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GRADUATE SCHOOL, KASETSART UNIVERSITY

Master of Engineering (Chemical Engineering)
DEGREE

Chemical Engineering	Chemical Engineering
FIELD	**DEPARTMENT**

TITLE: An Investigation of Activated Carbon Synthesized from Jatropha Seed Coat

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THESIS

AN INVESTIGATION OF ACTIVATED CARBON SYNTHESIZED FROM JATROPHA SEED COAT

NARUMON SOMBUN

A Thesis Submitted in Partial Fulfillment of
the Requirements for the Degree of
Master of Engineering (Chemical Engineering)
Graduate School, Kasetsart University
2012

Narumon Sombun 2012: An Investigation of Activated Carbon Synthesized from Jatropha Seed Coat. Master of Engineering (Chemical Engineering), Major Field: Chemical Engineering, Department of Chemical Engineering. Thesis Advisor: Associate Professor Penjit Srinophakun, Ph.D. 77 pages.

This research studiedon the characteristics of activated carbon prepared from jatropha seed coat. Three activation processes by physical activation in the presence of CO_2, chemical activation with KOH and physico-chemical activation were compared. The effects of activation temperature (600, 700, 800 and 900 °C) and holding time (1, 2 and 3 h) on the pore structure were studied. It was found that activated carbon obtained from the chemical and physico-chemical activations provided good iodine adsorption, high specific surface area and micropore pore volume, while the physical activation gave poor iodine adsorption. The results also showed that increasing the activation temperature from 600 to 900 °C developedthe iodine adsorption (from 501.56 to 1304.31 mg/g for chemical activation and from 385.42 to 1469.40 mg/g from physico-chemical activation) and the BET surface area (from 533.05 to 1400.23 m^2/g for chemical activation and 422.24 to 1397.11 m^2/g for physico-chemical activation).The highest iodine adsorption (1510.80 mg/g) and BET surface area (1497.06 m^2/g) were obtained when the activation temperature reached to 2 h. Activated carbon synthesized from jatropha seed coat by physico-chemical activation at the final temperature of 900 °C for 2 h was then used to examine the adsorption equilibrium and kinetics of methylene blue dye at 30 °C. Equilibrium data gave good fits with Langmuir adsorption isotherm with maximum monolayer adsorption capacity 909.09 mg/g. The rate of adsorption could be best described by the pseudo-second-order equation.

Student's signature　　Thesis Advisor's signature　　____ /____ /____

ACKNOWLEDGEMENTS

First of all, I wish to express my sincerest and appreciation to my thesis advisor, Associate Professor Dr. Penjit Srinophakun, for her valuable advice, guidance and encouragement in conducting this research. Secondly, I would like to thank Assistant Professor Dr. Samorn Hirunpraditkoon and Assistant Professor Dr. Anusith Thanapimmetha who are co-advisors for their suggestions, guidance and encouragement in conducting this research. Dr. Maythee Saisriyoot and Assistant Professor Dr. Sittinun Tawkaew for being members of the thesis committee whose comments are especially helpful.

The author sincerely appreciates the Jatropha school, Kasetsart University Kamphaeng Saen Campus and Center of Excellence for Jatropha for providing the jatropha seed coat used in this research. Also the Department of Chemical Engineering, Faculty of Engineering, King's Mongkut University of Technology North Bangkok and Kasetsart University for providing sample preparation and laboratory instruments, i.e., Thermal Gravimetric Analyzer (TGA) and UV-vis-Spectrophotometer etc. The author very grateful Mr. Suthat Turapan and all members of Bioprocess Engineering Laboratory for kindness help of them.

I specially would like to thank to my family (my mother, sister and brother in law) for their assistance and cheerful moral and social support.

Narumon Sombun

May 2012

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INTRODUCTION

In recent years, activated carbon has become widely used for many applications such as water and gas purifications, metal extraction and medicine as it has high surface area and low cost. However, most of the available commercial activated carbons in Thailand are produced from palm shell and it is expensive. Therefore, seeking for other low-cost carbonaceous materials for activated carbon production is highly desirable.

Jatropha curcas Linn. fruit is now become well known and high popularity for the production of biodiesel. It is an easily growing plant and its seed contains high oil yield. Generally, jatropha seed or physic nut seed consists of about 40-42 wt% seed coat and 58-60 wt% kernels which contain about 50 wt% oil for residue extraction (Singh *et al*., 2008). Its residues such as shell and seed coat and seed cake can be maximum beneficial by various applications. Shell can be used to produce briquette charcoal for combustion, and seed coat can be fed for gasification. Ultimately, seed cake can be used as organic manure and fertilizers. On the other hand, seed coat can be utilized as activated carbon for removal of heavy metals from wastewater (Dastgheib and Rockstraw, 2001).

This study, therefore, aims to synthesize activated carbon from jatropha seed coat and determined its adsorption capacity with methylene blue dye. Jatropha seed coat based activated carbon was prepared by physical, chemical and physico-chemical activations. The influences of activation temperature and holding time were investigated. Moreover, activated carbon was then treated with two different solutions acid after synthesized: boiled water and hydrochloric, in order to determine the activated carbon properties and the adsorption capacities. Finally, the adsorption isotherm and kinetics of methylene blue were also investigated.

OBJECTIVES

1. To investigate the effects of activation temperature and holding time on the properties of activated carbon synthesized from jatropha seed coat

2. To gain the appropriate synthesis method for the methylene blue adsorption

3. To illustrate the adsorption isotherm and kinetics of the optimum activated carbon obtained

LITERATURE REVIEW

1. Jatropha curcas

Jatropha curcas (Figure 1) is a species of flowering plant in the spurge family, Euphorbiaceae, which is a native plant for the American tropics, most likely Mexico and Central America. Common names include Barbados nut, purging nut and physic nut.

1.1 Botanic characteristic

Jatropha curcas is a small perennial plant which can be reached to a height of 6 meters and a trunk diameter of 2 meters. Life span is more than 20 years. Leaflets are purplish green while young trunk is green. When it becomes older, the height of trunk which is brown color can reach to 12 meters. Generally, *J. curcas* is resistant to a high degree of aridity.

Jatropha fruit is green with spherical shape whereas a ripe fruit has yellow skin. Inside the fruit,there are 3 jatropha seeds which are black and oval shape. The weight of 100 seeds is about 69.8 grams. With screw extruder, one jatropha seed consists of 35% by weight of oil. Therefore, the use of jatropha seed 4 kilograms can extract to be 1 liter of oil and 3 kilograms residue.

1.2 Utilization of *Jatropha curcas*

1.2.1 Seed cake: Normally, the residue of jatropha seed can be used in many applications, for example fuel, adsorbent, compost and feed.Its residue has a number of nutrient leftover whereas some poisonous substances are included too.

1.2.2 Leaf extract: Leaf extract is potentially used in folk medicine. In agriculture, it is utilized as insecticidal agents and a source of the production of insecticidal compounds.

Figure 1 *Jatropha curcas* Linn.

2. Activated carbon

Activated carbon is a synthesized highly porous product made from carbonaceous material by carbonization and activation processes. Raw materials which normally can be used to synthesized the activated carbon should contain high carbon and volatile (Lua and Guo, 2005). Constant properties and low cost materials are other factors of their used consideration.

Both organic and inorganic materials are able to use as raw material for the production of activated carbon. However, organic materials are mainly used and can be divided into 3 groups. These are cellulosic material from plant, coal and animal parts. The details of these materials are given in Table 1.

Although all organic materials can be converted into activated carbon,the specific characteristics are basis analyses prior to nature of the raw material and activation technique used in their production such as the nature of the activating agent, and the conditions of the carbonization and activation processes.

Table 1 Materials used as activated carbon products (Hassler, 1997).

Organic material			Inorganic material
Material from plant	Coal	Animal part	
Bagasse	Coal	Blood	Flue dust
Beet-sugar sludge	Graphite	Bone	Lampblack
Carbohydrate	Lignin	Leather waste	Rubber waste
Cereal	Lignite		Petroleum acid sludge
Coconut shell	Oil shale		Pulp-mill waste
Nut shell	Peat		Distillery waste
Coffee bean	Petroleum coke		
Corncob and corn stalk			
Seed hull and husk			
Fruit pit			
Kelp and seaweed			
Molasse			
Sawdust			

2.1 Types of activated carbon (Marsh and Reinoso, 2006)

Activated carbons are complex products which are difficultly classified on the basis of their behavior, surface characteristics and preparation methods. However, some classification can be made for general purpose based on their physical characteristics as follows:

2.1.1 Particle size

2.1.1.1 Powdered activated carbon (PAC) is active carbon made in form of powders or fine granules less than 150 micron in size. PAC is not commonly used in a dedicated vessel, due to the high head loss that would occur. It is generally added directly to other process units, such as raw water intakes, rapid mix basins, clarifiers, and gravity filters.

2.1.1.2 Granular activated carbon (GAC) has a relatively larger particle size compared to powdered activated carbon. This activated carbon is always used for gas and vapor adsorptions such as water treatment, deodorization and separation of components of flow system.

2.1.2 Pore size

2.1.2.1 Micropore is activated carbon which has pore size less than 2.0 nm. This active charcoal is usually used for gas adsorption.

2.1.2.2 Mesopore is active carbon containing pore with diameters between 2.0-50.0 nm. It is generally used for either catalytic reaction or decolorization.

2.1.2.3 Macropore is activated carbon which has pose size over 50.0 nm. It is used to promote adsorbate for the easily adsorbed into micropore.

2.1.3 Types of adsorbate

2.1.3.1 Gas adsorbent is activated carbon which is used for gas, odor and organic vapor.

2.1.3.2 Color adsorbent is activated carbon which is used for decolorization.

2.1.3.3 Metal adsorbent which is used to adsorb metal such as copper, lead and platinum etc.

2.2 Activated carbon structure

The basic structure of activated carbon is closely approximated by pure graphite, as can be seen in Figure 2. The graphite crystal composes of the layers of fused hexagons. Within hexagonal graphite, the layers of hexagonal arrangement of carbon atoms are described as graphene layers. These layers do not lie immediately above and below each other but are displaced to form an ABABAB sequence. The density of hexagonal graphite is around 2.25 g/cm^3. The distance between the layers is 0.335 nm and the distance between two bond carbon atoms is 0.142 nm. Within the graphite layers, the bonding is trigonal sp^2-hybrid σ-bonds with delocalized π-bonds within the layers. The interlayer spacing of 0.335 nm, being larger than the 0.142 nm of the C □ C bond, indicates no chemical bonding between the layers with the force of attraction being limited to van der Waals forces. This distance between the value of 0.153 and 0.132 nm for Csp^{3}□ □ Csp^3 bonding as in ethane, and Csp^2==Csp^2 bonding as in ethene. Resonance considerations indicate that the C to C bonding within a graphite layer has about one-third double bond character. Such graphite structure refers to as AB graphite.

