THESIS APPROVAL

GRADUATE SCHOOL, KASETSART UNIVERSITY

Master of Science (Chemistry)

DEGREE

Chemistry	Chemistry
FIELD	**DEPARTMENT**

TITLE: Preparation and Characterization of Activated Carbon from *Bambusa arundinacea (Retz) Wild*, *Bambusa oldhamii* and *Gigantochloa verticiliata*

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THESIS

PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON FROM *BAMBUSA ARUNDINACEA (RETZ) WILD, BAMBUSA OLDHAMII* AND *GIGANTOCHLOA VERTICILIATA*

PITSANU KHORBOOT

A Thesis Submitted in Partial Fulfillment of
the Requirements for the Degree of
Master of Science (Chemistry)
Graduate School, Kasetsart University
2009

Pitsanu Khorboot 2009: Preparation and Characterization of Activated Carbon from *Bambusa arundinacea (Retz) Wild*, *Bambusa oldhamii* and *Gigantochloa verticiliata.* Master of Science (Chemistry), Major Field: Chemistry, Department of Chemistry. Thesis Advisor: Associate Professor Apisit Songsasen, Ph.D. 197 pages.

Activated carbons were prepared from *Bambusa arundinacea (Retz) Wild* (BAW), *Bambusa oldhamii* (GO) and *Gigantochloa verticiliata* (GV), from Kanchanaburi province by chemical activation with phosphoric acid (H_3PO_4) and potassium hydroxide (KOH). The results showed that physical properties and adsorption capacities of the prepared activated carbon depended on types of chemical agents and activation conditions such as chemical concentrations and time of activation. For the adsorption of iodine, methylene blue and Cd (II) of all samples, the activated carbon which was prepared from 1-year-GV charcoal activated by 20%w/v KOH had the highest adsorption capacity in each adsorbate species (1,202 mg/g for iodine, 15.50 mg/g for methylene blue and 0.48 mg/g for Cd (II)). In case of the adsorption of phenol, the bamboo charcoal prepared from 1-year BAW provided the highest adsorption capacity, 4.90 mg/L, while commercial activated carbon was 2.59 mg/L.

The adsorption isotherms of iodine, phenol, methylene blue and Cd (II) were studied. Adsorption isotherm data of iodine, phenol and Cd (II) were fitted to both Langmuir and Freundlich models as considered from the correlation coefficient (R^2). On the other hand, adsorption isotherm data of methylene blue was well fitted with Langmuir isotherm more than Freundlich isotherm.
In addition, the concentration of organic acids found in wood vinegar was subject to types and ages of bamboo. Acetic acid was found with the highest concentration in organic acids.

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Student's signature Thesis Advisor's signature

ACKNOWLEDGEMENTS

I sincerely thank my advisor, Associate Professor Apisit Songsasen, for his tremendous support and meticulous attention throughout the duration of my graduate study and research. I also wish to express my appreciation to my advisory committee, Associate Professor Ranee Suwanapruk, Associate Professor Veerasak Udomchoke and Dr. Potjanart Suwanrujithe, for their worthy suggestion and constructive criticism.

I would like to express my deep gratitude to the Center of Excellence for Innovation in Chemistry: Postgraduate Education and Research Program in Chemistry (PERCH-CIC) for financial support and the Department of Chemistry, Faculty of Science, Kasetsart University for all research facilities.

Finally, I wish to thank my family and friends for their valuable advice, encouragement and understanding.

Pitsanu Khorboot

May, 2009

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LIST OF ABBREVIATIONS

AAS	=	Atomic Absorption Spectrophotometer
ASTM	=	American Society for Testing and Materials
AWWA	=	American Water Works Association
BAW	=	*Bambusa arundinacea (Retz) Wild*
BAWC 1	=	Bamboo charcoal prepared from one-year *Bambusa arundinacea (Retz) Wild*
BAWC 2	=	Bamboo charcoal prepared from two-year *Bambusa arundinacea (Retz) Wild*
BAWC 3	=	Bamboo charcoal prepared from three-year *Bambusa arundinacea (Retz) Wild*
BAWK 1	=	Activated carbon prepared from one-year *Bambusa arundinacea (Retz) Wild* by KOH activation
BAWK 2	=	Activated carbon prepared from two-year *Bambusa arundinacea (Retz) Wild* by KOH activation
BAWK 3	=	Activated carbon prepared from three-year *Bambusa arundinacea (Retz) Wild* by KOH activation
BAWP 1	=	Activated carbon prepared from one-year *Bambusa arundinacea (Retz) Wild* by H_3PO_4 activation
BAWP 2	=	Activated carbon prepared from two-year *Bambusa arundinacea (Retz) Wild* by H_3PO_4 activation
BAWP 3	=	Activated carbon prepared from three-year *Bambusa arundinacea (Retz) Wild* by H_3PO_4 activation
BDDT	=	Brunauer, Deming, Deming, and Teller
EAC	=	Extruded Activated Carbon
FT-IR	=	Fourier Transform Infrared Spectroscopy
GAC	=	Granular Activated Carbon
GO	=	*Bambusa oldhamii*
GOC 1	=	Bamboo charcoal prepared from one-year *Bambusa oldhamii*

LIST OF ABBREVIATIONS (Continued)

GOC 2	=	Bamboo charcoal prepared from two-year *Bambusa Oldhamii*
GOC 3	=	Bamboo charcoal prepared from three-year *Bambusa oldhamii*
GOK 1	=	Activated carbon prepared from one-year *Bambusa oldhamii* by KOH activation
GOK 2	=	Activated carbon prepared from two-year *Bambusa oldhamii* by KOH activation
GOK 3	=	Activated carbon prepared from two-year *Bambusa oldhamii* by KOH activation
GOP 1	=	Activated carbon prepared from one-year *Bambusa oldhamii* by H_3PO_4 activation
GOP 2	=	Activated carbon prepared from two-year *Bambusa oldhamii* by H_3PO_4 activation
GOP 3	=	Activated carbon prepared from three-year *Bambusa oldhamii* by H_3PO_4 activation
GV	=	*Gigantochloa Verticiliata*
GVC 1	=	Bamboo charcoal prepared from one-year *Gigantochloa verticiliata*
GVC 2	=	Bamboo charcoal prepared from two-year *Gigantochloa Verticiliata*
GVC 3	=	Bamboo charcoal prepared from three-year*Gigantochloa Verticiliata*
GVK 1	=	Activated carbon prepared from one-year *Gigantochloa verticiliata* by KOH activation
GVK 2	=	Activated carbon prepared from two-year *Gigantochloa verticiliata* by KOH activation
GVK 3	=	Activated carbon prepared from three-year *Gigantochloa verticiliata* by KOH activation

LIST OF ABBREVIATIONS (Continued)

GVP 1	=	Activated carbon prepared from one-year *Gigantochloa verticiliata* by H_3PO_4 activation
GVP 2	=	Activated carbon prepared from two-year *Gigantochloa verticiliata* by H_3PO_4 activation
GVP 3	=	Activated carbon prepared from three-year *Gigantochloa verticiliata* by H_3PO_4 activation
HPLC	=	High Performance Liquid Chromatography
IUPA	=	International Union of Pure and Applied Chemistry
PAC	=	Powdered Activated Carbon
SEM	=	Scanning Electron Microscopy
SPE	=	Solid Phase Extraction
UV-Vis	=	UV-Vis Spectrophotometry

PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON FROM *BAMBUSA ARUNDINACEA (RETZ) WILD, BAMBUSA OLDHAMII* AND *GIGANTOCHLOA VERTICILIATA*

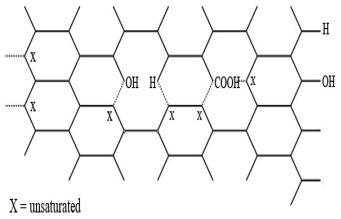
INTRODUCTION

1. Activated carbon

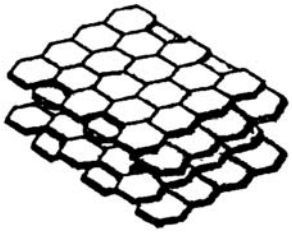
Activated carbon, also called activated charcoal or activated coal, is a form of carbon. Activated charcoal in its broadest sense includes a wide range of a processed amorphous carbon-based material. It is not truly an amorphous material but has a microcrystalline structure that starts to build up during the carbonization process. However, the activated carbon microcrystalline structure differs from that of graphite with respect to the interlayer spacing which is 0.335 nm in the case of graphite and ranges between 0.34 and 0.35 nm in activated carbon. The orientation of the microcrystallite layers is also different, being less ordered in activated carbon. The structures of activated carbon are shown in Figure 1. Activated carbon is a fine black odorless and tasteless powder made from wood or other materials that have been processed to make it extremely porous and thus to have a very large surface area available for adsorption. One gram of activated carbon has a surface area of approximately 500 m^2 (or about 2 tennis courts) as determined typically by nitrogen gas adsorption (Bansal and Goyal, 2005).

Activated carbon in the form of carbonized wood charcoal has been used for many centuries. The Egyptians used this charcoal about 1,500 BC as an adsorbent for medicinal purposes and also as a purifying agent. The ancient Hindus in India purified their drinking water by filtration through charcoal. The first industrial production of activated carbon started about 1900 for use in sugar refining industries. This activated carbon was prepared by the carbonization of a mixture of materials of vegetable origin in the presence of metal chlorides or by activation of the charred material by carbon dioxide (CO_2) or steam. Better quality gas-adsorbent carbon received attention

during World War I, when they were used in gas masks for protection against hazardous gases and vapours.



Source: Berl (1938)



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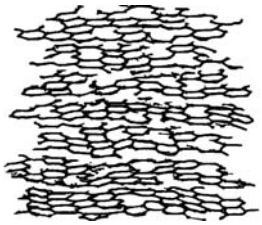
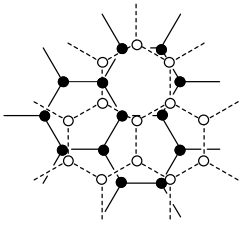


Source: Mattson and Mark (1971)



Source: Suzuki (1990)

Figure 1 The structures of activated carbon.

1.1 Classifications of activated carbon

Activated carbons are complex products which are difficult to classify on the basis of their behavior, surface characteristics and preparation methods. However, some on broad classification is made for general purpose based on their physical characteristics.

1.1.1 Powdered activated carbon (PAC)

Traditionally, activated carbons are made in particular form as powders or fine granules less than 1.0 mm in size with an average diameter between 0.15 and 0.25 mm. Thus, they present a large surface to volume ratio with a small diffusion distance. PAC is made up of crush or ground carbon particles, 95-100% of which will pass through a designated mesh sieve or sieve. The ASTM classifies

particle sizes corresponding to an 80-mesh sieve (0.177 mm) and smaller as PAC. PAC is not commonly used in a dedicated vessel, owing to the high headloss that would occur. PAC is generally added directly to other process units, such as raw water intakes, rapid mix basins, clarifiers and gravity filters (Bansal *et al*., 1988).

1.1.2 Granular activated carbon (GAC)

Granular activated carbon has a relatively larger particle size compared to powdered activated carbon and consequently presents a smaller external surface. Diffusion of the adsorbate is an important factor. These carbons are therefore preferred for all adsorption of gases and vapours as their rate of diffusion are faster. Granulated carbons are used for water treatment, deodourisation and separation of components of flow system. GAC can be either in the granular form or extruded.

1.1.3 Extruded activated carbon (EAC)

EAC consists of extruded and cylindrical shaped activated carbon with diameters from 0.8 to 45 mm. These are mainly used for gas phase applications because of their low pressure drop, high mechanical strength and low dust content.

1.1.4 Impregnated coated carbon

Porous carbons containing several types of inorganic impregnants such as iodine, silver, and cation (such as Al, Mn, Zn, Fe, Li, and Ca) have also been prepared for specific application in air pollution control especially in museums and galleries. Due to antimicrobial/antiseptic properties, silver loaded activated carbon is used as an adsorbent for purifications of domestic water. Drinking water can be obtained from natural water by treating the natural water with a mixture of activated carbon and flocculating agent $Al(OH)_3$. Impregnated carbons are also used for the adsorption of H_2S and mercaptans. Adsorption rates for H_2S as high as 50% by weight have been reported (Bansal *et al*., 1988).

1.1.5 Polymers coated carbon

This is one of the activated carbon using the process by which a porous carbon can be coated with a biocompatible polymer to give a smooth and permeable coat without blocking the pores. The resulting carbon is useful for hemoperfusion. Hemoperfusion is a treatment technique in which large volumes of the patient's blood are passed over an adsorbent substance in order to remove toxic substances from blood (Bansal *et al*., 1988).

1.2 Production

1.2.1 Carbonization

The carbonization is the process using the calcination of a carbonaceous raw material at temperatures below 800°C in an inert atmosphere. Thus, all carbonaceous materials can be converted into activated carbon, although the properties of the final product will be different, depending on the nature of the raw material used, the nature of the activating agent, as well as the conditions of the carbonization and activation process. During the carbonization process, most of the noncarbon element such as oxygen, hydrogen, and nitrogen are eliminated as volatile gaseous species by the pyrolytic decomposition of the starting material. The residual elementary carbon atoms group themselves into stacks of flat, aromatic sheets cross-linked in a random manner. These aromatic sheets are irregularly arranged, which leaves free interstices. These interstices give rise to pores, which make activated carbon as an excellent adsorbent. During carbonization, these pore are filled with the tarry matter or the products of decomposition or at least blocked partially by disorganizes carbon. This pore structure in carbonized char is further developed and enhanced during the activated carbon process, which converts the carbonized raw material into a form that contains the greatest possible number of randomly distributed pores of various sizes and shapes, producing an extended and extremely high surface area of the product (Bansal and Goyal, 2005).

1.2.2 Activation

Activated carbon is produced from carbonaceous source materials like nutshells, wood and coal. It can be produced by one of the following processes.

Physical reactivation: The precursor is developed into activated carbons using gases. This is generally done by using one or a combination of the following processes. Carbonization: Material with carbon content is pyrolyzed at temperatures in the range 600-900°C, in the absence of air (usually in inert atmosphere with gases like argon or nitrogen). Activation/Oxidation: Raw material or carbonized material is exposed to oxidizing atmospheres (carbon dioxide, oxygen, or steam) at temperatures above 250°C, usually in the temperature range of 600-1200°C.

Chemical activation: Impregnation with chemicals such as acids like phosphoric acid or bases like potassium hydroxide, sodium hydroxide or salts like zinc chloride, followed by carbonization at temperatures in the range of 450-900°C. It is believed that the carbonization/activation step proceeds simultaneously with the chemical activation. This technique can be problematic in some cases, because, for example, zinc trace residues may remain in the end product. However, chemical activation is preferred over physical activation owing to the lower temperatures and shorter time needed for activating material. Table 1 showed the examples of chemicals for the preparation of prepared activated carbon.

Table 1 Examples of chemicals for the preparation of activated carbon by chemical activation.

Acid	Base	Salt
Boric acid (H_3BO_3)	Sodium hydroxide (NaOH)	Ferric chloride ($FeCl_3$)
Phosphoric acid (H_3PO_4)	Calcium hydroxide ($Ca(OH)_2$)	Zinc chloride ($ZnCl_2$)
Nitric acid (HNO_3)	Potassium hydroxide (KOH)	Potassium sulfide (K_2S)
Sulfuric acid (H_2SO_4)		Potassium thiocyanate (KSCN)
		Calcium phosphate ($Ca_3(PO_4)_2$)
		Calcium chloride ($CaCl_2$)

Source: Wikipedia (2008)

1.2.2.1 Activation with H_3PO_4

The mechanism for the formation of pores in activated carbon by H_3PO_4 is as follows (Jibril *et al.,* 2008);

$$2H_3PO_4 \longrightarrow P_2O_5 + 3H_2O \quad \text{------ (1)}$$

$$2P_2O_5 + 5C \longrightarrow P_4 + 5CO_2 \quad \text{------ (2)}$$

1.2.2.2 Activation with KOH

The mechanism for the formation of pores in activated carbon by KOH is as follows (Yang and Lua, 2003). The first step of the mechanism is the thermal dehydration of KOH.

$$2KOH \longrightarrow K_2O + H_2O \quad \text{------ (3)}$$

The formation of CO_2 and H_2 will be then the consequence of the combined effect of the following reactions.

$$C + H_2O \longrightarrow H_2 + CO \quad \text{------ (4)}$$
$$CO + H_2O \longrightarrow H_2 + CO_2 \quad \text{------ (5)}$$

Metallic potassium will be formed by the reduction of K_2O by carbon or hydrogen at high temperature.

$$K_2O + C \longrightarrow 2K + CO \quad \text{------ (6)}$$
$$K_2O + H_2 \longrightarrow 2K + H_2O \quad \text{------ (7)}$$

1.3 Pore structure and surface of activated carbon

Activated carbons are associated with pores starting from less than a nanometer to several thousand nanometers. This classification of pores by International Union of Pure and Applied Chemistry (IUPAC) is based on their width, which represents the distance between the walls of a slit-shaped pore or the radius of a cylindrical pore. The pores are divided into three groups as the micropores, the mesopores, and the macropores. The classification of pores is shown in Figure 2.

Micropores have molecular dimensions and the effective radius is less than 2 nm. The adsorption in these pores occurs through volume filling, and there is no capillary condensation taking place. The adsorption energy in these pores is much larger compared to larger mesopores or to the nonporous surface because of the overlapping of adsorption forces from the opposite walls of the macropores. They generally have a pore volume of the 0.15 to 0.70 cm^3/g. Their specific surface area constitutes about 95% of the total surface area of the activated carbon.

Mesopores, also called transitional pores, have effective dimensions in the 2 to 50 nm range, and their volume usually varies between 0.1 and 0.2 cm^3/g. The surface area of these pores does not exceed 5% of total surface area of the carbon.

Macropores are not of considerable importance to the process of adsorption in activated carbon because their contribution to the surface area of the adsorbate is very small and does not exceed 0.5 m^2/g. they have effective radii larger than 50 nm, and frequently in the 500 to 2,000 nm range, with a pore volume between 0.2 and 0.4 cm^3/g. They act as transport for the adsorbate into the micorpore and mesopore.

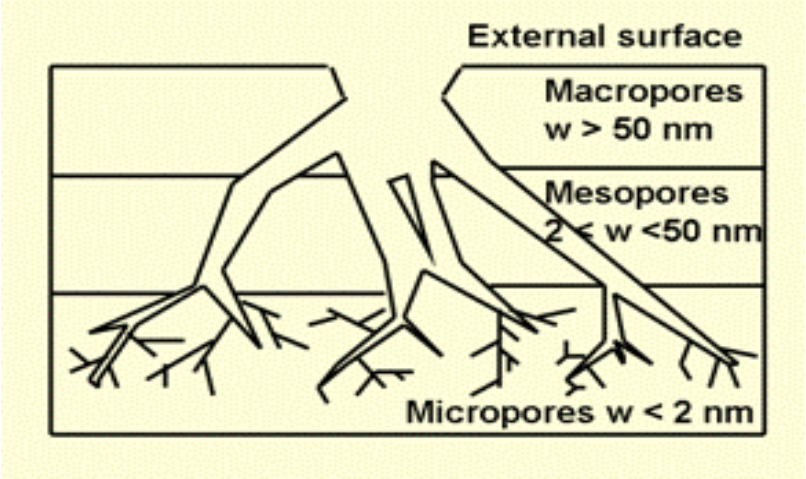


Figure 2 The classification of pores.

Source: Wikipedia (2008)

1.4 Chemical structure of the carbon surface

Besides the crystalline and porous structure, an activated carbon surface has a chemical structure. The adsorption capacity of activated carbon is determined by the physical or porous structure but it is strongly influenced by the chemical structure of carbon surface. In graphites that have a highly ordered crystalline surface, the adsorption capacity is determined mainly by the dispersion component of Van der Waals forces. But the random ordering of aromatic sheets in activated carbons causes a variation in the arrangement of electron clouds in the carbon skeleton and results in the creation of unpaired electrons and incompletely saturated valences, which would undoubtedly influence the adsorption properties of activated carbons (Bansal and Goyal, 2005).

Activated carbons are almost invariably associated with appreciable amounts of oxygen and hydrogen. In addition, they may be associated with atoms of sulfur, nitrogen, and halogens. These heteroatoms are derived from the starting material and become a part of the chemical structure as the result of imperfect carbonization, or they become chemically bonded to the surface during activation or during subsequent treatments. There is also evidence that the carbon can adsorb certain molecular species such as amines, nitrobenzene, phenol, and several other cationic species.

2. Adsorption

Adsorption is a process that occurs when a gas or liquid solute accumulates on the surface of a solid or a liquid (adsorbent), forming a film of molecules or atoms (adsorbate). It is different from absorption, in which a substance diffuses into a liquid or solid to form a solution (Bansal and Goyal, 2005).

2.1 Adsorption process

Adsorption arises as a result of the unsaturated and unbalanced molecular forces that are present on every solid surface. Thus, when a solid surface is brought into contact with a liquid or gas, there is an interaction between the fields of forces of the surface and that of the liquid or gas. The solid surface tends to satisfy these residual forces by attracting and retaining on its surface the molecules, atom, or ion of the gas or liquid. This results in a greater concentration of the gas or liquid in the near vicinity of solid surface than in the bulk gas or vapor phase, despite the nature of gas or vapor. The process by which this surface excess is caused is called adsorption. The adsorption characteristic into pores of adsorbent is also shown in Figure 3.

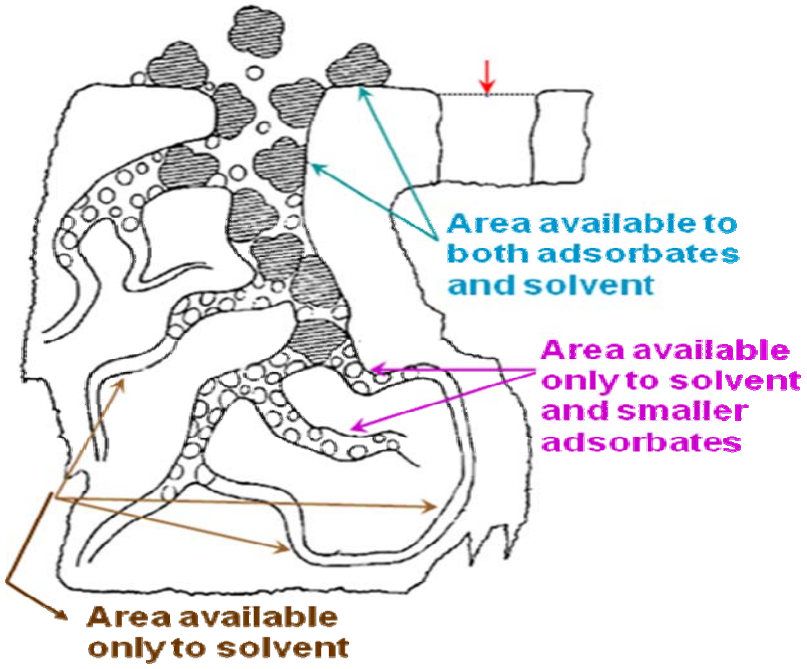


Figure 3 The adsorption characteristic into pores of adsorbent.

Source: Culp, G.L. and R.L. Culp (1974)

The adsorption involves two types of forces as physisorption (characteristic of weak Van der Waals forces) or chemisorption (characteristic of covalent bonding).

Physical adsorption: The adsorbate is bound to the surface by relatively weak Van der Waals forces, which are similar to the molecular force of cohesion and are involved in the condensation of vapors into liquids.

Chemical adsorption: Chemisorption involves exchange or sharing of electrons between the adsorbate molecules and the surface of the adsorbent resulting in a chemical reaction. The bond formed between the adsorbate and the adsorbent is essentially a chemical bond and is thus much stronger than in the physisorption.

2.2 Adsorption isotherm

To measure total surface area, nonspecific physical adsorption is required, but even with physical adsorption the isotherm varies somewhat with the nature of the adsorbent. Most physical adsorption isotherms may be grouped into six types, which are frequently referred to as the Brunauer, Deming, Deming, and Teller (BDDT) classification (Figure 4). In all cases the amount of vapor adsorbed gradually increases as its partial pressure is increased, becoming at some point equivalent to a monolayer, but then increasing to a multilayer, which eventually merges into a condensed phase. Types of adsorption isotherm are shown in Figure 4.

Type I is frequently called the Langmuir type. The asymptotic value was originally ascribed to a monolayer, as derived from the Langmuir equation. However, this isotherm is seldom encountered on nonporous materials. It is fairly common with certain activated carbon, silica gels, and zeolites that contain only very fine pores, and it is now generally believed that in these cases the asymptotic value represents the complete filling of microspores at a relative pressure substantially less than unity, rather than monolayer adsorption his type of isotherm would also be expected for reversible chemisorptions.

Type II, sometimes termed the sigmoid or S-shaped isotherm, is commonly encountered on nonporous structures. Point B occurs at a “knee” and is the stage at which monolayer coverage is complete.

Type III isotherm is convex over the entire range and does not exhibit a point B. It is relatively rare and is typical of a system where the forces of adsorption are relatively weak, as when the adsorbate is not wetted by the surface, e.g., water vapor on graphite.

Type IV is encountered with porous material. At low values of P/P_o the isotherm is similar to type II, but adsorption increases markedly at higher values of $P/P_o$where pore (capillary) condensation takes place. A hysteresis effect associated with this pore condensation is frequently, but not always, observed. Isotherms of this type are often encountered with industrial catalysts, and the capillary condensation curve may be used to determine a pore size distribution.

Type V is similar to type III, but with pore condensation taking place at higher values of P/P_o. It is also relatively rare.

Type VI is the stepped isotherm which is rarely found. This system involves the step-by-step adsorption on surface, like uniform adsorption. The isotherm shape depends on the system and temperature for adsorption.

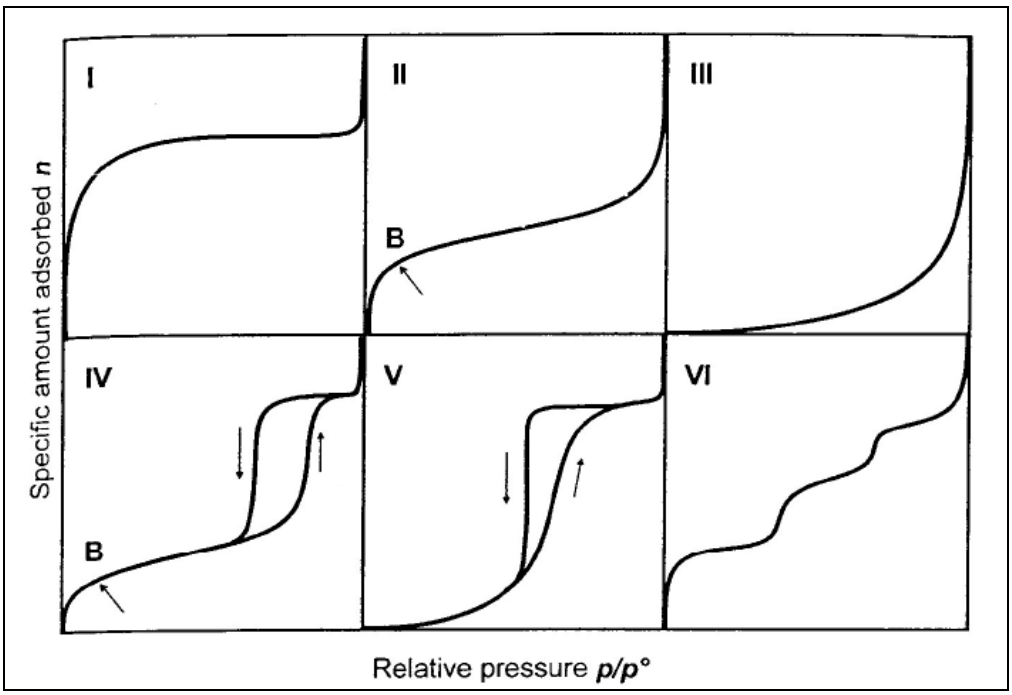


Figure 4 Types of adsorption isotherm.

Source: Masel (1996)

The adsorption isotherm indicates how the adsorption molecules distribute between the liquid phase and the solid phase when the adsorption process reaches an equilibrium state. The analysis of the isotherm data by fitting them to different isotherm models is an important step to find the suitable model that can be used for design purposes.

The procedures for the kinetic experiments were basically identical to those for the equilibrium test. The solution samples were taken at regular time interval and the concentrations of samples were measured. The plots of X/M versus C_e for adsorption are shown in Figure 5 and the amount of adsorption (q_t, mg/g) at time (t) was calculated by the following equation.

$$q_t = \frac{X}{m} = \frac{(C_o - C_t)V}{M} \qquad \text{------ (8)}$$

where q_t is the amount of adsorption at the time (mg/g)

X is the amount of absorbate (mg)

m is the mass of adsorbent used (g)

C_o is the liquid-phase concentrations of adsorbate at the initial (mg/L)

C_t is liquid-phase concentrations of samples at the time (mg/L)

V is the volume of solution (L)

For equilibrium

$$q_e = \frac{X}{m} = \frac{(C_o - C_e)V}{m} \qquad \text{------ (9)}$$

where q_e is the amount of adsorption at equilibrium (mg/g)

C_e is the liquid-phase concentrations of adsorbate at the time (mg/L)

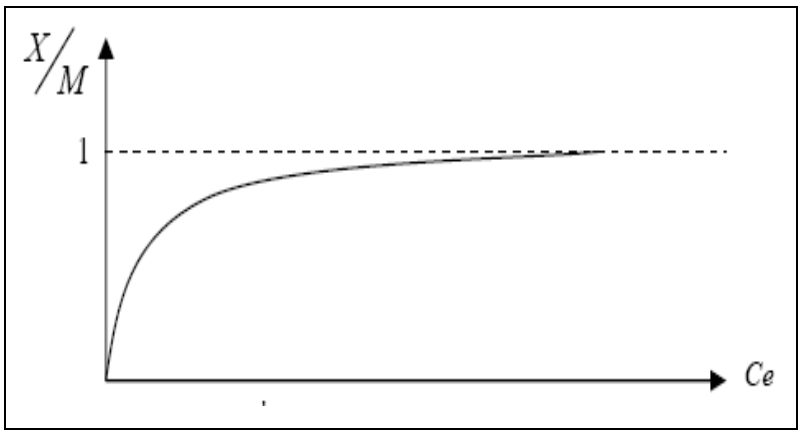


Figure 5 The plots of X/M versus C_e for adsorption.

Source: Cheremisinoff and Morresi (1987)

8.2.1 Langmuir isotherm

Langmuir isotherm assumes monolayer adsorption onto a surface containing a finite number of adsorption sites of uniform strategies of adsorption with no transmigration of adsorbate in the plane of surface. The linear form of the Langmuir isotherm equation is represented by the following equation;

$$q_e = \frac{Q_o b C_e}{1+bC_e} \qquad \text{------ (10)}$$

$$\frac{C_e}{q_e} = \frac{1}{Q_o b} + \frac{C_e}{Q_o} \qquad \text{------ (11)}$$

where C_e is the equilibrium concentration of the adsorbate (mg/L)

q_e is the amount of adsorbate adsorbed per unit mass of adsorbent (mg/g)

Q_o is the maximum surface coverage (formation of monolayer) of sorbent (mg/g)

b is the adsorption energy constant of Langmuir adsorption isotherm (L/mg)

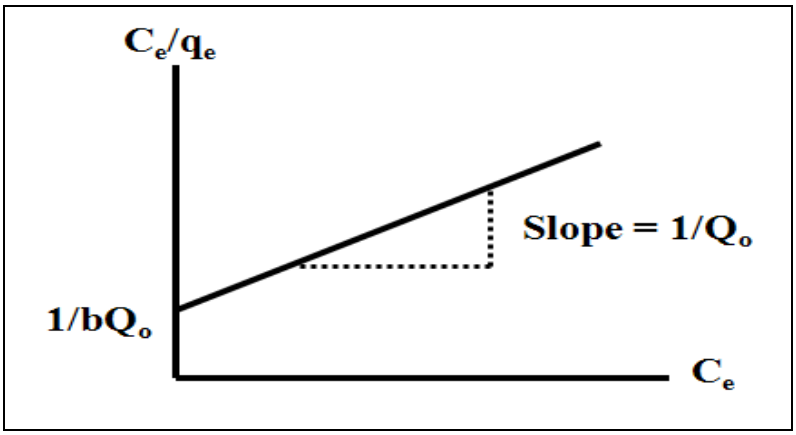


Figure 6 The Langmuir isotherm plots of C_e/ q_e versus C_e for adsorption.

Source: Cheremisinoff and Morresi (1987)

Langmuir isotherm plots of C_e/ q_e versus C_e for adsorption is shown in Figure 6. Furthermore, the plot of C_e/q_e versus C_e gives straight line with slope $1/Q_o$ is obtained. The Langmuir constants b and Q_o are calculated from this isotherm. When the molecule adsorbs on the surface of activated carbon based on the Langmuir isotherm, the specific surface area can be calculated as follows;

$$S = \frac{Q_o}{MW} \times N \times a \qquad \text{------ (12)}$$

where S is the specific surface area (m^2/g)

Q_o is the the maximum surface coverage (formation of monolayer) of sorbent (mg/g)

MW is the molecular weight (g/mol)

N is the Avogadro number (6.02×10^{23} molecule/mol)

a is the cross sectional area of adsorbate ($Å^2$)

2.2.2 Freundlich isotherm

Freundlich isotherm assumes heterogeneous surface energies, in which the energy term in Langmuir equation varies as a function of the surface coverage. The well-known logarithmic form of Freundlich model is given by the following equation.

$$q_e = K_F C_e^{1/n} \qquad \text{------ (13)}$$

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \qquad \text{------ (14)}$$

where q_e is the amount adsorbed at equilibrium (mg/g)

C_e is the equilibrium concentration of the adsorbate (mg/L)

K_F is the Freundlich isotherm constant related to adsorption capacity $((mg^{-1})(mg^{-1})^{1/n})$

n is the Freundlich isotherm constant related to adsorption intensity

The slope 1/n ranging between 0 and 1 is a measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero. A value for 1/n below one indicates a normal Langmuir isotherm while 1/n above one is indicative of cooperative adsorption. The Freundlich isotherm plot of log q_e versus log C_e for the adsorption is shown in Figure 7. Moreover, the plot of log q_e versus log C_e gives straight line with slope 1/n is obtained.

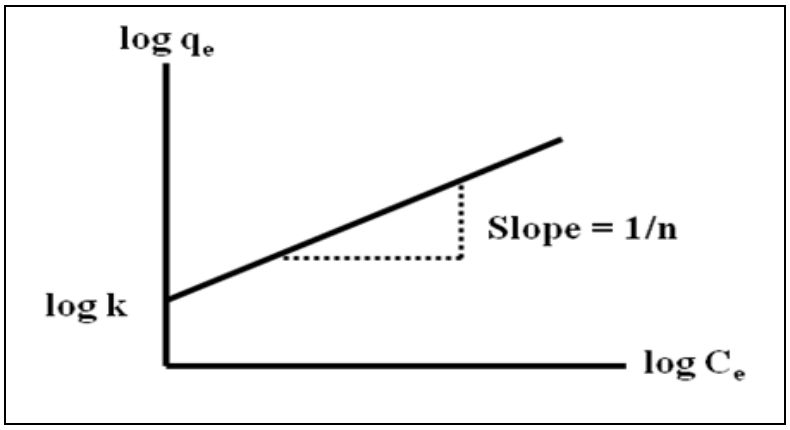


Figure 7 The Freundlich isotherm plots of log q_e versus log C_e for the adsorption.

Source: Cheremisinoff and Morresi (1987)

3. Bamboo

The bamboos are a group of woody perennial evergreen, except for certain temperate species, plants in the true grass family Poaceae, subfamily Bambusoideae, and tribe Bambuseae. Some are giant bamboos, the largest members of the grass family. Bamboos are the fastest growing woody plants in the world. Their growth rate (up to 60 centimeters/day) is due to a unique rhizome-dependent system, but is highly dependent on local soil and climate conditions. They are of economic and high cultural significance in East Asia and South East Asia where they are used extensively in gardens, as a building material, and as a food source.

3.1 *Bambusa arundinacea (Retz) Wild*

Familia: *Poaceae*
Genus: *Bambusa*
Synonyms: *Bambusa arundinacea (Retz) Wild*
Common name: Paipa

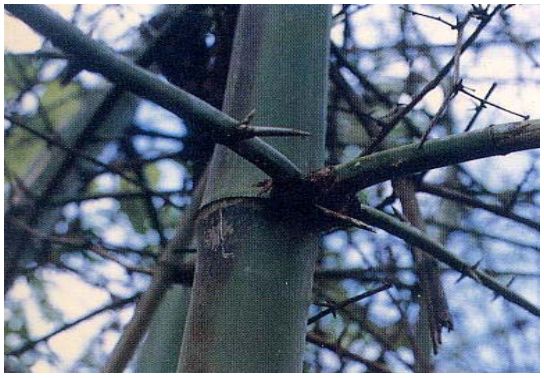


Figure 8 The images of *Bambusa arundinacea (Retz) Wild*.

Source: Wikipedia (2008)

Origin and geographic distribution: India to Southern China, including Singapore, Peninsular Malaysia, Thailand and the Philippines.

Stem: up to 10-24 m, 15-18 cm in diameter, wall up to 1-5 cm thick, internodes usually 20-40 cm long, and bright green.

The dimensions of fibres in the culm: length 1.73-2.52 mm, diameter 16.34-22.0 μm, and wall thickness 5.37-8.0 μm.

The chemical composition of the culm: holocellulose 58-67%, pentosans 10.37-12.12%, and lignin 22-30%.

Propagation and planting: Paipa is propagated by seed, rhizome cutting and tissue culture. Fresh seed germinates readily in 5-10 days after sowing, with a germination percentage of up to 80%. Seeds remain viable for about 6 months when stored at 5°C or when stored dry in sealed containers (with $CaCl_2$) at room temperature. Seed stored without lowering temperature or moisture content will lose viability completely within 3 months. The images of *Bambusa arundinacea (Retz) Wild* are shown in Figure 8.

3.2 *Bambusa oldhamii*

Familia: *Poaceae*

Genus: *Bambusa*

Synonyms: *Bambusa oldhamii*

Common name: Oldham bamboo or Green bamboo or Lu Chu

Figure 9 The images of *Bambusa oldhamii*.

Source: Wikipedia (2008)

Origin and geographic distribution:

Stem: up to 6-12 m, 5-12 cm in diameter, wall up to 0.4-1.2 cm thick, internodes usually 20-35 cm long, and green to yellow-green.

Dimensions of fibres in the culm: length 1.54-2.26 mm, diameter 16.34-19.71 μm, and wall thickness 5.21-7.59 μm.

The chemical composition of the culm: holocellulose 58-67%, pentosans 9.87-12.50%, and lignin 22-30%.

Propagation and planting: Lu Chu is only propagated vegetatively by rhizome, culm or branch cutting. Cuttings from flowering clumps should be avoided because they will start flowering soon after planting. Culm cuttings have shown a survival rate of nearly 100%. The best time for planting is in the rainy season. The images of *Bambusa oldhamii* are shown in Figure 9.

3.3 *Gigantochloa verticiliata*

Familia: *Poaceae*
Genus: *Gigantochloa*
Synonyms: *Gigantochloa verticiliata*
Common name: Manmoo

Figure 10 The images of *Gigantochloa verticiliata*.

Source: Wikipedia (2008)

Origin and geographic distribution: the origin of Mummoo is not known. It is only found in cultivation.

Stem: up to 8-15 m, 7-10 cm in diameter, wall up to 2 cm thick, internodes usually 40-60 cm long, and green to yellow-green.

Dimensions of fibres in the culm: length 2.75-3.27 mm, diameter 24.55-37.97 µm and wall thickness 5.51-7.61 µm.

The chemical composition of the culm: holocellulose 58-67%, pentosans 11.06%, and lignin 22-30%.

Propagation and planting: Manmoo is only propagated vegetatively by rhizome, culm or branch cutting. Cuttings from flowering clumps should be avoided because they will start flowering soon after planting. Culm cuttings have shown a survival rate of nearly 100%. The best time for planting is in the rainy season. The images of *Gigantochloa verticiliata* are shown in Figure 10.

4. Iodine

Iodine under standard condition is a shiny grey solid. It can be seen apparently sublimating at the standard temperature into a violet-pink gas that has an irritating odor. This halogen forms compounds with many elements, but is less reactive than the other members of its Group VII (halogens) and has some metallic light reflectance. Elemental iodine dissolves easily in chloroform and carbon tetrachloride. The solubility of elemental iodine in water can be vastly increased by the addition of potassium iodide. The molecular iodine reacts reversibly with the negative ion, creating the triiodide anion (I_3^-) which dissolves well in water. The properties of iodine are shown in Table 2. Iodine element is rare in the earth's crust. They are very soluble in water and the element is concentrated in seawater. Furthermore, the iodine and its compounds are primarily used in medicine, photography, and dyes. However, directly contacting iodine with skin can cause lesions. Iodine vapor is very irritating to the eye and to mucous membranes. Concentration of iodine in the air should not exceed 1 mg/m³. When mixed with ammonia, it can form nitrogen triiodide which is extremely sensitive and can explode unexpectedly. Iodine is fatal to humans if 2–3 grams of it are consumed. Iodides are similar in toxicity to bromides.

Table 2 The properties of iodine.

Properties	
General	
Name	iodine (I)
Number	53
Appearance	violet-dark gray, lustrous
Atomic weight (g/mol)	126.90447
Electron configuration	[Kr] $4d^{10}$ $5s^2$ $5p^5$
Electrons per shell	2, 8, 18, 18, 7
Physical properties	
Phase	solid
Density (g/cm^3)	4.933
Melting point (°C)	113.7
Boiling point (°C)	184.3
Heat of fusion (kJ/mol)	15.52
Heat of vaporization (kJ/mol)	41.57
Atomic properties	
Crystal structure	orthorhombic
Oxidation states	±1, 5, 7
Electronegativity	2.66
Ionization energies	
1^{st} (kJ/mol)	1,008.4
2^{nd} (kJ/mol)	1,845.9
3^{rd} (kJ/mol)	3,180.0
Atomic radius (pm)	140
Covalent radius (pm)	133
Van der Waals radius (pm)	198

Source: Wikipedia (2008).

5. Phenol

5.1 Characteristics of phenol

Phenol, also known as carbolic acid, is a white crystalline solid with a sweet tarry odor, commonly referred to as a “hospital smell”. Its chemical formula is C_6H_5OH and its chemical name is hydroxybenzene. The structure of phenol is that of a hydroxyl group (-OH) bonded to a phenyl ring. Thus, it is an aromatic compound which slightly acidic. The structure of phenol is shown in Figure 11. The phenol molecule has weak tendencies to lose the H^+ ion from the hydroxyl group, resulting in the highly water-soluble phenolate anion ($C_6H_5O^-$), and also called phenoxide anion. Compared to aliphatic alcohols, phenol shows much higher acidity. It even reacts with aqueous NaOH to lose H^+, whereas aliphatic alcohols do not. However, many carboxylic acids are more acidic than phenol. One explanation for the increased acidity over alcohols is 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 a sp^3 system, allows for great stabilization of the oxyanion. The properties of phenol are shown in Table 3.

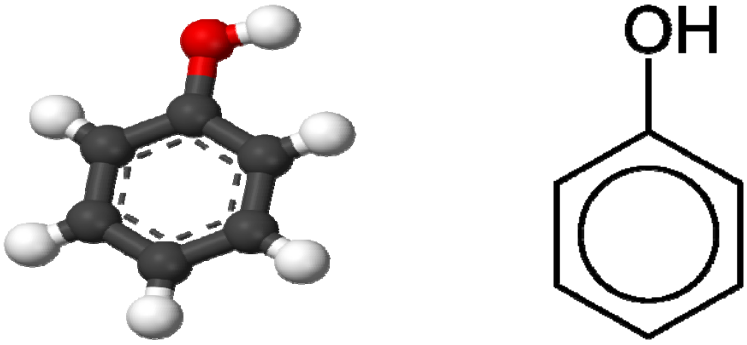


Figure 11 The structure of phenol.

Source: Wikipedia (2008).

5.2 Applications of phenol

Phenol is used in the production of drugs (it is the starting material in the industrial production of aspirin), herbicides, and synthetic resins (Bakelite, one of the first synthetic resins to be manufactured, is a polymer of phenol with formaldehyde). Phenol is the preferred chemical in use of embalming bodies for anatomical use and study because of its ability to preserve tissues for extended periods of time. Phenol is also used in the preparation of cosmetics including sunscreens, hair dyes, and skin lightening preparations. Compounds containing phenol moieties can be used to prevent ultraviolet light which induced damage to hair and skin due to the UV-absorbing properties of the aromatic ring of the phenol.

5.3 Toxicity of phenol

Inhalation and dermal exposure to phenol is highly irritating to the skin, eyes, and mucous membranes in humans. Phenol is considered to be very toxic to humans through oral exposure, with ingestion of 1 g reported to be lethal, with symptoms including muscle weakness and tremors, loss of coordination, paralysis, convulsions, coma, and respiratory arrest. Blood changes, liver and kidney damage, and cardiac toxicity including weak pulse, cardiac depression, and reduced blood

pressure have been reported in humans acutely exposed to phenol by the oral route. Acute (short-term) animal tests, such as the LD_{50} tests in rats, mice, and rabbits have shown phenol to have high acute toxicity from oral exposure. Long-term inhalation exposure to phenol in animal studies has shown effects on the liver, kidney, respiratory, cardiovascular, and central nervous systems.

Table 3 The properties of phenol.

Properties	
General	
Molecular formula	C_6H_5OH
Molar mass (g/mol)	94.11
Apprearance	white crystalline solid
Physical and chemical properties	
Density (g/cm^3)	1.07
Melting point (°C)	40.5
Boiling point (°C)	181.7
Solubility in water (g/100 ml)	8.3 (20 °C)
Acidity (pK_a)	9.95

Source: Wikipedia (2008)

6. Methylene blue

6.1 Characteristics of methylene blue

Methylene blue which is a heterocyclic aromatic chemical compound is shown in Figure 12. The molecular formula is $C_{16}H_{18}ClN_3S$ which is called 3,7-bis (dimethylamino)-phenazathionium chloride or tetramethylthionine chloride. At room temperature it appears as a solid, odorless, and dark green powder, that yields a blue solution when dissolved in water. The properties of methylene blue are shown in Table 4.

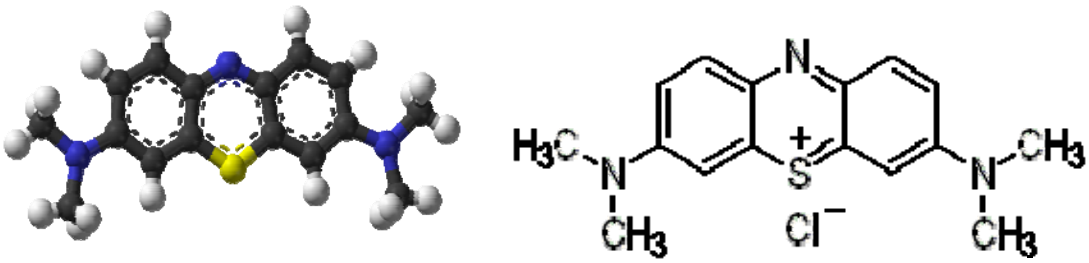


Figure 12 The structure of methylene blue.

Source: Wikipedia (2008)

6.2 Applications of Methylene blue

Methylene blue is widely used as a redox indicator in analytical chemistry, photosensitizer, medicine and dye.

6.3 Toxicity of Methylene blue

Methylene blue will be harmful if it is swallowed, inhaled or in contact with skin. It causes severe eye irritation, including carcinogenicity, reproductive and

developmental toxicity, neurotoxicity, and acute toxicity. Generally, methylene blue is mostly produced from the textile industries in terms of wastewater which is high in both color and organic contents. Therefore, the effluents discharged from such dyeing industries are highly colored; in which methylene blue can be toxic to aquatic life in receiving waters.

Table 4 The properties of methylene blue.

Properties	
General	
Molecular formula	$C_{16}H_{18}N_3ClS$
Molar mass (g/mol)	319.85
Apprearance	solid
Physical and chemical properties	
Melting point (°C)	100-110 (with decomposion)
Boiling point	Decomposes
Solubility in water (g/100 ml)	4 (20°C)

Source: Wikipedia (2008)

7. Cadmium

7.1 Characteristics of cadmium

Cadmium is a chemical element with the symbol Cd and atomic number 48. Cadmium is a soft, malleable, ductile, toxic, and bluish-white transition metal. It is similar in many respects to zinc but forms more complex compounds. The most common oxidation state of cadmium is +2, though rare examples of +1 can be found.

Cadmium burns in air to form brown amorphous cadmium oxide (CdO). The crystalline form of the same compound is dark red and changes colour when heated, similar to zinc oxide. Hydrochloric acid, sulfuric acid and nitric acid dissolve cadmium by forming cadmium chloride ($CdCl_2$) cadmium sulfate ($CdSO_4$) or cadmium nitrate ($Cd(NO_3)_2$). The oxidation state +1 can be reached by dissolving cadmium in a mixture of cadmium chloride and aluminium chloride, forming the Cd_2^{2+} which is similar to the Hg_2^{2+}. The physical properties of Cd are shown in Table 5.

7.2 Cadmium in natural resources

Cadmium-containing ores are rare and are found to occur in small quantities. Greenockite (CdS), the only cadmium mineral of importance, is nearly always associated with sphalerite (ZnS). As a consequence, cadmium is produced mainly as a byproduct from mining, smelting, and refining sulfide ores of zinc. Moreover, small amounts of cadmium about 10% of consumption are produced from secondary sources, mainly from dust generated by recycling iron and steel scrap.

7.3 Applications of cadmium

Cadmium is used largely in batteries and other uses include pigments, coatings, plating, (as stabilizers for plastics), and bearing alloys.

7.4 Toxicity of cadmium

Cadmium poisoning is an occupational hazard associated with industrial processes such as metal plating and the production of nickel-cadmium batteries, pigments, plastics, and other synthetics. The primary route of exposure in industrial settings is inhalation. Inhalation of cadmium-containing fumes can initially result in metal fume fever but may progress to chemical, pneumonitis, pulmonary edema, and death. Cadmium is also a potential environmental hazard.

Table 5 The physical properties of cadmium.

Properties	
General	
Name	cadmium (Cd)
Number	48
Apprearance	silvery gray metallic
Atomic weight (g/mol)	112.411
Electron configuration	[Kr] $5s^2$ $4d^{10}$
Electrons per shell	2, 8, 18, 18, 2
Physical properties	
Phase	solid
Density (g/cm^3)	8.65
Melting point (°C)	321.07
Boiling point (°C)	767
Heat of fusion (kJ/mol)	6.21
Heat of vaporization (kJ/mol)	99.87
Atomic properties	
Crystal structure	hexagonal
Oxidation states	+2, +1
Electronegativity	1.69
Ionization energies	
1^{st}: (kJ/mol)	867.8
2^{nd}:(kJ/mol)	1,631.4
3^{rd}: (kJ/mol)	3,616.0
Atomic radius (pm)	155
Covalent radius (pm)	148
Van der Waals radius (pm)	158

Source: Wikipedia (2008)

8. Wood vinegar

Wood vinegar or pyroligneous acid is a brown transparent liquid. It is a byproduct from charcoal production. It is normally generated from the gas and combustion of fresh wood burnt in airless condition. When the gas is cooled, it will condense into liquid. Raw wood vinegar consists of more than 200 chemical compounds, such as acetic acid, formaldehyde, ethyl-valerate, methanol, tar, etc. The components in wood venigar are shown in Table 6.

8.1 The primitive production process of wood vinegar

The wood that was heartwood and bark is cured for 5-15 days. Then, it is moved into a kiln, like a pile wood. The kiln needs to be tightly closed and the hole should be covered with clay. After that, the wood will be burnt at 120-430^{o}C for an hour. If brown or dark brown drops appear on the tile that was put on the top of the chimney, smoke should be flown through a bamboo pipe so that the hot stream may be condensed into liquid. Afterwards, the wood vinegar obtained is droped into a vessel via the bamboo pipe. If wood is burnt for 12-15 hours in a 200-liter oil drum kiln, it will produce 2-7 liters of wood vinegar. At this stage, it is called raw wood vinegar. Then, the raw wood vinegar was left for 3 months to become silted. The vinegar will turn yellow like vegetable oil into light brown and the tar will become silted. The top content will be light, clear oil. Remove the tar and light oil, as well as the dark brown translucent oil and the remainder will be sour vinegar.

8.2 The benefits of wood vinegar

8.2.1 Stimulates vegetable growth

8.2.2 Enriches soil fertility

8.2.3 Works as flavor enhancer for agricultural end products

8.2.4 Inhibits virus and soil disease when mixed in high concentration

8.2.5 Repels viruses and harmful insects to improve soil conditions

8.2.6 Prevents diseases caused by bacteria

8.2.7 Improves fruit quality and increases sugar content in fruit

8.2.8 Helps the animal live healthier and protected from disease

8.2.9 Improves the quality of meat and milk

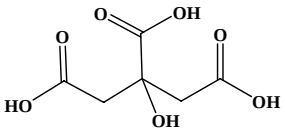
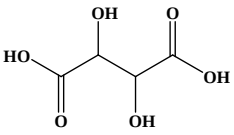
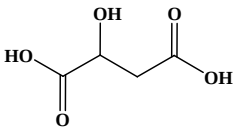
Table 6 The components in wood venigar.

	Component
Organic acid	formic acid, acetic acid, propionic acid, butyric acid, isobutyric acid, valeric acid, isovalenic acid, etc.
Phenol	phenol, o-cresol, m-cresol, p-cresol, 2,4-xylenol, 3,5-xylenol, 4-ethylphenol, 4-propylphenol, catechol, formaldehyde, acetaldehyde, isobutyldehyde, etc.
Carbonyl compound	butylaldehyde, glyoxal, acrolein, acetone, methylethylketone, etc.
Alcohol	methanol, ethanol, propanol, isopropanol, allylalcohol, etc.
Neutral ingredients	acetol, maltor, 4-methyl veratrole, 4-ethyl veratrole, 4-propyl veratrole, 3,4-benzopyrene, etc.
Basic ingredients	ammonia, methylamine, dimethylamine, pyridine, etc.

