USE OF ANIONIC SURFACTANT IN REGENERATION OF SPENT ACTIVATED CARBON

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ABSTRACT

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Keywords: Activated carbon/ Surfactant/ Regeneration/ Sodium dodecyl sulfate/ Methyl ester sulfonate

Surfactant-enhanced carbon regeneration (SECR) is a regeneration process that utilizes surfactant solution to dissolve adsorbed organic from spent activated carbon. Sodium dodecyl sulfate (SDS) and methyl ester sulfonate (MES) are anionic surfactants that are used in this investigation. The study is done in 3 steps: sample characterization, regeneration and water flushing step. The proximate analysis of spent activate carbon contains 27.5% moisture, 27.2% volatile matter content, 10% total ash content. The spent activated carbon also contains 18.4% benzene. In regeneration step, the benzene removal by SDS and MES solution were compared. The effect of concentration, solution flow rate and temperature are studied. As expected, the benzene removal increased as the concentration of SDS or MES increased and the benzene removal by MES is higher than that by SDS at the same concentration. However, SDS was easier to be removed in washing step with deionized water than MES.

บทคัดย่อ

บุษย์ กัลปดี : การใช้สารลดแรงตึงผิวประจุลบในการนำถ่านกัมมันต์ที่ใช้แล้วกลับมาใช้ใหม่ (Use of Anionic Surfactant in Regeneration of Spent Activated Carbon) อ.ที่ปรึกษา : รศ. ดร. บุนยรัชต์ กิติยานันท์ 42 หน้า

การนำถ่านกัมมันต์กลับมาใช้ใหม่โดยสารลดแรงตึงผิวเป็นกระบวนการใช้สารละลายของสารลดแรงตึงผิวในการชะสารอินทรีย์ที่ติดอยู่ออกจากถ่านกัมมันต์ด้วยวิธีโซลูบิไลเซชัน โดยสารลดแรงตึงผิวที่ใช้ในการทดลอง คือ โซเดียมโดเดซิลซัลเฟต (Sodium dodecyl sulfate) และ เมทิลเอสเตอร์ซัลโฟเนต (Methyl ester sulfonate) ซึ่งเป็นสารลดแรงตึงผิวชนิดประจุลบ การทดลองได้แบ่งออกเป็น 3 ขั้นตอน คือ การหาคุณลักษณะของถ่านกัมมันต์ตัวอย่าง, กระบวนการนำกลับมาใช้ใหม่ และการชะสารลดแรงตึงผิวตกค้างด้วยน้ำตามลำดับ โดยตัวอย่างถ่านกัมมันต์มีความชื้น 27.5 เปอร์เซ็นต์, ปริมาณสารอินทรีย์ที่ผิว 27.2 เปอร์เซ็นต์, ปริมาณขี้เถ้าหลังจากการเผา 10 เปอร์เซ็นต์ และมีปริมาณเบนซีนที่ดูดซับ 18.4 เปอร์เซ็นต์โดยมวล ในกระบวนการนำกลับมาใช้ใหม่ ปริมาณเบนซีนที่ถูกดึงออกโดยสารลดแรงตึงผิวทั้งสองถูกเปรียบเทียบจากผลกระทบของความเข้มข้น, อัตราการไหลของสารละลายสารลดแรงตึงผิว และผลของอุณหภูมิ จากผลการทดลองพบว่า เมื่อเพิ่มความเข้มข้นของสารละลายสารลดแรงตึงผิวจะทำให้ปริมาณเบนซีนที่ถูกดึงออกมาจากถ่านกัมมันต์เพิ่มขึ้น และเมทิลเอสเตอร์ซัลโฟเนตสามารถดึงเบนซีนออกมาได้มากกว่าโซเดียมโดเดซิลซัลเฟตที่ความเข้มข้นเท่ากัน อย่างไรก็ตามโซเดียมโดเดซิลซัลเฟตที่ตกค้างสามารถชำระออกจากถ่านกัมมันต์ได้ด้วยน้ำกลั่นง่ายกว่าเมทิลเอสเตอร์ซัลโฟเนต

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CHAPTER I
INTRODUCTION

Activated carbon has been widely used in removal of organic compounds form industrial and municipal wastewater. It is usually categorized as a physical and nonpolar adsorbent. Activated carbon has relatively high surface area and porosity for adsorbing contaminants from gas or liquid. The common organic impurities removed by activated carbon are aromatic hydrocarbons e.g. benzene, phenol which are carcinogenic compounds (Patrick, 1995).

After adsorption process, the contaminant is adhered to the surface of activated carbon, as a result, the adsorption efficiency of activated carbon is decreased. The spent activated carbon can be regenerated and reused. Surfactant-enhanced activated carbon regeneration is the regeneration method that utilizes surfactant solution to dissolve or remove adsorbate from activated carbon. Typically, a concentrated surfactant solution is passed through the spent activated carbon bed and the adsorbed organic compounds are removed from the carbon by solubilizing into the micelle of the surfactant solution. The advantages of this process are low energy consumption and no damage to the porous structure of the activated carbon. However, the activated carbon can contain residual surfactant after regeneration. Flushing by water is needed and the bed can be reused directly for liquid phase adsorption or dried prior to use in vapor phase adsorption.

In this study, sodium dodecyl sulfate (SDS) and methyl ester sulfonate (MES) are used and both surfactants are anionic surfactant. SDS is a common surfactant that has been used worldwide and MES can be derived from palm and/or coconut oils, there for it is environment friendly and also has good detergent properties. The investigated is done in 3 steps: spent activated carbon characterization, regeneration and water flushing step. Spent activated carbon characterization such as moisture content, volatile matter, total ash content, elemental composition and the amount of residual surfactant on the activated carbon surface are observed. The regeneration step is studied in effect of surfactant solution concentration, flow rate and column temperature. Deionized water is used as flushing water and to study the effect of flushing water flow rate.

CHAPTER II
LITERATURE REVIEW

2.1 Activated Carbon

Activated carbon (also called activated charcoal or activated coal) is a group of carbonaceous materials with a highly developed internal surface area and porosity that possess a large capacity for adsorbing contaminants from liquids and gasses (Patrick, 1995**)**. Production of activated carbon products can be accomplished by either thermal or chemical methods. Common feedstocks include peat, coal, wood, lignite, and nut shells. Criteria for selecting a feedstock include potential for producing high-quality carbon, availability, cost, storage life, and workability. The properties of the final activated carbon product are a function of both the feedstock and the preparation procedure.

2.1.1 Classification of Activated Carbon

Activated carbon can be classified into 2 groups

2.1.1.1 Powdered Activated Carbon

Powdered activated carbon (Figure 2.1) is generally produced by grinding a pile material prepared by mixing sawdust with zinc chloride. The shape of the particles depend on the method of grinding may differ which adsorptive properties. Powdered activated carbon have large pore diameters and small internal surface areas (Sufnarski, 1999). The size of powdered activated carbon is smaller than 325 mesh (0.025 mm) (Droste, 1997).

Figure 2.1 Powdered activated carbon.

2.1.1.2 Granular Activated Carbon

Granular activated carbon (Figure 2.2) has a larger particle size when compared with powdered activated carbon, but have higher internal surface. These carbons are preferred for all absorption of gasses and vapors as their rate of diffusion are faster. Granular activated carbon is used for separation of components of the flow system, deodorization, and water treatment. Granular activated carbon can be produced by crushing, pressing, and extruding. The size of granular activated carbon generally in from 40 mesh (0.425 mm) to 8 mesh (2.36 mm) (Droste, 1997).

Figure 2.2 Granular activated carbon.

2.1.2 Applicability of Activated Carbon

The porous carbon using dates starts back to 2000 B.C. in Egypt. They used for medicinal and purification (Patrick, 1995). In 1880, the carbon forms available. Porous carbons commonly obtained by carbonizing wood (charcoals), used as a medicine, deodorant, gunpowder, and pyrotechnics. At World War I, activated carbon was used for absorbing poisonous gasses in gas masks (Marsh and Rodriguez-Reinoso, 2006).

Nowadays, activated carbon has several applications in the food, pharmaceutical, chemical, petroleum, and wastewater industry. The most common impurities removed by activated carbon include aromatic hydrocarbon, phenol, pesticides, and carcinogenic compounds (Patrick, 1995).

In liquid application, granular activated carbon is desirable for continuous or cyclic processing using fixed-bed since pressure drop can be controlled by adjusting the particle-size distribution.