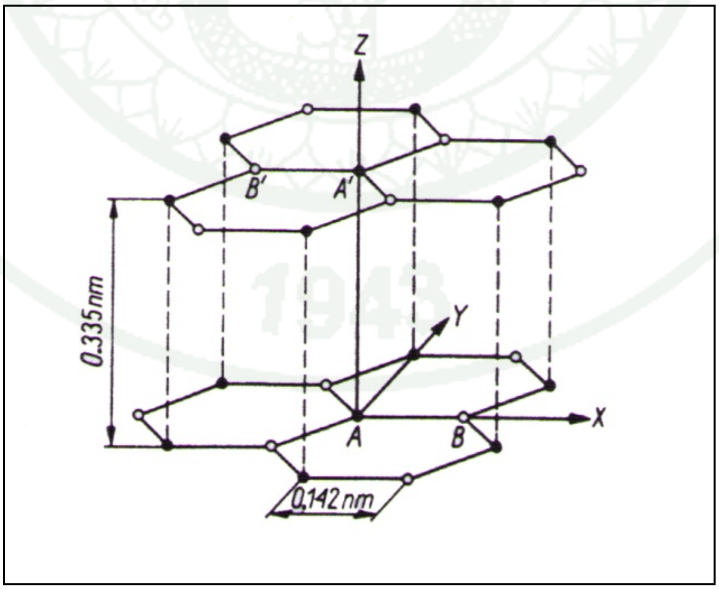

Figure 2 The structure of hexagonal graphite (Hassler, 1997).

2.3 Production of activated carbon

Activated carbon can be prepared by various techniques depending on the required properties. Generally, the activated carbon processing consists of 4 steps which are material preparation, carbonization, activation and washing (Nithasanacharukul, 2001), as shown in Figure 3.

2.3.1 Material preparation

The precursor in this step, which could be either direct raw material without any preparation or charcoal, should be grinded and sieved before carbonization to improve pyrolysis efficiency. However, the grinding precursorcan be done after carbonization process in case of the raw material is too hard or too sticky. Then, the precursor must be dehydrated before the carbonization.

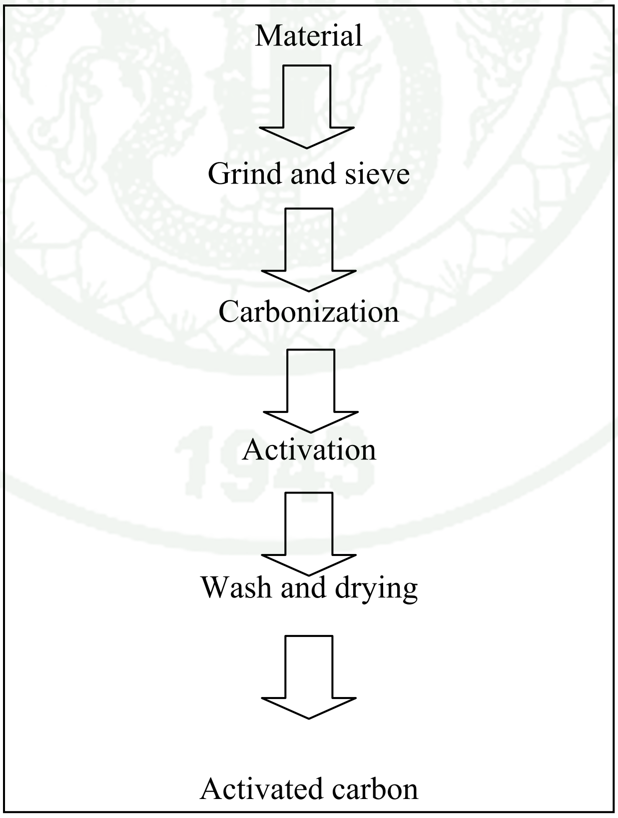

Figure 3 General activated carbon process (Nithasanacharukul, 2001).

2.3.2 Carbonization (Thapkanjana, 2005)

Carbonization is the process for eliminating some volatile elements from the material. In this step, the carbonaceous materials have to be heated in the absence of oxygen gas. During the process, most of non carbon elements such as oxygen, hydrogen and nitrogen are eliminated as volatile species by the pyrolytic decomposition of the starting raw material. The residual carbon atoms group themselves into the stack of plate, aromatic sheet cross-linked in a random manner. These aromatic sheets are irregular arranged which leaves free interstices. The carbonized char is filled with the tar or decomposition of the product, so it has to be further developed by activation process to give the rise in surface and pore volume. Three steps of the carbonization process are;

(i) Dehydration of the raw material under the temperature range 27-179 °C.

(ii) Removal of the pyrolytic products as a gas and tar oil in the structure under the temperature range 197-497 °C.

(iii) Formation of char structure with low density under the temperature range 497-847 °C.

However the char obtained from this process still has low adsorption property because of tar residue blocked in pore, therefore the activation process is introduced later to enhance the porosity of the char.

2.3.3 Activation (Marsh and Reinoso, 2006)

Activation is the step of tar removal that helps to develop the porosity and specific surface area of char. Therefore, higher adsorption property for activated carbon can be expected. Generally, the activation processcan be employed into 3 techniques as follows:

2.3.3.1 Physical activation

Physical activation is the process which the carbonized char is activated by thermal system and oxidizing gasto oxidize tar and then carbon structure is changed. This method found to be significantly developed the pores in a wide diameter range (Hu *et al*., 2007). The examples of oxidizing gas are saturated steam, carbon dioxide and oxygen.

(a) Steam activation

Steam activation is the technique using steam as an oxidizing gas. The stoichiometric equation for this reaction is

$$C_x + H_2O \rightarrow H_2 + CO + C_{x-1}\ ,\ \Delta \text{H} = 31 \text{ kcal/g mol} \tag{1}$$

In this process, oxygen atom in steam is eliminated as oxide of carbon and is endothermic reaction. It is generally operated at 750-950°C to prevent the shrinkage of pore from too high activation temperature.

(b) Carbon dioxide activation

This activation technique, carbon dioxide is used as an oxidizing agent and the reaction is endothermic, the reaction is given as follow:

$$C_x + CO_2 \rightarrow 2CO + C_{x-1},\ \Delta \text{H} = 38.2 \text{ kcal/g mol} \tag{2}$$

The equation indicates that operating temperature used in this process has to be higher than those steam activation because higher energy is required for the reaction. In general, it is operated at 1,000-1,200°C.

(c) Air activation

Air activation used oxygen in air as an oxidizing gas. The mechanism is suggested as follows:

$$C_x + O_2 \rightarrow 2CO + C_{x-2}\ ,\ \Delta \text{H} = -53.96 \text{ kcal/g mol} \tag{3}$$

$$C_x + O_2 \rightarrow CO_2 + C_{x-1}\ ,\ \Delta \text{H} = -92.4 \text{ kcal/g mol} \tag{4}$$

This method is now rarely used due to the exothermic reaction which is hard to the control temperature of the required condition. Moreover, activated carbon yield may be decreased from burning condition on carbon surface by oxygen gas.

2.3.3.2 Chemical activation

Chemical activation is a methodfor the activation of the char by chemical reagent. The chemical reagents which have been used for this technique are shown as follows **(**Hassler, 1997).

- Boric acid (H_3BO_3)
- Calcium chloride ($CaCl_2$)
- Calcium hydroxide ($Ca(OH)_2$)
- Calcuim phosphate ($Ca(PO_3)_2$)
- Dolomite ($CaMg(CO_3)_2$)
- Phosphoric acid (H_3PO_4)
- Potassium hydroxide (KOH)
- Manganese chloride ($MnCl_2$)
- Sulphuric acid (H_2SO_4)
- Zinc cholride ($ZnCl_2$)

Generally, the chemical activation step is divided into 2 types:

a) Type 1: The precursor is impregnated with chemical reagent prior to the carbonization. Later, the mixture is dried and then pyrolyzed together with activation process to form crystal.

b) Type 2: The precursor is carbonized before chemical activation to get the char. Later onthe chemical reagent is used to impregnate the char until its saturated adsorbed and the dried. Finally, the physical activation is used to develop the porosity.

In chemical activation, residue on surface of char is dissolved out while the chemical reagent is adsorbed into the pores after impregnation or soaking the precursor with chemical reagent. The mixture is then pyrolyzed at high temperature, commonly at about 500-800°C, to decompose the compoundsleft in the structure. Therefore, it results in pore development. Usually, the chemical reagents used are:

(a) Zinc chloride activation ($ZnCl_2$)

Zinc chloride acts as dehydrating agent that promotes the decomposition of carbonaceous materials during the pyrolysis process and inhibits the tar formation, and then can increase the yield of activated carbon (Yorgun *et al*., 2009). This technique, the activation temperature is generally used in the range of 500-700°C (Alhamed *et al*., 2009). However, the activated carbon needs to be washed by water or hydrochloric acid afterward to remove residue and ash for high pore and adsorption capacity.

(b) Potassium or sodium activation (KOH)

It is the method that potassium or sodium ion are penetrated between layer of crystal of carbon when potassium or sodium solution are added to raw material. At higher temperature than 700 °C, molecule of carbon dioxide, water and oxygen are eliminated and pores are developed. Although there is not clear in activated carbon formation by KOH activation, the mechanism can be illustrated as follows:

$$2KOH \rightarrow K_2O + H_2O \qquad \text{(Dehydration)}$$

$$C + H_2O \rightarrow H_2 + CO \qquad \text{(Water-gas reaction)}$$

$$CO + H_2O \rightarrow H_2 + CO_2 \qquad \text{(Water-gas shift reaction)}$$

$$K_2O + CO_2 \rightarrow K_2CO_3 \qquad \text{(Carbonate formation)}$$

2.3.3.3 Physico-chemical activation

Physico-chemical activation is the process which activates the carbon material using both chemical reagent for chemical activation such as phosphoric acid, zinc chloride and potassium hydroxide, and oxidizing gas for physical activation such as steam, carbon dioxide and air. The organic matters in char are decomposed as carbon dioxide at high temperature and then they react further with carbon atom to create pores.

Although chemical and physico-chemical activation are known as the method giving larger surface and well-developed microporosity than physical activation, the washing stage including acid solution and hot distilled water is very important and necessary to eliminate an excess chemical agent for synthesized activated carbon. The advantages and disadvantages of chemical and physical activations are shown in Table 2.

Table 2 Advantages and disadvantages of physical and chemical activation.

	Chemical activation	**Physical activation**
Advantages	- Larger surface area	- No activating agent waste
	- Lower energy cost	- Lower corrosive
	- Lower activation temperature	
Disadvantages	- Higher corrosive	- High activation temperature requirement

2.3.4 Washing and dry (Marsh and Reinoso, 2006)

Washing process is used to purify activated carbon by water or acid such as hydrochloric or nitric acid to remove either excess chemical agent in chemical activation or the ash components. This process results in to higher adsorption property of activated carbon. The washing is always done when the carbon is used in pharmaceutical preparations, food industries, wastewater treatment.

2.4 Activated carbon characteristics

The characteristics of activated carbon can be analyzed by various methods such as adsorption ability, using iodine and methylene blue tests, surface area using Brunauer, Emmet and Teller method (BET), proximate and ultimate analyses and surface morphology by scanning electron microscopy (SEM).

2.4.1 Iodine adsorption

It is a simple and quick test, giving an indication of the internal surface area of the carbon. Most of results from activated carbons the iodine number is close to the Brunauer, Emmet and Teller (BET) surface area. Therefore, iodine number is the most fundamental parameter used to characterize activated carbon performance prior to analyze by BET method as it is easy and cheaper method. It is a method of a measure of the micropore content of the activated carbon (0 to 20 Å, or up to 2 nm) by adsorption of iodine from solution. The iodine number, is expressed as milligrams of iodine per gram of carbon, is in typical range 500-1200 mg/g.

