

ADSORPTION OF PHENOL BY ACTIVATED CARBON PRODUCED FROM
DECANTER CAKE

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ADSORPTION OF PHENOL BY ACTIVATED CARBON PRODUCED FROM
DECANTER CAKE

ASMA SHUFIEYAH BT ZAINAL ABIDIN

A thesis submitted in fulfillment of the
requirements for the award of the degree of
Bachelor of Chemical Engineering

Faculty of Chemical and Natural Resources Engineering
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APRIL 2009

DECLARATION

I declare that this thesis entitled "*Adsorption of phenol by activated carbon produced from decanter cake*" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature :

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Date : APRIL 2009

DEDICATION

*Special dedication to my beloved father, mother, Alkarim, Syifa, Safwan,
Ubai, Fitry and Firdaus*

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ABSTRACT

Activated carbon from agricultural waste can be produce from various methods, either through physical activation or chemical activation. The studies involve on producing activated carbon from decanter cakes by using chemical activation method. The chemical agent use is zinc chloride as in previous research; the agent is very efficient in producing activated carbon with high surface area and large pores. The efficiencies of the activated carbon are tested with the removal capacities of organic waste such as phenol. Phenol is chosen because phenols are widely present in the industrial waste and its effect is harmful to the environment. For the preparation of activated carbon, the suitable ratio of zinc chloride to the decanter cakes is 3:5 and the effective temperature of pyrolysis are 700°C and 900°C. For the pH effect, the removal is increased as the pH increased from 2 to 6 and starts to decrease at pH of 8 until 12. Activation with zinc chloride can remove phenol more than 90% at optimum condition.

ABSTRAK

Karbon aktif daripada hasil buangan pertanian boleh dihasilkan melalui pelbagai kaedah samada melalui pengaktifan fizikal atau pengaktifan kimia. Kajian ini berkisar tentang menghasilkan karbon aktif daripada lumpur yang terhasil daripada proses pengasingan minyak dari lumpur kelapa sawit. Proses mengaktifkan karbon ini menggunakan kaedah pengaktifan kimia. Agen kimia yang digunakan ialah zink klorida kerana kajian yang lepas menunjukkan agen ini amat efisien dalam menghasilkan karbon aktif dengan luas permukaan dan liang yang besar. Keupayaan karbon aktif ini diuji melalui penjerapan komponen organik seperti fenol. Fenol dipilih kerana ia banyak dibuang oleh industri-industri dan ini memberikan impak berbahaya kepada alam sekitar. Dalam proses menyediakan karbon aktif, nisbah zink klorida kepada lumpur kelapa sawit yang sesuai ialah 3:5 dan suhu optima proses pirolisis ialah 700 dan 900°C. Untuk kesan pH, penjerapan fenol meningkat apabila pH larutan meningkat daripada 2 hingga 6. Ia mulai menurun pada pH 8 hingga 12. Pengaktifan dengan zink klorida boleh menyingkirkan fenol sehingga lebih daripada 90 peratus pada kondisi yang optima.

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Activated carbon (AC) is the carbonaceous material which plays an important role in adsorption process. Its ability to remove organic and inorganic chemical waste, odor, color and taste from any kind of chemical industry process is based on their amazing properties. Activated carbon has high degree of surface reactivity which can influence its interaction with polar or nonpolar adsorbates. Besides, it also has higher surface area and micro porous structure. Activated carbon are widely use in wastewater treatment to remove harmful chemicals and heavy metal, industrial waste water or industrial flue gas. Their application in industry includes removing organic and inorganic pollutants from drinking water, industrial wastewater treatment, decolorizing of syrups and purification of air and pharmaceutical products.

Because of their wide usage in industry, the demand of the activated carbon is increasing year by year. Commercial activated carbon is quite expensive. As such industry now seeks for the cheapest activated carbon derive from agriculture waste or residuals. The residuals can be rice husk, palm oil mill sludge, sawdust or other carbonaceous material. The raw material were processed and optimized to obtain excellent adsorptive properties. Activated carbon can be produced either by physical activation or chemical activation. Chemical activation only involves single step in heating process to activate the carbon. This process needs chemical activating agent such

as ZnCl_2 , KOH , H_3PO_4 or H_2SO_4 to enhance carbon yield and produce micro pores on the surface of the carbon. Physical activation involves two steps of heating process and does not involve any chemicals.

Phenol is an organic chemical usually present in industrial waste water. The substance is very dangerous to human even in small concentration. Phenols are long term effect pollutants and can be found in industries which produce chlorophenols that are widely used as fungicides and insecticides for agriculture sector. Phenolic compounds widely used for the commercial production of a variety of resins. Phenolic compounds in potable water emit an unpleasant odor and flavor in concentration as low as $5\mu\text{g/l}$ and are poisonous to aquatic life, plants and human as a product of the bio-concentration. Ingestion of phenols in concentration from 10 to 240 mg/l for a long time causes mouth irritation, diarrhea, and excretion of dark urine and vision problems. (Navaro *et al* 2007). Besides, World Health Organization (WHO) followed the EPA'S lead the same year by establishing the maximum level of phenol concentration in drinking water at $1\mu\text{g/l}$.

Hence activated carbon is the most suitable material to be used to adsorb this harmful chemical because of their larger surface area. Decanter cake is the rich carbonaceous material which remains after separation of oil from the sludge. The potential of decanter cake to adsorb organic chemical such as phenol is studied.

1.2 Identification of Problem

The activated carbon derives from the agriculture residual seems to be increasing and hence the activated carbon produced are cheap. It has the excellent properties to adsorb harmful chemical such as phenol, arsenic, and other heavy metal from drinking water and industrial wastewater stream. Malaysia is the third biggest producer of palm oil in the world. Tonnes of residual are producing include sludge disposes to the land. Besides it will consume more money for the mills to manage their waste. Phenol is highly toxic and present in most of industrial wastewater. To treat the wastewater that contains phenol is important for the environment. Phenol can cause damages to kidney, liver and pancreas when enter the human body through ingestion. It react with the body by degrade the protein and cause tissue erosion. (Qadeer and Rehan, 2002)

1.3 Statement of Objectives

- i. To produce and characterized the activated carbon from the palm oil sludge by using chemical activation process.
- ii. To study the optimal condition (temperature of pyrolysis process, the shaking time and the amount of activation agent i.e. $ZnCl_2$) of adsorption process.
- iii. To study the effect of initial concentration and initial pH of the phenol solution to the adsorption capacity.

1.4 Scope of Research

- i. Particle size of activated carbon is 125 μ m for chemical activation process.
- ii. ZnCl₂ to sludge mass ratio of 2:5 and 3:5
- iii. Temperature and time is 85°C and 8 h for impregnation sludge with zinc chloride by magnetic stirrer.
- iv. Temperatures of pyrolysis process are 300°C, 500°C, 700°C and 900°C.
- v. pH of initial phenol solution are ranging from 2 to 12
- vi. The amount of adsorbent ranging from 0.05 to 0.2g for adsorption analysis.
- vii. Concentration of initial phenol solution are 25, 50, 100, 150, 200 and 250 mg/L
- viii. For phenol analysis, by using UV-visible spectrophotometer, the wavelength used is 725nm.

1.5 Rationale and Significance

This research is done based on the significance of activated carbon production in Malaysia. In the future Malaysia would be the leading of producer of activated carbon from palm oil mill sludge. The sludge can be recycled into profitable and valuable product. It will give the benefit to the environment, country development and the palm oil mill itself if the sludge were converted to activated carbon. The palm oil mill industries will reduce the space for decanter cake's disposal. The activated carbon produce play an important role to remove carcinogenic substance such as phenol from the plastic, paint and dye industries effluent before discharging to the water streams, sea, lakes and rivers. Hence, this research would give benefit to the palm oil milling and chemical industries to manage their waste disposal.

“The processing and transformation of agricultural residues into activated carbon with good adsorption properties would alleviate problems of disposal and management

of these waste by-products, while providing a high quality end product for water and wastewater treatment that could potentially expand the carbon market (Kalderis and Bethanis, 2008).”

CHAPTER 2

LITERATURE REVIEW

2.1 Definition of Activated Carbon

Activated carbon is the carbonaceous material that is very effective adsorbents for organic compounds. Activated carbon is versatile adsorbents. It can be produced from carbonaceous material in order to provide adsorptive properties. The adsorptive properties depend on their high surface area, microporous structure and high degree of surface reactivity. The chemical structure on the carbon surface will influence the interaction between polar and nonpolar adsorbates. Activated carbon can be used to adsorb organic and inorganic species either from gaseous and aqueous phases. Activated carbon's properties depend on:

- i. Chemical and porous structure of carbon (nature and concentration of surface chemical groups).
- ii. The polarity of the surface.
- iii. The carbon surface area.
- iv. Pore size distribution.
- v. Physical and chemical characteristics of adsorbate. (Bansal, 2005)

2.1.1 Application of Activated Carbon

Activated carbon is widely use in industries. Examples of its usage as:

- a. Removal of Nitrogen from air.
- b. Removal of Hydrogen from syn-gas and hydrogenation process.
- c. Recovery of solvent vapors.
- d. Removal of SO_x and NO_x.
- e. Purification of helium.
- f. Decolorizing of syrups, sugars and molasses.
- g. Water purification in removing phenol, halogenated compounds, pesticides, caprolactum and chlorine.
- h. Purification of many chemical, food and pharmaceutical products.
- i. Air purification in inhabited spaces likes (restaurants and food processing industries).

2.2 Activated Carbon from Agriculture Waste Based

“Liquid and solid waste, are a huge and always increasing problem for the industries and the environment. One of the most popular ways of dealing with solid waste is its use as a raw material for the production of activated carbons that can be subsequently employed for the removal of pollutants from liquid waste. As adsorbents, activated carbons bear extremely large surface area, with varying porous structure consisting of a network of interconnected macropores, mesopores and micropores. Their surface area also presents great diversity in terms of surface charge and surface groups, depending on the original raw material and the way of activation. For a raw material to be used for activated carbon production, it should have high carbon content. Agricultural waste is commonly exploited this way (Michailof *et al*, 2008).”

“The activated carbon can be derived from agriculture waste for example palm oil mill waste, rice husk, olive waste, dates stone and other agriculture residuals. The carbonaceous material of agriculture waste made its very suitable for use as activated carbon. But the properties must be enhanced by the carbonization process. There are two steps in carbonization process, chemical activation process and physical activation process.” (Bansal, 2005)

Two processes involved:

a) Chemical activation process:

This process involve only single step of carbonization process. The activated agent must be used before carbonization takes place. The activated agent can be zinc chloride, sulphuric acid, potassium hydroxide and phosphoric acid. This agent will increase the surface area and reduce the ash content of final carbonized products. The chemical activation process is most popular because only required single heating and the temperature use usually lower than that of physical activation. But the process can generate secondary environmental pollution due use of chemical.

“Chemical activation presents two important advantages, a much higher yield (27–47% for chemical activation and 6% physical activation for $\sim 2500 \text{ m}^2/\text{g}$ activated carbon fibers and secondly the surface of the fibers prepared by chemical activation is less damaged than by physical activation.” (Maciá-Agulló *et al*, 2004).

b) Physical activation process:

The physical activation involve oxidizing the raw material at high temperatures before the presence of an oxidizing agent, usually, water steam. Because this is an endothermic reaction generally, a constant 800°C temperature must be generated. The temperature varies depending on the raw material.

2.3 Application of Other Waste as Activated Carbon

Any carbonaceous materials are potentially becomes activated carbon. Besides low cost of raw material, these materials are easily found in many agricultural wastes. It can be alternatives for the synthetic activated carbon as the efficiencies are similar or greater than the synthetic activated carbon.

Highly cost of synthetic activated carbon nowadays have been attempts to utilize the low cost, naturally occurring adsorbents to remove trace organic contaminants from wastewater. Organic wastes that can be used to remove organic contaminants from wastewater stream are fly ash, peat, soil, rice husk and activated sludges. In particular, dried activated sludge may be used as low cost, natural and abundant sources for the removal of phenol. Calace *et al* (2002) proved that papermill sludge has a potential to remove phenol from wastewater over a range of concentration more than 800mg/L. Papermill sludge also can be used as low cost and natural abundant sources for phenol removal.

“Biomass from wastewater treatment plant could be used as a biosorbent since it mainly consists of bacteria with cell walls that essentially contain various organic compounds such as acidic polysaccharides, lipids, amino acids, chitins, and other cellular components. These materials interact with chemicals resulting in a passive uptake of chemical from wastewater to the microbial cell. The main biosorption mechanisms appear to be extracellular interactions, complexation and subsequent accumulation, passive sorption at binding sites on the envelopes of cells, and intercellular accumulation.” (Lister and Line, 2001).

In Mediterranean, olive husk has been chosen to be an alternative source of activated carbon. Stavropoulos and Zabaniotou (2005) had claimed that activated carbon from olive kernels or olive cake presented interesting properties and suitable to be a low cost effectively activated carbon.

2.3.1 Preparation of Activated Carbon

The preparation of activated carbon by Kalderis, and Bethanis (1998) is derived from bagasse and rice husk. The methods are as follows:

4 g sample of raw material mixed with chemical reagent in glass beaker at ratio of reagent to raw material sample of 0.25, 0.5, 0.75 and 1.0. Distilled water was added at a quantity amounting to 10 times the total weight.

The mixture then was stirred and heated to become homogenized and impregnated at a temperature of 85°C until a thick uniform paste was obtained. The sample was weighed before placing inside the reactor on top of a metal mesh filter. Part of remaining paste was used to determine the moisture content.

