regeneration of Activated Carbon

After the adsorption capacity of activated carbon is consumed as reaching the breakthrough concentration (the minimum acceptable concentration of the VOCs in the effluent gas stream), the spent activated carbon must either be replaced or be regenerated to restore its adsorptive capacity. The carbon can be regenerated by many methods as followings:

- Thermal regeneration
- Chemical regeneration
- Solvent regeneration
- Supercritical fluid regeneration
- Ultrasonic regeneration
- Microwave regeneration
- Biological regeneration

For each regeneration techniques, several studies have been carried out in recent years. A few of relevant research works have been described herein.

2.3.1 Thermal Regeneration

Yang *et al.* (1993) regenerate activated carbon fiber by helium flushing at 200°C. The regenerated activated carbon could be used as an iodine removal adsorbent without significant change in the removal efficiency. Cannon *et al.* (1994) conducted thermal regeneration on the spent activated carbon in steam, carbon dioxide, or steam plus carbon dioxide, at 650-950°C. The regenerated carbon's pore structure maintained greater micropore volume at high temperatures (higher than 800°C) than at lower temperatures (lower than 750°C). Monreno-Castilla *et al.* (1995) studied the thermal desorption process of phenol, m-aminophenol, p-cresol, and p-nitrophenol from an activated carbon by heating to 1100°K in a helium flow. The results showed that during the heat treatment, part of the degradation reaction to light gases and to residue that remained on the surface of the activated carbon. Gomez-Serrano *et al.* (2000) regenerated carbon by heating at 250°C for 2 h in nitrogen atmosphere or by washing thoroughly with distilled water for 24 h. The mass losses in both cases are about 33.6 wt.%. San Mingual (2001) conducted regeneration in steam at 800°C over reaction time between 0-60 minutes. It was found that the steam gasification in the range of 5-10% burn-off had some positive effects on the characteristics of the spent carbon surface. Dranca *et al.* (2001) determined the temperature, in which p-nitroaniline undergo thermal desorption from the surface of activated carbon. It was established that the impregnation of the activated carbons facilitated the regeneration process in the sense that they can occur at lower temperatures. Bagreev *et al.* (2002) regenerated unmodified activated carbon, which adsorbed hydrogen sulfide in water treatment plant by washing with cold water and thermal treatment at 300°C. The results showed that thermal treatment is more efficient than cold water washing. Ferro-Garcia *et al.* (1996) regenerated activated carbon by using organic solvent treatments and subsequent heat treatments. The organic solvents used were acetone, methanol, and benzene. The heating temperature was at 1,073 K in an inert flow. The amount of adsorbate was removed increase in comparison to treatments without solvents.

2.3.2 Chemical Regeneration

Reed *et al.* (1995) conducted the regeneration of granular activated carbon in columns that were used for Pb removal using several regeneration schemes. Acid type (HCl and HNO₃), regenerant concentration (0.1N or 1 N) were used, and NaOH-only rinse were studied. The results showed that the type of acid did not affect column performance. Rinkus *et al.* (1997) studied regeneration of activated carbon, which high percentage of both Pb and phenol. A higher regenerant-to-carbon ratio resulted in increased regeneration. Gupa *et al.* (1998) regenerated activated carbon for removal of Hg²⁺, Cr⁶⁺, Pb²⁺, and Cu²⁺ by using 1 M HNO₃. Bagreev *et al.* (2000) regenerated activated carbon used as hydrogen sulfide adsorbent by using hot and cold water. The hot and cold water washing resulted in the similar capacity of hydrogen sulfide adsorption. Li *et al.* (2001) regenerated activated carbon saturated with phenol by using ethanol or sodium hydroxide solution with many times to obtain 90% efficiency of regeneration. Leng and Pinto (1996) investigated the mechanism of chemical regeneration using methanol, sodium dodecyl sulfate and CTAB. The results indicated that surface properties affected the regeneration efficiency.

2.3.3 Solvent Regeneration

Kilduff and King (1997) investigated the effect of surface properties on the uptake of phenol by carbonaceous adsorbent and on subsequent regeneration by acetone leaching. Matheickal *et al.* (1998) carried out the regeneration of activated carbon both batch and fixed-bed mode saturated with phenol wastewater by using ethanol and rinsing with water. In fixed-bed process, the regeneration efficiency was 98 % after the first regeneration cycle and 94% after the third regeneration cycle. Chinn and King (1999) explored solvent regeneration with acetone or methanol as a means to recover adsorbed multiple-OH compounds from activated carbons in batch and fixed-bed systems. With methanol as a regenerant, fixed-bed adsorption and regeneration could be operated over several cycles, because acetone is more strongly held on the bed than methanol.