2.4.2 Methylene blue adsorption

Methylene blue (MB) is a cationic dye that most commonly used for coloring (Cazetta *et al.*, 2011). The chemical structure of this dye is shown in Figure 4. The aromatic moiety of methylene blue contains nitrogen and sulfur atoms. In the aromatic unit, dimethylamino groups attach to it. MB can cause eyes burn in humans and animals, methemoglobinemia, cyanosis, convulsions, tachycardia, dyspnea, the skin irritation, and if ingested, irritation to the gastrointestinal, nausea, vomiting and diarrheas.

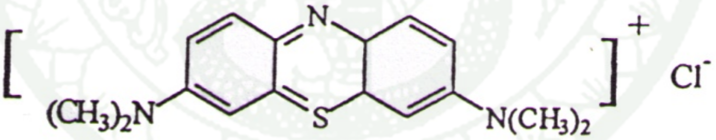

Figure 4 The chemical structure of Methylene blue.

The methylene blue adsorption gives an indication of the adsorption capacity for large molecules having similar dimensions to methylene blue. The methylene blue adsorption value is defines as the number of milliliters standard methylene blue solution decolorized by 0.1 g of activated carbon.

2.4.3 BET surface area

Preference internal surface area normally is measured by the method developed by Brunauer, Emmet and Teller. The adsorption is carried out by nitrogen at -196 C°. The average BET surface area for high quality activated carbon is about 1000 m^2/g.

2.5 Utilization of activated carbon (Srikun, 2007)

In industry, activated carbons are used in many applications which are described as follows:

2.5.1 Application for gas or vapor adsorption

- As adsorbent in toxic preventing mask
- To separate gasoline from natural gas
- To remove impurities such as hydrogen, nitrogen, helium, acethylene, ammonia, carbon dioxide, carbon monoxide
- To remove ordor from air in air conditioner
- Used to adsorb radioactivity relseased from nuclear reaction

2.5.2 Application for gas or vapor adsorption (Decolorization and liquid purification)

- Used in sugar and food production industries
- Used in chemical and medical industries
- To purify water
- To separate metal from solution

3. Adsorption

3.1 Adsorption on activated carbon (Marsh and Reinoso, 2006)

The adsorption on activated carbon is the way to separate required substances from liquid or gas solution using activated carbon as an adsorbent. Adsorption capacity depends on structure and pore size of adsorbent, chemical properties of adsorbate and adsorbent and a number of adsorbate layers on adsorbent surface. Adsorption mechanism can be divided into 2 types which are physical and chemical adsorptions.

3.1.1 Physical adsorption

Physical activation occurs from the interaction between adsorbent and adsorbate molecules and it is stronger than molecule interaction. Liquid or condensed gas can be adsorbed at site easily if adsorbent has high porosity, even though the vapor pressure of the system is lower than solution. Desorption occurs when the pressure of the system decreases or the temperature increases. The assumptions of physical activation are given as follows:

- Higher adsorption efficiency at low temperature
- The adsorption using van der Waals force
- Low adsorption energy($\Delta H < 20$ kJ mo1^{-1})
- Multilayer adsorption
- Reversible adsorption

3.1.2 Chemical adsorption

Similar to the physical activation, the chemical adsorption occurs from chemical reaction between adsorbent and adsorbate. This result is to decrease ofadsorbate and the large amount of heat is released from the recation. However, the adsorption capacity obtained is quite small due to the reaction occuring at some part of the adsorbent surface area. Assumptions of chemical activation are:

- The higher adsorption efficiency at greater temperature
- The adsorption using chemical reaction between adsorbate and adsorbent
- High adsorption energy (50 kJ mo1^{-1}< ΔH < 800 kJ mo1^{-1})
- Monolayer adsorption
- Irreversible adsorption

3.2 Adsorption isotherm (Marsh and Reinoso, 2006)

The isotherm shapes give information on the extent of adsorption and porous networks which occur in adsorption process. Adsorption isotherms are classified by the International Union of Pure and Applied Chemistry (IUPAC) and consist of six shapes which are represented in Figure 5.

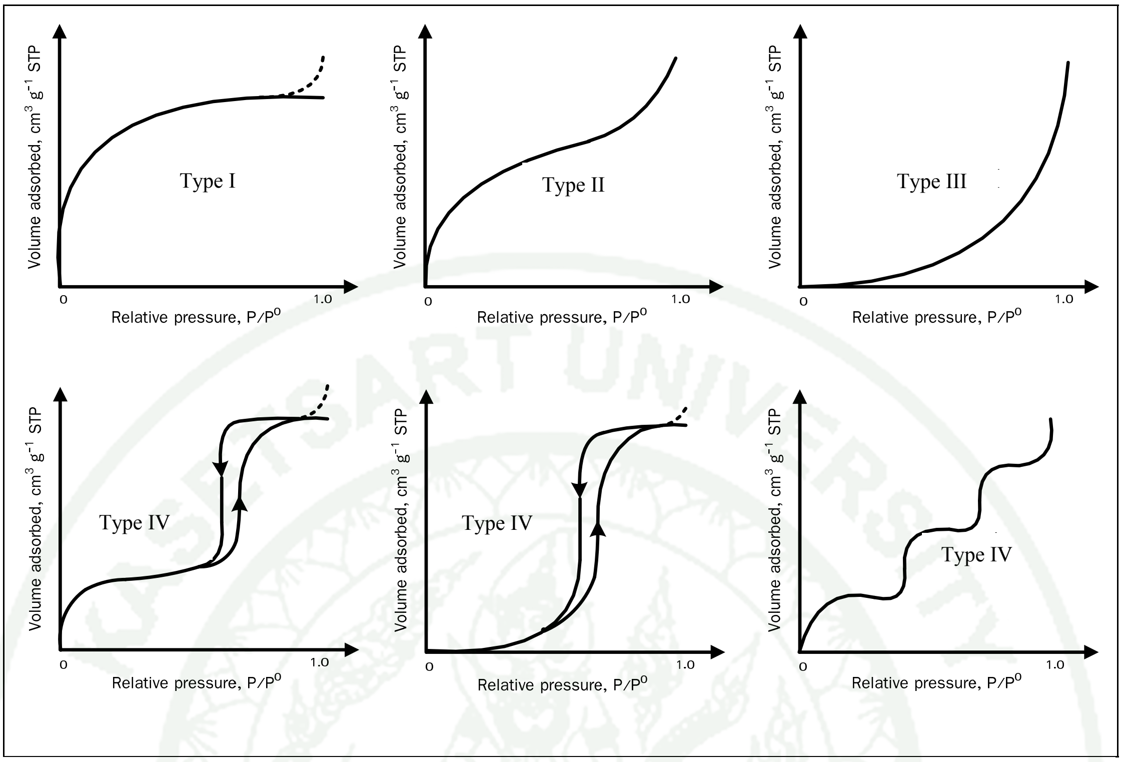

Figure 5 The IUPAC classification of adsorption isotherm shapes (Hirunpraditkoon, 2012).

3.2.1 Type I: Isotherm is called Langmiur or L-shaped. The isotherm reaches amaximum value of adsorption inflection. It characterizes the carbons which contain microporosity only. The gradient of initial part of the isotherm, from P/P_0 values from zero to about 0.05 indicates the dimension of microporosity. Example of this type is the adsorption of nitrogen on microporous activated carbon at temperature of -196 °C.

3.2.2 Type II: Isotherm shows an infection in the region of $P/P_0 > 0.1$, and at high relative pressure, $P/P_0 > 0.9$, where extents of adsorption increase rapidly. This isotherm characterized the adsorption on open surfaces with multilayer formation occuring in the stages of the process. This isotherm describes in mixed situations of micropores and open surfaces.

3.2.3 Type III: Isotherm is convex characterized adsorption at sites of low adsorption potential as in the case of adsorption on surfaces of organic polymeric systems.

3.2.4 Type IV: Isotherm is similar to type II isotherm, however, the adsorption takes place in mesoporosity. Moreover, isotherms may show hysteresis when the mechanism of filling by condensation in mesopores differs from that of empty mesopores. This isotherm is usual for activated carbon not to exhibit a plateau in the high relative pressure region.

3.2.5 Type V: Isotherm isa low energy and homogenous solid surface processing mesoporosity.

3.2.6 Type VI: Isotherm of the surfaces contains extremely homogeneous structure such as pyrolytic graphite which is used for argon and methane adsorptions.

Although these isotherm equations are used to describe adsorption equilibrium, two isotherms, Freundlich and Langmiur isotherms, are widely used for most adsorption processes especially in liquid-solid system.

a) Freundlich adsorption isotherm

It is most favorable, especially adsorption with diluted solution due to high accuracy. The equation is shown as follow:

$$q_e = K_F C_e^{1/n} \tag{5}$$

Where q_e is adsorption capacity at equilibrium (mg/gormmol/g).

C_e is concentrations of adsorbatein solution at equilibrium (mg/L or mmol/L).

K_F is a constant relating to the adsorption capacity (L/g).

n is a constant relating to the adsorption energy.

This equation can be derived to logarithmic equation, therefore:

$$\log(q_e) = \log(K_F) + \frac{1}{n}\log(C_e) \tag{6}$$

Plotting graph between $\log(C_e)$ and $\log(q_e)$, slope of linear equation and y-intercept are $1/n$ and $\log(K_F)$, respectively, which can be seen in Figure 6.

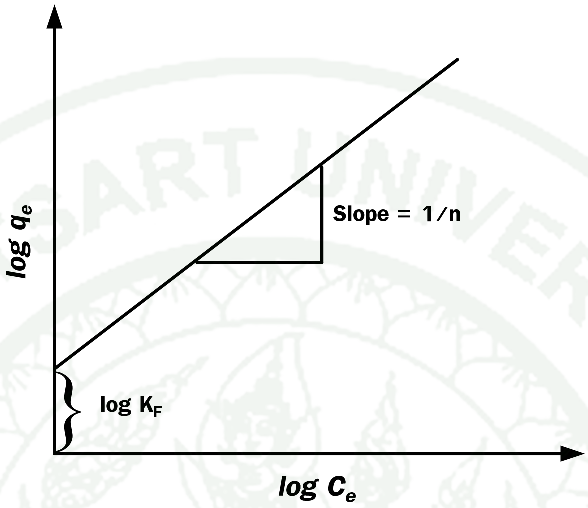

Figure 6 Freundlich adsorption isotherm (Hirunpraditkoon, 2012).

From the Freundlich adsorption isotherm model, the adsorption capacity is considered from slope between $\log(C_e)$ and $\log(q_e)$. Larger slope indicates greater adsorption at high concentration of solution. In general, activated carbon obtaining high slope has great adsorption efficiency when operates in carbon contacting system while those small slope should be operated as batch treatment.

b) Langmuir adsorption isotherm

Basic assumptions of Langmuir adsorption isotherm model are identified as following:

- Adsorption cannot proceed beyond monolayer coverage
- All surface sites are equivalent and can accommodate, at most, one adsorbed atom
- The ability of a molecule to adsorb at a given site is independent of the occupation of neighboring sites

The equation is represented as following:

$$\frac{1}{q_e} = \frac{1}{q_m} + \left(\frac{1}{K_L q_m}\right)\frac{1}{C_e} \qquad (7)$$

or

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{K_L q_m} \qquad (8)$$

Plottingrelation between C_e and C_e/q_e, slope of linear equation and y-intercept are $1/q_m$ and $1/K_L q_m$ which is shown in Figure 7.

