CERTIFICATION OF APPROVAL

Characterization and Adsorption Isotherm Measurement of Phenol On Activated Carbon

by

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CERTIFICATION OF ORIGINALITY

This is to verify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

NAZATUL SHEEMA BINTI CHE DIN

ABSTRACT

The research on adsorption isotherm measurement of phenol on activated carbon has been conducted prior to the increase in demand by the industry regarding the suitable method of removing this organic compound. Phenol is one of the components in industrial wastewaters which can bring detrimental effect to human life. Due to the increase in industrial wastewaters, the demand for removal of organic compounds including phenol has been increased. There are various separation method recognized such as coagulation, filtration, oxidation, aeration, ion exchange and activated carbon treatment. However, the latter method has been proven to be more efficient in removing these compounds, thus is adopted in this study.

The objectives of this research are to determine the adsorption isotherm measurement of phenol on various types of activated carbon at wide range of temperatures, to determine the most suitable adsorbent for this type of application and to characterize the physical properties of the adsorbents used in terms of their surface area, average pore diameter and microporous distribution. There are four types of activated carbons that have been selected which are 2.5 and 1.5 mm diameter of Granular Activated Carbon and GR and Powder Extra Pure of Powdered Activated Carbon. The scope of study is limited to three different temperatures.

The surface area, average pore diameter and microporous distribution are analyzed using the QUANTACHROME AUTOSORB analyzer whereas the amount of phenol removed by the activated carbons is determined from the adsorption isotherm measurement which shows that the amount of phenol adsorbed varies with the equilibrium phenol concentration. The analysis of concentration of phenol is done using UV-VIS Spectrophotometer.

From the result of adsorption isotherm measurement, 2.5 mm diameter of Granular Activated Carbon is the best option in removing phenol and the adsorption is proven to be favored at lower temperature. Since the diameter of phenol is less than the diameter of micropores, the adsorption capacity of phenol is very much affected by

the volume of microporous structure. This finding is supported by the results on the characterization of adsorbents which shows that 2.5 mm diameter Granular Activated Carbon has the highest micropore volume and area. Thus, it can be concluded that removal of adsorbate which has diameter less than the diameter of micropore increases with the increase in volume of micropores and is favored at lower possible temperature.

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ABBREVIATIONS

BET	Brunaeur, Deming and Teller
VOC	Volatile Organic Compound
BDDT	Brunauer, Deming, Deming and Teller
MSDS	Material Safety Data Sheet

NOMENCLATURES

Symbols

A	Structural constant
β	Affinity coefficient
c	Residual concentration of adsorbate in the fluid phase from which it has been partially removed, mg/L
C_o	Initial concentration of solution, mg/L
C	Final concentration of solution, mg/L
d_p	Pore diameter, nm
E_0	Characteristic energy of adsorption, KJ/mole
K	Empirical constant, mg/cm^3
m_p	Small mass of adsorbent, g
n	Adsorption constant that must be determined for each solute, adsorbent type and temperature
N_A	Avogadro's number = 6.023×10^{23} molecules/mol
P	Total pressure, atm
P_o	Saturation pressure of the gas, atm

P^s	Normal vapor pressure of liquid on flat surface, 760 torr
P^s_p	Vapor pressure of liquid in pore, torr
P/P_0	Relative pressure
R	Gas constant, KJ/mole.K
S	Surface area, m^2
S_g	Specific surface area, m^2/g
T	Absolute temperature, K
V	Volume of solution, L
V_a	Volume of gas adsorbed at STP (0°C, 760 mm Hg), $cm^3/mole$
V_{He}	Volumes of helium, cm^3
V_{Hg}	Volumes of mercury, cm^3
V_m	Quantity of gas adsorbed when the entire surface is covered with a monomolecular layer, $cm^3/mole$
V_p	Total pore volume of the adsorbent (cm^3/g)
q	Adsorbate adsorbed per unit weight of adsorbent, mg/g
q_o	Empirical constant, mg/g
q_1	Heat of adsorption of the first layer, KJ/mole
q_L	Heat of liquefaction of the adsorptive, KJ/mole
q_m	Saturated capacity of adsorption, mg/L
W	Weight of adsorbent, g
W_m	Volume of micropores filled at relative pressure, cm^3/g
W_0	Total volume of micropores, cm^3/g
x	Dimension of the micropore, nm

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Phenol is one of the compounds found in our wastewaters that have attracted public attention because of its possible link with health even at low concentrations. Carey, (2000), reported that phenol is a polar solute with high solubility in water which is 8.2g/100ml. Thus, it will produce negative ion at the hydroxyl group and correspondingly positive ion at the ethyl group. Solvation by water, for example, involves formation of a hydrogen bond between the positive hydrogen atoms of water and a negative group on a solute, along with some bonding in the reverse direction to the water oxygen (Sledjko, 1985). Roostaei and Tezel, (2004), had said that solvation of phenol in water will create an oxygen demand in receiving water, thus affecting the human and aquatic life. Besides, ingestion of this chemical may cause headache, excitement, fatigue, vomiting and coma while inhalation can cause severe irritation of upper respiratory tract with coughing, burns, difficulty in breathing and possible coma. Chronic inhalation and ingestion can bring to liver and kidney damage and repeated skin contact may cause dermatitis with dark pigmentation of the skin (Material Safety Data Sheet, Phenol Liquid (1), 2003).

By increasing the industrial wastewaters, the demand for removal of organic compounds including phenol has been increased. Sledjko, (1985), on his study has stated that with increased raw material prices, it has become increasingly important to not only develop a process which can accomplish an environmental control objective, but also permit the recovery of phenol in a useful form and thus optimize the operation of the plant. For example, a chemical manufacturer discharging a 100 gpm waste stream containing 3,000 ppm phenol is wasting over $400,000/year in unrecovered material (at 33cent/lb phenol). Some methods have been used with varying degrees of success for the control of organic chemicals in water. These include coagulation, filtration, oxidation, aeration, ion exchange and liquid phase adsorption method. However, it is well known that liquid phase adsorption is one of the most efficient methods for the removal of organic pollutants from the wastewaters (Juang, et. al., 2000). Among the adsorbents used in liquid phase adsorption, activated carbon has became a powerful adsorbent because it has a large surface area and pore volume, which allows the removal of liquid-phase contaminants, including organic compounds, heavy metal ions and colors (Hseih and Teng, 2000). Ying, (1989), had said that, in selecting whether to use the carbon treatment or other types of adsorbents, the adsorption capacities of carbons for the compounds to be removed is an important cost factor in applying the adsorption treatment. The capacities of carbon for contaminant compounds are significantly affected by the pores structures of activated carbon as well as the size of the adsorbate. Therefore, selecting a suitable carbon adsorber to remove specified compounds is an important issue for the treatment process (Hseih and Teng, 2000).

Prior to this research, some theories related to the adsorption process are being explained in this section. There are General Definitions of Adsorption, Adsorption Principle, Factors Affecting Adsorption Process and Adsorption on Activated Carbon. These general explanations are important to give an overall view about the basis of the research.

1.1.1 General Definitions of Adsorption

Sing, et. al., (1984), on their study on adsorption, had defined adsorption as the process of enrichment of one or more components in an interfacial layer. Physisorption (as distinct from chemisorption) occurs whenever an adsorbable gas (adsorbate) is brought in contact with the surface of a solid (adsorbent). It results from the action of Van der Waals, which are comprised of both London dispersion forces and classical electrostatic forces. Differs from physisorption, chemisorption involves a reaction between an adsorbate and an adsorbent which result in a change in the chemical form of the adsorbate. The resulting chemisorptive bond is usually stronger than the derived from the physical Van der Waals forces. It is convenient to regard the interfacial layer as comprising two regions which is the surface layer of the adsorbent (adsorbent surface) and the adsorption space in which the enrichment of the adsorbate can occur. The counterpart to adsorption, which is desorption, denotes the reverse process in which the amount adsorbed decreases (Sing, et. al., 1984).

Seader and Henley, (1998), had also done the research on adsorption and they had concluded that dynamic phase equilibrium is established for the distribution of the solute between the fluid and the solid surface. This equilibrium is usually expressed in terms of concentration (if the fluid is liquid) or partial pressure (if the fluid is gas) of the adsorbate in the fluid and solute loading on the adsorbent, expressed as mass, moles or volume of adsorbate per unit mass or per unit Brunauer, Deming and Teller (BET) surface area of adsorbent. The simplest equilibrium isotherms are Langmuir and Freundlich isotherm. Brunauer, et. al., (1940), had found that for pure gases, all adsorption isotherms fit into one of the five types. The topic on adsorption isotherm will be discussed further in Chapter 2 and 3.

Sing, et. al., (1984), had discovered that, in the context of physisorption, it is expedient to classify pores according to their size such as macropores (pore size > 50 nm), mesopores (pore size between 2 and 50 nm) and micropores (pore size < 2 nm). These limits are to some extend arbitrary since the pore filling mechanisms are dependent on the pore shape and are influenced by the properties of adsorbate, adsorbent, solution as well as the interaction between adsorbate-adsorbent. (Refer 1.1.2 and 1.1.3). The whole of the accessible volume present in micropores maybe regarded as adsorption space and the process which then occurs is micropore filling, as distinct from surface coverage which takes place on the walls of open macropores and mesopores. Micropore filling or primary physisorption process only focuses on monolayer adsorption while mesopores and macropores have monolayer-multilayer adsorption. In monolayer adsorption, all the adsorbed molecules are in contact with the surface layer of the adsorbent while in the multilayer adsorption, the adsorption space accommodates more than one layer of molecules so that not all adsorbed molecules are in direct contact with the surface layer of the adsorbent (Sing, et. al., 1984).

1.1.2 Adsorption Principles

As has been explained previously, the process of adsorption involves separation of a substance from one phase accompanied by its accumulation at the surface of another. Adsorption process will occur until equilibrium is achieved. A quantitative equilibrium distribution between phase concentrations is defined for each combination of a specific adsorbate, adsorbent, and set of system conditions. The character of the distribution, which markedly affects the feasibility of adsorption as a separation process for a particular application, is influenced by a variety of factors relating to the properties of adsorbate, adsorbent and the system in which adsorption occurs (Sledjko, 1985).

Adsorbate-Solvent Properties

An inverse relationship between extend of adsorption of a substance from a solvent and its solubility in that solvent can be generally anticipated. Adsorption from aqueous solution increases as the solubility reduces, largely because the expulsion of increasingly large hydrophobic molecules permits an increasing number of water-water bonds to reform. Besides, molecular size can also affect adsorption rates if these are controlled by intraparticle mass transport within porous adsorbents, which generally more rapid for smaller molecule. The properties of solute whether it is a polar or non-polar also effect the selection of adsorbent (Sledjko, 1985). A polar solute will be strongly adsorbed from a non-polar solvent by a polar adsorbent, and at the opposite end of the spectrum will prefer a polar solvent to a non-polar adsorbent. Polarity of organic compound is a charge separation within the molecule. Examples of high polarity compounds are hydroxyl, carboxyl, nitro, nitrile, carbonyl, sulfonate and amine. Solvation by water, for example, involves formation of a hydrogen bond between the positive hydrogen atoms of water and a negative group on a solute. Water solubility is thus expected to increase with increasing polarity and cause the adsorption from aqueous solution to decrease.

System Properties

Sledjko, (1985), had found out that the components of mixture of adsorbates may affect the adsorption capacity. This is because the adsorption of more than one substance generally reduces the number of surface sites available to each. Besides, he also discovered that for diffusion controlled rates of adsorption by porous adsorbents, very slowly diffusing species in mixed adsorbate systems can depress rates of uptake of those which diffuse more rapidly.