2.2 Surfactant

Surfactants are one of the most versatile products of the chemical industry, appearing in such diverse products as motor oils, pharmaceuticals, detergents, drilling muds, and flotation agents. The last decades have seen the extension of surfactant applications to such high-technology areas as electronic printing, magnetic recording, biotechnology, microelectronics, and viral research. (Rosen, 2012)

“Surfactant” is an acronym of “surface active agent”; which literally means active at a surface

Surfactants are amphipathic molecules that consist of a non-polar hydrophobic portion, usually a straight or branched hydrocarbon or fluorocarbon chain containing 8-18 carbon atoms, which is attached to a polar or ionic portion (hydrophilic). Therefore, a surfactant contains both a water-insoluble (or oil-soluble) component and a water-soluble component. Surfactants will diffuse in water and adsorb at interfaces between air and water or at the interface between oil and water, in the case where water is mixed with oil. The water-insoluble hydrophobic group may extend out of the bulk water phase, into the air or into the oil phase, while the water-soluble head group remains in the water phase.

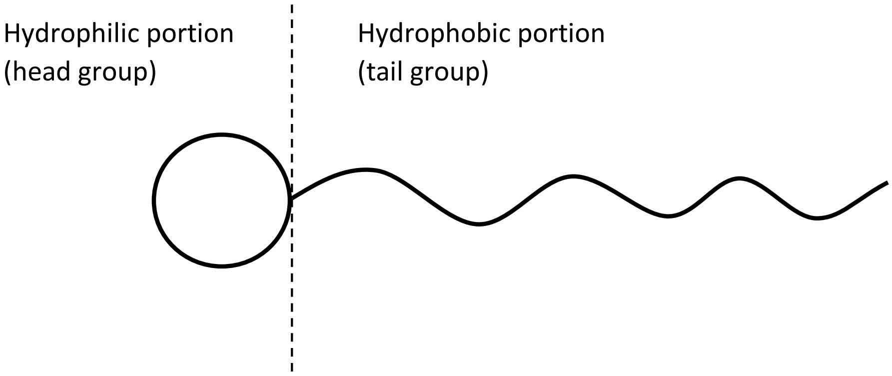

Figure 2.3 General representation of a surfactant molecule.

2.2.1 General Classification of Surfactants

A simple classification of surfactants based on the nature of the hydrophilic group is commonly used.

2.2.1.1 Anionic Surfactant

Anionic surfactant is the most widely used class of surfactants in industrial application due to their relatively low cost of manufacture and they are used in practically every type of detergent. The surface active portion of the molecule bears a negative charge, e.g. sodium dodecyl sulfate.

2.2.1.2 Cationic Surfactant

The most common cationic surfactant are the quaternary ammonium compounds with the general formula $R'R''R'''R''''N^+X^-$, where X^- is usually chloride ion and R represents alkyl group (Tadros, 2005). A widely used cationic surfactant is alkyl dimethyl ammonium chloride. Cationic surfactant is mainly used to adsorb at negatively charged surface, e.g. anticorrosive agents for steel.

2.2.1.3 Zwitterionic Surfactants

Zwitterionic (Amphoteric) surfactants are containing both cationic and anionic groups. The most common zwitterionic surfactants are the N-alkyl betaines. The main characteristic of zwitterionic surfactants is their dependence on the pH of the solution which they are dissolved. In acid solution, the molecule acquires a positive charge and behaves like a cationic surfactant, whereas in alkaline solution they become negatively charged and behaves like an anionic surfactant (Tadros, 2005).

2.2.1.4 Nonionic Surfactants

The most common nonionic surfactants are those based on ethylene oxide, referred to as ethoxylated surfactants, e.g. methyl ester ethoxylate.

2.2.2 General Effect of the Nature of the Hydrophobic Group

2.2.2.1 Length of the Hydrophobic Group

Increase in the length of the hydrophobic group decreases the solubility of the surfactant in water and increases its solubility in organic solvent, causes closer packing of the surfactant molecules at the interface, increases the tendency of the surfactant to adsorb an interface or to form aggregates, called micelle (Rosen, 2012)

2.2.2.2 Branching, Unsaturation

The introduction of branching or unsaturation into the hydrophobic group increases the solubility of the surfactant in water or in organic solvents, decreases the melting point of the surfactant and of the adsorbed film.

2.2.2.3 Aromatic Nucleus

The presence if an aromatic nucleus in the hydrophobic group may increase the adsorption of the surfactant onto polar surfaces and decrease its biodegradability.

2.2.3 Critical Micelle Concentration

The critical micelle concentration (CMC) is defined as the concentration of surfactants above which micelles form and all additional surfactants added to the system go to micelles. The CMC value determination can be made by using any of these physical properties, but most commonly, surface tension, the breaks in the electrical conductivity, light scattering (Ford *et al*., 1966).

2.2.4 Micellization

Micellization is the property that surfactant solutes have forming colloidal-sized clusters in the solution. In an aqueous media, when surfactants are at low concentration, the hydrophobic group distort the structure of the water and increase the free energy of the system. To minimize the free energy of the system, surfactants monomers orient hydrophobic group directed away from the solvent. The surfactant monomers concentrate at the surface by replacing molecules of the solvent at the interface. Therefore, the surface tension of solvent is decreased. If the surfactant concentration increases more than CMC. The interface is completely covered with surfactant monomers. After that, the energy of the system can be reduced by the aggregation of the surfactant monomers into clusters with their hydrophobic groups directed toward the solvents and form micelles.

2.2.5 Micellar Structure

A theory of micellar structure, base upon the geometry of various micellar shapes and the space occupied by the hydrophilic and hydrophobic groups of the surfactant molecules, has been developed by Israelachuili *et al*. (1976,1977) and Mitchell and Ninham (1981). The volume V_H occupied by the hydrophobic groups in the micellar core, the length if the hydrophobic group in core l_c, and the cross-sectional area a_o occupied by the hydrophilic group at the micelle-solution interface are used to calculate a “packing parameter,” V_H/l_ca_o, which determines the shape of the micelle (Rosen, 2012).

Table 2.1 Shape of the micelle at each value of packing parameter

Value of V_H/l_ca_o	**Structure of the Micelle**
$0 - \frac{1}{3}$	Spheroidal in aqueous media
$\frac{1}{3} - \frac{1}{2}$	Cylindrical in aqueous media
$\frac{1}{2} - 1$	Lamellar in aqueous media
>1	Inverse (reversed) micelle in nonpolar media

2.2.6 Solubilization by Solutions of Surfactants

Solubilization is one of the important properties of surfactants that is directly related to micelle formation. Solubilization may be defined as the spontaneous dissolving of a substance (solid, liquid, or gas) 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. Although both solvent-soluble and solvent-insoluble material may be dissolved by the solubilization mechanism. The importance of the phenomenon from the practical point of view is that it makes possible the dissolving of substances in solvents in which they are normally insoluble (Rosen, 2012).

This phenomenon has been widely used in industry as well as household application including detergency, agriculture, pharmaceuticals, cosmetics etc.

If the solubility of a normally solvent-insoluble material is plotted against the concentration of the surfactant solution that is solubilizing it, we find that the solubility is very low until reach a critical micelle concentration at which the solubility increases approximately linearly with concentration of the surfactant (Figure 2.4). This indicates that solubilization is a micellar phenomenon since it occurs only to a negligible extent at concentrations where micelles, of they exist at all, are found only in insignificant numbers.

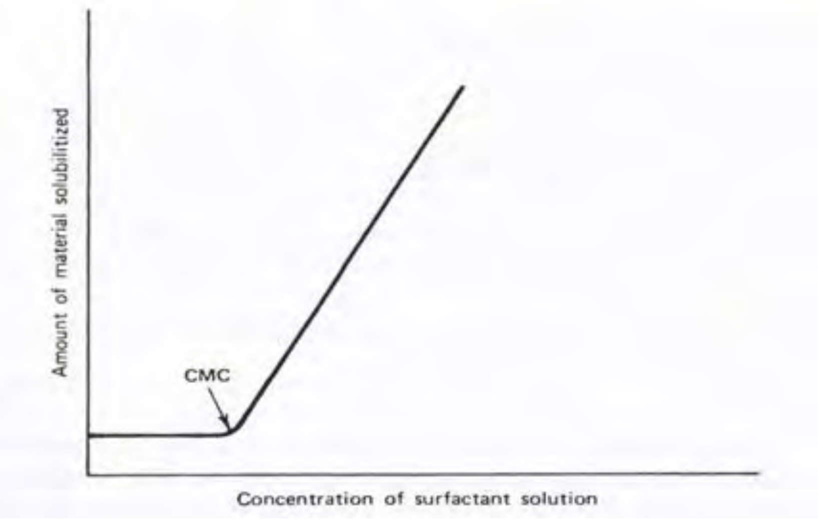

Figure 2.4 Plot of amount of material solubilized as a function of concentration of the surfactant in the bulk phase (Rosen, 2012).