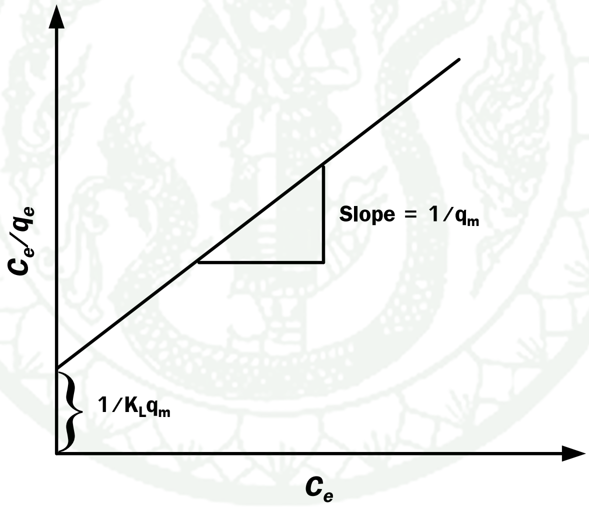

Figure 7 Langmuir adsorption isotherm (Hirunpraditkoon, 2012).

3.3 Adsorption kinetics

The adsorption process takes place in four steps which are bulk solution transport, film diffusion transport, pore and surface transport and adsorption. The process is shown in Figure 8.

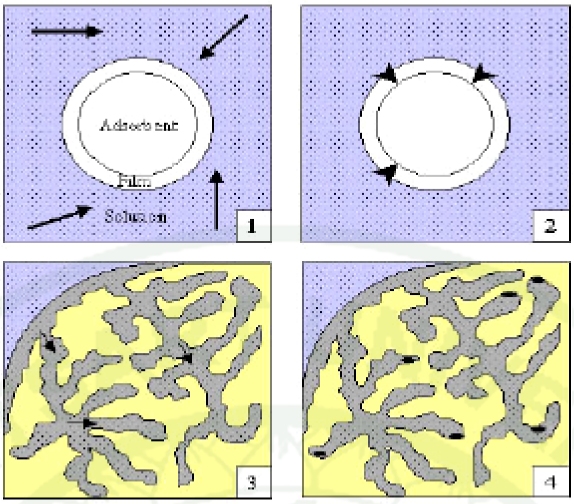

Figure 8 The adsorption process.

3.3.1 Bulk solution transportrefers to diffusion and mixing in fluid occupying the spaces between the adsorbents.Generally, it is a fast step.

3.3.2 Film Diffusion is the transport of the adsorbate across the liquid interface by molecular diffusion. Diffusion time depends on either solution flow rate or mixing rate which the diffusion time decreses with increasing solution flow rate or mixing rate.

3.3.3 Pore and surface transports refer to the diffusion of adsorbates within absorbent by either pore or surface diffusion.

3.3.4 The adsorption step involves the attachment of the material to be adsorbed to adsorbent at an available adsorption site. The adsorption rate is fast for physisorption while it is slow for chemisorption. The adsorption may be the rate limiting step if it is chemisorption.

In this research, pseudo-first orderandpseudo-second orderrate equations which describe adsorption step and intraparticle diffusion equation describing pore and surface transports are used to illustrate the adsorption kinetics. The equations are shown as follows:

a) The psudo-first order rate equation is generally expressed as follows:

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \tag{9}$$

where q_e and q_t are the adsorption capacity at equilibrium and at time *t*, respectively (mg/g).

k_1 is the rate constant of pseudo first-order adsorption (l/min).

b) The psudo-second order rate equation is generally expressed as follow:

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \tag{10}$$

Where k_2 is the rate constant of pseudo first-order adsorption (g/mg min).

For the boundary conditions $t = 0$ to $t = t$ and $q_t = 0$ to $q_t = q_t$, the integrated form of Eq. (10) becomes:

$$\frac{1}{(q_e - q_t)} = \frac{1}{q_e} + kt \tag{11}$$

which is the integrated rate law for a pseudo-second order reaction. Equation (11) can be rearranged to obtain Eq. (12), which has a linear form:

$$\left(\frac{t}{q_t}\right) = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}(t) \tag{12}$$

The plot of $\left(\frac{t}{q_t}\right)$ and *t* of Eq. (12) should give a linear relationship form which q_e and k_2 can be determined from the slope and intercept of the plot, respectively.

c) The intraparticle diffusion model is expressed as (Weber and Morris, 1963; Srivastava et al., 1989)

$$q_t = k_{id}\left(t\right)^{0.5} \tag{13}$$

where k_{id} is the intraparticle diffusion rate constant (mg/g $h^{0.5}$), which is obtained from the slope of the straightline of q_t vs. $t^{0.5}$.

4. Literature review

Tsai *et al*. (2001) prepared of activated carbons from corn cob catalyzed by potassium salts, later on gasification with $CO_2$was used. At carbonization temperature of 800°C, they found that 10.5 wt% KOH and 37.5 wt% K_2CO_3 were most effective which BET surface area were 1682 and 1266 m^2/g respectively. Additionally, they investigated the effect of washing treatment. The results also showed that the porosity created in acid-unwashed carbon product was lower than that of acid-washed carbon product.

Lua and Guo (2001) studied the preparation and characterization of activated carbons from oil-palm stones for gas-phase adsorption. The results showed that activation temperature and holding time played an important role on the surface area and pore size distribution of the activated carbon. The optimum conditions for CO_2 activation were found to be at an activation temperature of 900°C and a holding time of 30 min, which gave the largest BET surface area for the oil-palm stone-activated carbon of 1366 m^2 g^{-1}.

Carvolho *et al*. (2003) investigated activated carbon derived from cork waste by chemical activation with KOH. The effects of activating agent content and temperature and activation time on textural characteristics were studied. The results of N_2 adsorption isotherms found microporous materials were obtained for all impregnation ratios. BET surface area showed the increase of the KOH:cork weight ratio until it is equal to 1:1 rose surface area of sample, however for high content of KOH, the surface area became progressively smaller. For calcinations temperature and time, the highest BET surface area was obtained from 900°C and 2 h respectively.

Lua and Guo (2005) studied the effect of activation temperature on the textural and chemical properties of activated carbon prepared from pistachio-nut shell by potassium hydroxide activation. The results found that high temperature was required to develop a highly porous structure, where maximum BET surface area was 1946 m^2/g. However, too high activation temperature will result in the burn-off of some carbon structures and the widening of micropores to meso- and macropores.

Hameed *et al*. (2007) investigated activated carbon synthesized from rattan sawdust by physic-chemical activation with KOH and CO_2. Langmuir and Freundlich models were used to study the adsorption isotherm. The result showed that the equilibrium data fitted with Langmuir adsorption modelof adsorption. The adsorption kinetics followed with thepseudo-second-order model.

Tan *et al*. (2008) illustrated the adsorption of methylene blue on activated carbon prepared from oil palm shell by physic-chemical activation with KOH and CO_2. Adsorption isotherms were investigated by comparing Langhmuir, Freundlich, Temkin and Dubinin-Radushkevich. The result showed that it was best fitted with the Langmuir isotherm model. Kinetics studies found that the adsorption capacity increased with increasing the initial dye concentrations, while the adsorption equilibrium decreased with the increase in temperature, indicating that the exothermic nature of the adsorption reaction. Adsorption kinetics was studied by psudo-first order and psudo-second order rate equations and intraparticle diffusion equation. The adsorption kinetics followed closely to thepseudo-second-order model.

Chandra *et al.* (2009) characterized the activated carbon prepared from durian shell by chemical activation with potassium hydroxide. The effects of impregnation and activation temperature were studied. Adsorption isotherm showed that activated carbons obtained at low chemical impregnation ratio of 0.25 were microporous, while mesoporous became predominantly at higher impregnation ratio. Moreover, the KOH to durian shell ratio of 0.5 and activation temperature of 773 K was found as the optimum condition to synthesize high surface area activated carbon.

MATERIALS AND METHODS

Materials

1. Chemicals

1.1 Potassium hydroxide, AR grade, Merck
1.2 37% Hydrochloric acid, Fisher chemicals
1.3 Methylene blue, AR grade, Carol Erba
1.4 99.995% Nitrogen
1.5 99.9% Carbon dioxide
1.6 Sodium thiosulfate, AR grade, QRëC
1.7 Iodine, AR grade, Ajax finechem
1.8 Potassium iodide, AR grade, J. T. Baker
1.9 Sodium carbonate, AR grade, QRëC
1.10 Strach, AR grade, QRëC

2. Equipments

2.1 Furnace, model PF 2, Vecstar
2.2 Stainless steel retort
2.3 Analytical balance, model PG 503-s, Metler Toledo
2.4 Desiccator
2.5 Oven, model UL 40, Memmert
2.6 Sieve shaker, model GmbH, PT
2.7 Water bath and shaker, model Memmert
2.8 Moltar
2.9 UV/Vis Spectrophotometer, model UV/Vis 500,Unicam anltech
2.10 Centrifuge, EBA III, Hettich

Methods

1. Activated carbon preparation

1.1 Precursor

The jatropha seed coat received from National Corn and Sorghum was washed for several times with water and dried overnight at the temperature of 105 °C to remove residual moisture. Chemical composition, proximate and ultimate analyses of raw material were determined.

1.2 Synthesized activated carbon

To study the effect of activation method and temperature on the activated carbon characterization, an activation time was fixed at 1 h.

1.2.1 Physical activation

The washed jatropha seed coat was pyrolyzed in the furnace with a heating rate of 10°C/min at N_2 flow rate of 200 ml/min. The carbonization temperatures were varied at the temperature of 600, 700, 800 and 900 °C. Subsequently, 200 ml/min of CO_2 gas was fed for activation for 1 h. Then the activated carbon product was cooled down to the room temperature under N_2 atmosphere.

1.2.2 Chemical activation

The washed jatropha seed coat was firstly soaked with 25 wt% KOH in a 1000 ml beaker at a ratio of 1:1 precursor to KOH. The mixture was hold in the temperature of 105 °C for 8 h in an oven for complete reaction. The impregnated seed coat was then dried at 110 °C overnight. The resulting sample was placed in the furnace. The carbonization was carried out at heating rate of 10 °C/min under N_2 gas flow at 200 ml/min at the temperature of 600, 700, 800 and 900 °C. The final temperature was held for 1 h for the activation process. Finally, the product was cooled down to the room temperature under N_2 atmosphere.

1.2.3 Physico-chemical activation

The washed precursor was soaked with 25 wt% KOH at the impregnation ratio of 1 for about 8 h at 105 °C. The sample was dried at 110 °C for 12 h. The resulting sample was then placed in the furnace and the carbonization was initiated by heating the sample at a heating rate of 10 °C/min under N_2 gas flow of 200 ml/min from the room temperature to the final temperature (600 °C, 700 °C, 800 °C and 900 °C). When the desired temperature was reached, CO_2 gas flow of 200 ml/min was subsequently fed for 1 h for activation. Once the activation was completed, the activation gas was switched to N_2 gas to cool down the activated carbon sample.

The derived activated carbon was then washed by 0.1 M HCl and hot distilled water until the filtrate became neutral. Then, the product was air dried at 110 °C overnight. The final carbon product was grounded and sieved before characterization.

To investigate the influence of holding time, only experimental condition which resulted the optimized physical and chemical properties was done. The activation time was varied at 1, 2 and 3 h, respectively. Overall research methodology is shown in Figure 9.