Adsorbent Properties

The performance characteristics of adsorbents relate in large measure to their intraparticle properties. Total surface area, total pore volume and the distribution of area with respect to pore size generally are primary determinants of adsorption capacity (Sledjko, 1985). The physical properties of adsorbent are usually determined by the application of BET model of physical adsorption using nitrogen as the adsorptive (Brunaeur, et. al., 1940). The actual surface area available for adsorption is dependent on the specific nature of the adsorbate and can be considerably less than the total. The nature of the intraparticle surface area markedly affects the types of adsorption interactions that will be operative for an adsorbent, and is a major distinguishable factor to compare different types of adsorbents.

1.1.3 Factors Influence Adsorption Process

Further research done by Sledjko, (1985), was on the factors which influence adsorption process. He said in his report that the characteristics of adsorbate, adsorbent and solution have to be taken into consideration since they tend to have particular impact on the process. The explanations on these factors are discussed in this section.

Characteristics of the Adsorbate

The chemical character of the adsorbate especially on the molecular size is important for a number of reasons. First, as size increases, the solubility decreases. A material which has low solubility in water will have greater tendency to be adsorbed since it has higher affinity for solid surfaces than for the water. Second, molecular size is important to ensure full use of its adsorption capabilities. If the molecular size is too large, adsorption capacities will decrease as very large molecule blocks the pores within the adsorbent. Besides, larger molecule will tend to diffuse more slowly from solution and therefore will require longer time to complete the adsorption process until it reaches the equilibrium (Sledjko, 1985).

Characteristics of the Adsorbents

As being explained in section 1.1.2, physical properties such as surface area, size and distribution of pores in the particles directly affect the adsorption performance. Since different adsorbents will have different physical properties, the amount of adsorption capacity for different types of adsorbents will be different. By determining the amount of adsorbent capacity available as well as the molecular size which can be allowed to be adsorbed, the adsorption performance of different types of adsorbents can be differentiated roughly.

Characteristics of the Solution

For the characteristics of the solution, Sledjko, (1985), said that the two major waste water solution characteristics that have particular impact on adsorption are the solution pH and its temperature. Both adsorbate and adsorbent may have chemical characteristics which are affected by the concentration of hydrogen ions (H^+) in the solution. Some adsorbents have affinity for H^+ and OH^- ions and can directly affect the solution pH and therefore the solubility and adsorption capacity as has been discussed before. The affect of temperature on the adsorption process can be explained by directing the discussion to the heat of reaction of the process. Since adsorption reaction is normally exothermic, the equilibrium extent or capacity of adsorption in a given system is thus generally found to increase with decreasing temperature. Conversely, because adsorption kinetic is generally controlled by diffusive mass transfer, rates of approach to equilibrium normally increase with increasing temperature (Sledjko, 1985).

1.1.4 Adsorption on Activated Carbon

Among adsorbents that are currently used, activated carbons (granular and powdered) are the most widely used adsorbents because of their excellent adsorption ability (Juang, et. al., 2000). The use of activated carbon for adsorption treatment was first recorded more than two hundred years ago. Activated carbon is now broadly employed for product purification (such as sugar refining, food processing and the production of water) and pollution control (such as water and wastewater treatment and the removal of volatile organics from industrial process gases) (Hseih and Teng, 2000). Activated carbon is a carbonaceous adsorbent with a high internal porosity, and hence a large internal surface area. According to Baker, et. al., commercial activated carbon grades have specific surface area of 500 to 2000 m^2/g, but values as high as 3500 to 5000 m^2/g have been reported for some activated carbon. Related to the type of application, two major product groups exists that is powdered activated carbon, which has particle size between 1 to 150 micron and granular activated carbon with particle size in the range of 0.5 to 4 mm (Activated Carbon, http://www.norit.com/Techinfo/techinfo.html).

Almost any carbonaceous material can be converted into activated carbon. Some of the raw materials of activated carbon are coal, peat and coconuts (Activated Carbon, http://www.norit.com/Techinfo/techinfo.html). Bansal, et. al., (1988), on their research on activated carbon, had stated that manufacturing of activated carbon involves carbonization of raw carbonaceous materials in an inert atmosphere and activation of carbonized product. Carbonization results in production of fines and closed pores while activation enlarges the diameters of fine pores and creates new pores. Through high manufacturing techniques and perfect quality control and research development, it is possible for the production of high pore volume, wide surface area and elevated adsorption efficiency of activated carbon.

1.2 Problem Statement

Due to the increased in the industrial wastewaters, the demand for removal of organic compounds including phenol has been increased. Although liquid-phase adsorption on activated carbon has become one of the most common approach to solve this problem, not all types of activated carbon has been applied in industry. Since the surface area, dimensions and distribution of the pores depend on the condition of carbonization and activation, different types of activated carbon might be produced from different ways of manufacturing. The different in the physical properties cause the activated carbon to perform differently in adsorbing a particular adsorbate.

In order to evaluate the performance of the activated carbon in adsorption process, it is vital to analyze the physical properties of the adsorbents as well as the size of the adsorbate used in the adsorption. The adsorption capacity of the same adsorbate will be different on various types of activated carbon.

Temperature is also one of the factors that contribute to the effectiveness of the adsorption process. Theoretically, adsorption capacity tends to decrease as the temperature increases. However, the adsorption capacity for different types of activated carbon will not be the same although the experiment is conducted at the same temperature.

Based on the literature review that had been done, there are no information on industrial application regarding the adsorption of phenol on few activated carbons such as 2.5 and 1.5 mm diameter Granular Activated Carbon as well as GR and Powder Extra Pure of Powdered Activated Carbon. The characterization of physical properties of these types of activated carbon has not been analyzed yet. Their performance in removing phenol at various temperatures has also not yet known. If it can be proven that any of the activated carbons selected is able to remove phenol more than the existing activated carbon used in industry, thus it can be said that this activated carbon is capable to be commercialized in industry.

1.3 Objectives

The objectives of this research are:

i. To characterize various types of activated carbons used in terms of their surface area, pore diameter and micropore size distribution.
ii. To compare the degree of phenol removal on various types of activated carbon.
iii. To analyze the effect of various temperatures on the adsorption capacity of phenol on different types of activated carbon.

1.4 Scope of Study

In this research, the adsorption capacity of phenol using various types of adsorbents and the characterization properties of the adsorbents which affect the adsorption capacity such as surface area, pore diameter and micropore size distribution are being investigated. Since no information is available regarding the performance of four types of activated carbons in adsorbing phenol, which are 2.5 and 1.5 mm diameter of Granular Activated Carbon and GR and Extra Pure Powder of Powdered Activated Carbon, thus there arise need for the analysis and compilation of well developed analysis methods, covering both the adsorption isotherm measurement at various temperatures as well as the characterization of physical properties of the adsorbents. The concentration of phenol remaining after reaching equilibrium is determined using UV-VIS Spectrophotometer while the analysis of surface area, pore diameter and micropore size distribution are analyzed using Quantachrome AUTOSORB-1 analyzer. The experiment is evaluated at three different temperatures (35°C, 55 and 65°C).

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

There are very scarce published literatures found relating to the adsorption isotherm measurement of phenol. In this section, few works and investigations relating to liquid phase adsorption will be reviewed based on findings through websites as well as literatures. These findings are helpful for this early preliminary research to give an insight on what direction of work on isotherm measurement and characterization of adsorbents should be focused on. This section can be classified into two major parts. There are Adsorption Isotherm and Adsorption of Phenol in Liquid-Phase Adsorption.

2.2 Adsorption Isotherm

Brunauer, et. al., (1940), on their extensive literature survey on adsorption, had found that all adsorption isotherms fit into one of the five types shown in Figure 2.1 where W is the weight adsorbed, P is the adsorbate equilibrium pressure, P_o is the adsorbate saturated equilibrium vapor pressure and P/P_o is the relative pressure. Each of these five isotherms and the conditions leading to its occurrence are discussed below.

Type I isotherms are encountered when adsorption is limited to, at most only a few molecular layers. This condition is encountered in chemisorption where the asymptotic approach to a limiting quantity indicates that all of the surface sites are occupied. In the case of physical adsorption, Type I isotherms are encountered with microporous powders whose pore size does not exceed a few adsorbate molecular diameters. A gas molecule, when inside pores of these small dimensions, encounters the overlapping potential from the pore walls which enhances the quantity of gas adsorbed at low relative pressures. At higher pressures, the pores are filled by adsorbed or condensed adsorbate leading to the plateau, indicating little or no additional adsorption after the micropores have been filled. Physical adsorption that produces the Type I isotherm indicates that the pores are microporous and that the exposed surface resides almost exclusively within the micropores, which once filled with adsorbate; leave little or no external surface for additional adsorption (Lowell and Shields, 1991).

Type II isotherms are most frequently encountered when adsorption occurs on nonporous powders or on powders with pore diameters larger than micropores. The inflection point or knee of the isotherm usually occurs near the completion of the first adsorbed monolayer and with increasing relative pressure; second and higher layers are completed until at saturation where the number of adsorbed layers become infinite (Lowell and Shields, 1991). The heat of adsorption for the first adsorbed layer is greater than that for the succeeding layers, each of which is assumed to have a heat of adsorption equal to the heat of condensation (vaporization). Seader and Henley, (1998), reported that both Types I and II are desirable isotherm, exhibiting strong adsorption.

Type III isotherms are characterized principally by heats of adsorption which are less than the adsorbate heat of liquefaction. Thus, as adsorption proceeds, additional adsorption is facilitated until the saturation pressure is approached because the adsorbate interaction with an adsorbed layer is greater than the interaction with the adsorbent surface (Lowell and Shields, 1991).

The adsorption isotherm for Types I, II and III preclude the possibility of capillary condensation. In a development by Brunauer, subsequent to BET equation (refer equation 3.6 in Theory), the number of layers is restricted by pore size and capillary condensation is assumed to occur at reduced pressure. It predicts the adsorption isotherm of Type IV and V, where we see the maximum extent of adsorption occurs before the saturation pressure is reached. Type IV is the capillary condensation version of Type II and Type V is the capillary condensation version of Type III (Lowell and Shields, 1991).

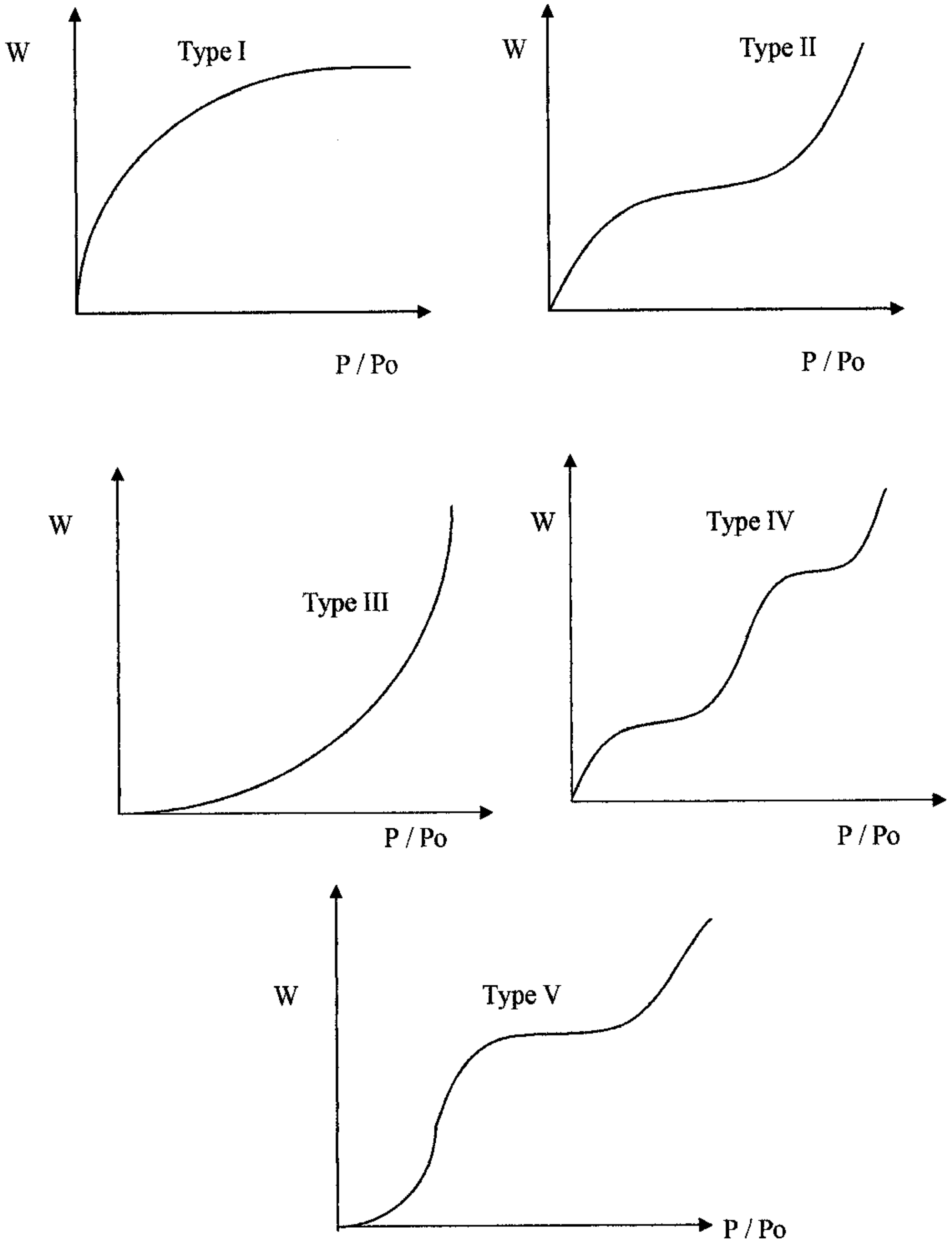

Figure 2.1 The Five Isotherm Classifications according to BDDT (Brunaeur, et. al., 1940)