In concentrated aqueous surfactant solution, although the shape of micelles may be different model that in dilute solution, the locus of solubilization for a particular type of solubilizate appears to be analogous in dilute solution that is polar molecules are solubilized mainly in outer regions of the micellar structures, whereas nonpolar solubilizates are contained in the inner portions.

Like surfactant monomers, organic solutes also distribute between aqueous phase and micellar phase. The solubilization equilibrium constant is defined as follows:

$$K = X_o/C_o$$

where $X_o = C_s/(C_m+C_s)$

K = solubilization equilibrium constant

C_o = the concentration of unsolubilized organic solute in the aqueous phase

C_s = the concentration of solubilizaed solute in the micelle

C_m = the concentration of surfactant in micelle form

2.2.7 Solubility-Temperature Relationship for Surfactants

Many Ionic surfactants show dramatic temperature-dependent solubility. The solubility may be very low at low temperatures and then increases by orders of magnitude in a relatively narrow temperature range. This phenomenon is generally denoted as the Krafft phenomenon, with the temperature for the onset of increasing solubility being known as the Krafft temperature. The latter may vary dramatically with subtle changes in the surfactant's chemical structure. In general, the Krafft temperature increases rapidly as the alkyl chain length of the surfactant increases. It also depends on the head group and counter ion. Addition of electrolytes increases the Krafft temperature.

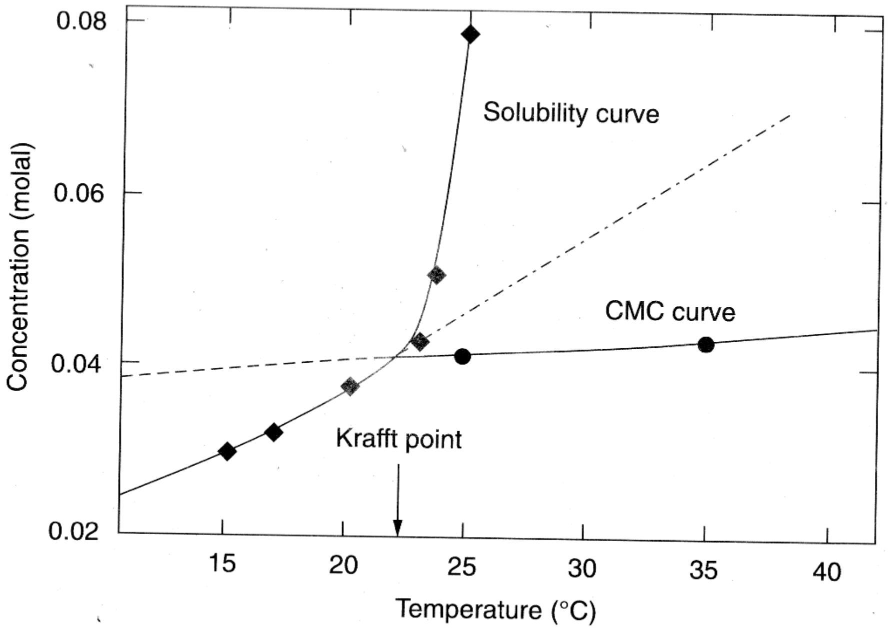

Figure 2.5 Solubility and CMC versus temperature for sodium decyl sulfonate in water (Tadros, 2005).

Figure 2.5 illustrates the rising of solubility with increasing of temperature for sodium decyl sulfonate in water. The solubility of the surfactant clearly increases rapidly above 22 °C. The Krafft temperature is very important in the application of surfactants. As mention above, the solubility of a surfactant increases significantly above the Krafft temperature and, hence, most industrial applications require surfactants with a low Krafft temperature.

2.2.8 Adsorption of Surfactant on Activated Carbon

Adsorption isotherms for well-purified monofunctional anionic and cationic surfactants are similar on activated carbon and are the Langmuir-type adsorption. They appear to show surface saturation in the vicinity of the CMC of the adsorbate with an orientation of the adsorbate perpendicular to the substrate. Adsorption onto these substrates in mainly by dispersion forces. The orientation of adsorbate initially maybe parallel to the surface of the solid or slightly tilted or L-shaped, with the hydrophobic groups close to the surface and the hydrophilic group oriented toward the aqueous phase. As adsorption continues, the adsorbed molecules may become more oriented and perpendicular to the surface with hydrophilic heads oriented toward the water.

The rate of adsorption has been shown to be a function of the portion of the hydrophilic group in the molecules, with surfactants containing the hydrophilic group in a central location in the molecule adsorbing faster than those in which the hydrophilic group is terminally located. Moreover, the rate of adsorption on carbon also have been shown to be dependent on the presence in the aqueous phase of additives that affect the structure of water.

Neutral electrolyte addition increases both efficiency of adsorption of ionic surfactants by decreasing the electrical repulsion between the similarly charged adsorbed ions and oncoming ions. The addition of small amounts of cationic to aqueous solution of anionic, or small amounts of metal carboxylates to cationic solutions has also been shown to increase the adsorption of the predominant ionic surfactant.

2.3 Surfactant-Enhanced Carbon Regeneration

In surfactant-enhanced carbon regeneration, the surfactant solution contains surfactant above the critical micelle concentration. The adsorbate solute solubilizes into the micelles in the surfactant solution.

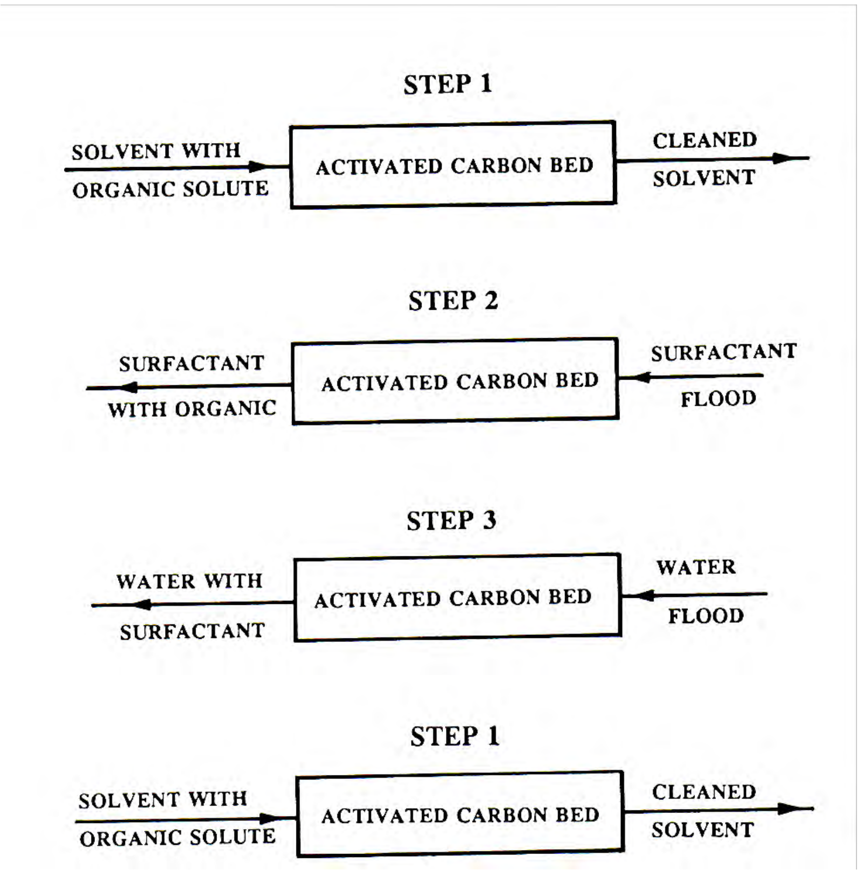

Figure 2.6 Surfactant-enhanced carbon regeneration process (Roberts, 1989).