Fixed bed reactor consisted of U-shaped stainless steel reactor (480mm length and 30mm internal diameter) was set up. The reactor equipped with gas inlet pipes and an outlet pipe for pyrolysis gaseous by-products.

Nitrogen gas was allowed to pass through the reactor (200ml/min) in order to remove air from the system. The furnace was heated to the activation temperature, once the targeted temperature reached after 20 min, the gas supply was changed from nitrogen to carbon dioxide. The reactor then was inserted in the furnace for activation process.

Effect of the impregnating agents zinc chloride, sodium hydroxide and phosphoric acid on carbon surface was investigated with ratio of agent to raw material of 0.25, 0.5, 0.75 and 1. Activation was performed at temperature of 400, 600, 700 and 800°C and retention time of 30 min and 60 min were examined.

2.4 Reviews on Palm Oil Milling

In the early 1970s, Malaysia's economic development was based on the agricultural sector. Large areas of forest were converted into oil palm estates. By the end of 1980s, the oil palm plantation covered one third of the country's cultivated area. As a result of expansion in oil palm based industry, during 1975-1985, crude palm oil production rose from 1.3 million tonnes to 4.1 million tonnes making it the country second largest earner of foreign exchange by 1984. (Jayantha *et al*, 2006)

With the growth of agro based industries the oil palm related industries become a major pollution to the 42 rivers in Malaysia. Consequently, this situation led to significant impacts on coastal areas and coastal resources, which in turn affected the socio-economy of the local communities.

The government's efforts to alleviate poverty and at the same time to protect river systems for various beneficial uses saw the following measures being implemented towards the second half of the decade of 1970s (Jayantha *et al*, 2006):

- a) License Fee: A licensing system was introduced to control the pollution load into rivers and onto land. Due to the lack of a proven technology for the treatment of effluents from agro-based industries, a progressive reduction programmed was allowed under the regulation. Under this regulation, a license is necessary before effluents can be dumped into the water or land. The license to discharge effluents was given based on:
 - i. Class of premises
 - ii. Location of premise
 - iii. Quality of waste discharged
 - iv. Pollutants or class of pollutants discharged

v. The existing level of pollution

- b) Phasing in the Standards: In order to give time to the industry to use technology to dispose off their effluents, a mechanism in the progressive phasing in the standards were adopted. The standards set by government have become increasingly stringent overtime.
- c) Effluent-Related Fee: In this implementation, the "polluter pays principle" was adopted in the fee structure. The amount of effluent-related fee payable to the Government was linked to the BOD load of the effluent discharged either onto land, watercourse or both.
- d) Research and Development: The government provided a provision in the regulations authorizing it to grant a partial or full waiver of effluent-related license fees to industries conducting research on effluent treatment as an incentive for the industries to use new technology in their effluents disposal.

“Malaysian palm oil industry continues to contribute significantly to the country’s economic development and foreign exchange earnings. Export earnings of palm oil products was recorded at RM 30.41 billion in 2004 in spite of having strong competition from other palm oil producing countries (MPOB 2005). Environmental impacts from the palm oil industry in Malaysia can be identified from three distinct perspectives. Waste audits identify flow rates and compositions of materials that could be the targets to environmental impacts. Life cycle analysis (LCA) examines individual products, determining rates of waste generation, energy consumption and raw material usage. Industrial ecology examines the uses and wastes associated with particular materials.”
(Jayantha *et al*, 2006)

2.4.1 Palm Oil Milling Process

Figure 2.1 below shows the overall process in palm oil milling factory. The fresh fruit bunch (FFB) was collected from the plantation and processed to produce crude palm oil. The processes involve sterilization of FFB, digestion, pressing, clarification, purification, drying and storage of crude palm oil. Decanter cake is the waste from decanting or centrifuging process.

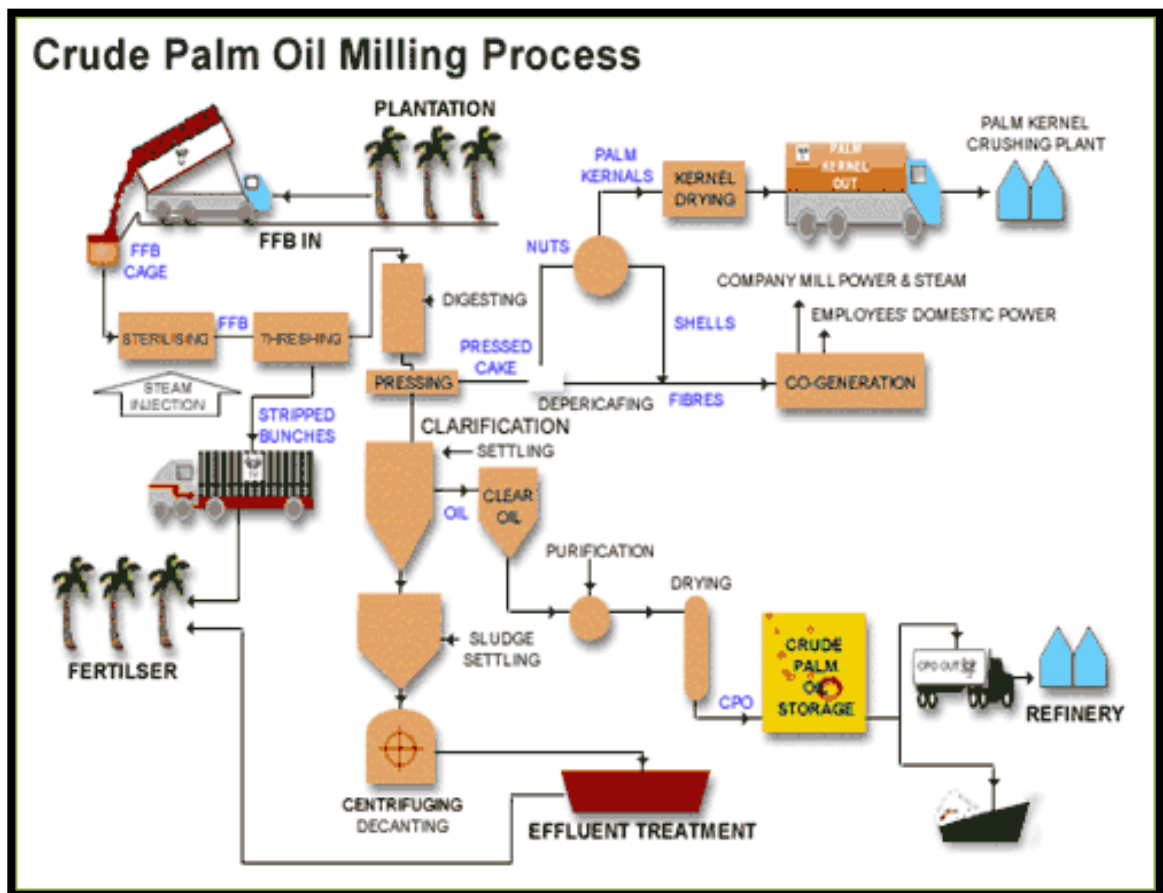


Figure 2.1: Overall Process of Crude Palm Oil Milling

2.4.2 Palm Oil Waste Milling Waste

“The entire crude palm oil process does not need any chemical as a processing aid. Therefore, all substances found in the products, by products and residues are originated from the fresh fruit bunch. However, there are a number of pollution problems at the facility, such as high noise level from machine, high water consumption, generation of high organic content of wastewater, generation of large quantity of solid waste and air pollution (Table 2.1).” (Chavalparit, 2006)

Table 2.1 Summaries of the Emission Associated with the Crude Palm Oil Production

Process	Air emission	Waste water	Solid waste
Loading ramp	-	Oil contaminated wastewater	-
Sterilization	Steam blow down	High organic wastewater	-
Bunch stripping	-	-	Empty fruit bunch
Oil extraction	-	-	Fiber/shell
Oil clarification	-	High organic wastewater	Decanter cake
Steam generation	Vapor	High organic wastewater	-
	Black smoke	-	Ash

2.4.2.1 Solid waste and by-products

“Solid waste and by – products generated in the palm oil extraction process are empty fruit bunches; fibers; shell; decanter cake and ash from boiler. In 2002, Thai crude palm oil industry produces 0.6 million tonnes per year of palm oil from 3.5 million ton of fresh fruit bunches. Palm oil production generates large amounts of process residues

such as fiber 0.5 million ton, shell 0.16 million ton, and empty fruit bunches 0.9 million ton. Solid waste can be reused in production process, oil palm plantation and sold to other industry. A large fraction of the fiber and some of the shell are used as fuel to generate process steam and electricity in the palm processing mill itself. However, much is wasted through dumping in areas adjacent to the mill, or utilized as manure in the palm oil plantation. Palm oil mills in Thailand operate on cogeneration system using biomass residue as fuel in the boiler. The boiler produces high pressure and temperature steam which expands in a backpressure steam turbine and produces enough electric power for the internal needs of the mill. The exhaust steam from the turbine goes to an accumulator which distributes the steam to various processes in the mill.” (Chavalparit, 2006).

Table 2.2: Wastes generated in Jugra Palm Oil Mill, Selangor in June 2006

Waste	Percentage from fresh fruit bunch	For 1 ton of FFB	For 45 ton/hr
• Empty Fruit Bunch	• 23%		• 10.35 ton/hr
• Fiber	• 12%		• 5.4 ton/hr
• Shell	• 7%		• 3.15 ton/hr
• POME	• 50%		• 22.5 ton/hr
• Decanter cake		• 42Kg	• 1.89 ton/hr
• Ash		• 48Kg	• 2.16 ton/hr

2.4.2.2 Application of palm oil mill waste

Table 2.3 below shows the application of palm oil mill waste with different types of waste. There are five wastes produced from palm oil milling that can be used such as empty fruit bunch (EFB), fiber, shell decanter cake and ash. The usage of waste can minimize the palm oil milling cost of disposing beside can reduce the quantity of wastes.

Table 2.3: Applications of Palm Oil Mill Waste (Chavalparit, 2006)

Types of waste	Application
Empty fruit bunch	<ul style="list-style-type: none"> • Soil cover material • Sold for edible mushroom cultivation • As an organic fertilizer (for palm plantation) • As fuel (for milling process)
Fiber	<ul style="list-style-type: none"> • Fuel in milling factory
Shell	<ul style="list-style-type: none"> • Fuel in milling factory • Sold for fuel (other industry) • Charcoal • As activated carbon
Decanter cake	<ul style="list-style-type: none"> • Soil conditioning • Animal feed
Ash	<ul style="list-style-type: none"> • Land filling purpose • Soil conditioning

2.4.2.3 Properties of Palm Oil Milling Waste

Table 2.4 below shows the average nutrient in palm oil mill effluent residue. The nutrient contain in decanter cake are nitrogen (N), phosphorus (P), kalium (K) and magnesium (Mg). The data also include nutrient content after anaerobic and biological treatment which is reducing.

Table 2.4: Average Nutrient Contents in Palm Oil Mill Effluent Residue

Residue	N	P	K	Mg	Quantity kg/ton FFB
<ul style="list-style-type: none"> • EFB, k/ton dry water (30% moisture content) 	8	0.6	24.1	1.8	240.0
<ul style="list-style-type: none"> • Decanter cake ,kg /ton dry (70 % moisture) 	20	8.0	2.0	4.0	42.0
<ul style="list-style-type: none"> • Raw wastewater, kg/m3. 	1.1	0.047	0.1-0.3	2.0	630.0
<ul style="list-style-type: none"> • After anaerobic treatment, kg/m3 	0.34	0.028	0.1-0.3	2.0	
<ul style="list-style-type: none"> • After full biological treatment, kg/m3 	0.03	0.003	0.1	2.0	
<ul style="list-style-type: none"> • Nutrient from EFB application, tons/year 	402	30	1230	90	
<ul style="list-style-type: none"> • WW after anaerobic, ton/1000 ton FFB/ year 	64.3	5.3	37.8	378	
Total	466.3	35.3	1268	468	

Note: * data from Kittikhun, A. et al 2000.

2.5 Introduction of Decanter Cakes

From milling process, decanter cakes are produced from the oil and sludge separation. It contains about 70% of moisture and 30% of solid (25% of the solid are oil). It also contains amino acid and proteins. The color of decanter cake is dark brown. 5 tonnes of decanter cakes are produced from 100 tonnes of fresh fruit bunch. In a day, 800 tonnes of FFB will produce about 40 tonnes of decanter cakes from one palm oil milling plant. Decanter cake is used as soil conditioner and fertilizer by most palm plantation. The industries need to reduce the cost of buying chemical fertilizer for their plantation. Besides, decanter cakes also can be used as animal feed. Further investigation is to convert the decanter cakes into activated carbon. The figure 2.2 below shows the image of decanter cake produce in palm oil milling factory.



Figure 2.2: Decanter Cakes Produce from Lepar Hilir Palm Oil Milling Factory

2.6 Introduction to Phenol

Phenol is a toxic aromatic compound. It is a colorless, crystalline solid with a sweet tarry odor. The chemical formula of phenol is C_6H_5OH and its structure is that of hydroxyl group (-OH) bonded to phenyl ring and thus becomes an aromatic compound. The IUPAC name is Phenol while the other names carbolic acid, benzenol, phenylic acid, hydroxybenzene and phenic acid.