Source: Wikipedia (2008)

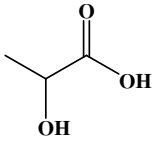
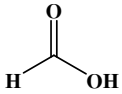
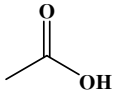
There are nine important organic acid compounds from which this work will focus. Moreover, the significant physical properties and the utilization of all nine organic acid compounds are shown in Table 7 and 8.

Table 7 The significant physical properties of all nine organic acid compounds.

Properties	Types of organic acids		
	citric acid	tartaric acid	malic acid
General			
Molecular formula	$C_6H_8O_7$	$C_4H_6O_6$	$C_4H_6O_5$
Chemical structure			
IUPAC	2-hydroxypropane-1,2,3-tricarboxylic acid	3-dihydroxy butanedioic acid	hydroxy butanedioic acid
Molar mass (g/mol)	192.123	150.087	134.090
Physical and chemical properties			
Apprearance	white crystal	white powder	white crystal
Melting point (K)	426.15	443.15	401.15
Solubility in water (g/100 ml)	soluble	soluble	excellent soluble
Liquid density (g/cm^3)	1.542	1.800	1.609

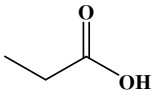
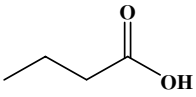
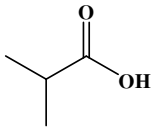
Source: Wikipedia (2008)

Table 7 (Continued)

Properties	Types of organic acids		
	lactic acid	formic acid	acetic acid
General			
Molecular formula	$C_3H_6O_3$	CH_2O_2	$C_2H_4O_2$
Chemical structure			
IUPAC	2-hydroxypropanoic acid	methanoic acid	ethanoic acid
Molar mass (g/mol)	90.080	46.025	60.050
Physical and chemical properties			
Apprearance	yellow crystal	transparent liquid	transparent liquid
Melting point (K)	395.15	281.55	289.6
Solubility in water (g/100 ml)	good soluble	soluble	excellent soluble
Liquid density (g/cm^3)	1.2	1.22	1.049

Source: Wikipedia (2008)

Table 7 (Continued)

Properties	Types of organic acid		
	propionic acid	butyric acid	isobutyric acid
General			
Molecular formula	$C_3H_6O_2$	$C_4H_8O_2$	$C_4H_8O_2$
Chemical structure			
IUPAC	propanoic acid	butyric acid	2-methylpropanoic acid
Molar mass (g/mol)	74.08	88.10	88.10
Physical and chemical properties			
Apprearance	transparent liquid	transparent liquid	transparent liquid
Melting point (K)	252.00	265.10	226.15
Solubility in water (g/100 ml)	good soluble	soluble	soluble
Liquid density (g/cm^3)	0.99	0.95	0.97

Source: Wikipedia (2008)

Table 8 The utilization of all nine organic acid compounds**.**

Types of organic acid	Utilization
Citric acid	food and water additive, pharmaceutical industry, and bathroom cleaning solutions
Tartaric acid	photographic chemicals, ceramics industry, flavoring agent in foods
Malic acid	adjunct acid in food, pH control reagent
Lactic acid	admixture in cosmetic and solvent in laboratory
Formic acid	insecticide, antibacterial agent in livestock feed, organic latex (sap) into raw rubber
Acetic acid	catalyst and admixture in industries such as plastic and dye industries
Propionic acid	food additive and solvent in laboratory, preparation of various butanoate esters.
Butyric acid	derivative of butyric acid as 4-(2,4-dichlorophenoxy)-butyric acid which can be used in term of perticides
Isobutyric acid	solvent in laboratory

Source: Wikipedia (2008)

OBJECTIVES

There are four main objectives for this work.

1. To prepare the activated carbon from *Bambusa arundinacea (Retz) Wild*, *Bambusa oldhamii* and *Gigantochloa verticiliata* by chemical activation.

2. To study the properties of the prepared activated carbon.

3. To study the adsorption of iodine, phenol, methylene blue, and cadmium onto the prepared activated carbon.

4. To study the components of organic acids in wood vinegar from *Bambusa arundinacea (Retz) Wild*, *Bambusa oldhamii* and *Gigantochloa verticiliata*.

LITERATURE REVIEW

The reviews studied the previous researches on preparation and characterization of activated carbon by several methods. The types of adsorption onto activated carbon were also studied with adsorption isotherms.

Hayashi *et al*. (2000) prepared activated carbon from corncob by $ZnCl_2$, H_3PO_4, K_2CO_3, Na_2CO_3, KOH, and NaOH. The results indicated that the maximum surface areas of the activated carbons prepared by $ZnCl_2$ activation and by H_3PO_4 activation observed at 600°C were as large as values for commercial activated carbons. The micropores were well developed. In contrast, the maximum surface areas of activated carbons prepared with alkali metal compounds occurred at 800°C and was generally larger than the surface areas of the commercial activated carbons. However, the mesopore volumes were seen to increase with carbonization temperature.

Vernersson *et al*. (2002) prepared activated carbon from canes in Arundo donax by H_3PO_4 activation under a self-generated atmosphere. The influence of the carbonization temperature in the range of 400–550 °C and of the weight ratio phosphoric acid to precursor (R = 1:5–2:5) on the developed porous structure of resulting carbons was studied for 1 hour of carbonization time. Under selected conditions (R = 2, T = 500 °C) and carbonization times shorter than 1 hour led to carbons with relatively larger surface area (1300 m^2/g) and smaller mean pore radius. Activated carbons obtained under flowing N_2 possessed predominant microporous structures and larger ash contents than the samples derived in the self-generated atmosphere.

Salame and Bandosz (2003) studied the adsorption of phenol onto two activated carbons of wood origin manufactured by Westvaco. The first carbon sample (WVA 1100) was obtained by using H_3PO_4 activation at 900 K and was referred to as W. The second carbon sample (UMC) was a developmental adsorbent manufactured by further KOH activation of WVA 1100 at 1300 K and was referred to as U. Both

carbons were oxidized with ammonium persulfate. The result showed that phenol adsorption from solution on carbons with acidic pH depends on both the porosity and surface chemistry of the carbons. Oxidation resulted in a decrease in the uptake of phenol, which was linked to a decrease in the average energy of adsorption sites. This decrease was a combination of two factors. The first factor was related to a decrease in the micropore volume as a result of destruction of thin pore walls. The second factor was an increase in the number of acidic groups on the carbon surface. Furthermore, phenol adsorption showed a strong dependence on the number of carboxylic groups due to two factors: (1) phenol reacts with carboxylic groups on the carbon surface, forming an ester bond and (2) carboxylic groups on the carbon surface remove the π-electron from the activated carbon aromatic ring matrix, causing a decrease in the strength of interactions between the benzene ring of phenol and the carbon's basal planes, which decreases the uptake of phenol.

Yang and Lua (2003) prepared activated carbon in granular from pistachio-nut shells by chemical activation with KOH. It was found that the porosity of the carbons was highly dependent on the preparation conditions such as the chemical impregnation ratio, the activation temperature, and the activation hold time. The best conditions for preparing activated carbons with high-surface area and pore volume by char impregnation were an impregnation ratio of 0.5 (KOH to raw material on mass basis), an activation hold time of 3 h, and an activation temperature of 800 °C. With these experimental conditions, the activated carbon with a BET surface area of the 2259.4 m^2/g and a total pore volume of the 1.10 cm^3/g were obtained. Furthermore, too high activation temperature and impregnation ratio resulted in the burn-off of carbon structures and widening of micropores to mesopores and macropores.

Budinova *et al*. (2006) prepared activated carbon from woody biomass birch by using various activation procedures: a) treatment with H_3PO_4 and pyrolysis at 600°C in inert atmosphere, b) the same as in (a) followed by steam activation at the same temperature and c) treatment with H_3PO_4 and direct pyrolysis in a steam of water vapour at 700 °C. The results indicated that the surface area and the porosity of the activated carbons were strongly dependent on the treatment after impregnation

with H_3PO_4. Activated carbon, prepared by impregnation with H_3PO_4 followed by steam pyrolysis (steam activation) had highly developed porous structure and the largest surface area among all prepared carbons (iodine number 1280 mg/g and BET surface area 1360 m^2/g). The adsorption followed Langmuir isotherms and the adsorption capacity for Hg(II) at 293 K was 160 mg/g.

Ucer *et al*. (2006) studied the adsorption of Cu (II), Cd (II), Zn (II), Mn (II), and Fe (III) ions onto tannic acid immobilised activated carbon. The results indicated that adsorption of the toxic metal ions depending on pH, contact time, carbon dosage, adsorption capacity, and adsorption isotherms. The adsorption data was correlated to Langmuir and Freundlich isotherm for each metal ion and the data fitted better to the Langmuir isotherm model.

Chandra *et al*. (2007) studied the adsorption equilibrium of methylene blue from aqueous solutions onto activated carbon. It was prepared from durian shell using chemical activation method with KOH. The activation was conducted at 673.15 K for 1 hour with mass ratio of chemical activating agent to durian shell 1:2. The Langmuir and Freundlich isotherm model were used to describe the equilibria data. For the adsorption equilibrium, it was found that Langmuir model could represent the data well compared with Freundlich isotherm.

Dincer *et al*. (2007) studied the adsorption of the dye from aqueous solution onto granular activated carbon (GAC) and coal-based bottom ash (CBBA). The results indicated that the capability of dye adsorption onto CBBA was higher than GAC. The extent of dye removal increased with decreased initial concentration of the dye and also increased with increased contact time. However, the adsorption of Vertigo Navy Marine (VNM) onto GAC occurred faster and reached higher equilibrium levels as compared to the CBBA. The GAC had a higher adsorption capacity than CBBA at lower and higher VNM concentrations.

Hameed *et al*. (2007) prepared activated carbon from bamboo in Malaysia by chemical activation with KOH and CO_2 as the activating agents at 850°C for 2 hours. It was found that the equilibrium data for methylene blue adsorption was well fitted to the Langmuir equation, with maximum monolayer adsorption capacity of 454.2 mg/g.

Chan *et al*. (2008) studied the adsorption of acid dye onto activated carbons which produced by thermal activation of bamboo with H_3PO_4. Two acid dyes with different molecular sizes were used, namely Acid Yellow 117 (AY117) and Acid Blue 25 (AB25). It was found that the high surface area carbon showed nearly three times higher adsorption capacity for small dye molecule (AB25) than the commercial carbon (F400) due to the potential of high BET surface area microporous activated carbon. For AY117, it has similar capacity as F400. However, the low surface area carbon showed poor adsorption for both dyes. Both surface area and porosity of the carbon played an important role in the adsorption of the dyes.

Fierro *et al*. (2008) studied the adsorption of phenol onto activated carbons. Six types of activated carbons were used half of them were commercial and other. Three were obtained from Kraft lignin chemically activated with NaOH, KOH or H_3PO_4. The results indicated that the high surface areas of activated carbons prepared from chemical activation of Kraft lignin with NaOH, KOH or H_3PO_4, were highly microporous materials between 940 and 2340 m^2/g. The high phenol adsorption capacities of activated carbons which prepared by activation of Kraft lignin with NaOH and KOH were 238 and 213 mg/g, respectively. However, the phenol uptake was found to depend not only on the micropore volume and the ratio of acid to basic groups but also on the total number of basic and carbonyl groups. Freundlich, Langmuir and Tempkin equations were tested for modelling the adsorption isotherms at equilibrium and it was concluded that Langmuir model fitted adequately with the experimental data.

Hameed and Rahman (2008) studied the adsorption of phenol from aqueous solution onto activated carbon which was prepared from rattan sawdust (ACR). Equilibrium studies were conducted in the range of 25–200 mg/L initial phenol concentrations, 3–10 solution pH and at temperature of 30°C. The experimental data were analyzed by the Langmuir, Freundlich, Temkin and Dubinin–Radushkevich isotherm models. The obtained results showed that the solution pH played a significant role in influencing the capacity of adsorbent towards phenol molecules. A decrease in the pH of solutions led to a significant increase in the adsorption capacity of activated carbon. The ACR possessed a high adsorption capacity to remove phenol. In addition, equilibrium data fitted well to the Langmuir model with a maximum adsorption capacity of 149.25 mg/g. The dimensionless separation factor R_L revealed the favorable nature of isotherm of the phenol-activated carbon system.

Jibril *et al*. (2008) prepared activated carbon from palm stems (Phoenix dactylifera) at Nizwa region. They were carbonized at different temperatures (400–600 °C) to investigate the effects of their impregnation with aqueous solution of either H_3PO_4 (85 wt %) or KOH (3 wt %). In the presence of either H_3PO_4 or KOH, the layer of fibrous cellular structure was destroyed at all temperatures with corresponding to the generation of porous surfaces. Generally, H_3PO_4 treated precursor yielded larger number of more regular pores. Surface area of 1100 m^2/g was obtained from the sample carbonized at 500 °C. On the other hand, KOH impregnation led to more heteroporous surface.

Klijanienko *et al*. (2008) prepared activated carbon with well-developed mesoporosity from oak and birch by H_3PO_4 promoted activation in a steam atmosphere. It was demonstrated that increasing impregnation ratio favors the development of micropores and small mesopores of 2–5 nm, whereas the soaking time promotes the creation of large mesopores with a width of 10–50 nm. The birch, containing lower amount of lignin compared to oak, is more susceptible to develop mesoporosity on activation with H_3PO_4.

Tseng *et al*. (2008) prepared activated carbon from corncob by the KOH activation method. It was found that the element residue ratio of C, H, and O physical activation caused a large drop in the residue ratio of H and O while KOH etching in chars shown high O and H residual ratios, attributabled to the main consumption of C in this activation method. The information of chemical composition and surface oxygen functional groups on activated carbons proposes the reaction mechanism of KOH activation. From the adsorption study for five organics with molecular weights varying from 129 to 466 g/mol, the specific adsorption capacity of activated carbons for organics were independent of their specific surface area.

MATERIALS AND METHODS

Materials

1. Apparatus

1.1 Atomic absorption spectrophotometer (AAS : Perkin Elmer, AA analyst 800)

1.2 UV-Vis spectrophotometer (Perkin Elmer, Lambda 35)

1.3 Fourier transform infrared spectrometer (FT-IR : Perkin Elmer, System 2000)

1.4 Scanning electron microscope (SEM : LEO, 1450VP)

1.5 High performance liquid chromatography (HPLC : Varian, Prostar 210)

1.6 Reverse phase column (Supelco C-610H : sulfonate polystylene/divinylbenzene)

1.7 Ultrasonic bath (Branson 1210)

1.8 Microbalance (Mattler Toledo, AL 204)

1.9 Rotary shaker (Clifton, NE5-2BD CE)

1.10 pH meter (Inolab level 1, 8F93)

1.11 Muffle furnace (Lenton thermal)

1.12 Oven (Precision,16EG)

1.13 Sieve mesh 150 μm

2. Reagents

2.1 Phosphoric acid (H_3PO_4, AR. grade, Ajax Finechem, Aukland, New Zealand)

2.2 Potassium hydroxide (KOH, AR. grade, Ajax Finechem, Aukland, New Zealand)

2.3 Iodine (I_2, AR. grade, Univar, Seven Hills, Australia)

2.4 Methylene blue ($C_{16}H_{18}N_3ClS$, AR. grade, Fluka, Steinheim, Switzerland)

2.5 Phenol (C_6H_6O, AR. grade, Carlo Erba, Rodano, Milan, Italy)

2.6 Cadmium standard for atomic absorption 1000 mg/l ($CdCl_2.2.5H_2O$ in diluted hydrochloric acid, AA. Grade, Carlo Erba, Rodano, Milan, Italy)

2.7 Sodium acetate (CH_3COONa, AR. grade, Merck, Darmstadt, Germany)

2.8 Sodium carbonate (Na_2CO_3, AR. grade, Merck, Darmstadt, Germany)

2.9 Sodium thiosulfate ($Na_2S_2O_3.5H_2O$, AR. grade, Merck, Darmstadt, Germany)

2.10 Potassium bromate ($KBrO_3$, AR. grade, BDH, Poole, United Kingdom)

2.11 Potassium bromide (KBr, AR. grade, Merck, Darmstadt, Germany)

2.12 Potassium iodate (KIO_3, AR. grade, Univar, United State of America)

2.13 Potassium iodide (KI, AR. grade, VWR International, England)

2.14 Potassium hydrogen phosphate (K_2HPO_4, AR. grade, Merck, Darmstadt, Germany)

2.15 Acetone (C_3H_6O, Lab. grade, Lab Scan, Bangkok, Thailand)

2.16 Diethyl ether ($C_4H_{10}O$, AR. grade, Merck, Darmstadt, Germany)

2.17 Hexane (C_6H_{12}, AR. Grade, Mallinckrodt, United State of America)

2.18 Hydrochloric acid (HCl, AR. grade, Carlo Erba, Rodano, Milan, Italy)

2.19 Starch ($(C_6H_{10}O_5)_n$, AR. Grade, Merck, Darmstadt, Germany)

2.20 Acetic acid (CH_3COOH, AR. grade, Merck, Darmstadt, Germany)

2.21 Boric acid ($B(OH)_3$, AR. grade, Fisher, Leicestershire, United Kingdom)

2.22 Butyric acid ($C_4H_8O_2$, AR. grade, Merck, Darmstadt, Germany)

2.23 Citric acid ($C_6H_8O_7$, AR. grade, Univar, Seven Hills, Australia)

2.24 Formic acid (CH_2O_2, AR. grade, Fluka, Steinheim, Switzerland)

2.25 Isobutyric acid ($C_4H_8O_2$, AR. grade, Fluka, Steinheim, Switzerland)

2.26 Lactic acid ($C_3H_6O_3$, AR. grade, Merck, Darmstadt, Germany)

2.27 Malic acid($C_4H_6O_5$, AR. grade, Merck, Darmstadt, Germany)

2.28 Propionic acid ($C_3H_6O_2$, AR. Grade, Darmstadt, Germany)

2.29 Tartaric acid ($C_4H_6O_6$, AR. grade, Fluka, Steinheim, Switzerland)

2.30 Commercial activated carbon (C, AR. grade, Fluka, Steinheim, Switzerland)

2.31 Bamboo charcoal powder (C, Lab. Grade, Bunton, Bangkok, Thailand)

Methods

1. Preparation of bamboo charcoal

Raw materials (bamboos) used for preparation of charcoal which were cultivated at Kanchanaburi province were washed, dried and crushed to desired size (1-2 mm.). It was then carbonized at 450°C by MES 20 as shown in Figure 13. The carbonized materials were sieved to 150 μm sizes and dried at 120°C for 1 hour. The charcoal was kept in a desiccator.

Figure 13 The MES 20 for carbonization.

2. Preparation of activated carbon

2.1 Activation by phosphoric acid (H_3PO_4)

The bamboo charcoal was activated by refluxed at 250°C using 1 g of charcoal per 4 ml of H_3PO_4 for 4 hour. The concentrations of H_3PO_4 were 20, 40, 60 and 85 %v/v. The activated carbon was washed with deionized water until the pH of washing solution was approximately neutral and dried at 120°C for 1 hour. Then, the prepared activated carbon was characterized by the adsorption capacity towards iodine using the standard test method for determination of iodine number of activated carbon (American Society for Testing and Materials [ASTM], 1999) for optimal concentrations of H_3PO_4 for the activation.

The bamboo charcoal was activated by the same procedure as previously described except using H_3PO_4 at the optimal concentration. The prepared activated carbon was characterized by the adsorption capacity towards iodine using the standard test method for determination of iodine number of activated carbon (ASTM, 1999) for optimal concentrations of H_3PO_4 for the activation.

2.2 Activation by potassium hydroxide (KOH)

The bamboo charcoal was activated by reflux at 250°C using 1 g of char per 4 ml of KOH for 4 hour. The concentrations of KOH were 20, 40, 60 and 80 %w/v. The activated carbon was washed with deionized water until the pH of washing solution was approximately 7 and dried at 120°C for 1 hour. The prepared activated carbon was characterized by the adsorption capacity towards iodine using the standard test method for determination of iodine number of activated carbon (ASTM, 1999) for optimal concentrations of KOH for the activation.

The bamboo charcoal was activated by the same procedure as previously described except using KOH at the optimal concentration. The preparation activated carbon was characterized by the adsorption capacity towards iodine determined using the standard test method for determination of iodine number of activated carbon (ASTM, 1999) for optimal times of activation.

3. Proximate analysis properties of charcoal and activated carbon

3.1 Moisture of activated carbon

The activated carbon samples were determined using standard test method for moisture in activated carbon (ASTM, 2004) which is shown in Appendix A. The example of calculation is also shown in Appendix H.

3.2 Ash content of activated carbon

The activated carbon samples were determined using standard test method for ash content of activated carbon (ASTM, 1999) which is shown in Appendix B. The example of calculation is also shown in Appendix H.

3.3 Volatile matter content of activated carbon

The activated carbon samples were determined using standard test method for volatile matter content of activated carbon (ASTM, 2003) which is shown in Appendix C. The example of calculation is also shown in Appendix H.

3.4 Fixed carbon

The fixed carbon of activated carbon samples were calculated from the following equation.

% fixed carbon = 100 – (% ash content + % volatile matter content) ------ (14)

4. Adsorption properties of charcoal and activated carbon

4.1 Iodine adsorption

4.1.1 Iodine number

The iodine number of all samples and bamboo charcoal were determined using the standard test method for determination of iodine number of activated carbon (ASTM, 1999) which is shown in Appendix D. The example of calculation is also shown in Appendix I.

4.1.2 Adsorption Isotherms

The activated carbon samples from each preparation method and bamboo charcoal that had high iodine number were chosen for construct adsorption isotherms that were performed in a set of 50 ml flasks, where solutions of iodine (25 ml) with different initial concentrations (0.005-0.025 N) were placed in these flasks. Equal mass of 0.5 g of activated carbon of particle size 150 μm were added to iodine solutions at pH 7 and kept in an isothermal shaker of 200 rpm at 30±1°C for 25 minutes to reach equilibrium of the solid–solution mixture. A similar procedure was followed for another set of flask containing the same concentration of iodine without activated carbon, which was used as a blank. The flasks were then removed from the shaker and the samples were filtered prior to analysis. The final concentrations of iodine in the solutions were titrated with sodium thiosulfate solutions which observed from equation (15).

$$2\ Na_2S_2O_3\ (aq) + I_2 \rightarrow Na_2S_4O_6\ (aq) + 2NaI\ (aq) \qquad \text{------ (15)}$$

Afterwards, the Langmuir plots of C_e/ q_e against C_e yields straight line for the adsorption of iodine onto the activated carbon. The Langmuir constant Q_o and b are obtained from slope and intercept of the respective plot. The Freundlich plots of log q_e versus log C_e yields straight line for the adsorption of iodine onto the activated carbon. The Freundlich constant n and K_F are obtained from the slope and intercept of respective plot. The applicability of isotherm models of the adsorption was compared by judging the correlation coefficient (R^2) values.

4.2 Phenol adsorption

4.2.1 Phenol value

The activated carbon samples from each preparation method and bamboo charcoal that had high iodine number were chosen for measuring phenol value that was determined according to standard test method for determination of

phenol value of activated carbon (American Water Works Association, 1995) which is shown in Appendix E. The example of calculation is also shown in Appendix I.

4.2.2 Adsorption isotherms

The best phenol value of activated carbon samples from 4.2.1 were chosen for measuring adsorption isotherms that were performed in a set of 50 ml flasks, where solutions of phenol (25 ml) with different initial concentrations (10-50 mgl^{-1}) were placed in these flasks. Equal mass of 0.3 g of activated carbon of particle size 150 μm were added to phenol solutions at pH 7 and kept in an isothermal shaker of 200 rpm at 30±1°C for 5 hours to reach equilibrium of the solid–solution mixture. A similar procedure was followed for another set of flask containing the same concentration of phenol without activated carbon, which was used as a blank. The flasks were then removed from the shaker and the samples were filtered prior to analysis. The final concentrations of phenol in the solutions were measured by UV-Vis spectrophotometer at 270 nm wavelength.

Afterwards, the Langmuir plots of C_e/ q_e against C_e yields straight line for the adsorption of phenol onto the activated carbon. The Langmuir constant Q_o and b are obtained from the slope and intercept of respective plot. The Freundlich plots of log q_e versus log C_e yields straight line for the adsorption of phenol onto the activated carbon. The Freundlich constant n and K_F are obtained from the slope and intercept of respective plot. The applicability of isotherm models of the adsorption was compared by judging the correlation coefficient (R^2) values.

4.3 Methylene blue adsorption

4.3.1 Methylene blue value

The activated carbon samples from each preparation method and bamboo charcoal that had high iodine number were chosen for measuring methylene blue value. Activated carbon was first dried at 120°C for 1 hour. 0.04 g of activated

carbon of particle size 150 µm were added to 25 ml of a 25 ppm of dye solution and kept in an isothermal shaker of 200 rpm at 30±1°C for 2 hours. The flasks were then removed from the shaker and the samples were filtered prior to analysis. The final concentrations of dye in the solutions were measured by UV-Vis spectrophotometer at 665 nm wavelength.

4.3.2 Adsorption isotherms

The methylene blue value of activated carbon samples from 4.3.1 were chosen for measuring adsorption isotherms that were performed in a set of 50 ml flasks, where solutions of dye (25 ml) with different initial concentrations (75-175 mgl^{-1}) were placed in these flasks. 0.04 g of activated carbon with particle size of 150 µm were added to dye solutions at pH 7 and kept in an isothermal shaker of 200 rpm at 30±1°C for 6 hours to reach equilibrium of the solid–solution mixtures. A similar procedure was followed for another set of flask containing the same concentration of dye without activated carbon which was used as a blank. The flasks were then removed from a shaker and the samples were filtered prior to analysis. The final concentrations of dye in the solutions were measured by UV-Vis spectrophotometer at 665 nm wavelength.

Afterwards, the Langmuir plots of C_e/ q_e against C_e yields straight line for the adsorption of Methylene blue onto the activated carbon. The Langmuir constant Q_o and b are obtained from the slope and intercept of respective plot. The Freundlich plots of log q_e versus log C_e yields straight line for the adsorption of Methylene blue onto the activated carbon. The Freundlich constant n and K_F are obtained from the slope and intercept of respective plot. The applicability of isotherm models of the adsorption was compared by judging the correlation coefficient (R^2) values.

4.4 Cadmium (II) adsorption

4.4.1 Cd (II) value

The activated carbon samples from each preparation method and bamboo charcoal that had high iodine number were chosen for measuring Cd (II) value. Activated carbon was first dried at 120°C for 1 hour. 1 g of activated carbon of particle size 150 µm were added to 25 ml of a 25 ppm of Cd (II) solution and kept in an isothermal shaker of 200 rpm at 30±1°C for 4 hours. The flasks were then removed from the shaker and the samples were filtered prior to analysis. The final concentrations of Cd (II) in the solutions were measured by AAS.

4.4.2 Adsorption Isotherms

The activated carbon samples from each preparation method and bamboo charcoal that had high iodine number were chosen for measuring adsorption isotherms that were performed in a set of 50 ml flasks, where solutions of Cd (II) (25 ml) with different initial concentrations (5-25 mgl^{-1}) were placed in these flasks. Equal mass of 1 g of activated carbon of particle size 150 µm were added to Cd (II) solutions at pH 7 and kept in an isothermal shaker of 200 rpm at 30±1°C for 4 hours to reach equilibrium of the solid–solution mixture. A similar procedure was followed for another set of flash containing the same concentration of Cd (II) without activated carbon, which was used as a blank. The flasks were then removed from the shaker and the samples were filtered prior to analysis. The final concentrations of Cd (II) in the solutions were measured by AAS**.**

Afterwards, the Langmuir plots of C_e/ q_e against C_e yields straight line for the adsorption of Cd (II) onto the activated carbon. The Langmuir constant Q_o and b are obtained from the slope and intercept of respective plot. The Freundlich plots of log q_e versus log C_e yields straight line for the adsorption of Cd (II) onto the activated carbon. The Freundlich constant n and K_F are obtained from

the slope and intercept of respective plot. The applicability of isotherm models of the adsorption was compared by judging the correlation coefficient (R^2) values.

5. Analyses of structure and functional groups of charcoal and activated carbon

5.1 Scanning electron micrograph analyses

The activated carbon samples from each preparation method and bamboo charcoal that had high iodine number were chosen for measuring SEM analysis that was carried out for the carbon samples that was scattered on a stub and coated with gold to study the surface morphology and to verify the presence of porosity.

5.2 FT-IR spectroscopy

The activated carbon samples from each preparation method and bamboo charcoal that had high iodine number were chosen for analyzed using FT-IR spectroscopy to study the information on the surface chemistry of carbon samples. The samples were mixed with potassium bromide and the mixture was pressed into pellets to be used in the analysis.

6. Analysis of organic acids in wood vinegar from bamboos

6.1 Preparation of 0.1 %v/v phosphoric acid as a mobile phase

Concentrated phosphoric acid 1.2 ml was diluted and made up to 1000 ml by deionized water in a volumetric flask. After that 0.1%v/v phosphoric acid was filtrated and degased by ultra-sonic bath for 30 minute.

6.2 Preparation of wood vinegar

The wood vinegar was filtered and then extracted with mixed solvent between hexane and diethylether (2:1) at ratio of 1:1. The wood vinegar was distilled

at 90-95°C for 15-20 minute and then filtrated with 0.45 μm HPLC filters. The other samples were flowed into the solid phase extraction at vacuum.

6.3 Analysis of organic acid in wood vinegar

HPLC analysis of organic acids of wood vinegar was performed using Varian Prostar 210 equipment with a quaternary pump (Varian Prostar 210/215). Detection was performed using a UV-Vis detector (Varian Prostar model 320). The column was a reverse phase (sulfonate polystyrene/divinylbenzene). The chromatographic conditions for the wood vinegars were: 0.5 ml/min flow rate at room temperature, 20 μl injection volume, and eluents: 0.1%v/v phosphoric acid. Detection was by means of measurement of UV absorption at 210 nm. Identification of the organic acid was carried out by comparing their retention times and UV spectra to those of standard. Quantitation was performed by the external standard method.

RESULTS AND DISCUSSIONS

1. Preparation of bamboo charcoal

The bamboo charcoal was prepared by kiln at 450°C. The final product was a black solid with fragileness and had a charcoal yields as shown in Table 9.

Table 9 The charcoal yield from carbonization process.

Types of bamboo	Age of bamboo (year)	Yield $_{charcoal}$ (%)
BAW	1	57.92
	2	54.75
	3	51.64
GO	1	47.13
	2	52.89
	3	51.26
GV	1	50.58
	2	48.45
	3	49.39

Table 9 showed that the ages and types of bamboos had small influence on the percentage of charcoal yield. Since the composition of each bamboo was not different (Anapanurak *et al*., 2007), the carbonization process provided a quite similar amount of charcoal.

2. Preparation of activated carbon

2.1 Activation by H_3PO_4

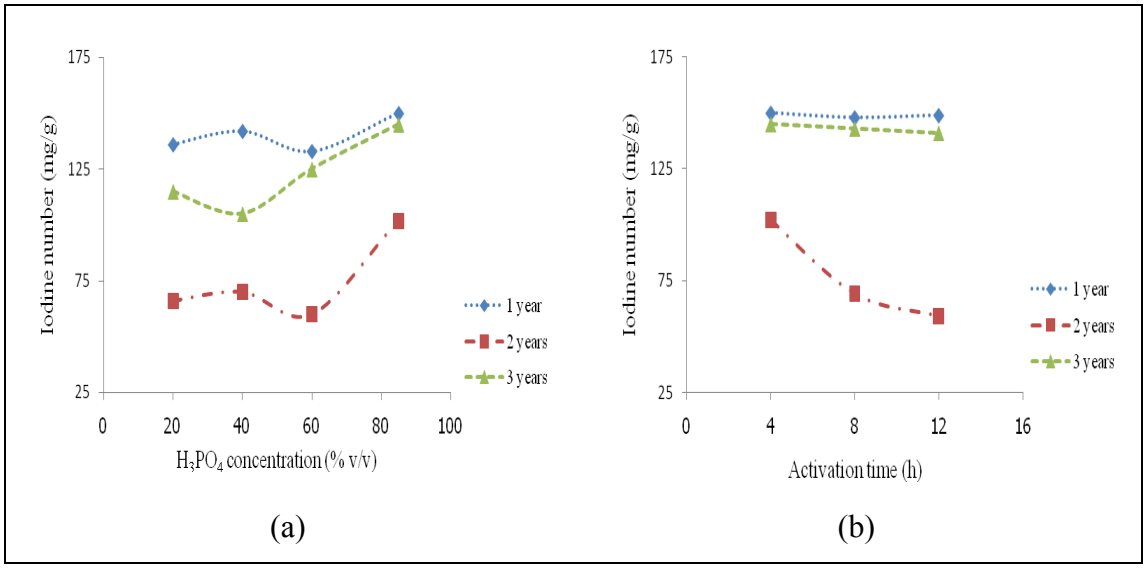


Figure 14 Iodine number of BAWP of all three ages (a) when activated by various concentration of H_3PO_4 for 4 hours (b) when activated by 85 %v/v H_3PO_4 at different activation time.

Figure 14a illustrated that 85% v/v H_3PO_4 concentration was the optimal concentration for activating the BAW of all three ages. It could be assumed that H_3PO_4 had a high opportunity to react with charcoal when its concentration was increased, which finally caused the large number of micropores. Therefore, it gave the maximum iodine number for each BAW's age 1-3 years as 150, 102 and 145 mg/g, respectively. This concentration was chosen for continually searching for optimal activation time.

Figure 14b illustrated that the activation time for 4 hours was an optimal time for activating the BAW of all three ages. It could be implied that the activation with H_3PO_4 led to a larger pore size when the activation time extended. Thus, the adsorption of iodine onto activated carbon was decreased. It was expected that the

reaction between H_3PO_4 and carbon of charcoal was increased with increasing time. Therefore, the pore size of activated carbon was larger. The highest iodine number for each BAWP's age 1-3 years were 150, 102 and 145 mg/g, respectively. Therefore, these conditions were chosen for preparing activated carbon in this work.

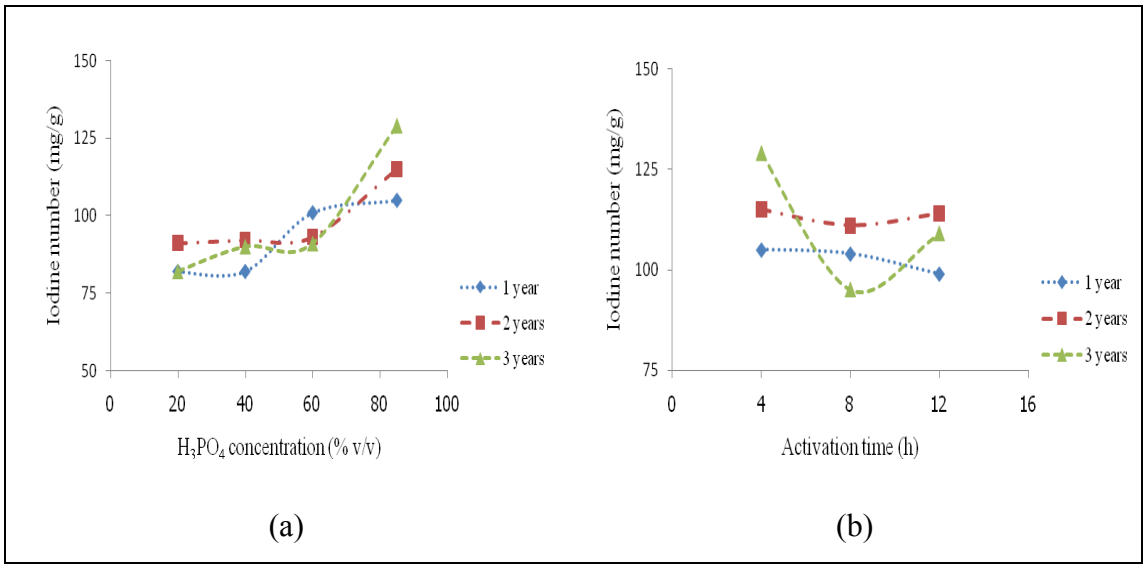


Figure 15 Iodine number of GOP of all three ages (a) when activated by various concentration of H_3PO_4 for 4 hours (b) when activated by 85 %v/v H_3PO_4 at different activation time.

Figure 15a illustrated that 85% v/v H_3PO_4 concentration was the optimal concentration for activating the GO of all three ages. It could be assumed that H_3PO_4 had a high opportunity to react with charcoal when its concentration was increased, which finally caused the large number of micropores. Therefore, it gave the maximum iodine number for each GOP's age 1-3 years were 105, 115 and 129 mg/g, respectively. Thus this concentration was chosen for continually searching optimal activation time.

Figure 15b illustrated that the activated time for 4 hours was an optimal time for activating the GO of all three ages. It could be implied that the activation with H_3PO_4 led to a larger pore size when the activation time extended. Thus, the

adsorption of iodine onto activated carbon decreased. The highest iodine number for each GO's age 1-3 years were 105, 115 and 129 mg/g, respectively. Therefore, these conditions were chosen for preparing activated carbon in this work.

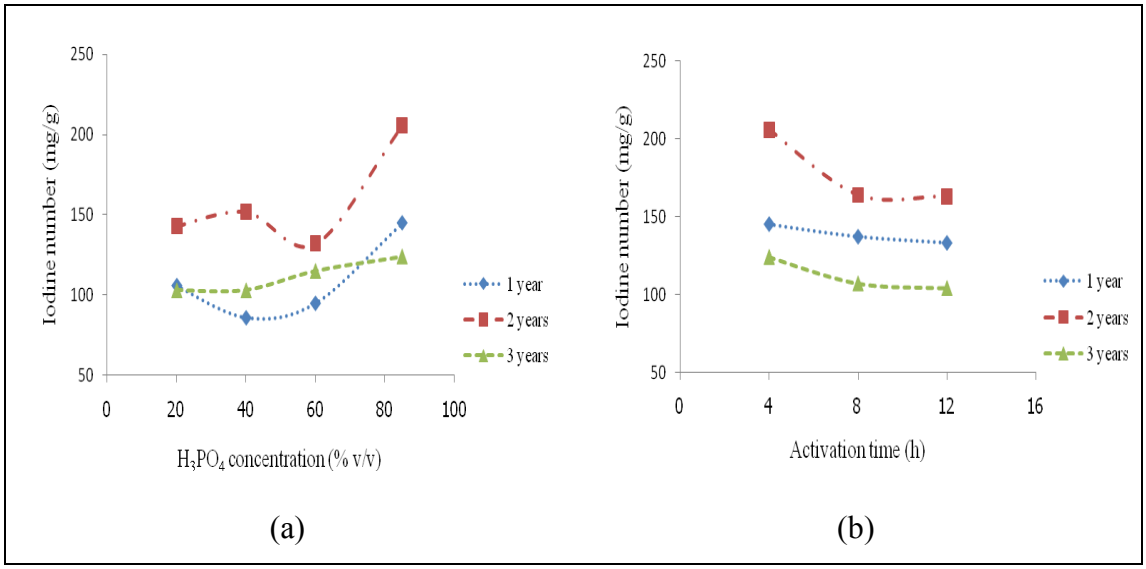


Figure 16 Iodine number of GVP of all three ages (a) when activated by various concentration of H_3PO_4 for 4 hours (b) when activated by 85 %v/v H_3PO_4 at different activation time.

Figure 16a illustrated that the 85% v/v H_3PO_4 concentration was the optimal concentration for activating the GV of all three ages. It could be assumed that H_3PO_4 had a high opportunity to react with charcoal when its concentration was increased, which finally caused the large number of micropores. Therefore, it gave the maximum iodine number for each GVP's age 1-3 years were 145, 206 and 124 mg/g, respectively. Thus this concentration was chosen for continually searching for optimal activation time.

Figure 16b illustrated that the activation time for 4 hours was the optimal time for activating the GV of all three ages. It could be implied that the activation with H_3PO_4 led to a larger pore size when the activation time extended. Thus, the adsorption of iodine onto activated carbon was decreased. The highest iodine number

for each GV's age 1-3 years were 145, 206 and 124 mg/g, respectively. Therefore, these conditions were chosen for preparing activated carbon in this work.

2.2 Activation by KOH

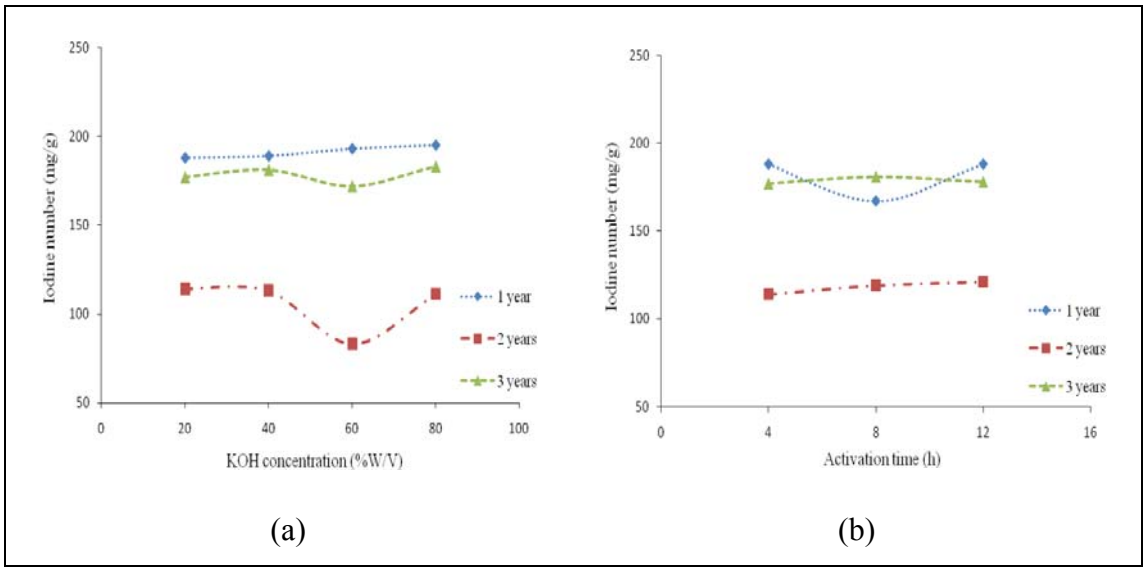


Figure 17 Iodine number of BAWK of all three ages (a) when activated by various concentration of KOH for 4 hours (b) when activated by 20 %w/v KOH at different activation time.

Figure 17a illustrated that the 20 %w/v KOH concentration was the optimal concentration for activating the BAW of all three ages. From the similar iodine adsorption, it could be assumed that KOH created the similar number of micropores when its concentration was increased. Thus, this concentration was chosen for continually searching for optimal activation time.

Figure 17b illustrated that the activation time for 4 hours was the optimal time for activating the BAW of all three ages. It could be implied that the activation with KOH caused the same number of pores when the activation time extended. Hence, the iodine number of BAWC that activated for 4 hours was similar to the other

types of charcoal which were activated for 8 or 12 hours. Therefore, these conditions were chosen for preparing activated carbon in this work.

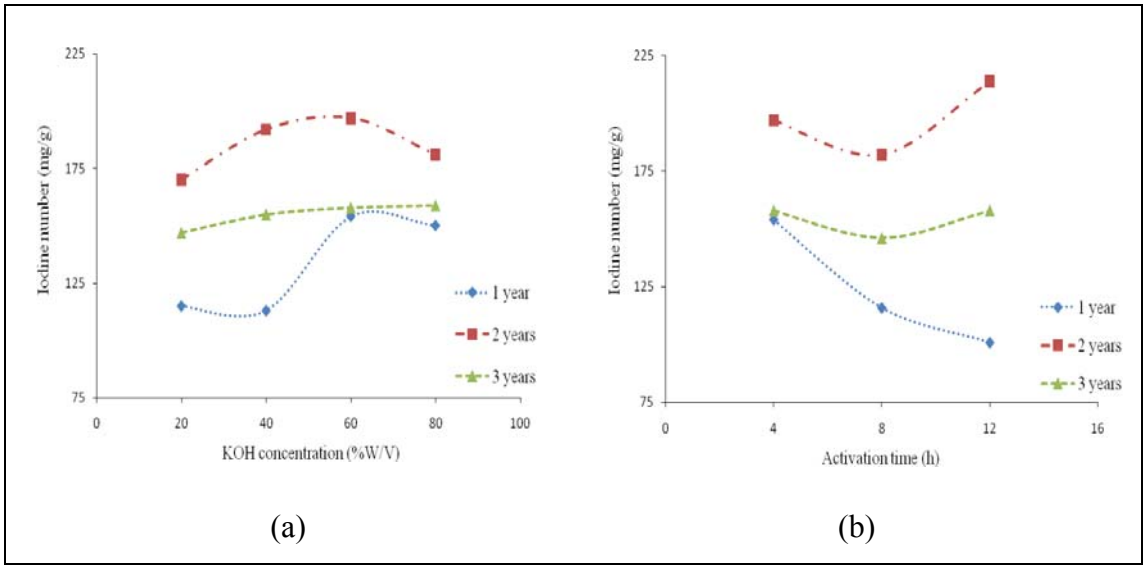
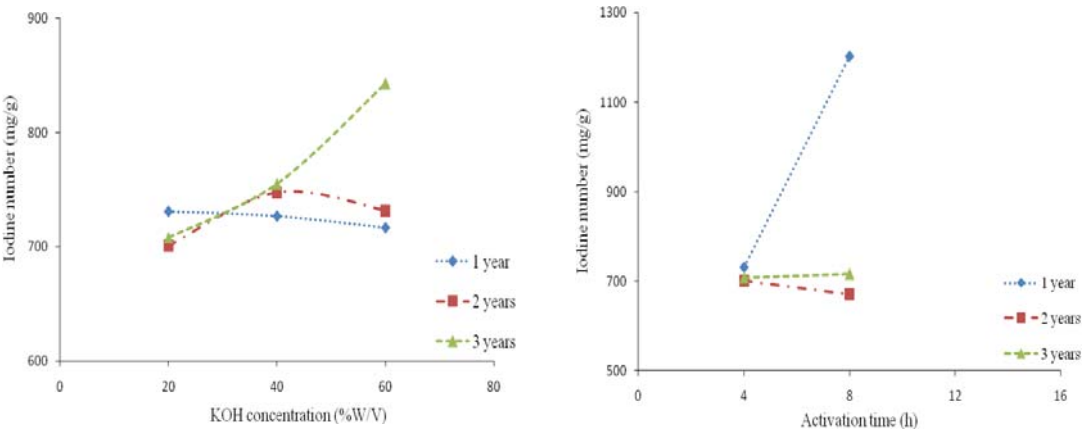


Figure 18 Iodine number of GOK of all three ages (a) when activated by various concentration of KOH for 4 hours (b) when activated by 60 %w/v KOH at different activation time.

Figure 18a illustrated that the 60 %w/v KOH concentration was an optimal concentration for activating the GO of all three ages. It could be assumed that 60 %w/v KOH had a high opportunity to react with charcoal which caused the high number of micropores. Moreover, it gave the maximum iodine number for each GO's age 1-3 years 154, 197 and 158 mg/g, respectively. Thus, this concentration was chosen for continually searching for optimal activation time.

Figure 18b illustrated that the activation time for 4 hours was the optimal time for activating the GO of all three ages. However, the variation of the activation time had a minor effect on the number of micropores because the iodine numbers of activated carbon for 4 hours did not differ from the others which activated at 8 and 12 hours of each GO's age 1-3 years. Therefore, these conditions were chosen for preparing activated carbon in this work.



(a) (b)

Figure 19 Iodine number of GVK of all three ages (a) when activated by various concentration of KOH for 4 hours (b) when activated by 20 %w/v KOH concentration at different activation time of one-year, 40 %w/v KOH concentration at different activation time of two-years, 60 %w/v KOH concentration at different activation time of three-years.

Figure 19a illustrated that GV charcoal which was activated by 20 %w/v KOH concentration of one-year, 40 %w/v KOH concentration of two-years and 60 %w/v KOH concentration of three-years were the optimal concentration for activating the GV charcoal. It could be assumed that these conditions had a high opportunity to react with charcoal finally caused the large number of micropores. Thus, the activated carbon activated with these conditions gave the maximum iodine number for each GV's age as 731, 748 and 843 mg/g, respectively. Thus these concentrations were chosen for continually searching for optimal activation time.

Figure 19b illustrated that the activated time for 4 hours (two-years and three-years) and 8 hours (one-year) was an optimal time for activating the GV since the activated carbon activated with optimal concentration for 4 and 8 hours gave the highest iodine number for each GV's age as 1202, 748 and 843mg/g, respectively. Therefore, these conditions were chosen for preparing activated carbon in this work.

3. Proximate analysis properties

Table 10 The proximate analysis properties of activated carbon.

Charcoal			Moisture (%)	Proximate analysis properties (Wt %)		
Type of bamboo	Age of bamboo	Chemical activation		Ash	Volatile matter	Fixed carbon
Fluka [a]			6.05	2.12	26.59	71.29
Bunton [b]			7.29	8.69	38.21	53.10
BAW	1 year	-	7.29	8.00	44.95	47.05
		H_3PO_4	4.81	6.37	37.35	56.28
		KOH	6.35	5.59	34.42	59.99
	2 years	-	8.32	11.26	46.31	42.43
		H_3PO_4	6.16	10.25	46.21	43.54
		KOH	6.63	9.87	45.18	44.95
	3 years	-	8.74	8.48	44.91	46.61
		H_3PO_4	8.28	4.98	39.99	55.03
		KOH	6.04	5.03	38.23	56.74

[a] Activated carbon was produced by Fluka Company.

[b] Bamboo charcoal was produced by Thai Agard Dee Bamboo Charcoal Products.

Table 10 (Continued)

Charcoal			Moisture (%)	Proximate analysis properties (Wt %)		
Type of bamboo	Age of bamboo	Chemical activation		Ash	Volatile matter	Fixed carbon
GO	1 year	-	4.81	11.7	48.12	40.18
		H_3PO_4	4.66	8.71	47.93	43.36
		KOH	7.04	7.16	44.98	47.86
	2 years	-	6.37	11.42	44.49	44.09
		H_3PO_4	5.27	8.32	40.79	50.89
		KOH	8.08	10.81	40.4	48.79
	3 years	-	5.84	7.05	44.13	48.82
		H_3PO_4	5.82	5.52	40.21	54.27
		KOH	9.36	6.7	43.82	49.48
GV	1 year	-	5.21	9.66	38.79	51.55
		H_3PO_4	6.81	7.12	37.95	54.93
		KOH	6.57	7.51	34.85	57.64
	2 years	-	6.46	8.54	39.22	52.24
		H_3PO_4	5.95	6.36	37.3	56.34
		KOH	7.62	8.36	34.79	56.85
	3 years	-	5.62	10.73	45.3	43.97
		H_3PO_4	7.68	8.64	44.8	46.56
		KOH	6.93	8.24	35.5	43.50

The results of proximate analysis properties are shown in Table 10. The ash and volatile of activated carbon were slightly decreased, compared to bamboo charcoal and Bunton. On the contrary, Fluka has the lowest amount of ash (2.12 wt%) and volatile (26.59 wt%) excepting for moisture, indicated that it had a few amount of organic compounds and volatile matters. It is expected that Fluka was prepared at higher temperature so the major organic compounds and volatile matters were decomposed at the high extent. In addition, Fluka has the highest fixed carbon. This is due to the same reason that mentioned above.

Fixed carbon of activated carbon slightly increased when compared to charcoal, because the activated carbon was activated at a low temperature (250°C) in which a few organic compounds and volatile matters were decomposed. However, fixed carbon was the value which displayed the amount of remaining carbon after the substraction of moisture, ash and volatile matters. Besides, it is very difficult to use the amount of fixed carbon as the indicator for the adsorption ability of the activated carbon. For example, Gurdal and Yalcm (2000) found that the adsorption ability decreased when the amount of fixed carbon increased up to 69%. Nevertheless, the adsorption ability would increase when the amount of fixed carbon increased more than 69% (Figure 20). Levy *et al*. (1997) found that the adsorption ability increased with increasing the amount of fixed carbon (Figure 21). The ability of the adsorbent relied on the porous and chemical structure of adsorbent surface more than the amount of fixed carbon.

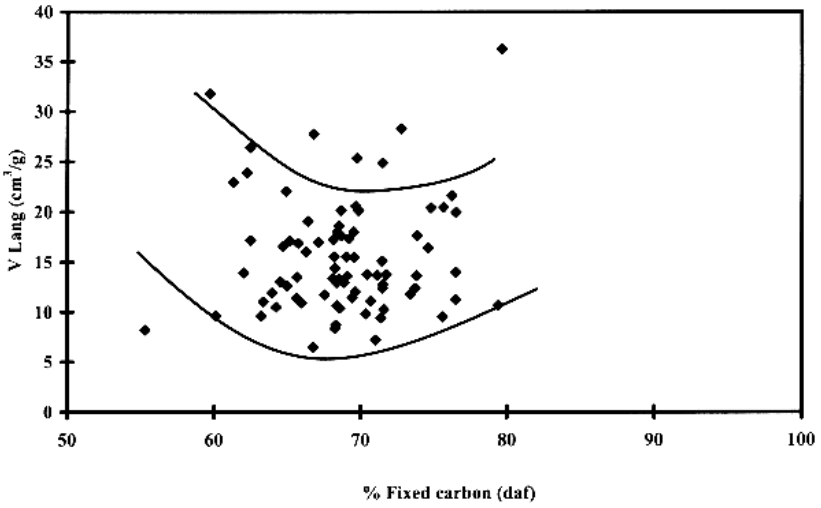


Figure 20 Relation between fixed carbon and Langmuir gas adsorption capacity for Zonguldak coals.