From Figure 2.5, organic solute was adsorbed by activated carbon. The cleaned solvent come out from adsorber. The regeneration process was started at step 2. Surfactant solution flow though activated carbon bed and adsorbates are removed from activated carbon. For the third step, the activated carbon was washed with water to remove the remaining surfactant.

A surfactant solution is passed through the spent activated carbon bed. The organic adsorbate is desorbed and solubilized into micelle in the solution. The micelle has a hydrocarbon like interior into which organic molecules will dissolve or solubilize as shown in Figure 2.6.

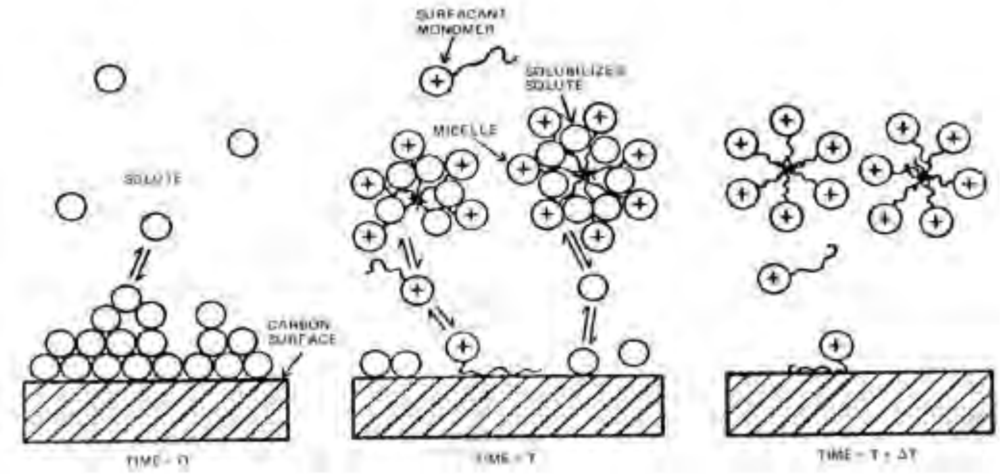

Figure 2.7 Equilibria present at the various stages of a surfactant-enhanced carbon regeneration (Scamehorn, 1989).

The surfactant can be removed from the effluent solution exiting the adsorber by a precipitation process. This allows the surfactant to be recycled to the feed, which greatly improves the economics of this process. The desorbed organic may now be present at concentrations greater than saturation, resulting in a separate organic-rich phase splitting out for direct reuse. The saturated water could be recycled fir use in the next regeneration cycle. Alternatively, if the organic solute is very soluble in the water, a very concentrated aqueous stream results from which the organic can be separated by conventional techniques.

Blakeburn and Scamehorn (1989) found that a surfactant solution stream containing a cationic surfactant can remove of approximately 80% tert-butylphenol with a reasonable volume of surfactant solution. Removal of the last 20% of the adsorbate from the carbon is very difficult due to these adsorbate being chemisorbed instead of physically adsorbed. The residual surfactant can be washed off by a water flush step using a large volume of water.

Roberts *et al.* (1989) investigated the regeneration of activated carbon containing toluene and amyl acetate. The effect of the surfactant solution flow rate and surfactant concentration show that the removal of toluene was nearly equilibrium limited, while amyl acetate showed mass transfer resistance. The residual surfactant on carbon was removed by water flush.

Bhummasobhana *et al.* (1995) also studied the effect of regeneration and flushing conditions on the breakthrough of the phenol in subsequent adsorption cycles. And hence, on effective adsorption capacity upon repeated usage. It showed that the reduction of the effective carbon adsorption capacity is less if a higher surfactant solution concentration, more pore volume of the surfactant solution, or more pore volumes of flushing water are used. The activated carbon regenerated by the SECR in liquid phase application can be effective in subsequent adsorptions.

Sutad na Ayoothaya *et al.* (1996) studied the carbon regeneration by using sodium dodecyl sulfate. The effect of the surfactant solution flow rate and surfactant concentration indicate that the trichloroethylene removal of the solute exhibits significant mass transfer resistant during the regeneration step. However, a moderate reduction in adsorption capacity in subsequent loadings was found after the regeneration compared to virgin carbon.

2.4 Sodium Dodecyl Sulfate

Sodium dodecyl sulfate (SDS) is a synthetic organic compound with the formula $CH_3(CH_2)_{11}SO_4Na$. It is an anionic surfactant used in many cleaning and hygiene products. The sodium salt is of an organosulfate class of organics. It consists of a 12-carbon tail attached to a sulfate group. SDS is a common component of many domestic cleaning, personal hygiene and cosmetic, pharmaceutical, and food products, as well as of industrial and commercial cleaning and product formulations.

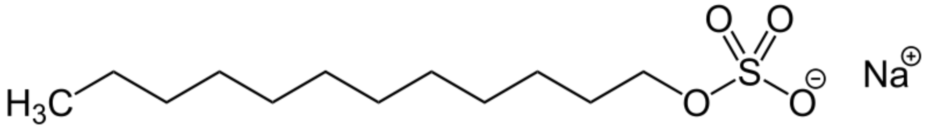

Figure 2.8 Sodium dodecyl sulfate.

SDS is synthesized by treating lauryl alcohol with sulfur trioxide gas, oleum, or chlorosulfuric acid to produce hydrogen lauryl sulfate. The resulting product is then neutralized through the addition of sodium hydroxide or sodium carbonate. Lauryl alcohol can be used in pure form or may be derived from either coconut or palm kernel oil by hydrolysis (which liberates their fatty acids), followed by hydrogenation.

The molecular weight of SDS is 288.37 g/mol. The critical micelle concentration (CMC) in pure water at 25 °C is 8.2×10^{-3} M.

2.5 Methyl Ester Sulfonate

Methyl Ester Sulfonate is a new anionic surfactant made from natural plant, animal oil, or fat. It is characterized by good wetting, emulsifying, softening and resistance to hard water, and good solubility, high degradation rate, little stimulation to skin, high frothing ability. Especially in hard water, its frothing ability is better than Linear Alkyl Benzene Sulphonate (LAS) and SDS. With good decontaminating ability, especially under the existence of non-phosphor agent, it is suitable for confecting low-phosphor and non-phosphor scour. It has high dispersing power of calcium soap, used as the active matter and disperser of calcium soap in soap, compound soap, and soap powder. It can improve and adjust the viscosity of paste detergent and wash powder slurry. The liquid detergent confected with it has high cleaning power and good low-temperature stability. It is also applicable in many industrial fields, such as emulsifier agent of plastic industry, demolding agent of rubber product, dyeing agent, pigment, dye of printing and dyeing industry, wetting agent and dispersant of agricultural chemicals, etc.

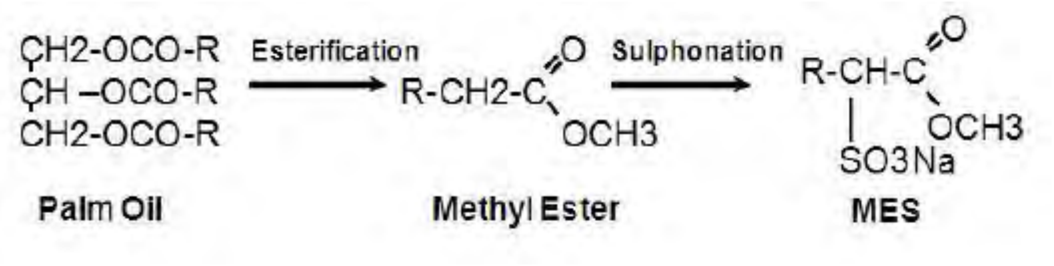

Figure 2.9 MES synthesis reaction.

MES is produced by the reaction of SO_3 with fatty acid methyl ester (derived from triglycerides by transesterification with methanol). Generally, MES has 12-18 carbon atoms.

CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Spent Activated Carbon

Sample of spent activated carbon was used in this study is a granular activated carbon contaminated mainly by benzene and phenol in liquid phase application. The size of spent sample 8x30 mesh.

3.1.2 Sodium Dodecyl Sulfate (SDS)

Commercial grade SDS (96% purity) was purchased from Sigma-Aldrich. It has an average molecular weight of 288.37 and critical micelle concentration or CMC at 25°C is $8.2\text{x}10^{-3}$ M and the Krafft temperature of SDS is 16°C.