Phenol has a limited solubility in water (8.3 g/100 ml). It is slightly acidic: The phenol molecule has weak tendencies to lose the H^+ ion from the hydroxyl group, resulting in the highly water-soluble phenoxide anion $C_6H_5O^-$. Compared to aliphatic alcohols, phenol shows much higher acidity; it even reacts with aqueous NaOH to lose H^+ , whereas aliphatic alcohols do not. One explanation for the increased acidity is the resonance stabilization of the phenoxide anion by the aromatic ring. In this way, the negative charge on oxygen is shared by the ortho and para carbon atoms. In another explanation, increased acidity is the result of orbital overlap between the oxygen's lone pairs and the aromatic system. In a third, the dominant effect is the induction from the sp^2 hybridized carbons, the comparatively more powerful inductive withdrawal of electron density that is provided by the sp^2 system compared to the sp^3 system allows for great stabilization of the oxyanion. In making this conclusion, one can examine the pK_a of the enol of acetone, which are 10.9 in comparison to phenol with a pK_a of 10.0. (Wikipedia)

2.6.1 Characteristic of Phenol

The characteristic of phenol are summarizing in Table 2.5 below:

Table 2.5: Table Characteristic of Phenol

IUPAC name	Phenol
Other names	Carbolic Acid Benzenol Phenylic Acid Hydroxybenzene Phenic acid
Molecular formula	C ₆ H ₅ OH
Molar mass (g/mol)	94.11
Appearance	White Crystalline Solid
Density (g/cm³)	1.07
Melting point (°C)	40.5
Boiling point (°C)	181.7
Solubility in water	8.3 g/100 ml (20 °C)
Acidity (pK_a)	9.95
Dipole moment	1.7 <u>D</u>

2.6.2 Phenol in Industries Waste Water

Discharge of wastewater/effluent containing organic pollutants into natural surface waters poses serious risk to aquatic organisms and human beings. Phenols find their way into surface water from industrial effluents such as those from coal tar, gasoline, plastic, rubber-proofing, disinfectant, pharmaceutical and steel industries, domestic wastewaters, agricultural runoff and chemical spillage. The health effects following repeated exposure to low levels of phenol in water include liver damage, diarrhea, mouth ulcers, dark urine and hemolytic anemia.

Even present in small concentration, phenol is considered toxic and can effects human health. Ingestion of phenol from contaminated drinking water source may cause tissue erosion, tissue degradation and paralysis of central nervous system. Phenol also can damage the kidney, liver and pancreas. The threshold value of phenol in water is 4000 $\mu\text{g/L}$. Therefore, it is considered necessary to remove the phenol from industrial effluents before discharging into the water stream. (Qadeer, and Rehan, 2002)

2.6.3 Phenol Adsorption by Activated Carbon

Michailof *et al* (2008) have found that most popular and extensively researched families of compounds that adsorbed onto activated carbon as they are highly toxic and present in many industrial wastewater

2.6.4 Effect of Initial pH on Phenol Removal Efficiencies

The carbon surface of activated carbon is protonated and positively charged at low pH values. As the pH increased, the sites of carbon become negatively charged. The isoelectric point of activated sludge usually between pH of 1 and 3 (Aksu and Yerner,1998,2001). It means that the surface of the sludge is positively charged at pH of 1 until 3 and becomes negatively charged at pH more than 3.

“Phenol is a weak acid with pK_a of 9.99~ 10 at temperature of 25°C and it ionized in the solution and become negative phenolate ion at pH of 10 and above. At pH of 3 until 6, phenol exists in neutral form as polar compound. Hence phenol is attracted to the negative sites of sludge and generating positively charged sites on its surface. Subsequently, the surface of sludge interacted with the phenolate ions, which increased in concentration when pH was increased. Hence the adsorption capacity also increased as the pH increased from pH of 2 until 6. At pH above 10, the solution contained phenolate ions while the surface of sludge was also negatively charged. This caused repulsion between negatively charged phenolate ions and the negative surface of sludge. The adsorption capacity hence decreased as the pH of the solution 10 and above (Thawornchaisit and Pakulanon, 2007).”

2.7 Effect of Impregnating Reagent and Reagent to Raw Material Ratio on Activated Carbon Surface Area

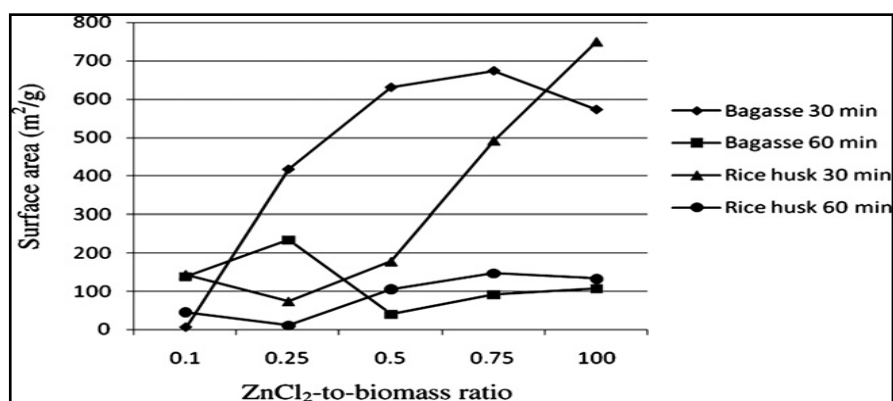


Figure 2.3: Effect of ZnCl₂-To-Biomass Ratio on the Development of the Surface Area of the Activated Carbon at 30 And 60 Min Residence Time.

Table 2.6: Results of the Single-Stage Production of Activated Carbon from Bagasse and Rice Husks Using H₃PO₄ and NaOH as Reagents

Reagent	% reagent	Activation temperature (°C)	Activation time (min)	S _{BET} (m ² /g)
<i>Bagasse</i>				
H ₃ PO ₄	50	600	30	24
H ₃ PO ₄	100	600	30	13
H ₃ PO ₄	75	700	30	173
NaOH	0-100	700	30	<10
<i>Rice husk</i>				
H ₃ PO ₄	50	600	30	9
H ₃ PO ₄	100	600	30	91
H ₃ PO ₄	75	700	30	56
NaOH	0-100	700	30	<10

From Fig. 2.3 and Table 2.6 show the results of the single-stage activation process of sugarcane bagasse and rice husk, respectively. The impregnating agent, the

reagent to raw material ratio, the activation temperature and time, and the surface area for each carbon obtained are shown. Chemical treatment with zinc chloride produced activated carbons with significant surface areas. The addition of ZnCl_2 and the thermal treatment of the mixture in the form of a thick paste enhanced the attack of the chemical on the matrix of the precursor, fragmenting cellulose, hemicelluloses and lignin. Fragmentation of these components led to reorganization of the precursor's matrix, swelling of the particles, dehydration and gradual development of a porous structure upon activation.

Fragmentation of celluloid compounds and lignin also restricted the formation of tars and volatiles, therefore blocking of pores and fissures was also restricted. It can be seen that the carbons with the highest surface area were obtained when the raw materials were impregnated with ZnCl_2 and then activated at $700\text{ }^\circ\text{C}$ for 30 min. Despite the relatively high quantity of ash in rice husk, carbons with high surface area were still produced. Fig. 2.3 shows the effect of the ZnCl_2 -to-raw material ratio during the impregnation step on the surface area. The optimum ratio of zinc chloride-to-bagasse is 0.75 while optimum ratio zinc chloride-to-rice husk is 1.00. The impregnation time of zinc chloride with bagasse at 60 min give the highest surface area. 30 min is the optimum time of impregnation of zinc chlorides with rice husk which gives highest surface area.

From Table 2.6, the activation agent uses in impregnation with bagasse and rice husk are NaOH and H_3PO_4 . Both bagasse and rice husk shows higher surface area when impregnation with H_3PO_4 . Impregnation with NaOH gives the BET surface area of bagasse and rice husk less than $10\text{ m}^2/\text{g}$. (Kalderis *et al*, 2008).

2.8 Effect of Pyrolysis Temperature on Activated Carbon

The volatile materials and carbon of elutrilithe decrease with increasing of temperature but moisture content increase from 500 to 800°C, but decrease in the range of 800 to 1000°C. The sample of elutrilithe has the highest water content but intermediate carbon content. This implies that the heating temperature has significant influences on the carbon development. In the range of 500 until 800°C the energy increases but decrease from temperature of 800 until 1000°C. This indicates that the porosity was developed by burning off part of the carbon to produce new pores and open closed pores at temperature between 500 until 800°C. Above the temperature of 800°C, the inner carbon was volatilized to widen the pores. Inner surface of carbon constructs the wall of each pore. Adsorbent with a small average pore size together with a large adsorption capacity can be obtained at temperature of 800°C. (Hu and Vansant, 1995)

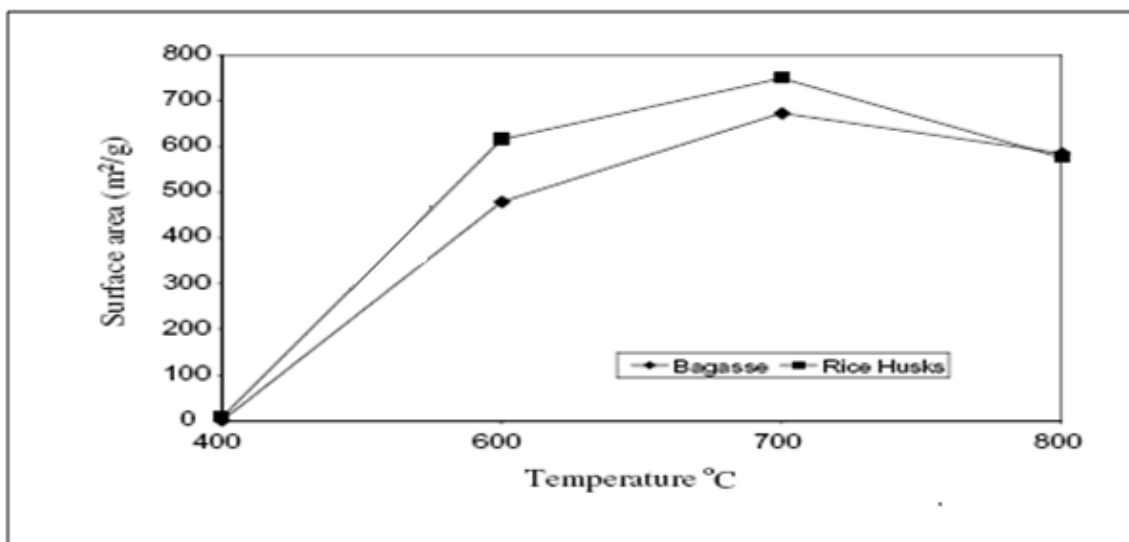


Figure 2.4: Effect of Activation Temperature on the Surface area of Acs Prepared With Optimum ZnCl₂ to Biomass Ratio

The effect of activation temperature is presented in Fig. 2.4. The activating agent was ZnCl₂ at ratios of 0.75:1 (w/w) (agent:precursor) and 1:1 for bagasse and rice husk, respectively. The results indicated that an increase in temperature resulted in an increase of the surface area because of the generation of new micro- and mesopores. The carbons with the highest surface area were obtained at 700 °C. A further increase in temperature at 800 °C yielded a decrease in the surface area. At this temperature, the surface area of bagasse AC decreased by more than 13%, whereas in the AC obtained from rice husk, the decrease in surface area was comparable. As temperature increased, ash formation also increased, resulting in a decrease of surface area. Pore widening and pore collapse could also occur and result in a decrease of surface area. (Kalderis *et al*,2008)

2.9 Effect of Carbonization Time

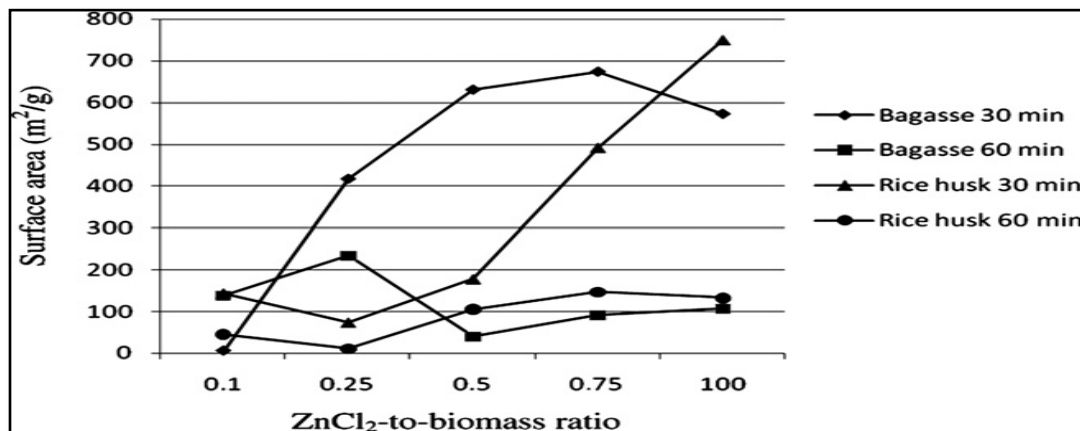


Figure 2.5: The Effect of Activation Time to the Surface Area of Bagasse and Rice Husk

As seen in Fig. 2.5, increasing the activation time from 30 to 60 min resulted in lower surface areas, especially at higher impregnating ratios. From these plots it can be concluded that at lower ZnCl₂-to-raw material ratios (60.5), ZnCl₂ equally competes

with the formation of tars. This explains the similar trend that was observed in both 30 and 60 min graphs at impregnation ratios below 0.5. However, at higher ratios (>0.5) and in longer carbonization periods the formation of more volatiles and tars predominated over the positive effect of $ZnCl_2$ and consequently the surface area was reduced. The positive effect of the $ZnCl_2$ impregnation was counteracted, to some degree, by the more lengthy emission of tars and volatiles. These observations are consistent with the results from Ahmadpour and Do (1996), where it was concluded that in the case of $ZnCl_2$ -impregnated precursor, time had a negative effect in the surface area and pore volume of activated carbons produced from coal.