Source: Gürdal and Yalcm (2000)

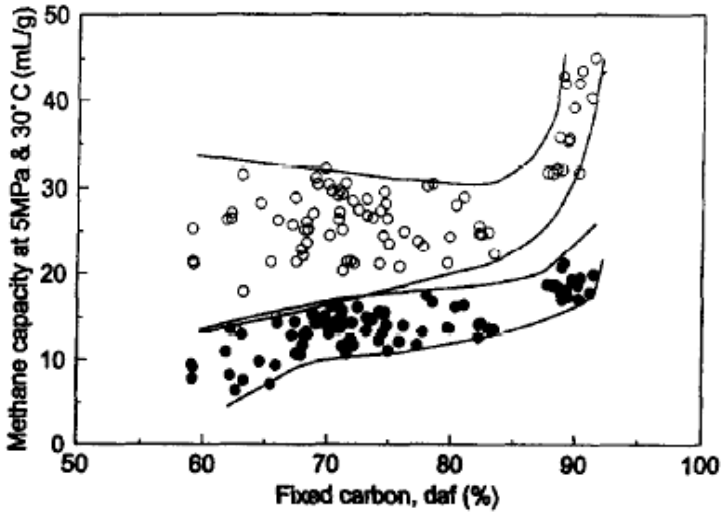


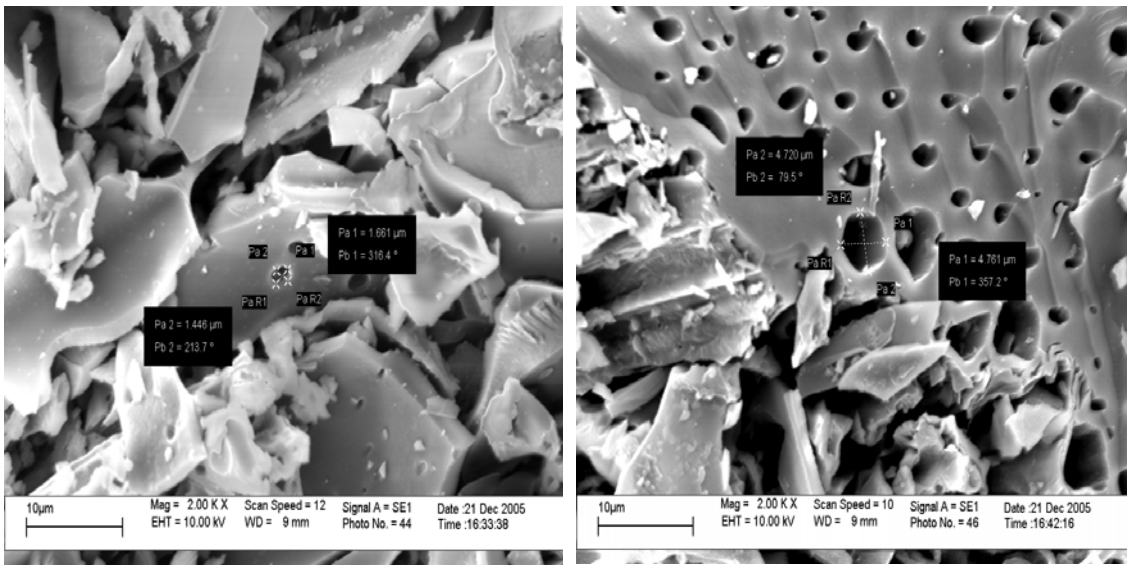
Figure 21 Relation between fixed carbon and Langmuir methane adsorption capacity when ● and ○ are methane adsorption capacity of activated carbon with moisture and without moisture, respectively.

Source: Levy *et al*. (1997)

4. Analyses of structure and functional group of charcoal and activated carbon

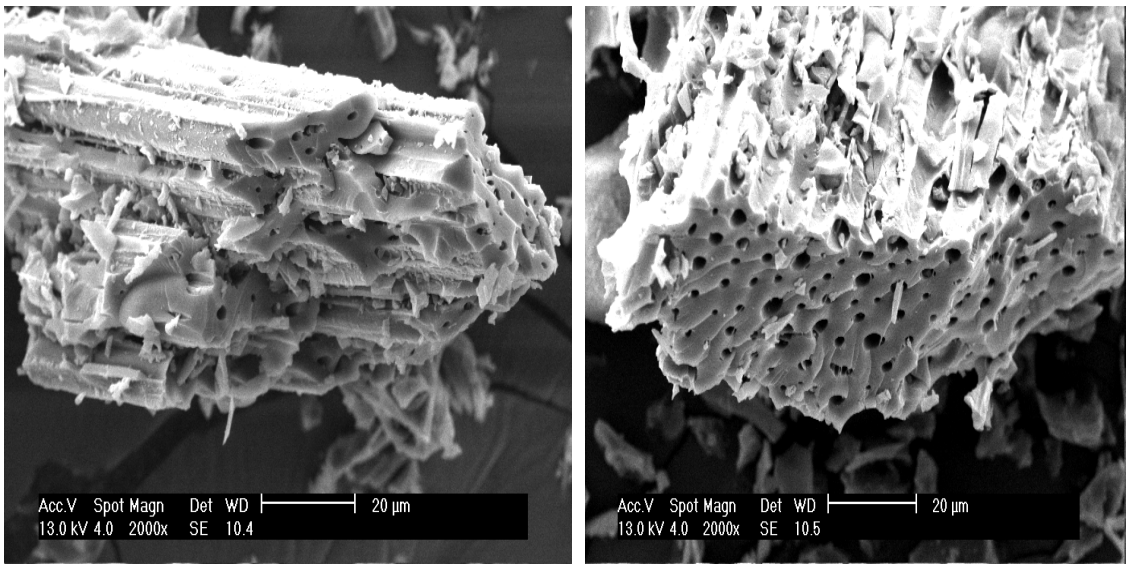
4.1 Scanning electron micrograph analyses

Pore structures and structural changes after chemical activation could be observed by SEM. Figure 22-25 showed SEM micrographs of Fluka, activated carbon and bamboo charcoal, respectively. It was obvious that the activated carbon surface exhibited various types of pores. The pore sizes of activated carbon were in the range of 1-10 μm. The activated carbon had a greater number of pores than bamboo charcoal due to the activation process used, which involved chemical activating agents of H_3PO_4 and KOH (Figure 22-24). Furthermore, Fluka and GVK 1 contained the highly number of pores which indicated relatively large surface areas for adsorption (Figure 22 and 25).



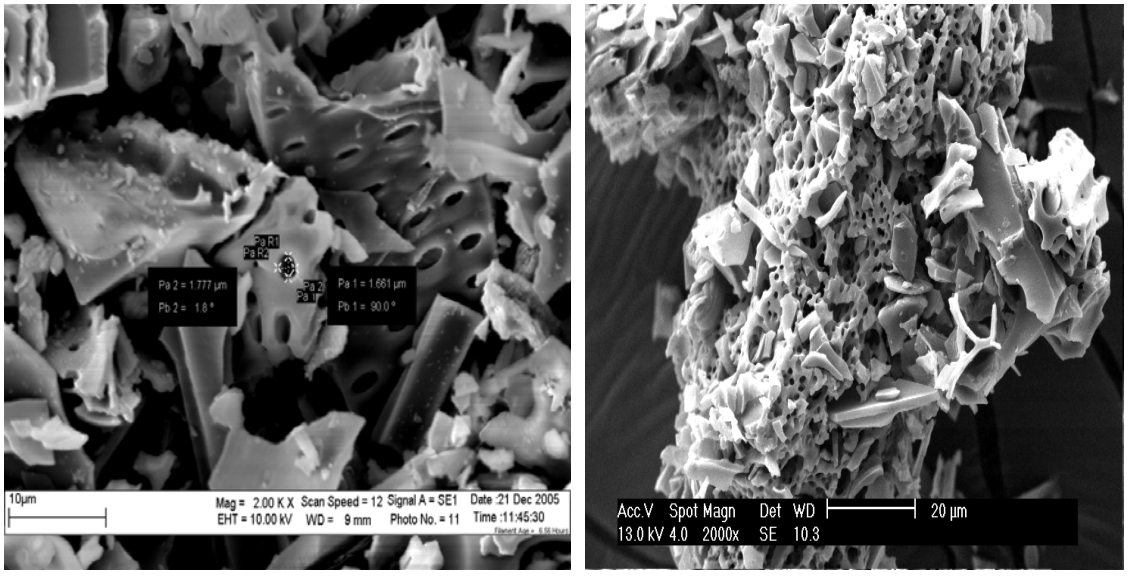
(a) (b)

Figure 22 SEM micrograph of (a) BAWC 3 and (b) BAWP 3 at 2000 x magnification.



(a) (b)

Figure 23 SEM micrograph of (a) GOC 1 and (b) GOP 1 at 2000 x magnification.



(a) (b)

Figure 24 SEM micrograph of (a) GVC 1 and (b) GVK 1 at 2000 x magnification.

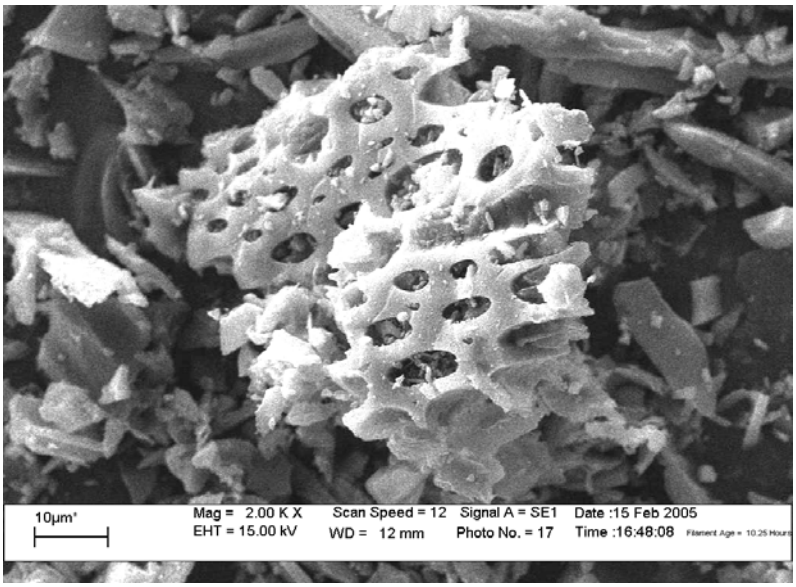


Figure 25 SEM micrograph of Fluka at 2000 x magnification

4.2 FT-IR spectroscopy

The different in the adsorption of activated carbon and bamboo charcoal might be attributed from functional groups on surface that could enhance the adsorption. Therefore, the functional groups on surface were studied by FT-IR. The band assignments are shown in Table 11.

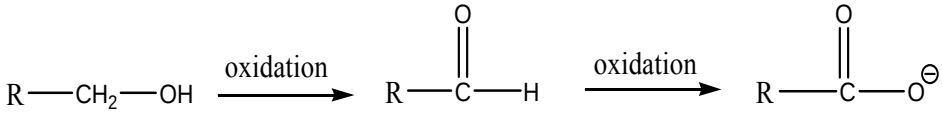
Table 11 Band assignments for FT-IR spectra.

Functional group	Wavelength (cm^{-1})
Hydroxyl group (O-H Stretching, ROH)	3,450 – 3,350
Alkane (C-H Stretching)	2,950 - 2800
Ester (ROOR´)	1,750 – 1,650
Aromatic (C-C Stretching)	
Asymmetric and symmetric carboxylate ($(C-O)_2$ Streching)	1,580 – 1,540
Alkane (C-H Bending)	
Alcoholic (C-O Stretching)	1,200 – 1,050
Ether (ROR)	
Cyclic group	880 - 650

Source: Pavia *et al*. (2001)

The FT-IR spectra and their assigned peaks of all samples were presented in Appendix G and Table 12, respectively. It could be seen that the activated carbon spectrum was closely identical to bamboo charcoal. A board peak in the range of 3,450 – 3,350 cm^{-1} was attributed to O-H stretching vibration of hydroxyl group. Presence of aromatics could be observed through C-C Stretching vibrations in

the range of 1,580 – 1,540 cm^{-1}. The bands in the 1200–1000 cm^{-1} region were difficult to assign because there were a number of broad overlapping bands. The very weak absorption which observed at 880–650 cm^{-1} was attributed to the cyclic group. It indicated that the bamboo charcoal and activared carbon gave rise to such peaks more than the Fluka and Bunton. This pointed out that the charcoal and activated carbon had higher aromaticity than Fluka and bunton, influencing the adsorption ability of activated carbon. The high aromaticity could cause the negative environment on the surface of activated carbon, which enhanced the positive molecule to adsorb on the surface. However, the band in the range of 1,750 – 1,650 cm^{-1} was ascribed to C=O or COO^- groups. Some kinds of bamboo charcoals showed the small presence of those bands; some did not but this band occurred after the activation by H_3PO_4 and KOH due to the oxidation reaction by –OH group change to C=O or COO^- groups as shown in equation.



------ (16)

C=O or COO^- groups can increase the adsorption capacity of activated carbon since C=O or COO^- groups has a negative property. Therefore, it could attract a positive matter such as metal ions or cationic molecules.

The FT-IR results obtained for the Fluka and Bunton were similar to activated carbon but the band of Fluka and Bunton in the range of 1,750 – 1,650 cm^{-1} which was attributed to C=O or COO^- groups was absence. It pointed out that Fluka and Bunton were prepared by pyrolysis at high temperature so the major organic compounds and volatile matters are decomposed at the high extent. This reason causes a high amount of carbon in Fluka and Bunton. Furthermore, the band in the range of 2,950 – 2800 cm^{-1} was also identified as C-H stretching of alkane which was only found in the spectra of Fluka and Bunton.

Table 12 The FTIR results of activated carbon and bamboo charcoal.

Sample	Wavelength (cm^{-1})					
	3,450 – 3,350	2,950 - 2800	1,750 – 1,650	1,580 – 1,540	1,200 – 1,000	880 - 650
Fluka [a]	broad, medium 3,432	very weak 2,917, 2,850	(-)	very weak 1,559	broad, strong 1,115, 1,019	very weak 881, 836, 802, 720
Bunton [b]	broad, medium 3,432	very weak 2,917, 2,850	(-)	weak 1,559 1,541	broad, medium 1,078	weak 873, 802, 668, 616
BAWC 1	broad, medium 3,372	(-)	(-)	strong 1,583	broad, weak 1,116	broad 871, 805, 750
BAWP 1	broad, medium 3,357	(-)	broad, weak 1,690	broad, strong 1,561	broad 1,103	broad, weak 875, 805, 750
BAWK 1	broad, medium 3,052	(-)	broad, weak 1,694	medium 1,569	broad, weak 1,212	very weak 875, 809, 750

[a] Activated carbon was produced by Fluka Company.

[b]Bamboo charcoal was produced by Thai Agard Dee Bamboo Charcoal Products.

Table 12 (Continued)

Sample	Wavelength (cm^{-1})					
	3,450 – 3,350	2,950 - 2800	1,750 – 1,650	1,580 – 1,540	1,200 – 1,000	880 - 650
BAWC 2	broad, medium 3,364	(-)	(-)	medium 1,582	broad, weak 1,116	very weak 879, 809, 750
BAWP 2	broad, medium 3,394	(-)	broad, weak 1,690	medium 1,558	broad, weak 1,196	very weak 875, 750, 669
BAWK 2	broad, medium 3,048	(-)	broad, weak 1,690	medium 1,565	broad, weak 1,224	very weak 879, 812, 753
BAWC 3	broad, medium 3,342	(-)	(-)	strong 1,586	weak 1,117	very weak 875, 750, 613
BAWP 3	broad, medium 3,433	(-)	broad, weak 1,686, 1,620	medium 1,557	broad, weak 1,222	very weak 665
BAWK 3	broad, medium 3,302	(-)	broad, weak 1,698	broad, strong 1,574	broad, weak 1,226	very weak 886, 816, 753, 665

Table 12 (Continued)

Sample	Wavelength (cm^{-1})					
	3,450 – 3,350	2,950 - 2800	1,750 – 1,650	1,580 – 1,540	1,200 – 1,000	880 - 650
GOC 1	broad, medium 3,170	(-)	very weak 1,686	strong 1,575	broad, weak 1,220, 1,115	very weak 875, 812, 750
GOP 1	broad, medium 3,179	(-)	broad, weak 1,694	strong 1,561	broad, medium 1,179	very weak 879, 812, 750
GOK 1	broad, strong 3,147	(-)	broad, weak 1,702	strong 1,588	broad, weak 1,229	very weak 890, 823, 746
GOC 2	broad, medium 3,182	(-)	very weak 1,690	strong 1,578	broad, weak 1,221, 1,115	very weak 879, 783, 669
GOP 2	broad, medium 3,201	(-)	broad, weak 1,690	strong 1,588	broad, medium 1,220	very weak 882, 779, 669
GOK 2	broad, medium 3,194	(-)	broad, weak 1,697	strong 1,588	broad, weak 1,228	very weak 894, 823, 761

Table 12 (Continued)

Sample	Wavelength (cm-1)					
	3,450 – 3,350	2,950 - 2800	1,750 – 1,650	1,580 – 1,540	1,200 – 1,000	880 - 650
GOC 3	broad, medium 3,200	(-)	very weak 1,690	strong 1,590	broad, weak 1,115	very weak 882, 783, 665
GOP 3	broad, medium 3,186	(-)	broad, weak 1,694	strong 1,592	broad, weak 1,217	very weak 890, 790, 665
GOK 3	broad, medium 3,061	(-)	broad, weak 1,697, 1,664	strong 1,588	broad, weak 1,226	very weak 886, 757
GVC 1	broad, medium 3,180	(-)	broad, weak 1,697	strong 1,580	broad, weak 1,240, 1,115	very weak 879, 757, 613
GVP 1	broad, strong 3,422	(-)	broad, weak 1,694	medium 1,558	broad, weak 1,229	very weak 886, 753, 665
GVK 1	broad, strong 3,047	(-)	broad, weak 1,669	strong 1,588	broad, weak 1,240	very weak 886, 768

Table 12 (Continued)

Sample	Wavelength (cm^{-1})					
	3,450 – 3,350	2,950 - 2800	1,750 – 1,650	1,580 – 1,540	1,200 – 1,000	880 - 650
GVC 2	broad, medium 3,215	(-)	very weak 1,694	strong 1,586	broad, weak 1,236, 1,115	very weak 879, 779
GVH 2	broad, medium 3,213	(-)	broad, weak 1,694	strong 1,560	broad, weak 1,221	very weak 882, 776
GVK 2	broad, strong 3,043	(-)	very weak 1,690	medium 1,588	broad, weak 1,238	very weak 869, 810, 787, 668
GVC 3	broad, strong 3,419	(-)	(-)	medium 1,561	broad, weak 1,222	very weak 875, 779
GVH 3	broad, medium 3,336	(-)	broad, weak 1,694	broad, strong 1,567	broad 1,223	very weak 886, 779, 665
GVK 3	broad, medium 3,041	(-)	broad, weak 1,705, 1,671	strong 1,589	broad 1,238	very weak 886, 827, 772

5. Adsorption properties of charcoal and activated carbon

5.1 Iodine adsorption

5.1.1 Iodine number

The adsorption ability of activated carbon depended on the pore size that had to be suitable to the size of adsorbate. For example, when the pore size was smaller than size of adsorbate, the adsorbate could not intrude into the pore so it had a low adsorption. The number of micropore inside activated carbon had a profound effect on iodine adsorption because the small particle diameter of iodine molecule could be adsorbed inside the micropore of adsorbent. Therefore, the value of iodine number informed about a number of micropore.

Table 13 The iodine number of bamboo charcoal and bamboo charcoal for adsorption ability of iodine.

Charcoal			Iodine number (mg/g)
Type of bamboo	Age of bamboo	Chemical activation	
Fluka [a]			708±6.08
BAW	1 year	-	178± 3.61
		H_3PO_4	150± 5.29
		KOH	188± 2.64
	2 years	-	92± 2.64
		H_3PO_4	102± 2.64
		KOH	114± 3.46
	3 years	-	140± 2.00
		H_3PO_4	145± 1.73
		KOH	177± 2.64

Table 13 (Continued)

Charcoal			Iodine number (mg/g)
Type of bamboo	Age of bamboo	Chemical activation	
GO	1 year	-	71± 7.55
		H_3PO_4	105± 3.46
		KOH	154± 3.46
	2 years	-	82± 2.64
		H_3PO_4	115± 3.00
		KOH	158± 3.61
	3 years	-	89± 4.58
		H_3PO_4	129± 4.58
		KOH	197± 2.64
GV	1 year	-	106± 7.00
		H_3PO_4	145± 3.61
		KOH	1,202
	2 years	-	105± 3.61
		H_3PO_4	206± 5.57
		KOH	748
	3 years	-	103± 3.46
		H_3PO_4	124± 2.00
		KOH	643

[a] Activated carbon was produced by Fluka Company.

Table 13 showed the iodine number of activated carbon, bamboo charcoal and Fluka. It could be seen that the iodine number of activated carbon was increased, compared to charcoal. This might be attributed to the activation process with H_3PO_4 and KOH, creating a higher number of micropores. Therefore, the activated carbon had a high surface for adsorption of iodine. This was also confirmed by SEM indicating that after the activation by H_3PO_4 and KOH, the activated carbon had higher number of pores. Moreover, the iodine number of activated carbon which activated by KOH was higher than the iodine number of activated carbon which activated by H_3PO_4 due to the difference in chemical activation processes.
The activation with KOH brought about the higher number of micropore than H_3PO_4. Thus, the efficiency for activating charcoal with KOH was higher than in case of H_3PO_4.

By considering types of bamboo used in preparation of activated carbon, the iodine numbers of activated carbon which were prepared from all three types of bamboo were in the range of 100-200 mg/g, but the iodine number of activated carbon of GVC which activated by KOH was in the range of 600-1,200 mg/g. In terms of ages of bamboo used in preparation of activated carbon, the iodine numbers of activated carbon of all three ages were similar. This might due to the same amount of adsorption surface for iodine.

The studies of activated carbon and Fluka indicated that the activated carbon had lower iodine number than Fluka excepting GVK 1 (1,202 mg/g) because the activated carbon was prepared from chemical activation. Therefore, the surface on activated carbon was negative due to the increasing of C=O or COO^- functional groups on surface of activated carbon. Accordance to the iodine anion occurred from iodine dissolved in water resulting in the repulsion between these negative charges. On the other hand, the Fluka migth be prepared by stream activation. Thus, Fluka had no C=O or COO^- functional groups on surface. Consequently, the adsorption capability of activated carbon decreased more than that of Fluka. This was also confirmed by IR. However, the GVK 1 had the highest iodine

number indicating the numerous adsorption surfaces. This was also confirmed by SEM (Figure 24) that GVK 1 had a large numbers of pores since GV mainly consisted of 51.00% of moisture. Meanwhile, the moisture of BAW and GO were 13.80 and 20.60%, respectively (Anapanurak *et al*., 2007). When the carbonization was preceded, the amount of water contained in the bamboo would be evaporated and pores were finally generated. Therefore, the activated carbon prepared from GV provided the large amount of pores.

5.1.2 Adsorption isotherms of iodine onto activated carbon and bamboo charcoal.

The adsorption isotherm indicated how the adsorption molecules distribute between the liquid phase and the solid phase when the adsorption process reaches an equilibrium state. The analysis of the isotherm data by fitting them to different isotherm models was an important step to find the suitable model that could be used for the design purposes.

Adsorption isotherm study was carried out on two well-known isotherm models which were Langmuir and Freundlich isotherm. Langmuir isotherm assumes monolayer adsorption onto a surface containing a finite number of adsorption sites of uniform strategies of adsorption with no transmigration of adsorbate in the plane of surface. Meanwhile, Freundlich isotherm model assumed heterogeneous surface energies. The applicability of the isotherm models to the adsorption study was compared by judging the correlation coefficient (R^2) values.

Figures 26-34 showed the Langmuir plots of C_e/ q_e versus C_e and Freundlich plots of log q_e versus log C_e for the adsorption of iodine onto the activated carbon.

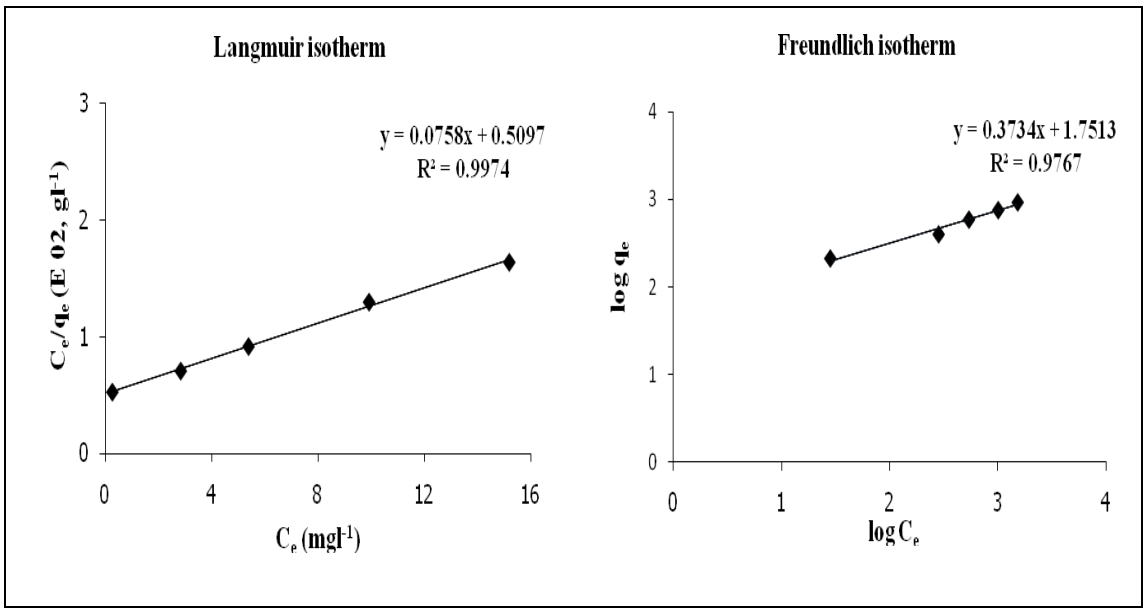


Figure 26 Langmuir and Freundlich adsorption isotherm of iodine onto BAWC 1.

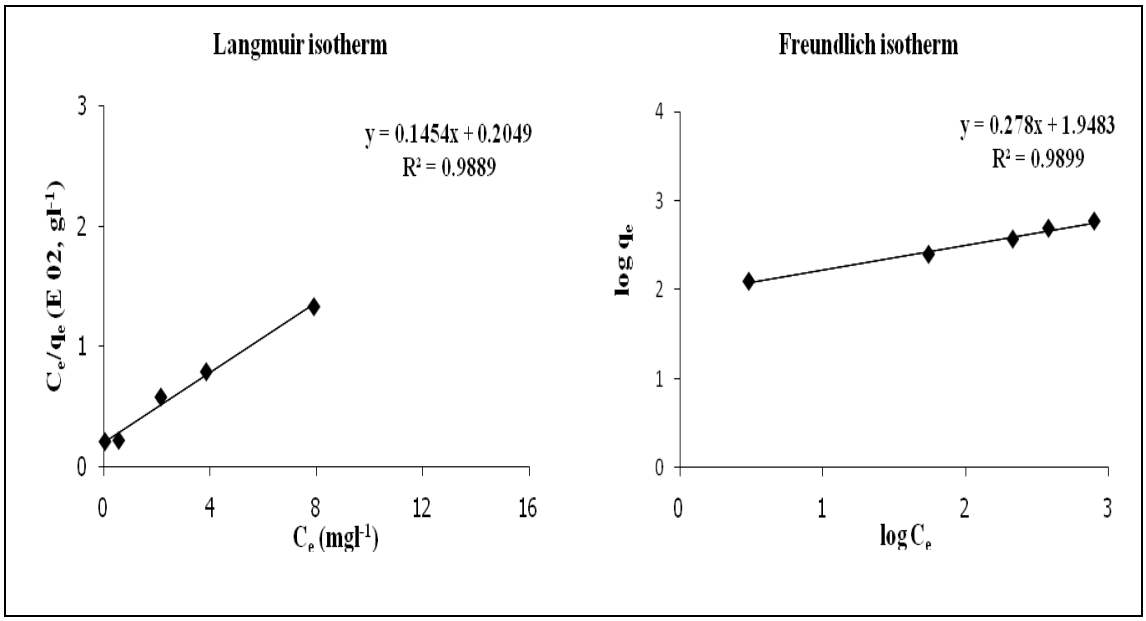


Figure 27 Langmuir and Freundlich adsorption isotherm of iodine onto BAWP 1.

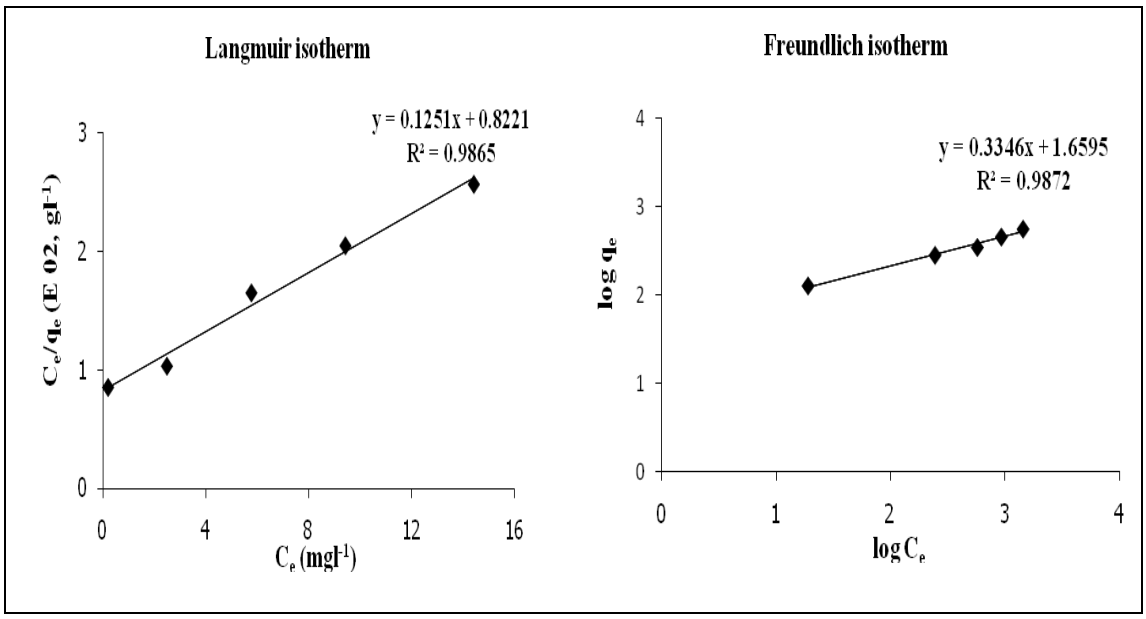


Figure 28 Langmuir and Freundlich adsorption isotherm of iodine onto BAWK 1.

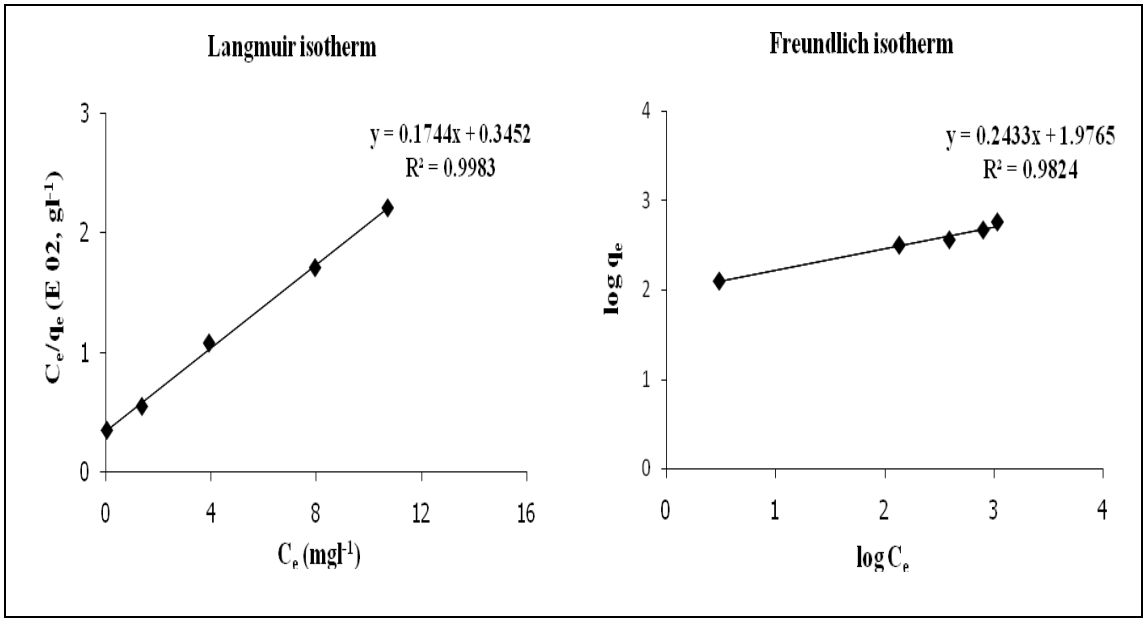


Figure 29 Langmuir and Freundlich adsorption isotherm of iodine onto GOC 3.

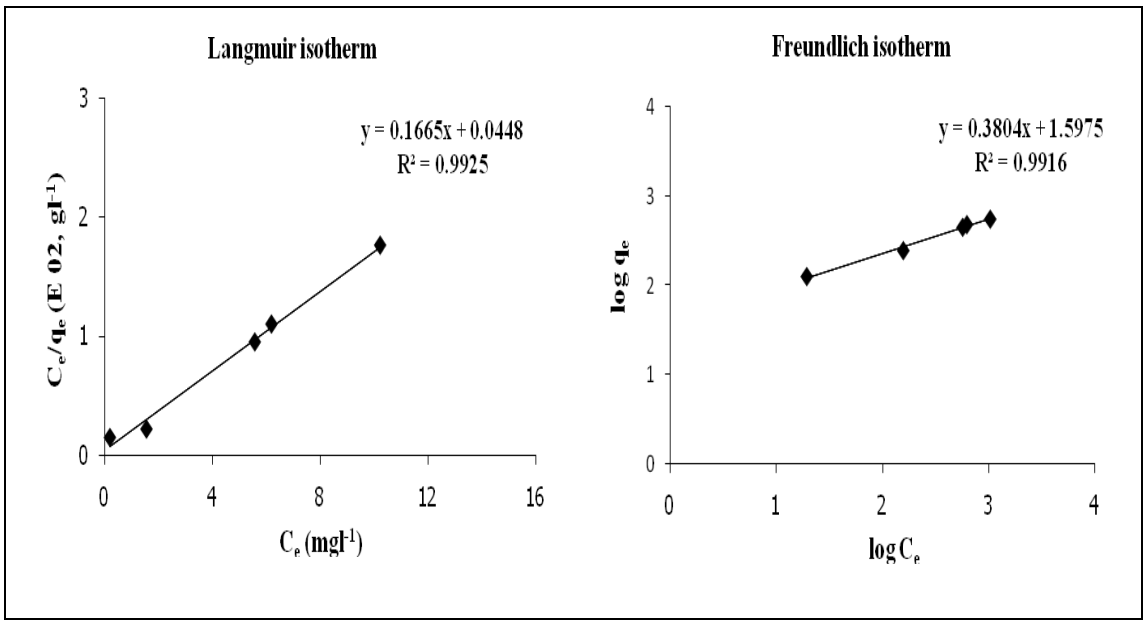


Figure 30 Langmuir and Freundlich adsorption isotherm of iodine onto GOP 3.

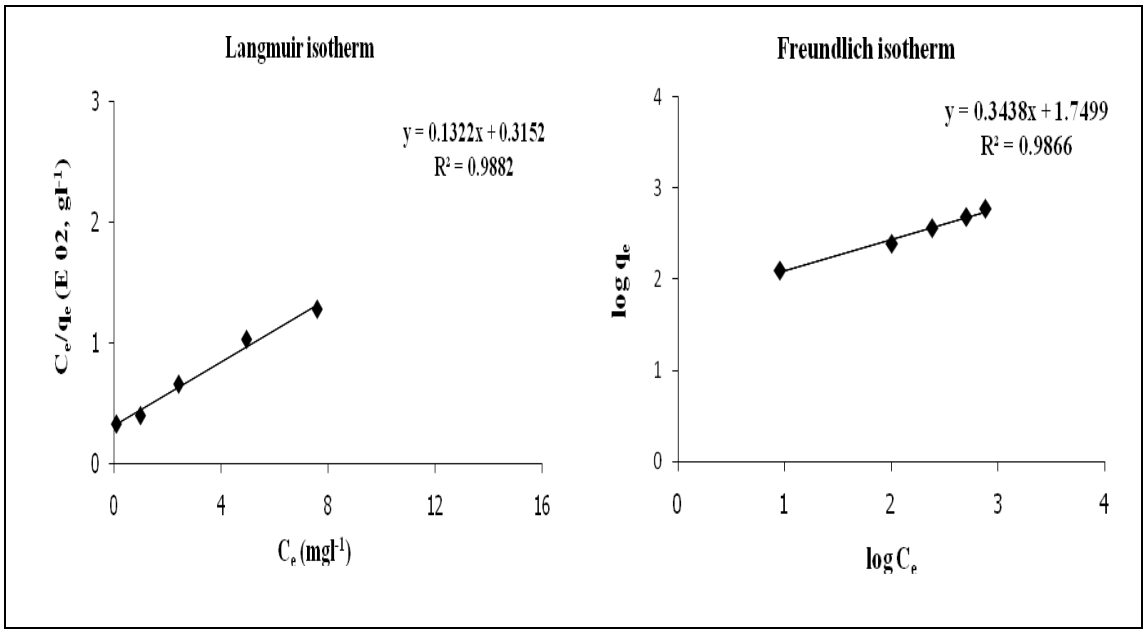


Figure 31 Langmuir and Freundlich adsorption isotherm of iodine onto GOK 3.

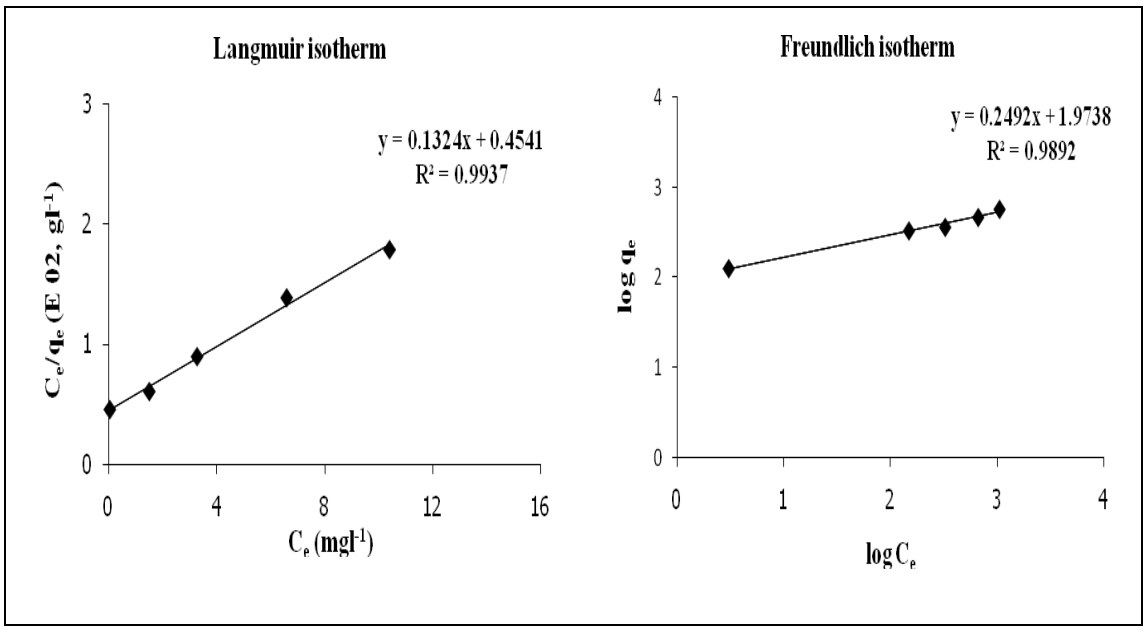


Figure 32 Langmuir and Freundlich adsorption isotherm of iodine onto GVC 1.

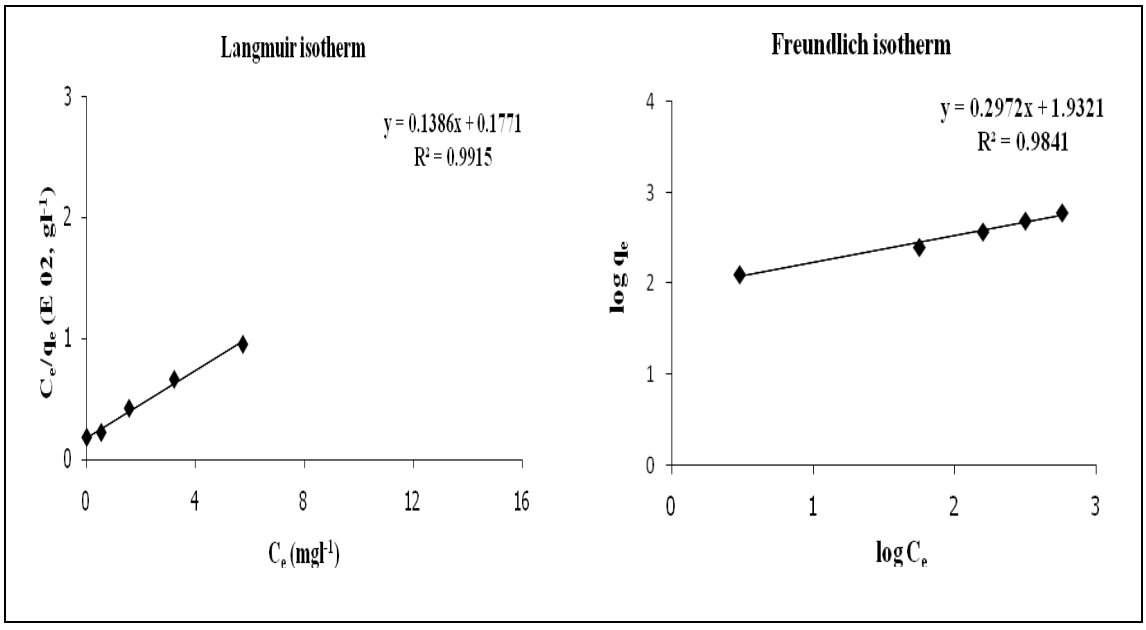


Figure 33 Langmuir and Freundlich adsorption isotherm of iodine onto GVP 2.

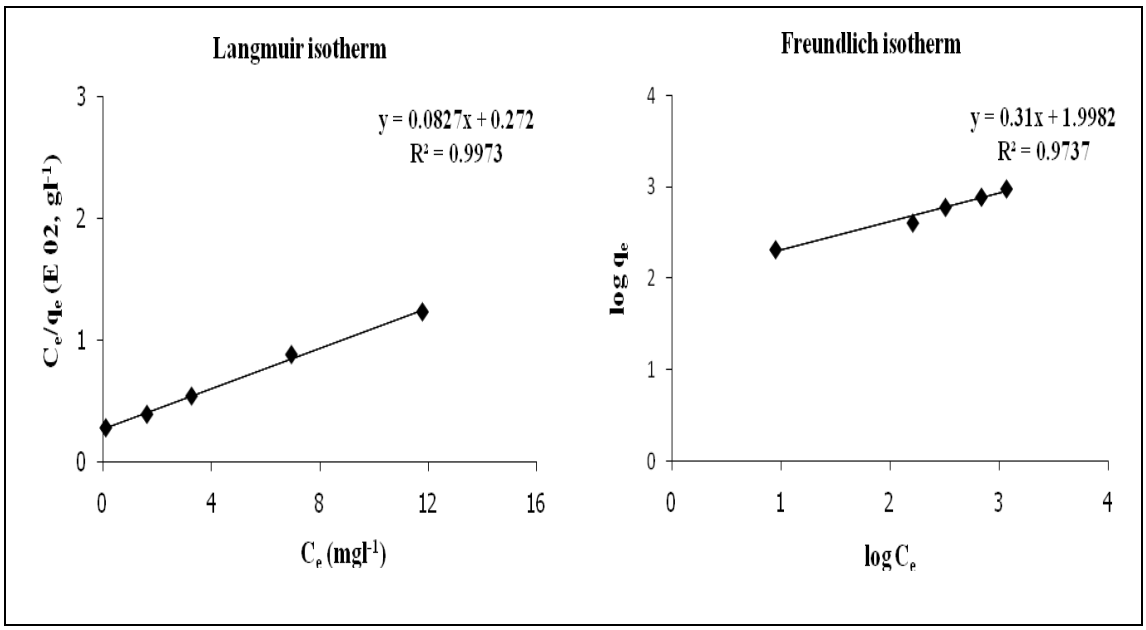


Figure 34 Langmuir and Freundlich adsorption isotherm of iodine onto GVK 1.

The Langmuir and Freundlich adsorption isotherm constants for iodine adsorption are given in Table 14. Apparently, the fitting to Langmuir adsorption isotherm model is similar in comparison to Freundlich model according to R^2 values. Therefore, the types of adsorption could be either monolayer or multilayer coverage of iodine molecules onto surface of activated carbon, bamboo charcoal, Bunton and Fluka. The Langmuir constants showed that the Q_o of all samples were in the range of 5-13 mg/g. Furthermore, the b value of Fluka and Bunton were higher than that of activated carbon and bamboo charcoal, indicating that the Fluka and Bunton had the high adsorption energy. The Freundlich constants showed that all the 1/n values were in the range of 0-1, suggesting favorable multilayer adsorption of iodine. Furthermore, the K_F value of Fluka and Bunton were higher than that of activated carbon and bamboo charcoal, indicating that the Fluka and Bunton had the higher adsorption capacity.

Table 14 Langmuir and Freundlich isotherm constants for iodine**.**

Sample	Langmuir isotherm			Freundlich isotherm		
	Q_o (mg/g)	b (L/mg)	R^2	1/n	K	R^2
Fluka [a]	9.69	4.691	0.997	0.203	2,771.41	0.993
Bunton [b]	6.86	4.600	0.993	0.189	1,790.61	0.993
BAWC 1	12.98	0.156	0.996	0.373	56.36	0.976
BAWP 1	6.62	0.878	0.985	0.278	88.72	0.989
BAWK 1	7.35	0.194	0.978	0.334	45.60	0.987
GOC 3	6.29	0.511	0.959	0.243	94.62	0.982
GOP 3	5.85	2.440	0.974	0.380	39.54	0.991
GOK 3	7.09	0.536	0.982	0.343	56.10	0.986
GVC 1	7.25	0.339	0.990	0.249	93.97	0.989
GVP 2	6.99	0.923	0.988	0.297	85.51	0.984
GVK 1	11.90	0.331	0.996	0.310	99.54	0.973

[a] Activated carbon was produced by Fluka Company.

[b] Bamboo charcoal was produced by Thai Agard Dee Bamboo Charcoal Products.

Table 15 The specific surface area of samples for adsorption of iodine**.**

Sample	Specific surface area (m^2/g)
Fluka	14.71
Bunton	10.41
BAWC 1	19.70
BAWP 1	10.05
BAWK 1	11.16
GOC 3	9.55
GOP 3	8.88
GOK 3	10.76
GVC 1	11.01
GVP 2	10.61
GVK 1	18.06

According to the Langmuir isotherm in case of iodine, this pointed out the fact that the iodine adsorption was the mono-layered adsorption, in which the specific surface area could be calculated by replacing the Q_o value as shown in Table 15 on the equation 12. The obtained specific surface area of all activated carbon and bamboo charcoal were in the range of 8-19 m^2/g. The specific surface area value reflected the iodine adsorption ability of the activated carbon, in that, the higher this value, the more adsorption ability. However, in some cases, the lower specific surface area could cause the higher iodine adsorption ability owing to the multi-layered adsorption of iodine molecules on the surface of activated carbon.

5.2 Phenol adsorption

5.2.1 Phenol value

Table 16 The phenol value of of bamboo charcoal and bamboo charcoal for adsorption ability of phenol.

Charcoal			Phenol value (mg/L)
Type of bamboo	Age of bamboo	Chemical activation	
Fluka [a]			2.32±0.13
Bunton [b]			7.71±0.32
BAW	1 year	-	4.90±0.31
		H_3PO_4	24.62±2.22
		KOH	13.30±0.87
	2 years	-	28.01±0.58
		H_3PO_4	13.45±0.79
		KOH	11.27±2.95
	3 years	-	12.64±0.94
		H_3PO_4	24.86±0.83
		KOH	35.52±2.01

[a] Activated carbon was produced by Fluka Company.

[b] Bamboo charcoal was produced by Thai Agard Dee Bamboo Charcoal Products.

Table 16 (Continued)

Charcoal			Phenol value (mg/L)
Type of bamboo	Age of bamboo	Chemical activation	
GO	1 year	-	11.37±2.30
		H_3PO_4	10.97±1.18
		KOH	10.30±2.36
	2 years	-	19.08±3.46
		H_3PO_4	56.84±6.39
		KOH	12.41±0.73
	3 years	-	17.81±0.53
		H_3PO_4	17.52±7.09
		KOH	22.53±7.23
GV	1 year	-	23.40±1.37
		H_3PO_4	23.23±0.97
		KOH	18.27±1.67
	2 years	-	11.04±1.97
		H_3PO_4	7.34±1.23
		KOH	12.56±0.77
	3 years	-	13.45±1.18
		H_3PO_4	14.65±1.46
		KOH	29.39±0.93

The phenol value was the value which indicated the adsorption ability of an adsorbent on phenol molecule. A low phenol value suggested the high adsorption ability. Based on the process for calculation of the phenol value, this value was calculated from the 10% remaining concentration from the initial phenol concentration that the activated carbon adsorbed. Hence, if the remaining concentration of phenol was minute, the activated carbon should adsorb phenol with the high extent. Table 16 showed the phenol values of activated carbon, bamboo charcoal, Bunton and Fluka for adsorption ability of phenol. It was supposed that the activated carbon had a low capability for phenol adsorption. This might be attributed to the preparation of activated carbon from chemical activation, causing C=O or COO^- functional groups on surface of activated carbon. The carboxylate groups on the carbon surface removed the π-electron from the activated carbon aromatic ring matrix, causing a decrease in the strength of interactions between the benzene ring of phenol and the carbon's basal planes, which decreases the uptake of phenol (Salame and Bandosz, 2003). However, the phenol value of GVP 2 was similar to that of Bunton.

The Fluka had the lowest phenol value, compared to activated carbon and bamboo charcoal. However, the phenol value of BAWC 1 was 4.9 mg/L similar to that of Fluka. This might be attributed to the high surface of BAWC 1 for adsorption of phenol because a large number of pores having bigger pore size than the phenol molecule (Salame and Bandosz, 2003). Those pores originated from the carbonization process.

5.2.2 Adsorption isotherms of phenol onto activated carbon and bamboo charcoal.

Figure 35-43 showed the Langmuir plots of C_e/ q_e versus C_e and Freundlich plots of log q_e versus log C_e for the adsorption of phenol onto the activated carbon.

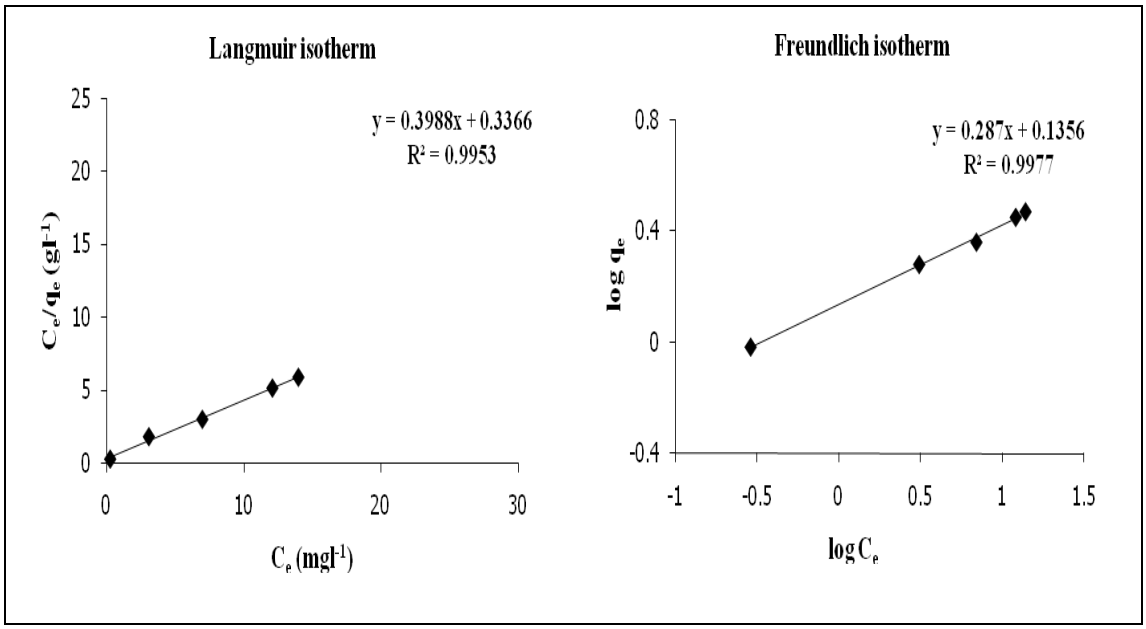


Figure 35 Langmuir and Freundlich adsorption isotherm of phenol onto BAWC 1.