3.1.3 Methyl Ester Sulfonate (MES)

Commercial grade MES was obtained from Bellestra SpA, Italy with a total active matter higher than 89.4%. The MES contained a narrow distribution of hydrophobic chain lengths between C_{16} to C_{18} (approximately 45% C_{16} and 55% C_{18}). It was reported to have an average molecular weight of 382 and critical micelle concentration at 25°C was $3.5\text{x}10^{-4}$ M. the Krafft Temperature of this MES is 20°C.

3.2 Equipment

3.2.1 Adsorption Column

A 1.9 cm diameter, 30 cm long jacketed column made from acrylic was used for the experiment. The column can contain 50 g of activated carbon. The column temperature was controlled by water bath. A Masterflex laboratory/standard pump maintained a constant flow rate as the surfactant solution or flushing water was pumped through the bed. The diagram of adsorption column is shown in Figure 3.1.

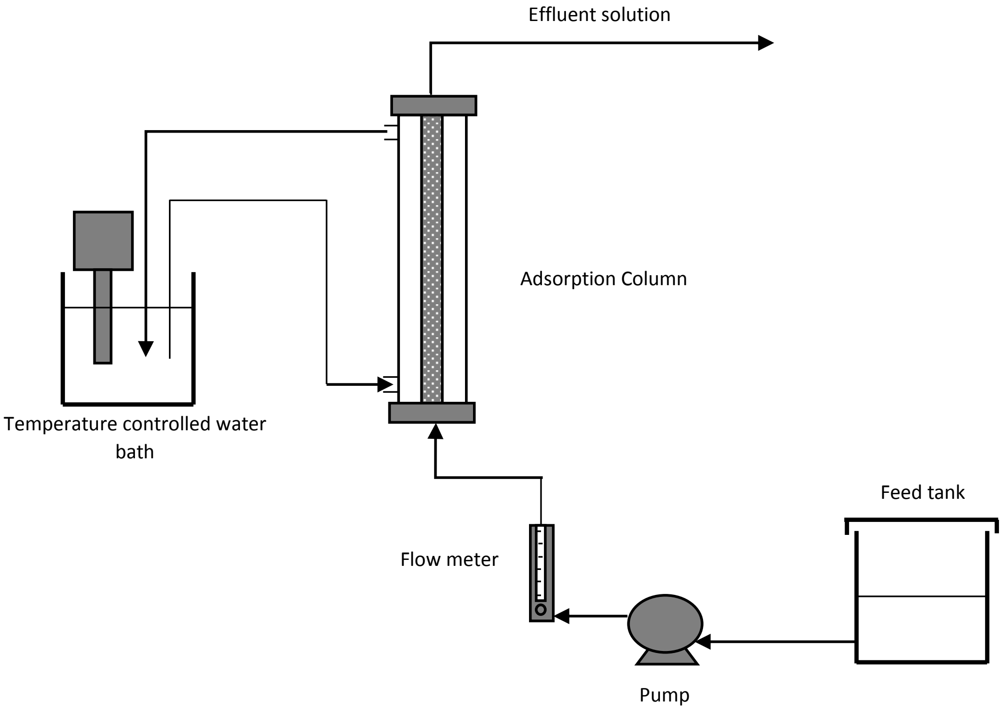

Figure 3.1 Schematic diagram of experimental apparatus.

3.2.2 UV-Visible Spectrophotometer

UV-Visible spectrophotometer (UV-1800), Shimadzu, was used in this experiment for analyzing organic compound. The samples were diluted before the analysis for keeping the absorbance of sample lower than 1 (Lambert's Law). The wavelength of maximum absorbance (λ_{max}) of benzene and phenol are 255 and 271 nm.

3.2.3 X-Ray Fluorescence Spectrometer

In order to find the elemental composition, the sample was investigated by using X-Ray fluorescence spectrometer (XRF). The sample was mashed and mixed with a binder (boric acid). Since the XRF cannot detect the elements that have atom number lower than 11, total ash content analysis is needed to find the composition of carbon, organic compound and moisture.

3.2.4 Conductivity Meter

Conductivity meter was used to find the amount of surfactant concentration. The electrical conductivity increases as the concentration of surfactant increases. The relationship between the surfactant concentration (SDS and MES) and the electrical conductivity were created for using as the indicator to find the concentration of surfactant in the effluent.

3.3 Methodology

3.3.1 Spent Activated Carbon Characterization

The activated carbon was characterized by following methods before the regeneration step.

Table 3.1 Method for spent activated carbon characterization

Characterization	Method
Moisture content	ASTM D 2867
Volatile matter	ASTM D 5832
Total ash content	ASTM D 2866
Elemental composition	X-ray Fluorescence Spectrometer (XRF)
Organic compound on the activated carbon surface	Dissolved the compound on the activated carbon surface by n-hexane and analyzed by using UV-Visible Spectrophotometer (UV-1800)

In order to estimate the amount of adsorbed benzene and phenol on the spent activated carbon, 10 g of spent activated carbon was soaked in 100 ml of n-hexane to extract the organic compounds. The extract was analyzed and the process was repeated until no further benzene or phenol was detected in the extractant.

3.3.2 Regeneration

Prior to the regeneration step, the salts on the spent activated carbon was washed with deionized water until the conductivity is zero because the subsequent surfactant concentration is known by conductivity. However, this step may not be necessary if the process is used in the real application. Subsequently, the amount of organic compound was analyzed again. To perform the regeneration step, 50 g of spent activated carbon was placed into the adsorption column. Surfactant solution (SDS or MES) flowed through the packed of spent activated carbon for 5 hours. The parameters are the surfactant concentration (0.01, 0.03 and 0.05 M), solution flow rate (20 and 40 ml/min) and column temperature (30 and 60°C). The effluent was collected and analyzed for the amount of organic compound by using UV-Visible Spectrophotometer. The surfactant concentration in the effluent during the regeneration process was observed by using conductivity meter.

3.3.3 Water Flushing

After the regeneration step, deionized water was flowed through the packed of activated carbon for 1 hour to wash out the residual surfactant. The water flushing flow rate was varied at 100 and 120 ml/min. The effluent was collected and investigated for the amount of surfactant by using conductivity meter.

CHAPTER IV
RESULTS AND DISCUSSION

4.1 Spent Activated Carbon Characterization

The spent activated carbon for this study was characterized and the results are shown in Table 4.1. The moisture and volatile matter are about 55% weight of the spent activated carbon. After treating in furnace at 950°C, 90% weight of the spent activated carbon was burned off and the remained ash is 10% by weight. The ash composition is inorganic compound and from the XRF data, calcium, phosphorus, silicon, and iron are the main component of the ash.

Table 4.1 Spent activated carbon characterization

Characterization	**wt %**
Moisture content	27.5
Volatile matter	27.2
Total ash content	10.0
Elemental composition (in total ash content)	Calcium 3.1 Phosphorus 1.5 Silicon 1.5 Iron 1.5 Other (Al, Cl, K, and etc.) 2.4

For the amount of organic compound on the activated carbon surface (Table 4.2), the organic compound was leached from the surface of the activated carbon by using n-hexane and analyzed by UV-Visible Spectrophotometer (UV-1800). The results were calculated to weight percentage base on the volatile matter. The main organic compound was benzene at 18.4% in spent activated carbon. The amount of benzene removed from the spent activated carbon was selected to be the main factor for regeneration step.

Table 4.2 The amount of organic compound

Organic compound	% in spent carbon
Benzene	18.4
Phenol	7.4

4.2 Regeneration

Before the regeneration step, activated carbon sample was washed by using deionized water to remove the salts on the sample that interfere the value of electrical conductivity during the regeneration step. After that, the amount of benzene was recalculated. The new value of the amount of benzene is 13% in the spent activated carbon. During the process, the adsorbate started to be removed from the surface of spent activated carbon by surfactant solution and came out with the effluent.

4.2.1 Effect of Surfactant Concentration

At the initial stage, the outlet surfactant concentration was less than the inlet surfactant concentration until the steady state has reached the inlet and outlet surfactant concentration are equaled. After 5 hour of the regeneration process, the percentage of benzene removed was calculated from the total amount of benzene in the effluent solution received from UV-1800 results.