CHAPTER 3

RESEARCH METHODOLOGY

3.1 Chemical and Apparatus Used

During the research, few chemical and apparatus are used in order to complete the research process. The chemical and apparatus used are as following:

List of glassware and apparatus used:

- i. Volumetric flask 25ml, 100ml and 1000ml
- ii. Beaker 50ml and 100ml
- iii. Measuring cylinder 10ml, 50 and 100ml
- iv. Magnetic stirrer
- v. Glass rod
- vi. Cone cylinder
- vii. Hot plate and stirrer
- viii. Whatman filter paper
- ix. Micropipette 10 to 1000 μ L
- x. pH meter
- xi. Hitachi U-1800 UV-Visible Spectrophotometer with cuvette
- xii. Orbital Shaker
- xiii. Memmet oven and furnace
- xiv. Blender

List of chemical used:

- i. Phenol extra pure
- ii. Zinc chloride
- iii. Sodium Carbonate
- iv. Hydrochloric Acid
- v. Sodium Hydroxide
- vi. Folin-Ciocalteu reagent
- vii. Distilled water

3.2 The Overall Methodology

The experimental procedures can be divided into three categories.

1. Preparation of activated carbon.
2. Preparation of phenol calibration curve.
3. Performance of activated carbon on phenol adsorption.

3.2.1 Preparation of activated carbon

The fresh decanter cake taken from palm oil milling factory is dried at 120°C for 24 hour to ensure the moisture content of decanter cake can be reduced. The dry decanter cake then crushed by using blender and sieve to the size of 125µm.

The chemical activation process was the next step to increase the surface area, porosity and reduce the ash content of final carbonized products. The activation agent such as zinc chloride will be used to mix with the material in mass ratios of 3:5 and 2:5. The slurry mixture will be stirred in magnetic stirrer at 85°C for about 8 hour. Then it will be dried and crushed again until it becomes powder before exposure to light and humidity for about a day.

The activated carbon then will be pyrolysed by using the furnace at temperature ranging from 300 until 900°C for 1 hour. After complete the pyrolysis process, the sample were let to cool before removed from the furnace and compress using mortar and pestle.

The activated carbon then stored in seal containers for further testing phenol adsorption capacity analysis and characterization of adsorbents analysis.

3.2.2 Preparation of Calibration Curve

Standard or calibration curve is a method used to determine the unknown concentration of phenol solution by comparing the unknown concentration with a standard sample of known concentration. The method can convert the value of absorption (ABS) from UV-Visible Spectrophotometer reading into the value of concentration. Hence final concentration of phenol solution can be known.

Standard curve was prepared by dilution of phenol solution into 2 until 50 mg/L. About 25 samples of phenol at different known concentration was added with 10ml distilled water, 3ml sodium carbonate solution and 1 ml folin reagent. All the samples then allowed until the maximum blue color appeared. The time estimation for maximum blue color is about 1 hour. Then measure the ABS by using UV-Visible Spectrophotometer. Then the graphs of absorption (ABS) are plotted against initial phenol concentration.

3.2.3 Analysis of Phenol

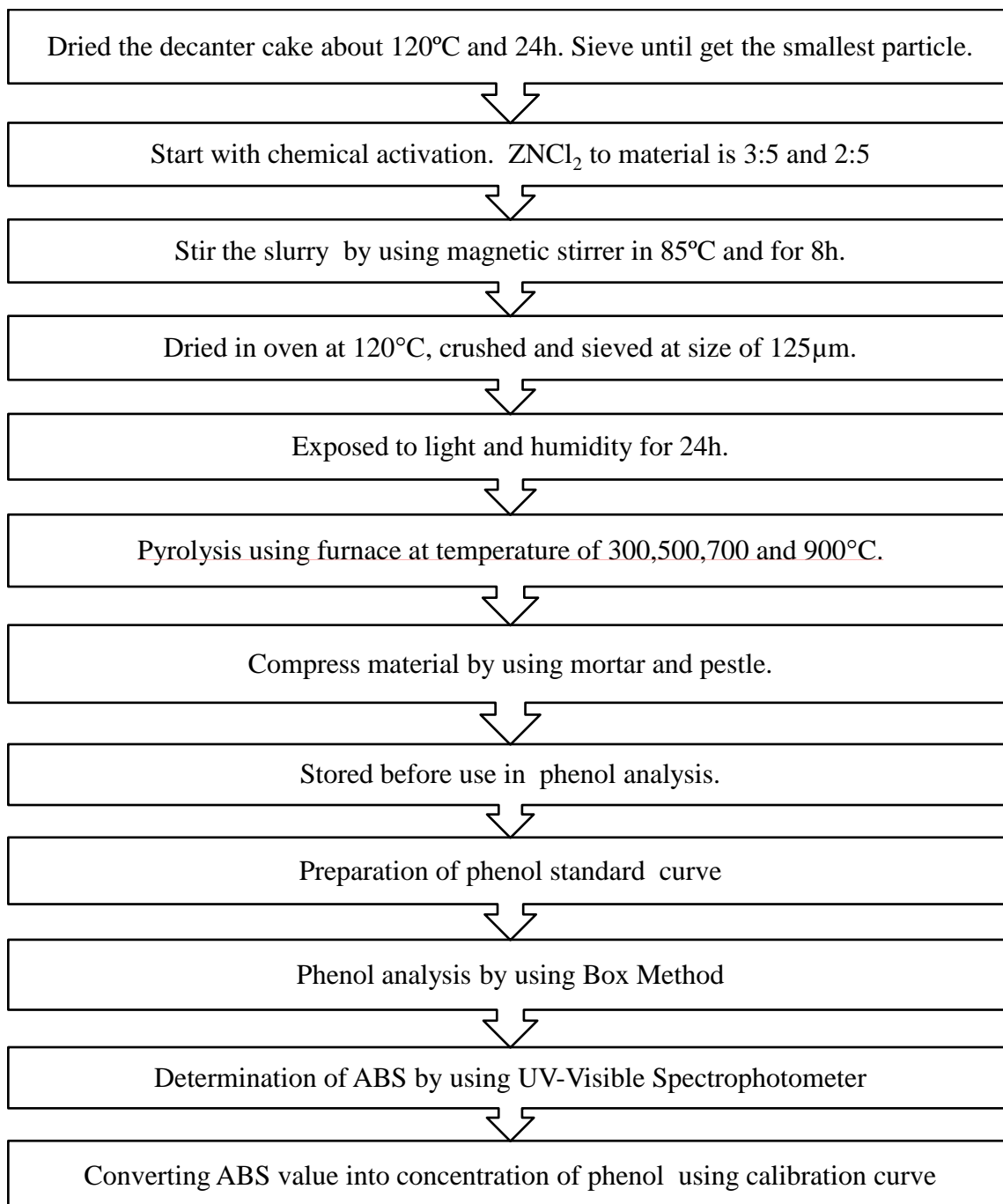
Analysis of phenol is done based on Box method (1983). About 0.1 g of adsorbed was added into the 50 ml conical flask with known concentration of phenol. Then the solution was shake for 3 hours by using the orbital shaker at 200 rpm. The solution then filtered with Whatman filter paper. The filtrate then was measured for 10 ml and placed into 25ml volumetric flask. The solution then was added with 10ml of distilled water, 3ml of sodium carbonate and 1ml of folin reagent. After 1 hour of reaction the maximum blue color is observed, placed into cuvette and measured with UV-Visible spectrophotometer at wavelength of 725nm.

The experiments were repeated for different sample of activated carbon such as at different temperature of pyrolysis and different ratio of decanter cakes to zinc chloride. For Langmuir isotherm, the experiment was repeated with different initial concentration of phenol and constant amount of activated carbon. The initial phenol concentration and equilibrium concentration were measured using the UV-Visible Spectrophotometer. The ABS data are then converted into concentration value by using the calibration curve of phenol.



Figure 3.1: UV-Visible Spectrophotometer

3.3 Summary of Research Procedure



3.4 Research Design Calculation

3.4.1 Percent Removal Equation

To calculate the percent removal of phenol, the equation as follow:

$$\%ABS = [(C_{Ph})_0 - (C_{Ph})_e] / (C_{Ph})_0 \times 100$$

Where:

$(C_{Ph})_0$ = Initial concentrations of phenol in mg/L.

$(C_{Ph})_e$ = Equilibrium concentrations of phenol in mg/L.

Reference*: Alhamed (2006)

3.4.2 Langmuir Isotherm Equation

To calculate Langmuir Isotherm data, the equation as follow:

$$\frac{ce}{q} = \frac{1}{q_{max}} ce + \frac{1}{kL \times q_{max}}$$

Where:

C_e = Equilibrium concentration of phenol in mg/L

q = Amount adsorbed at equilibrium in mg/g

q_{max} = Maximum adsorption capacity

k_L = Adsorption equilibrium constant in L/mg

The equation is then compared with the straight line graph of C_e/q against C_e to get the value of maximum adsorption capacity, q_{max} and adsorption equilibrium constant, k_L

Reference: *Ayar,A. *et al* (2008)

CHAPTER 4

RESULT AND DISCUSSION

4.1 Equilibrium Adsorption Data (Contact Time)

Table 4.1 below shows the absorption data from UV-Visible Spectrophotometer increased with time. As the time increased up to 180 minutes (3 hours), the absorption shows constant reading until 360 minutes (6 hours).

Table 4.1: Equilibrium Contact Time

Time (min)	Initial Absorption	Final Absorption	ABS
10	1.135	0.887	0.248
20	1.135	0.843	0.292
30	1.135	0.836	0.299
60	1.135	0.803	0.332
120	1.135	0.752	0.383
180	1.135	0.750	0.385
240	1.135	0.750	0.385
300	1.135	0.749	0.386
360	1.135	0.749	0.386

Figure 4.1 shows the ABS against contact time. From the figure, as the time is increased the value of ABS also increased. At certain time, the value of ABS starts constant and reaches equilibrium state.

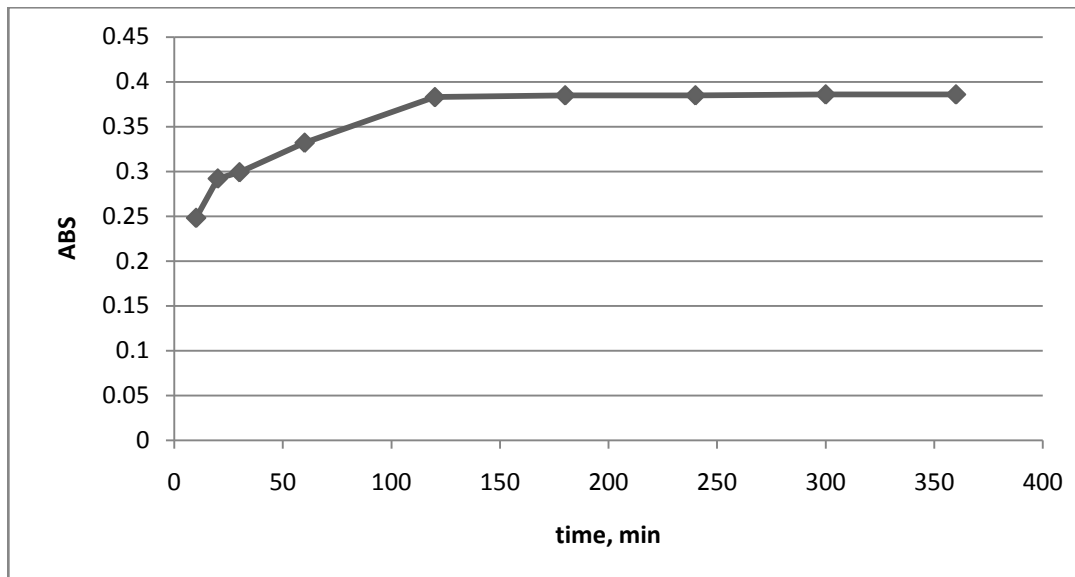


Figure 4.1: Graph of Equilibrium Contact Time

From the Table 4.1 and Figure 4.1 above, the graph shows that the contacting time between phenol solution and activated carbon reach the equilibrium state at 3 hour time of shaking. There was equilibrium state between adsorbate and adsorbent where the removal of phenol does not change much. As the final ABS increased with time, the percent removal is decreased because the remaining of phenol concentration is high at time less than 3 hours. As the time increase, the contacting time between phenol and activated carbon is longer and adsorption process is efficient. After the equilibrium contact time is reached, all the experiment was repeated with 3 hours of shaking between phenol solution and activated carbon.

4.2 Calibration Curve of Phenol

Table 4.2 shows the calibration data on phenol at different initial concentration ranging from 0 until 50 mg/L. The ABS values are increased as the concentration increased.

Table 4.2: Calibration Data of Phenol at Initial Concentration 0 until 50 mg/L

Concentration, mg/L	ABS
0	0.000
10	0.411
20	0.813
30	1.109
40	1.676
50	2.092

Figure 4.2 shows the straight line graph of ABS against initial concentration of phenol ranging from 0 until 50 mg/L.