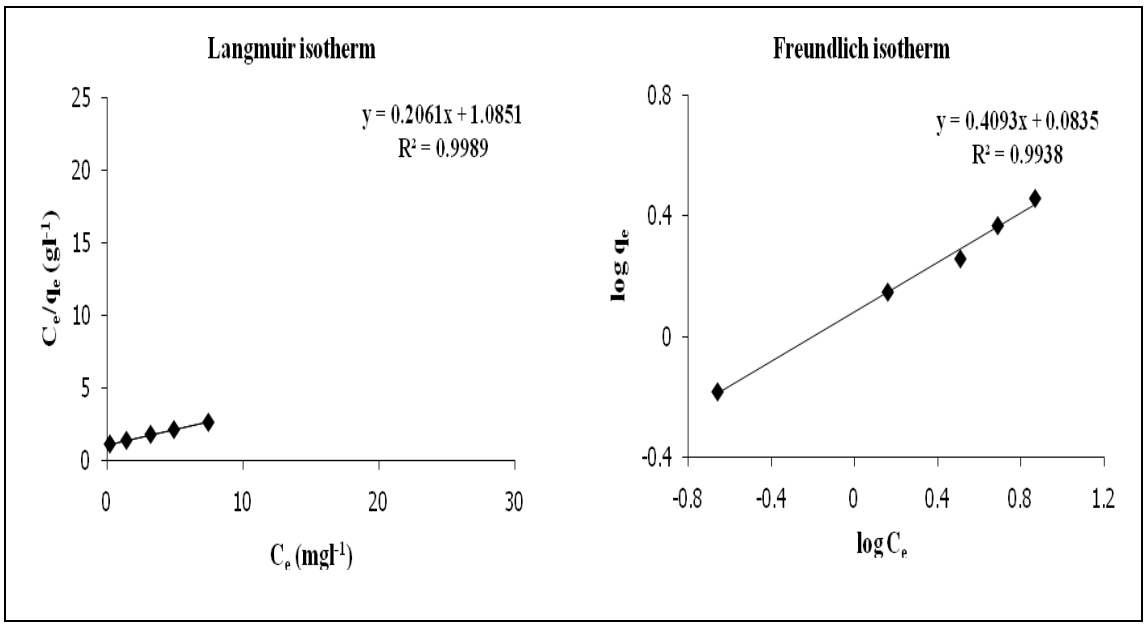


Figure 36 Langmuir and Freundlich adsorption isotherm of phenol onto BAWP 2.

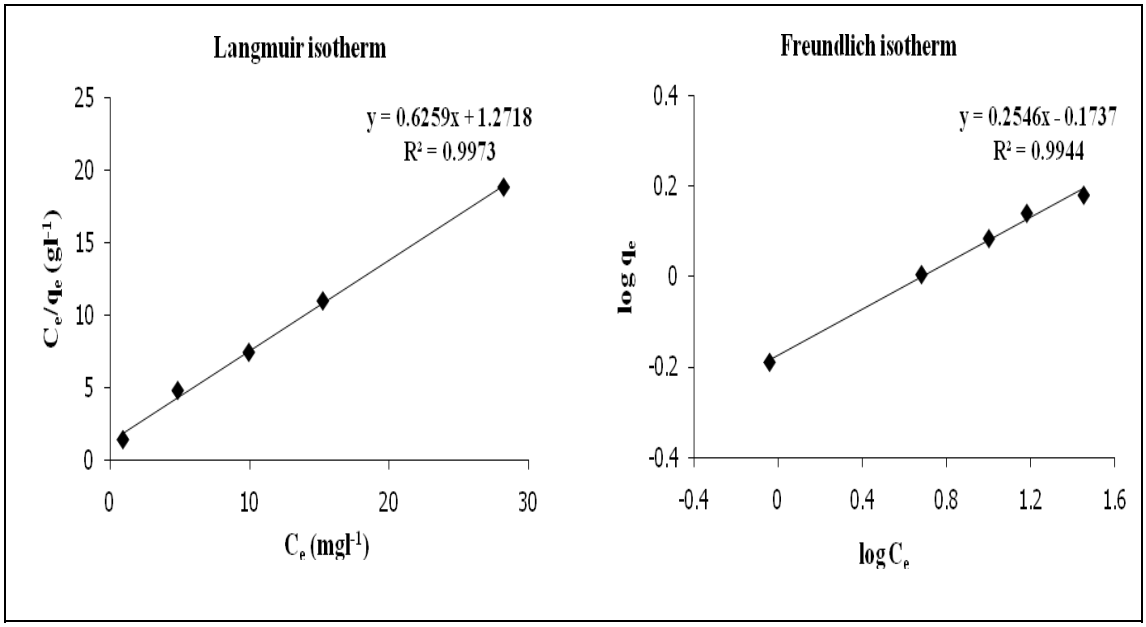


Figure 37 Langmuir and Freundlich adsorption isotherm of phenol onto BAWK 2.

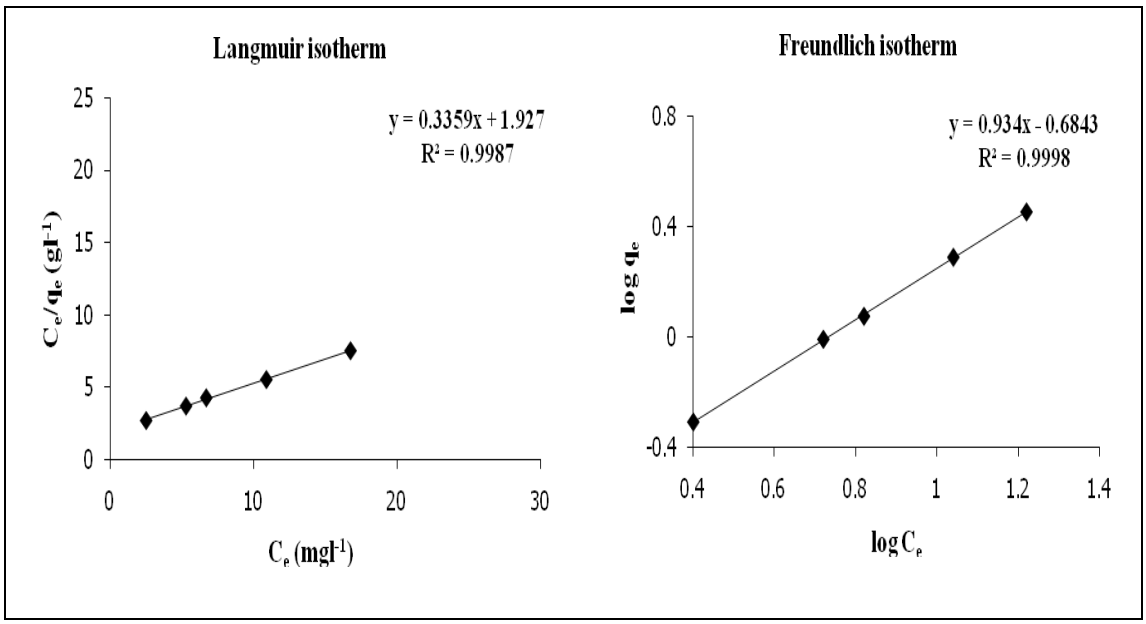


Figure 38 Langmuir and Freundlich adsorption isotherm of phenol onto GOC 1.

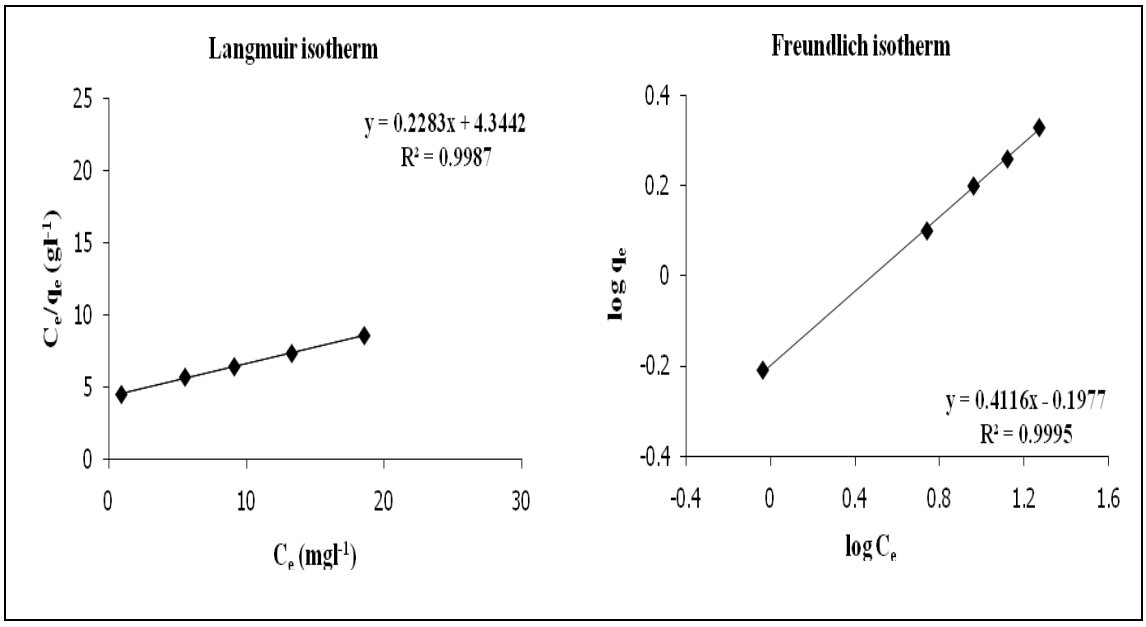


Figure 39 Langmuir and Freundlich adsorption isotherm of phenol onto GOP 1.

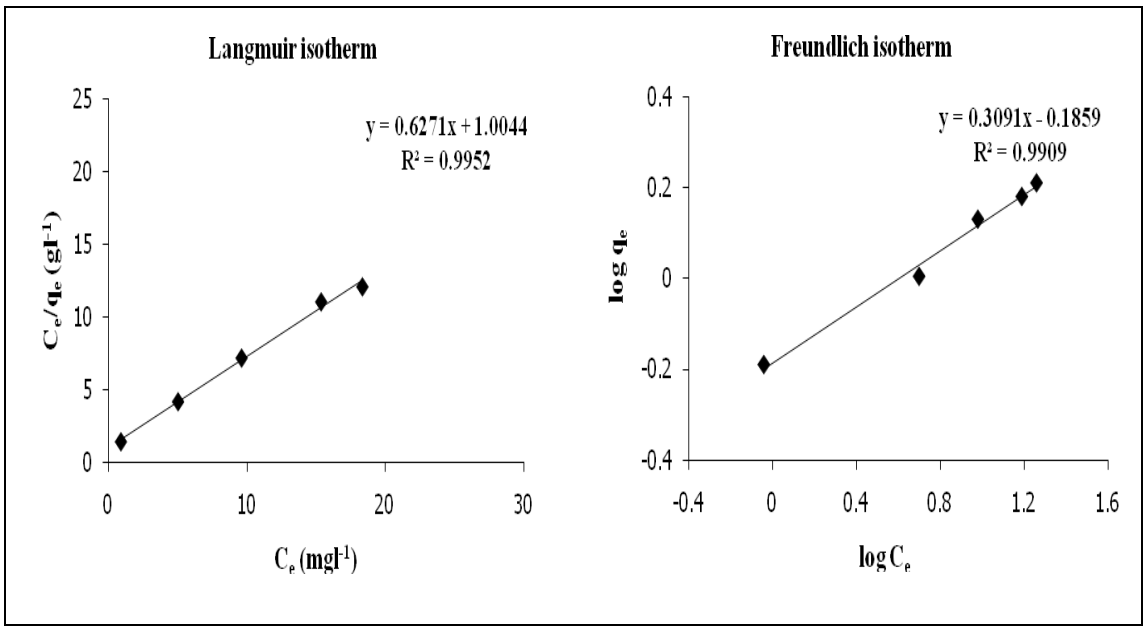


Figure 40 Langmuir and Freundlich adsorption isotherm of phenol onto GOK 1.

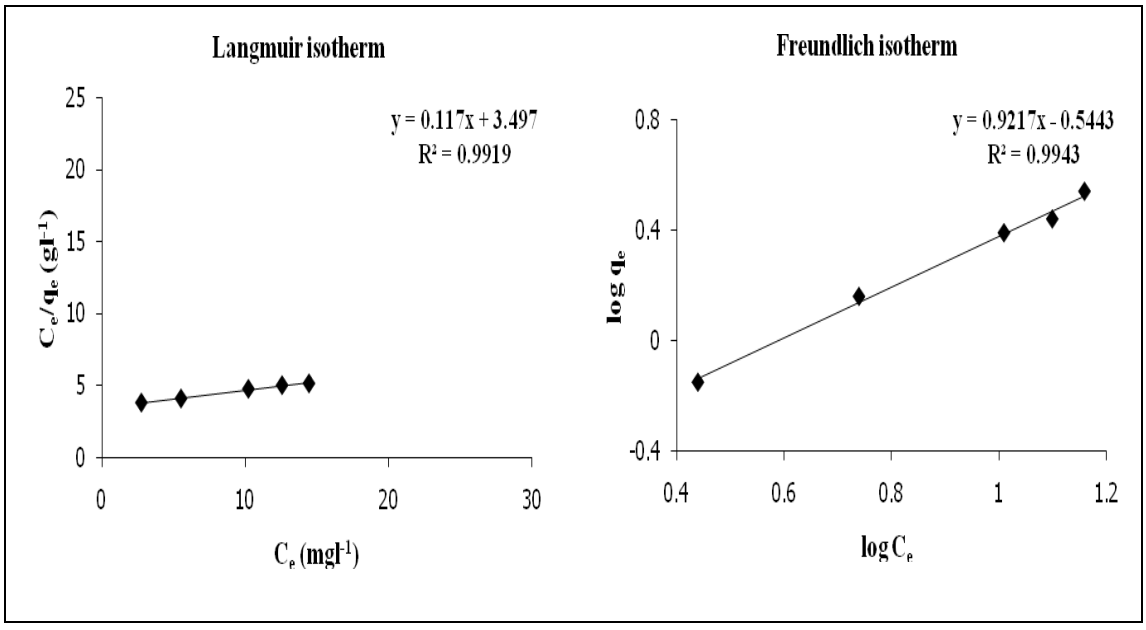


Figure 41 Langmuir and Freundlich adsorption isotherm of phenol onto GVC 2.

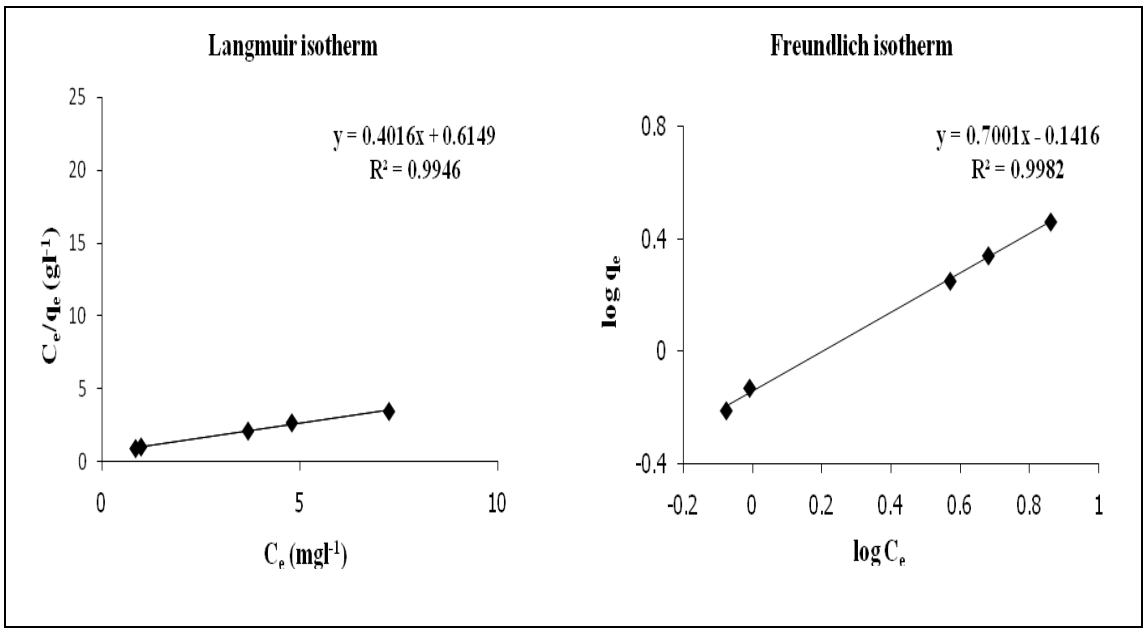


Figure 42 Langmuir and Freundlich adsorption isotherm of phenol onto GVP 2.

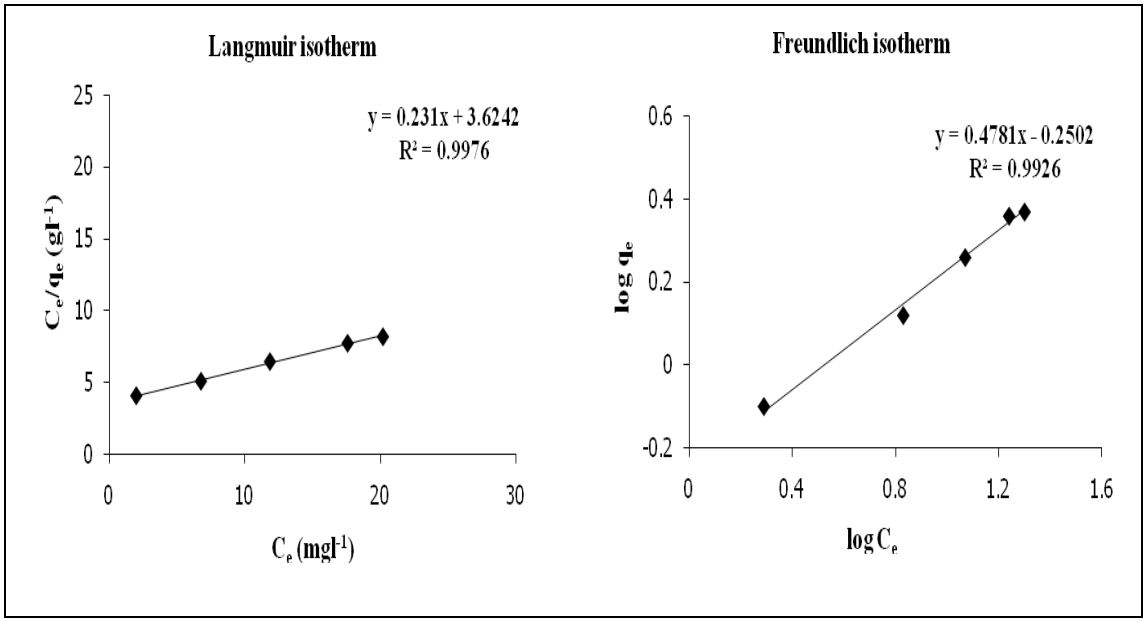


Figure 43 Langmuir and Freundlich adsorption isotherm of phenol onto GVK 2.

The Langmuir and Freundlich adsorption isotherm constants for phenol adsorption are given in Table 17. Obviously, the fitting to the Langmuir adsorption isotherm model was similar in comparison to the Freundlich model according to R^2 values. Therefore, the types of adsorption could be either monolayer or multilayer coverage of phenol molecules onto surface of activated carbon, bamboo charcoal, Bunton, and Fluka. The Langmuir constants showed that the Q_0 of GVC 2 was the highest, compared to all samples. Furthermore, the b value of Fluka and Bunton was higher than that of activated carbon and bamboo charcoal, indicating that the Fluka and Bunton had the higher adsorption energy. The Freundlich constants showed that the all 1/n value was in the range of 0-1 shows favorable multilayer adsorption of phenol (Hameed *et al*., 2007). Furthermore, the K_F value of activated carbon was similar to that of Fluka, Bunton, and bamboo charcoal. However, the K_F value of GOC 1 was the highest value, pointing out that the GOC 1 had high adsorption capacity.

Table 17 Langmuir and Freundlich isotherm constants for phenol**.**

Sample	Langmuir isotherm			Freundlich isotherm		
	Q_o (mg/g)	b (L/mg)	R^2	1/n	K_F	R^2
Fluka [a]	2.59	2.171	0.983	0.447	1.60	0.981
Bunton [b]	2.51	2.328	0.988	0.404	1.56	0.988
BAWC 1	2.51	1.186	0.995	0.287	1.36	0.997
BAWP 2	4.85	0.190	0.998	0.409	1.21	0.993
BAWK 2	1.60	0.492	0.997	0.254	1.48	0.994
GOC 1	2.98	0.174	0.998	0.934	4.83	0.999
GOP 1	4.38	0.052	0.998	0.411	1.57	0.999
GOK 1	1.59	0.626	0.995	0.309	1.53	0.990
GVC 2	8.55	0.033	0.991	0.921	3.50	0.994
GVP 2	2.49	0.654	0.994	0.700	1.38	0.998
GVK 2	4.33	0.064	0.997	0.478	1.78	0.992

[a] Activated carbon was produced by Fluka Company.

[b] Bamboo charcoal was produced by Thai Agard Dee Bamboo Charcoal Products.

Table 18 The specific surface area of samples for adsorption of phenol**.**

Sample	Specific surface area (m^2/g)
Fluka	8.65
Bunton	8.38
BAWC 1	8.38
BAWP 2	16.19
BAWK 2	5.34
GOC 1	9.95
GOP 1	14.62
GOK 1	5.31
GVC 2	28.55
GVP 2	8.31
GVK 2	14.46

According to the Langmuir isotherm in case of phenol, this pointed out the fact that the phenol adsorption was the mono-layered adsorption, in which the specific surface area could be calculated by replacing the Q_o value as shown in Table 18 on the equation 12. The obtained specific surface area of all activated carbon and bamboo charcoal were in the range of 5-28 m^2/g. The specific surface area value reflected the phenol adsorption ability of the activated carbon, in that, the higher this value, the more adsorption ability. In case of Fluka, it had the highest adsorption ability although its specific surface area was lower than the specific surface area of other activated carbons which may due to the multi-layered adsorption on the surface area of Fluka.

5.3 Methylene blue adsorption

5.3.1 Methylene blue value

Some activated carbon had a mesopore (2 to 5 nm) structure which adsorbs medium size molecules, such as methylene blue. Thus, the quantity of methylene blue which is adsorbed onto absorbent also indicated the number of mesopore.

Table 19 The methylene blue value of of bamboo charcoal and bamboo charcoal for adsorption ability of iodine.

Charcoal			Methylene blue value (mg/g)
Type of bamboo	Age of bamboo	Chemical activation	
Fluka [a]			15.50±0.61
BAW	1 year	-	13.80±1.58
		H_3PO_4	6.33±0.95
		KOH	14.00±1.85
	2 years	-	7.25±1.60
		H_3PO_4	6.66±1.17
		KOH	9.45±1.11
	3 years	-	7.62±1.83
		H_3PO_4	7.31±0.90
		KOH	6.06±0.68

[a] Activated carbon was produced by Fluka Company.

Table 19 (Continued)

Charcoal			Methylene blue value (mg/g)
Type of bamboo	Age of bamboo	Chemical activation	
GO	1 year	-	7.28±1.20
		H_3PO_4	5.77±1.21
		KOH	6.73±1.65
	2 years	-	6.94±0.42
		H_3PO_4	5.53±0.43
		KOH	8.82±1.61
	3 years	-	6.43±0.47
		H_3PO_4	6.25±1.79
		KOH	6.13±0.86
GV	1 year	-	14.50±1.21
		H_3PO_4	7.61±0.64
		KOH	15.50±1.63
	2 years	-	8.60±1.48
		H_3PO_4	7.97±2.50
		KOH	10.01±1.31
	3 years	-	7.22±1.06
		H_3PO_4	7.16±2.62
		KOH	8.77±1.58

Table 19 showed the methylene blue values of activated carbon, bamboo charcoal and Fluka for adsorption ability of methylene blue. It could be obviously seen that the activation of charcoal with H_3PO_4 and KOH had a minor effect on the adsorption capability of methylene blue. It was assumed that the chemical activation created the less numbers of pores which had bigger pore size than the dye molecule. The dye molecule could not enter into the pores of activated carbon. Thus, the methylene blue values of all activated carbon were similar to that of bamboo charcoal. However, the methylene blue values of BAWP 1 and GVP 1 were decreased because they were prepared by activated with H_3PO_4, causing C=O or COO^- functional groups on surface of activated carbon. The carboxylate groups on the carbon surface removed the π-electron from the activated carbon aromatic ring matrix, causing a decrease in the strength of interactions between the benzene ring of methylene blue and the carbon's basal planes, which decreases the uptake of methylene blue (Salame and Bandosz, 2003).

The Fluka had the highest methylene blue value, compared to activated carbon and bamboo charcoal. However, the methylene blue value of GVK 1 which was similar to that of Fluka was 15.5 mg/g. The activated carbon had a higher surface for adsorption of methylene blue because a large numbers of pores. This was also confirmed by SEM, indicating that after the activation by KOH, the activated carbon had higher number of pores.

For types of bamboo, which were used in preparation for activated carbon, the results indicated that the methylene blue values of all activated carbon were similar. This might be due to the same amount of adsorption surface for methylene blue. However, the methylene blue values of BAWC 1, BAWK 1, GVC 1, and GVK 1 were higher than those of as-prepared activated carbon because the large number of bigger pores which were called mesopores was occurred. For ages of bamboo that were used in preparation of activated carbon, the methylene blue values of activated carbon of all three ages of bamboo were similar. This might be due to the same amount of adsorption surface for methylene blue.

5.3.2 Adsorption isotherms of methylene blue onto activated carbon and bamboo charcoal.

Figures 44-52 showed the Langmuir plots of C_e/ q_e versus C_e and Freundlich plots of log q_e versus log C_e for the adsorption of methylene blue onto the activated carbon.

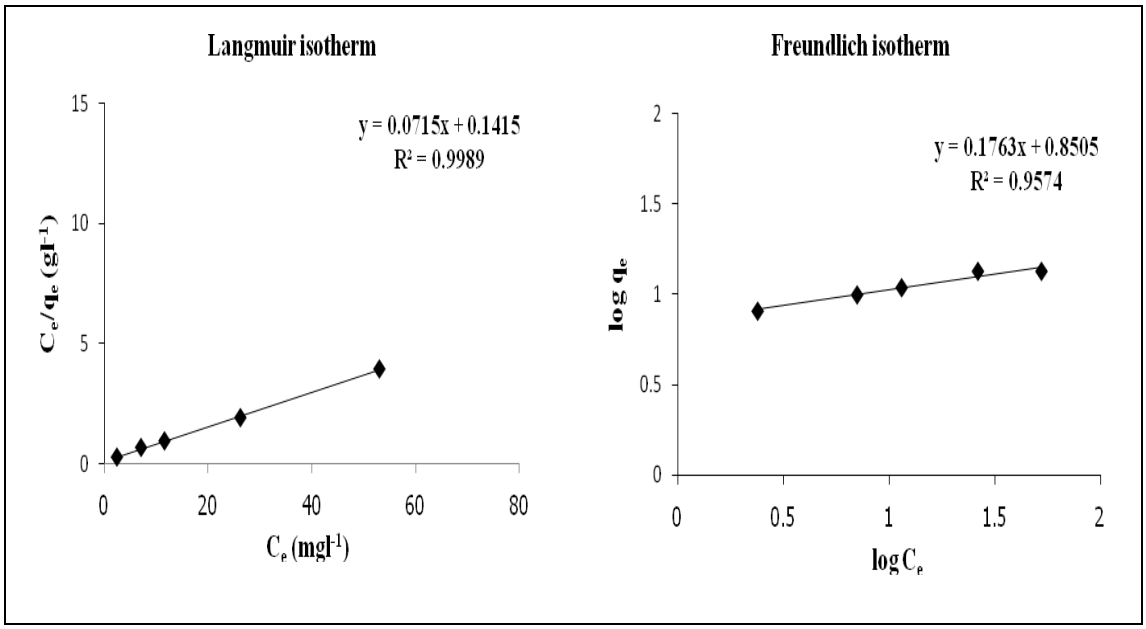


Figure 44 Langmuir and Freundlich adsorption isotherm of methylene blue onto BAWC 1.

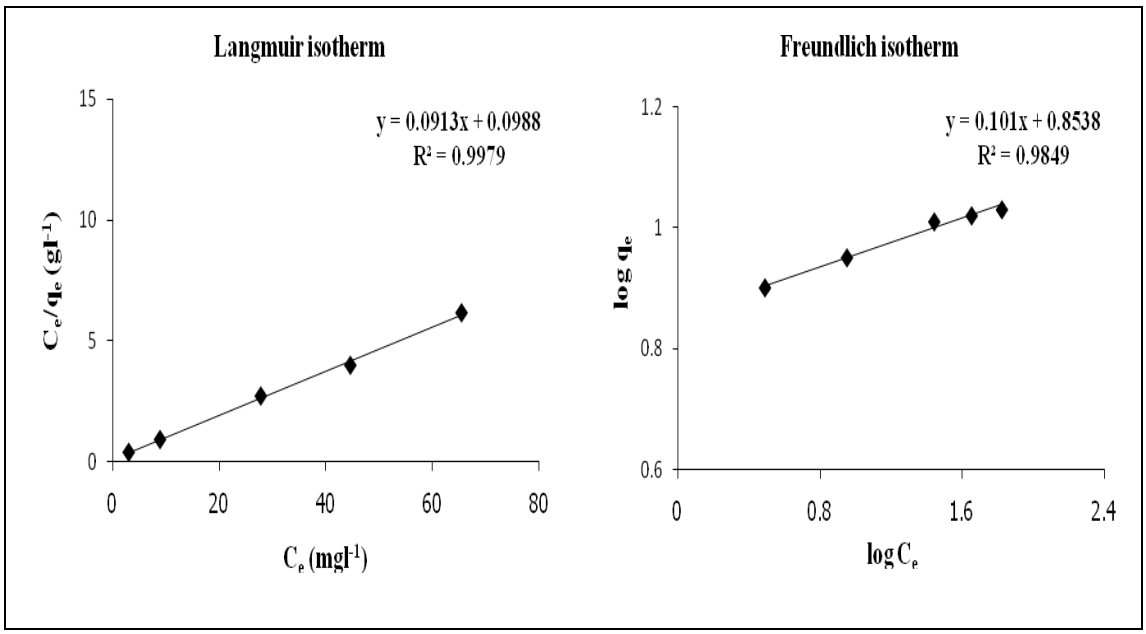


Figure 45 Langmuir and Freundlich adsorption isotherm of methylene blue onto BAWP 3.

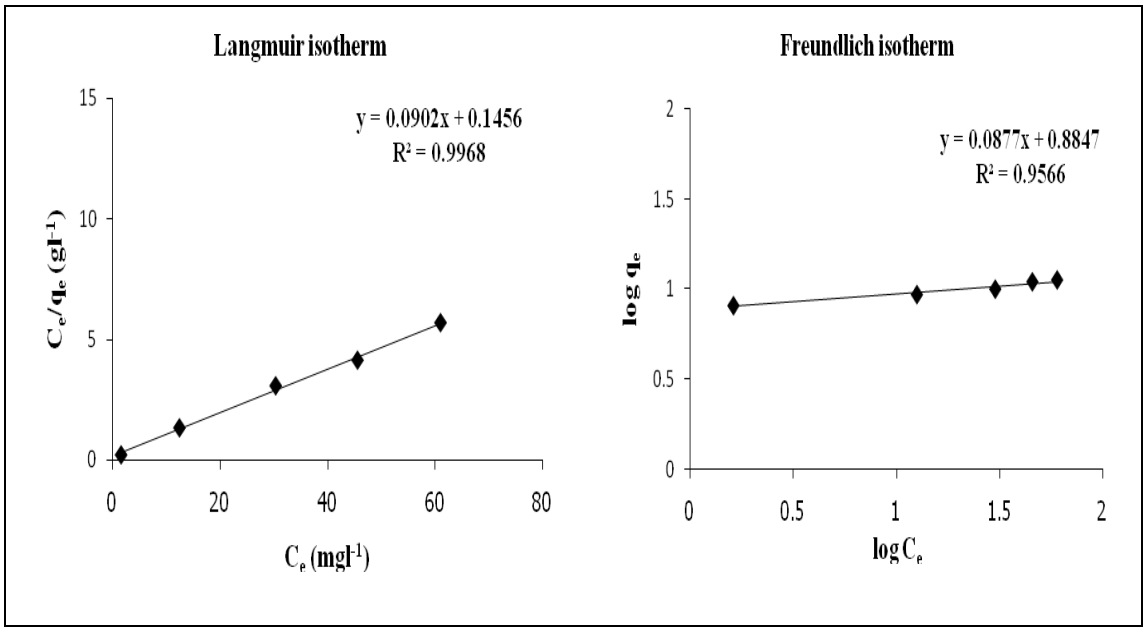


Figure 46 Langmuir and Freundlich adsorption isotherm of methylene blue onto BAWK 1.

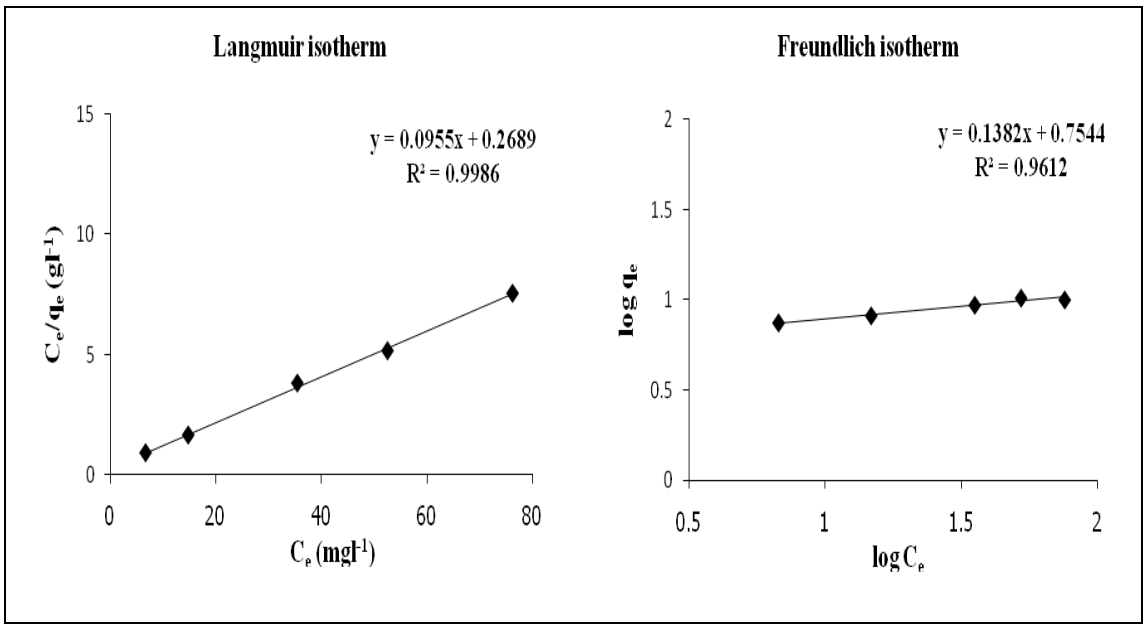


Figure 47 Langmuir and Freundlich adsorption isotherm of methylene blue onto GOC 1.

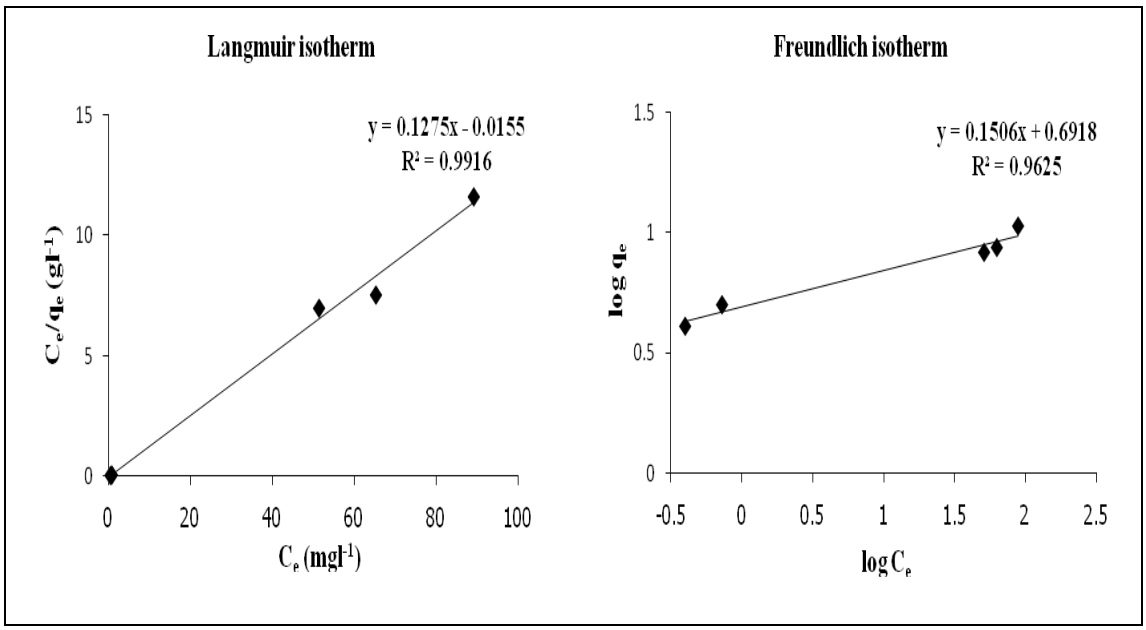


Figure 48 Langmuir and Freundlich adsorption isotherm of methylene blue onto GOP 3.

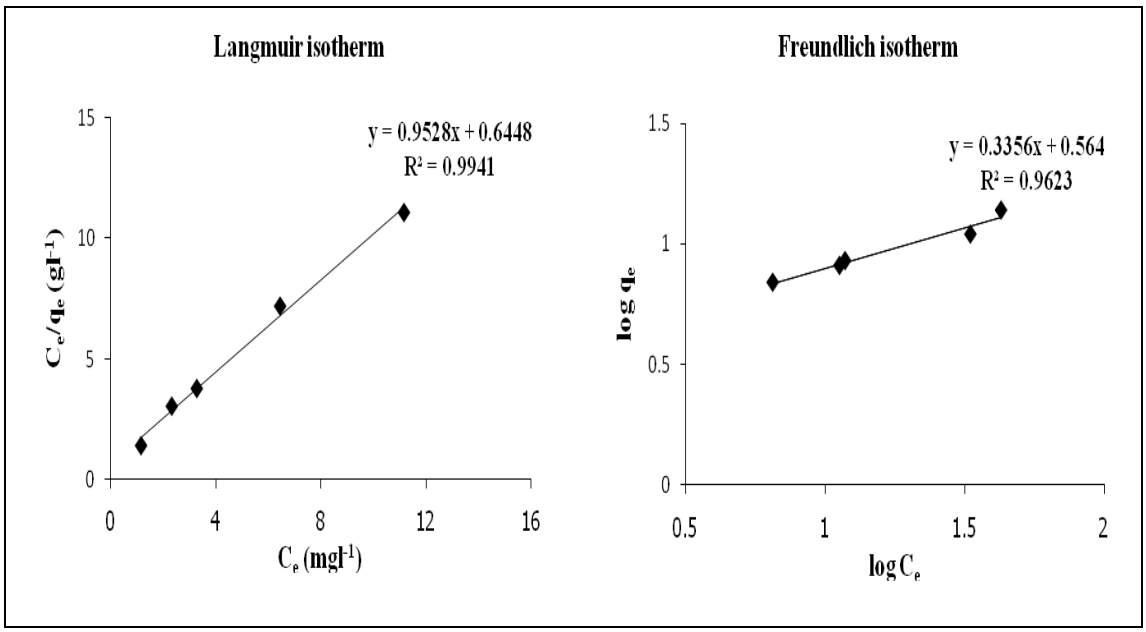


Figure 49 Langmuir and Freundlich adsorption isotherm of methylene blue onto GOK 2.

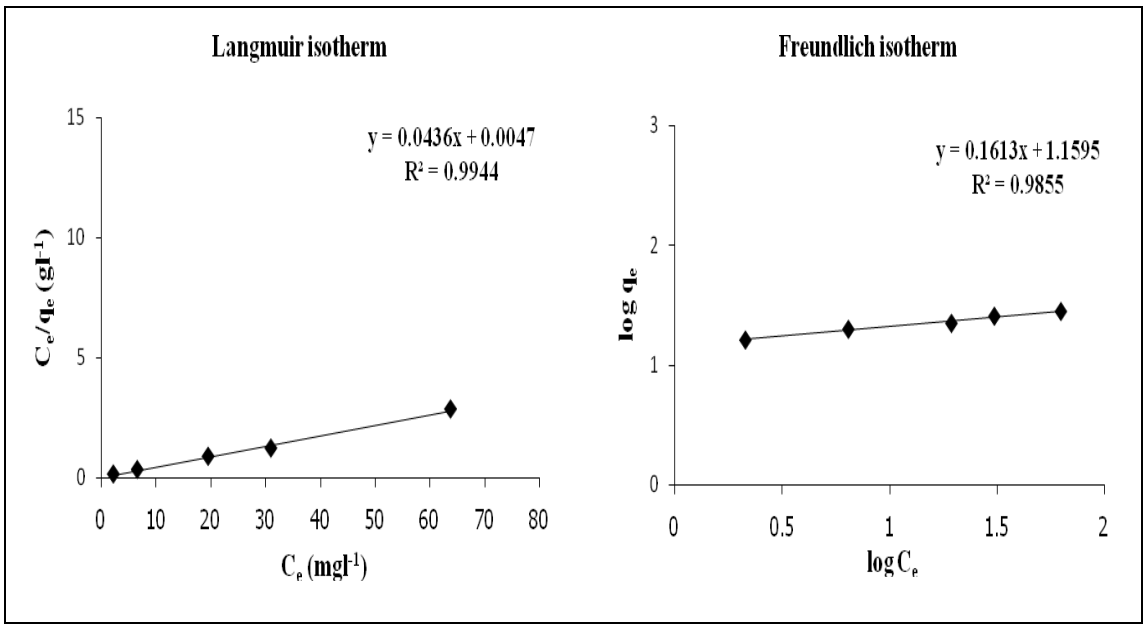


Figure 50 Langmuir and Freundlich adsorption isotherm of methylene blue onto GVC 1.

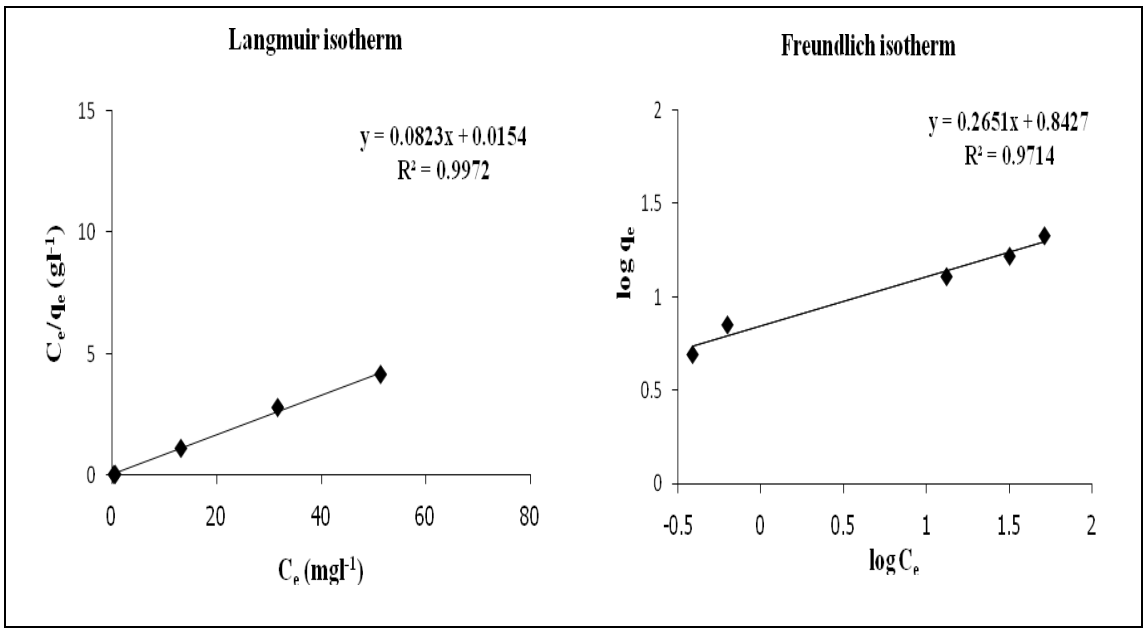


Figure 51 Langmuir and Freundlich adsorption isotherm of methylene blue onto GVP 2.

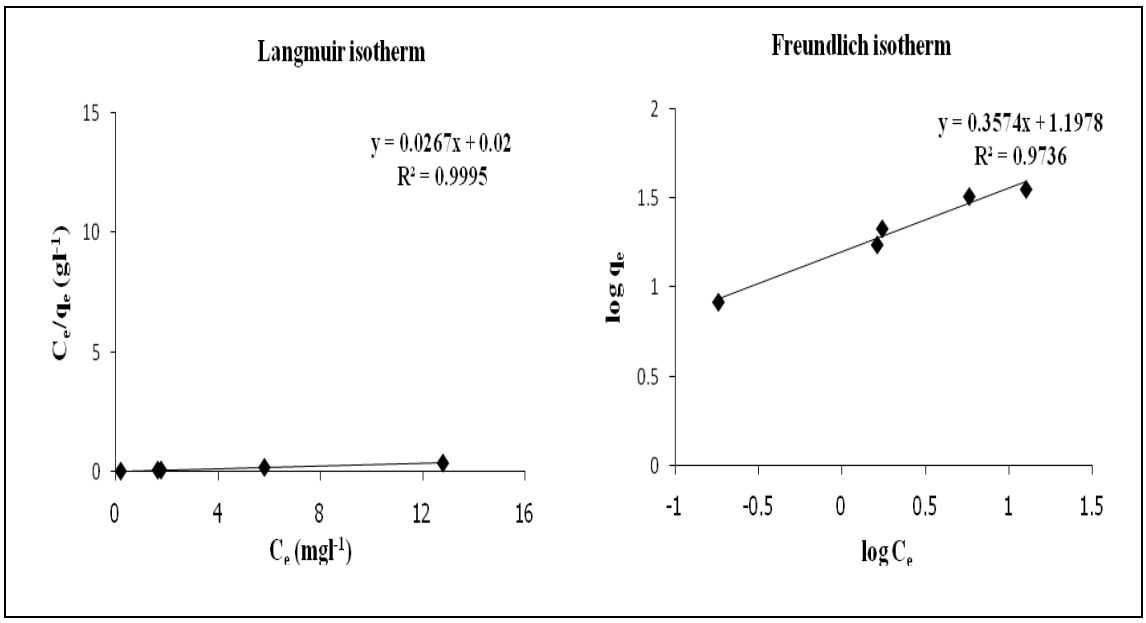


Figure 52 Langmuir and Freundlich adsorption isotherm of methylene blue onto GVK 1.

The Langmuir and Freundlich adsorption isotherm parameters for methylene blue adsorption of activated carbon were given in Table 20. It could be implied that the sorption data were better fitted with Langmuir adsorption isotherm model than Freundlich model according to R^2 values. Therefore, the type of adsorption was monolayer coverage of dye molecules onto surface of activated carbon and bamboo charcoal. The Langmuir constants, GVK 1 had the highest Q_o value (37.48 mg/g), compared to activated carbon, bamboo charcoal and Bunton. However, the Q_o value of GVK 1 was lower than that of Fluka (333.33 mg/g). Furthermore, the b value of GVC 1 was highest, indicated that the GVC 1 had highest adsorption energy.

Table 20 Langmuir and Freundlich isotherm constants for methylene blue**.**

Sample	Langmuir isotherm			Freundlich isotherm		
	Q_o (mg/g)	b (L/mg)	R^2	1/n	K	R^2
Fluka [a]	333.33	0.750	0.992	0.172	175.27	0.984
Bunton [b]	15.29	0.512	0.995	0.072	10.80	0.990
BAWC 1	13.99	0.505	0.999	0.176	7.08	0.957
BAWP 3	10.95	0.924	0.998	0.101	7.13	0.984
BAWK 1	11.09	0.619	0.997	0.087	7.66	0.956
GOC 1	10.47	0.355	0.999	0.138	5.68	0.961
GOP 3	7.84	8.230	0.992	0.150	4.91	0.962
GOK 2	1.05	1.480	0.994	0.335	3.66	0.962
GVC 1	22.94	9.270	0.994	0.161	14.42	0.985
GVP 2	12.15	5.340	0.997	0.265	6.95	0.971
GVK 1	37.45	1.340	0.999	0.357	15.74	0.973

[a] Activated carbon was produced by Fluka Company.

[b] Bamboo charcoal was produced by Thai Agard Dee Bamboo Charcoal Products.

Table 21 The specific surface area of samples for adsorption of methylene blue**.**

Sample	Specific surface area (m^2/g)
Fluka	752.84
Bunton	34.53
BAWC 1	31.60
BAWP 3	24.73
BAWK 1	25.05
GOC 1	23.65
GOP 2	17.71
GOK 3	2.37
GVC 1	51.81
GVP 2	27.44
GVK 1	84.58

According to the Langmuir isotherm in case of methylene blue, this pointed out the fact that the methylene blue adsorption was the mono-layered adsorption, in which the specific surface area could be calculated by replacing the Q_o value as shown in Table 21 on the equation 12. The obtained specific surface area of all activated carbon and bamboo charcoal were in the range of 2-85 m^2/g. The specific surface area value reflected the methylene blue adsorption ability of the activated carbon, in that, the higher this value, the more adsorption ability. Also, the multi-layered adsorption had an impact on the adsorption ability of the GVK 1. Therefore, the GVK 1 provided the adsorption ability of methylene blue similar to the Fluka.

5.4 Cd (II) adsorption

5.4.1 Cd (II) value

Table 22 The Cd (II) value of activated carbon and bamboo charcoal for adsorption ability of Cd (II).

Activated carbon	Cadmium value (mg/g)
BAWC 1	0.35±0.08
BAWP 1	0.43±0.08
BAWK 1	0.36±0.04
GOC 3	0.28±0.03
GOP 3	0.44±0.07
GOK 3	0.47±0.06
GVC 1	0.47±0.06
GVP 2	0.41±0.02
GVK 1	0.48±0.06

Table 22 showed the Cd (II) value of activated carbon, bamboo charcoal and Fluka for adsorption ability of Cd (II). It could be seen that the Cd (II) value of activated carbon was increased, compared to charcoal. This might be attributed to the preparation of activated carbon by chemical activation. Therefore, the surface on activated carbon was negative due to the increasing of C=O or COO^- functional groups on surface of activated carbon. Additionally, the Cd (II) ions were positive resulting in the attraction between negative and positive charges. For all type of bamboo, they were used in preparation for activated carbon. The results indicated that the types of bamboo seemed to be a minor effect on adsorption of Cd (II) onto activated carbon due to the same components of each types of bamboo.

5.4.2 Adsorption isotherms of Cd (II) onto activated carbon and bamboo charvoal.

Figures 53-61 showed the Langmuir plots of C_e/ q_e versus C_e and Freundlich plots of log q_e versus log C_e for the adsorption of Cd (II) onto the activated carbon.

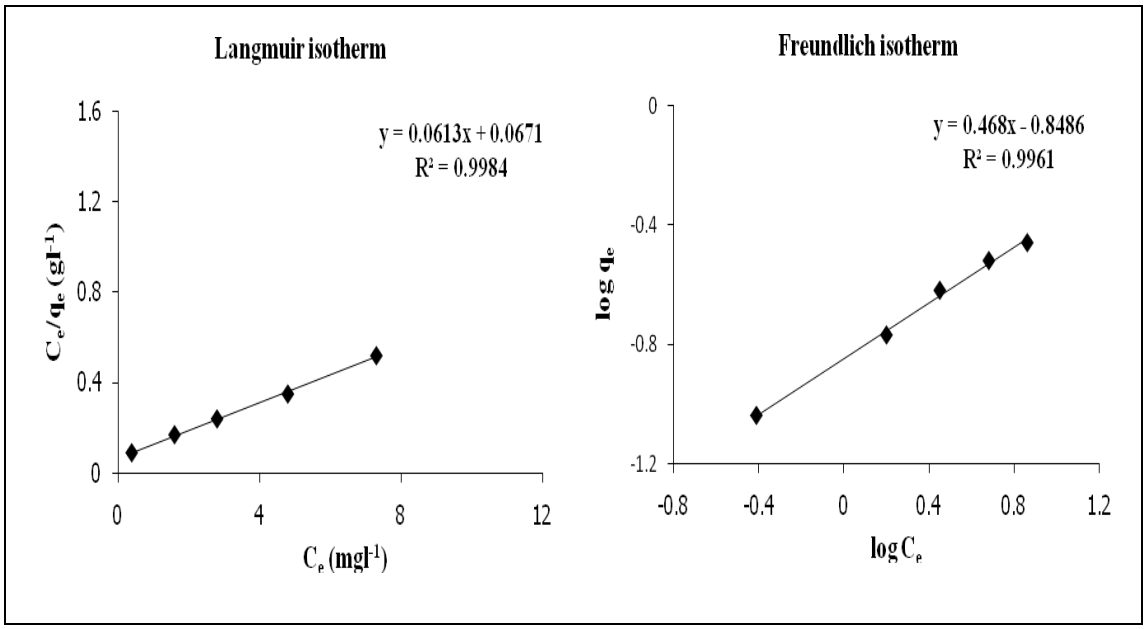


Figure 53 Langmuir and Freundlich adsorption isotherm of Cd (II) onto BAWC 1.