2. Characterization

2.1 Physical and chemical properties

2.1.1 Proximate and ultimate analyses

The content of C, H and N elements by the ultimate analysis was determined using CHNO analyzer, Perkin Elmer PE2400 SeriesII. The proximate analysis (moisture, volatile, fixed carbon and ash contents) was determined in accordance with the ASTM E1131 using simultaneous DSC-TGA analyzer, Perkin Elmer SDT 2960. Details of these methods are shown in see Appendix A.

2.1.2 Yield of activated carbon

Yield of activated carbon is calculated on a chemical-free basis.

$$Yield = \frac{W_1}{W_0} \tag{14}$$

Where W_0 is the original mass of raw jatropha on dry basis (g)

W_1 is mass of the carbon after activation, washing and drying (g)

2.1.3 Iodine number

Iodine number was determined accordance with ASTM D 4607-94: see in appendix B

2.1.4 Specific surface area and pore volume

Textural characterization of activated carbon was carried out by nitrogen adsorption at -196 °C using Autosorp-1, Quantachrome. The Brunauer-

Emmett-Teller (BET) surface area was calculated from the isotherms using BET equation. The total pore volume was estimated to be the liquid volume of adsorbate (N_2) at the relative pressure of 0.988. The cross-sectional area of a nitrogen molecule assumed to be 0.162 nm^2. The Dubinin-Radushkevich (DR) equation was used to calculate the micropore volume. The pore size distribution was determined using Barrett, Joyner and Halenda (BJH) model. The average pore size was calculated as 4 times the total pore volume divided by the BET surface area.

2.1.5 Methylene blue adsorption

Methylene blue adsorption was investigated accordance with JIS K 1470-1975. The methodology can be seen in appendix C.

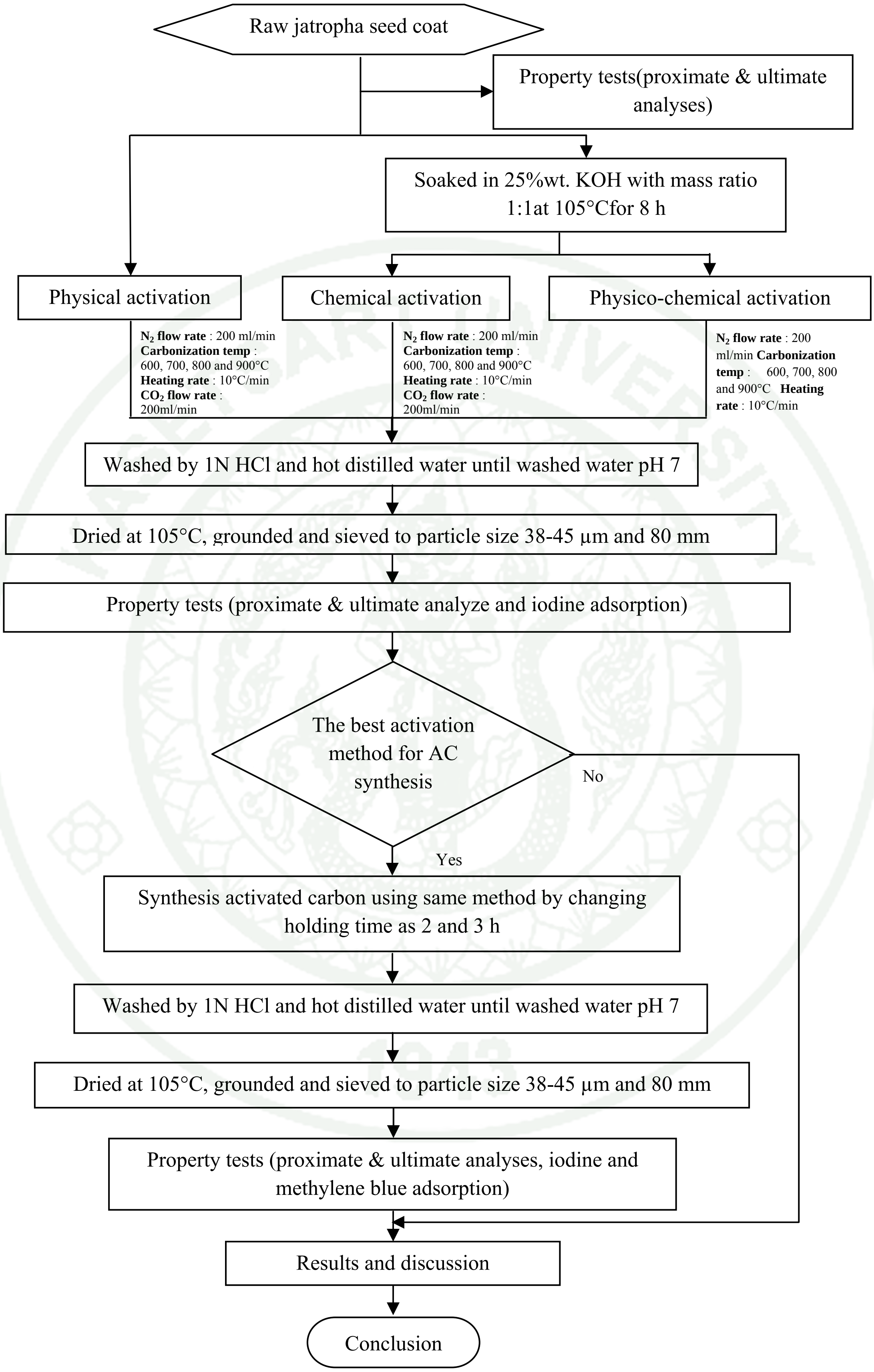

Figure 9 Methodology flowchart.

RESULTS AND DISCUSSION

1. Properties of jatropha seed coat

The proximate and ultimate analyses of the jatropha seed coat used in this study are given in Table 3. The results show that jatropha seed coat contains high carbon and low ash contents. Table 4 shows the comparison of proximate analysis of jatropha seed coat with the other raw material used for the study of activated carbon. It can be seen that its properties are similar to that of coconut shell which is generally used as the main source for the production of activated carbon in the industry (Sudaryanto*et al*, 2006). This indicates that jatropha seed coat can be used as a precursor for the production of activated carbon.

Table 3 Properties of the jatropha seed coat.

Properties	**Jatropha seed coat**
Chemical composition (%)	
Lignin	33.80
Cellulose	39.40
Hemicellulose	10.50
Proximate analysis (%)	
Moisture content	12.07
Volatile matter	76.16 [a]
Ash	3.85 [a]
Fixed carbon	19.99 [a]

Table 3 (Continued).

Properties	**Jatropha seed coat**
Ultimate analysis (%)	
Carbon	50.90
Hydrogen	5.80
Nitrogen	0.80
Oxygen, sulfer and others	42.50

[a]Dry basis

[b] by difference

Table 4 Proximate analysis of various raw materials.

Precursor	**Proximate analysis (%)**		
	Volatile	**Fixed carbon**	**Ash**
Cassava peel (Sudaryanto*et al.*, 2006)	59.40	28.90	0.30
Cherry stone (Marín *et al.*, 2006)	79.97	19.59	0.44
Coconut shell (Guo*et al.*, 2009)	78.30	20.96	0.74
Macademia nutshell (Ahmadpour and Do, 1997)	74.49	24.49	1.02

Table 4 (Continued).

Precursor	Proximate analysis (%)		
	Volatile	**Fixed carbon**	**Ash**
Pitsachio shell (Okutucua*et al.*, 2011)	86.80	13.10	0.10
Sunflower oil cake (Karag□z*et al.*, 2008)	74.71	19.54	5.75
Jatropha seed coat (This study)	76.16	19.99	3.85

2. Effect of activation temperature on carbon yield

The results of yields of activated carbons prepared at different temperatures are shown in Figure 10. The results show that the carbon yields slightly decrease with the increasing in the activation temperature for all activation methods. It is expected that an increasing in the temperature increases the amount of volatiles released.It can be seen that yields of activated carbon from chemical and physico-chemical activations are not different while yield from the physical activation is higher than those both methods. This is because KOH used for chemical and physico-chemical activations can promote the devolatilization during pyrolysis process. Moreover, the physico-chemical activation gives lower carbon yield than KOH activation alone.It is because of more chemical reaction occurred and higher carbon consumption in the physico-chemical activation (Sabio*et al.*, 1995).

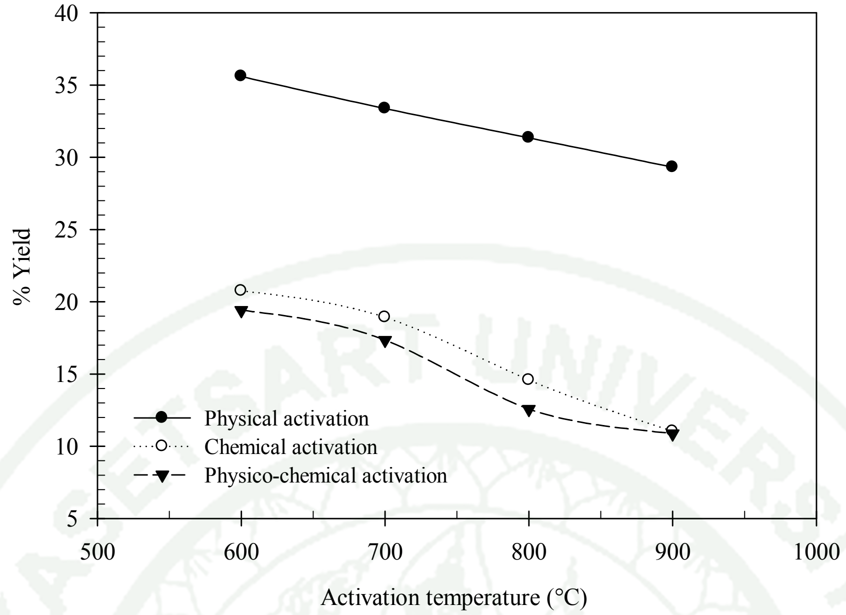

Figure 10 Effect of the activation temperature on the yield of activated carbon.

3. Effect of activation temperature on the iodine number

Iodine adsorption is a basic methodused to study the micropore content of the activated carbon. Results of the iodine adsorption of activated carbon derived from jatropha seed coat are illustrated in Figure 11. It is evident that increasing in activation temperature from 600 to 900 °C continuously increases in the iodine adsorption. This indicates that in the internal microporous cavities at higher temperature are developed (Tongpoothong *et al.*, 2011). The maximum iodine adsorption obtained at the activation temperature of 900 °C for physical, chemical and physico-chemical activations are 287.79, 1304.31 and 1469.40 mg/g, respectively. However, it can be observed that the iodine number of activated carbon prepared by chemical and physico-chemical activations is much higher than those of activated carbon from physical activation. This is because of the effect of the gasification process with KOH that can promote more new pores. Therefore, the greater iodine adsorption in this temperature range is occurred.

Since the results of physical activation could not developgood porous structure in the temperature range used, therefore only chemical and physico-chemical activations were selected to investigate the influence of other effects on the pore structure.