2.3.4 Microwave Regeneration

Mocho *et al.* (1996) studied the use of electromagnetic induction to heat activated carbon. For a given current at a frequency of 263 kHz, the energy efficiency was found to be in the range of 40 to 80%. Bradshaw *et al.* (1997) regenerated granular activated carbon by microwave at 650, 700 and 800°C in multimode cavity at 2450 MHz. The acceptable carbon performance was obtained by regeneration at 650°C for zero minute. Van Wyk *et al.* (1998) regenerated spent activated carbon at 750-850°C for 5-30 minutes by using microwave oven. The results showed that microwave-regenerated carbon generally performed better than mine-regenerated carbon.

2.3.5 Supercritical Carbon Dioxide Regeneration

Chihara *et al.* (1997) evaluated the adsorption equilibrium and adsorption dynamics for some typical organic pollutants under some critical CO₂ conditions. The results showed that the adsorption equilibrium constants of some organics varied with CO₂ density and their boiling points. Grajek (2000) investigated the influence of the adsorption and desorption temperature, the pressure and the density of the extraction solvent, the solubility of the adsorbate in the extraction solvent, the activation energy for adsorbate desorption and the particle size of the adsorbent on the adsorbate desorption efficiency by this method. The results indicated that the temperature and pressure affected the desorption efficiency of adsorbate.

2.3.6 Ultrasonic Regeneration

Rege *et al.* (1998) studied the feasibility of using ultrasound to accomplish the difficult desorption of phenol from activated carbon and polymeric resin adsorbent. The desorption rates of activated carbon were found to significantly increase by ultrasound at 40 kHz and 1.44 MHz. Schueller and Yang (2001) investigated the effects of ultrasound on the adsorption and desorption of phenol using activated carbon and polymeric resin as adsorbents and ultrasound at a power of 60 W and frequency of 1 MHz. Hamdaoui *et al.* (2003) evaluated the effects of

ultrasound on p-chlorophenol adsorption-desorption on granular activated carbon. Results indicated that desorption rates of p-chlorophenol from activated carbon were favored by increasing ultrasound intensity. The rise is more noticeable as temperature increases.

2.3.7 Bioregeneration

Ivancev-Tumbas *et al.* (1998) regenerated activated carbon which desorbed phenol from wastewater at the substrate concentration ranging from 1.9 to 1053 mg/L. A 92% efficiency of phenol removal was achieved irrespective of whether the fresh or bioregenerated activated carbon was used. The process regeneration for the once-used activated carbon was faster than for the one that was used several times.

Recently, several novel regeneration methods have been investigated, for example, wet oxidative regeneration and catalytic regeneration. Shende and Mahajani (2002) studied the regeneration of 10% w/v of spent activated carbon by wet oxidation in the temperature range of 150-250°C using oxygen partial pressure between 0.69-1.38 MPa in an autoclave. It was observed that the regeneration mainly occurred due to the oxidation of the adsorbate taking place on the surface of carbon. It is possible to regenerate the spent activated carbon to the extent of more than 98% by wet oxidation. While Matatove-Meytal and Sheintuch (2000) investigated regeneration of spent granular activated carbon saturated with chloroorganic compound by catalytic regeneration. Complete recovery of the adsorption capacity was achieved in three tested cycles of desorption and regeneration in batch and column.

2.4 Surfactant-Enhanced Carbon Regeneration

Regeneration of spent activated carbon for gas-phase application and polymeric resin using surfactant is one of the promising alternative methods. Since surfactant possesses two predominant properties, which lowers interfacial tension

and increases solubilization (Rosen, 1989). Lowering interfacial tension is caused by adsorption of surfactant monomer at the interface of two surfaces while solubilization is caused by preferential solubilization of organic compounds into the core or the palisade layer of micelles (aggregates of surfactant molecules). Its lowering interfacial tension property provides surfactant monomers to reach in to the micropore of the activated carbon by diffusion to replace organic compounds adsorbed on the activated carbon surface by adsorption of surfactant monomer. This phenomenon causes desorption of the organic compounds from the activated carbon surface. The desorbed organic compounds are migrated out from the activated carbon pore into the bulk solution by the stream of surfactant solution. Then, the organic compounds solubilize into the core or palisade layer of the micelles of surfactant. This regeneration technique is called surfactant-enhanced carbon regeneration (SECR).

Surfactant-enhanced carbon regeneration (SECR) is a novel, *in-situ*, low-energy method of regenerating spent adsorbents. In SECR, a concentrated surfactant solution (regenerant solution) is passed through the spent adsorbent. The organic adsorbate desorbs by adsorption of surfactant and is solubilized into the micelles in the regenerant solution, as shown in Figure 2.1. The organic contained in the solution and the surfactant can be recovered. The residual surfactant on the carbon is washed off with a water flushing step. The process strategy of SECR for gas-phase application, activated carbon consists of three major steps: desorption of sorbate organic compound, flushing with water, and drying as shown in Figure 2.2.