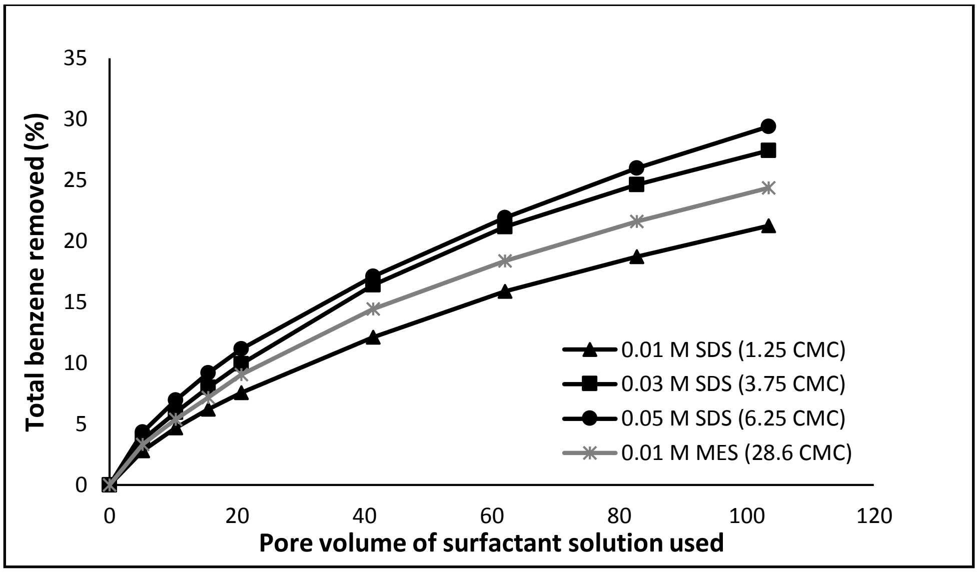

Figure 4.1 The effect of surfactant concentration on the total benzene removed at flow rate 20 ml/min and 30°C.

As shown in Figure 4.1, the effect of surfactant concentration on the total benzene removed is presented and compared with the pore volume of surfactant solution used. In this research, the pore of the spent activated carbon bed in the regeneration column can contained the surfactant solution feed 58 ml. In consequence, 1 pore volume of surfactant solution used is 58 ml of surfactant solution that was fed through the carbon bed. As expected, the benzene concentration in the effluent is extremely high when it first appeared and then slightly lower over time. After 5 hour, the benzene removals are 21, 28, and 30% for 0.01, 0.03, and 0.05 M of SDS respectively. The total benzene removed increases as the concentration of SDS increased because the higher amount of surfactant leads to more surfactant micelle formation and then more benzene is solubilized from the spent activated carbon. Nevertheless, benzene removal between concentration of SDS 0.03 M and 0.05 M are not significant possibly due to the limit of mass transfer. At the same concentration, the benzene removal by MES is higher than SDS because MES has a much lower CMC than SDS. Moreover, MES has longer hydrophobic tail group (C_{16} and C_{18}) than the SDS (C_{12}). Therefore, the solubility of organic compounds (e.g. benzene) in MES micelle should be higher (Rosen and Kunjappu, 2012).

However, the process is required longer operation time because of the low total benzene removed in order to maximize the benzene removal of this process.

4.2.2 Effect of Surfactant Solution Flow Rate

The effect of surfactant solution flow rate is shown in Figure 4.2, the total benzene removed increases as the surfactant flow rate is increased and it took up to 31% at 40 ml/min flow rate for 0.01 M of SDS. With a higher flow rate of the surfactant solution, there are more moles of SDS passing through the bed, so the rate of benzene removal is higher. In other words, the rate of benzene removal depends only on the total number of mole of SDS in the surfactant solution passing through the bed (Thamtarai, 2007).

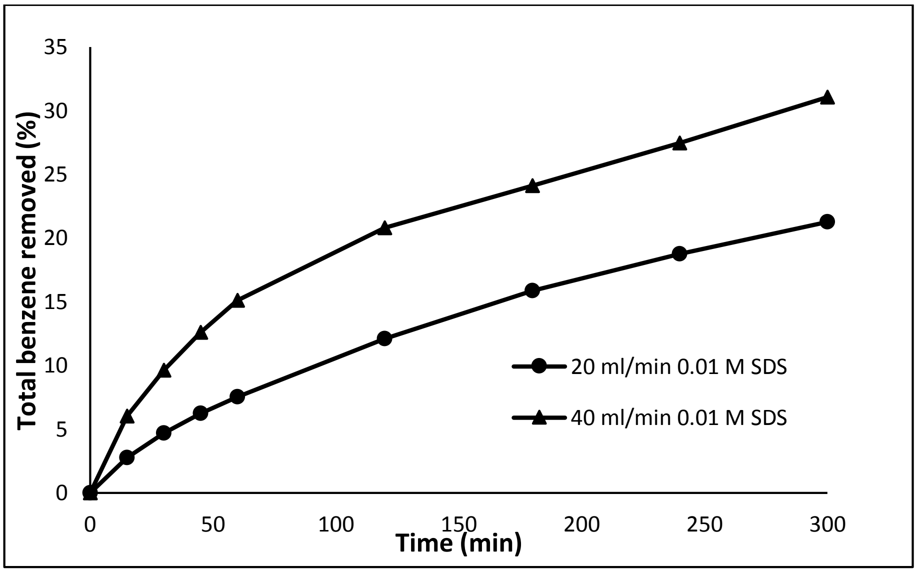

Figure 4.2 The effect of surfactant solution flow rate on the total benzene removed at 30°C per time.

On the other hand, the results at the same amount of surfactant solution (pore volume of surfactant solution used) was compared as shown in Figure 4.3, the total benzene removed of both flow rate are less different. Hence, the results is supported the previous reason as well. The lower flow rate used, the longer regeneration time required to remove benzene to specific levels.

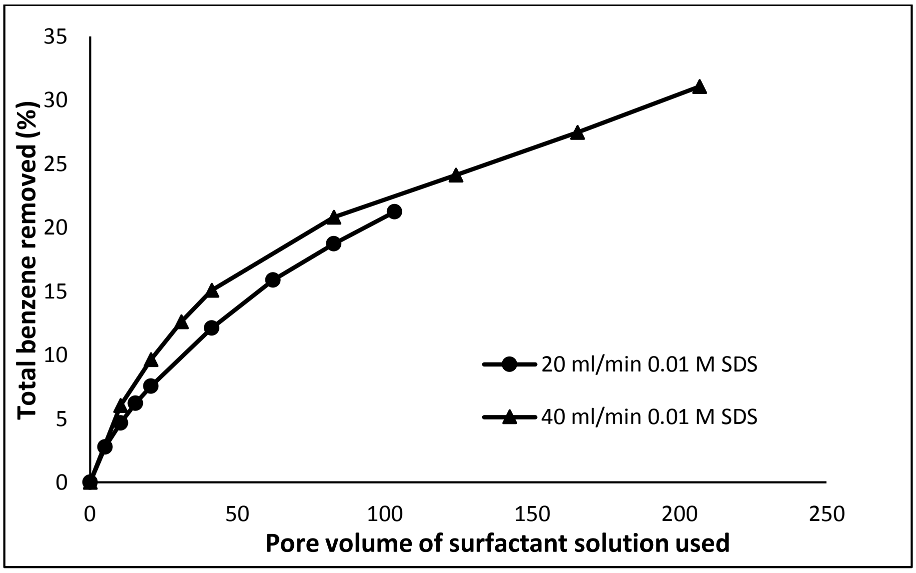

Figure 4.3 The effect of surfactant solution flow rate on the benzene removal at 30°C per pore volume of surfactant solution used.

4.2.3 Effect of Column Temperature

For the effect of column temperature, the total benzene removed increases as the column temperature increased as illustrated in Figure 4.4. The operation temperatures were selected over the Krafft temperature of both surfactants (16 °C for SDS and 20 °C for MES). Therefore, all of the surfactant molecules could form the micelles. The heat from the jacket of adsorption column transferred to the surfactant solution and heated it to higher temperature (at 60°C). Increasing of the column temperature from 30 °C to 60 °C can raised the benzene removal 7% for SDS and 10% for MES because the solubility of the surfactant in an organic solvent was increased and also basically because of an increased thermal agitation which increases the available space for solubilization in the micelle (Singh, 2012). Even though the Krafft temperature of SDS is lower than MES, The removal of benzene of MES is still higher than SDS at the same feed pore volume and temperature because the CMC of MES is very low when compared with the CMC of SDS.