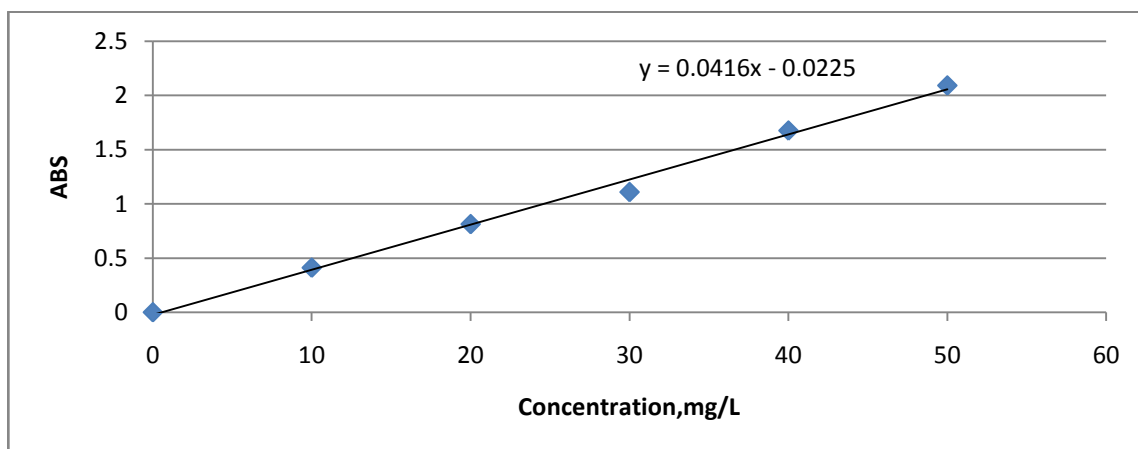


Figure 4.2: Calibration Curve of Phenol at Initial Concentration 0 until 50 mg/L

4.3 Effect of Temperature on Activated Carbon

4.3.1 Temperature Effect at Ratio of 2:5

The table 4.3 shows the percent removal of phenol from based initial concentration of 25 mg/L at different temperature of pyrolysis process which are 300, 500, 700 and 900°C. The correction initial concentration reading from UV-Visible spectrophotometer is 30.77 mg/L. The ratio of impregnation is 2:5.

Table 4.3: Temperature Effect at Ratio of 2:5

Temperature (°c)	Initial Concentration, mg/L	Initial Reading Concentration, mg/L	Final Concentration, mg/L	Percent Removal (%)
300	25	30.77	26.05	15.34
500	25	30.77	14.82	51.84
700	25	30.77	7.54	75.50
900	25	30.77	2.44	92.07

Figure 4.3(a) shows the graph of percent removal of phenol against the pyrolysis temperature at ratio of impregnation 3:5. The percent removal of phenol increased as the temperature of pyrolysis increased. While figure 4.3(b) shows the color comparison of phenol solution at different temperature of pyrolysis. As the intensity blue color of the solution increase, the phenol present in the solution also increase.

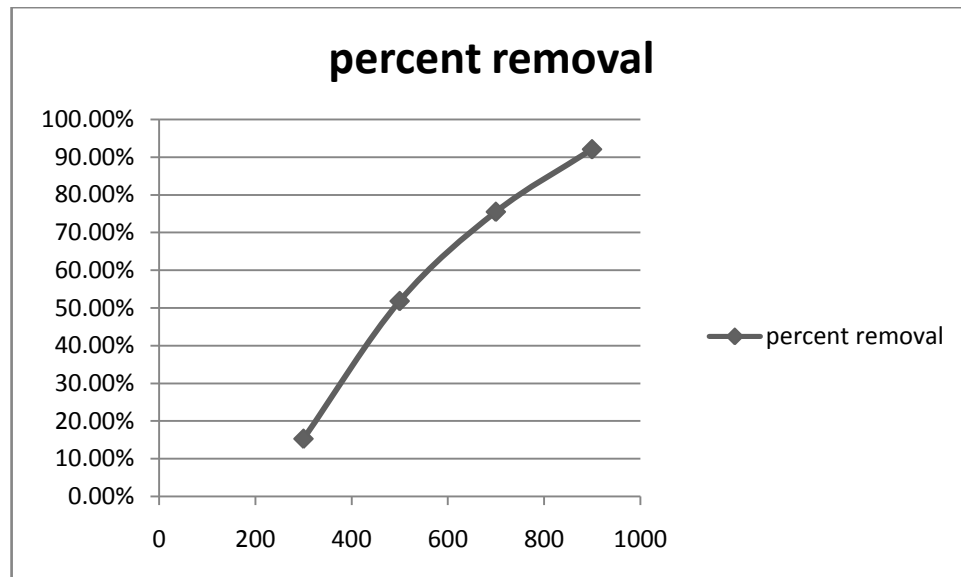


Figure 4.3(a): Graph of Temperature Effect at Ratio of 2:5

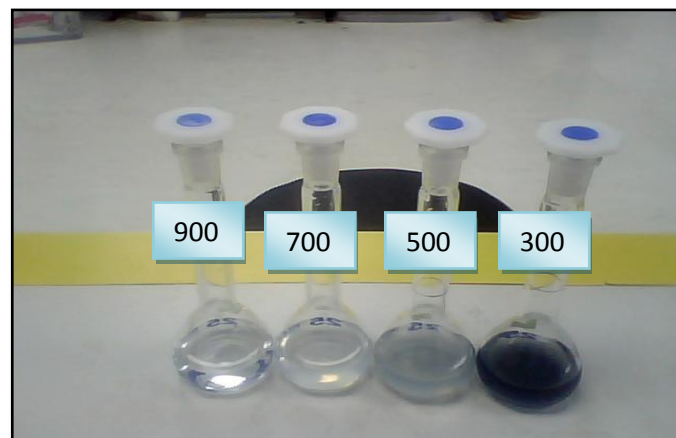


Figure 4.3 (b): Final Concentration of Phenol at Different Pyrolysis Temperature and Ratio of Impregnation 2:5

4.3.2 Temperature Effect at Ratio Impregnation of 3:5

The table 4.4 below shows the percent removal of phenol from based initial concentration of 25 mg/L at different temperature of pyrolysis process which are 300, 500, 700 and 900°C. The correction initial concentration reading from UV-Visible spectrophotometer is 30.77 mg/L. The ratio of zinc chloride to decanter cake (impregnation) is 3:5.

Table 4.4: Table of Temperature Effect at Ratio Impregnation of 3:5

Temperature (°c)	Initial concentration, mg/L	initial reading concentration, mg/L	final concentration, mg/L	Percent removal
300	25	30.77	24.22	21.29
500	25	30.77	9.12	70.36
700	25	30.77	2.15	93.01
900	25	30.77	1.41	95.42

Figure 4.4(a) shows the graph of percent removal of phenol against the pyrolysis temperature at ratio of impregnation 3:5. The percent removal of phenol increased as the temperature of pyrolysis increased. While figure 4.4(b) shows the color comparison of phenol solution at different temperature of pyrolysis. As the intensity blue color of the solution increase, the phenol present in the solution also increase.

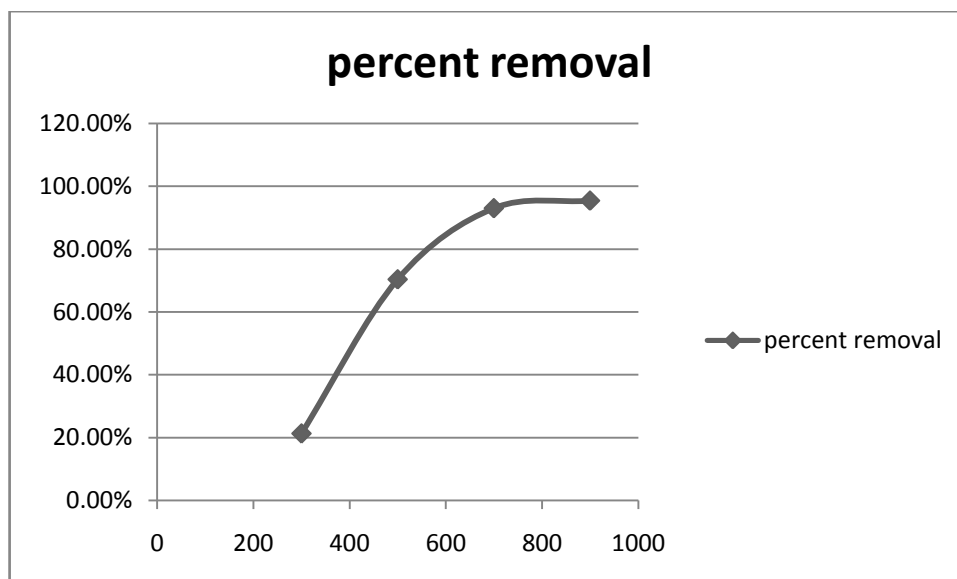


Figure 4.4(a): Graph of Temperature Effect at Ratio Impregnation of 3:5

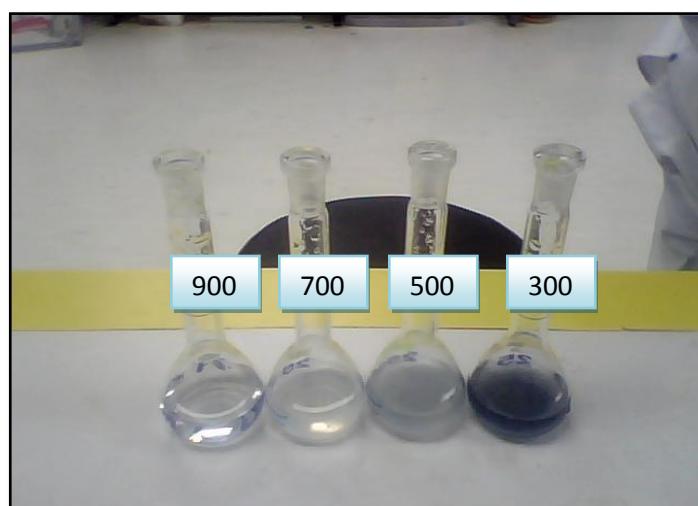


Figure 4.4(b): Final Concentration of Phenol at Different Pyrolysis Temperature and Ratio of Impregnation 3:5

Table 4.5 below shows the comparison data at both ratio of zinc chloride to decanter cake (impregnation) 2:5 and 3:5. Ratio of 3:5 shows the higher percentage of phenol removal compared with ratio of 2:5.

Table 4.5: Table of Temperature Effect at Both Ratio of Impregnation

Temperature °C	300	500	700	900
Percent removal at 2:5	15.34	51.84	75.5	92.07
Percent removal at 3:5	21.29	70.36	93.01	95.42

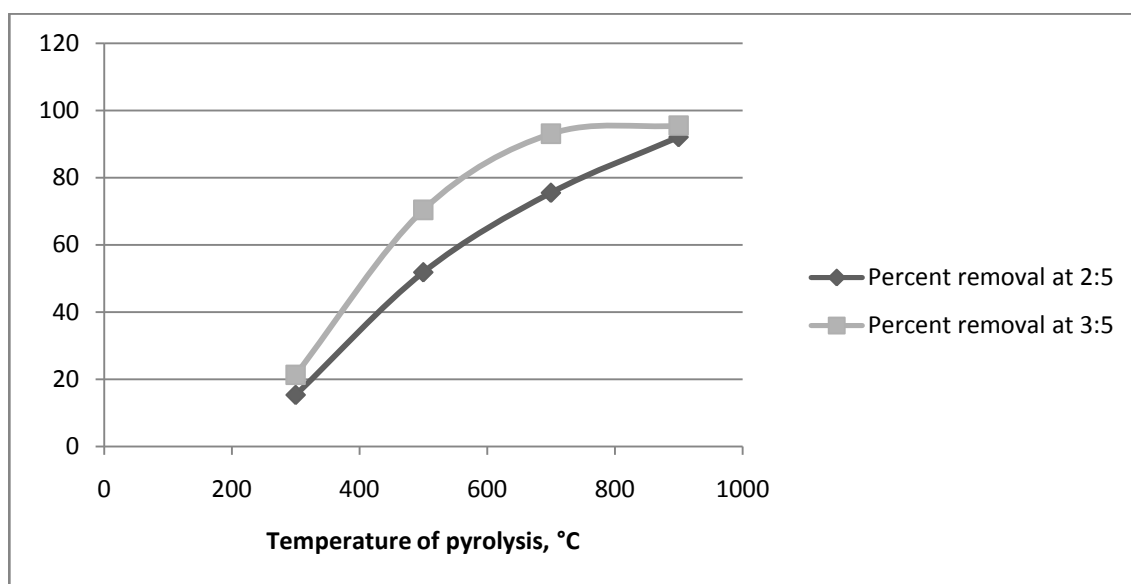


Figure 4.5: Comparison Graph of Both Ratio of Impregnation

From Figure 4.5, as the temperature of pyrolysis process increased, the percent removal of phenol also increased. At both ratios, temperature of 700 and 900°C is the optimal temperature of phenol removal. The removal shows up to 90 percent of removal. Temperature of pyrolysis play important role in producing activated carbon with larger

pores and high surface area. Because at high temperature, the material is burn off and the high temperatures also open the new pores of the carbon surface. The ratios of impregnation also affect the carbonization process. Between the ratio of 2:5 and 3:5, the ratio of 3:5 is efficient to produce activated carbon with greater removal. Because the less amount of activated agent, there are insufficient amount of zinc chloride to react with the decanter cakes in order to enlarge the surface area and pores.