2.3 Adsorption of Phenol in Liquid-Phase Adsorption

Various researches had been done on the adsorption of phenol in liquid-phase adsorption. The results are presented in many ways. Some effects such as concentration of adsorbate, solubility of adsorbate, particle size, temperature of solution as well as physical characteristics of the adsorbent had been studied. However, in this paper, only the studies on the effect of temperature and the physical characteristics of the adsorbent are presented since these two conditions are directly related to the thesis. The main purpose is that to have the comparison between the literature reviews and also between the results that been obtained from experiment. This will help to ensure that the result is similar to the expectation. If not, the flaws of the research can be studied and discussed for further improvements. Below is the results obtained by previous conducted researches.

2.3.1 Effect of Temperature

Roostaei and Tezel, (2004), had done investigations regarding the removal of phenol from aqueous solution by adsorption. They had used few types of adsorbents such as silicagel, HiSiv 3000, activated alumina, activated carbon, Filtrasorb-400 and HiSiv 1000. The adsorption isotherm model of the Langmuir-Freundlich type was the best to describe adsorption equilibrium data for phenol for the adsorbents studied. The effect of temperature on adsorption of phenol was studied by determining the equilibrium isotherms for HiSiv 1000 at 25, 40 and 55°C. The results showed that adsorption capacity decreased with increasing temperature (refer Figure 2.2). Zogorski and Faust, (1978) observed the same results on the effect of temperature to the adsorption capacity. Their study of phenol on granular activated carbon at different temperatures showed that adsorptive capacity of phenol increased with decreasing temperature. Besides, the reduction or increment in temperature also gives effect on the adsorption rate. Sing and Rawat, (1994), stated that the rate of adsorption of phenol by fly ash was increased as the temperature increased in the range of 30 to 50°C. This is because the kinetic rate increased with increasing temperature. He also said that due to the diffusion-limited process, the rate of adsorption of the organics from solution increases as the temperature of the system is increased. Zogorski, et. al., (1976), on his study, had concluded that the removal rate of phenol tend to increase by 21% after raising the temperature of the system from 10 to 30°C.

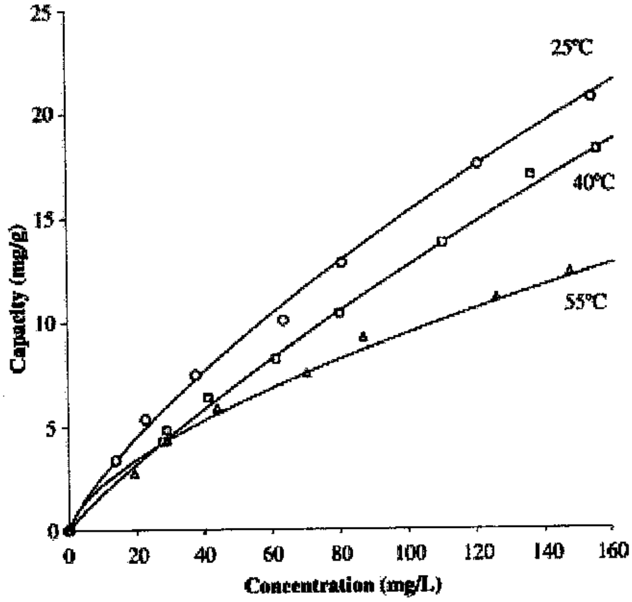

Figure 2.2 Adsorption isotherms of phenol with HiSiv 1000 (50×70 mesh), at different temperatures. Points represent experimental data and curves represent three-parameter isotherm fits for 25 and 40 °C and Freundlich fit for 55 °C (Roostaei and Tezel, 2004).

2.3.2 Effect of Physical Properties of Adsorbent

Juang, et. al., (2000), had done investigation on the role of microporosity of activated carbons on their adsorption abilities for phenol and 4-chlorophenol. The carbons that they used were prepared from cane piths and were activated by steam with the activation temperature and time was in the ranges of 750 to 840°C and 2 hour, respectively. The results showed that the adsorption of phenols mostly, rather than completely, occurred within the micropores of the adsorbents. The literature review from Adsorption of Phenols and Dyes by High Surface Area Activated Carbon, (http://www.lenntech.com/adsorption.htm) also proved the same theory. For this research, phenol was chosen as representative microporous adsorbate and methylene blue and erythrosine red as the mesopores adsorbate. The adsorption was carried out on five different types of activated carbon. The result showed that the difference in adsorption capacity for phenol is not very large, between Filtrasorb 100 and CZ124. However, CZ126 and CK22 have much larger adsorption capacities. Thus, the extent of the uptake of phenol conforms to the micropore volume of the carbon. The micropore volume, and also the phenol uptake, is in the order of CK22 > CZ126 > CZ124 > Filtrasorb 100. The tabulated data for this research is shown in Appendix A. This investigation had demonstrated that adsorption of phenol is favored to occur in the microporous structure. This was due to the diameter of the phenol (0.62 nm) that is smaller than the diameter of the micropore which is less than 2 nm.

CHAPTER 3

THEORY

3.1 Liquid Phase Adsorption Isotherm

Seader and Henley, (1998), on their research on the dynamic of the adsorption process, had discovered than when a quantity of adsorbent is contacted with a given volume of liquid containing an absorbable solute, adsorption occurs until equilibrium is reached. The common manner in which to depict this distribution is to express the amount of substance adsorbed per unit weight of adsorbent, q, as a function of the residual equilibrium concentration, c, of substance remaining in the liquid phase. The equation for the adsorption isotherm for liquid-phase adsorption is as follows:

$$q = V(C_o - C)/W \qquad \text{(equation 3.1)}$$

where q is the adsorbate adsorbed per unit weight of adsorbent (mg/g), V is the volume of solution (L), C_o is the initial concentration of solution (mg/L), C is the final concentration of solution (mg/L) and W is the weight of adsorbent (g).

A wide range of q against c values maybe obtained depending on the volume of solution involved, the initial and final concentration of solution as well as the weight of adsorbent (refer equation 3.1). These values of q and c can be fitted into one or more standard isotherm equations, so that the q and c relationship can be expressed in mathematical form. Valenzuela and Myers, (1989), had done literature research for the equilibrium adsorption of 25 different binary liquid mixtures. With one exception, that is the mixture of cyclohexane and n-heptane with silica gel, all 25 mixtures give composite isotherms of the forms shown in Figure 3.1b and c. The composite curve with the negative adsorption is obtained due to the adsorption of the solvent itself.

Numerous equilibrium adsorption isotherm based on different concepts have been proposed. In the case which involves binary mixture in dilute region in which the amount of the adsorption of solvent is constant and all changes in the total amount adsorbed are due to just the solute, the adsorption isotherm formed is shown in Figure 3.1a which resembles the form obtained with pure gases (Seader and Henley, 1998). It is then common to fit the data into Langmuir or Freundlich equation:

Langmuir Isotherm $q = q_o c / (K + c)$ (equation 3.2)

Freundlich Isotherm $q = Kc^n$ (equation 3.3)

For Langmuir isotherm, q_o and K represents the empirical constant and for Freundlich isotherm, q and n represents the constants which can be determined experimentally. Figure 3.2 shows the adsorption isotherm of both Langmuir and Freundlich isotherms (Geankoplis, 1993).

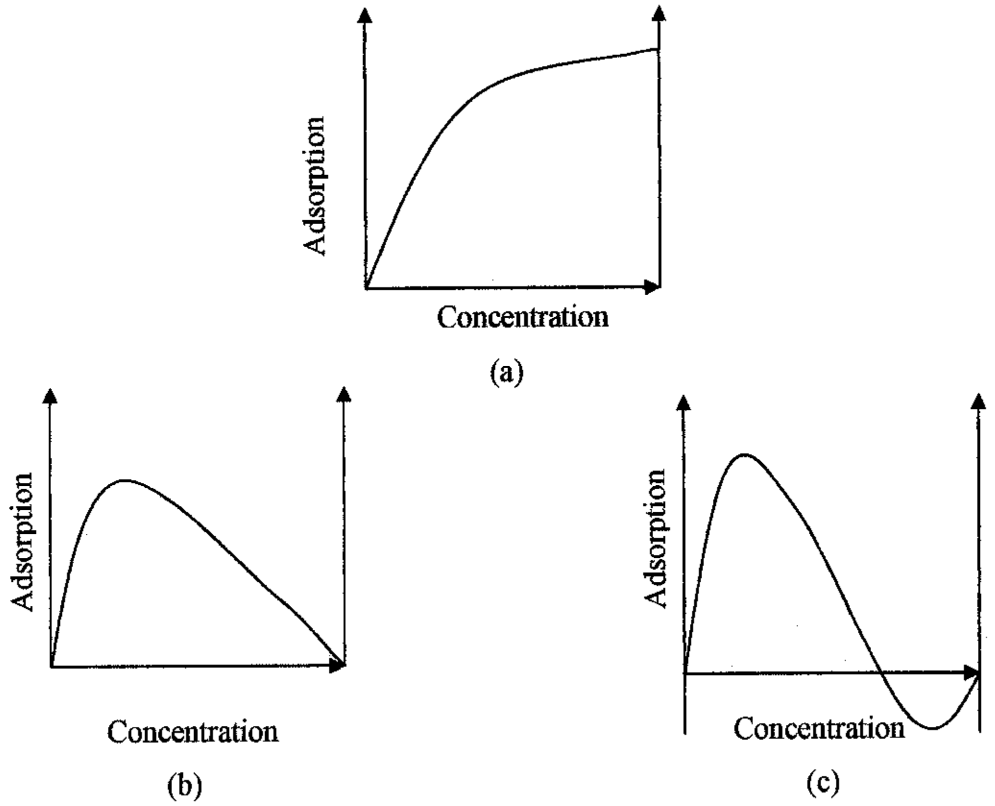

Figure 3.1 Representative Isotherm of Concentration Change for Liquid Adsorption (Seader and Henley, 1998)

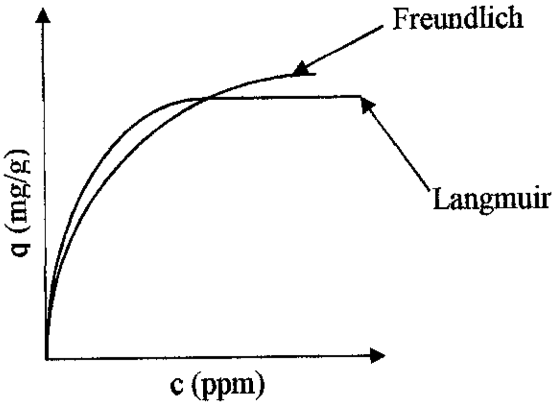

Figure 3.2 Graphical Representation of Isotherm Model (Geankoplis, 1993)

However, for this research, the scope of adsorption isotherm model is not been covered.