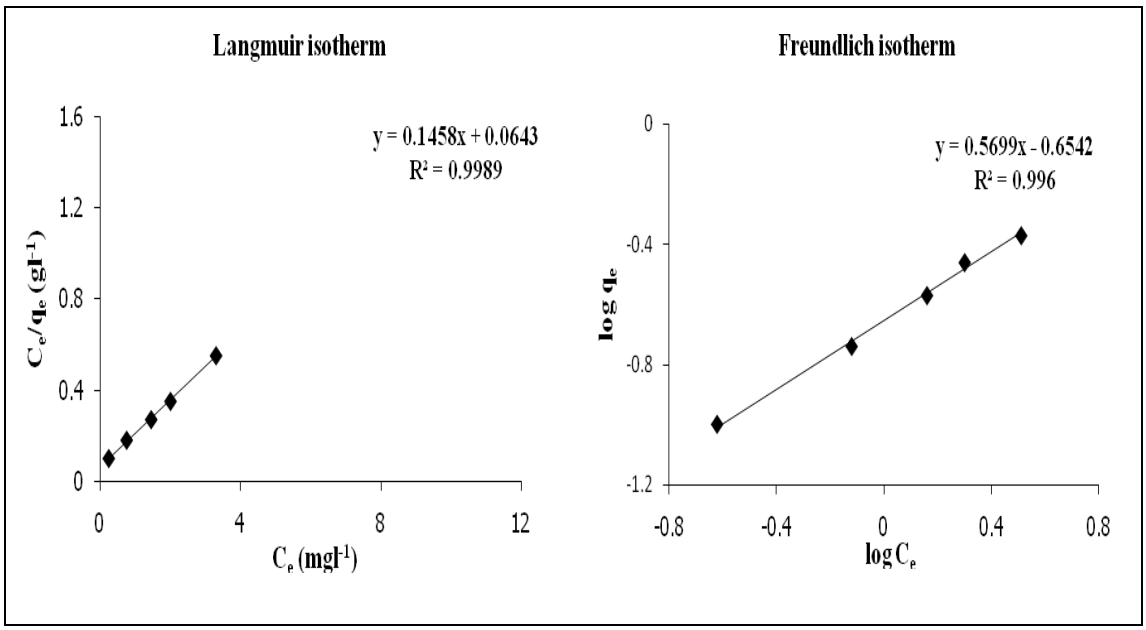


Figure 54 Langmuir and Freundlich adsorption isotherm of Cd (II) onto BAWP 1.

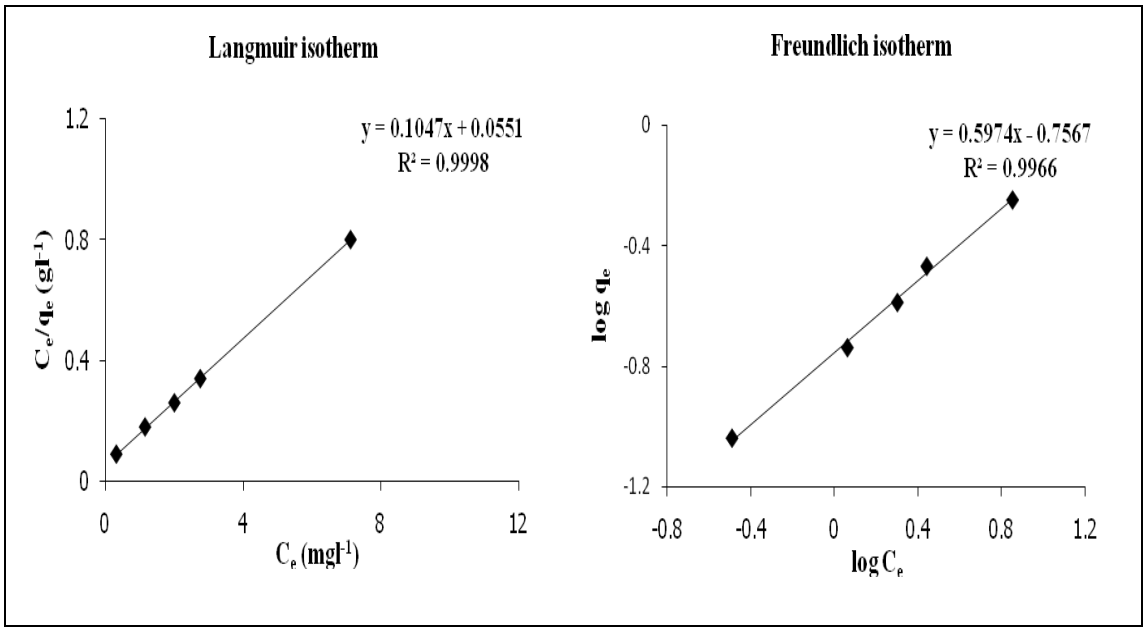


Figure 55 Langmuir and Freundlich adsorption isotherm of Cd (II) onto BAWK 1.

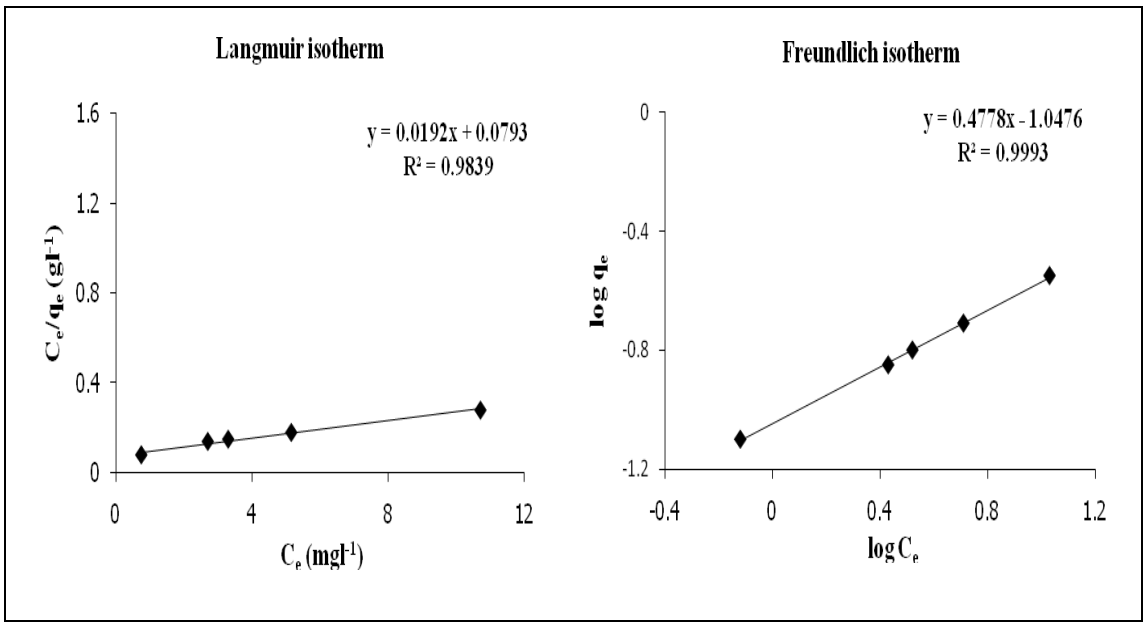


Figure 56 Langmuir and Freundlich adsorption isotherm of Cd (II) onto GOC 3.

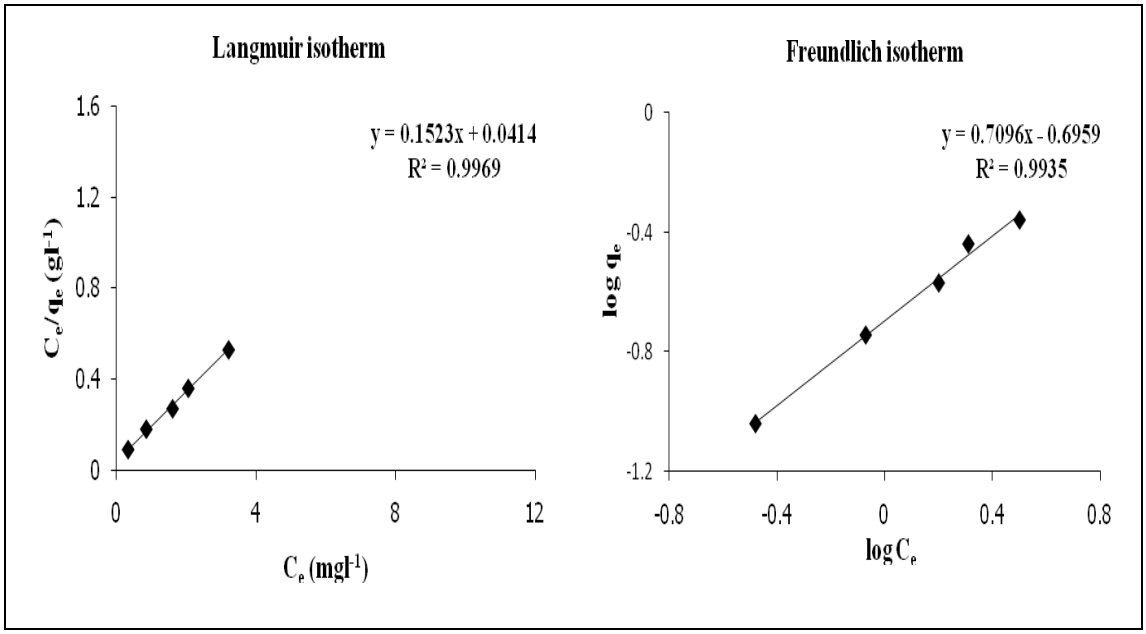


Figure 57 Langmuir and Freundlich adsorption isotherm of Cd (II) onto GOP 3.

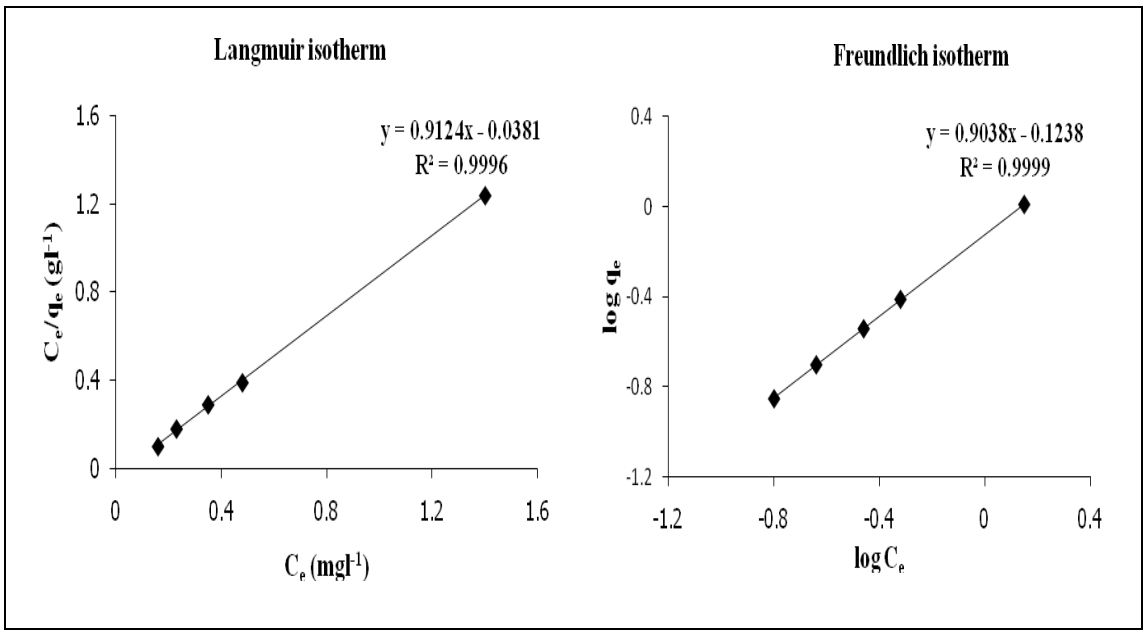


Figure 58 Langmuir and Freundlich adsorption isotherm of Cd (II) onto GOK 3.

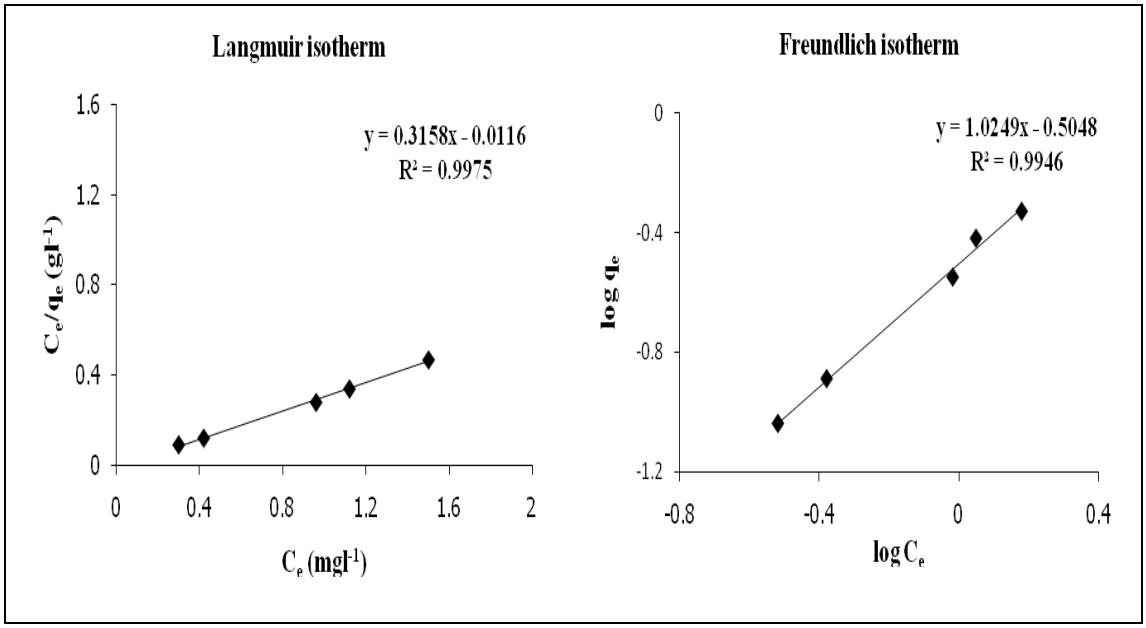


Figure 59 Langmuir and Freundlich adsorption isotherm of Cd (II) onto GVC 1.

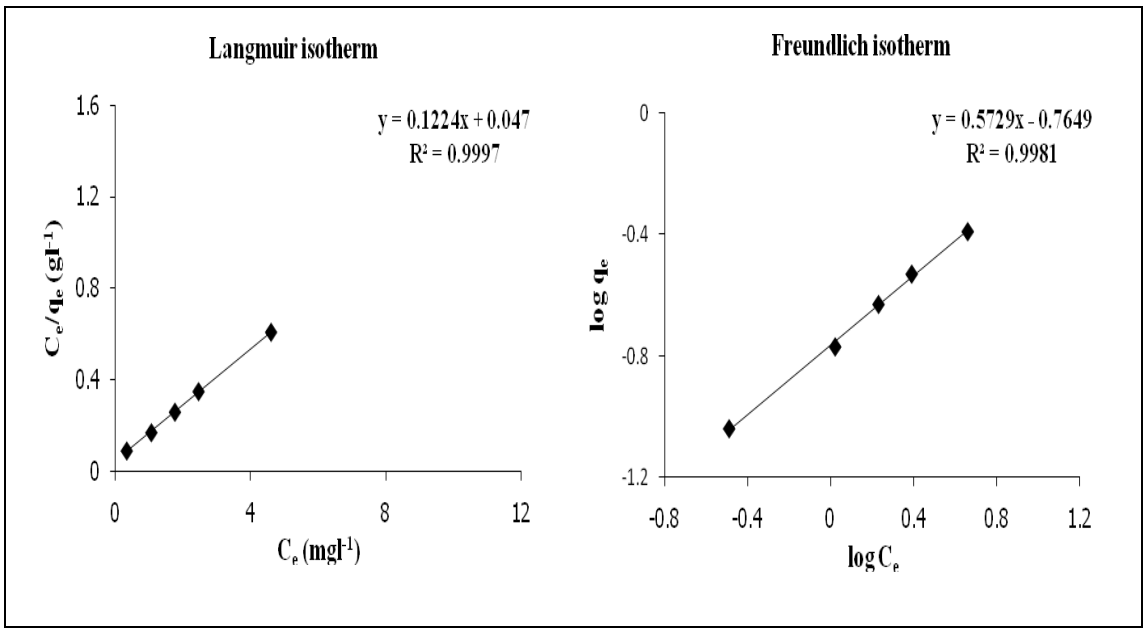


Figure 60 Langmuir and Freundlich adsorption isotherm of Cd (II) onto GVP 2.

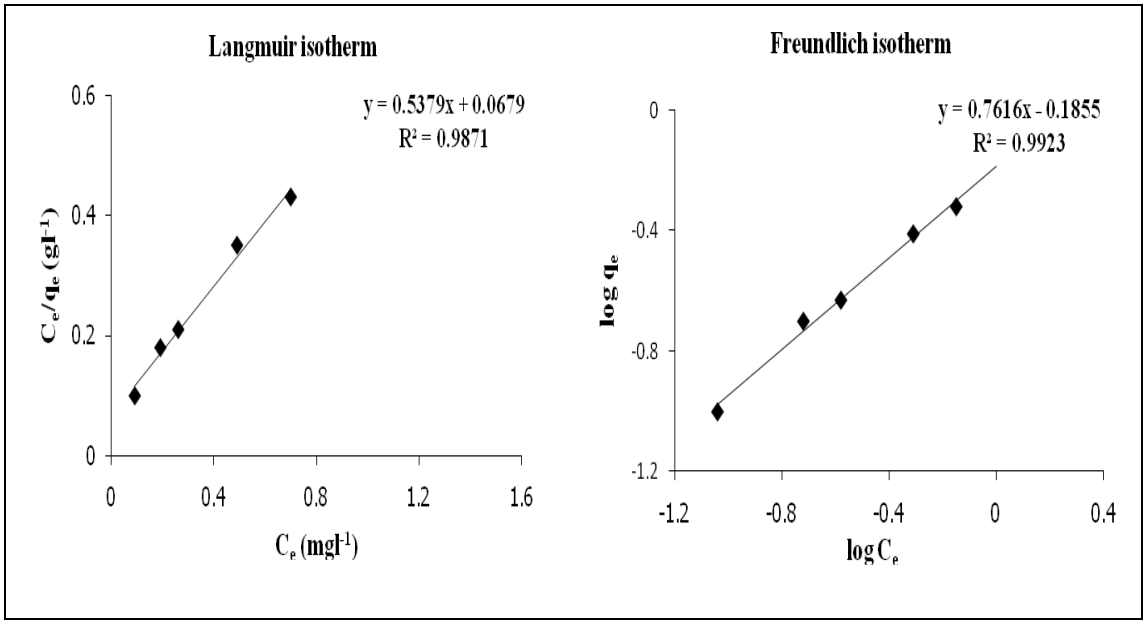


Figure 61 Langmuir and Freundlich adsorption isotherm of Cd (II) onto GVK 1.

The Langmuir and Freundlich isotherm parameters for Cd (II) adsorption of activated carbon were given in Table 20. For activated carbon and bamboo charcoal, the fitting to Langmuir adsorption isotherm model was similar in comparison to Freundlich model according to R^2 values. Therefore, the type of adsorption could be either monolayer or multilayer coverage of iodine molecules onto surface of activated carbon and bamboo charcoal. On the other hand, the sorption data of Fluka and Bunton were better fitted by the Langmuir adsorption isotherm model than the Freundlich model based on R^2 values. The Langmuir constant of the GOC 3 that had the highest Q_o value was 52.63 mg/g. Furthermore, the b value of Fluka was higher than that of activated carbon and bamboo charcoal, showing that the Fluka had the higher adsorption energy. The Freundlich constants showed that all 1/n value were in the range of 0-1 and indicated favorable multilayer adsorption of Cd (II). Furthermore, the K_F value of Fluka and Bunton were higher than that of activated carbon and bamboo charcoal, illustrating that the Fluka and Bunton had high adsorption capacity.

Table 23 Langmuir and Freundlich isotherm constants for Cd (II).

Sample	Langmuir isotherm			Freundlich isotherm		
	Q_o (mg/g)	b (L/mg)	R^2	1/n	K	R^2
Fluka[a]	0.12	23.072	1.000	0.136	13.84	0.784
Bunton[b]	0.08	5.210	0.999	0.142	8.57	0.782
BAWC 1	16.39	0.911	0.998	0.468	7.05	0.996
BAWP 1	6.90	2.264	0.998	0.569	4.51	0.966
BAWK 1	9.62	1.890	0.999	0.597	5.70	0.996
GOC 3	52.63	0.241	0.983	0.477	11.14	0.999
GOP 3	6.58	1.000	0.996	0.709	4.95	0.993
GOK 3	1.10	0.997	0.999	0.903	1.33	0.999
GVC 1	3.17	1.000	0.997	1.024	3.19	0.994
GVP 2	8.20	1.000	0.999	0.572	5.81	0.998
GVK 1	1.86	8.024	0.987	0.761	1.53	0.992

[a] Activated carbon was produced by Fluka Company.

[b] Bamboo charcoal was produced by Thai Agard Dee Bamboo Charcoal Products.

Table 24 The specific surface area of samples for adsorption of Cd (II)**.**

Sample	Specific surface area (m^2/g)
Fluka	0.39
Bunton	0.26
BAWC 1	53.54
BAWP 1	22.54
BAWK 1	31.42
GOC 3	171.93
GOP 3	21.50
GOK 3	3.59
GVC 1	10.36
GVP 2	26.79
GVK 1	6.08

According to the Langmuir isotherm in case of methylene blue, this pointed out the fact that the Cd (II) adsorption was the mono-layered adsorption, in which the specific surface area could be calculated by replacing the Q_o value as shown in Table 24 on the equation 12. The obtained specific surface area of all activated carbon and bamboo charcoal were in the range of 2-85 m^2/g. Nevertheless, the GVK 1 had the highest adsorption ability of Cd (II) even though it had the low specific surface area.

6. Study of the concentration of organic acids in wood vinegar obtained from *Bambusa arundinacea (Retz) Wild, Bambusa oldhamii* and *Gigantochloa verticiliata* by chemical activation.

From the analysis of the amount of nine-organic acid components in wood vinegar by HPLC, it was typically found that the concentration of nine organic acids was dependable on types and ages of bamboo as shown in Table 21. Acetic acid, one of the nine organic acids, had the highest concentration in all kinds of wood vinegar because it mainly originated from the thermal decomposition of cellulose which was the principal composition of bamboo. The thermal reaction was shown in equation 17. Moreover, the order of the amount of organic acid that was found was acetic acid > butyric acid > fomic acid > isobutyric acid > propionic acid ~ citric acid ~ tartaric acid > malic acid > lactic acid.

$$(C_6H_{12}O_5)_n + nH_2O \rightarrow nC_6H_{12}O_6 \rightarrow 2nCH_3CH_2OH + 2nCO_2 \rightarrow 2nCH_3COOH \quad \text{------ (17)}$$

Source: Wade (2003)

In this work, there were two different methods used to analyse the amount of nine-organic acids in wood vinegar as elaborated in the section 4.2. The results in Table 22 showed that the detectable amount of nine organic acids was not quite different. However, there was still something different, especially the analytical time. In the first method, wood vinegar did not pass through the solid phase extraction process in order to remove unwanted non-polar organic molecules prior to entering into the reverse phase HPLC column. It, therefore, took a long time to clean the column after used because there were the non-polar organic molecules adsorbed in the column at the high extent. On the contrary, in terms of the second method, wood vinegar needed to pass the solid extraction process. Hence, the non-polar organic molecules were annihilated and only polar molecules, especially nine organic acids; were able to pass further into the HPLC column. Therefore, the time for cleaning the column was less than that of the first method.

Regarding the selection of wood vinegar in its application, the amount of each organic acid was the main factor to select the most applicable wood vinegar. For example, the wood vinegar produced from 1-year GV indicated that there was a large amount of formic acid which can be used in term of insecticides. Additionally, in wood vinegar, there was a large amount of butyric acid which derivative of butyric acid as 4-(2,4-dichlorophenoxy)-butyric acid which can be used in term of perticides. In other word, in case of wood vinegar obtained from 2-year GV, isobutyric acid was the main composition with a large quantity, which could be implemented as a solvent in laboratory research.

Table 25 The amount of nine organic acid compounds in the wood vinegar.

Type of bamboo	years	Amount of organic acids (ppm)								
		Citric acid	Tartaric acid	Malic acid	Lactic acid	Fomic acid	Acetic acid	Propionic acid	Isobutyric acid	Butyric acid
BAW	1	10,900	6,123	5,965	4,080	46,404	164,929	10,618	45,467	92,498
	2	12,314	11,672	9,966	2,644	52,383	268,691	12,696	41,877	138,442
	3	8,650	10,054	1,880	2,157	50,517	273,409	18,135	44,780	123,547
GO	1	12,689	13,162	8,960	2,148	33,426	179,332	10,829	26,934	100,419
	2	8,622	9,188	7,952	2,276	38,537	181,872	16,479	31,107	89,465
	3	9,972	107,277	8,246	16,757	33,433	166,801	17,562	28,795	106,927
GV	1	11,501	12,416	15,625	9,645	93,306	323,915	19,631	57,645	146,980
	2	8,292	8,569	12,109	3,990	34,540	175,283	12,436	255,113	106,732
	3	9,940	7,505	9,588	-[a]	71,723	199,626	11,761	22,980	59,514

[a] Lower than detection limit.

Table 26 The amount of nine organic acid compounds in the wood vinegar which passed through SEP.

Type of bamboo	years	Amount of organic acids (ppm)								
		Citric acid	Tartaric acid	Malic acid	Lactic acid	Fomic acid	Acetic acid	Propionic acid	Isobutyric acid	Butyric acid
BAW	1	12,578	5,503	11,163	49,999	22,502	179,701	22,744	24,294	85,333
	2	15,174	5,391	22,074	13,034	64,603	333,912	20,187	18,167	80,042
	3	11,768	4,418	10,329	10,374	43,038	277,873	27,421	25,135	119,296
GO	1	10,893	5,423	6,173	57,974	47,754	183,961	17,374	15,873	75,023
	2	9,198	4,684	3,788	8,722	35,518	187,952	25,208	18,905	71,241
	3	10,347	12,814	6,189	38,949	14,520	166,475	18,910	8,591	103,067
GV	1	10,488	9,263	9,403	13,580	69,212	340,321	31,513	45,834	107,321
	2	8,446	8,216	4,580	31,846	16,855	198,858	21,341	17,834	78,369
	3	8,653	6,386	2,799	34,661	34,504	224,899	20,932	17,506	43,410

CONCLUSION AND RECOMMENDATION

Conclusion

From the result of proximate analysis properties, it could be seen that the bamboo charcoal and activated carbon had a higher amount of ash and volatile matters but a lower amount of fixed carbon than the commercial activated carbon. This indicated that there were organic content and volatile matters in a high extent. Moreover, from the SEM result, the surface morphology of the activated carbon prepared from BAW, GO and GV had higher number of pores when the charcoals were activated by H_3PO_4 or KOH. The increase of a number of pores resulted in the increasing of the adsorption surface area. However, the pore size of the activated carbon should have the appropriate size for each of the adsorbate as well. Nevertheless, the activation by H_3PO_4 or KOH brought about the variation of functional groups on the activated carbon resulting from oxidation reaction, which was able to be seen in the IR results at 1730-1650 cm^{-1} (C=O) and 1200-1050 cm^{-1} (C-O). The occurrence of COO^- on the activated carbon surface had an impact on the adsorption efficiency. On the contrary, the bamboo charcoal showed the absence of such these IR peaks.

In terms of the efficiency of iodine adsorption, the activated charcoal provided higher iodine number than non-activated charcoal, in particular the 1-year-GV charcoal activated by KOH (20 %w/v) for 4 hours. Moreover, this as-prepared activated charcoal had higher I_2 adsorption efficiency than the Fluka commercial activated carbon. As to the efficiency of phenol adsorption by the activated carbon, determined by the phenol value, the 1-year-BAW charcoal had the lowest phenol value, which indicated the higher phenol adsorption efficiency, when comparing with other as-prepared activated/non-activated carbon, including Bunton commercial activated carbon. However, it had lower phenol adsorption efficiency than the Fluka commercial activated carbon. Regarding to the methylene blue adsorption efficiency, determined by the methlene blue value, activating the charcoal had few effects on the adsorption efficiency. However, the 1-year-GV charcoal activated by KOH (20 %w/v)

for 4 hours gave rise to the highest methylene blue value compared to the other as prepared charcoal, which was the same efficiency as the Fluka commercial activated carbon. Besides, in case of Cd (II) adsorption efficiency, determined by the cadmium (II) value, the activated charcoal by H_3PO_4 or KOH gave the higher cadmium (II) value than non-activated charcoal, especially the 1-year-GV charcoal activated by KOH (20 %w/v) for 4 hours. Furthermore, when considering the adsorption isotherm, three kinds of the adsorbates such as iodine, phenol and Cd (II) provided both Langmuir and Freundlich isotherms. Whilst the methylene blue adsobate had also the adsorption which could be fitted with the Langmuir isotherm better than the Freundlich isotherm.

The activated carbon which was prepared from GV and activated by KOH had a large number of micropores due to the high adsorption ability of iodine. Meanwhile, some as-prepared activated carbon preferred to adsorb methylene blue because they provided mesopores with a high extent. Moreover, the negative surface of activated carbon improved the adsorption of Cd (II). Nevertheless, the presence of C=O and COO^- groups on the activated carbon surface after activation brought about the decreasing of phenol adsorption ability. Therefore, the preparation of activated carbon for the best quality succeeded in using the 1-year GV and KOH which was the best chemical reagent for activation.

In addition, the concentration of organic acids in wood vinegar was subject to type and age of bamboo. Acetic acid was found with the highest concentration in organic acid. The solid phase extraction succeded in eliminating non-polar organic molecules in wood vinegar and the time used to clean column was reduced even though the detected amount of nine organic acids did not differ from the absence of solid phase extraction.

Recommendation

1. In the carbonization process of bamboo, inert or N_2 atmosphere should be adopted instead of O_2 atmosphere because it will expectedly decrease the amount of ash originating from the oxidation of bamboo but provide the larger amount of charcoal.

2. Temperature or pH of solution should be considered because they might have the impacts on the adsorption abilities of each adsorbate.

3. The used activated carbon for adsorption should be cleaned and reused in order to study the adsorption ability of activated carbon after the first-time usage.

4. In the real situation, the activated carbon is applied to use in stagnant water because some molecules have effects on the adsorption ability of activated carbon.

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APPENDICES

APPENDIX A

Standard Test Method for Moisture of Activated Carbon

ASTM D 2867 - 04

Designation: D 2867 – 04

Standard Test Methods for Moisture in Activated Carbon[1]

This standard is issued under the fixed designation D 2867; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods provide two procedures for the determination of the moisture content of activated carbon. The procedures may also be used to dry samples required for other tests. The oven-drying method is used when water is the only volatile material present and is in significant quantities, and the activated carbon is not heat-sensitive (some activated carbons can ignite spontaneously at temperatures as low as 150°C). The xylene-extraction method is used when a carbon is known or suspected to be heat sensitive or to contain nonwater-miscible organic compounds instead of or in addition to water. The oven-drying method described in these test methods may be used as the reference for development of instrumental techniques for moisture determination in activated carbon.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Summary of Test Methods

3.1 *Oven-Drying Test Method*—A sample of carbon is put into a dry, closed capsule (of known weight) and weighed accurately. The capsule is opened and placed with the lid in a preheated oven. The sample is dried to constant weight then removed from the oven and with the capsule closed, cooled to ambient temperature. The closed capsule is weighed again accurately. The weight loss is expressed as a percentage of the weight of the original sample.

3.2 *Xylene-Extraction Test Method*— A known, accurate weight of carbon is put into a boiling flask. A known volume of xylene is added to the flask and the flask then connected to a water trap. A hot plate is used to heat the xylene until boiling. The temperature is controlled to allow steady reflux. Reflux continues until no further water can be collected in the trap. The weight of water collected is expressed as a percentage of the weight of the original sample.

4. Significance and Use

4.1 The moisture content of activated carbon is often required to define and express its properties in relation to the net weight of the carbon.

4.2 The moisture content of activated carbon packed in typical shipping containers will usually increase during transportation and storage. Users of activated carbon in applications where low moisture content is important should be aware of this effect.

OVEN-DRYING METHOD

5. Apparatus

5.1 *Moisture Oven*—Most commercial, electrically heated, forced-circulation drying ovens capable of temperature regulation between 145 and 155°C may be used.

5.2 *Capsules with Covers*—Low-form glass weighing bottles with ground-glass stoppers or seamless metal boxes with covers may be used. They should be as shallow as possible, consistent with convenient handling.

5.3 *Desiccator*.

6. Materials

6.1 *Desiccant*—Anhydrous calcium chloride or other suitable desiccant.

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[1] These test methods are under the jurisdiction of ASTM Committee D28 on Activated Carbon and are the direct responsibility of Subcommittee D28.04 on Gas Phase Evaluation Tests.

Current edition approved April 1, 2004. Published May 2004. Originally approved in 1970. Last previous edition approved in 1999 as D 2867 – 99.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

7. Procedure for Activated Carbon Passing A No. 50 Sieve

7.1 Dip out with a spoon or spatula from the sample bottle a 1 to 2-g representative sample. Put this into a predried tared capsule with lid, close and weigh at once to the nearest 0.5 mg. The depth of the carbon in the capsule must not exceed 1.25 cm.

7.2 Remove the cover and place the capsule and cover in a preheated forced circulation oven (at 145 to 155°C). Close the oven and dry to constant weight (3 h normally sufficient). Open the oven and cover the capsules quickly. Cool in a desiccator to ambient temperature and weigh.

8. Procedure for Activated Carbon Larger Than A No. 50 Sieve

8.1 Use a 5 to 10-g representative sample and weigh to the nearest 2 mg. Complete the determination as described in Section 7.

9. Calculation

9.1 Calculate the moisture content as follows:

$$\textit{Moisture, weight \%} = [(C - D) / (C - B)] \times 100$$

where:

B = weight of capsule with cover, g,
C = weight of capsule with cover plus original sample, g, and
D = weight of capsule with cover plus dried sample, g.

XYLENE–EXTRACTION METHOD

10. Apparatus

10.1 *Boiling Flask*—A 300-mL flat-bottom Erlenmeyer flask with ground-glass joints.

10.2 *Condenser*—A 300-mm water-cooled condenser of the Allihn type with ground-glass joints.

10.3 *Drying Tube*, containing a suitable desiccant with fiber-glass filter.

10.4 *Water Trap*—A Bidwell and Sterling 10-mL or a Dean and Stark receiver with ground-glass joints. The water trap should be clean so that the shape of the meniscus at the end of the test is the same as at the beginning.

NOTE 1—The trap may be coated with a silicone resin to give a uniform meniscus. To coat the trap, first clean it with a suitable cleaner. Rinse the clean trap with a silicone resin and after draining for a few minutes, bake for 1 h at approximately 200°C.

10.5 *Hot Plate*—An electrically heated hot plate with enclosed elements and temperature control.

11. Reagent

11.1 *Xylene*—Reagent grade in accordance with the specifications of the Committee on Analytical Reagents of the American Chemical Society.[3]

12. Hazards

12.1 The use of hot xylene presents a continual fire hazard and suitable fire extinguishing equipment should be available.

13. Preparation of Apparatus

13.1 Clean the condenser, flask, and trap and carefully dry to ensure that it is free of water. Assemble the condenser and water trap as shown in Fig. 1.

14. Procedure

14.1 Weigh the sample bottle. Dip out with a spoon from the sample bottle 25 to 50 g of the sample. Put this into the boiling flask and reweigh the sample bottle to the nearest 0.1 g. Add 100 mL of xylene and connect the boiling flask to the water trap. For carbons having density less than 0.30 g/cm ^{3}, 200 mL of xylene should be used for a 25-g sample.

14.2 Place the hot plate under the boiling flask and heat to boiling. Adjust the temperature control so as to reflux the xylene at the rate of about 1 drop/s from the tip of the condenser. Continue to reflux until there is no further increase in the water layer in the trap over a 30-min period (from 2 to 8 h may be required).

15. Calculation

15.1 Calculate the moisture content as follows:

$$\textit{Moisture, weight \%} = [V / (C - E)] \times 100$$

where:

V = water collected, mL,
C = initial weight of sample bottle, g, and
E = weight of sample bottle after removing moisture sample, g.

15.2 Calculate for the correction for moisture in carbon to determine the weight of a carbon sample on the dry basis as follows:

$$\text{Corrected weight (dry basis)} = \frac{\text{Initial weight of Carbon(undried)} \times (100\% - \%\ \text{moisture from 15.1})}{(100\%)} \quad (1)$$

[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

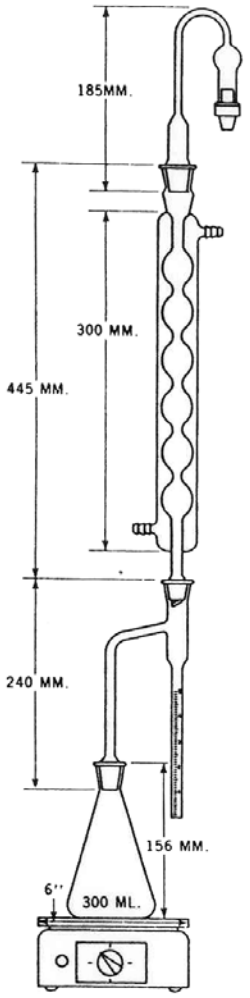

FIG. 1 Moisture Determination Apparatus

16. Precision and Bias

16.1 An interlaboratory test which included four laboratories testing in triplicate, activated carbon samples with nominal moisture levels of 1 weight %, 5 weight %, and 12 weight %, was conducted according to Practice E 691. Results of these tests yielded repeatability and reproducibility coefficients at 95 % confidence levels as listed in Table 1.

16.1.1 The high coefficients of variation for both test methods when the moisture is nominally 1 weight % suggests that values in this range be reported as "1 weight % or less." If greater precision is needed, the amount of carbon sample can be increased over that specified in Sections 7, 8, and 14.

17. Keywords

17.1 activated carbon; moisture

TABLE 1 Within- and Between-Laboratory Precision

Nominal Moisture, weight %	1	5	12
Oven Drying Method			
Repeatability Coefficient, $CV\%_r$[A]	19	3	6
Reproducibility Coefficient, $CV\%_R$[A]	51	13	10
Xylene Extraction Method			
Repeatability Coefficient, $CV\%_r$[A]	51	5	6
Reproducibility Coefficient, $CV\%_R$[A]	54	6	6

[A] Defined in Practice E 177, Section 28.

APPENDIX B

Standard Test Method for Total Ash Content of Activated Carbon

ASTM D 2866 - 94

Designation: D 2866 – 94 (Reapproved 2004)

Standard Test Method for Total Ash Content of Activated Carbon[1]

This standard is issued under the fixed designation D 2866; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes a procedure for the determination of total ash content of activated carbon.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 2867 Test Methods for Moisture in Activated Carbon

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E 691 Practice for Conducting an Interlaboratory Study to Determine Precision of a Test Method

3. Summary of Test Method

3.1 An accurately weighed sample of dried activated carbon is placed in a controlled-temperature muffle furnace for a period of several hours. When constant weight has been achieved, the crucible is cooled to ambient temperature in a desiccator and reweighed. The weight of the ashed carbon is expresesed as a percentage of the weight of the original carbon sample.

4. Significance and Use

4.1 In specific end uses, the amount and composition of the ash may influence the capabilities and certain desired properties of activated carbon.

5. Apparatus

5.1 *Muffle Furnace*, having air circulation, capable of temperature regulation of ±25°C at 650°C.

5.2 *High-Temperature Crucible*, high-form.

5.3 *Analytical Balance*, having a sensitivity of 0.1 mg.

5.4 *Desiccator.*

5.5 *Oven*, forced-air circulation, capable of temperature regulation between 145 and 155°C.

6. Procedure

6.1 Ignite the crucible in the muffle furnace at 650± 25°C for 1 h. Place the crucible in the desiccator. Cool to room temperature and weigh to the nearest 0.1 mg.

6.2 Dry an adequate sample of activated carbon to constant weight at 150 ± 5°C (3 h is usually sufficient).

NOTE 1—Some carbons can ignite spontaneously at 150°C. In this case, moist carbon should be used with a correction for moisture (in accordance with Methods D 2867) applied in the calculations. In this case, the ashing should be started in a cold muffle furnace.

6.3 Weigh out to the nearest 0.1 mg sufficient dried activated carbon, so that the estimated amount of ash will be 0.1 g, into the ignited crucible and place the crucible in the furnace at 650 ± 25°C. Ashing will require from 3 to 16 h, depending on the size and type of activated carbon. Ashing can be considered complete when constant weight is achieved.

6.4 Place the crucible in the desiccator and allow to cool to room temperature. After the sample has cooled in the desiccator, admit air slowly to avoid loss of ash from the crucible. Weigh to the nearest 0.1 mg.

7. Calculation

7.1 Calculate the ash content as follows:

$$\text{Total ash, \%} = [(D - B)/(C - B)] \times 100 \quad (1)$$

where:

B = weight of crucible, g,
C = weight of crucible plus original sample, g, and
D = weight of crucible plus ashed sample, g.

8. Precision and Bias [3]

8.1 *Precision*:

8.1.1 *Interlaboratory Test Program*—An interlaboratory study was run in which representative samples of three types of

[1] This test method is under the jurisidiction of ASTM Committee D-28 on Activated Carbon and is the direct responsibility of Subcommittee D28.04 on Gas Phase Evaluation Tests.

Current edition approved April 1, 2004. Published June 2004. Originally approved in 1970. Last previous edition approved in 1999 as D 2866 – 94 (1999).

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting RR: D 28-1004.

TABLE 1 Precision

Material	A	B	C
Average Test Value	7.74	1.88	4.61
95 % Repeatability Limit[A] (Within Laboratory)	0.27	0.22	0.22
95 % Reproducibility Limit[A] (Between Laboratories)	0.41	0.54	0.48

[A] The terms *repeatability limit* and *reproducibility limit* are used in accordance with Practice E 177. The respective standard deviations among test results may be obtained by dividing the above limit values by 2.8.

activated carbon (coconut-shell based (*A*), coal-based (*B*), and wood-based (*C*)) were testerd for ash content by six laboratories with each laboratory making three observations of each activated carbon type over three days. Practice E 691 was followed for the design and analysis of the data.

8.1.2 *Test Result*—The precision information given in Table 1 in units of measurement (percent minus weight ash content) is for the comparison of two test results, each of which is the average of three test determinations.

APPENDIX C

Standard Test Method for Volatile Matter Content of Activated Carbon Sample

ASTM D 5832 – 98

Designation: D 5832 – 98 (Reapproved 2003)

Standard Test Method for Volatile Matter Content of Activated Carbon Samples[1]

This standard is issued under the fixed designation D 5832; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the percentage of gaseous products, exclusive of moisture vapor, present in virgin and used activated carbons which are released under specific conditions of the test.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 2652 Terminology Relating to Activated Carbon[2]

D 2867 Test Method for Moisture in Activated Carbon[2]

D 3175 Test Method for Volatile Matter in the Analysis Sample of Coal and Coke[3]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method relating to activated carbon, refer to Terminology D 2652.

4. Summary of Test Method

4.1 Volatile matter is determined by establishing the loss in mass resulting from heating an activated carbon sample under rigidly controlled conditions. The measured mass loss, corrected for moisture as determined in Test Method D 2867, establishes the volatile matter content of the activated carbon sample.

5. Significance and Use

5.1 Volatile matter, when determined as herein described, may be used as a relative measure of the extent of carbonization in an activated carbon and the extent of loading of volatile material on an activated carbon that has been used in an adsorption application.

5.2 Combined with other information, the volatile matter of an activated carbon may be useful in evaluating its performance in an adsorption application.

5.3 Other automated methods for the determination of the volatile content of solids, such as using a thermogravimetric analyzer (TGA), can be used in place of this test method with equally reliable results.

6. Apparatus

6.1 *Crucible and Cover*, high temperature porcelain, high form, 30 cc capacity.

6.2 *Oven*, forced-air circulation, capable of temperature regulation up to 250°C.

6.3 *Moisture Determination Apparatus*, as described in Test Method D 2867.

6.4 *Muffle Furnace*, gravity circulation, capable of temperature regulation at 950 ± 25°C. An electric furnace similar to the one described in Test Method D 3175 is suitable for use in this test method.

6.5 *Desiccator*, glass with indicating type desiccant.

6.6 *Balance*, analytical, capable of 0.1 mg sensitivity.

7. Hazards

7.1 The furnace used in this test method should be located in a well ventilated area to eliminate exposure to possible toxic vapors that may evolve from the carbon sample during the high temperature heating.

7.2 Exercise care when working with the high temperature furnace to eliminate the possibility of burns.

8. Procedure

8.1 Determine the moisture content of an as-received representative portion of the sample using the Xylene-Extraction Test Method described in D 2867. If the as-received sample is wet, drain it of all free liquid before the representative sample is taken.

8.2 Weigh to 0.1 mg accuracy a crucible and cover that have been ignited in a muffle furnace regulated at 950°C for 30 min and cooled in a desiccator. Record the weight.

8.3 Using a spoon or spatula, dip from the sample bottle approximately 1 g of the as-received sample and place it in the pre-dried and tared crucible. Cover it with a lid and immediately weigh it to the nearest 0.1 mg.

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[1] This test method is under the jurisdiction of ASTM Committee D28 on Activated Carbon and is the direct responsibility of Subcommittee D28.04 on Gas Phase Evaluation Tests.

Current edition approved March 10, 2003. Published July 2003. Originally approved in 1995. Last previous edition approved in 1998 as D 5832 – 98.

[2] *Annual Book of ASTM Standards*, Vol 15.01.

[3] *Annual Book of ASTM Standards*, Vol 05.05.

8.4 Place the covered crucible in the muffle furnace regulated at 950 ± 25°C for 7 min ± 10 s.

8.5 Remove the covered crucible from the muffle furnace and cool to room temperature in a desiccator.

8.6 Weigh the covered crucible to the nearest 0.1 mg. Record the weight.

9. Calculation

9.1 Calculate the weight loss percent as follows:

$$\text{Weight loss, \%} = [(C - D)/(C - B)] \times 100 \quad (1)$$

where:

B = mass of crucible and cover, g,
C = mass of crucible, cover, and sample, g, and
D = mass of crucible, cover, and de-volatilized sample, g.

9.2 Calculate the volatile matter content of the sample as follows:

$$VM, \% = E - F \quad (2)$$

where:

VM = volatile matter content of as-received sample, %,
E = weight loss, % (as defined in 9.1), and
F = moisture, % (as measured in 8.1).

10. Precision and Bias

10.1 An interlaboratory study of this test method was conducted in 1996. Each of seven laboratories tested three randomly drawn specimens from each of three different activated carbons containing volatile matter content. Carbon A was a coconut shell gas phase carbon containing gasoline vapors. Carbon B was a coal based liquid phase carbon containing organic components from gasoline. Carbon C was a coconut shell vapor phase carbon containing chlorinated organic compounds. The average volatile matter contents were 24.7 %, 9.1 % and 12.9 %, respectively. In order to determine the volatile matter content of the samples, their moisture contents were determined according to Test Method D 2867 and were found to be 3.54 %, 35.2 % and 3.87 %, respectively. Practice E 691 and E 691 computer software were used to design the study and analyze the resulting data.

10.2 *95 % Limit on Repeatability (Within Laboratory), %:*

	Activated Carbon		
	A	B	C
Voltatile Matter Content, %	1.38	0.63	0.44

10.3 *95 % Limit on Reprodicibility (Between Laboratories), %:*

	Activated Carbon		
	A	B	C
Volatile Matter Content, %	1.54	1.32	1.47

NOTE 1—The terms "limit on repeatability" and "limit on reproducibility" are used as specified in Practice E 177.

11. Keywords

11.1 activated carbon; volatile matter

APPENDIX D

Standard Test Method for Determination of Iodine Number of Activated Carbon
ASTM D 4607 – 94

Designation: D 4607 – 94 (Reapproved 1999)

Standard Test Method for Determination of Iodine Number of Activated Carbon[1]

This standard is issued under the fixed designation D 4607; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the relative activation level of unused or reactivated carbons by adsorption of iodine from aqueous solution. The amount of iodine absorbed (in milligrams) by 1 g of carbon using test conditions listed herein is called the iodine number.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*

C 819 Test Method for Specific Surface Area of Carbon or Graphite[2]

D 1193 Specification for Reagent Water[3]

D 2652 Terminology Relating to Activated Carbon[4]

D 2867 Test Method for Moisture in Activated Carbon[4]

D 3860 Practices for Determination of Adsorptive Capacity of Carbon by Isotherm Technique[4]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[5]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[5]

E 287 Specification for Laboratory Glass Graduated Burets[5]

E 288 Specification for Laboratory Glass Volumetric Flasks[5]

E 300 Practice for Sampling Industrial Chemicals[6]

2.2 *NIST Publication:*

Circular 602—Testing of Glass Volumetric Apparatus[7]

3. Summary of Test Method

3.1 This test method is based upon a three-point adsorption isotherm (see Practices D 3860). A standard iodine solution is treated with three different weights of activated carbon under specified conditions. The carbon treated solutions are filtered to separate the carbon from the treated iodine solution (filtrate). Iodine remaining in the filtrate is measured by titration. The amount of iodine removed per gram of carbon is determined for each carbon dosage and the resulting data used to plot an adsorption isotherm. The amount of iodine adsorbed (in milligrams) per gram of carbon at a residual iodine concentration of 0.02 *N* is reported as the iodine number.

3.2 Iodine concentration in the standard solution affects the capacity of an activated carbon for iodine adsorption. Therefore, the normality of the standard iodine solution must be maintained at a constant value (0.100 ± 0.001 *N*) for all iodine number measurements.

3.3 The apparatus required consists of various laboratory glassware used to prepare solutions and contact carbon with the standard iodine solution. Filtration and titration equipment are also required.

4. Significance and Use

4.1 The iodine number is a relative indicator of porosity in an activated carbon. It does not necessarily provide a measure of the carbon's ability to absorb other species. Iodine number may be used as an approximation of surface area for some types of activated carbons (see Test Method C 819). However, it must be realized that any relationship between surface area and iodine number cannot be generalized. It varies with changes in carbon raw material, processing conditions, and pore volume distribution (see Definitions D 2652).

4.2 The presence of adsorbed volatiles, sulfur; and water extractables may affect the measured iodine number of an activated carbon.

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[1] This test method is under the jurisdiction of ASTM Committee D-28 on Activated Carbon and is the direct responsibility of Subcommittee D28.02 on Liquid Phase Evaluation.

Current edition approved Nov. 15, 1994. Published January 1995. Originally published as D 4607 – 86. Last previous edition D 4607 – 86 (1990).

[2] *Discontinued*—See *1986 Annual Book of ASTM Standards*, Vol 15.01.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 15.01.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

[6] *Annual Book of ASTM Standards*, Vol 15.05.

[7] Available from National Institute of Standards and Technology, U.S. Department of Commerce, Gaithersburg, MD 20899.

5. Apparatus

NOTE 1—All volumetric measuring equipment should meet or exceed the requirements of NIST Circular 602. Volumetric glassware meeting these specifications is generally designated as "Class A". See also Specifications E 287 and E 288.

5.1 *Analytical Balance*, accuracy ±0.0001 g.

5.2 *Buret*, 10-mL capacity or 5-mL precision buret.

5.3 *Flasks*, Erlenmeyer 250-mL capacity with ground glass stoppers.

5.4 *Flask*, Erlenmeyer wide-mouthed, 250-mL capacity.

5.5 *Beakers*, assorted sizes.

5.6 *Bottles*, amber, for storage of iodine and thiosulfate solutions.

5.7 *Funnels*, 100-mm top inside diameter.

5.8 *Filter Paper*, 18.5-cm prefolded paper, Whatman No. 2V or equivalent.

5.9 *Pipets*, volumetric type, 5.0, 10.0, 25.0, 50.0, and 100.0-mL capacity.

5.10 *Volumetric Flasks*, 1 L.

5.11 *Graduated Cylinders*, 100 mL and 500 mL.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[8] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—References to water shall be understood to mean reagent water conforming to Specification D 1193 for Type II reagent water.

6.3 *Hydrochloric Acid*, concentrated.

6.4 *Sodium Thiosulfate*, ($Na_2S_2O_3 \cdot 5H_2O$).

6.5 *Iodine*, United States Pharmacopeia, resublimed crystals.

6.6 *Potassium Iodide*.

6.7 *Potassium Iodate*, primary standard.

6.8 *Starch*, soluble potato or arrowroot.

6.9 *Sodium Carbonate*.

7. Hazards

7.1 Several potential hazards are associated with conducting this test procedure. It is not the purpose of this standard to address all potential health and safety hazards encountered with its use. The user is responsible for establishing appropriate health and safety practices before use of this test procedure. Determine the applicability of federal and state regulations before attempting to use this test method.

7.2 Personnel conducting the iodine number procedure should be aware of potential safety and health hazards associated with the chemicals used in this procedure. The "Material Safety Data Sheet" (MSDS) for each reagent listed in Section 6 should be read and understood. Special precautions to be taken during use of each reagent are included on the "Material Safety Data Sheet" (MSDS). First aid procedures for contact with a chemical are also listed on its "MSDS." A "Material Safety Data Sheet" for each reagent may be obtained from the manufacturer. Other safety and health hazard information on reagents used in this procedure is available.[9,10,11]

7.3 Careful handling and good laboratory technique should always be used when working with chemicals. Avoid contact with hydrochloric acid or acid vapor. Care should also be taken to prevent burns during heating of various solutions during this test procedure.

7.4 The user of this test method should comply with federal, state, and local regulations for safe disposal of all samples and reagents used.

8. Preparation of Solutions

8.1 *Hydrochloric Acid Solution* (5 % by weight)—Add 70 mL of concentrated hydrochloric acid to 550 mL of distilled water and mix well. A graduated cylinder may be used for measurement of volume.

8.2 *Sodium Thiosulfate* (0.100 *N*)—Dissolve 24.820 g of sodium thiosulfate in approximately 75 ± 25 mL of freshly boiled distilled water. Add 0.10 ± 0.01 g of sodium carbonate to minimize bacterial decomposition of the thiosulfate solution. Quantitatively transfer the mixture to a 1-L volumetric flask and dilute to the mark. Allow the solution to stand at least 4 days before standardizing. The solution should be stored in an amber bottle.