2.4.1 Adsorption of Surfactant at Solid-Liquid Interfaces

One of the characteristic features of surfactants is their tendency to adsorb at interfaces in an oriented fashion. In general, the adsorption of surfactants involves single ions rather than micelles (Rosen, 1989). The adsorption of surfactants at the solid-liquid interface is strongly influenced by a number of factors, which are the nature of the structural groups on the solid surface, the molecular structure of the surfactant being adsorbed, the environment of aqueous phase. Furthermore, there are a number of mechanisms by which surfactant may adsorb onto solid substrates from

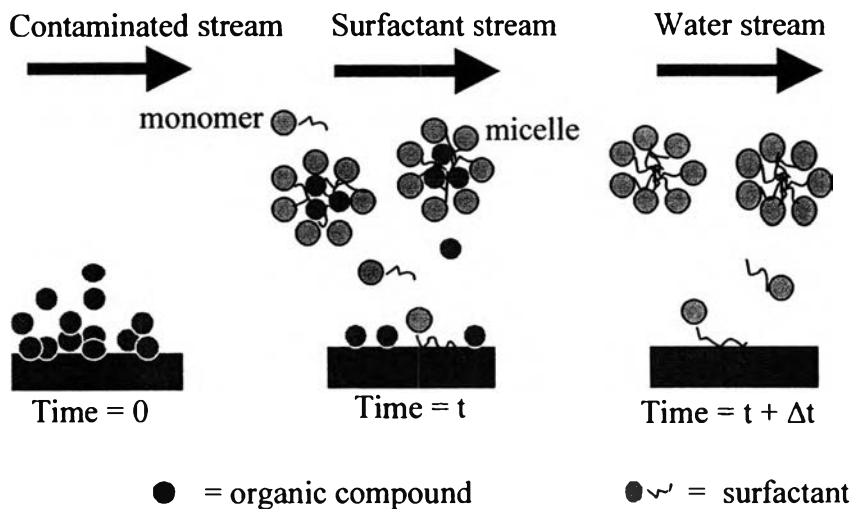


Figure 2.1 Principle of SECR.

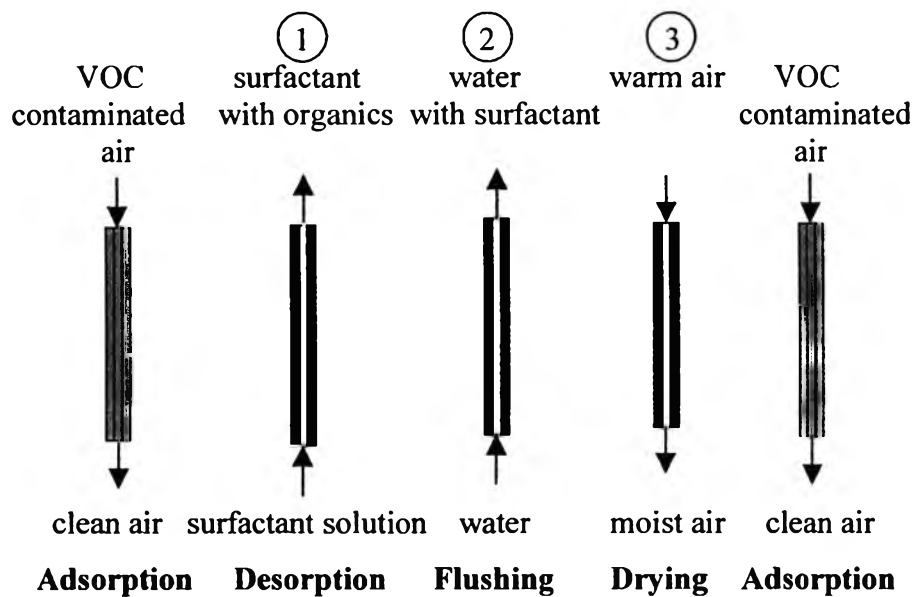


Figure 2.2 Schematic diagram of the process strategy of SECR.

aqueous solution. The mechanisms are ion exchange, ion pairing, acid-base interaction, adsorption by polarization of (π) electron, adsorption by dispersion forces, and hydrophobic bonding. Among these mechanisms, for surface of activated carbon, which its surface is hydrophobic substrate, the adsorption by dispersion forces is predominant. However, the surface of carbon can be oxidized by oxygen in the air at high temperatures during its manufacturing, so another mechanisms, ion

pairing or ion exchange, would be involved as a minor. An increase in the length of hydrophobic group increases efficiency and effectiveness of adsorption. The increase in efficiency is due to the increase in magnitude of the Gibb's free energy ($-\Delta G$) of adsorption with increase in number of unit in the hydrophobic chain. The increase in effectiveness may be due to tighter packing of the hydrophobic chain (Rosen, 1989).