3.2 Determination of Surface Area, Pore Volume and Pore Size Distribution

The surface area value of an adsorbent can be determined via the gas adsorption method. For this research, the isotherm data points is fitted in BET isotherm to plot linear graph in order to calculate the average pore diameter and total surface area. Besides, it is also fitted in Dubinin-Radushkevich (DR) plot for the micropore analysis.

3.2.1 BET theory

The research done by Langmuir, (1918), was more properly applies to chemisorption situations than to physical adsorption ones because of its first assumptions that gases form only one molecular layer on a solidA major advance in adsorption theory generalized the treatment of Langmuir and incorporated the concept of multimolecular layer adsorption. It is known as the BET theory. The fundamental assumption is that the forces active in the condensation of gases also are responsible for the binding energy in multimolecular adsorption. By equating the rate of condensation of gas molecules onto an already adsorbed layer to the rate of evaporation from that layer and summing for an infinite number of layers, the expression is obtained

$$V_a = \frac{V_m C P}{(P_o - P)[\, 1 + (C-1)\, P/P_o]} \qquad \text{(equation 3.4)}$$

where C is a constant related to the heat of adsorption (exp [$(\Delta H_{cond} - \Delta H_{ads})/RT$]), P is the total pressure, P_o is the saturation pressure of the gas, V_a is the volume of gas adsorbed at STP (0°C, 760 mm Hg) and V_m is the quantity of gas adsorbed when the entire surface is covered with a monomolecular layer (Brunaeur, et. al., 1940).

The value of C in simplest terms is

$$C \quad \propto \quad exp \frac{q_1 - q_L}{RT} \qquad \text{(equation 3.5)}$$

where q_1 is the heat of adsorption of the first layer, q_L is the heat of liquefaction of the adsorptive, R is the gas constant, and T is the absolute temperature.

Equation 3.4 can be rewritten in the linear form as

$$\frac{P}{V_a (P_o - P)} = \frac{1}{V_m C} + \frac{C - 1}{V_m C} (P/P_o) \qquad \text{(equation 3.6)}$$

From which, if applicable, a plot P / [V_a $(P_o\text{-}P)$] versus P/Po should yield a straight line with intercept $1/V_m C$ and slope $(C\text{-}1)/V_m C$. The values of V_m and C may then be obtained from the plot of a straight line. Further finding by Brunaeur, et. al., 1940), bring to the calculation of the value of specific surface area, S_g, using the value of constant V_m that is get from previous calculation.

$$S_g = \sigma\, V_m N_A / V \qquad \text{(equation 3.7)}$$

where N_A is the Avogadro's number, V is the volume of gas per mole at STP conditions (0°C, 1atm) and σ is the area of surface occupied by a single adsorbed gas molecule.

Brunaeur, et. al., (1940), also found out that the pore diameter of an adsorbent can be calculated as follow:

$$dp\ (\text{Å}) = 17.9 / \ln\ (P^s / P^s_p) \qquad \text{(equation 3.8)}$$

where P^s is the normal vapor pressure and P^s_p is the vapor pressure of liquid in pore.

3.2.2 DR method

In the case of physisorption on microporous solids, Dubinin and Radushkevich, (1947), said that the origin of the shape of the adsorption isotherm is due to micropore filling. The micropores tend to be filled with adsorbate at low relative pressures. The adsorption isotherm on the microporous solids is described by the DR equation shown in equation 3.9 and 3.10 (Quantachrome AUTOSORB 1, Laboratory Manual).

$$W_m = W_0 \exp\ [-A\ (T / \beta)^2 \log^2 (P_0 / P)] \qquad \text{(equation3.9)}$$

$$E_0 = 0.01915 / B^{1/2} \qquad \text{(equation 3.10)}$$

where W_m is the volume of micropores filled at relative pressure P/P_0 and temperature T, W_0 is the total volume of micropores, A is the structural constant, β is the affinity coefficient and E_0 is the characteristic energy of adsorption. Equation 3.9 is rewritten for experimental use as equation 3.11:

$$\log W_m = \log W_0 - D \log^2 (P_0 / P) \qquad \text{(equation 3.11)}$$

A plot of log W_m versus $\log^2 (P_0 / P)$ is called the DR plot and the total volumes of micropores is calculated from the intercept A. DR plot is shown in Figure 3.3 (Quantachrome AUTOSORB 1, Laboratory Manual). The characteristic dimension of the micropore (x), nm corresponding to half width of the slit pore, is related to the characteristic energy of adsorption, E_0 by equation 3.12:

$$x = k / E_0 \qquad \text{(equation 3.12)}$$

where the value of the parameter k depends on E_0

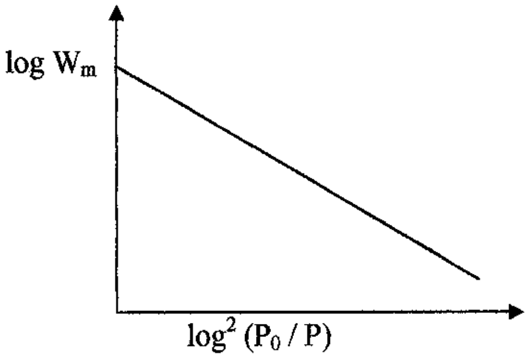

Figure 3.3 DR Plot of Adsorption of Argon at 77 on Activated Carbon (Quantachrome AUTOSORB 1, Laboratory Manual)

3.3 Effect of Temperature to Adsorption Isotherm

Slejko, (1985), from his investigation regarding the factors which influence the adsorption process, said that any increment or decrement in the temperature of a solution will affect both the adsorption capacity and adsorption rate. Since the adsorption reaction is normally exothermic, the equilibrium capacity of adsorption in a given system tends to increase with decreasing temperature. Further, since solubility and adsorption are inversely related, as temperature affects solubility, it will therefore affect the extent of adsorption for the particular adsorbate. However, in the case of the kinetic rate of adsorption, it is usually increased at higher temperatures. This is due primarily to the increased rate of diffusion of adsorbate molecules through the solution to the adsorbent.

CHAPTER 4

METHODOLOGY

4.1 Introduction

This chapter provides the detail explanation on the equipments, apparatus and raw materials required as well as the experimental procedures needed prior to conducting the experimental laboratory work. The laboratory work can be divided into 4 parts that is sample preparation, calibration curve preparation, characterization of adsorbent and adsorption isotherm measurement. For this research, 2 types of Granular Activated Carbon (2.5 and 1.5mm diameter) and 2 types of Powdered Activated Carbon (GR and Powder Extra Pure) have been used.

4.2 Equipments, Apparatus and Raw Materials Required

For different laboratory works, various apparatus, equipment and raw materials were required and are summarized in Table 4.1.

	Apparatus	Equipment	Raw Material
Sample Preparation	Volumetric flasks of 1000 mL, filter funnel, beakers of 1000 mL, beakers of 250 mL, analytical balance capable of measuring to the nearest of 0.1 mg, stirrer and vacuum oven		2.5 mm and 1.5 mm diameter of Granular Activated Carbon, GR and Powder Extra Pure of Powdered Activated Carbon, solid phenol and distilled water

Calibration Curve Preparation	Volumetric cylinders of 100 mL	UV-VIS Spectrophotometer	100, 50, 25 and 10 ppm phenol solution
Adsorption Isotherm Measurement	Aluminium foil, analytical balance capable of measuring to the nearest of 0.1 mg, desicaters with silica gel, patry discs, water bath shaker, volumetric cylinders of 100 mL, conical flasks of 250 mL, filter papers of 0.4 to 0.45 µm, filter funnels and gravity filters	UV-VIS Spectrophotometer	100 ppm phenol solution, 2.5 mm and 1.5 mm diameter of Granular Activated Carbon, GR and Powder Extra Pure of Powdered Activated Carbon
Characterization of Adsorbent	Weighing machine capable of measuring to the nearest 0.1 mg	Quantachrome AUTOSORB-1 analyzer	2.5 mm and 1.5 mm diameter of Granular Activated Carbon, GR and Powder Extra Pure of Powdered Activated Carbon

Table 4.1 Apparatus, Equipments and Raw Materials needed for each parts of laboratory works

4.3 Properties and Composition of Adsorbate and Adsorbents

Prior to the characterization of adsorbents and adsorption isotherm measurement, the knowledge on the physical and chemical composition of adsorbate and adsorbents should be established. From this knowledge, the insight to the basic composition required for the purpose of the application shall be known. The composition data, specification and synthesis method of adsorbents used in this project were obtained from the manufacturer and presented in this section. The physical and chemical properties of adsorbate used were obtained from literature reviews.

4.3.1 Adsorbents

Four types of activated carbon, two each from granular and powdered activated carbon used in this experiment were obtained from Merck, (2000), vendor of the laboratory chemicals. General precautions have to be taken into account while handling these chemicals since it can affect human life (Material Safety Data Sheet, Activated Carbon). Table 4.2 shows the hazard of these chemicals to eyes and skin and the effect to health if being ingested while Table 4.3 shows some first aid measures that should be taken during emergency. These activated carbons were made from the finest grades of bituminous coal which were in granular form and were activated by steam process. Their high activity and surface area made these products ideal for many purification applications including chemical treatment, gas adsorption, waste water, potable water, etc. Some specifications of these chemicals are listed in Table 4.4 for Granular Activated Carbon and Table 4.5 for Powdered Activated Carbon.

	Hazard Identification
Eyes	It is not corrosive, should be treated like most particulate materials, it could cause mild physical irritation.
Skin	It is not corrosive and not a primary skin irritant. There could be mild irritation due to abrasive action of dust.
Ingestion	There is no known deleterious effect.
Inhalation	There could be possible mild irritation of respiratory tract due to drying and abrasive action of dust.

Table 4.2 Hazard Identification of Activated Carbon (MSDS, Activated Carbon)

	First Aid Measure
Eyes	Use soap and water to wash material off the skin and seek medical attention if irritation occurs.
Skin	Use copious amounts of water to flush. If irritation occurs, seek medical attention.
Ingestion	Drink one or two glasses of water. If gastrointestinal symptoms develop, seek medical attention.
Inhalation	Move to fresh air. If cough or respiratory symptoms develop, seek medical attention.

Table 4.3 First Aid Measure of Activated Carbon (MSDS, Activated Carbon)

Specifications	**2.5 mm diameter**	**1.5 mm diameter**
Solubility in water (%)	≤ 2	≤ 2
Arsenic (%)	≤ 0.002	≤ 0.0005
Heavy metals (as Pb) (%)	≤ 0.01	≤0.01
Residue on ignition (600°C) (%)	≤ 5	≤ 5
Bulk density (g/100ml)	40	40
Loss on drying (120 °C) (%)	≤ 10	≤ 10
Fe (Iron) (%)	≤ 0.6	≤ 0.05

Table 4.4 Specifications of Granular Activated Carbon (Merck, 2000)

Specifications	GR	Powder Extra Pure
Solubility in water (%)	≤ 0.5	≤ 0.5
pH (5% in water)	4-7	5-7
Chloride (Cl)	≤ 0.01	≤0.02
Residue on ignition (600°C) (%)	≤ 1	≤ 2
Methylene blue adsorption (ml/0.1g)	12	12
Loss on drying (120 °C) (%)	≤ 10	≤ 10
Fe (Iron) (%)	≤ 0.03	≤ 0.05

Table 4.5 Specifications of Powdered Activated Carbon (Merck, 2000)

4.3.2 Adsorbate

Brown, et. al., (2000), from their research, had claimed that phenol is a compound with an OH group attached to an aromatic ring. It is a colorless liquid with sweetish odor. Phenol is about 1 million times more acidic in water than a typical non-aromatic alcohol such as ethanol with 6 pH. It is used industrially in the making of several kinds of plastics and in the preparation of dyes. It is also used as a topical anesthetic in many sore throat sprays. The hazard identification of this chemical is listed in Table 4.6 while the first aid measure is shown in Table 4.7. Besides, some physical properties of phenol are summarized in Table 4.8.