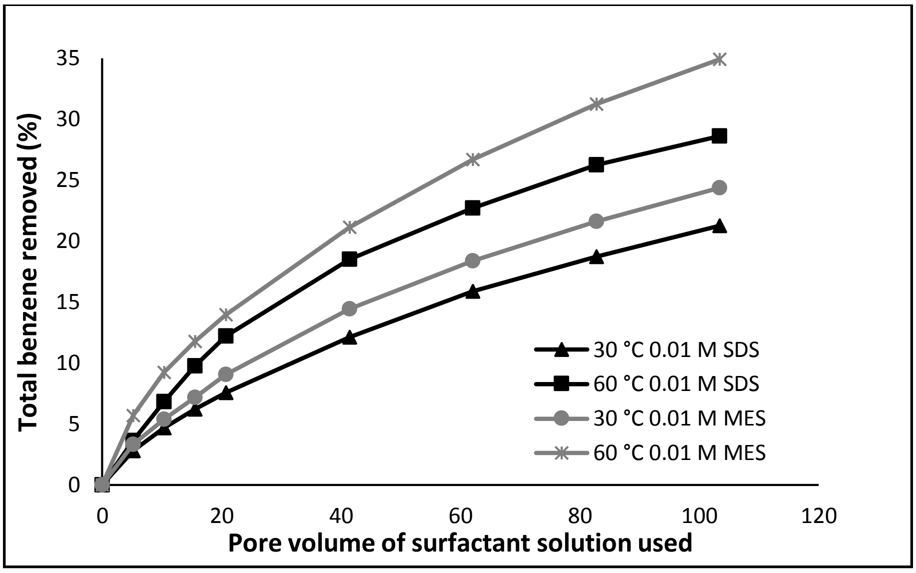

Figure 4.4 The effect of column temperature on the total benzene removed at flow rate 20 ml/min.

4.3 Water Flushing

Before starting the water flushing step, the surfactant solution was drained from the column and the pipeline was cleaned by deionized water to prevent the surfactant solution mixed with flushing water.

4.3.1 Effect of Flushing Water Flow Rate

During the water flushing step, the adsorbed surfactant is removed from activated carbon by deionized water. The flushing water can removed only 82% of SDS and 77% of MES from the surface of activated carbon. As shown in Figure 4.5, the flushing water flow rate has small effect on the surfactant removal for both SDS and MES since the less different flushing water flow rate. The higher flow rate can remove the surfactant faster. The SDS has slightly higher surfactant removal because of the smaller size of surfactant molecule and higher solubility of SDS in water. The deionized water cannot removed all of the remaining surfactant on the activated carbon surface, indicating that this step is also mass transfer limited.

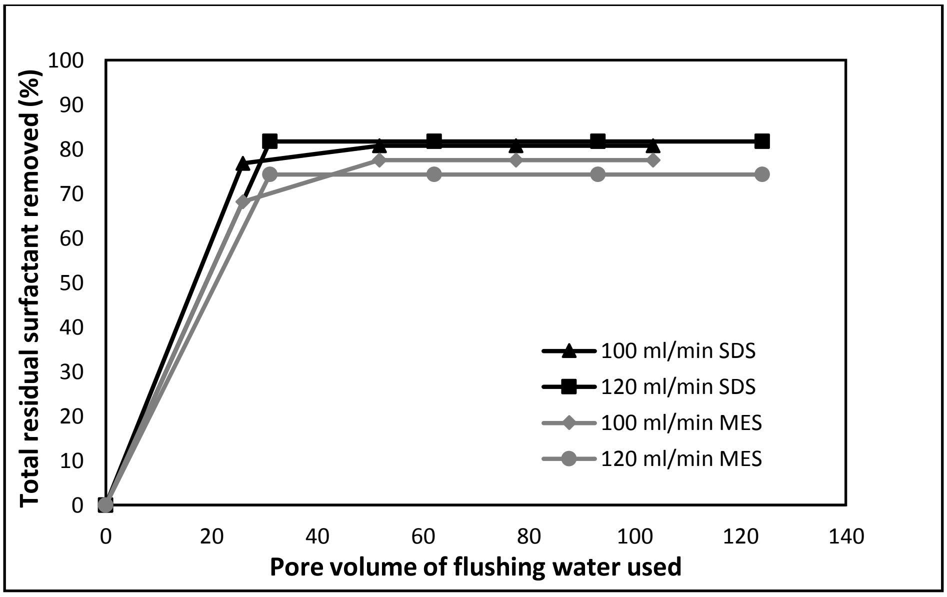

Figure 4.5 The effect of flushing water flow rate on the total residual surfactant removed at 30˚C.

CHAPTER V
CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The benzene is the main organic compound on the surface of the spend activated carbon. The moisture content and the volatile matter of the spent activated carbon are 27.5 and 27.2 wt %. In the ash, the main elements are calcium, phosphorus, silicon, and iron.

The benzene on the spent activated carbon is removed by using anionic surfactant such as SDS and MES by using surfactant-enhanced carbon regeneration process and the highest total benzene removed in this experiment is 35% in 5 hr at flow rate 20 ml/min and 60°C by using MES. The concentration of surfactant, solution flow rate and column temperature affect to the benzene removal. The higher surfactant concentration, flow rate and column temperature leads to higher total benzene removed from the surface of spent activated carbon for both SDS and MES. MES is more effective than SDS due to faster regeneration rate at the same condition. After the regeneration process, the water flushing step is need since some of surfactant was remained on the surface of the regenerated activated carbon and it can be removed by water flushing like deionized water but not all of the residual surfactant can be removed and total residual surfactant removed increases as the flow rate of flushing water increased.

In summary, Surfactant-enhanced carbon regeneration shows great promise as an alternative way to regenerate spent activated carbon and nondestructive carbon regeneration technique that may find wide use in future applications.

5.2 Recommendations

In the regeneration step, the process is required longer operation time because of the low benzene removed in order to maximize the efficiency of this process. The process can be studied in other parameter and type of surfactant to increase the capacity of the process. For water flushing step, the range of flushing water flow rate should be more different to make the results are easier to be observed.

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APPENDIX

Table A-1 X-Ray fluorescence spectrometer result

Activated carbon weight = 2.007 g Weight after pressing = 7.009 g

Compound Formular	Concentration (%)
Al	7.174
Si	14.658
P	14.824
Cl	5.999
K	2.746
Ca	32.114
Ti	0.905
Mn	0.863
Fe	14.450
Ni	0.801
Cu	1.042
Zn	0.704
Sr	0.610
Zr	0.518
La	2.591
	99.999

Table A-2 Total benzene removed at SDS concentration 0.01 M on effect of concentration

Total benzene (salt removed) = 13.05 g Surfactant solution flow rate = 20 ml/min

Column temperature = 30°C 1 pore volume = 58 ml of SDS solution

Time (min)	Pore volume	Concentration (g/L)	Benzene mass rate (g/min)	Benzene removed (g)	Acc. benzene removed (g)	Benzene removal (%)
0	0	0.73484	0.01470	0	0	0
15	5.17	0.46767	0.00935	0.18038	0.18038	2.76520
30	10.34	0.36397	0.00728	0.12475	0.30512	4.67757
45	15.52	0.30198	0.00604	0.09989	0.40502	6.20893
60	20.69	0.27651	0.00553	0.08677	0.49179	7.53918
120	41.38	0.21851	0.00437	0.29701	0.78880	12.09242
180	62.07	0.19086	0.00382	0.24562	1.03442	15.85784
240	82.76	0.12143	0.00243	0.18737	1.22180	18.73030
300	103.45	0.15138	0.00303	0.16369	1.38548	21.23963

Table A-3 Total benzene removed at SDS concentration 0.03 M on effect of concentration

Total benzene (salt removed) = 13.05 g Surfactant solution flow rate = 20 ml/min

Column temperature = 30°C 1 pore volume = 58 ml of SDS solution

Time (min)	Pore volume	Concentration (g/L)	Benzene mass rate (g/min)	Benzene removed (g)	Acc. benzene removed (g)	Benzene removal (%)
0	0	1.09840	0.02197	0	0	0
15	5.17	0.51432	0.01029	0.24191	0.24191	3.70848
30	10.34	0.45123	0.00902	0.14483	0.38674	5.92878
45	15.52	0.44126	0.00883	0.13387	0.52061	7.98108
60	20.69	0.40221	0.00804	0.12652	0.64713	9.92066
120	41.38	0.30109	0.00602	0.42198	1.06911	16.38969
180	62.07	0.21790	0.00436	0.31139	1.38051	21.16338
240	82.76	0.15913	0.00318	0.22622	1.60673	24.63133
300	103.45	0.14287	0.00286	0.18120	1.78793	27.40915