2.4.2 Solubilization of Organic Compounds

Solubilization may be defined as the spontaneous dissolving of a substance by reversible interaction with the micelles of a surfactant in a solvent to form a thermodynamically stable isotropic solution with reduced thermodynamic activity of the solubilized material. The important of the solubilization is that it makes possible the dissolving of substances in solvents, in which they are normally insoluble or less soluble. Solubilization into aqueous media is of practical important in such areas as the formulation of products containing water-insoluble ingredients. It can replace the use of organic solvents or cosolvents; in detergency, where solubilization is believed to be one of the major mechanism involved in the removal of oily soil (Rosen, 1989); and in enhanced oil recovery. Solubilization is distinguished from emulsification by the fact that in solubilization, the solubilized material (solubilizate) is in the same phase as the solubilizing solution and the system is consequently thermodynamically stable (Rosen, 1989). The solubility of normally solvent-insoluble material will be improved by solubilization. The solubility is slightly low until critical micelle concentration is reached. The increase of the solubility is approximately linear with the concentration of surfactant.

2.4.3 Previous Works

Previous studies on surfactant-enhanced carbon regeneration (SECR) have demonstrated that this promising technique can provide an effective removal of the organic adsorbate (Blakeburn and Scamehorn, 1989 and Robert *et al.*, 1989). In liquid phase, Blakeburn and Scamehorn (1989) found that approximately 80% of *tert*-butylphenol can be removed using a regenerant stream containing a cationic surfactant with a reasonable volume of regenerant. The majority of

surfactant can be removed by water flush, but large volumes of water are needed for reaching complete removal of surfactant from the carbon. The effective adsorption capacity of the carbon upon subsequent cycles was found to be reduced by this method. However, details of the breakthrough curves were not obtained. Bhummasobhana *et al.* (1996) found that 50% of phenol can be removed using 0.1 M SDS at 500 pore volumes of regenerant and nearly 90% of phenol can be removed at 2,000 pore volumes. The breakthrough curves for the adsorption of phenol on fresh and regenerated activated carbon were also obtained.

In vapor phase application, very few studies have been done. For example, Roberts *et al.* (1989) studied the application of SECR to regenerate carbon containing either toluene or amyl acetate. The results showed that toluene and amyl acetate were desorbed from carbon. However, the details of the breakthrough curves were not generated.

2.5 Regeneration of Polymeric Hydrophobic Adsorbent

Besides activated carbon, polymeric resin adsorbent has been widely investigated to remove organic compounds from aqueous solution and gas stream and have been extensively used in many applications (Bilgili, 2006). It has been used to remove chlorinated organics, pesticides and hydrophobic compounds, steroids, amino acids, polypeptides, and antibiotics from process streams for purification and recovery in pharmaceutical manufacturing. The resin is rigid, nonionic, crosslinked macroreticular copolymer of styrene and divinyl benzene with high surface area and aromatic nature of its surface. It possesses excellent physical, chemical, and thermal stability and it is also stable over a wide pH range in aqueous solution. Its characteristic pore size distribution makes this resin an excellent adsorbent for organic substances of relatively low molecular weight.

The adsorption experiments using polymeric adsorbent have been reported. For example, Juang and Shiau (1999) conducted adsorption isotherms experiment of phenol and chlorophenol from water on resin at 288-318 K. It showed that the

isotherm data could not fit by any conventional equation over the entire range of concentration. The different fitting results could be related to the solute-resin interaction. Young and Lee, (2000) carried out experiments on removal of phenols from aqueous solution by XAD-4 resin. The results indicated that phenols were effectively removed at acidic condition. Deosakar and Pangarkar (2004) studied the removal and recovery of organics from actual industrial effluents. An attempt was made to correlate multi-solute equilibrium data using the Langmuir model.

The regenerations of the resin have been studied recently. For example, (Schueller and Yang, 2001) studied desorption of phenol from polymeric resin using ultrasound. The effects of all parameters were found to be dependent on ultrasound power. Deosakar and Pangarkar (2004) studied on the regeneration of resin using acetone as a regenerant. The results showed that the regeneration was accomplished by 4 to 5 bed volumes. Garcia *et al.* (2002) used 0.1, 0.2, and 0.3 M sodium hydroxide as a regenerant with regenerant flow rates of 43-117 mL/min to remove single and binary mixture of phenol and m-cresol by performing sets of desorption. The results indicated that the sodium hydroxide was efficient and faster than conventional desorption with organic solvents.

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