4.4 Effect of pH on Activated Carbon

Table 4.6 below shows the percent removal of phenol at initial concentration of 25 mg/L and ratio of zinc chloride to decanter cake of 2:5 at different initial pH of phenol solution.

Table 4.6: pH Data on Phenol Removal

pH	Initial concentration mg/L	Final concentration mg/L	Percent removal
2	25	1.8162	92.74
4	25	1.5537	93.79
6	25	0.8874	96.45
8	25	1.3163	94.73
10	25	1.5776	93.69
12	25	1.5776	93.69

Figure 4.6 shows the graph of percent phenol removal against different initial pH of phenol solution. The pH ranging from 2 until 12 and the percentage of removal is more than 92%.

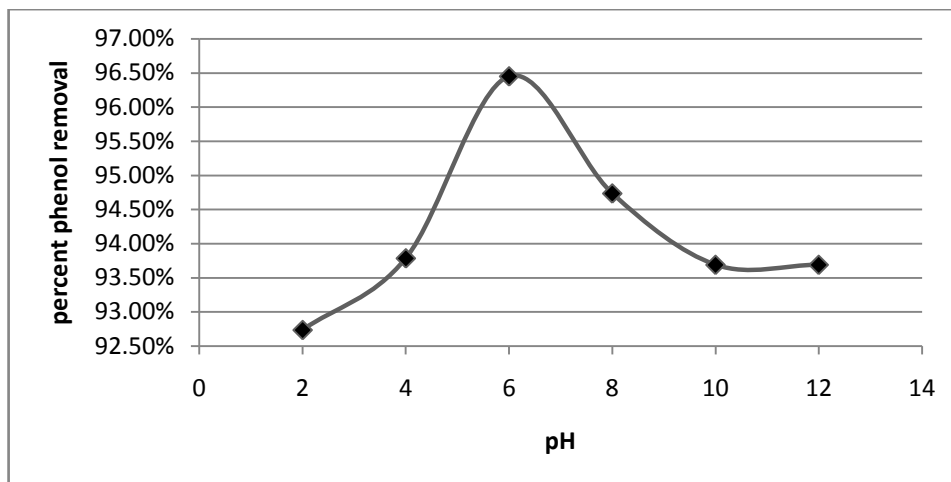


Figure 4.6: Graph of Percent removal against pH

From the Table 4.6 and Figure 4.6 above, the percent removal are increased as the initial pH phenol solution increased from 2 until 6. At pH of 6 the percent removal of phenol is the highest where the removal is about 96.45%. The result shows that molecule attraction happen between phenolate ions with the carbon surface. At low pH values, the carbon surface is positively charged, hence the nonpolar phenolate ion will attract to the positive charge of carbon surface. Hence the carbon will adsorb the phenol from the solution. At pH of 8 and below, the percent removal of phenol by the activated carbon is decreased. It shows that there is repulsion between the phenolate ions with the carbon surface. The reason why repulsion happens is that the carbon surface becomes negatively charged as the pH increased and the phenolate ions also becomes negatively charged. This phenomenon happens due to the nature of the phenol as a weak acid with Pk_a nearly 10. Hence it becomes negative at pH of 10 and above. When both of phenolate ions and carbon surface is negatively charged, the repulsion happens and phenol ions not attract to the carbon surface. As a result the removal of phenol decreased. The experiment shows an error where the removal of phenol decreased from pH of 8, which supposed to decrease at pH of 10. The result shows that phenolate ions become negative at pH of 8. This maybe because of some error while preparing the sample or the reading taken from the UV-Visible Spectrophotometer. The device is very sensitive as any changing in the solution; hence it will give the different readings.

4.5 Langmuir Isotherm

4.5.1 Langmuir Isotherm at Temperature 900°C and Ratio Impregnation of 2:5

Six samples of phenol solution at different concentration have been prepared. The sample of activated carbon was prepared at pyrolysis temperature of 900°C and ratio zinc chloride to decanter cake (impregnation) of 2:5. Initial and final ABS readings are taken from UV-Visible Spectrophotometer and converted into concentration values. The equilibrium concentrations are tabulated in table 4.7 below.

Table 4.7: Data of Langmuir Isotherm at Temperature 900°C and Ratio Impregnation of 2:5

Concentration, mg/L	Initial concentration C_o , mg/L	Equilibrium concentration C_e , mg/L	q $\frac{(C_o-C_e)V}{W}$ mg/g	ce/q g/L
25	22.85	0.81	4.408	0.184
50	50.83	1.12	9.942	0.113
100	109.50	4.58	20.9984	0.218
150	172.00	11.42	32.116	0.356
200	214.78	22.24	38.508	0.576
250	241.42	32.82	45.72	0.718

From the table 4.7, the straight line graph of C_e/q against C_e is plotted in figure 4.7 (a). Figure 4.7 (b) shows the final concentration of phenol at different initial concentration of phenol. As the blue color increase, the phenol concentration in the solution also increases. The sample of activated carbon is at temperature of pyrolysis 900°C and ratio of impregnation of 2:5.

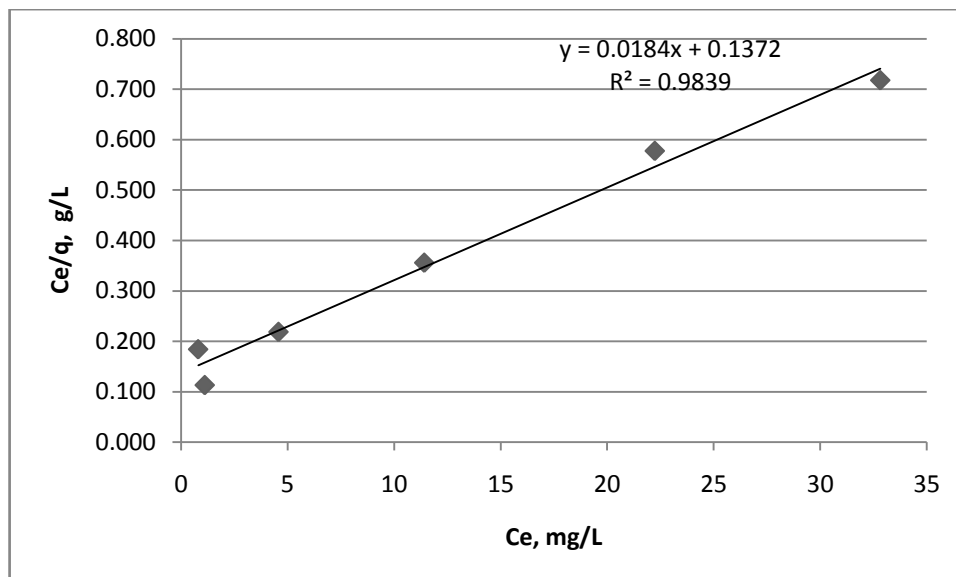


Figure 4.7 (a): Graph of Langmuir Isotherm at Temperature of pyrolysis 900°C and Ratio Impregnation of 2:5

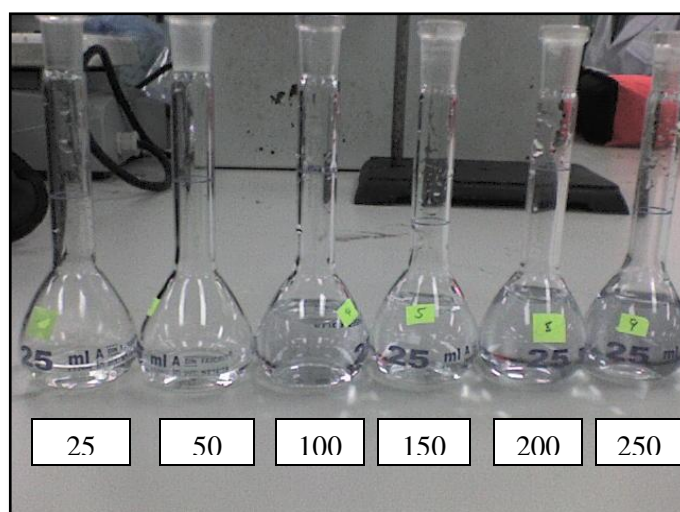


Figure 4.7 (b): Final Concentration of Phenol at Different Initial Concentration in mg/L Respectively

4.5.2 Langmuir Isotherm at Temperature 900°C and Ratio Impregnation of 3:5

Six samples of phenol solution at different concentration have been prepared. The sample of activated carbon was prepared at pyrolysis temperature of 900°C and ratio zinc chloride to decanter cake (impregnation) of 3:5. Initial and final ABS readings are taken from UV-Visible Spectrophotometer and converted into concentration values. The equilibrium concentrations are tabulated in table 4.8 below.

Table 4.8: Data of Langmuir Isotherm at Temperature 900°C and Ratio Impregnation of 3:5

Concentration, mg /L	Initial concentration C_o , mg/L	Equilibrium concentration C_e , mg/L	q mg/g	Ce/q g/L
25	22.85	7.42	3.086	2.404
50	50.83	19.72	6.222	3.169
100	109.50	49.15	12.07	4.072
150	172.00	96.75	15.05	6.429
200	214.78	179.45	7.066	25.396
250	241.42	250.12	2.26	110.673

From the table 4.8, the straight line graph of Ce/q against Ce is plotted in figure 4.8(a). Figure 4.8(b) shows the final concentration of phenol at different initial concentration of phenol. As the blue color increase, the phenol concentration in the solution also increases. At concentration of 150, 200 and 250 mg/L, final phenol solution had been diluted because the UV-Visible Spectrophotometer cannot measure the solution at higher of concentration. The sample of activated carbon is at temperature of pyrolysis 900°C and ratio of impregnation of 3:5.

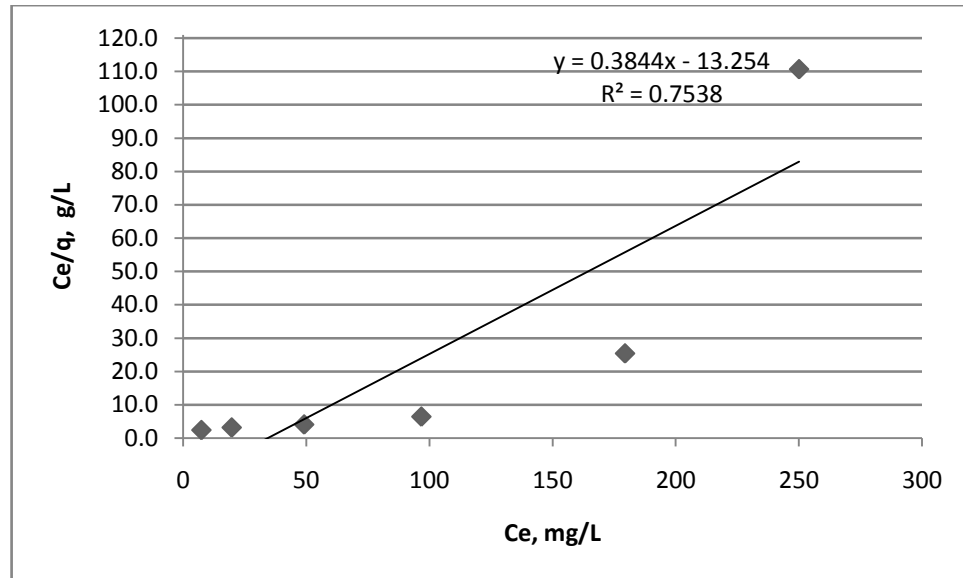


Figure 4.8(a): Graph of Langmuir Isotherm at Temperature 900°C and Ratio Impregnation of 3:5

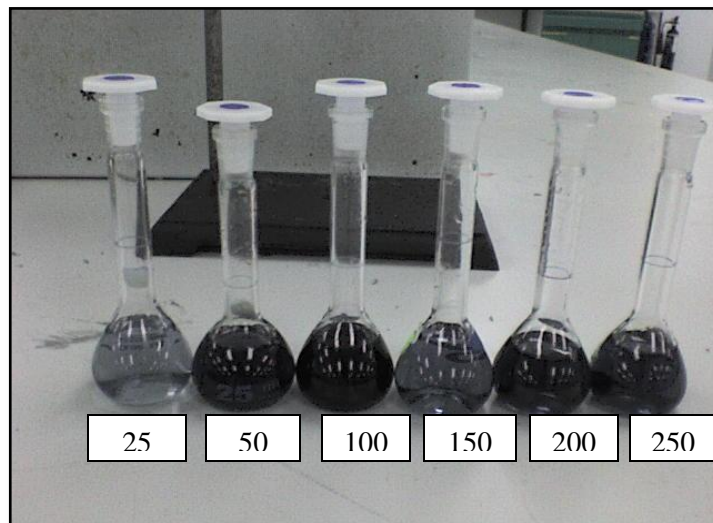


Figure 4.8 (b): Final Concentration of Phenol at Different Initial Concentration in mg/L Respectively

4.5.3 Langmuir Isotherm at Temperature 700°C and Ratio Impregnation of 2:5

Six samples of phenol solution at different concentration have been prepared. The sample of activated carbon was prepared at pyrolysis temperature of 700°C and ratio zinc chloride to decanter cake (impregnation) of 2:5. Initial and final ABS readings are taken and converted into concentration values. The equilibrium concentrations are tabulated in table 4.9 below.