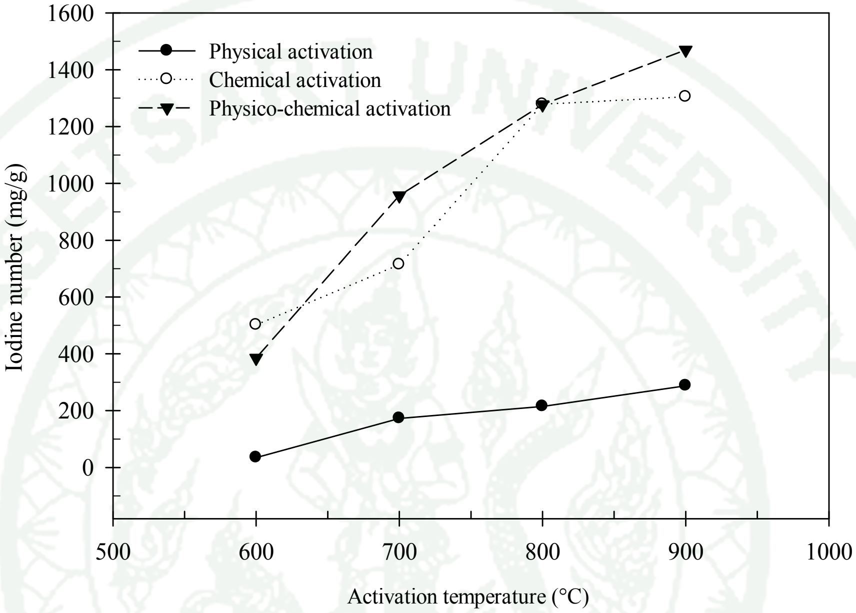

Figure 11 Effect of activation temperature on the iodine number.

4. Effect of the activation temperature on the pore structure of activated carbon

The effects of activation temperature on the pore and surface of activated carbon synthesized by chemical and physico-chemical activation methods are shown in Figures 12 to 15. Upon the increasing of the temperature from 600 to 900 °C, predominantly micropores but some mesopores and macropores are also formed at higher temperature, as a result of higher BET surface area and micropore volume for both chemical and physico-chemical activations. The results are shown in Figure 12 and 13.

For the chemical activation, the increase in the final temperature from 600 to 700 °C developed the BET surface area and micropore volume while non-micropore volume remained constant. At the activation temperature of 800 °C, there was a progressive increase in the BET surface, micropore volume whereas non-micropore volume decreased. The reduction in the non-micropore volume was due to an enhancement C-KOH reaction, resulting in the burning-off of some wall. On the other hand, creating of new micropores gave more effect than those of the non-micropore volume reduction, therefore the increase in the micropore volume percentage. For the maximum temperature used at 900 °C, the increases in the BET surface area, micropore volume and non-micropore volume were observed. These werebecause of the excessive burning of some pore walls from C-KOH reaction combined with continuous devolatilization at high activation temperature, resulting in the widening of pores and even the loss some walls between the pores. This is similar to previous work by chemical activation of cassava peel with potassium hydroxide (Sudaryanto *et al*., 2006). However, creating the micropores is still more dominant than that of the effect of widening micropores to be mesopores and macropores. The maximum surface area of activated carbon obtained at activation temperature of 900 °C is 1400.23 m^2/g.

For the physico-chemical activation, increasing in the final temperature from 600 to 700 °C progressively increased in the BET surface area and micropore volume while there was a decrease in the non-micropore volume. This may be because of the more devolatilization and the effect of combined chemical reaction and CO_2 activation at higher temperature. Therefore, micropores formation was predominant. At activation temperature of 800 °C, there was a significant increase in the non-micropore volume, BET surface area and micropore volume. These increasing were due to the excessive carbon burn off resulting in the widening of some pores. Consequently, evolution of volatiles from the raw material was increased with the increasing of temperature. The pores widening has more influence over the formation of new pores which indicated the decrease in the micropore volume percentage. This is in consistency with the work of activated carbon from cherry stones by Marín *et al*.(2006). For the activation temperature up to 900 °C, the BET surface area and micropore volume continuously increased while the non-micropore volume become decreased. The decrease in non-micropore volume was caused by an enhanced C-

KOH combined with C-CO_2 reaction at high temperature, leading to the burning-off some walls and the wall became thinner. However, creating micropore was still predominant therefore the micropore volume percentage increased.

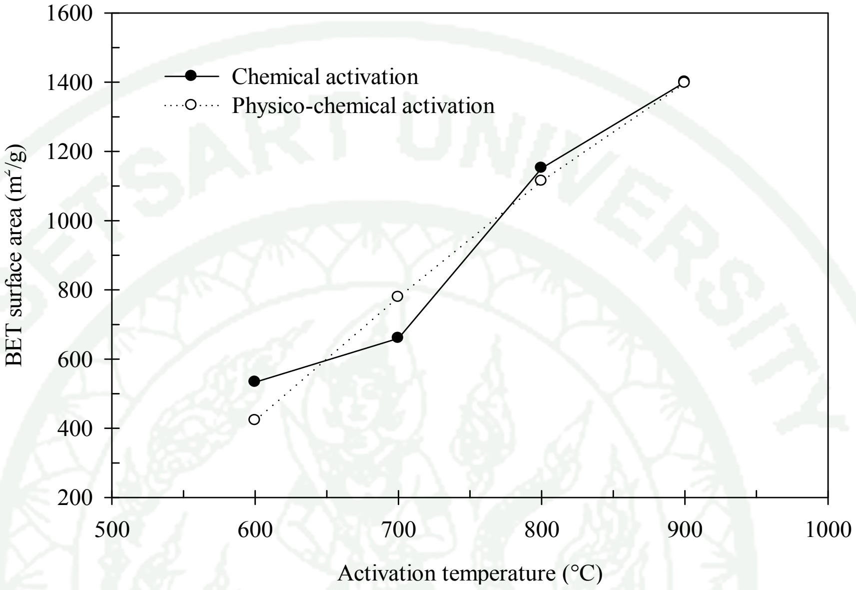

Figure 12 Effect of activation temperature on the BET surface area.

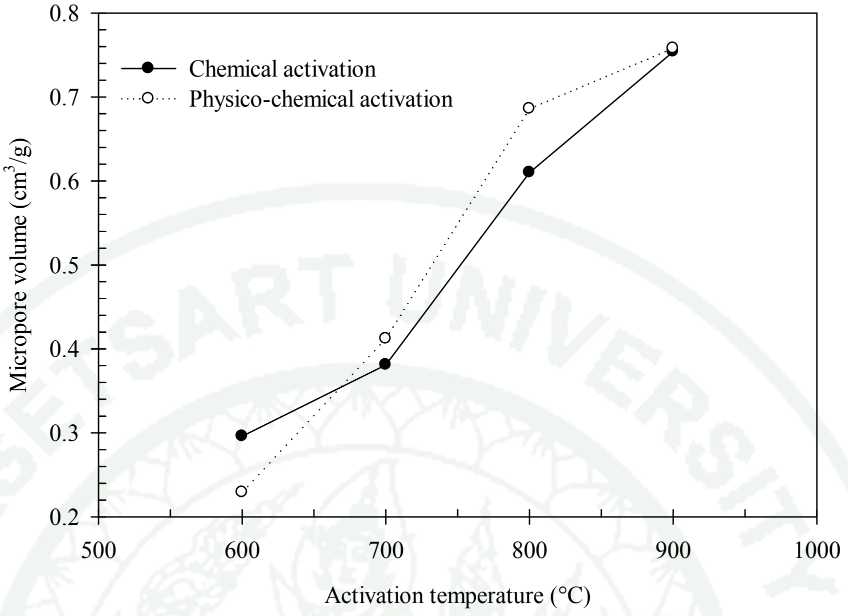

Figure 13 Effect of activation temperature on micropore volume.

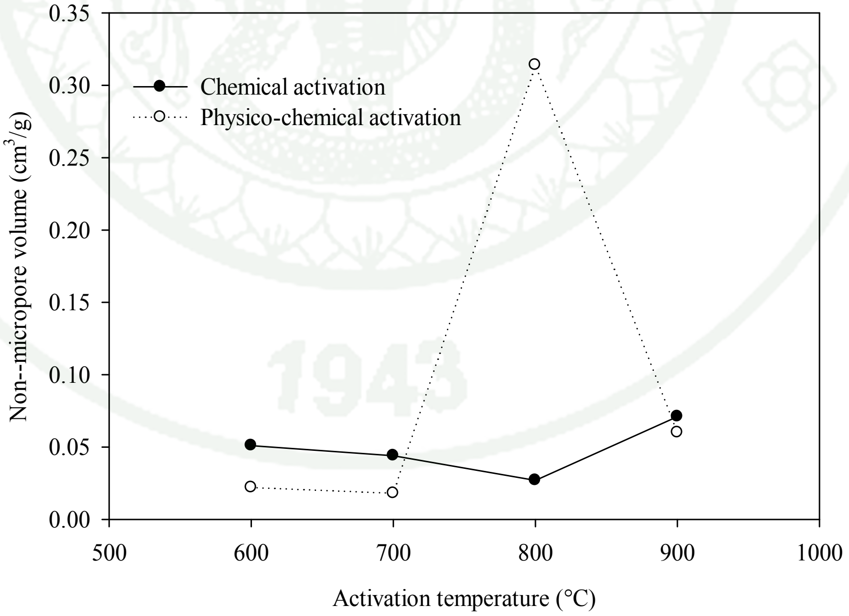

Figure 14 Effect of activation temperature on non-micropore volume.

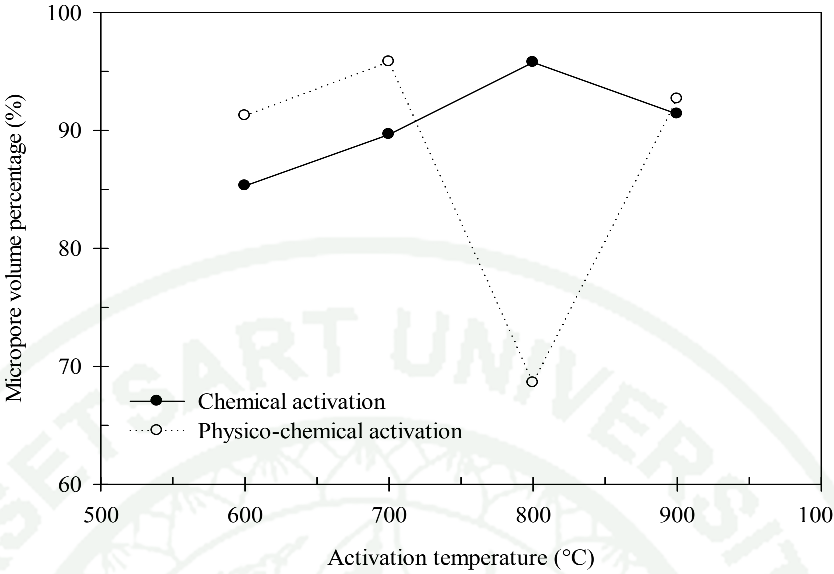

Figure 15 Effect of activation temperature on micropore volume percentage.

4. Proximate analysis

Proximate analysis of activated carbon prepared by chemical and physico-chemical activations with activation temperature is shown in Table 5. The results showed that the volatile contents gradually decreased whereas fixed carbon increased with the activation temperature increase. These phenomena can be observed by the predominance of the loss of volatiles over carbon consumption for the increase in the activation temperature (Yang and Lua, 2003).