8.3 *Standard Iodine Solution* (0.100 ± 0.001 *N*)—Weigh 12.700 g of iodine and 19.100 g of potassium iodide (KI) into a beaker. Mix the dry iodine and potassium iodide. Add 2 to 5 mL of water to the beaker and stir well. Continue adding small increments of water (approximately 5 mL each) while stirring until the total volume is 50 to 60 mL. Allow the solution to stand a minimum of 4 h to ensure that all crystals are thoroughly dissolved. Occasional stirring during this 4-h period will aid in the dissolution. Quantitatively transfer to a 1-L volumetric flask and fill to the mark with distilled water. It is important that the standard iodine solution has an iodide-to-iodine weight ratio of 1.5 to 1. Store the solution in an amber bottle.

8.4 *Potassium Iodate Solution* (0.1000 *N*)—Dry 4 or more grams of primary standard grade potassium iodate (KIO_3) at 110 ± 5°C for 2 h and cool to room temperature in a desiccator. Dissolve 3.5667 ± 0.1 mg of the dry potassium iodate in about 100 mL of distilled water. Quantitatively transfer to a 1-L volumetric flask and fill to the mark with distilled water. Mix thoroughly and store in a glass-stoppered bottle.

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[8] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

[9] The "Chemical Safety Data Sheet" for the subject chemical is available from the Manufacturing Chemists Association, Washington, DC.

[10] Sax, N. I., *Dangerous Properties of Industrial Materials*, 4th edition, 1975, Van Nostrand Reinhold Company, New York, NY.

[11] *NIOSH/OSHA Pocket Guide to Chemical Hazards*, 1978, U.S. Department of Labor, Occupational Safety and Health Administration, Washington, DC. Available from U.S. Government Printing Office, Washington, DC.

8.5 *Starch Solution*—Mix 1.0 ± 0.5 g of starch with 5 to 10 mL of cold water to make a paste. Add an additional 25 ± 5 mL of water while stirring to the starch paste. Pour the mixture, while stirring, into 1 L of boiling water and boil for 4 to 5 min. This solution should be made fresh daily.

9. Standardization of Solutions

9.1 *Standardization of 0.100 N Sodium Thiosulfate*—Pipet 25.0 mL of potassium iodate (KIO_3) solution from 8.4 into a 250-mL titration (or wide-mouthed Erlenmeyer) flask. Add 2.00 ± 0.01 g of potassium iodide (KI) to the flask and shake the flask to dissolve the potassium iodide crystals. Pipet 5.0 mL of concentrated hydrochloric acid into the flask. Titrate the free iodine with sodium thiosulfate solution until a light yellow color is observed in the flask. Add a few drops of starch indicator (8.5) and continue the titration dropwise until one drop produces a colorless solution. Determine sodium thiosulfate normality as follows:

$$N_1 = (P \cdot R)/S \quad (1)$$

where:
N_1 = sodium thiosulfate, *N*,
P = potassium iodate, mL,
R = potassium iodate, *N*, and
S = sodium thiosulfate, mL.

The titration step should be done in triplicate and the normality results averaged. Additional replications should be done if the range of values exceeds 0.003 *N*.

9.2 *Standardization of 0.100 ± 0.001 N Iodine Solution*—Pipet 25.0 mL of iodine solution (8.3) into a 250-mL widemouthed Erlenmeyer flask. Titrate with standardized sodium thiosulfate (9.1) until the iodine solution is a light yellow color. Add a few drops of starch indicator and continue titration dropwise until one drop produces a colorless solution. Determine the iodine solution normality as follows:

$$N_2 = (S \cdot N_1)/I \quad (2)$$

where:
N_2 = iodine, *N*,
S = sodium thiosulfate, mL,
N_1 = sodium thiosulfate, *N*, and
I = iodine, mL.

The titration step should be done in triplicate and the normality results averaged. Additional replications should be done if the range of values exceeds 0.003 *N*. The iodine solution concentration must be 0.100 ± 0.001 *N*. If this requirement is not met, repeat 8.3 and 9.2.

10. Procedure

10.1 The procedure applies to either powdered or granular activated carbon. When granular carbon is to be tested, grind a representative sample (see Practice E 300) of carbon until 60 wt % (or more will pass through a 325-mesh screen) and 95 wt % or more will pass through a 100-mesh screen (U.S. sieve series, see Specification E 11). Carbon received in the powdered form may need additional grinding to meet the particle size requirement given above.

10.2 Dry the ground carbon from 10.1 in accordance with Test Method D 2867. Cool the dry carbon to room temperature in a desiccator.

10.3 Determination of iodine number requires an estimation of three carbon dosages. Section 11.4 describes how to estimate the carbon dosages to be used. After estimating carbon dosages, weigh three appropriate amounts of dry carbon to the nearest milligram. Transfer each weighed sample of carbon to a clean, dry 250-mL Erlenmeyer flask equipped with a ground glass stopper.

10.4 Pipet 10.0 mL of 5 wt % hydrochloric acid solution into each flask containing carbon. Stopper each flask and swirl gently until the carbon is completely wetted. Loosen the stoppers to vent the flasks, place on a hot plate in a fume hood, and bring the contents to a boil. Allow to boil gently for 30 ± 2 s to remove any sulfur which may interfere with the test results. Remove the flasks from the hot plate and cool to room temperature.

10.5 Pipet 100.0 mL of 0.100 *N* iodine solution into each flask. Standardize the iodine solution just prior to use. Stagger the addition of iodine to the three flasks so that no delays are encountered in handling. Immediately stopper the flasks, and shake the contents vigorously for 30 ± 1 s. Quickly filter each mixture by gravity through one sheet of folded filter paper (Whatman No. 2V or equivalent) into a beaker. Filtration equipment must be prepared in advance so no delay is encountered in filtering the samples.

10.6 For each filtrate, use the first 20 to 30 mL to rinse a pipet. Discard the rinse portions. Use clean beakers to collect the remaining filtrates. Mix each filtrate by swirling the beaker and pipet 50.0 mL of each filtrate into a clean 250-mL Erlenmeyer flask. Titrate each filtrate with standardized 0.100 *N* sodium thiosulfate solution until the solution is a pale yellow. Add 2 mL of the starch indicator solution and continue the titration with sodium thiosulfate until one drop produces a colorless solution. Record the volume of sodium thiosulfate used.

11. Calculation

11.1 The capacity of a carbon for any adsorbate is dependent upon the concentration of the adsorbate in solution. The concentrations of the standard iodine solution and filtrates must be specified or known. This is necessary to determine an appropriate carbon weight to produce final concentrations agreeing with the definition of iodine number. The amount of carbon sample to be used in the determination is governed by the activity of the carbon. If filtrate normalities (*C*) are not within the range of 0.008 *N* to 0.040 *N*, repeat the procedure using different carbon weights.

11.2 Two calculations are required for each carbon dosage, as *X/M* and *C*.

11.2.1 To calculate the value of *X/M*, first derive the following values:

$$A = (N_2)\,(12693.0) \quad (3)$$

where:
N_2 = iodine, *N* (from 9.2).

$$B = (N_1)\,(126.93) \quad (4)$$

where:
N_1 = sodium thiosulfate, *N* (from 9.1).

$$DF = (I + H)/F \quad (5)$$

where:
DF = dilution factor,
I = iodine, mL (from 9.2),
H = 5 % hydrochloric acid used, mL, and
F = filtrate, mL.

For example, if 10 mL of HCl and 50 mL of filtrate are used: $DF = (100 + 10)/50 = 2.2$.

11.2.1.1 Calculate the value of X/M as follows:

$$X/M = [A - (DF)(B)(S)]/M \quad (6)$$

where:
X/M = iodine absorbed per gram of carbon, mg/g,
S = sodium thiosulfate, mL, and
M = carbon used, g.

11.2.2 Calculate the value of C as follows:

$$C = (N_1 \cdot S)/F \quad (7)$$

where:
C = residual filtrate, N,
N_1 = sodium thiosulfate, N, and
F = filtrate, mL.

11.3 Using logarithmic paper, plot X/M (as the ordinate) versus C (as the abscissa) for each of the three carbon dosages (see Fig. 1). Calculate the least squares fit for the three points and plot. The iodine number is the X/M value at a residual iodine concentration (C) of 0.02 N. The regression coefficient for the least squares fit should be greater than 0.995.

11.4 Carbon dosage may be estimated as follows:

$$M = [A - (DF)(C)(126.93)(50)]/E \quad (8)$$

where:
M = carbon, g,
A = (N_2) (12693.0),
DF = dilution factor (see 11.2.1),
C = residual iodine, and
E = estimated iodine number of the carbon.

Three carbon dosages are calculated using three values of C (usually 0.01, 0.02, and 0.03).

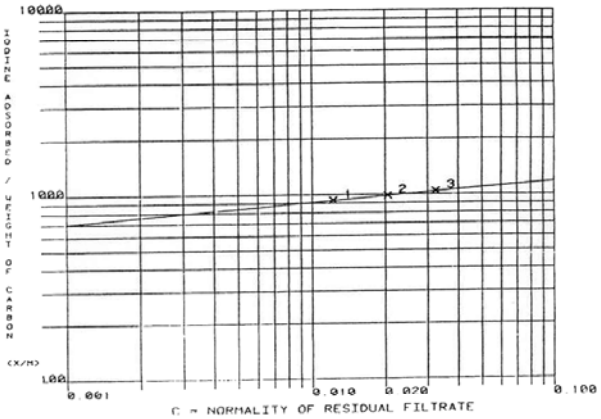
12. Report

12.1 The reports should include the following:

12.1.1 Complete identification of the sample, including source, manufacturer's lot number, and carbon type.

12.1.2 The iodine number adsorption isotherm, the iodine value with a 95 % confidence limit for the determination.

13. Precision and Bias [12]



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TECH. PRP

POINT 1		
X/M	–	932
C	–	0.012
POINT 2		
X/M	–	964
C	–	0.020
POINT 3		
X/M	–	1040
C	–	0.032
IODINE NO.	–	964
SLOPE	–	0.111
CORR. COEF.	–	0.999

FIG. 1 Activated Carbon Iodine Adsorption Isotherm

13.1 Interlaboratory precision for this test method was determined by a round robin in which six laboratories participated. Activated carbon made from five different raw materials was used in the round robin.

13.2 The following criteria should be used in determining the acceptability of the results:

13.2.1 *Repeatability (Within Laboratories)*—Precision of this test method in the determination of iodine number of activated carbons ranging from 600 to 1450 iodine number is ±5.6 % of the average value measured in milligrams iodine absorbed per gram of carbon. This range corresponds to 2*S*, or the 95 % confidence limits, as defined in Practice E 177. If two results determined in the same laboratory differ by more than 5.6 %, they should be considered suspect.

13.2.2 *Reproducibility (Between Laboratories)*—The between laboratory precision of this test method in the determination of iodine number of activated carbons ranging from 600 to 1450 iodine number, is ±10.2 % of the average value, as measured in milligrams of iodine absorbed per gram of carbon. This range corresponds to 2 *S* or the 95 % confidence limits, as defined in Practice E 177. Results obtained by two different laboratories which differ by more than 10.2 % should be considered suspect.

APPENDIX E

Standard Test Method for Determination of Phenol Value of Activated Carbon

AWWA B600

APPENDIX B
Phenol and Tannin Values

This appendix is for information only and is not a part of AWWA B600.

Section B.1 Phenol Value

Sec. B.1.1 Phenol Value Test

The phenol value test procedure from ANSI/AWWA B600-78 is reprinted below for the information of ANSI/AWWA B600-96 users. ANSI/AWWA B600-78 stated, "Unless otherwise specified by the purchaser, the phenol value of the activated carbon shall not be greater than 3.5." The modified phenol value used in this standard is defined as the concentration of activated carbon in grams per litre required to reduce the standard phenol concentration from 200 mg/L to 20 mg/L.

Sec. B.1.2 Reagents and Equipment

a. Stock phenol solution. Dissolve 1 g of reagent-grade phenol in distilled water and dilute to 1 L. Phenol should be weighed on a glass weighing dish. Rinse the weighing dish with distilled water several times to ensure the removal of all phenol from the weighing dish into the solution. The solution need not be buffered. Standardize the solution. After two weeks, the solution should be discarded and a new solution prepared. (Reagent-grade phenol must be stored in a refrigerator.)

b. Sodium thiosulfate, 0.1*N*. Dissolve 25 g of reagent-grade sodium thiosulfate and 1 g of reagent-grade sodium carbonate, as a preservative, in boiled distilled water, and make up to 1 L. Store in a brown bottle. Standardize the solution.

c. Sodium thiosulfate, 0.01*N*. Dilute 100 mL of the standardized 0.1*N* sodium thiosulfate solution to 1 L with boiled distilled water. Store in a brown bottle. Standardize the solution.

d. Potassium bromate–bromide, 0.1*N*. Dissolve 2.784 g of reagent-grade potassium bromate and 10 g of reagent-grade potassium bromide (bromate-free) in distilled water, and dilute to 1 L. Store in a brown bottle.

e. Potassium biniodate, 0.1*N*. Dissolve 3.250 g of potassium biniodate, primary standard, in distilled water, and make up to 1 L.

f. Potassium biniodate, 0.025*N*. Dissolve 0.8125 g of potassium biniodate, primary standard, in distilled water, and make up to 1 L.

g. Potassium iodide, 12.5 percent. Dissolve 25 g of reagent-grade potassium iodide in 175 mL of distilled water and dilute to 200 mL. Store solution in a brown bottle in a dark place.

h. Starch solution. Dissolve 2.5 g of soluble potato-powder starch and 1.25 g of reagent-grade salicylic acid in 50 mL of distilled water. Add the dissolved starch and salicylic acid slowly, while stirring, to 950 mL of boiling distilled water. Rinse the 150-mL beaker with some of the hot starch solution to ensure removal of all of the starch.

i. Buffer solution, 8 strength. Dissolve 728 g of anhydrous disodium phosphate (Na_2HPO_4), or equivalent weight of crystalline phosphate, in 2 L of hot distilled water. When the phosphate is completely dissolved, acidify with about 100 mL of

concentrated reagent-grade phosphoric acid and make up to 7 L. Make final adjustment to pH 6.5 ± 0.1.

Sec. B.1.3 Standardization of Reagents

The standardization of reagents follows:

a. Sodium thiosulfate. Add 100 mL of distilled water, 4 mL of concentrated reagent-grade hydrochloric acid, and 8 mL of 12.5 percent potassium iodide solution to a 500-mL iodine flask and mix. Rinse the sides of the flask with distilled water.

When standardizing 0.1*N* sodium thiosulfate solution, add 25 mL of 0.1*N* potassium biniodate solution.

When standardizing 0.01*N* sodium thiosulfate solution, add 10 mL of 0.025*N* potassium biniodate solution.

Using a transfer pipette, add the potassium biniodate solution to the flask. Mix and allow to stand for 3 min. Titrate with the sodium thiosulfate solution, using starch as the indicator.

$$\text{normality sodium thiosulfate solution} = \frac{\text{millilitre potassium biniodate solution} \times \text{normality factor}}{\text{millilitre sodium thiosulfate solution used}}$$

b. Stock phenol solution. Pipette 25 mL of stock phenol solution into a 500-mL iodine flask. Using a pipette or a burette, add 25 mL of 0.1*N* potassium bromate–bromide solution. Shake flask. Add 5 mL of concentrated hydrochloric acid. After 3 min, add 8 mL of 12.5 percent potassium iodide solution. Allow to stand for 3 min and titrate the liberated iodine with 0.1*N* sodium thiosulfate solution, using starch solution as the indicator.

$$\text{concentration of phenol (grams per litre)} = \frac{\left[\begin{array}{l}(\text{millilitre bromate–bromide} \times \text{normality factor}) - \\ (\text{millilitre sodium thiosulfate} \times \text{normality factor})\end{array}\right]}{\text{millilitre stock phenol solution}} \times 15.685$$

Sec. B.1.4 Phenol Test Procedure

The phenol test procedure follows:

a. Prepare a test phenol solution with a concentration of 200 mg/L of phenol by diluting one volume of stock phenol solution (1,000 mg/L) with four volumes of single-strength buffer solution. Single-strength buffer solution is prepared by diluting one volume of 8-strength buffer solution with seven volumes of distilled water. For example, to prepare 1 L of test phenol solution use 200 mL of stock phenol solution and 800 mL of single-strength buffer solution (100 mL of 8-strength buffer solution plus 700 mL of distilled water.) If the stock phenol solution is not of the exact concentration (1 g/L), adjust the volume used so that the test solution is maintained at 200 mg/L of phenol.

When preparing test phenol solution, prepare enough to conduct all of the sample for the day. A four-point isotherm for a single activated carbon requires 800 mL of test phenol solution (200 mL for each activated carbon dosage). It is advisable to make up some solution in excess of the amount required to allow for spillage, and so forth.

b. Use the activated carbon as received, and determine the moisture content according to Sec. 5.2.3 of ANSI/AWWA B600.

Use four dosages for each activated carbon. At least one dosage should adsorb 90 percent or more of the phenol in the test phenol solution. Activated carbon weights of 0.4 g, 0.5 g, 0.6 g, and 0.7 g are usually satisfactory. If these weights do not give the desired range of phenol adsorption, higher or lower weights may be used. Each activated carbon dosage is weighed into an Erlenmeyer flask.

c. Using pipette, add 200 mL of test phenol solution (200 mg/L of phenol) to each Erlenmeyer flask. Add only about 100 mL of solution initially to thoroughly wet the activated carbon, and rinse the sides of the flask with the remaining portion. Close the flasks with rubber caps and place them in an Eberback Water Bath Shaker (or equivalent) at ambient temperature, without water in the bath, and shake for 30 min. Samples are to be filtered by gravity using Whatman No. 2V, 24-cm fluted paper (or equivalent) on a 5-in. (127-mm) funnel. Allow the samples to filter completely.

Sec. B.1.5 Residual Phenol Determination

Determine the residual phenol concentration for each activated carbon treated filtrate by either the titration method or the spectrophotometric method according to the following procedures:

a. Titration method. Transfer a 150-mL portion of each filtrate, measured with a graduated cylinder, to a 500-mL iodine flask. Add 10 mL of concentrated, reagent-grade hydrochloric acid to each flask.

Add from a burette 0.1*N* potassium bromate–bromide solution until a light yellow color develops, indicating that an excess has been added. Replace the stopper immediately, shake the flask vigorously for a few seconds, and then place the flask in cold water. The potassium bromate–bromide solution should be added as quickly as possible so that the liberated bromine does not escape. Record the potassium bromate–bromide titration.

Place the flasks in a sink with a stoppered drain or in a cold-water bath, with cold tap water running into it to cool the flasks. Keep the water as cold as possible, taking care not to fill the sink, causing the flasks to upset. After the flasks are cooled, add 12.5 percent potassium iodide solution to the flange top of the flask. Completely fill the top. Loosen the stopper to allow the potassium iodide solution to be drawn into the flask by the vacuum. Immediately drop the stopper back into place. (The flask-cooling period must be adequate to develop the required vacuum. If multiple activated carbons are tested, add potassium bromate–bromide solution to at least eight of the samples, and then add the potassium iodide to the first four samples, while allowing the last four samples to cool.) After the potassium iodide solution has been drawn into the flask, rinse the top of the flask (using a wash bottle) with distilled water. Loosen the stopper and allow the water to run into the flask, rinsing all the potassium iodide into the flask. Remove the flasks from the cold water. Titrate the liberated iodine with 0.01*N* sodium thiosulfate solution, using starch as the indicator. Record the sodium thiosulfate titration.

$$\text{residual phenol (milligram per litre)} = \left[\begin{array}{l}(\text{millilitre bromate–bromide} \times \text{normality factor}) - \\ (\text{millilitre sodium thiosulfate} \times \text{normality factor})\end{array}\right] \times 104.6$$

b. Spectrophotometric method. Prepare 1,500 mL of buffer solution mixture by adding one volume of 8-strength buffer solution to nine volumes of distilled

water. The buffer solution mixture is used as the reference solution in the spectrophotometer and as the diluent in preparing the standard phenol reference curve.

Prepare standards containing 20 mg/L, 40 mg/L, 60 mg/L, 80 mg/L, 100 mg/L, and 120 mg/L phenol. To make these standards, measure from a burette 4 mL, 8 mL, 12 mL, 16 mL, 20 mL, and 24 mL of stock phenol solution (1,000 mg/L), respectively, into 200-mL volumetric flasks. Dilute each to exactly 200 mL with the buffer solution mixture and mix thoroughly.

The optical density of each filtrate and each dilution of the standard phenol reference curve is read on the spectrophotometer at a wavelength of 270 μm using silica cells with a 10-mm light path. Buffer solution mixture is used as the reference to balance the spectrophotometer.

Plot the data for the phenol reference curve, milligram per litre phenol versus optical density, on arithmetic paper.

Determine the residual phenol concentration milligram per litre for each activated carbon treated filtrate from the phenol reference curve.

NOTES: The following notes list some aids for more accurate results when using a spectrophotometer:

1. Let the instrument warm up sufficiently before balancing it.
2. Be sure that no foreign particles or air bubbles are present in the cell.
3. Dry the cells thoroughly and polish with lens tissue.
4. Always use the same matched cell for the reference cell.
5. Rinse the cells thoroughly with distilled water.
6. Avoid making fingerprints on the cell face while placing the cell in the instrument.

Sec. B.1.6 Calculations

a. Determine the percentage of residual filtrate normality (remaining phenol) (see B600-78, Table 1, page 8) in each activated carbon treated filtrate:

$$\%\ \text{of residual filtrate normality} = \frac{\text{milligram per litre residual phenol} \times 100}{200\ (\text{milligram per litre phenol in test solution})}$$

b. Determine percentage of X (adsorbed phenol):

$$\%\ \text{of}\ X = 100 - \%\ \text{of residual filtrate normality}$$

c. Activated carbon dosages, per 200 mL of phenol solution, are multiplied by 5 to obtain activated carbon dosage, M, as grams per litre.

d. Calculate the percentage of X/M value for each activated carbon dosage.

e. Plot isotherm: percentage of residual filtrate normality on abscissa and percentage of X/M on ordinate on 2 × 2 cycle logarithmic paper and draw best straight line through the points.

f. Determine the percentage of X/M at 10 percent of residual filtrate normality.

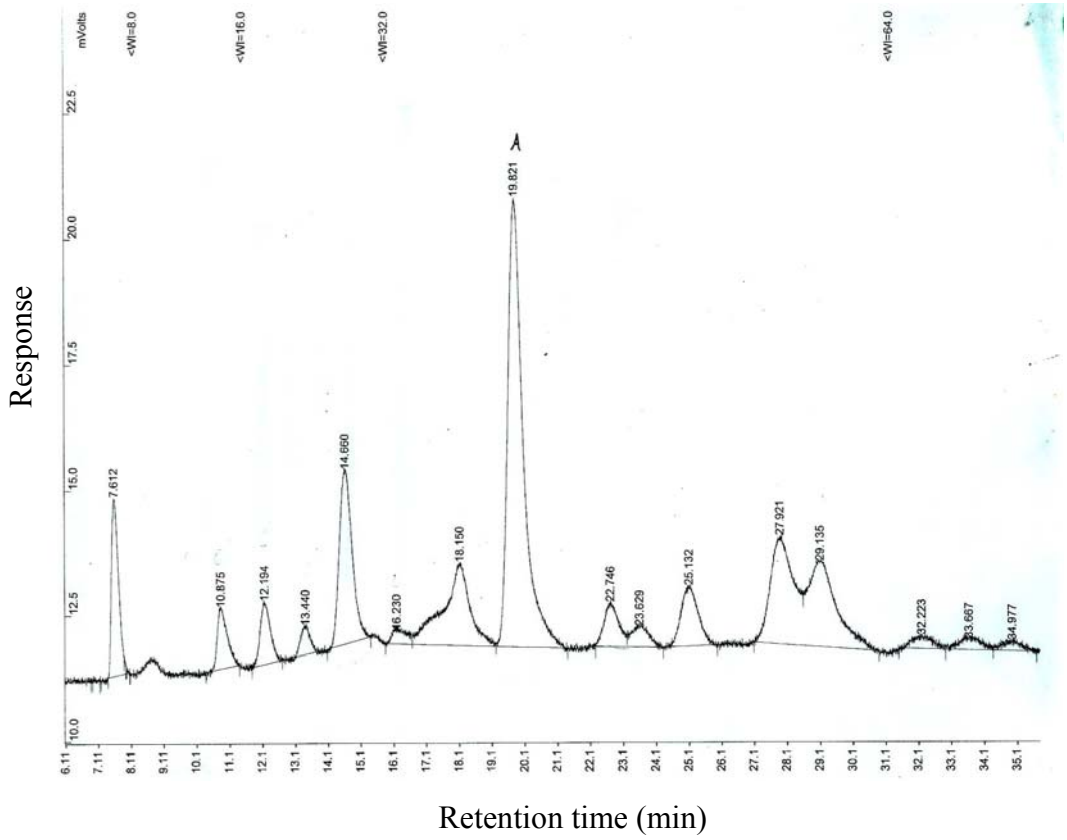
g. Phenol value in grams per litre =

$$\frac{90}{\%\ \text{of}\ X/M\ \text{in grams per litre at 10\% of residual filtrate normality}} \times \frac{100\% - \%\ \text{of moisture}}{100\%}$$

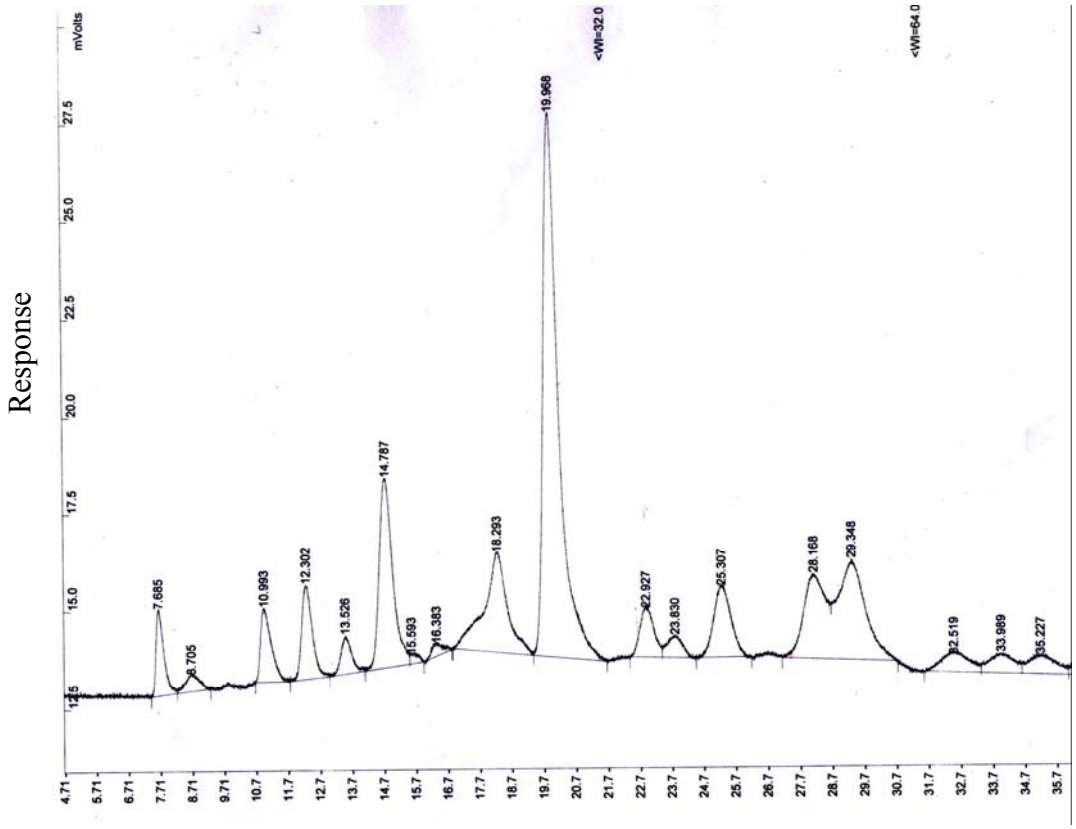
As shown, this formula is on a dry activated carbon basis.

APPENDIX F

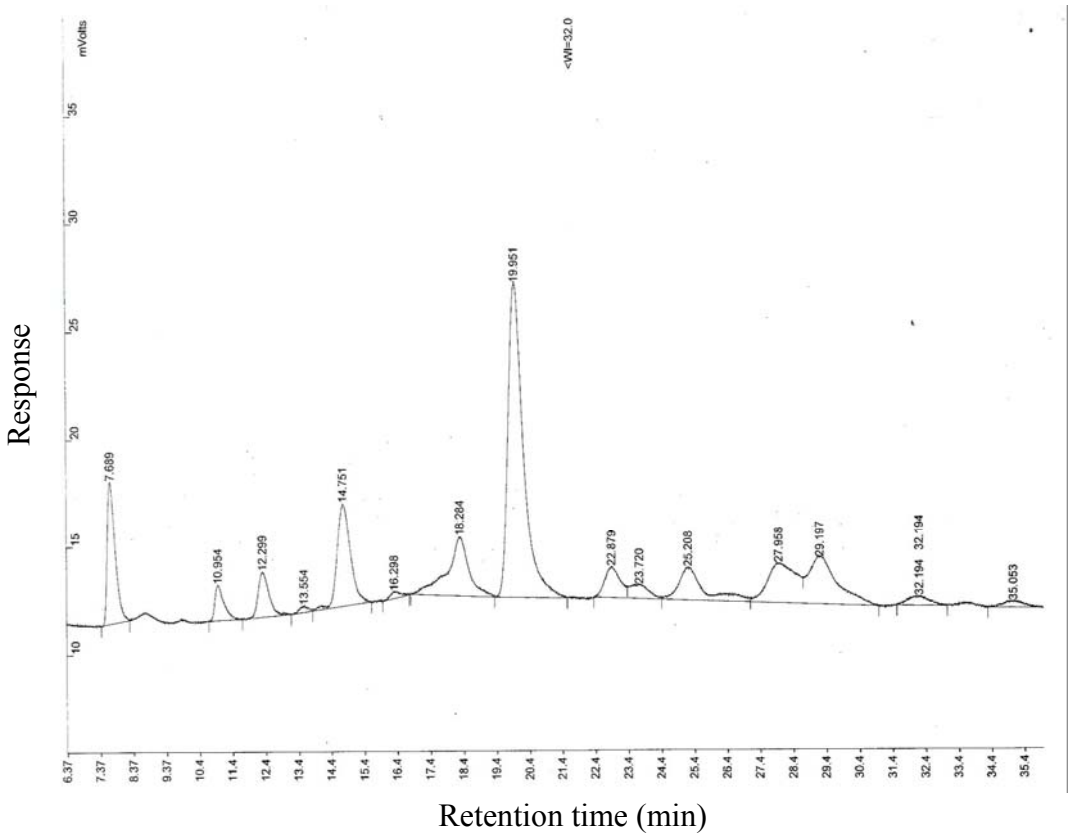
The HPLC chromatogram of wood vinegar prepared from bamboos



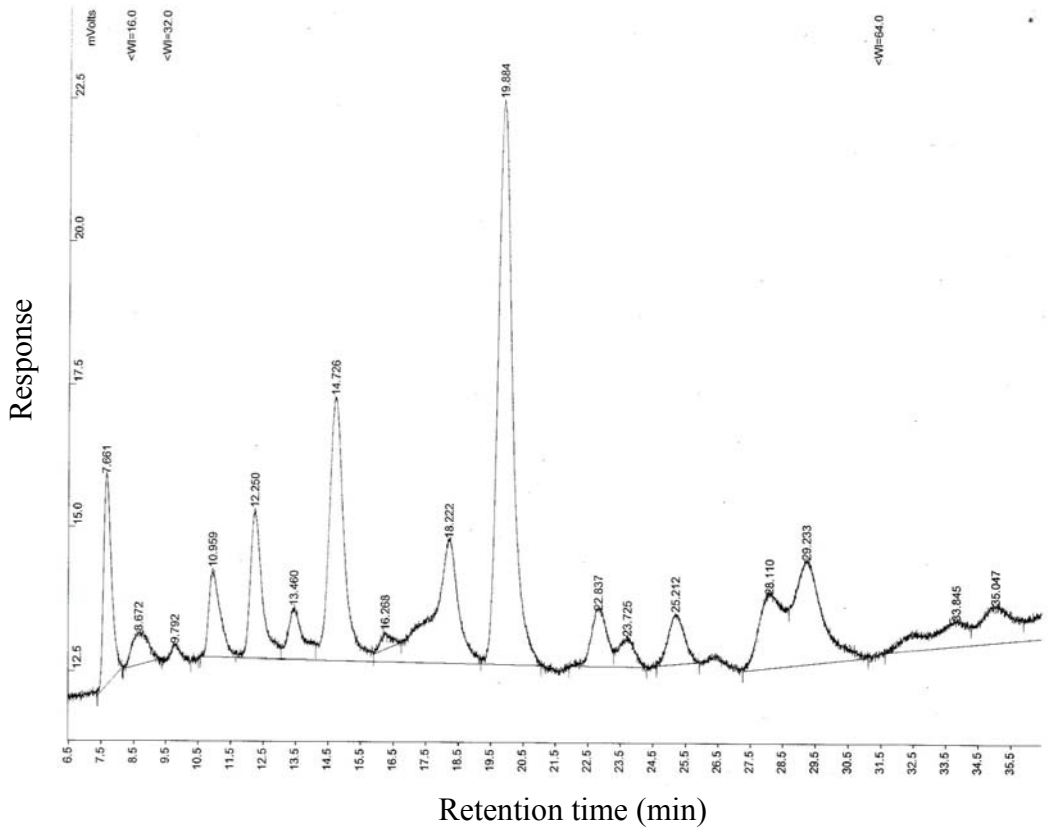
Appendix Figure F1 The HPLC chromatogram of wood vinegar prepared from one-year BAW.



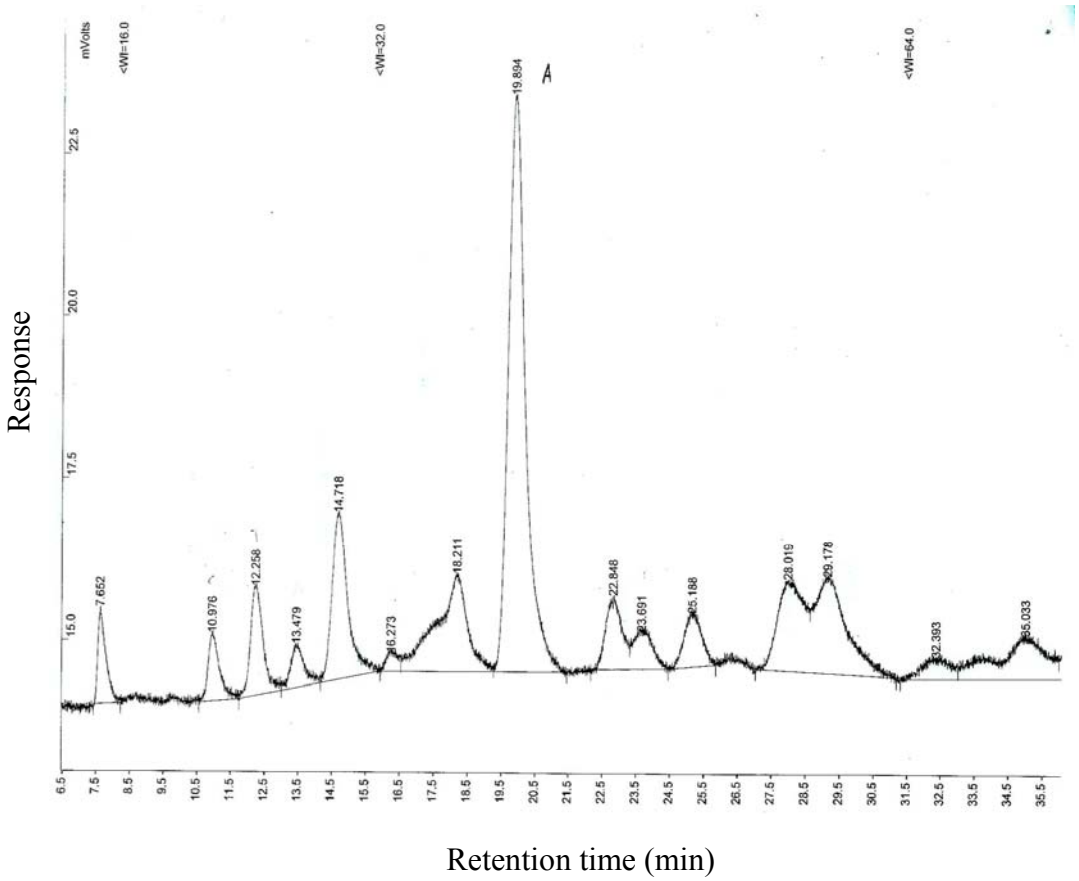
Appendix Figure F2 The HPLC chromatogram of wood vinegar prepared from two-year BAW.



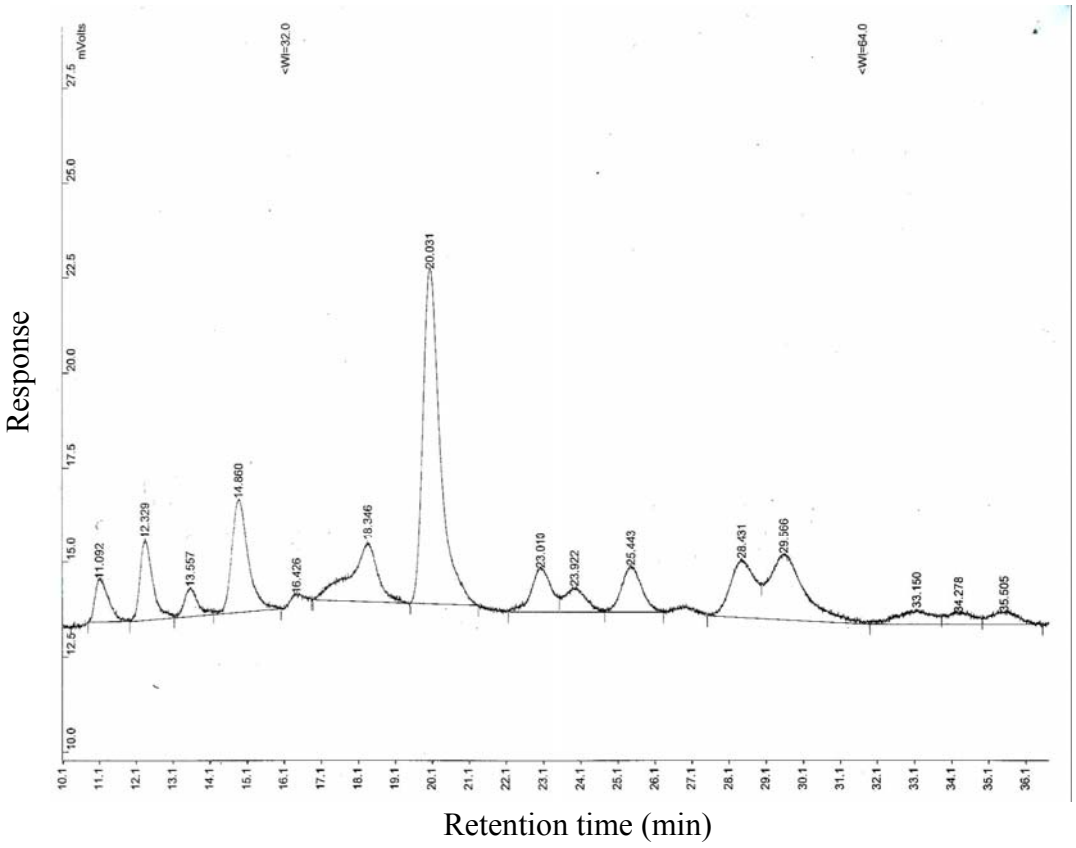
Appendix Figure F3 The HPLC chromatogram of wood vinegar prepared from three-year BAW.



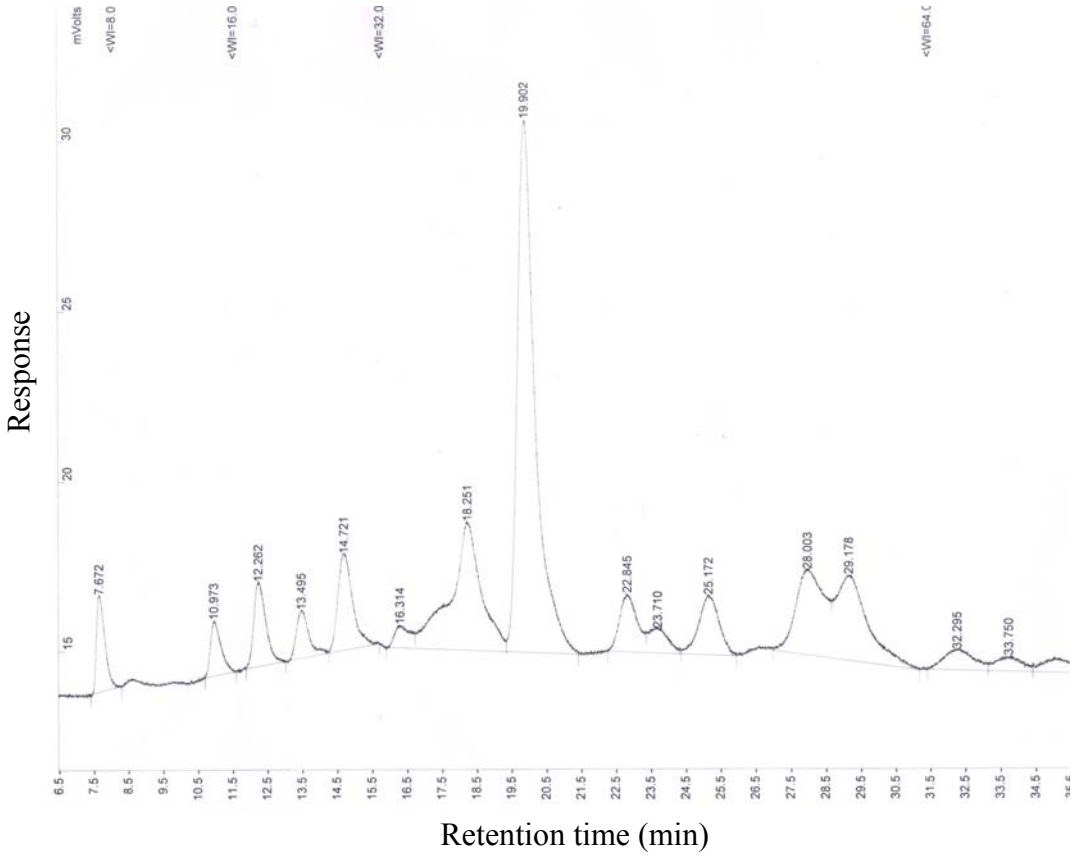
Appendix Figure F4 The HPLC chromatogram of wood vinegar prepared from one-year GO.



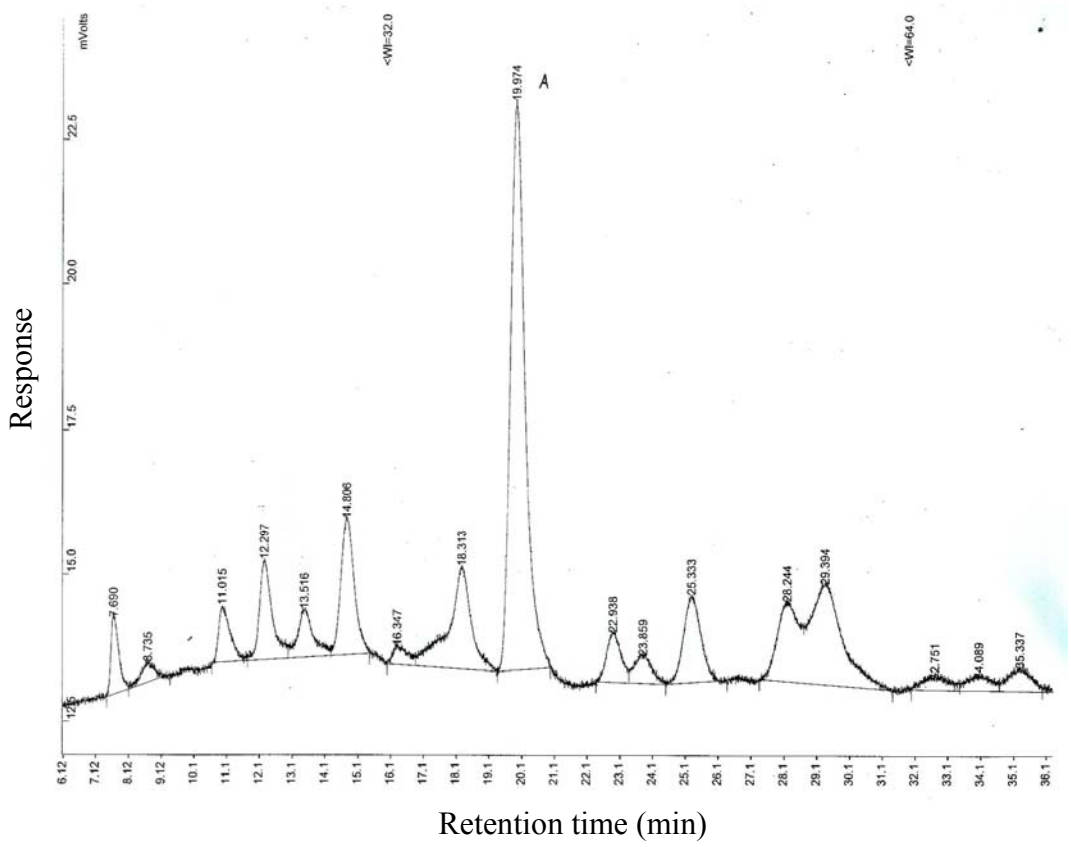
Appendix Figure F5 The HPLC chromatogram of wood vinegar prepared from two-year GO.



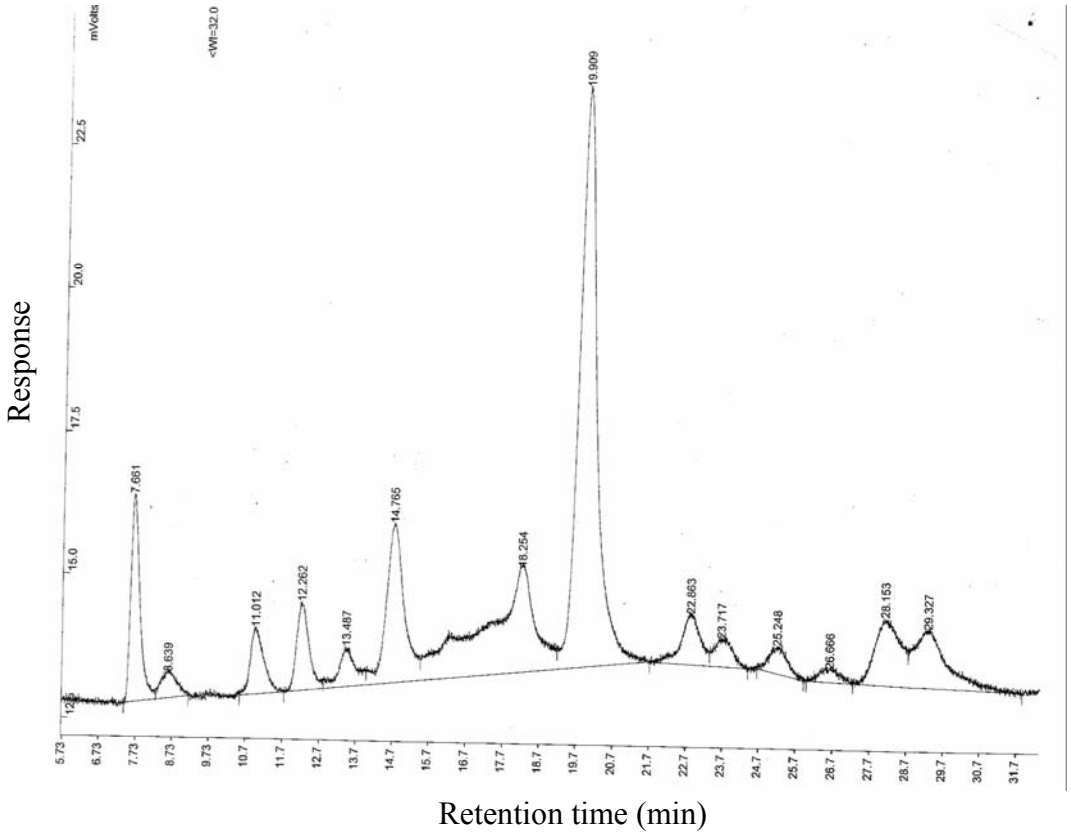
Appendix Figure F6 The HPLC chromatogram of wood vinegar prepared from three-year GO.



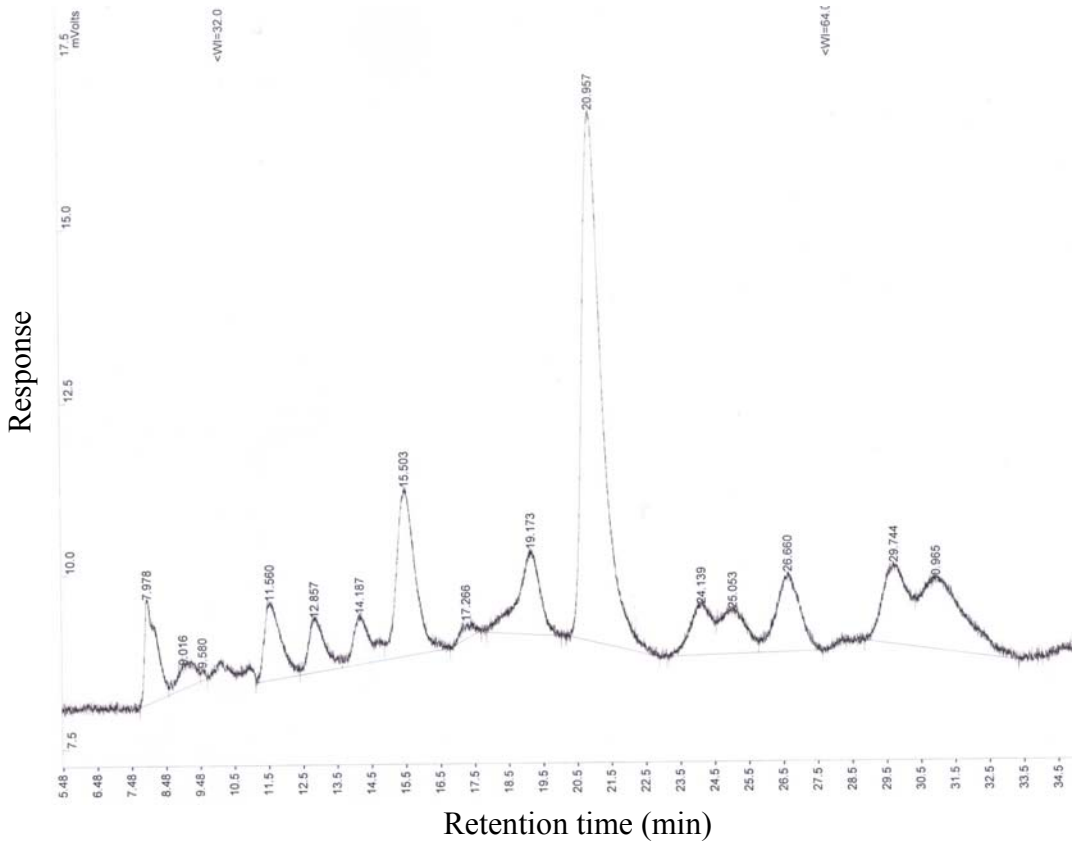
Appendix Figure F7 The HPLC chromatogram of wood vinegar prepared from one-year GV.



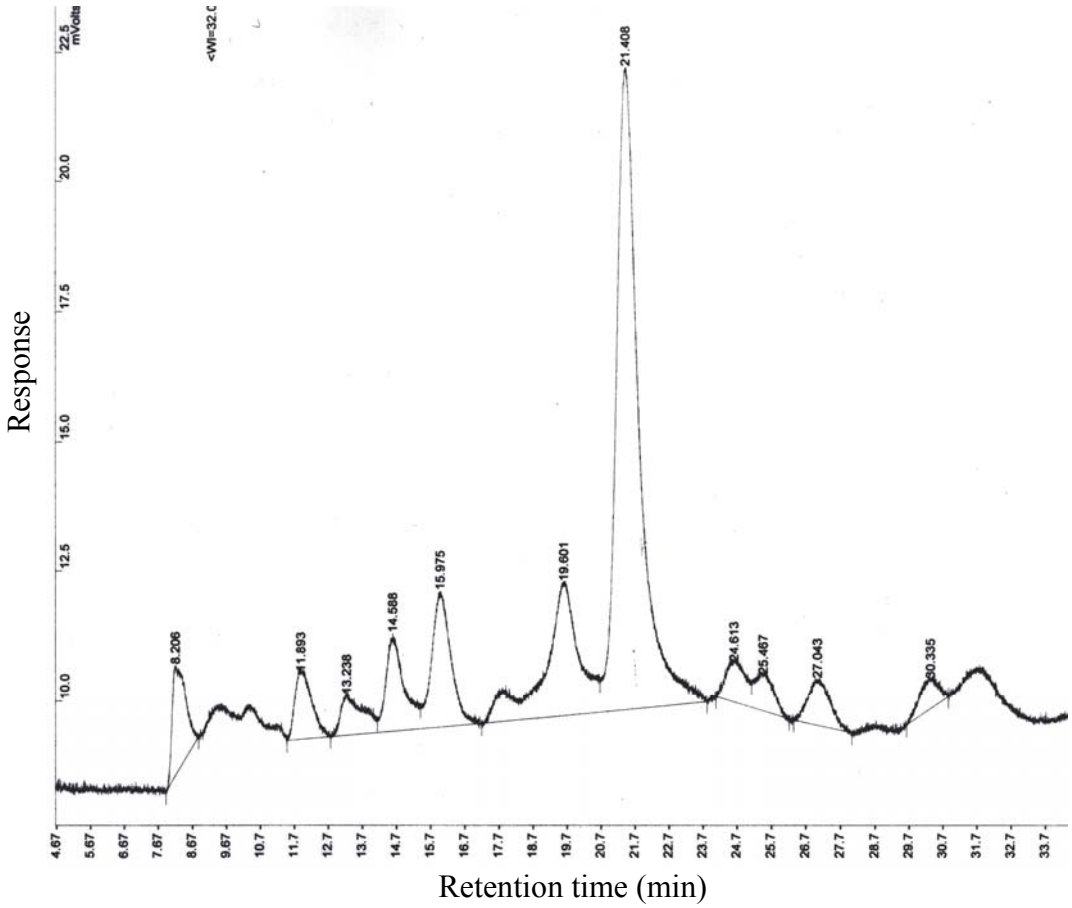
Appendix Figure F8 The HPLC chromatogram of wood vinegar prepared from two-year GV.