Table 4.9: Data of Langmuir Isotherm at Temperature 700°C and Ratio Impregnation of 2:5

Concentration mg/L	Initial concentration C_0 , mg/L	Equilibrium concentration C_e , mg/L	q mg/g	C_e/q g/L
25	22.85	2.08	4.154	0.501
50	50.83	8.500	8.466	1.004
100	109.50	31.13	15.674	1.986
150	172.00	66.47	21.106	3.149
200	214.78	101.08	22.740	4.445
250	241.42	140.26	24.232	5.788

From the table 4.9, the straight line graph of C_e/q against C_e is plotted in figure 4.9(a). Figure 4.9(b) shows the final concentration of phenol at different initial concentration of phenol. As the blue color increase, the phenol concentration in the solution also increases. At concentration of 100, 150, 200 and 250 mg/L, final phenol solution had been diluted because the UV-Visible Spectrophotometer cannot measure the solution at higher of concentration. The sample of activated carbon is at temperature of pyrolysis 700°C and ratio of impregnation of 2:5.

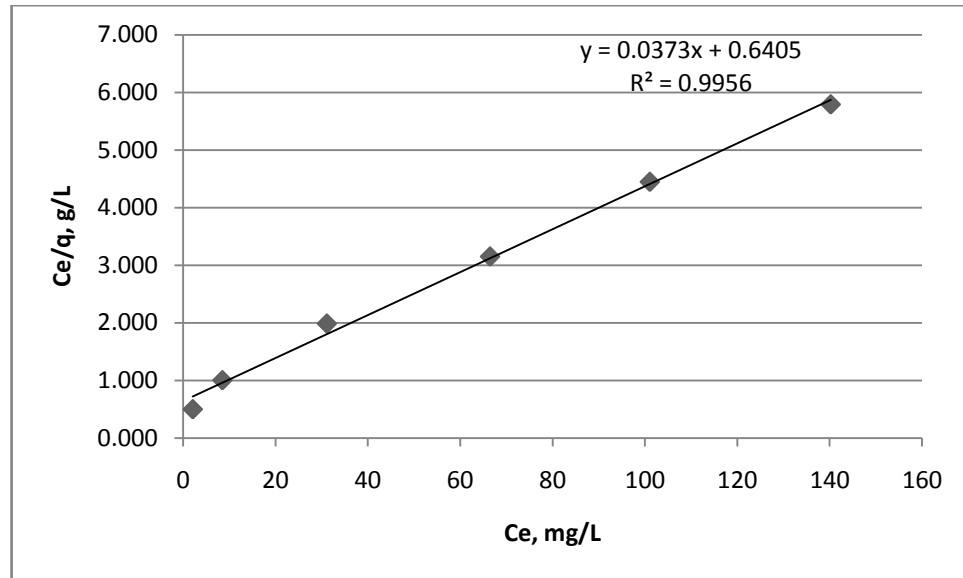


Figure 4.9(a): Graph of Langmuir Isotherm at Temperature 700°C and Ratio Impregnation of 2:5

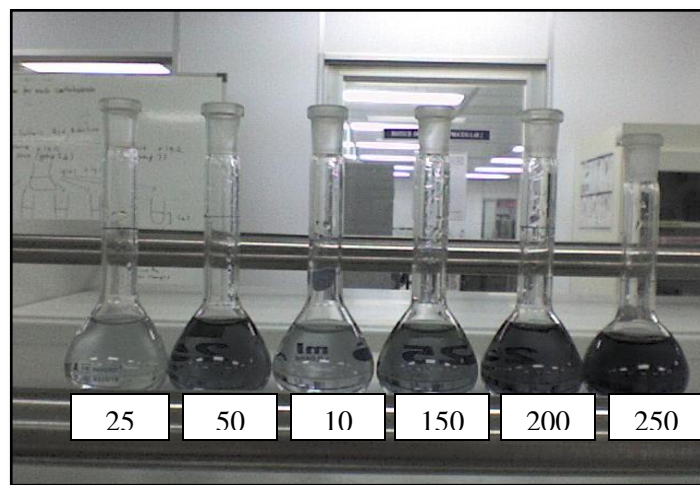


Figure 4.9 (b): Final Concentration of Phenol at Different Initial Concentration in mg/L Respectively

4.5.4 Langmuir Isotherm at Temperature 700°C and Ratio Impregnation of 3:5

Six samples of phenol solution at different concentration have been prepared. The sample of activated carbon was prepared at pyrolysis temperature of 700°C and ratio zinc chloride to decanter cake (impregnation) of 3:5. Initial and final ABS readings are taken and converted into concentration values. The equilibrium concentrations are tabulated in table 4.10 below.

Table 4.10: Data of Langmuir Isotherm at Temperature 700°C and Ratio Impregnation of 3:5

Concentration mg/L	Initial Concentration, C_0 mg/L	Equilibrium concentration, C_e mg/L	q mg/g	ce/q g/L
25	22.85	2.61	4.048	0.645
50	50.83	7.01	8.764	0.800
100	109.50	20.18	17.864	1.130
150	172.00	57.81	22.838	2.531
200	214.78	76.32	27.692	2.756
250	241.42	129.45	26.394	4.905

From Table 4.10, the straight line graph of C_e/q against C_e is plotted in figure 4.10(a). Figure 4.10(b) shows the final concentration of phenol at different initial concentration of phenol. As the blue color increase, the phenol concentration in the solution also increases. At concentration of 150, 200 and 250 mg/L, final phenol solution had been diluted because the UV-Visible Spectrophotometer cannot measure the solution at higher of concentration. The sample of activated carbon is at temperature of pyrolysis 700°C and ratio of impregnation of 3:5.

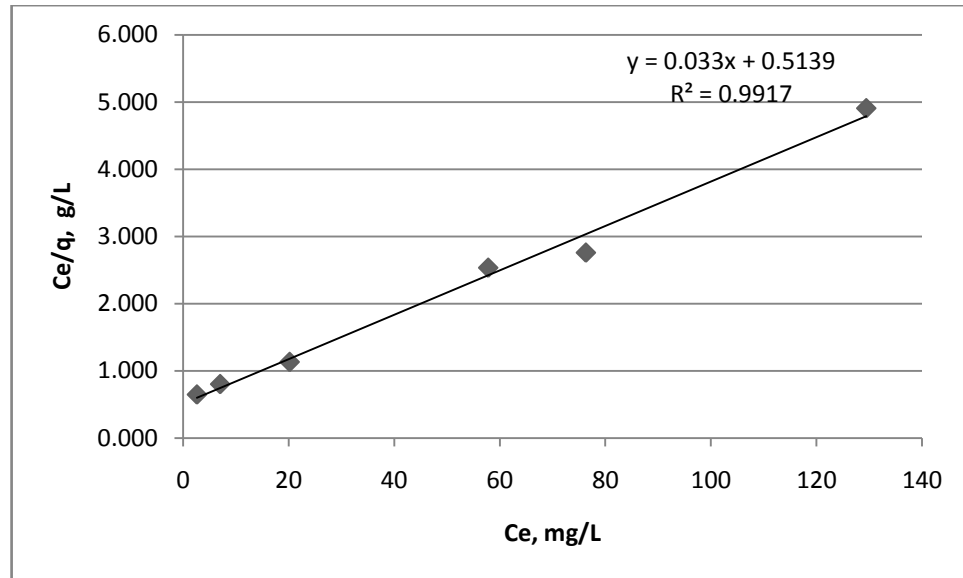


Figure 4.10(a): Graph of Langmuir Isotherm at Temperature 700°C and Ratio Impregnation of 3:5

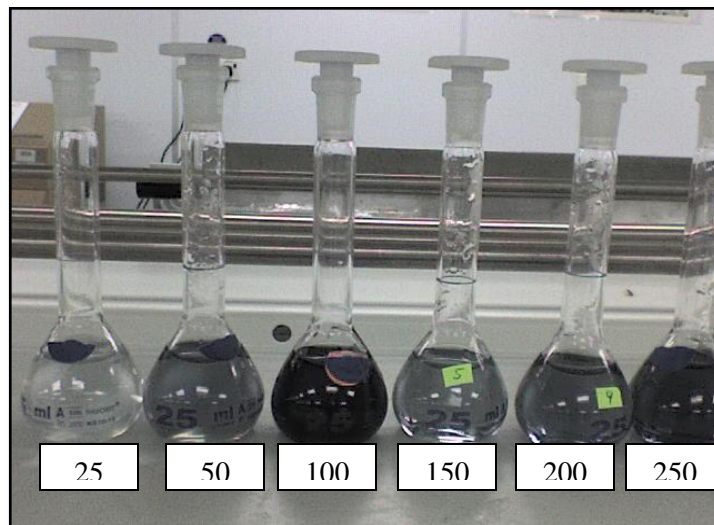


Figure 4.10(b): Final Concentration of Phenol at Different Initial Concentration in mg/L Respectively

4.5.5 Summary of Langmuir Isotherm

Tables 4.11 below summarize the Langmuir Isotherm data for sample of activated carbon at different temperature of pyrolysis and ratio of impregnation.

Table 4.11: Table summary of Langmuir Isotherm

Sample	1	2	3	4
Ratio impregnation	2:5	3:5	2:5	3:5
Temperature pyrolysis	900	900	700	700
q_{\max}	54.35	2.60	26.81	30.30
k_L	0.134	-0.029	0.058	0.064
R^2	0.984	0.754	0.996	0.991

The result on Langmuir isotherm shows the adsorption capacity of phenol by the activated carbon. About four samples of two different temperatures and two different ratios are studied. The optimal temperature of pyrolysis was at 700 and 900°C. The ratios are 2:5 and 3:5 respectively. Each of the samples contains about 0.1g adsorbent in 20 ml phenol solution. Initial phenol solution ranging from 25mg/L until 250 mg/L. The data of UV-Visible Spectrophotometer may vary with the initial prepared phenol concentration. Hence to avoid error in Langmuir isotherm, the initial readings of phenol are taken.

From the result obtained temperature of 900°C. The maximum adsorption capacity is 54.35. The result at first sample, i.e. at temperature of 900 °C and ratio of 3:5 shows the opposite result. The maximum capacity of the sample must be higher than the sample 2, 3 and 4. Maybe the activated carbons are no longer effective, as it might be exposed to air because the lack of storing the carbon. The containers seem to be not tight and as a result the activated carbon no longer effective in adsorb the phenol. By comparing with the ratio of impregnation, at temperature of 700°C, the ratio of 3:5 show

the better adsorption capacity that is 30.30 compared with the capacity at ratio of 2:5 that is 26.81. The higher temperature of pyrolysis give the better performance to the activated carbon produced from decanter cakes. Hence the optimal temperature was at 900°C.

CHAPTER 5

CONCLUSION

5.1 Conclusion

The result obtained from the experiment shows that the optimum contacting time for phenol and activated carbon produce from decanter cake is at 3 hours. With the data, all the experiment is done based on the constant shaking time.

The suitable impregnation ratio between activating agent, $ZnCl_2$ to decanter cakes is 3:5 and the optimum temperature of pyrolysis is occurred at $900^\circ C$. Even the temperature of $700^\circ C$ also gives the high percentage of phenol removal but at temperature of $900^\circ C$ it is slightly increase.

For the pH effect, the experiment shows that the optimum of phenol removal occurred at pH of 6 and it start to decrease after pH of 6. The previous research shows that the optimum pH is happen at pH of 6 until 10. "The adsorption capacity increased from pH of 2 until 6 and decreased at pH 10 and above. (Thawornchaisit and Pakulanon, 2007)."

Langmuir Isotherm are plotted to determine the capacity of adsorption of decanter cake as activated carbon. From the result, the optimum temperature which

adsorption capacity is higher is at temperature of pyrolysis at 900°C. At temperature of 700°C, the optimum ratio of zinc chloride to decanter cake is at 3:5.

5.2 Recommendation and Future Work

There will be a few suggestions for the improvement of this research in the future. There a few suggestions in order to improve the phenol removal capacities:

1. The pyrolysis process can be done in the quartz reactor with the flow of nitrogen gas instead of using the furnace with absence of oxygen gas only. This may reduce the temperature of pyrolysis for the optimum removal of phenol.
2. The activated carbon must be placed in the desiccators for a certain period until it is used for analysis because the air would reduce the surface area of the activated carbon as the particles will combine together into the bigger particles of sizes.
3. For the future work, the activated carbon produced from this experiment should be compared with the synthetic activated carbon such as coal and activated carbon which undergo physical treatment which means without adding chemical activation.
4. For industrial application, the sufficient amount of activated carbon is suggested to be placed in packed bed types of tank to adsorb the organic compound at certain contacting time before it were discharge into the water stream. The tank should be design until meet the specification of organic limitation in water stream.
5. Besides phenol, other organic waste also can be investigated such as acrylic acid and acetic acid.