Table 5 Proximate analysis of activated carbon at difference activation temperature.

	Activation temperature (°C)	Proximate analysis (%)		
		Volatile	Fixed carbon	Ash
Chemical activation	600	32.79	62.34	4.87
	700	28.59	65.90	5.51
	800	24.03	72.49	3.48
	900	22.58	73.19	4.23
Physico-chemical activation	600	30.56	67.43	2.01
	700	26.69	69.58	3.37
	800	23.73	76.00	0.27
	900	21.22	75.39	3.39

5. Effect of activation time on yield of activated carbon

This study only the activated carbon prepared by physico-chemical activation at the activation temperature of 800 and 900 °Cwere investigatedsince the results of average pore size and the BET surface area were maximum.Figure 16 illustrates the results of holding time and the yield of activated carbon derived from jatropha seed coat at activation temperature of 800 and 900 °C. The results show that the yields of all activated carbon derived from different activation temperature present the same trends. The yields continuously decrease with increasing the activation time from 1 to 3 h. This is expected as longer activation time leads to higher volatiles released from the sample and a longer C-CO_2 reaction by higher carbon burning-off (Yang*et al.*, 2010).

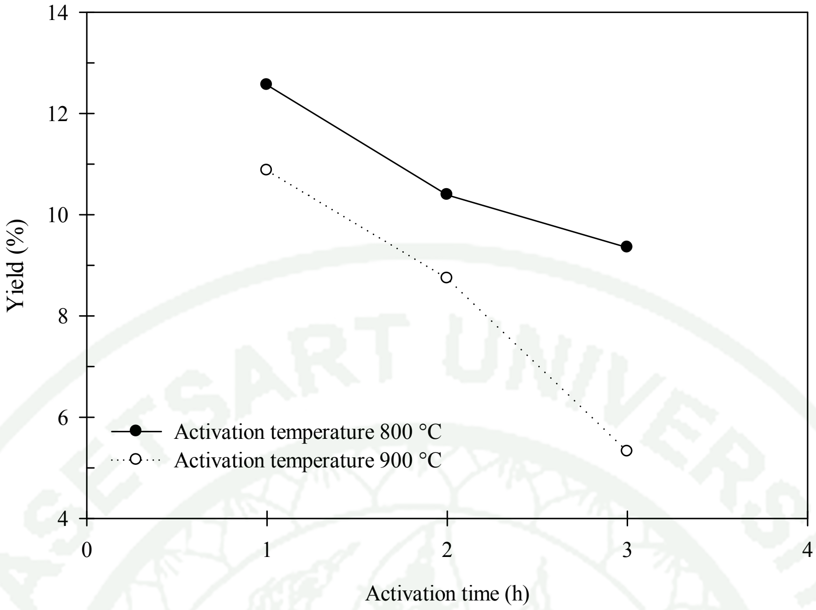

Figure 16 Effect of the activation time on the yield of activated carbon.

6. Effect of activation on the iodine number

The iodine adsorption of activated carbon prepared at activation temperature of 800 and 900 °C with various holding time is shown in Figure 17. It can be seen that a slight increase in the iodine number is observed when the activation time raised to 2 h. The maximum iodine adsorptions for activated carbon at activation temperature of 800 and 900 °C are 1407.79 and 1510.80 mg/g, respectively. This was due to the continuous devolatilization created new pores with time. However, extension in the activation time to 3 h caused a progressive decrease in the iodine number for both activation temperatures which indicated that excessive activation time caused excessive release of volatile matter due to too long time.

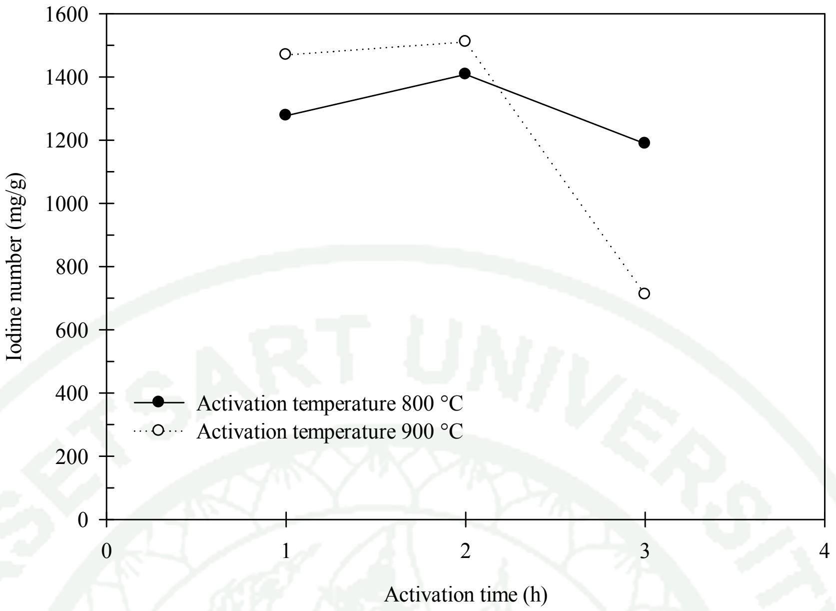

Figure 17 Effect of activation time on the iodine number.

7. Effect of activation time on pore structure of activated carbon

The pore development and surface area characteristics of activated carbon prepared by physico-chemical activation at the different activation time are shown from Figure 18 to 21.

For the activation temperature at 800 °C, the BET surface area and micropore volume increased while the non-micropore pore volume decreased with increasing the activation time from 1 h to 2 h. This was expected as longer holding time may cause higher volatiles released from the sample and a longer C-CO_2 reaction which resulting in a higher carbon burning- off. The progressive deceased in the BET surface area, the micopore volume and non-micropore volume were observed when the activation time reached up to 3 h.

For the activation temperature of 900 °C, increasing the holding time from 1 h to 2 h increased the BET surface area, the micopore volume and non-micropore volume. These properties increased with the continuous devolatilization coupled with the pore enhancement and the formation of new pores.This was because the combined C-KOH and C-CO_2 reactionswere enhanced, as a result the carbon was burning-off. The BET surface area obtained in this condition was 1479.06 m^2/g. However, the BET surface area, the micopore volume and non-micropore volume become decreased when the activation time increased to 3 h. The decrease was due to the widening of pores through the complete burning-off some walls between neighbor pores and the pore wall thinning effect from too long holding time period.

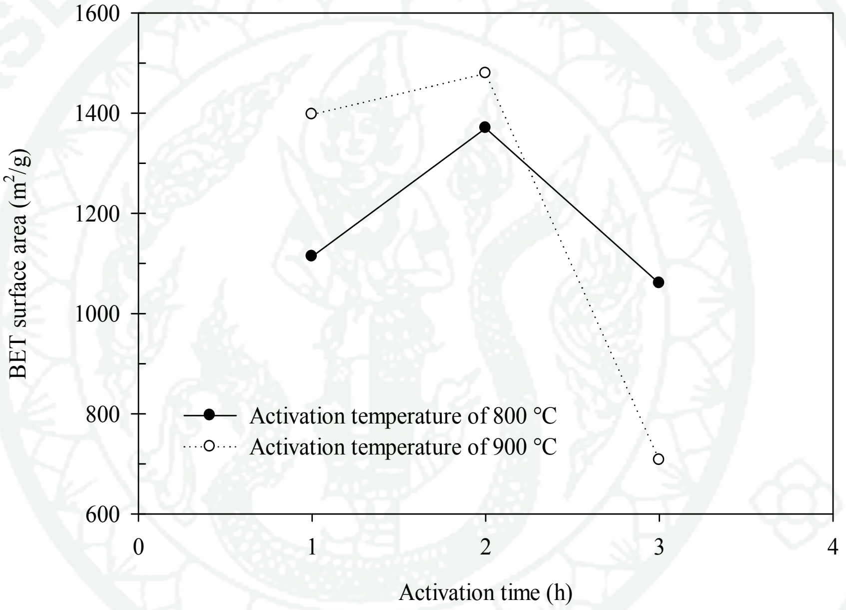

Figure 18 Effect of activation time on the BET surface area.

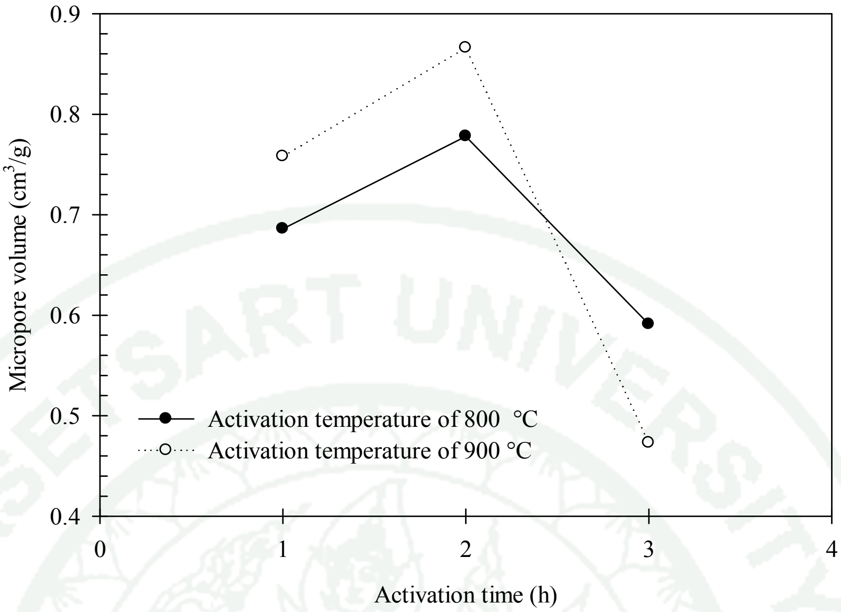

Figure 19 Effect of activation time on micorpore volume.

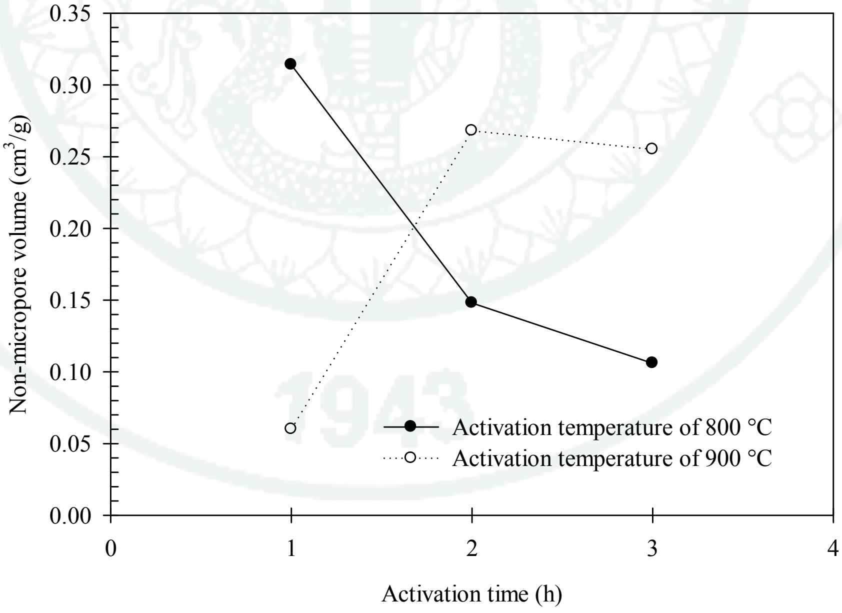

Figure 20 Effect of activation on the non-micropore volume.