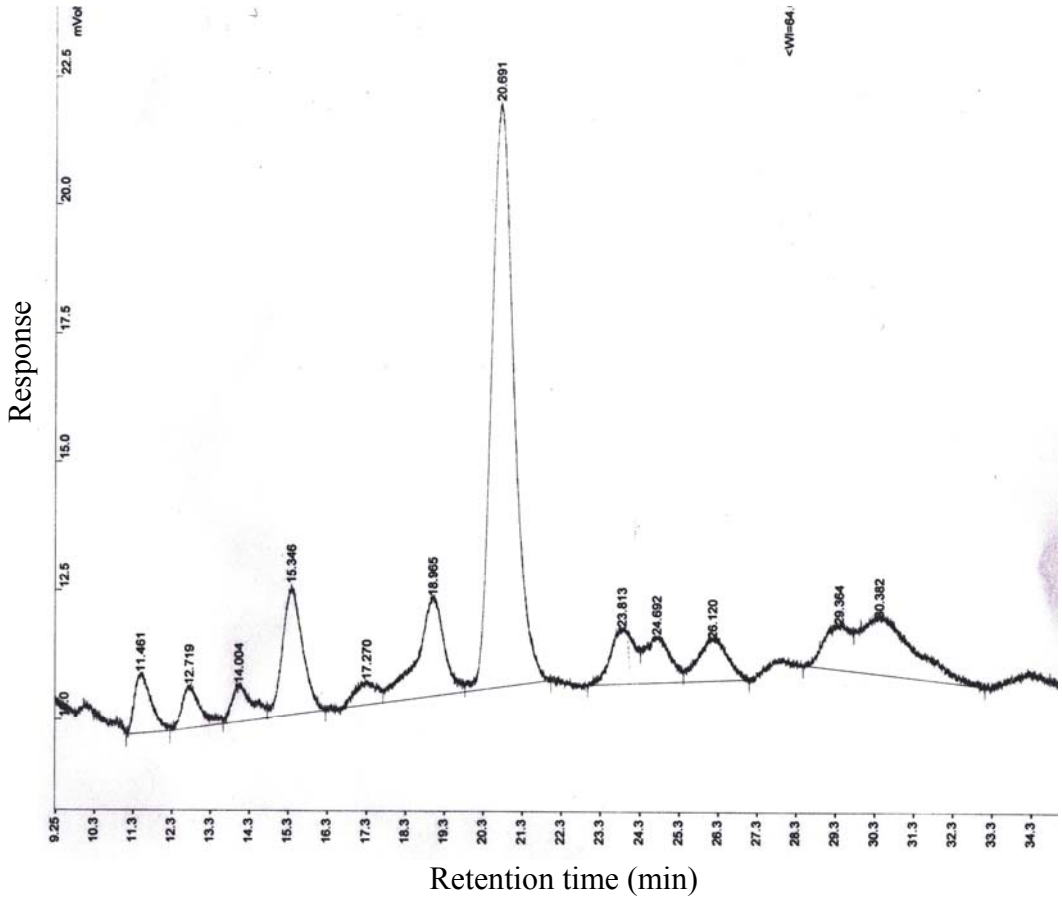
Appendix Figure F9 The HPLC chromatogram of wood vinegar prepared from three-year GV.



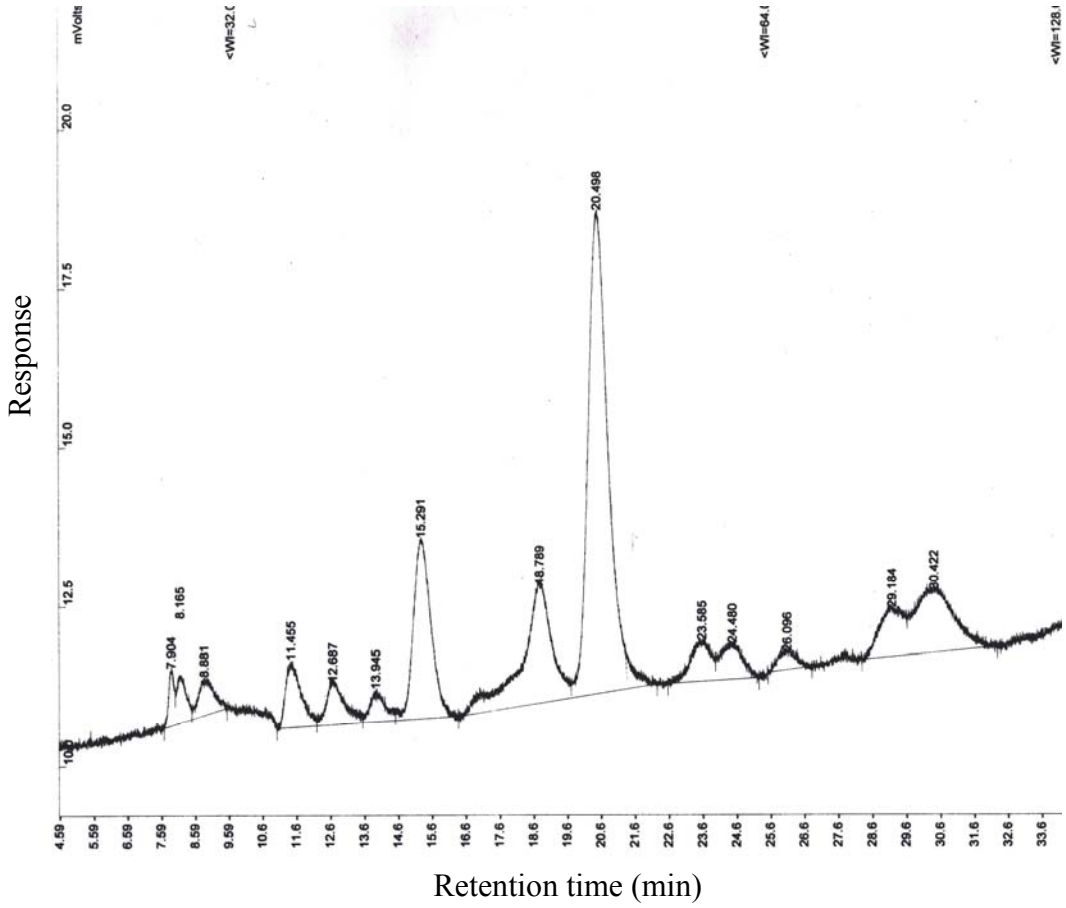
Appendix Figure F10 The HPLC chromatogram of wood vinegar prepared from one-year BAW which through SEP.



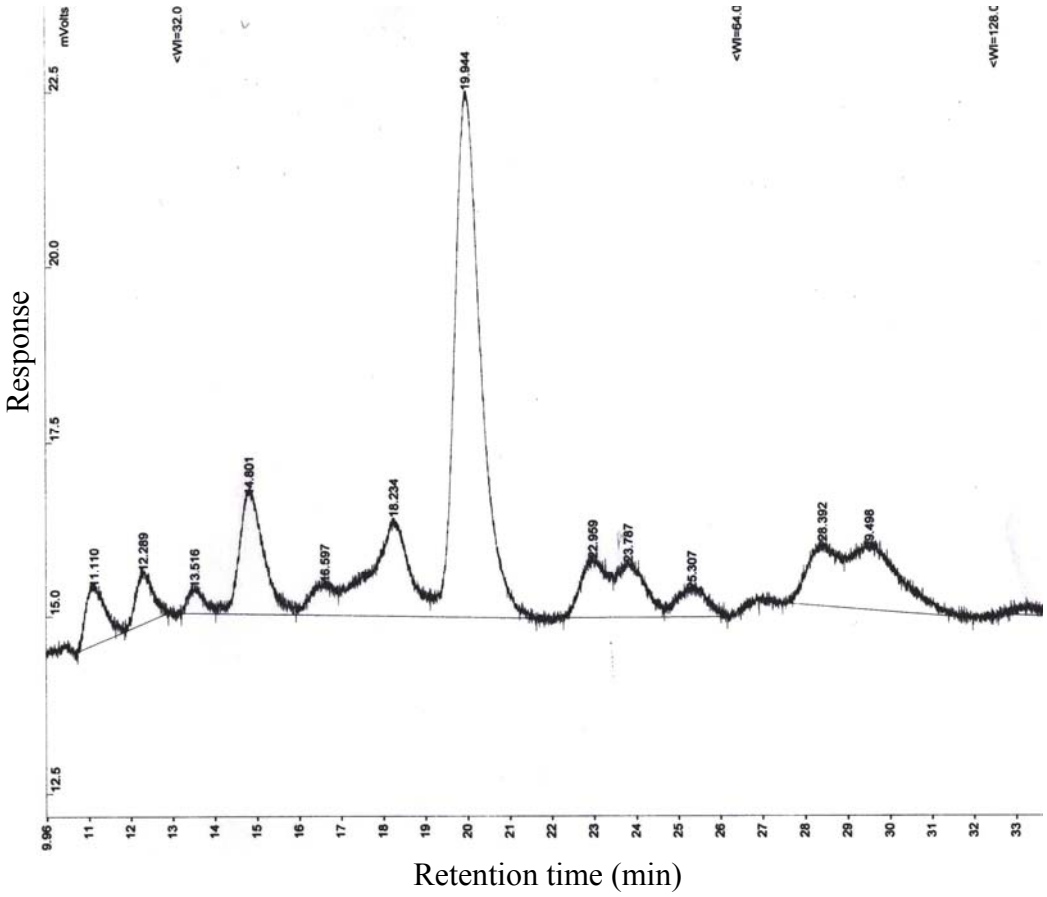
Appendix Figure F11 The HPLC chromatogram of wood vinegar prepared from two-year BAW which through SEP.



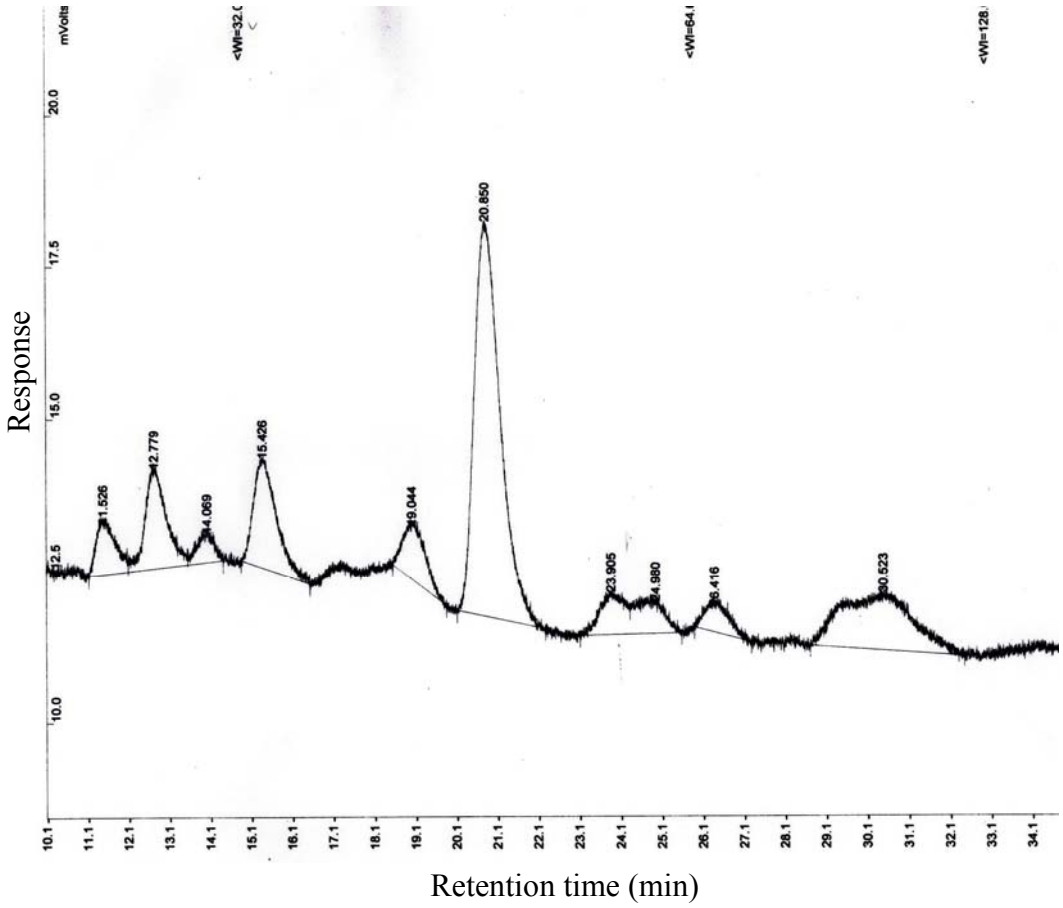
Appendix Figure F12 The HPLC chromatogram of wood vinegar prepared from three-year BAW which through SEP.



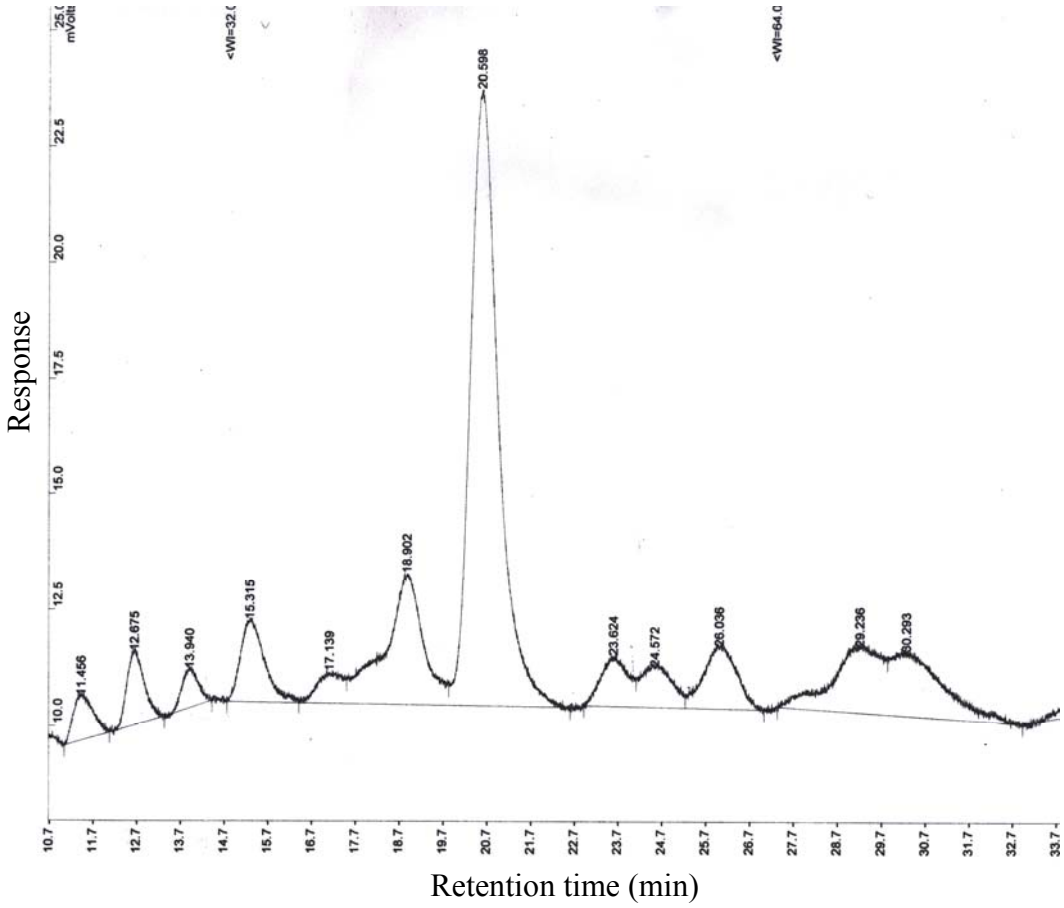
Appendix Figure F13 The HPLC chromatogram of wood vinegar prepared from one-year GO which through SEP.



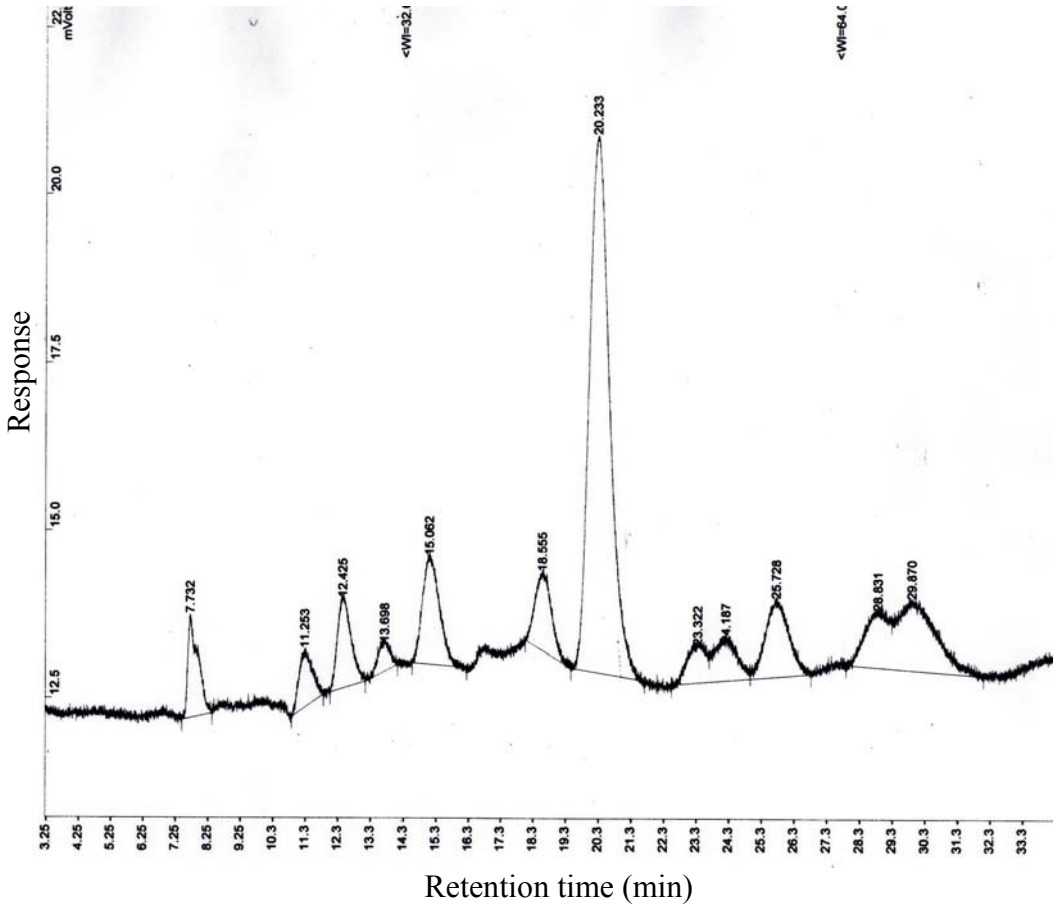
Appendix Figure F14 The HPLC chromatogram of wood vinegar prepared from two-year GO which through SEP.



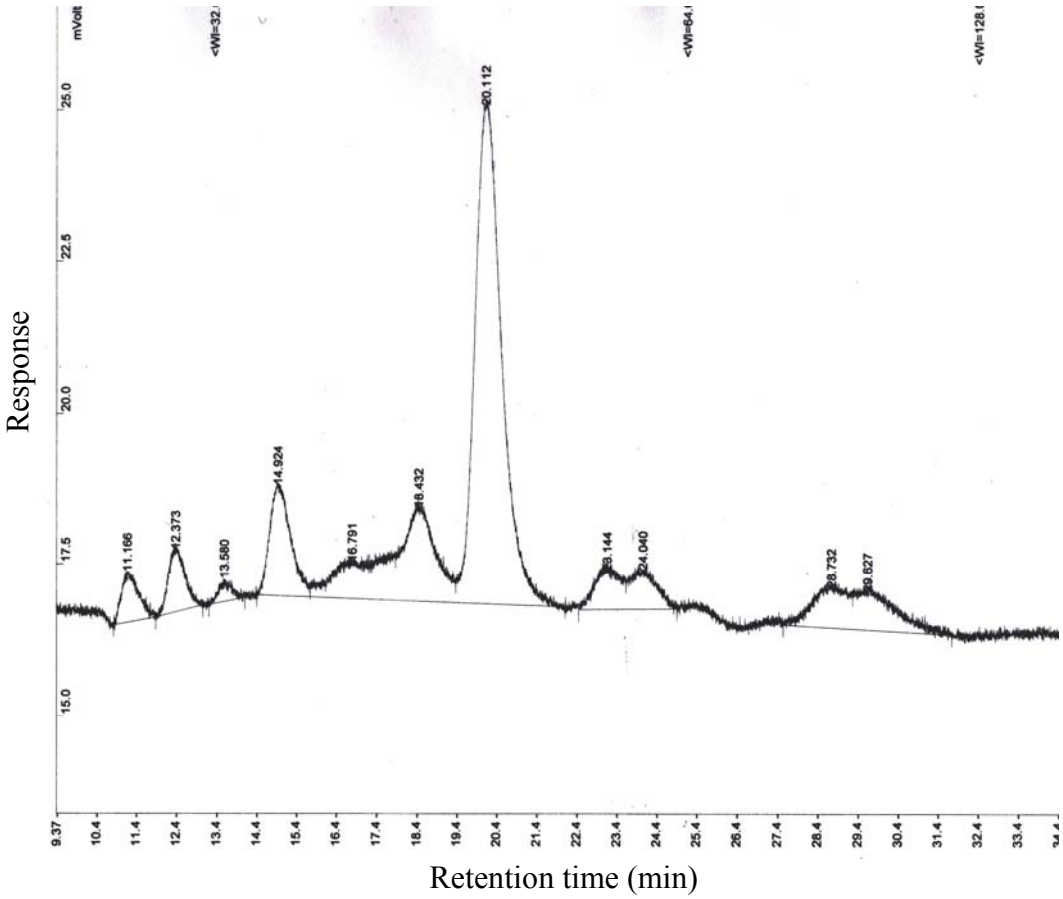
Appendix Figure F15 The HPLC chromatogram of wood vinegar prepared from three-year GO which through SEP.



Appendix Figure F16 The HPLC chromatogram of wood vinegar prepared from one-year GV which through SEP.



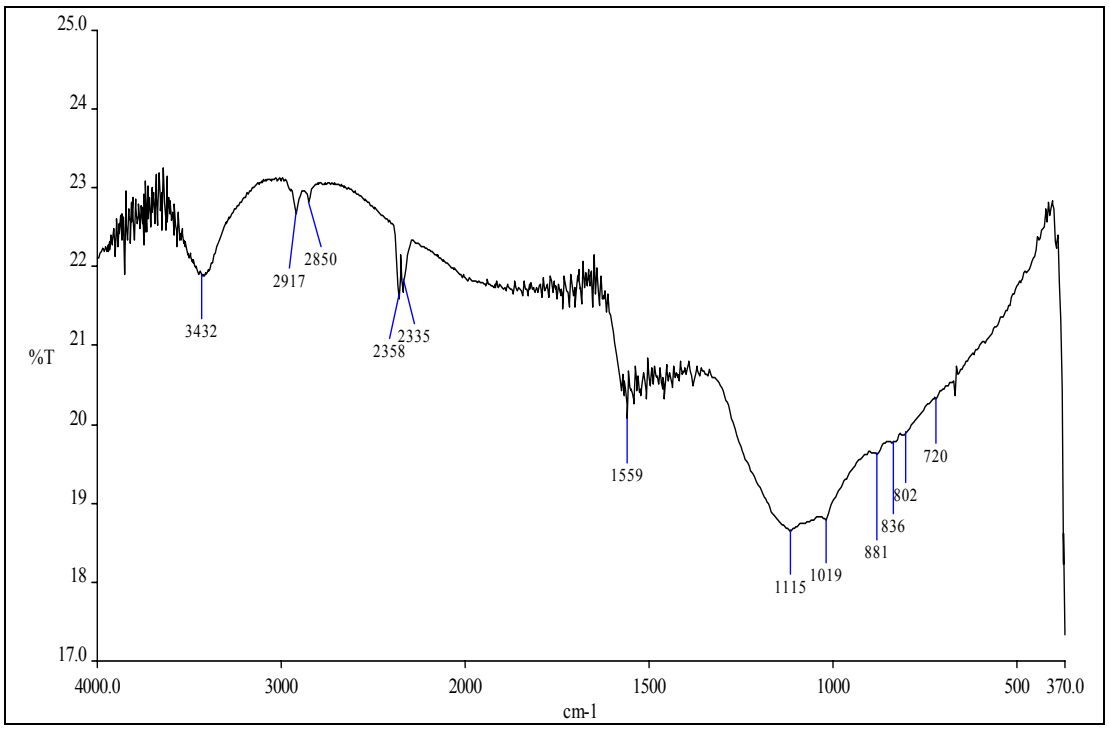
Appendix Figure F17 The HPLC chromatogram of wood vinegar prepared from two-year GV which through SEP.



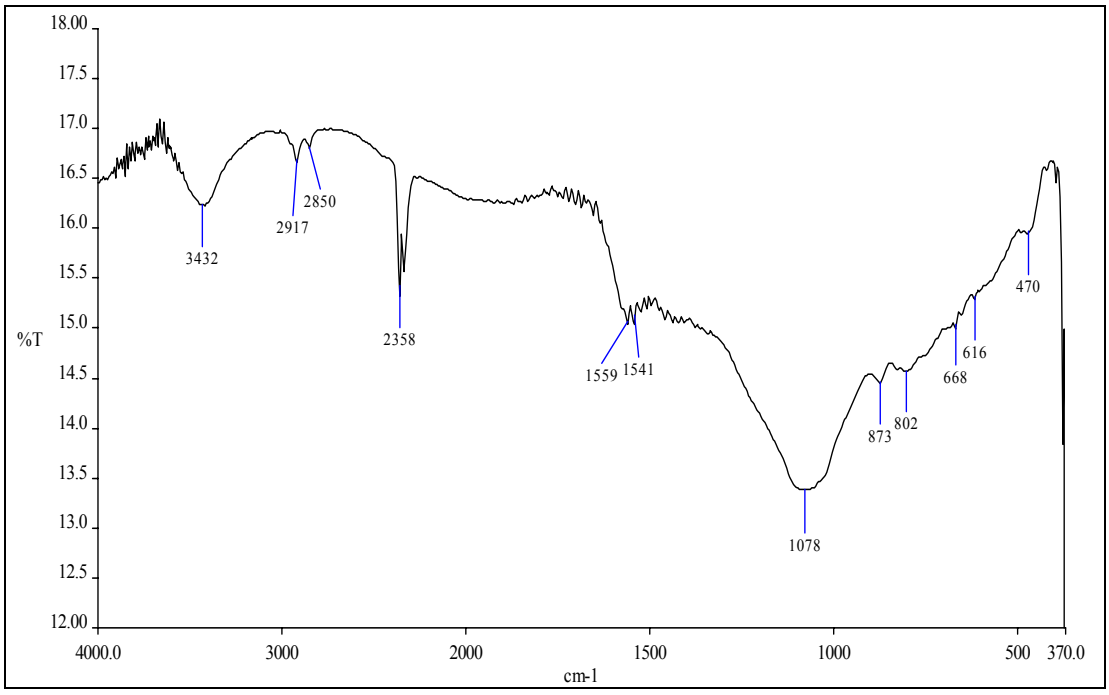
Appendix Figure F18 The HPLC chromatogram of wood vinegar prepared from three-year GV which through SEP.

APPENDIX G

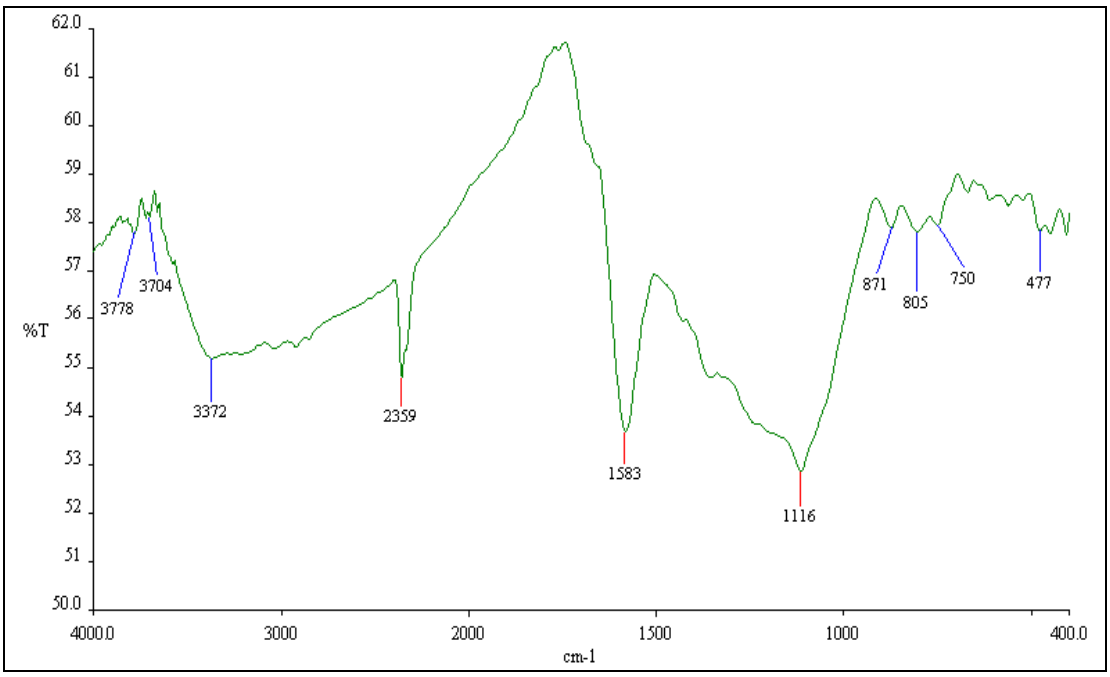
The FT-IR spectrum of all samples



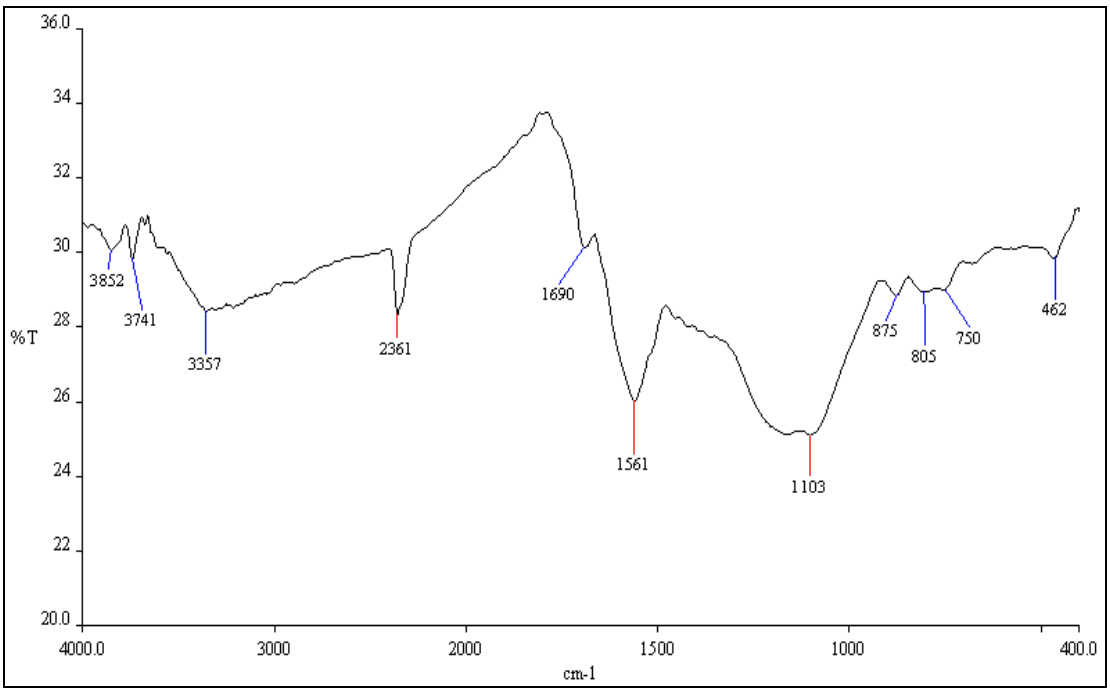
Appendix Figure G1 The FT-IR spectrum of Fluka.



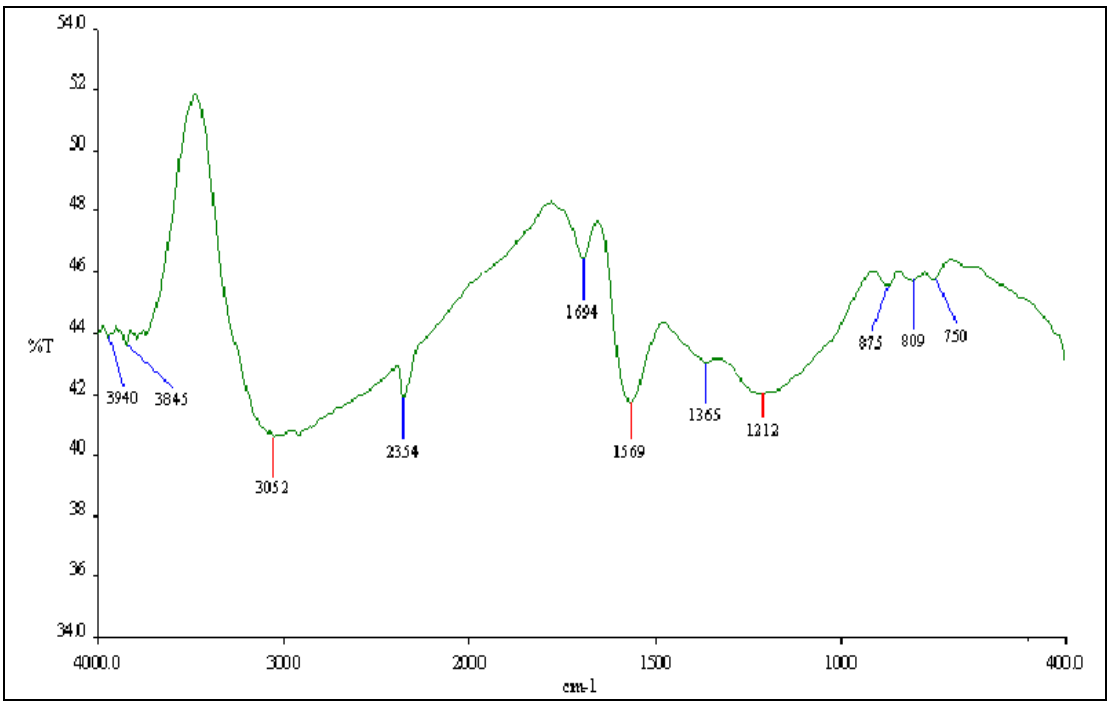
Appendix Figure G2 The FT-IR spectrum of Bunton**.**



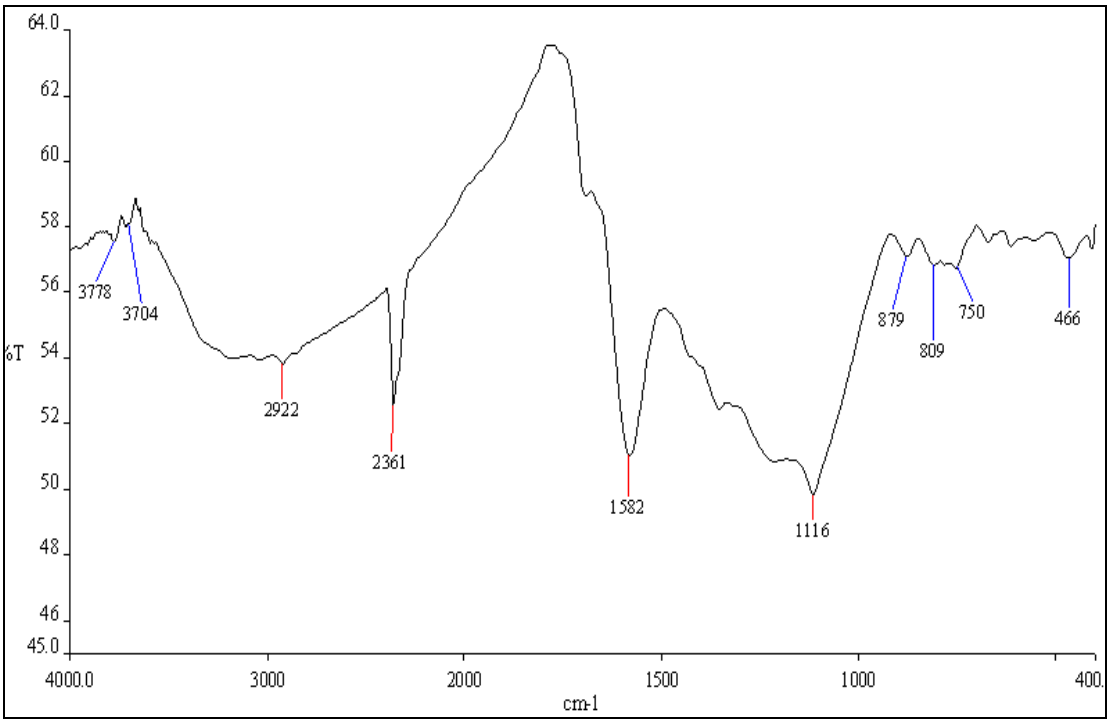
Appendix Figure G3 The FT-IR spectrum of BAWC 1.



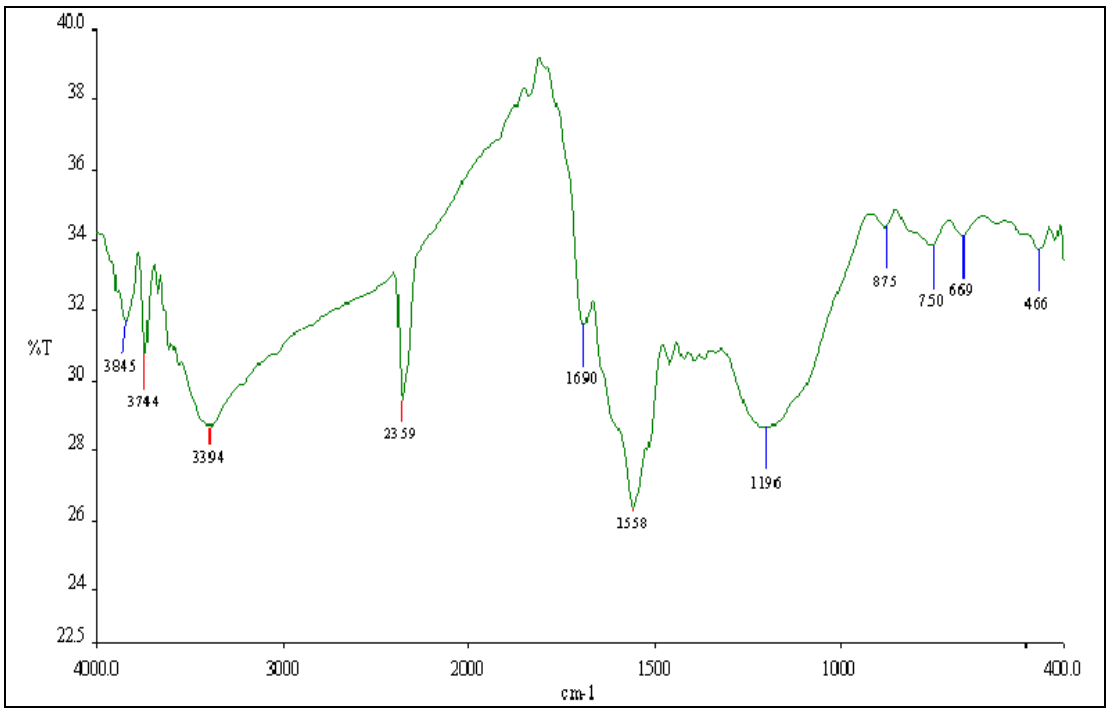
Appendix Figure G4 The FT-IR spectrum of BAWP 1.



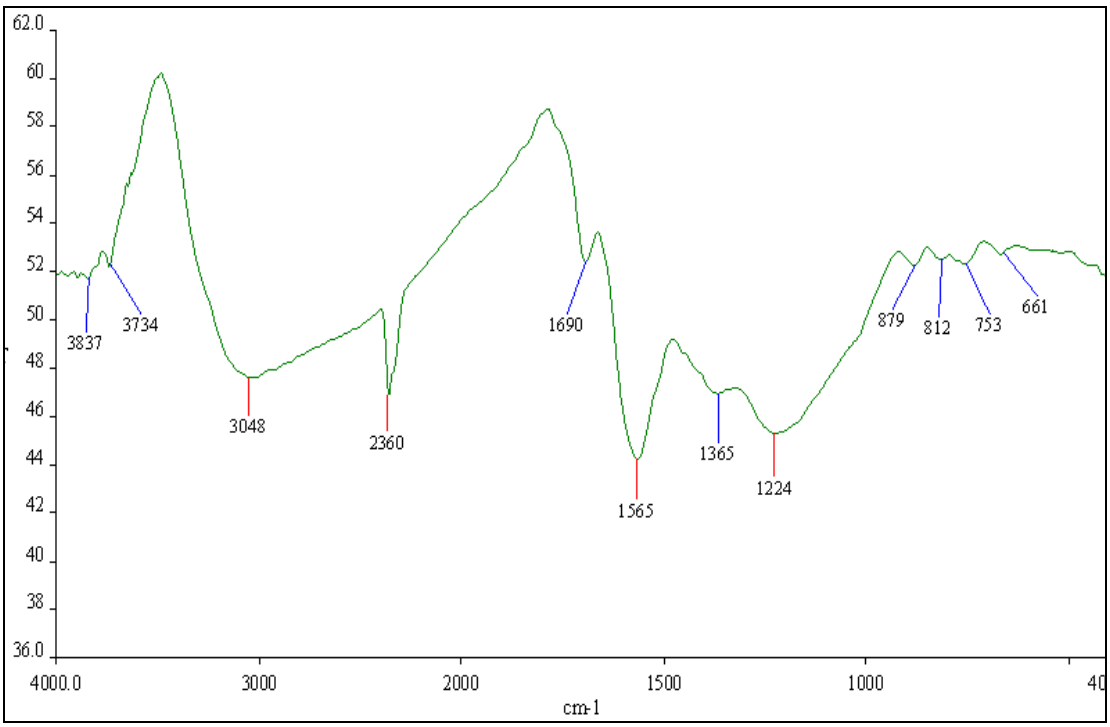
Appendix Figure G5 The FT-IR spectrum of BAWK 1.



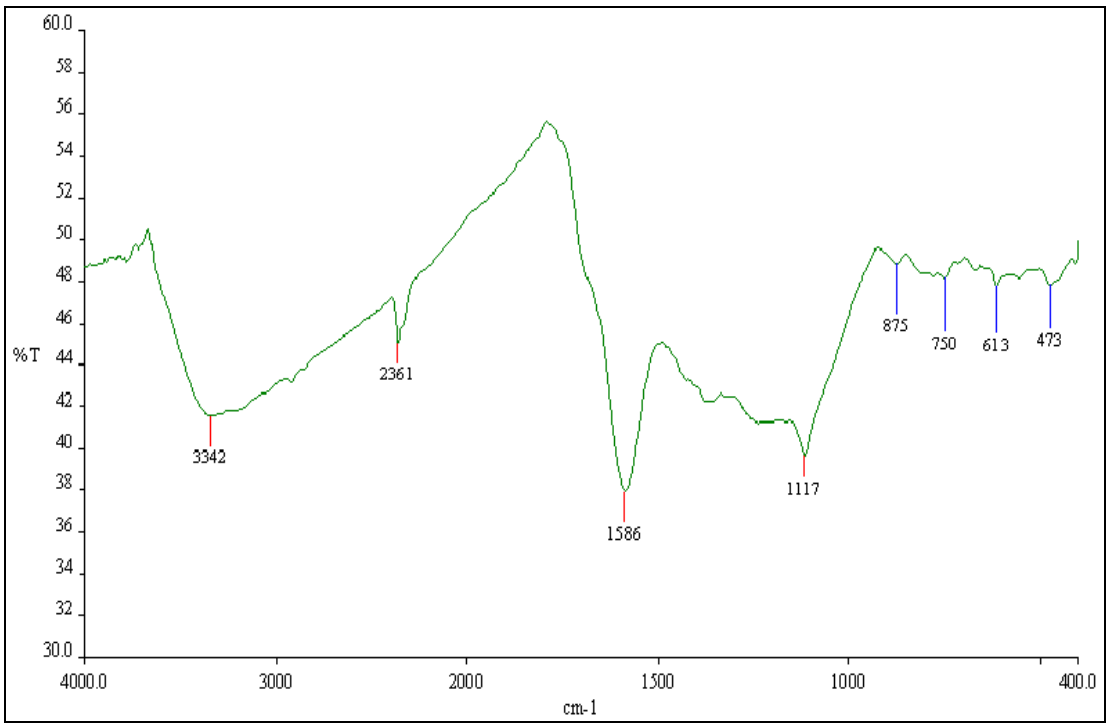
Appendix Figure G6 The FT-IR spectrum of BAWC 2.



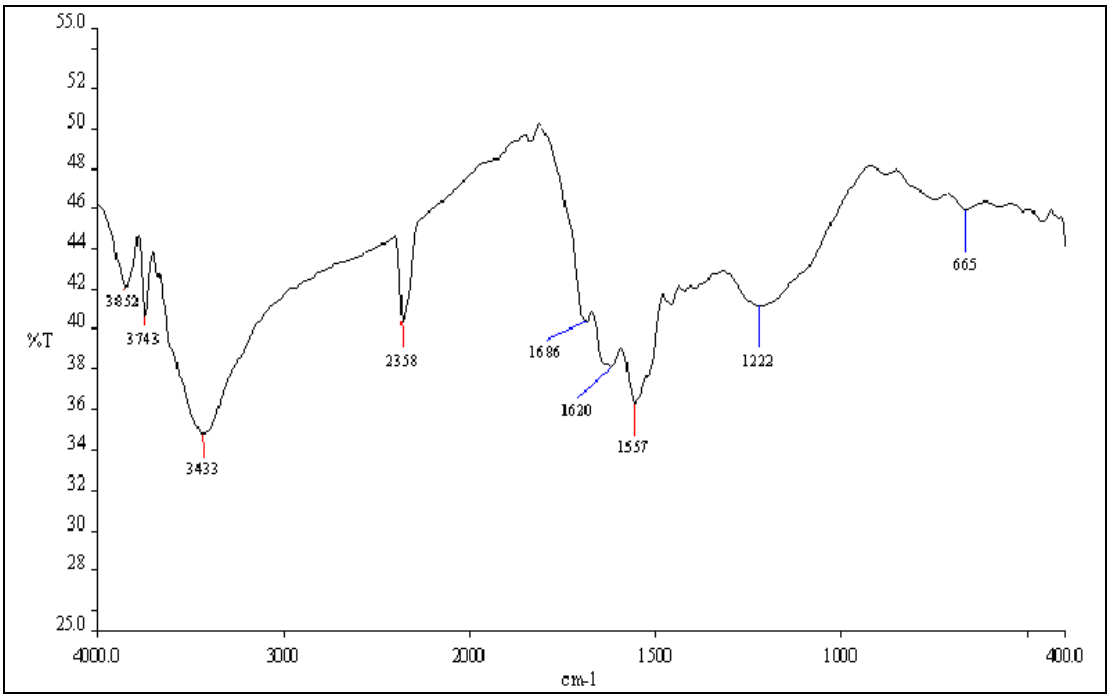
Appendix Figure G7 The FT-IR spectrum of BAWP 2.



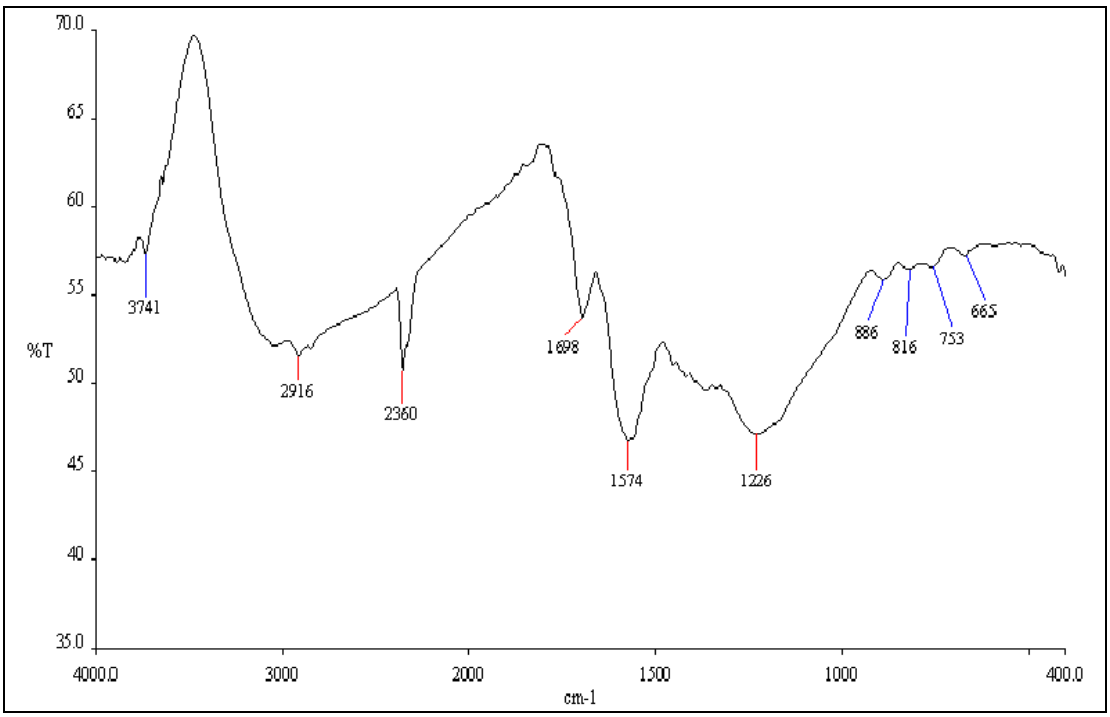
Appendix Figure G8 The FT-IR spectrum of BAWK 2.



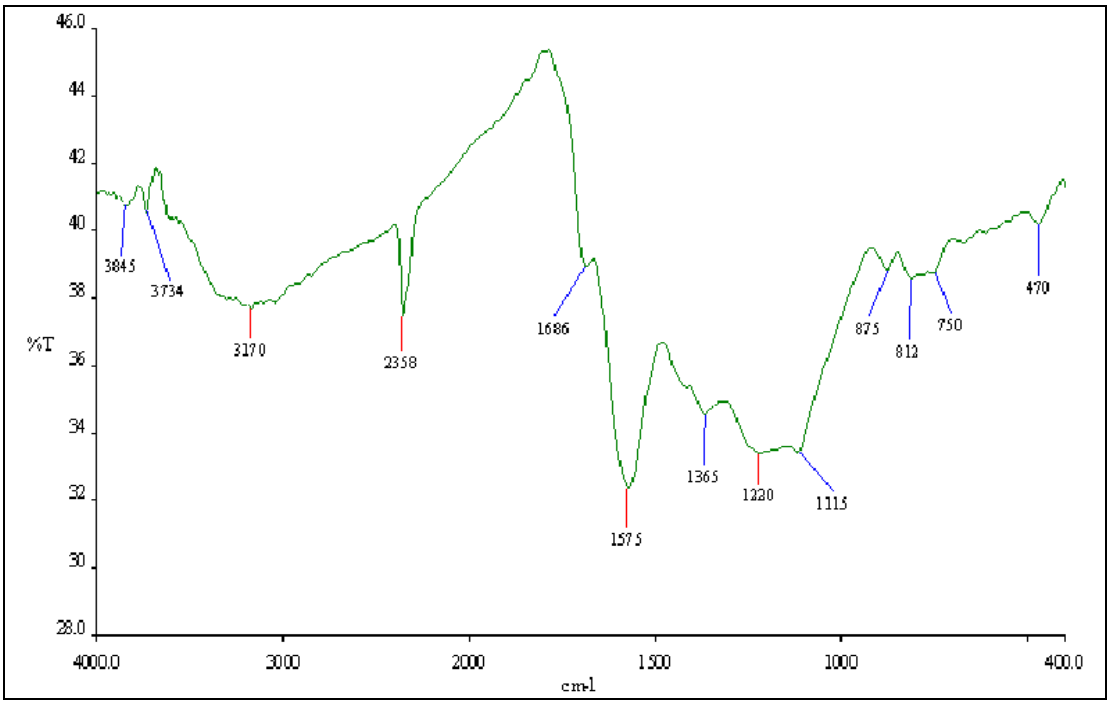
Appendix Figure G9 The FT-IR spectrum of BAWC 3.