	Hazard Identification
Eyes	Exerts a strong corrosive action. May cause corneal damage or blindness.
Skin	Phenol is rapidly absorbed through the skin. A serious burn or poisoning through skin absorption may occur if the chemical is not removed promptly and thoroughly. If the phenol is left on the skin, the exposed area will burn or itch intensely which may be followed by local anesthesia and gabfrene. Absorption of phenol through skin may cause collapse or sudden death.
Ingestion	Ingestion of phenol causes intense burning of mouth and throat followed by marked abdominal pain and distress.

	Lethal oral doses of phenol for adults have ranged from 1 to 10 gm.: for infants 50 to 500 mg. Cyanosis muscular weakness and collapse may occur within a few minutes after ingestion.
Inhalation	In animals, prolonged inhalation of vapors (30-60ppm) has induced respiratory difficulties, lung damage, loss of weight & weight & paralysis. Breathing of phenol vapors can cause irritation of the mucous membranes.

Table 4.6 Hazard Identification of Phenol (MSDS, Phenol (2))

	First Aid Measure
Eyes/Skin	Immediately flush eyes and/or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Obtain medical attention promptly.
Ingestion	Do not induce vomiting. If conscious, give patient milk, milk of magnesia or whites of eggs beaten with water. Keep patient warm and quiet, call a physician. Obtain medical attention promptly. If inhaled, remove to fresh air. If not breathing, give artificial respiration, preferably mouth-to-mouth. If breathing is difficult, give oxygen only by trained personnel. Obtain medical attention promptly.
Inhalation	If inhaled, remove to fresh air. If not breathing, give artificial respiration, preferably mouth-to-mouth. If breathing is difficult, give oxygen only by trained personnel. Obtain medical attention promptly.

Table 4.7 First Aid Measure of Phenol (MSDS, Phenol (2))

Specifications	Phenol
Appearance	Colorless crystals with a characteristic odor
Explosion limit	1.5 – 8.6%
Specific gravity	1.07
Melting point (°C)	40 - 42
Boiling point (°C)	182
Vapor pressure (mmHg at 20 °C)	0.35
Flash point (°C)	79

Table 4.8 Specifications of Phenol (Physical & Theoretical Chemistry Lab. Safety, Phenol)

4.4 Experimental Procedures

The experimental procedures were referred from American Standards of Testing Method, (ASTM) on Activated Carbon, (Graham, 2000) and Equipment Manual (Quantachrome AUTOSORB 1, Laboratory Manual).

4.4.1 Sample Preparation

In sample preparation section, phenol solution and activated carbon samples were prepared. Below are the brief procedures for both samples preparations.

Activated Carbon Sample Preparation

The selected activated carbons should be free of moisture, gases and any impurities. To remove all the moisture, the activated carbons were dried in vacuum oven at 150 °C overnight. Then the adsorbents were kept in desicaters to prevent them from being exposed to the environment which can cause the adsorbents to adsorb any possible particulates and chemicals. Silica gel was included in order to maintain the moisture free condition in the desicaters.

Phenol Solution Sample Preparation

In laboratory, phenol was stored in solid form. For the purpose of conducting this experiment, 100 ppm phenol solution has to be prepared. Most important criteria that should be taken into account while preparing the solution was the solubility of phenol in water. (Carey, 2000), had investigated the solubility of phenol and he found that phenol is more soluble in water than arenas and aryl halides of comparable molecular weight. The solubility of phenol is 8.2g / 100ml water. The method of calculating the required amount of phenol solid to produce a specific concentration of phenol solution is shown in Appendix B.

4.4.2 Calibration Curve Preparation

To prepare the calibration curve which functioned as the basis in measuring the concentration of phenol remained in the solution after adsorption taken place, four different concentrations of phenol solutions were prepared which are 100, 50, 25 and 10 ppm. Then the Absorbance Value (AU) for each unit concentration was measured using UV-VIS Spectrophotometer and the graph of AU versus concentration phenol (ppm) was plotted.

4.4.3 Characterization of Adsorbents

The properties of activated carbon such as surface area, pore volume and pore size distributions were analyzed in order to support the experimental result. For measuring these properties, the AUTOSORB analyzer with N_2 liquid as adsorbate at 77 K was used. This analyzer functioned by measuring the N_2 adsorption-desorption isotherms of the activated carbons using static vacuum procedures. The partial pressure of N_2 above the samples was gradually increased and the amount of N_2 adsorbed at each pressure after the equilibrium was noted, thereby generating the isotherm curve.

A sample cell with the narrowest diameter that will comfortably admit the adsorbent sample was chosen. Prior to the analysis, the weights of adsorbents were recorded. The sample was placed in the AUTOSORB analyzer and the instructions on the equipment were followed. In the AUTOSORB, the sample was degassed at high temperature to remove the remaining moisture and vapors adsorbed on the sample.

The temperature was set at 150°C. (Time for completing degassing was determined by the AUTOSORB software). A sample was considered ready for analysis when the sample degas test of no more than 50 microns mercury per minute (at elevated temperature) which was then be initialized by the AUTOSORB for further analysis. Upon completing the degassing, N_2 liquid was used as adsorbate as a backfill gas to minimize buoyancy errors. The heating mantle was switched off using toggle switch on the front of AUTOSORB. The 'unload' button in the degasser control panel was clicked. The mantle was cooled below 100 °C before unloading because warm sample cell could cause weighting error. The adsorption method required was selected from built in AUTOSRB software. For this research, 10 points of adsorption and 10 points of desorption were chosen. The isotherm data were collected and was fitted with BET plot to obtain the surface area and pore diameter. The data was also been used to plot Dubinin-Radushkevich (DR) plot in order to determine the micropore porosity. The AUTOSORB software calculates the identified physical properties automatically using the equations 3.4 to 3.12 presented in Chapter 3.

4.4.4 Adsorption Isotherm Measurement

The water bath shaker was filled with distilled water and left with initial temperature set at 35 °C. 100 mL phenol solution was measured using volumetric cylinder and be poured quickly into 250 mL conical flask. This flask was sealed tightly with aluminium foil. The same procedure was repeated 5 times to prepare 6 flasks of conical flasks. Then, the freshly dried activated carbon (example 2.5 mm diameter of GAC) was weighted accordingly (refer Table 4.9) using analytical balance capable of measuring to the nearest 0.1 mg. This activated carbon was added to the conical flasks before being sealed again to prevent any contamination of impurities from the atmosphere and evaporation of phenol. All the conical flasks were left in the water bath shaker with the speed set at 150 rpm for 24 hours to reach equilibrium.

After 24 hours, the shaker was switched off and the samples were taken out to be filtered using gravity filter. During this step, the samples were prevented from being exposed to the environment in order to reduce the potential of phenol to be evaporated to the atmosphere. The filtered samples were then analyzed using UV-VIS Spectrophotometer to find the concentration of phenol left in the solution.

In the UV-VIS Spectrophotometer, there were UV cells (cuvettes) which must be clean and dry before used in order to minimize moisture and solvent interference during the analysis. The best way to achieve to this result was to soak the cuvettes in hot water for few hours and dried in oven before used. Then the power was turned on and the unit was allowed to warm up for a couple of minutes. In 'Method' tab button, Wavelength Scan Method was chosen and the required parameters such as the wavelength range scan cycle and number of samples are filled in the edit button. For phenol, the wavelength was set at 269.90 nm. The scan cycle and number of sample were set at 50/cycle and 1 sample respectively. There were two Spectronic spaces available to place the sample (cuvettes). One was used for the solvent blank (distilled water), while the other was used as sample holder. The cuvettes were handled with extreme care. They were wiped with soft tissue paper and touching the wall of the cuvettes with fingers must be avoided as the fingerprints will absorb and scatter light. The cuvettes were closed with its cover lid. The analysis space was closed. On the monitor, the 'Measure' button was clicked. The concentrations of the samples were measured using UV-VIS Spectrophotometer by referring to the calibration curve which has been prepared before. The experiment was repeated for other types of activated carbons. The experiment was conducted at two other temperatures (55 and 65°C).

Concentration of Adsorbable Constituents, ppm	**Solution Sample Volume, mL**	**Carbon Weights (Dry Basis), g**
>10; 100	100	0.03, 0.04, 0.1, 0.2, 0.3

Table 4.9 Solution Sample Volume and Carbon Weights for Determining Adsorbable Constituents (Graham, 2000)

CHAPTER 5

RESULTS AND DISCUSSIONS

5.1 Introduction

This chapter presents the results and the detailed discussion of the adsorption isotherm measurement of phenol on four types of activated carbon at three different temperatures. The relationship between the physical properties of each adsorbents such as surface area, pore diameter and micropore structures to their performance in absorbing phenol molecule also been studied. Among these four types of activated carbon, the most effective one has been identified.

5.2 Results

This section provides the results obtained upon analysis of the data acquired from the experiment stated above. It is presented in graphical and tabular form to ease the comparison and understanding.

5.2.1 Results on Calibration Curve of Solutions

The standard curve of AU versus concentration is obtained by measuring the AU of phenol solutions at different concentrations (100, 50, 25 and 10 ppm). This curve is used in the adsorption isotherm measurement to determine the equilibrium concentration of the phenol left in the solution since UV-VIS Spectrophotometer will only measure the AU of the phenol solution. 100 ppm phenol solution is selected as the highest concentration since the equilibrium isotherm measurement is conducted using 100 ppm phenol solution. It is expected that the concentration of phenol left in the solution after equilibrium is reached will be less than 100 ppm. In order to reduce the error of the curve, additional points should be added by measuring the AU of few other concentrations of phenol in a range of 0 to 100 ppm. The result is shown in Figure 5.1 and Table C.1.1 (refer Appendix C.1).

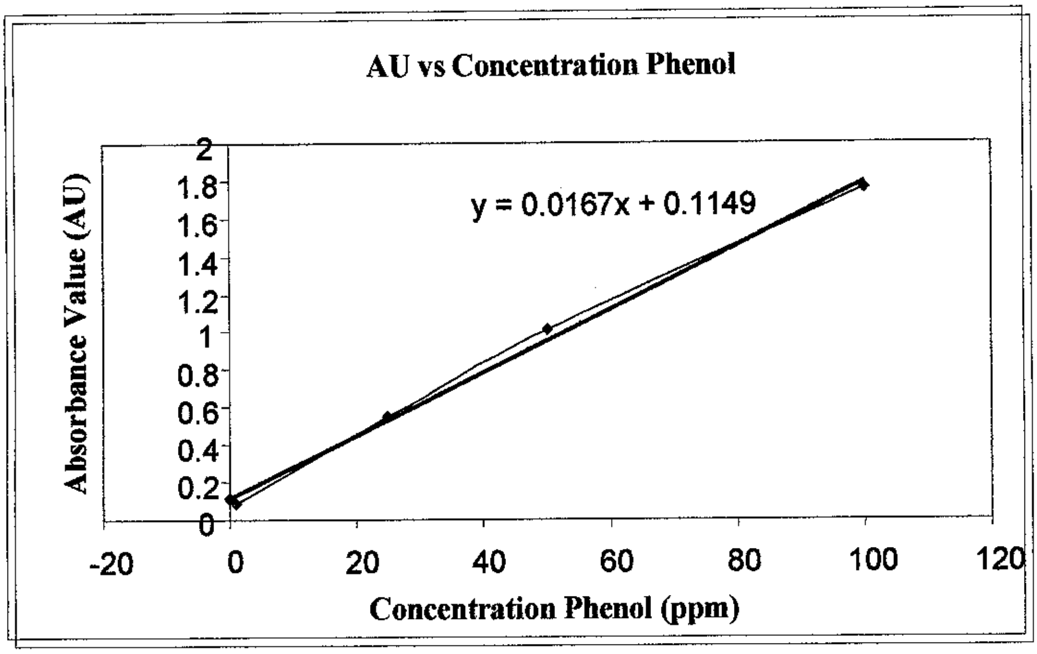

Figure 5.1 Standard Curve of Phenol

5.2.2 Results on Adsorption Isotherm Measurement

The data obtained from the experimental section has been transferred into graphical form for easier understanding. The graph of adsorbate adsorbed, q (mg/g) versus the final equilibrium concentration, c (ppm) are plotted in two different ways. First graphs (refer Figure 5.2 to 5.4) vary the types of activated carbon used with the temperature maintained at the same value while second graphs (refer Figure 5.5 to Figure 5.8) vary the temperatures for the same type of activated carbons. From these graphs, it is easier to make comparison between the adsorption capacity of each activated carbon in removing phenol at the same temperature as well as the effect of temperature on the adsorption capacity of each activated carbon.