Table A-4 Total benzene removed at SDS concentration 0.05 M on effect of concentration

Total benzene (salt removed) = 13.05 g

Surfactant solution flow rate = 20 ml/min

Column temperature = 30°C

1 pore volume = 58 ml of SDS solution

Time (min)	Pore volume	Concentration (g/L)	Benzene mass rate (g/min)	Benzene removed (g)	Acc. benzene removed (g)	Benzene removal (%)
0	0	1.22840	0.02457	0	0	0
15	5.17	0.65084	0.01301	0.28189	0.28189	4.32135
30	10.34	0.48740	0.00975	0.17074	0.45262	6.93876
45	15.52	0.47896	0.00958	0.14495	0.59758	9.16092
60	20.69	0.37408	0.00748	0.12796	0.72553	11.12250
120	41.38	0.27667	0.00553	0.39045	1.11598	17.10815
180	62.07	0.24414	0.00488	0.31249	1.42847	21.89861
240	82.76	0.19672	0.00393	0.26451	1.69298	25.95367
300	103.45	0.17418	0.00348	0.22254	1.91552	29.36524

Table A-5 Total benzene removed at MES concentration 0.01 M on effect of concentration

Total benzene (salt removed) = 13.05 g Surfactant solution flow rate = 20 ml/min

Column temperature = 30^{o}C 1 pore volume = 58 ml of MES solution

Time (min)	Pore volume	Concentration (g/L)	Benzene mass rate (g/min)	Benzene removed (g)	Acc. benzene removed (g)	Benzene removal (%)
0	0	0.73484	0.01470	0	0	0
15	5.17	0.46767	0.00935	0.18038	0.18038	2.76520
30	10.34	0.36397	0.00728	0.12475	0.30512	4.67757
45	15.52	0.30198	0.00604	0.09989	0.40502	6.20893
60	20.69	0.27651	0.00553	0.08677	0.49179	7.53918
120	41.38	0.21851	0.00437	0.29701	0.78880	12.09242
180	62.07	0.19086	0.00382	0.24562	1.03442	15.85784
240	82.76	0.12143	0.00243	0.18737	1.22180	18.73030
300	103.45	0.15138	0.00303	0.16369	1.38548	21.23963

Table A-6 Total benzene removal at surfactant solution flow rate 40 ml/min on effect of surfactant solution flow rate

Total benzene (salt removed) = 13.05 g SDS concentration = 0.01 M

Column temperature = 30°C 1 pore volume = 58 ml of SDS solution

Time (min)	Pore volume	Concentration (g/L)	Benzene mass rate (g/min)	Benzene removed (g)	Acc. benzene removed (g)	Benzene removal (%)
0	0	0.84151	0.03366	0	0	0
15	10.34	0.46812	0.01872	0.39289	0.39289	6.023041
30	20.69	0.31561	0.01262	0.23512	0.62801	9.627447
45	31.03	0.32984	0.01319	0.19364	0.82164	12.5959
60	41.38	0.21101	0.00844	0.16226	0.98390	15.08329
120	82.76	0.10022	0.00401	0.37348	1.35737	20.80873
180	124.14	0.07971	0.00319	0.21592	1.57329	24.11875
240	165.52	0.10154	0.00406	0.21750	1.79079	27.45305
300	206.90	0.09413	0.00377	0.23480	2.02559	31.05263

Table A-7 Total benzene removed at column temperature 60°C on effect of column temperature (SDS)

Total benzene (salt removed) = 13.05 g SDS concentration = 0.01 M

Surfactant solution flow rate = 20 ml/min 1 pore volume = 58 ml of SDS solution

Time (min)	Pore volume	Concentration (g/L)	Benzene mass rate (g/min)	Benzene removed (g)	Acc. benzene removed (g)	Benzene removal (%)
0	0	0.86625	0.01733	0	0	0
15	5.17	0.70269	0.01405	0.23534	0.23534	3.60781
30	10.34	0.69380	0.01388	0.20947	0.44482	6.81907
45	15.52	0.58410	0.01168	0.19169	0.63650	9.75762
60	20.69	0.47124	0.00943	0.15830	0.79480	12.18440
120	41.38	0.21341	0.00427	0.41079	1.20559	18.48186
180	62.07	0.24394	0.00488	0.27441	1.48000	22.68861
240	82.76	0.14310	0.00286	0.23222	1.71223	26.24863
300	103.45	0.11197	0.00223	0.15304	1.86527	28.59479

Table A-8 Total benzene removed at column temperature 60°C on effect of column temperature (MES)

Total benzene (salt removed) = 13.05 g MES concentration = 0.01 M

Surfactant solution flow rate = 20 ml/min 1 pore volume = 58 ml of MES solution

Time (min)	Pore volume	Concentration (g/L)	Benzene mass rate (g/min)	Benzene removed (g)	Acc. benzene removed (g)	Benzene removal (%)
0	0	1.52510	0.03050	0	0	0
15	5.17	0.94645	0.01893	0.37073	0.37073	5.68338
30	10.34	0.59143	0.01183	0.23068	0.60142	9.21977
45	15.52	0.50037	0.01001	0.16377	0.76519	11.73038
60	20.69	0.46541	0.00931	0.14487	0.91005	13.95121
120	41.38	0.31208	0.00624	0.46649	1.37655	21.10263
180	62.07	0.29451	0.00589	0.36395	1.74050	26.68209
240	82.76	0.20158	0.00403	0.29765	2.03815	31.24517
300	103.45	0.19413	0.00388	0.23743	2.27558	34.88494

Table A-9 Total residual surfactant removed at flushing water flow rate 100 ml/min (SDS)

Total residual surfactant on the activated carbon surface = 0.002605 mole

1 pore volume = 58 ml of deionized water

Time (min)	Pore volume	Conductivity (μS)	Acc. surfactant removed (mole)	Residual surfactant removal (%)
0	0	271	0	0
15	25.86	15	0.0020	76.79
30	51.72	0	0.0021	80.83
45	77.59	0	0.0021	80.83
60	103.45	0	0.0021	80.83

Table A-10 Total residual surfactant removed at flushing water flow rate 120 ml/min (SDS)

Total residual surfactant on the activated carbon surface = 0.004735 mole

1 pore volume = 58 ml of deionized water

Time (min)	Pore volume	Conductivity (μS)	Acc. surfactant removed (mole)	Residual surfactant removal (%)
0	0	461	0	0
15	31.03	0	0.0039	81.74
30	62.07	0	0.0039	81.74
45	93.10	0	0.0039	81.74
60	124.14	0	0.0039	81.74

Table A-11 Total residual surfactant removed at flushing water flow rate 100 ml/min (MES)

Total residual surfactant on the activated carbon surface = 0.002972 mole

1 pore volume = 58 ml of deionized water

Time (min)	Pore volume	Conductivity (µS)	Acc. surfactant removed (mole)	Residual surfactant removal (%)
0	0	214	0	0
15	25.86	34	0.0020	68.17
30	51.72	0	0.0023	77.52
45	77.59	0	0.0023	77.52
60	103.45	0	0.0023	77.52

Table A-12 Total residual surfactant removed at flushing water flow rate 120 ml/min (MES)

Total residual surfactant on the activated carbon surface = 0.005662 mole

1 pore volume = 58 ml of deionized water

Time (min)	Pore volume	Conductivity (µS)	Acc. surfactant removed (mole)	Residual surfactant removal (%)
0	0	429	0	0
15	31.03	0	0.0042	74.29
30	62.07	0	0.0042	74.29
45	93.10	0	0.0042	74.29
60	124.14	0	0.0042	74.29

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Proceedings:

1. Kallapadee, B. and Kitiyanan, B. (2015, April 21) Use of anionic surfactant in regeneration of spent activated carbon. Proceedings of the 6th Research Symposium on Petrochemical and Materials Technology and the 21st PPC Symposium on Petroleum, Petrochemicals, and Polymers, Bangkok, Thailand.