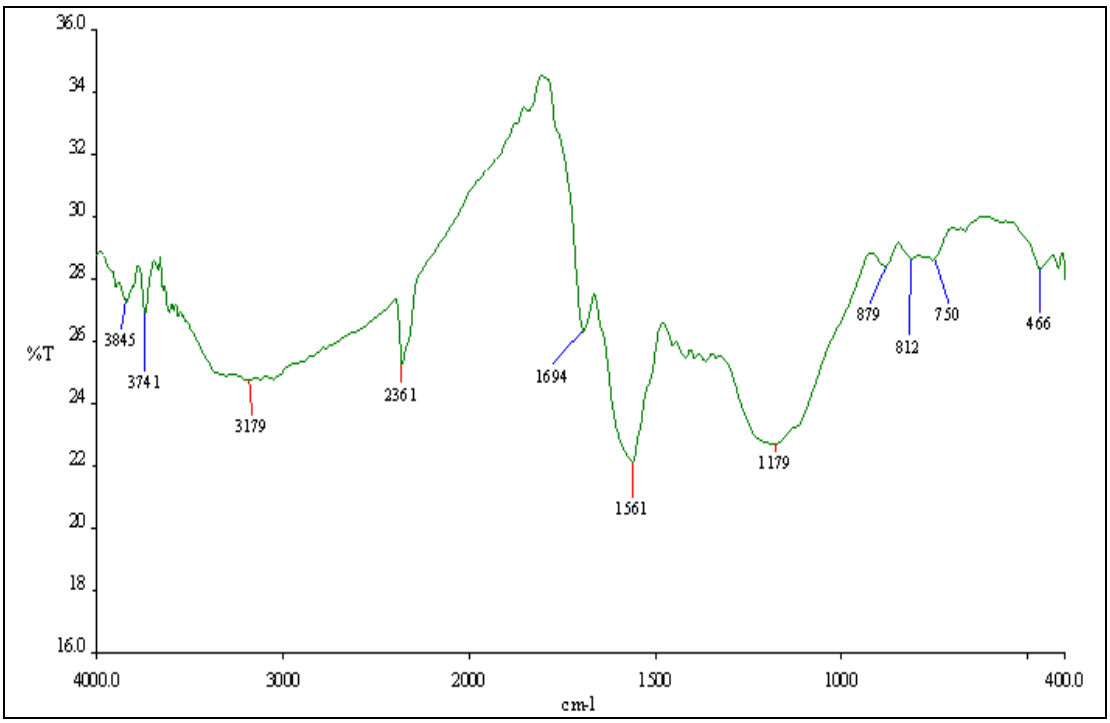
Appendix Figure G10 The FT-IR spectrum of BAWP 3.



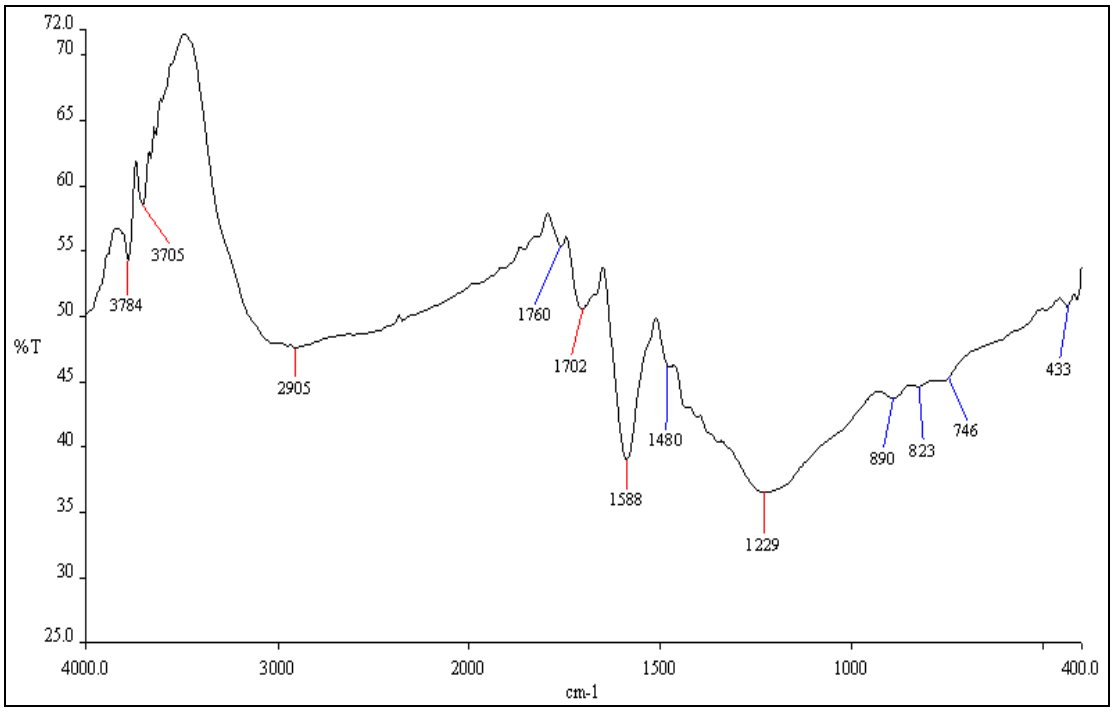
Appendix Figure G11 The FT-IR spectrum of BAWK 3.



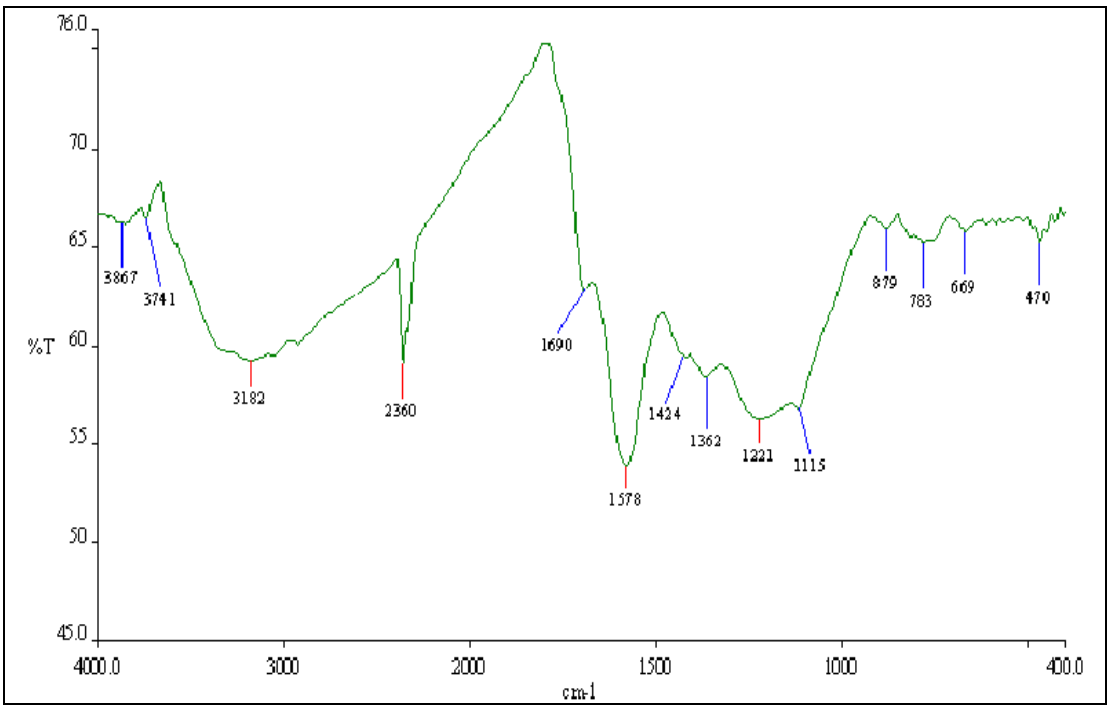
Appendix Figure G12 The FT-IR spectrum of GOC 1.



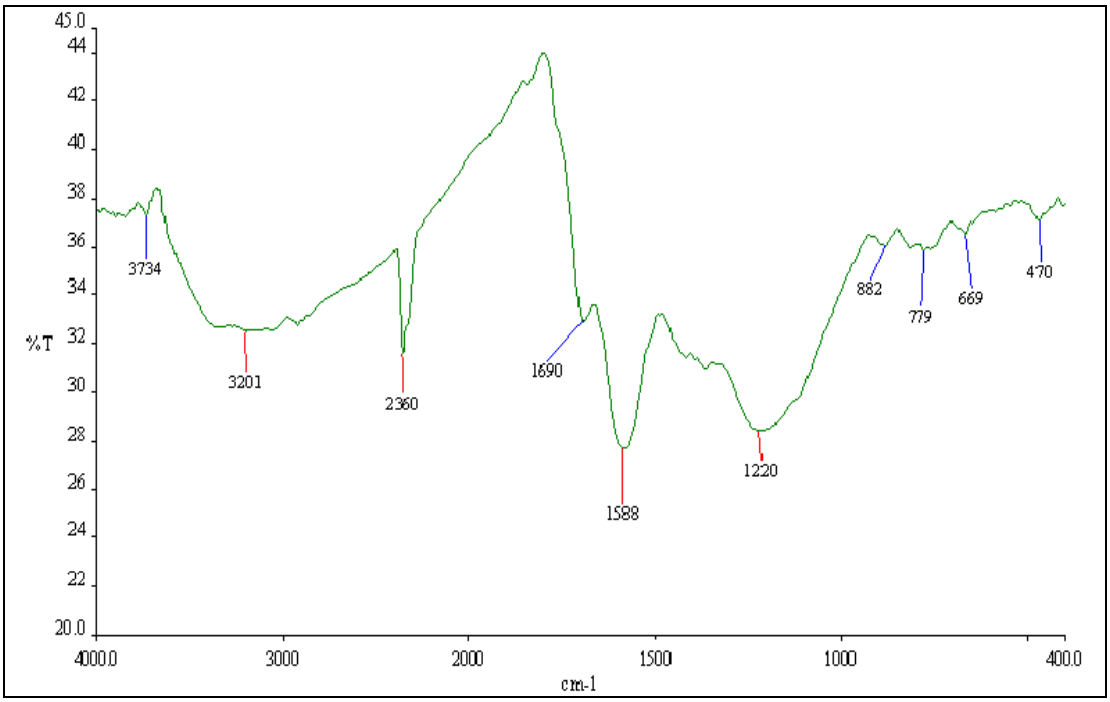
Appendix Figure G13 The FT-IR spectrum of GOP 1.



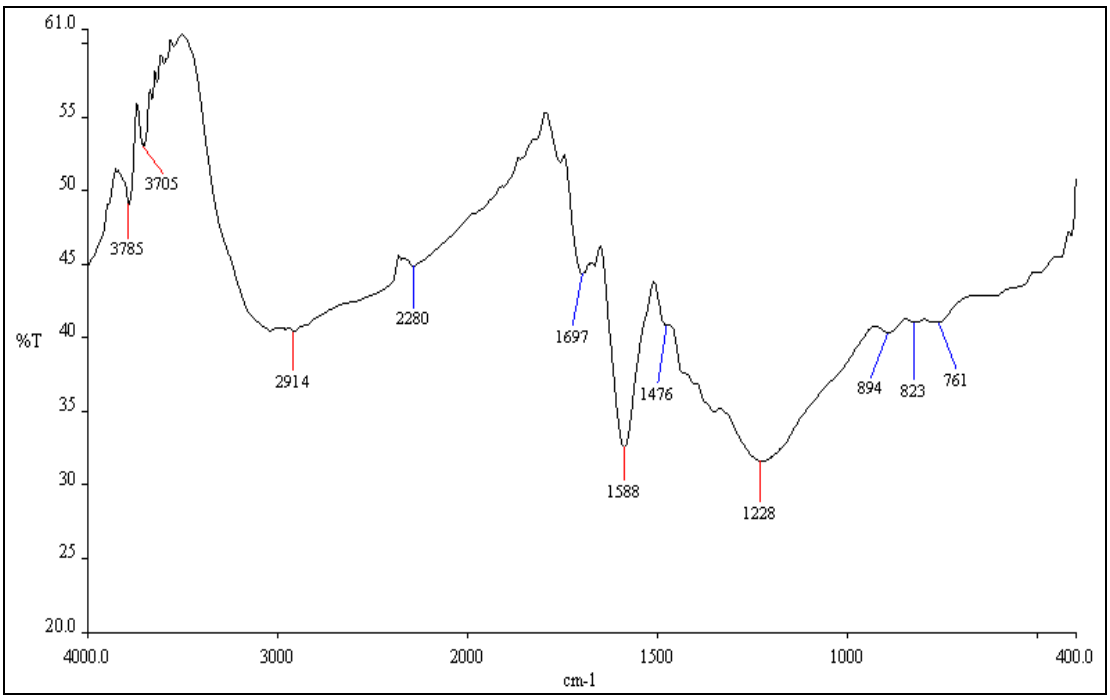
Appendix Figure G14 The FT-IR spectrum of GOK 1.



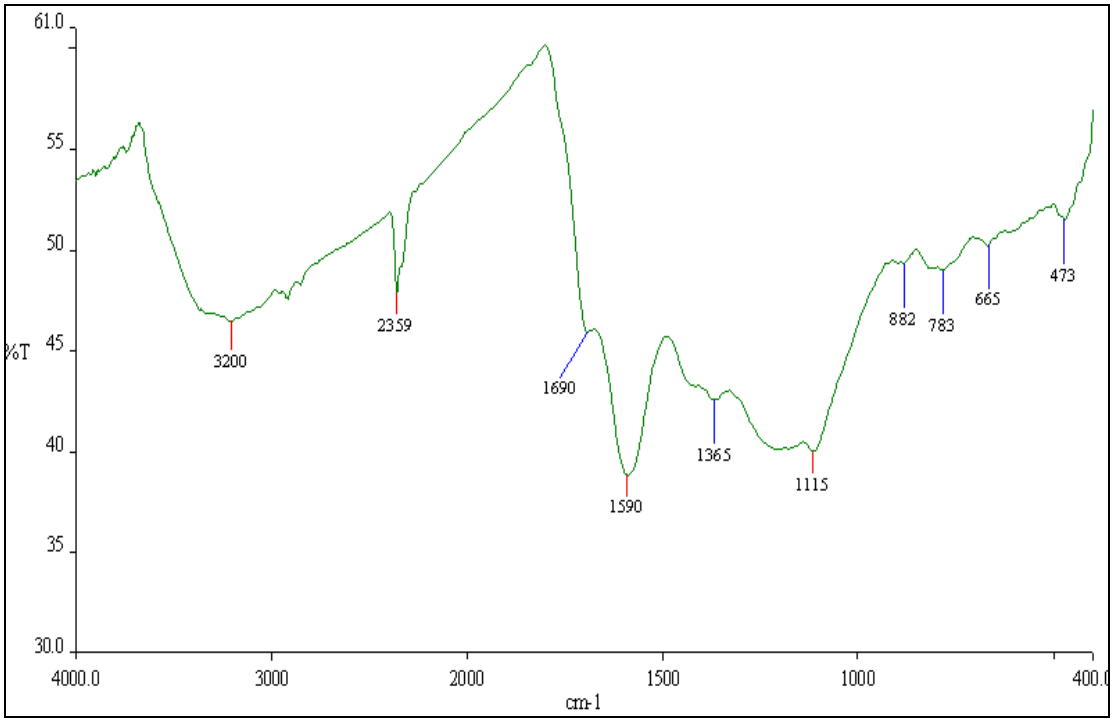
Appendix Figure G15 The FI-IR spectrum of GOC 2.



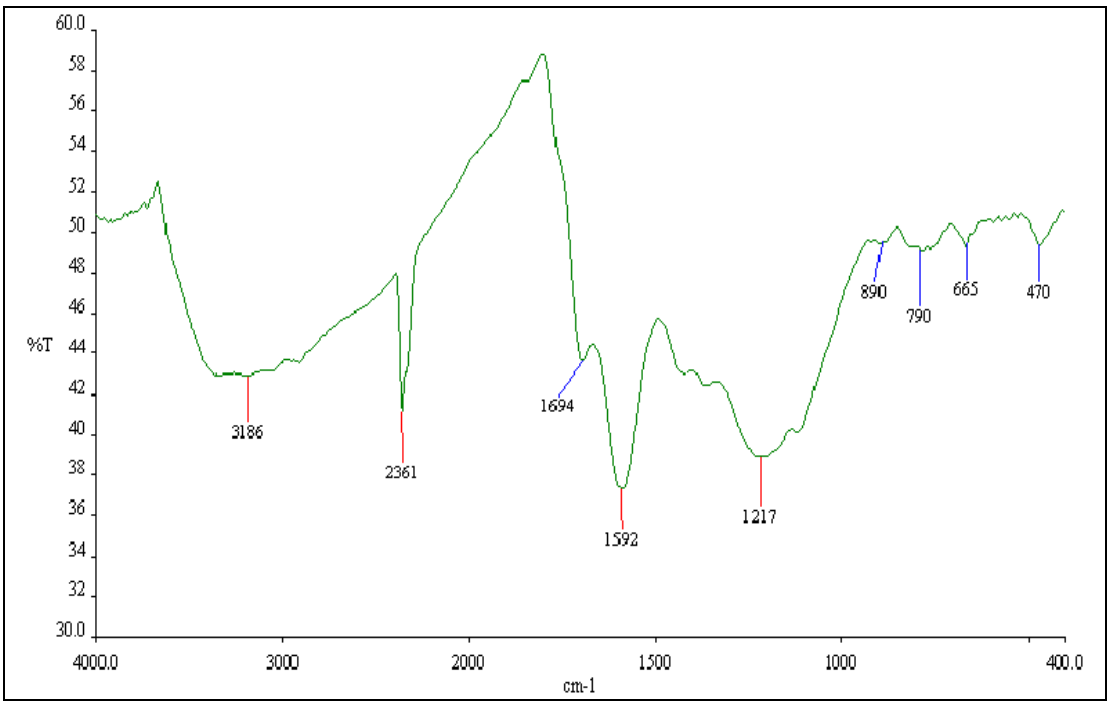
Appendix Figure G16 The FT-IR spectrum of GOP 2.



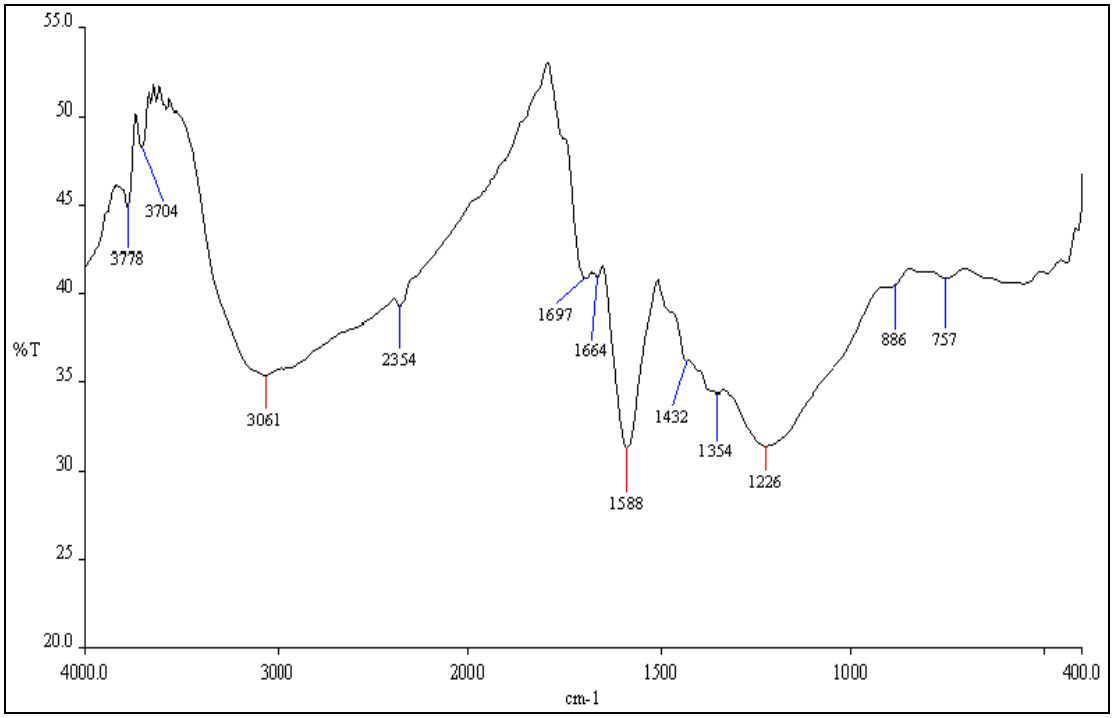
Appendix Figure G17 The FT-IR spectrum of GOK 2.



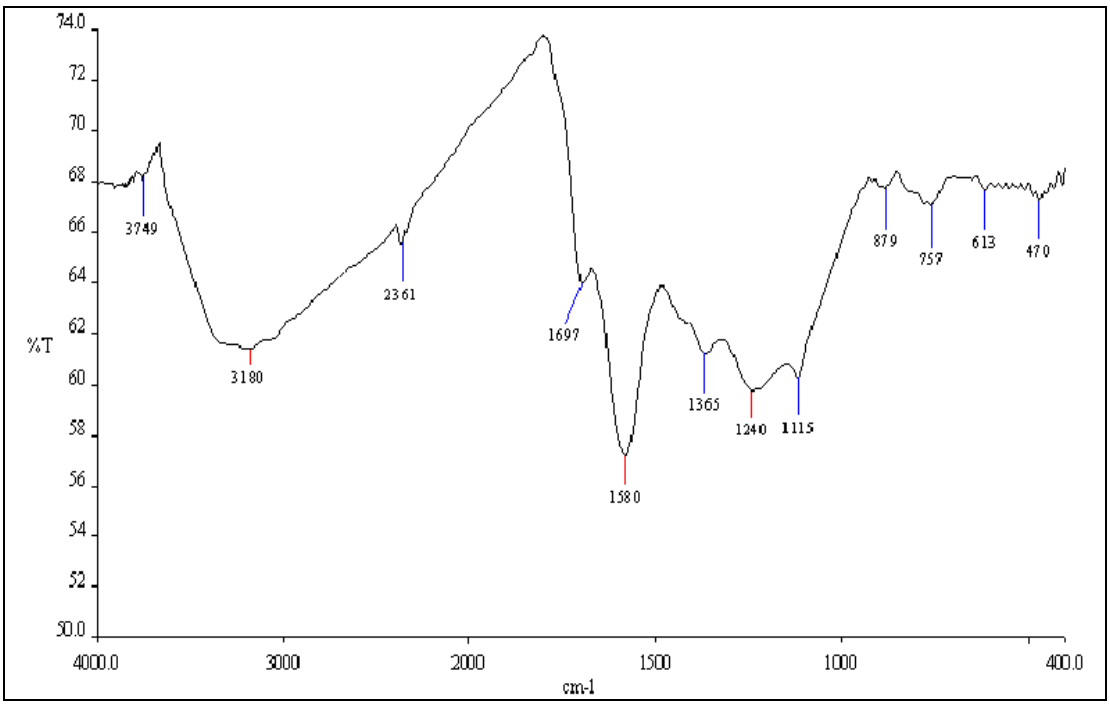
Appendix Figure G18 The FT-IR spectrum of GOC 3.



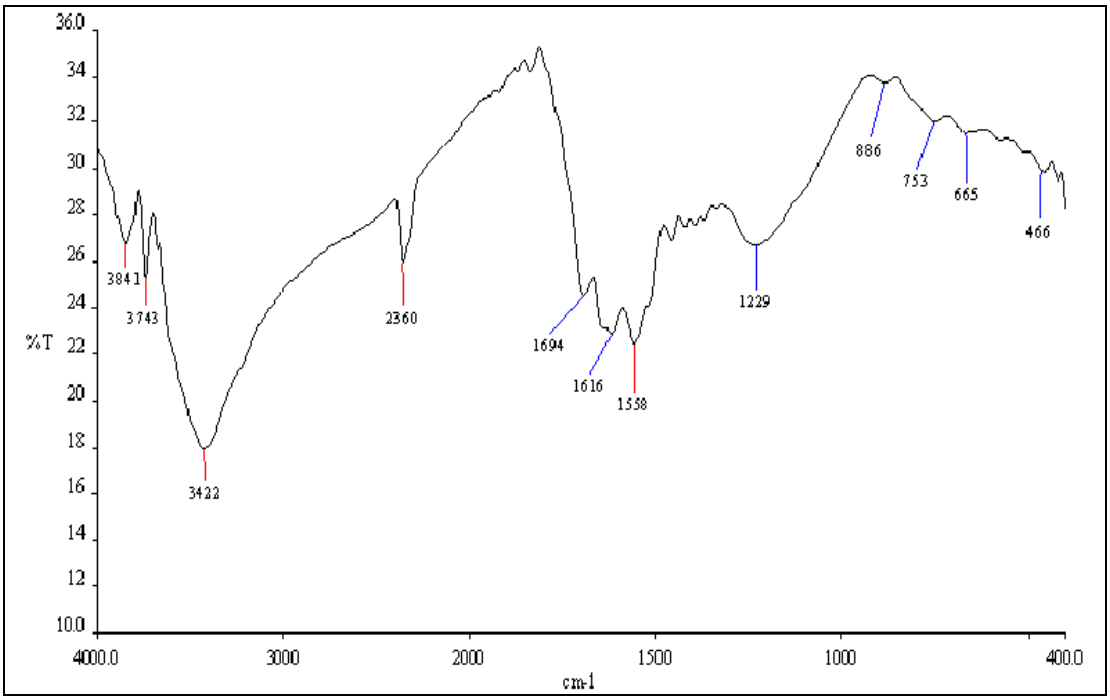
Appendix Figure G19 The FT-IR spectrum of GOP 3.



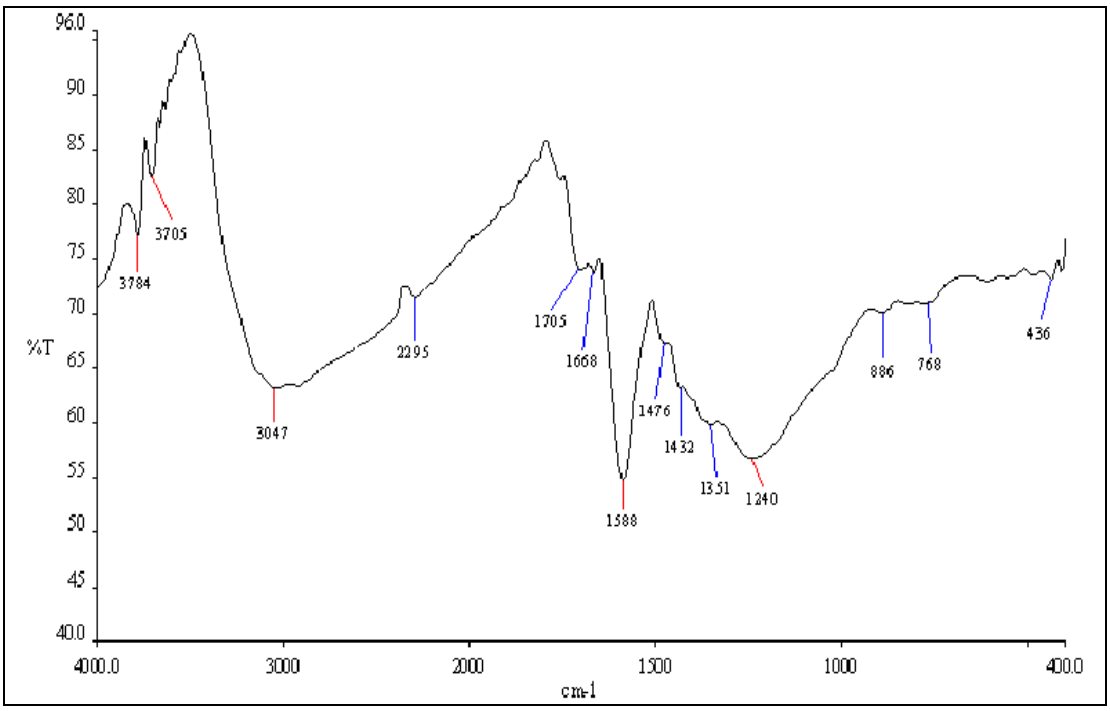
Appendix Figure G20 The FT-IR spectrum of GOK 3.



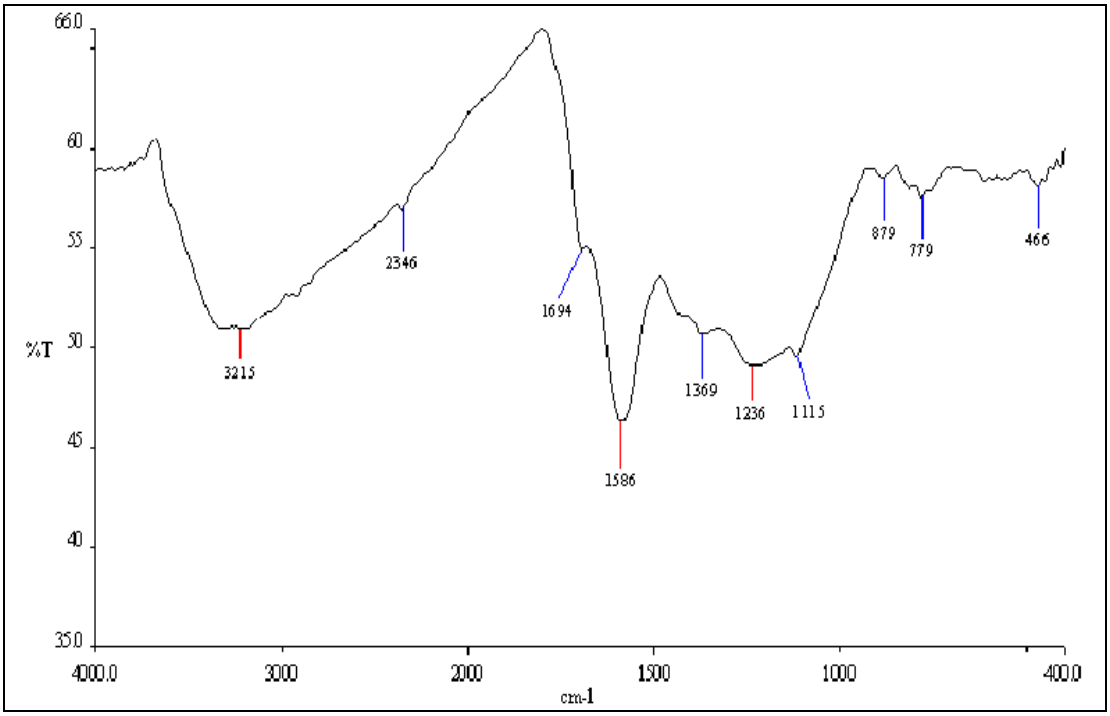
Appendix Figure G21 The FT-IR spectrum of GVC 1.



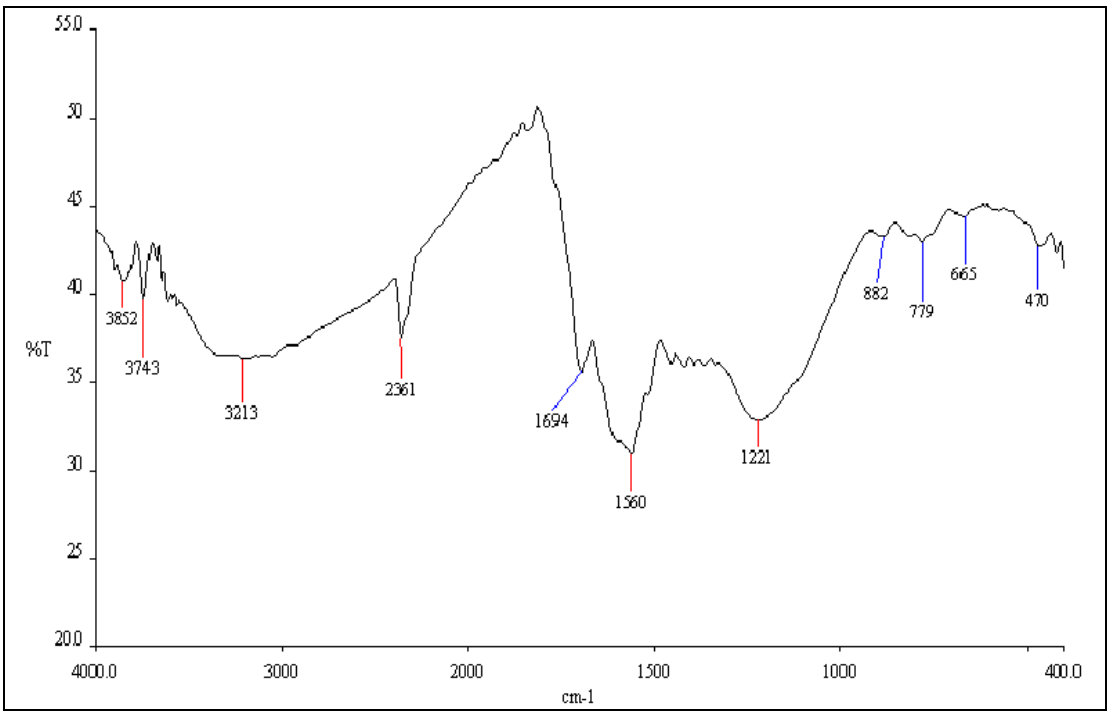
Appendix Figure G22 The FT-IR spectrum of GVP 1.



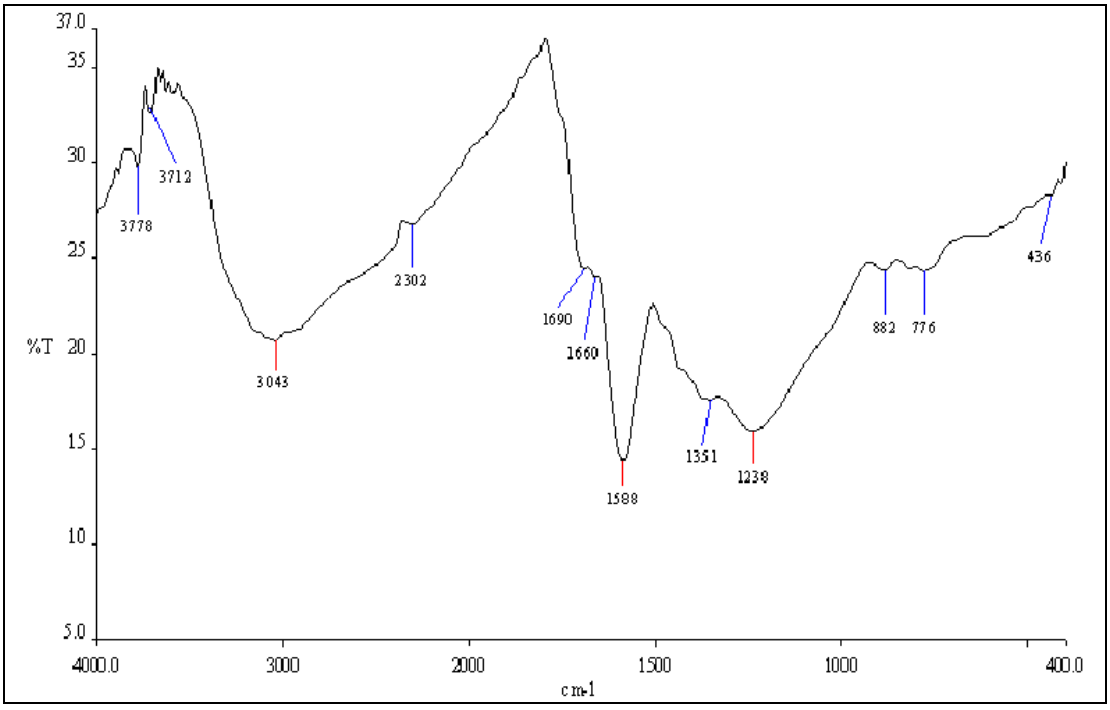
Appendix Figure G23 The FT-IR spectrum of GOK 1.



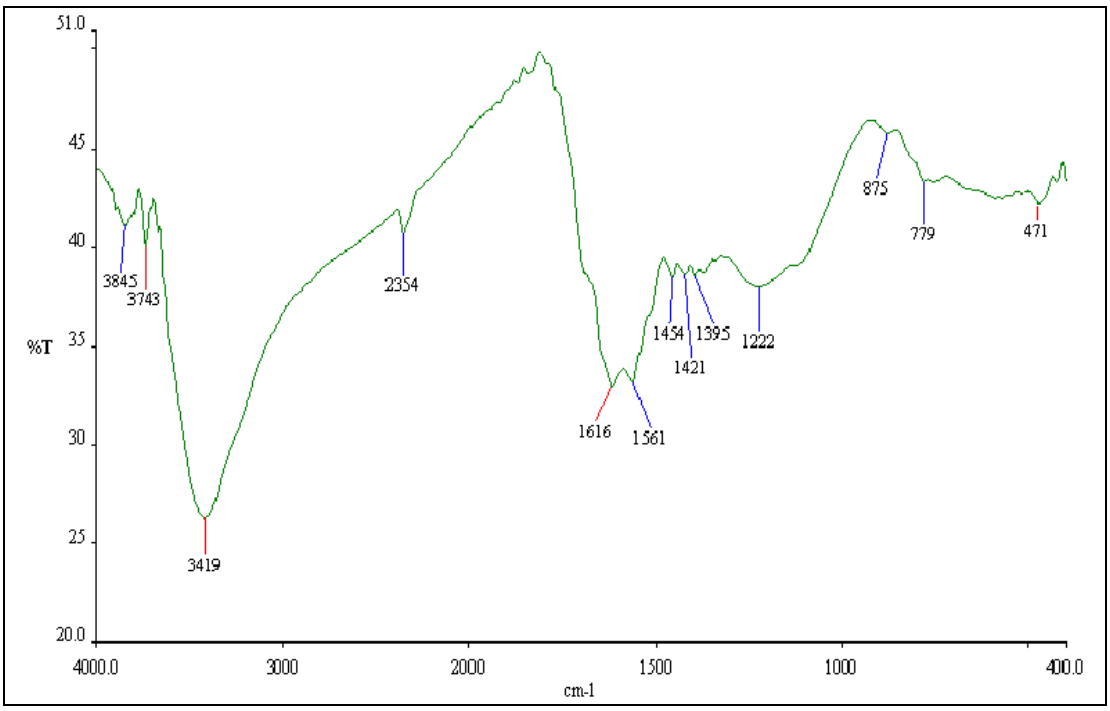
Appendix Figure G24 The FT-IR spectrum of GOC 2.



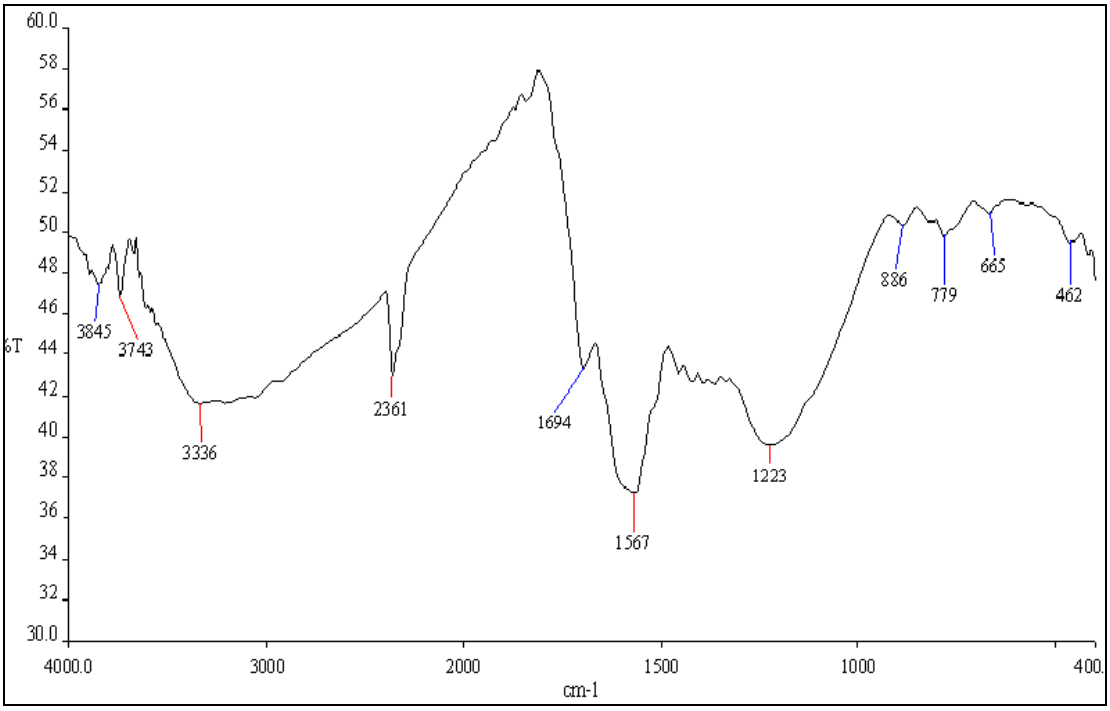
Appendix Figure G25 The FT-IR spectrum of GVP 2.



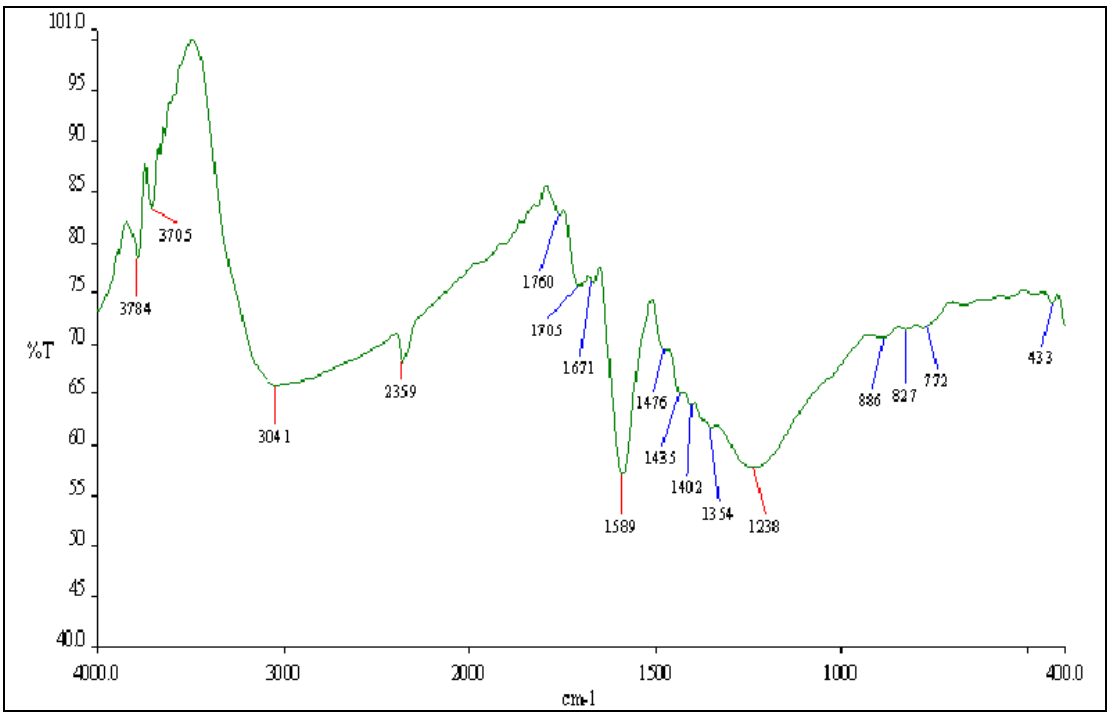
Appendix Figure G26 The FT-IR spectrum of GVK 2.



Appendix Figure G27 The FT-IR spectrum of GVC 3.



Appendix Figure G28 The FT-IR spectrum of GVP 3.



Appendix Figure G29 The FT-IR spectrum of GVK 3.

APPENDIX H

Calculation of moisture, ash and volatile matter content in activated carbon

1. Calculation of moisture

Calculate the moisture content as following;

$$\text{Moisture (weight \%)} = [(C - D)/(C - B)] \times 100$$

where B = weight of capsule with cover (g)
C = weight of capsule with cover plus original sample (g)
D = weight of capsule with cover plus dried sample (g)

For example, the moisture content of BAWC 1 was calculated as follows;

B = 3.5271 g
C = 5.5278 g
D = 5.3820 g

$$\text{Moisture} = [(C - D)/(C - B)] \times 100$$
$$= [(5.5278 - 5.4316)/(5.5278 - 3.5271)] \times 100$$
$$= 7.29\ \%$$

Therefore, the moisture content of BAWC 1 was 7.29 %.

2. Calculation of ash content

Calculate the ash content as following;

$$\text{Total ash (\%)} = [(D - B)/(C - B)] \times 100$$

where B = weight of crucible (g)
C = weight of crucible plus original sample (g)
D = weight of crucible plus ashed sample (g)

For example, the ash content of BAWC 1 was calculated as follows;

B = 4.8526 g , D = 4.9327 g
C = 5.8533 g

Total ash (%) = [(D – B)/(C – B)] x 100
= [(4.9327– 4.8526)/(5.8533 – 4.8526)] x 100
= 8.00 %

Therefore, the ash content of BAWC 1 was 8.00 %.

3. Calculation of volatile matter content

Calculate the volatile matter content as following;

Volatile matter (%) = [(C – D)/(C – B)] x 100

where B = mass of crucible with cover (g)
C = mass of crucible with cover plus original sample (g)
D = mass of crucible with cover plus de-volatilized sample (g)

For example, the volatile matter content of BAWC 1 was calculated as follows;

B = 5.1484 g , D = 5.6992 g
C = 6.1490 g

Volatile matter (%) = [(C – D)/(C – B)] x 100
= [(6.1490 – 5.6992)/(6.1490 – 5.1484)] x 100
= 44.95 %

Therefore, the volatile matter content of BAWC 1 was 44.95 %.

APPENDIX I

Calculation of iodine number, phenol value and specific surface area

1. Calculation of iodine number

$$X/M = \frac{A-(DF \times B \times S)}{M}$$

where X/M = iodine number (mg/g)

A = $12693N_2$

B = $126.93N_1$

C = $N_1/(50 \times S)$

C = residual iodine (N)

S = sodium thiosulfate (ml)

M = carbon used (g)

N_1 = concentration of sodium thiosulfate (N)

N_2 = concentration of iodine (N)

DF = dilution factor

= (I + H)/F

I = initial iodine (ml)

H = 5% hydrochloric acid used (ml)

F = filtrate (ml)

From the result, the iodine number of BAWC 1 was calculated by ASTM D 4607 – 94.

The first result;

N_1 = 0.101 N

N_2 = 0.099 N

M = 5.0040 g

I = 100.0 ml

H = 20.0 ml

F = 50.0 ml

S = 10.90 ml

A	=	12693 x 0.101	=	1281.993
B	=	126.93 x 0.099	=	12.566
DF	=	(100 + 30)/50	=	2.6

$$X/M = \frac{A-(DF \times B \times S)}{M} = \frac{1281.993-(2.6 \times 12.566 \times 10.90)}{5.0040}$$

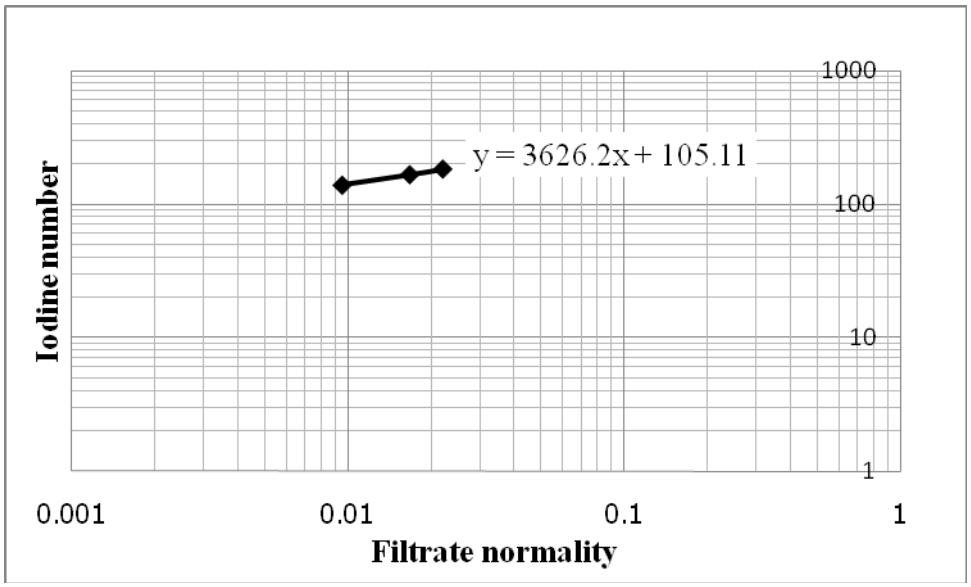
$$= 185.03 \text{ mg/g}$$

$$C = \frac{N_1}{50 \times S} = \frac{0.101}{50 \times 10.90}$$

$$= 0.0185 \text{ mg/L}$$

The second result; X/M = 167.03 mg/g, C = 0.0167 mg/L and the third result; X/M = 138.98 mg/g, C = 0.0095 mg/L

Then, the correlation between X/M and C from all results as follows;



Appendix Figure H1 The relationship between iodine number and filtrate normality**.**

The iodine number was calculated by replacing C = 0.02 mg/g in y = 3626.2x + 105.11 as follows;

$$y = 3626.2x + 105.11 = 3626.2(0.02) + 105.11$$
$$= 177.63 \sim 178 \text{ mg/g}$$

Therefore, the iodine number of BAWC 1 was 178 mg/g.

2. Calculation of phenol value

From the result, the phenol value of BAWK 2 was calculated by AWWA B600

The first result;

carbon used = 0.0503 g

initial concentration of phenol = 9.98 mg/g

initial phenol = 25 ml

$$\% \text{ of residual filtrate normality} = \frac{\text{residual phenol (mg/L) x 100}}{\text{phenol in test solution (mg/L)}}$$

$$= (5.247 \times 100)/9.98$$

$$= 52.58 \%$$

$$\% \text{ of adsorbed phenol (X)} = 100 - \% \text{ of residual filtrate normality}$$

$$= 100 - 52.58$$

$$= 47.42 \%$$

$$\text{carbon used (M)} = \frac{1000 \times \text{carbon weigh (g)}}{\text{phenol used (ml)}}$$

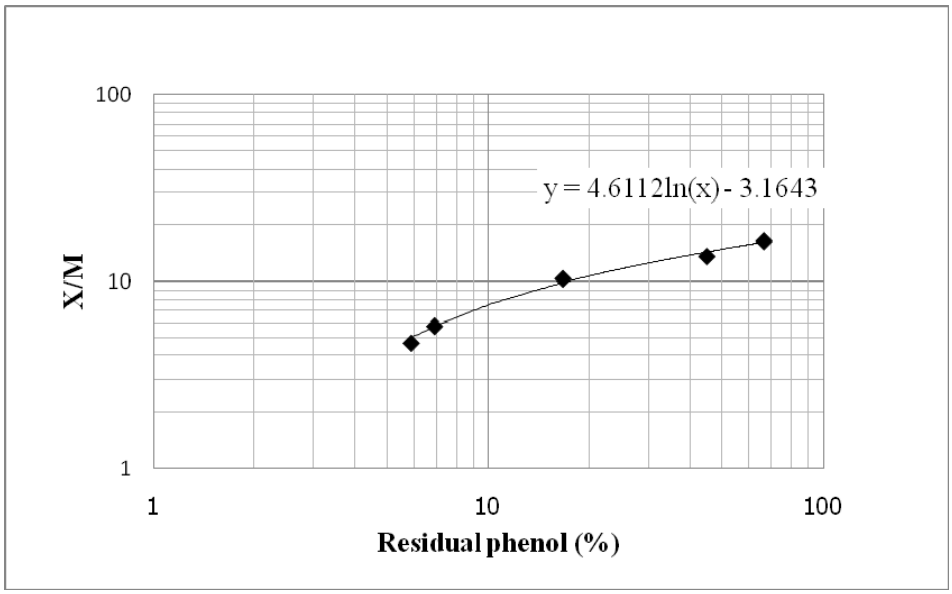
$$= (1000 \times 0.0503)/25$$

$$= 2.012 \text{ g/L}$$

$$\text{X/M} = 47.42/2.012$$

$$= 23.475$$

The next experiment was made like the first one but the amount of charcoal was increased until the phenol adsorption ability had high than 90 %. Then, the correlation between X/M and residual phenol (%) from all results as follows;



Appendix Figure H2 The relationship between X/M and residual phenol.

The X/M of 10 % residual phenol was calculated as follows;

$$y = 4.6112\ln(x) - 3.1643 = 4.6112\ln(10) - 3.1643$$
$$= 7.4534$$

Then, moisture (%) calculated by ASTM D 2867 - 04 was 6.63.

$$\text{Phenol value (mg/L)} = \frac{90}{\text{\% of X/M at 10\% of residual filtrate normality}} \times \frac{100 - \text{\% of moisture}}{100}$$

$$\text{Phenol value} = \frac{90}{7.4534} \times \frac{100 - 6.63}{100}$$

$$= 11.27 \text{ mg/L}$$

Therefore, the phenol value of BAWK 2 was 11.27 mg/L.

3. Calculation of specific surface area

$$S = \frac{Q_o}{MW} \times N \times a$$

where S = the specific surface area (m^2/g)

Q_o = the the maximum surface coverage (formation of monolayer) of sorbent (mg/g)

MW = the molecular weight (g/mol)

N = the Avogadro number (6.02×10^{23} molecule/mol)

a = the cross sectional area of adsorbate ($Å^2$)

For example, the specific surface area of BAWC 1 for adsorption of iodine was calculated as follows;

Q_o = 12.98 mg/g

MW = 126.90 g/mol

N = 6.02×10^{23} molecule/mol

a = 32.0 $Å^2$

$$S = \frac{12.98}{126.90} \times 6.02 \times 10^{23} \times 32.0 = 1.9704 \times 10^{21}\ Å^2/g$$

$$= 19.70\ m^2/g$$

Therefore, the specific surface area of BAWC 1 for adsorption of iodine was 19.70 m^2/g.

Note: Phenol; MW = 94.11 g/mol, a = 52.2 $Å^2$

Methylene blue; MW = 319.85 g/mol, a = 120.0 $Å^2$

Cd (II); MW = 112.41 g/mol, a = 61.0 $Å^2$

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