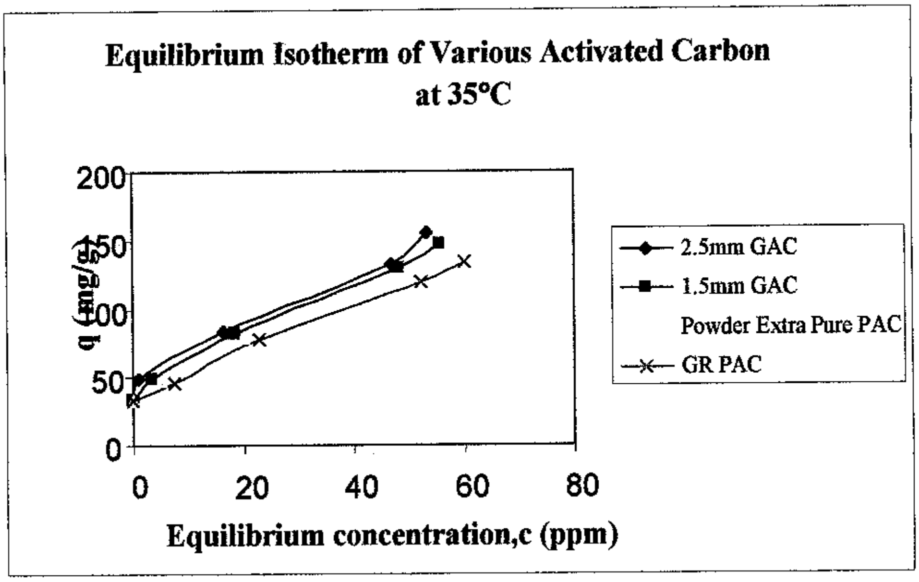

Figure 5.2 Isotherm Plot of Phenol on 4 types of Activated Carbon at T=35°C

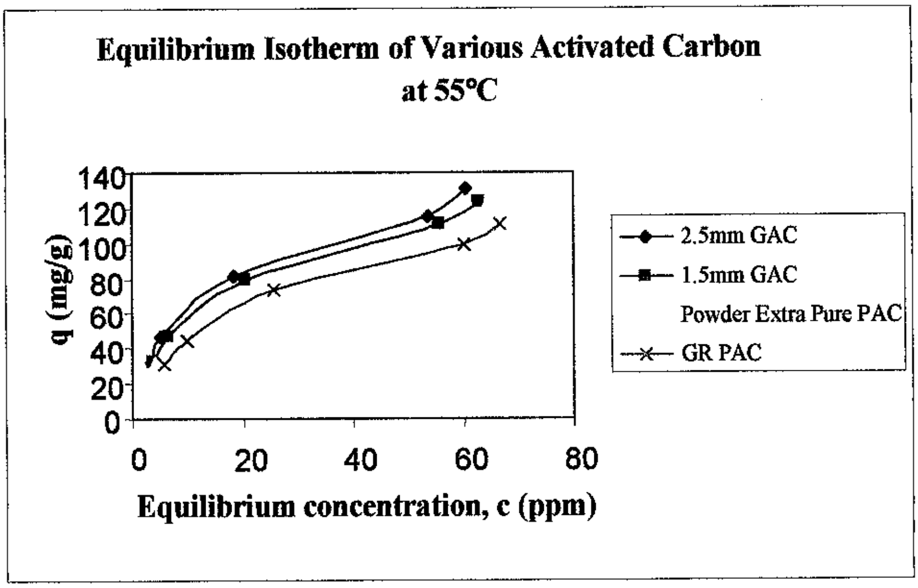

Figure 5.3 Isotherm Plot of Phenol on 4 types of Activated Carbon at T=55°C

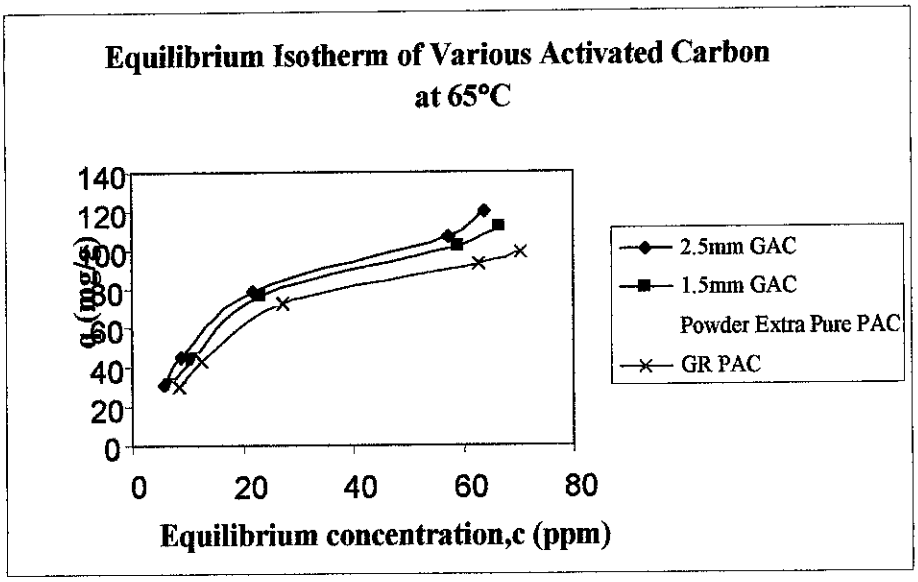

Figure 5.4 Isotherm Plot of Phenol on 4 types of Activated Carbon in T=65°C

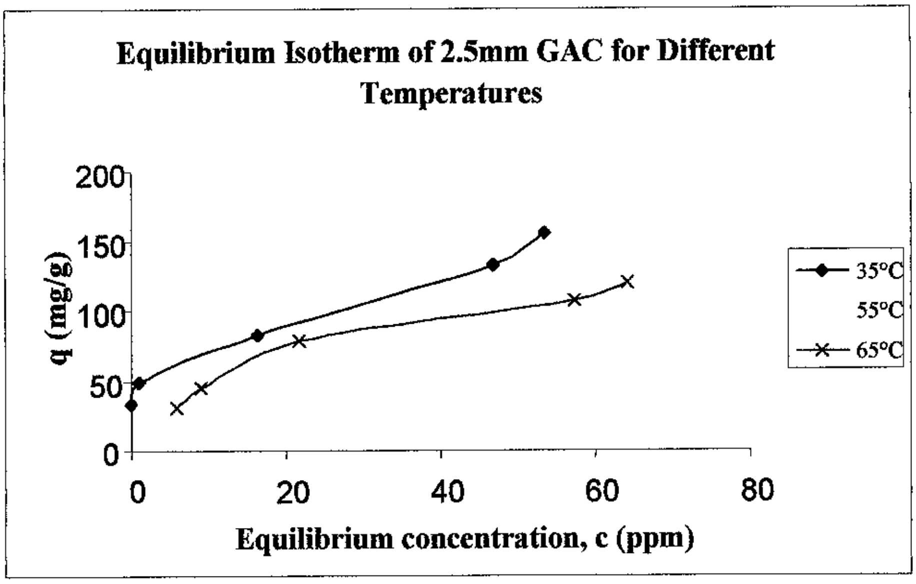

Figure 5.5 Isotherm Plot of Phenol on 2.5mm Granular Activated Carbon at Various Temperatures

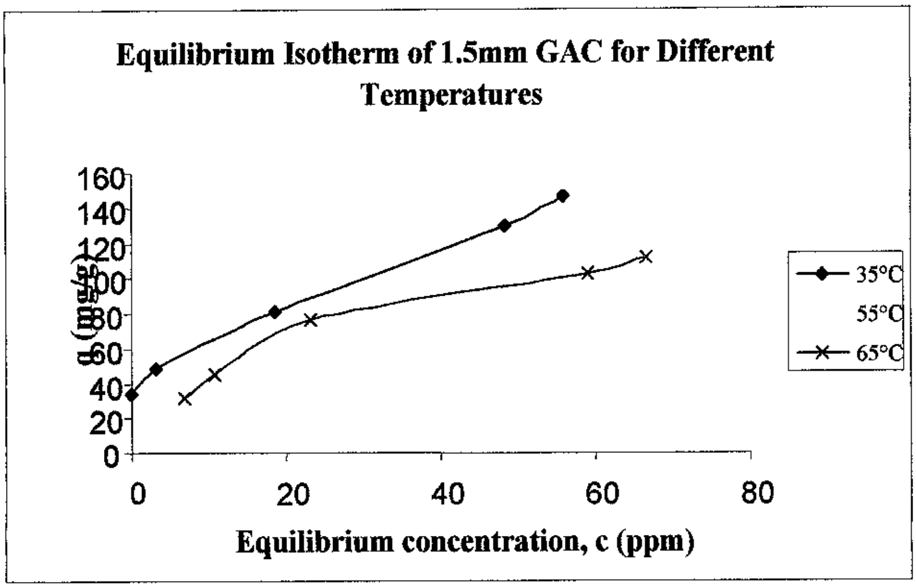

Figure 5.6 Isotherm Plot of Phenol on 1.5mm Granular Activated Carbon at Various Temperatures

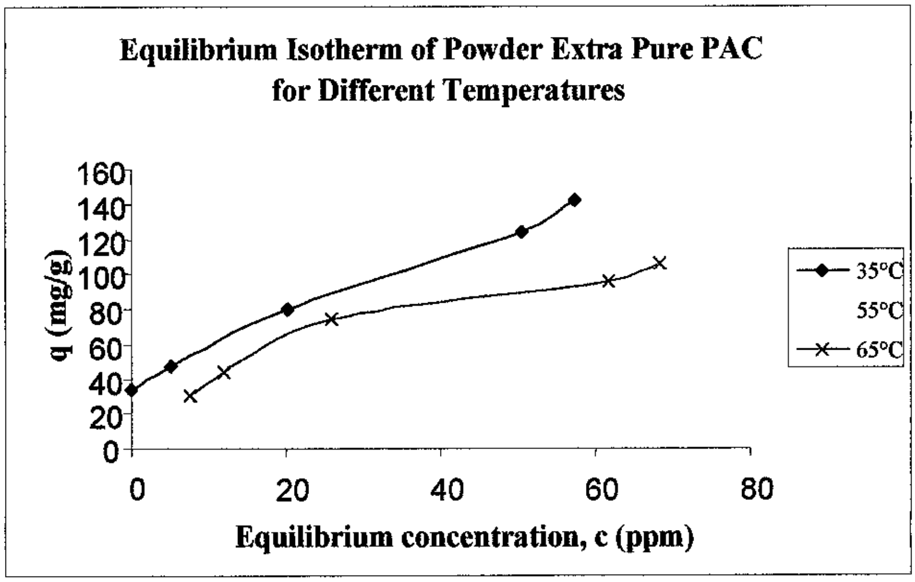

Figure 5.7 Isotherm Plot of Phenol on Powder Extra Pure of Powdered Activated Carbon at Various Temperatures