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APPENDIX A
RESEARCH CALCULATION

Preparation of Phenol Stock Solution

About six stock solutions with different concentration of phenol are prepared. The preparation is by dilution certain amount of phenol crystal in 1 L volumetric flask. The calculations are as following:

For phenol concentration of 100mg/L, 150mg/L, 200mg/L, 250 mg/L

0.1g phenol crystals in 1L distilled water

0.15g phenol crystal in 1L distilled water

0.20g phenol crystal in 1L distilled water

0.25g phenol crystal in 1L distilled water

For phenol concentration of 25 and 50 mg/L

$$M_1V_1 = M_2V_2$$

$$(100\text{mg/L})V_1 = (25\text{mg/L})(100\text{ml})$$

$$V_1 = 25\text{ml stock solution} + 75\text{ml distilled water}$$

$$M_1V_1 = M_2V_2$$

$$(100\text{mg/L})V_1 = (50\text{mg/L})(100\text{ml})$$

$$V_1 = 50\text{ml stock solution} + 50\text{ml distilled water}$$

Langmuir Adsorption Isotherm

Before graph of Langmuir isotherm are plotting, value for q must be calculated as following:

$$q = \frac{(C_0 - C_e) V}{W} \dots \dots \dots (1)$$

Where C_0 is initial phenol concentration, C_e is the equilibrium concentration; V is volume of phenol solution in L and W is the weight of the activated carbon in g.

$$V = 0.02 \text{ L}$$

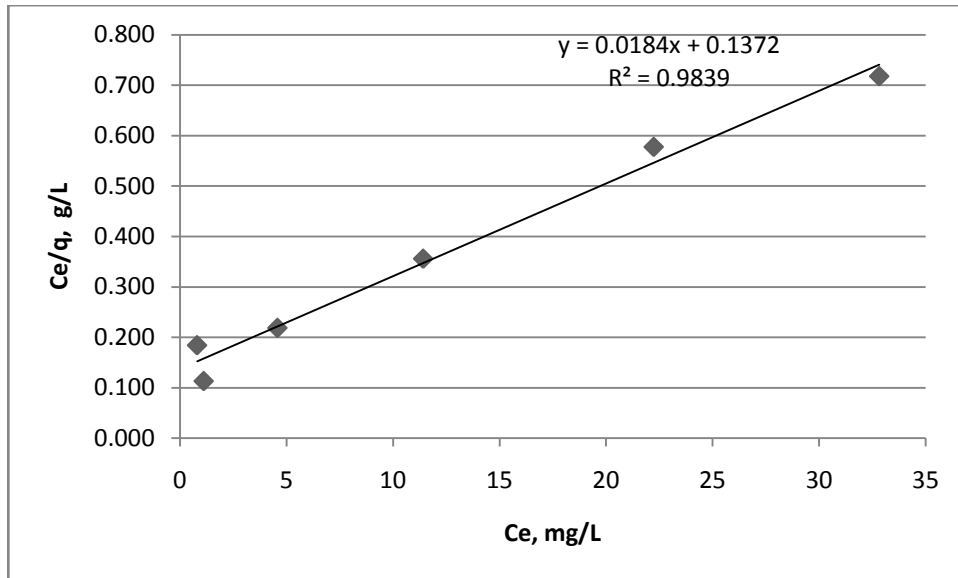
$$W = 0.1 \text{ g}$$

Condition 1

Temperature of pyrolysis = 900°C

Ratio of impregnation = 2:5

Concentration, mg/L	Initial concentration C_0 , mg/L	Equilibrium concentration C_e , mg/L	q $\frac{(C_0 - C_e)V}{W}$ mg/g	ce/q g/L
25	22.85	0.81	4.408	0.184
50	50.83	1.12	9.942	0.113
100	109.50	4.58	20.9984	0.218
150	172.00	11.42	32.116	0.356
200	214.78	22.24	38.508	0.576
250	241.42	32.82	45.72	0.718



From the graph, $y=mx + c$

From Langmuir equation, $\frac{ce}{q} = \frac{1}{q_{max}} ce + \frac{1}{KL \times q_{max}}$

Hence equation for graph, $y= 0.0184x + 0.1372$

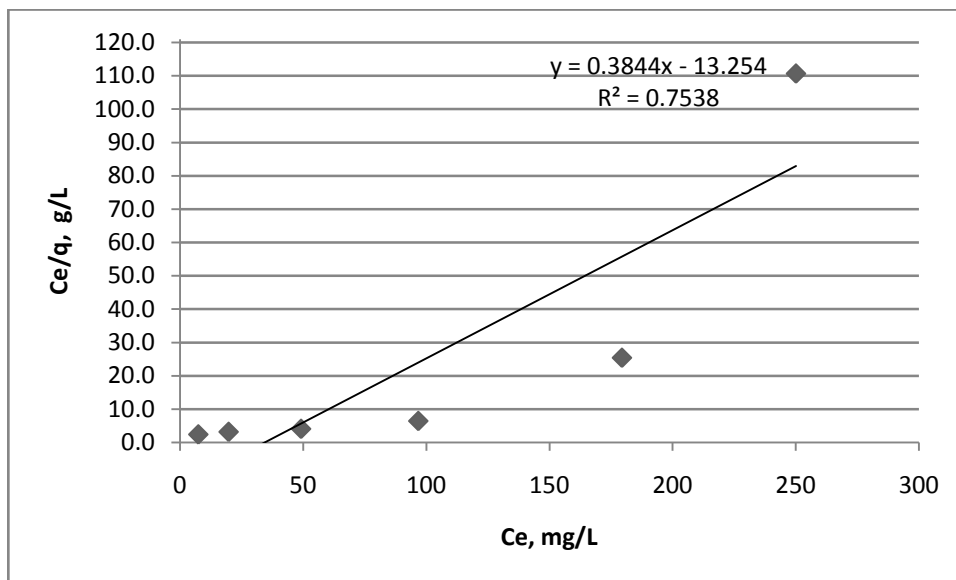
$q_{max}= 54.35$ and $k_L= 0.134$

Condition 2

Temperature of pyrolysis = 900°C

Ratio of impregnation = 3:5

Concentration mg /L	Initial concentration Co, mg/L	Equilibrium concentration Ce, mg/L	q mg/g	Ce/q g/L
25	22.85	7.42	3.086	2.404
50	50.83	19.72	6.222	3.169
100	109.50	49.15	12.07	4.072
150	172.00	96.75	15.05	6.429
200	214.78	179.45	7.066	25.396
250	241.42	250.12	2.26	110.673



From the graph, $y = mx + c$

From Langmuir equation, $\frac{ce}{q} = \frac{1}{q_{max}} ce + \frac{1}{KL \times q_{max}}$

Hence equation for graph, $y = 0.3844x - 13.254$

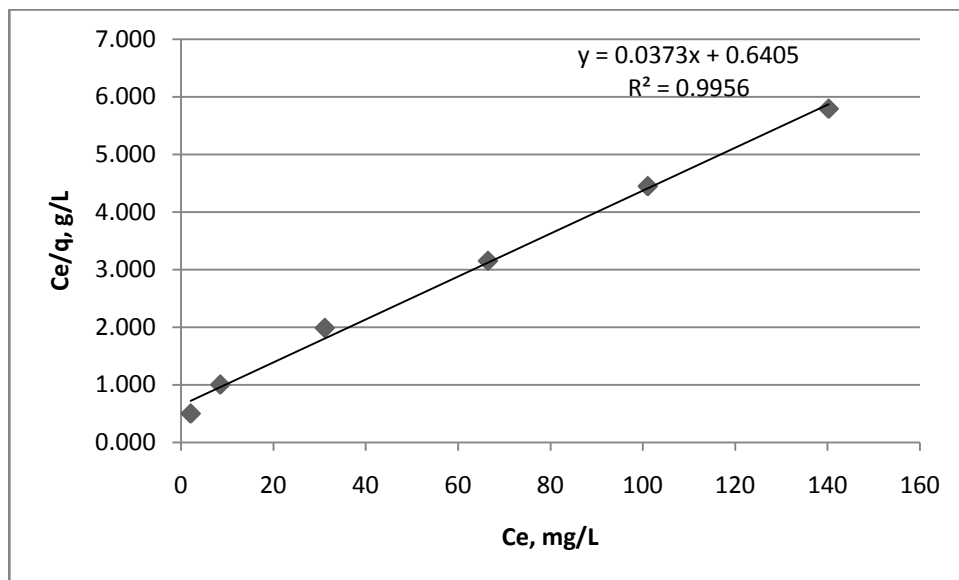
$q_{max} = 2.60$ and $K_L = -0.029$

Condition 3

Temperature of pyrolysis = 700°C

Ratio of impregnation = 2:5

Concentration mg/L	Initial concentration Co, mg/L	Equilibrium concentration Ce, mg/L	q mg/g	Ce/q g/L
25	22.85	2.08	4.154	0.501
50	50.83	8.500	8.466	1.004
100	109.50	31.13	15.674	1.986
150	172.00	66.47	21.106	3.149
200	214.78	101.08	22.740	4.445
250	241.42	140.26	24.232	5.788



From the graph, $y = mx + c$

From Langmuir equation, $\frac{ce}{q} = \frac{1}{q_{max}} ce + \frac{1}{KL \times q_{max}}$

Hence equation for graph, $y = 0.0373x + 0.6405$

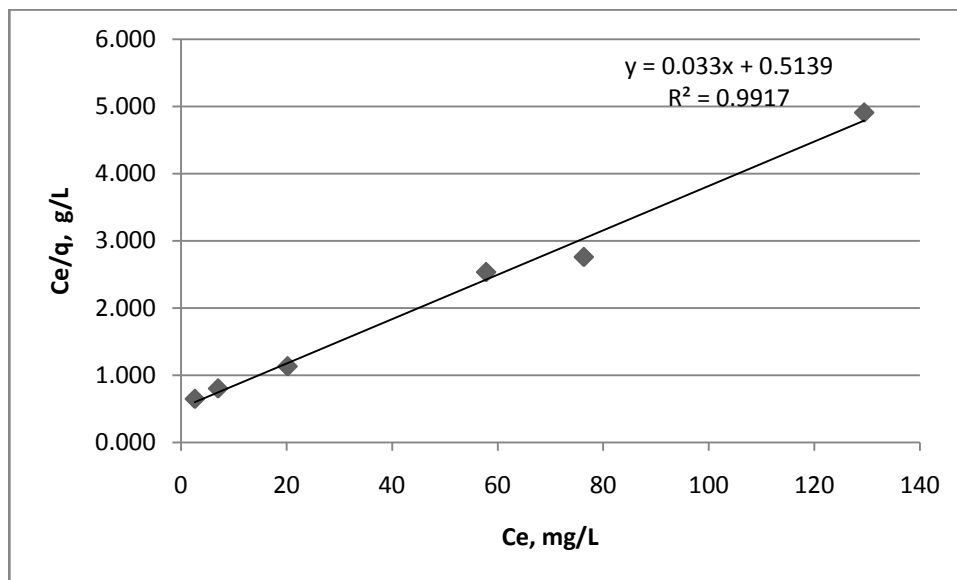
$q_{max} = 26.81$ and $K_L = 0.058$

Condition 4

Temperature of pyrolysis = 700°C

Ratio of impregnation = 3:5

Concentration mg/L	Initial Concentration, Co mg/L	Equilibrium concentration, Ce mg/L	q mg/g	ce/q g/L
25	22.85	2.61	4.048	0.645
50	50.83	7.01	8.764	0.800
100	109.50	20.18	17.864	1.130
150	172.00	57.81	22.838	2.531
200	214.78	76.32	27.692	2.756
250	241.42	129.45	26.394	4.905



From the graph, $y = mx + c$

From Langmuir equation, $\frac{ce}{q} = \frac{1}{q_{max}} ce + \frac{1}{K_L \times q_{max}}$

Hence equation for graph, $y = 0.033x + 0.5139$

$q_{max} = 30.30$ and $K_L = 0.064$

APPENDIX B

Material Safety Data Sheet

Safety Data for Phenol

General

Synonyms: benzenol, carbolic acid, hydroxybenzene, monohydroxybenzene, monophenol, oxybenzene, phenic acid, phenylic acid, phenyl alcohol, phenyl hydrate, phenyl hydroxide and phenylic alcohol.

Molecular formula: C₆H₅OH

CAS No: 108-95-2

EC No: 203-632-7

Annex I Index No: 604-001-00-2

Physical Properties

Appearance: colorless crystals with a characteristic odor

Melting point: 40 - 42°C

Boiling point: 182°C

Specific gravity: 1.07

Vapor pressure: 0.35 mm Hg at 20°C

Flash point: 79°C

Explosion limits: 1.5 % - 8.6 %

Auto ignition temperature: 715°C

Stability

Stable. Substances to be avoided include strong oxidizing agents, strong bases, strong acids, alkalies, calcium hypochlorite. Flammable. May discolor in light.

Risk Phrases

- R24 Toxic in contact with skin
- R25 Toxic if swallowed
- R34 Causes burns
- R36 Irritating to eyes
- R37 Irritating to respiratory system
- R38 Irritating to skin

Toxicology

This material is a systemic poison and constitutes a serious health hazard. The risks of using it in the laboratory must be fully assessed before work begins. Acute poisoning by ingestion, inhalation or skin contact may lead to death. Phenol is readily absorbed through the skin. Highly toxic by inhalation. Corrosive and can causes burns. Severe irritant.

Personal protection

Person should wear safety glasses, gloves and work in good ventilation.

Symbol



Material Safety Data Sheet
Safety Data for Zinc Chloride

General

Synonyms: zinc dichloride, zinc (II) chloride, butter of zinc, zinc butter, zinc chloride fume

Molecular formula: ZnCl_2

CAS No: 7646-85-7

EC No: 231-592-0

Annex I Index No: 030-003-00-2

Physical Properties

Appearance: white powder, crystals or granules

Melting point: 293°C

Boiling point: 732°C

Vapor densities: 4.7 (air = 1)

Vapor pressure: 1 mmHg at 428°C

Densities (g cm^{-3}): 2.9

Water solubility: substantial

Stability

Stable. Incompatible with potassium.

Risk Phrases

R20 Harmful by inhalation

R22 Harmful if swallowed

R34 Causes burns

R50 Very toxic to aquatic organisms

R53 May cause long-term adverse effects in the aquatic environment.

Toxicology

Corrosive. Causes burns. Harmful if swallowed or inhaled and in contact with skin.

Irritant.

Environmental information

Very harmful in the environment - very toxic to aquatic organisms.

Personal protection

Safety glasses, adequate ventilation.

Symbol