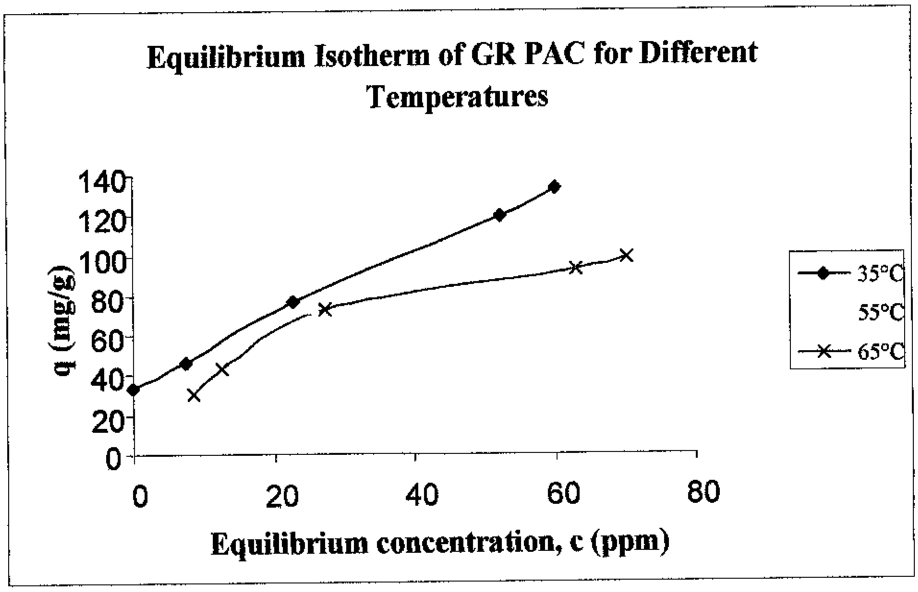

Figure 5.8 Isotherm Plot of Phenol on GR of Powdered Activated Carbon at Various Temperatures

5.2.2 Results of Characterization of Adsorbents

Some of the physical properties of adsorbents have been analyzed using QUANTACHROME AUTOSORB-1. The results are shown in Table 5.1.

Sample	Specific surface area, Sg (m^2/g)	Average pore diameter, dp (nm)	Micropore analysis		
			Micropore volume (cm^3/g)	Micropore surface area, S_{micro} (m^2/g)	Average pore width (nm)
2.5mm GAC	3419.66	1.995	1.868	5255	5.641
1.5mm GAC	2046.77	1.979	1.073	3021	3.563
Powder Extra Pure PAC	1927.00	2.114	1.018	2865	4.51
GR PAC	1876.51	2.088	0.9901	2786	4.761

Table 5.1 Physical Characteristic of Four Types of Activated Carbon

5.3 Discussions

As stated in Section 1.3, the objectives of this research are to determine the adsorption isotherm of phenol and to compare the degree of adsorbate removal by using various types of activated carbon, to characterize various types of activated carbons used, in term of their surface area, pore diameter and micropore size distribution and lastly to analyze the effect of various temperatures on the adsorption capacity of phenol on different types of activated carbon. This section provides detailed discussion and justification on the experimental works and finding to achieve these three objectives.

5.3.1 Discussion on Adsorption Isotherm Measurement

As been explained in Section 5.1, the graphical datas for adsorption isotherm measurement are presented in two different ways. From Figure 5.2, it is clear that at 35°C, 2.5 mm diameter Granular Activated Carbon (GAC) has the highest adsorption capacity, followed by 1.5 mm diameter GAC, Powder Extra Pure Powdered Activated Carbon (PAC) and lastly GR of PAC. The same results are observed from Figure 5.3 and 5.4.

In this experiment, 100 ppm phenol solution is used and the amount of activated carbon is added to the solution in five different mass, ranging from 0.03 to 0.3 g. The solution of phenol and activated carbon is then left for one night to ensure that the adsorption process is completed and equilibrium is reached. From the result obtained from Figure 5.2 to 5.4, it is observed that, the highest mass of activated carbon, that is 0.3 g shows the least concentration of phenol left in the solution due to more phenol has been adsorbed on the activated carbon. In contrast, the smallest mass of activated carbon used, that is 0.03 g resulted in the highest concentration of phenol remaining in the solution. For example, (refer Figure 5.2 and Appendix C.2.1), if 0.03 g 2.5 mm diameter GAC is used as adsorbent at , the concentration of phenol left in the solution is 53.344 ppm, which means that the concentration of phenol that has been adsorbed is only 46.656 ppm. However, the same type of adsorbent which used 0.3 g tends to adsorbed all the phenol. The limited surface area available for small mass of adsorbent restricts the amount of phenol molecule to be adsorbed on the surface. Bigger mass produces larger bonding area which allow larger amount of phenol to be adsorbed, provided that other factor such as temperature is constant. Once all of the bonding sites are filled, the adsorption on the activated carbon will stop.

The characteristic of the phenol itself also has direct impact on the adsorption process. Since phenol is a polar solute, it has two poles that differ in its affinity for the water molecules, so one side of the molecule is hydrophilic (likes water) and the other one is hydrophobic (dislikes water – likes lipids). It tends to ionize in water and produce negatively charge ion of hydroxyl group and correspondingly positive ion of phenyl group. Solvation by water will cause the formation of a bond between the positive hydrogen ion from water and a negative group of solute and hydrophilic nature exists. Water solubility is thus expected to increase with increasing polarity and causes the adsorption capacity of phenol to reduce. Although phenol has high solubility in water, the activated carbons used in this research are still capable in adsorbing the phenol molecule in quite a large amount due to its polar behavior. This is because when the phenol molecule approaches the polar surface of activated carbon, it is attached on it by its hydrophobic (lipophilic) side and thus is removed from the water solution that contained it. It is expected that these activated carbons will adsorb larger amount of adsorbate which has less hydrophilic nature (less solubility in water) compared to its hydrophobic nature.

Beside solubility, the molecular size of phenol also affects the adsorption rates. From literature review done by Hsieh and Teng, (2000), they found that phenol molecules are relatively small with estimated diameter of 0.62 nm. This small molecule of phenol enhances its capability of intraparticle diffusive mass transport within porous activated carbon and is more rapid for smaller molecule. It is assumed that there are no other components in the solution. Sledjko, (1985), had found out that the components of mixture of adsorbates may affect the adsorption capacity since the adsorption of more than one substance generally reduces the number of surface sites available to each. Besides, very slowly diffusing species in mixed adsorbate on porous adsorbents can depress rates of uptake of those which diffuse more rapidly.

5.3.2 Discussions on Characterization of Adsorbents

The physical characteristics of the activated carbons also affect their capability in adsorbing phenol molecule. Table 5.1 shows experimental data regarding some of the physical characteristics of the activated carbons used in this research which focuses on the surface area, pore diameter and micropore analysis such as micropore volume, micropore surface area and micropore width. Focusing on the micropore analysis, it is observed that 2.5 mm diameter GAC has the highest micropore volume which is 1.868 cm^3/g and micropore surface area which is 5225 m^2/g compared to other types of activated carbon.

Since activated carbon is well known for its microporous nature (average pore diameter < 2 nm) as stated by Bansal, et. al., (1988), it is not very efficient in removing larger molecules such as dyes, humid acids, etc. However, since phenol has small molecular size as reported by Hsieh and Teng, (2000), which is less than the average pore diameter of microporous structure, it tends to be adsorbed directly into the micropores and generates either monolayer or multilayer adsorption. It is generally believe that micropores are the major provider of adsorptive sites in the phenol solution. This theory is directly proportional to the experimental result which shows that 2.5 mm of GAC has the highest adsorption capacity due to the highest micropores volume as shown in Table 5.1. Thus, the extent of uptake of phenol conforms to the micropores volume of the activated carbons. The micropores volume, and also the phenol uptake, is in the order of 2.5 mm of GAC > 1.5 mm of GAC > Powdered Extra Pure of PAC > GR of PAC.

The conclusion that adsorption of phenol is limited by the micropore volumes of the activated carbons satisfy with the result of other researches (refer Chapter 2).

5.3.3 Discussion on the Effect of Temperatures on Adsorption

As seen in Figure 5.5 through 5.8, an increase in temperature reduces the adsorption capacity. This behavior can directly be related to the adsorption reaction itself. Since the adsorption reactions is exothermic, in tends to release heat whenever phenol molecule is adsorbed on the surface of activated carbon. However, as the temperature of the phenol solution increases, more energy will be gained by the molecules, thus favor the molecules to be escaped from the surface of the activated carbon. Thus, at high temperature, more phenol molecules will be evaporated, or so call desorption process, as evidence to less amount of phenol adsorbed. The desorption process, which is an endothermic reaction is the reverse of adsorption. Since adsorption is the main concern, the position of the equilibrium has to be shifted as far as possible to the right in order to produce maximum capacity of adsorption.

According to Le Chatelier's Principle, this will only be favored if the temperature is lowered (Carey, 2000). In an endothermic reaction, heat can be considered as a reactant, whereas in an exothermic reaction, heat is a product.

Endothermic : Reactants + heat ⇔ products

Exothermic : Reactants ⇔ products + heat

As the temperature is increased, the equilibrium shifts in the direction that absorbs heat. In an exothermic reaction such as adsorption of phenol on activated carbon, heat is released as phenol molecule is adsorbed on the surface of the activated carbon. Thus, increasing the temperature causes the equilibrium to shift to the left, in the direction of desorption process which cause less amount of phenol adsorbed.

However, cooling a reaction has the opposite effect of heating it. As the temperature is lowered, the equilibrium shifts to the side that produces heat. Thus cooling the adsorption process causes the reaction to be shifted to the right which favors the adsorption to occur instead of desorption.

From Figure 5.5 and Appendix C.2, it is observed that for the adsorption of phenol using 0.03 g of 2.5 mm diameter GAC, the concentration of phenol left in the solution at equilibrium is higher for 65°C that is 64.076 ppm compared to only

53.344 ppm for 35°C. From this, it can be concluded that more phenol is adsorbed on the activated carbon for 35°C compared to for 65°C. This result conforms to the theory which has been discussed previously. As the conclusion, increasing the temperature reduces the adsorption capacity and favors desorption process to occur.

Besides, any increment in temperature causes the increase in rate of adsorption. This is because, an increase in temperature increases the speed of adsorption, thus the time taken to reach equilibrium. This phenomenon is called diffusivity. In almost every adsorption process, diffusion is the rate limiting step, thus the role of diffusion in adsorption process is quite important. Since most practical solid used in industries such as activated carbon are porous and the overall adsorption rate is limited by the ability of the adsorbate to diffuse into the particle interior, it is very valuable to study the effect of diffusivity to adsorption process. Yet, a system having high mobility in both free and adsorbed phase does not necessarily mean that it would approach equilibrium faster. This is because, for the equilibrium to occur, it also depends on the quantity which can be accommodated by the solid at equilibrium.

From this, it can be concluded that the speed to approach equilibrium depends on two factors that is rate and capacity of adsorption. However, the effect of temperature on the rate of adsorption is not covered in this research.

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

6.1 Conclusions

The complete adsorption isotherm measurement of phenol using different types of activated carbons at various temperatures was successfully achieved. The correlation between the adsorption capacity of each activated carbon to its physical characteristics as well as temperatures effect was effectively determined.

Among four different types of activated carbons used, 2.5 mm diameter Granular Activated Carbon (GAC) appeared to be the best option for removal of phenol. This is followed by 1.5 mm diameter GAC, Powder Extra Pure of Powdered Activated Carbon (PAC) and lastly GR of PAC. This result is directly affected by the size of the phenol molecule itself as well as the microporous nature of the adsorbent. It is expected that the same types of activated carbon will be more efficient in removing other types of adsorbate found in the wastewaters stream.

Besides, this experiment also proved the theory that adsorption is favored at lower temperature. This is due to the exothermic reaction of the adsorption process, which favor cooler environment in order to allow adsorption process to occur, instead of desorption. At higher temperature, desorption is much more favored compared to adsorption. However, the rate of adsorption tends to increase for higher temperature.

The outcome of this project is not only useful for preliminary studies of adsorption phenomenon, but it also provides the reliable methodology and procedures which can be followed in future studies and analysis of similar applications.

6.2 Recommendations for Future Work

The study on the removal of phenol in the wastewaters stream using various types of activated carbon at different temperatures has been conducted prior to the increased in the water pollution in many countries, which is caused by this compound. Thus, search has to be done to find the efficient and economical control strategies for removal of this organic compound. As this is only the preliminary study which aiming toward the application in waste water effluent treatment, lot of improvements and adjustments should be made to predict the actual performance.

The correlations of the experimental data were performed from 35 °C up to the temperature of only 65 °C. In the actual process condition, temperature might be higher or lower than this range. The findings obtained in this study can predict the adsorption equilibrium at these temperatures but it is by extrapolation. Thus, to confirm the predicted result, the isotherm measurement at the respective temperature needs to be performed.

Further, by considering the adequate contact time between the adsorbent and adsorbate, it might be possible to know the exact amount of adsorbent needed to treat per unit weight of adsorbate at the specific contact time in order to allow the adsorption equilibrium to occur. This is advantage since enough time can be allowed for every dosage of adsorbent to yield valid data points. Besides, it is also recommended to consider the particle size of adsorbent since it tends to affect the rate of equilibrium, although the equilibrium capacity is not changed. Since smaller particle simply reach equilibrium faster than larger ones, pulverized carbon should be used in order to increase the rate of adsorption so that laboratory testing time is minimized.

Since the process stream condition has certain pH that will affect the adsorption process, it might be necessary to conduct the experiment by ranging the pH of the initial solution. In this way, it is possible to predict whether the added cost of pH adjustment will be offset with the savings in the quantity of adsorbent used.

It is also possible to consider other physical characteristics such as macropores and mesopores analysis to confirm that the adsorption of phenol is mostly occur on the microporous structure.

To ensure the accuracy of the analysis on the physical characteristics of the activated carbons used, the experiment using Quantachrome AUTOSORB-1 analyzer should be repeated, probably three times. Besides, it is also better to compare the physical characteristics that had been analyzed with the information from the vendor itself.

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APPENDIX A

DATA FOR LITERATURE REVIEW

Sample	S_{gBET} m^2/g	$S_{gmesopores}$ m^2/g	$V_{micropore}$ cm^3/g	V_{total} cm^3/g	$V_{mesopore}$ cm^3/g	V_{me}/V_{tot} %
CZ124	2191	1208	0.549	1.913	1.364	71
CZ126	2728	664	0.907	1.594	0.687	43
CK17	2334	569	0.67	1.565	0.895	57
CK22	2450	148	1.049	1.216	0.167	14
Filtasorb 100	937	74	0.391	0.494	0.103	21

Table A.1 Porosity of Activated Carbon, (http://www.lenntech.com/ adsorption.htm)

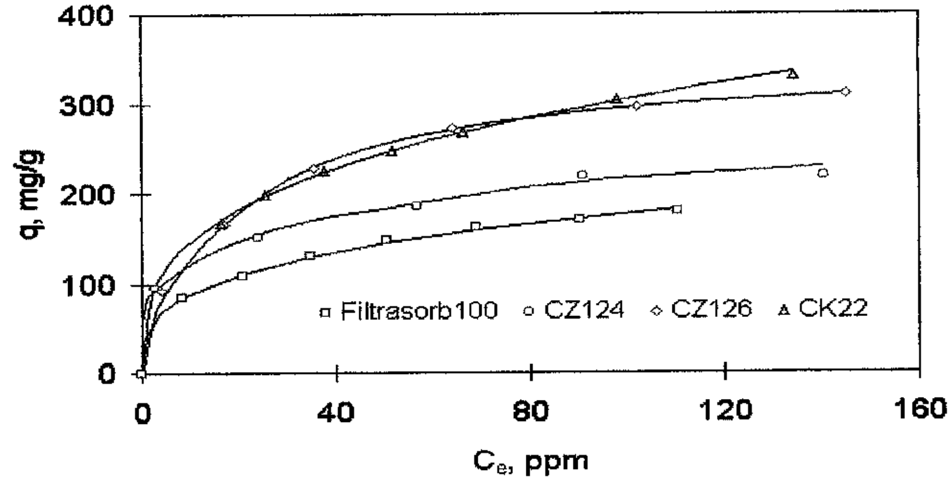

Figure A.1 Adsorption Isotherms for Phenol on Various Adsorbents (http://www.lenntech.com/adsorption.htm)

APPENDIX B

DATA FOR METHODOLOGY

Since the concentration of solid phenol in the laboratory was much higher than 100 ppm that is required, the solid had to be diluted with water (maintaining 1L volume of water). The sample calculation is shown below:

Required concentration = 100 ppm

Volume water = 1L

Required amount of phenol = (100 mg/L × 1L)

= 100 mg

APPENDIX C

DATA FOR THE ADSORPTION ISOTHERM MEASUREMENT

C.1 Experimental Data for Calibration Curve

Standard sample	
Concentration phenol (ppm)	**AU**
100	1.754
50	1.009
25	0.552
1	0.088
0	0.1149

Table C.1.1 Data for the calibration curve

C.2 Experimental Data for Adsorption Equilibrium

35 degree					
2.5 mm of Granular Activated Carbon					
Carbon Weight (g)	**Volume solution, (L)**	**AU**	**Phenol remained, c (mg/L)**	**Phenol removed (mg/L)**	**q (mg/g)**
0.03	0.1	0.975	53.344	46.656	155.52
0.04	0.1	0.833	46.854	53.146	132.865
0.1	0.1	0.388	16.349	83.651	83.651
0.2	0.1	0.133	1.08	98.92	49.46
0.3	0.1	0.096	0	100	33.333333
1.5 mm of Granular Activated Carbon					
Carbon Weight (g)	**Volume solution, (L)**	**AU**	**Phenol remained, c (mg/L)**	**Phenol removed (mg/L)**	**q (mg/g)**
0.03	0.1	1.032	55.897	44.103	147.01
0.04	0.1	0.935	48.233	51.767	129.4175
0.1	0.1	0.424	18.519	81.481	81.481
0.2	0.1	0.185	3.29	96.71	48.355
0.3	0.1	0.199	0	100	33.333333
POWDER EXTRA PURE of Powdered Activated Carbon					
Carbon Weight (g)	**Volume solution, (L)**	**AU**	**Phenol remained, c (mg/L)**	**Phenol removed (mg/L)**	**q (mg/g)**
0.03	0.1	1.143	57.345	42.655	142.18333
0.04	0.1	0.87	50.543	49.457	123.6425
0.1	0.1	0.312	20.178	79.822	79.822
0.2	0.1	0.095	5.034	94.966	47.483
0.3	0.1	0.087	0	100	33.333333
GR of Powdered Activated Carbon					
Carbon Weight (g)	**Volume solution, (L)**	**AU**	**Phenol remained, c (mg/L)**	**Phenol removed (mg/L)**	**q (mg/g)**
0.03	0.1	1.148	60.035	39.965	133.21667
0.04	0.1	0.989	52.312	47.688	119.22
0.1	0.1	0.266	22.756	77.244	77.244
0.2	0.1	0.135	7.456	92.544	46.272
0.3	0.1	0.104	0	100	33.333333

Table C.2.1 Experimental Data for Adsorption Equilibrium at 35 °C

55 degree

2.5 mm of Granular Activated Carbon

Carbon Weight (g)	Volume solution, (L)	AU	Phenol remained, c (mg/L)	Phenol removed (mg/L)	q (mg/g)
0.03	0.1	1.061	60.534	39.466	131.55333
0.04	0.1	0.958	53.652	46.348	115.87
0.1	0.1	0.287	18.456	81.544	81.544
0.2	0.1	0.123	4.973	95.027	47.5135
0.3	0.1	0.093	3.252	96.748	32.249333

1.5 mm of Granular Activated Carbon

Carbon Weight (g)	Volume solution, (L)	AU	Phenol remained, c (mg/L)	Phenol removed (mg/L)	q (mg/g)
0.03	0.1	1.117	62.911	37.089	123.63
0.04	0.1	0.943	55.801	44.199	110.4975
0.1	0.1	0.341	20.567	79.433	79.433
0.2	0.1	0.138	6.456	93.544	46.772
0.3	0.1	0.105	3.929	96.071	32.023667

POWDER EXTRA PURE of Powdered Activated Carbon

Carbon Weight (g)	Volume solution, (L)	AU	Phenol remained, c (mg/L)	Phenol removed (mg/L)	q (mg/g)
0.03	0.1	1.148	64.039	35.961	119.87
0.04	0.1	1.03	57.855	42.145	105.3625
0.1	0.1	0.431	22.954	77.046	77.046
0.2	0.1	0.147	8.956	91.044	45.522
0.3	0.1	0.108	4.113	95.887	31.962333

GR of Powdered Activated Carbon

Carbon Weight (g)	Volume solution, (L)	AU	Phenol remained, c (mg/L)	Phenol removed (mg/L)	q (mg/g)
0.03	0.1	1.136	66.587	33.413	111.37667
0.04	0.1	1.106	60.278	39.722	99.305
0.1	0.1	0.486	25.749	74.251	74.251
0.2	0.1	0.204	10.034	89.966	44.983
0.3	0.1	0.138	5.89	94.11	31.37

Table C.2.2 Experimental Data for Adsorption Equilibrium at 55 °C

65 degree					
2.5 mm of Granular Activated Carbon					
Carbon Weight (g)	**Volume solution, (L)**	**AU**	**Phenol remained, c (mg/L)**	**Phenol removed (mg/L)**	**q (mg/g)**
0.03	0.1	1.137	64.076	35.924	119.74667
0.04	0.1	0.973	57.367	42.633	106.5825
0.1	0.1	0.41	21.735	78.265	78.265
0.2	0.1	0.19	8.908	91.092	45.546
0.3	0.1	0.137	5.815	94.185	31.395
1.5 mm of Granular Activated Carbon					
Carbon Weight (g)	**Volume solution, (L)**	**AU**	**Phenol remained, c (mg/L)**	**Phenol removed (mg/L)**	**q (mg/g)**
0.03	0.1	1.179	66.544	33.456	111.52
0.04	0.1	1.052	59.135	40.865	102.1625
0.1	0.1	0.436	23.265	76.735	76.735
0.2	0.1	0.189	10.786	89.214	44.607
0.3	0.1	0.153	6.751	93.249	31.083
POWDER EXTRA PURE of Powdered Activated Carbon					
Carbon Weight (g)	**Volume solution, (L)**	**AU**	**Phenol remained, c (mg/L)**	**Phenol removed (mg/L)**	**q (mg/g)**
0.03	0.1	1.176	68.367	31.633	105.44333
0.04	0.1	1.062	61.789	38.211	95.5275
0.1	0.1	0.46	25.783	74.217	74.217
0.2	0.1	0.187	11.983	88.017	44.0085
0.3	0.1	0.126	7.521	92.479	30.826333
GR of Powdered Activated Carbon					
Carbon Weight (g)	**Volume solution, (L)**	**AU**	**Phenol remained, c (mg/L)**	**Phenol removed (mg/L)**	**q (mg/g)**
0.03	0.1	1.232	70.377	29.623	98.743333
0.04	0.1	1.119	63.064	36.936	92.34
0.1	0.1	0.557	27.278	72.722	72.722
0.2	0.1	0.242	12.635	87.365	43.6825
0.3	0.1	0.184	8.563	91.437	30.479

Table C.2.3 Experimental Data for Adsorption Equilibrium at 65 °C