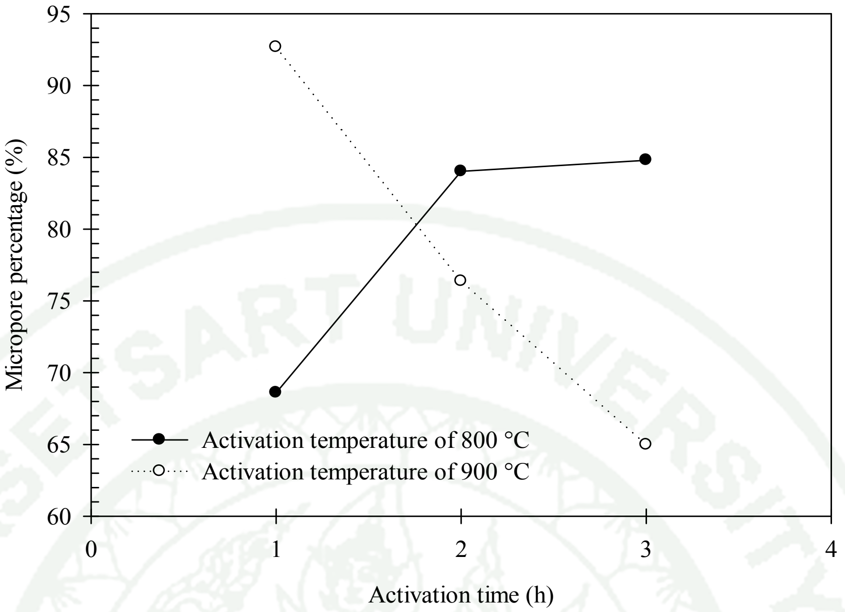

Figure 21 Effect of activation time on micropore percentage.

The proximate analysis results of the activated carbon at different activation time are given in Table 6. Similar trends are observed for both activation temperatures in which increasing holding time from 1 h to 2 h reduced volatile content but fixed carbon was increased. The release of volatile matter from the simple was greater than the carbon burning-off due to the C-CO_2 reaction. However, increasing the dwell time from 2 h to 3 h decreased fixed carbon content but increased the percentage of volatile content because the carbon burn off become more dominant.

Since the maximum BET surface area of the activated carbon at the activation temperature of 900 °C with the holding time of 2 h was observed, therefore the study of methylene blue adsorption was continued to investigate.

Table 6 Proximate analysis of activated carbon at difference activation time.

Activation temperature (°C)	Activation time (h)	Proximate analysis (%)		
		Volatile	Fixed carbon	Ash
800	1	23.73	76.00	0.27
	2	19.23	76.81	3.96
	3	20.23	75.24	3.83
900	1	21.22	75.39	3.39
	2	15.96	78.49	5.55
	3	19.13	75.25	5.52

9. Methylene blue adsorption

9.1 Effect of initial concentration dye on adsorption

The adsorption data for methylene blue adsorption withcontact time interval at different concentration is shown in Figure 22. The results showed that the amount of MB adsorbed per unit mass increased with the increasing in the MB concentration. This increase was due to increase in the driving force of the concentration gradient (Doğan *et al*., 2009).

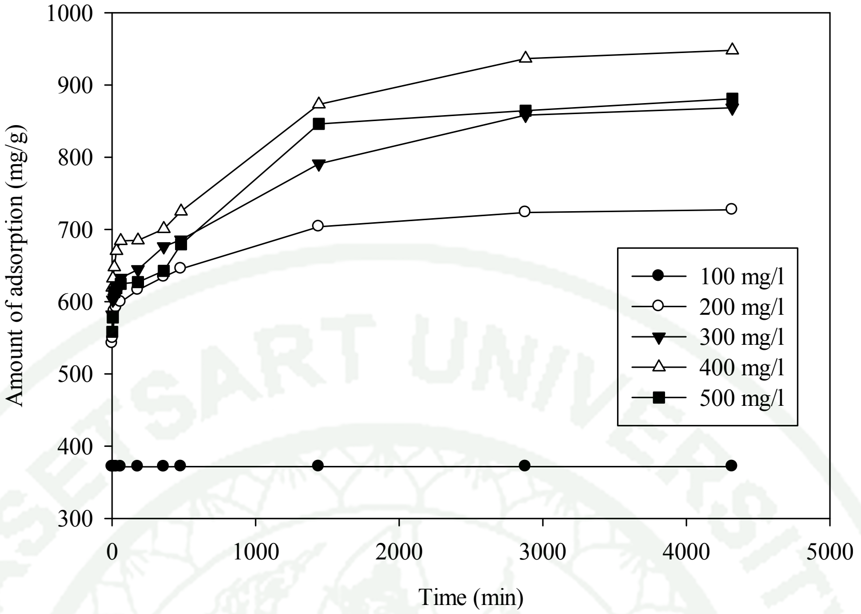

Figure 22 The variation of adsorption capacity with adsorption time at various initial concentrations.

3.2 Adsorption isotherm

The adsorption isotherm generally is used to indicate the adsorption molecules distribute between the liquid and solid phase when the adsorption process reaches equilibrium.In this work, only the Langmuir and Frundlich model are used to describe the relationship between the amount of dye adsorbed and its equilibrium concentration.

The Langmuir isotherm equation is represented by the following equation:

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{K_L q_m} \tag{15}$$

Where C_e is the equilibrium concentration of the methylene blue (mg/l).

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

q_m and K_L are Langmuir constants related to adsorption capacity and rate of adsorption, respectively.

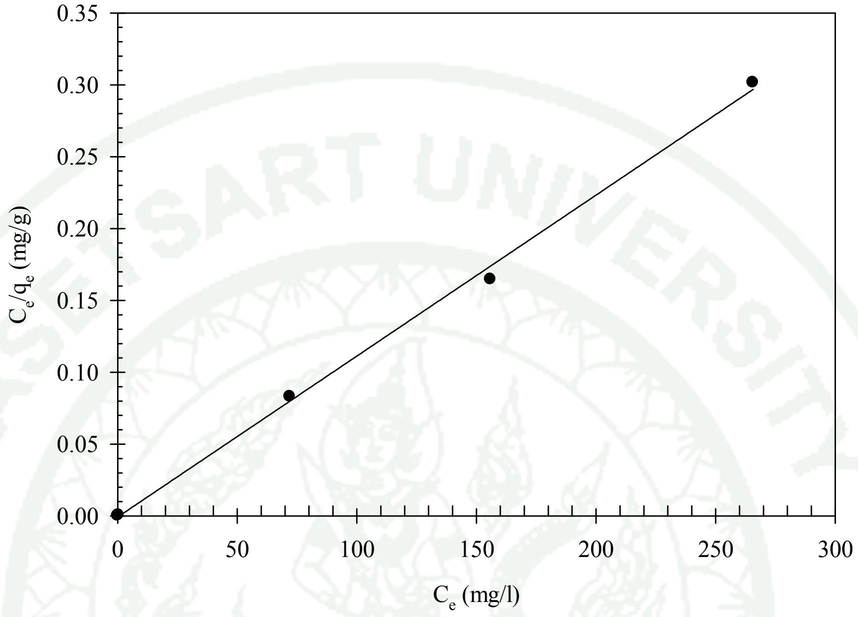

Figure 23 Langmuir adsorption isotherm of methylene blue activated carbon adsorption.

When Plottingrelation between C_e and C_e/q_e, slope of linear equation and y-intercept are $1/q_m$ and $1/K_L q_m$. The value of q_m and K_L are calculated and presented in Table 8.

The Freundlich isotherm equation is represented by the following equation:

$$\log(q_e) = \log(K_F) + \frac{1}{n}\log(C_e) \tag{16}$$

where q_e is adsorption capacity at equilibrium (mg/g).

C_e is concentrations of adsorbatein solution at equilibrium (mg/l).

K_F is a constant relating to the adsorption capacity (l/g).

n is a constant relating to the adsorption energy.

Plotting graph between $\log(C_e)$ and $\log(q_e)$, slope of linear equation and y-intercept are $1/n$ and $\log(K_F)$, respectively. The Freundlich adsorption isotherm of methylerne blue-activated carbon adsorption is shown in Figure 24 and the value of Freundlich constants are listed in Table 8.

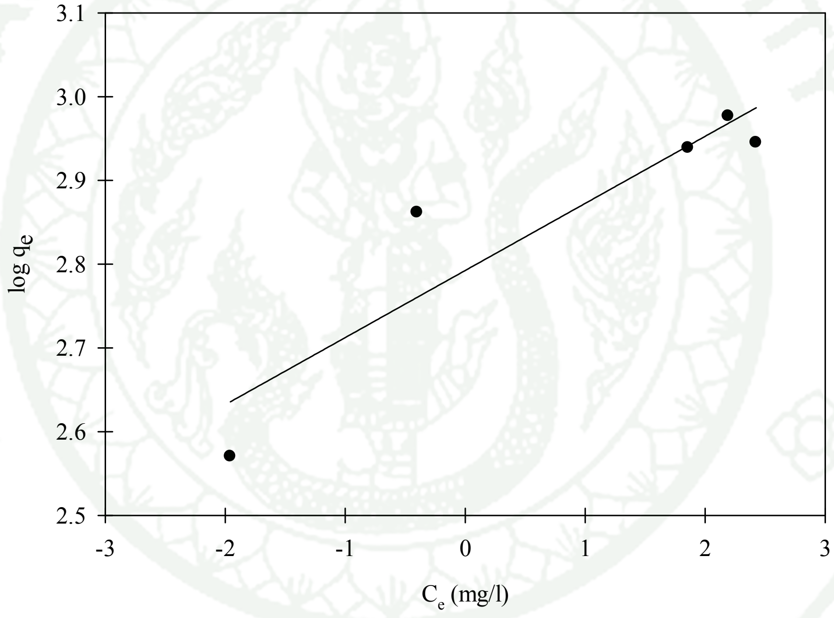

Figure 24 Freundlich adsorption isotherm of methylerne blue-activated carbon adsorption.

Table 7 Langmuir and Freundlich isotherm constants for MB dye on activated carbon at 30 °C.

Langmuir isotherm			Fruendlich isotherm		
q_m (mg/g)	K_L (l/mg)	R^2	n	K_F	R^2
909.09	0.73	0.998.	12.48	620.01	0.853

From Table 7, the Langmuir isotherm model fits quite well with the experimental data (correlation coefficient R^2= 0.998), whereas the low correlation coefficient (R^2= 0.853) shows poor agreement of Fruendlich isotherm with the experimental data. It indicates that homogeneous nature of dye molecule/jatropha seed coat carbon surface adsorption has equal adsorption activation energy. The results also show the formation of monolayer coverage of the dye molecule at outer surface of activated carbon.

3.3 Adsorption kinetics

Psudo-first-order reaction kinetics model is given as follows;

$$\frac{1}{(q_e - q_t)} = \frac{1}{q_e} + kt \tag{17}$$

where q_e and q_t are the adsorption capacity at equilibrium and at time *t*, respectively (mg/g)

k_1 is the rate constant of pseudo first-order adsorption (l/min)

The slope and intercept of the plot of $\log(q_e - q_t)$ versus t are used to determine the first order equation rate constant, k which can be seen in Figure 24.

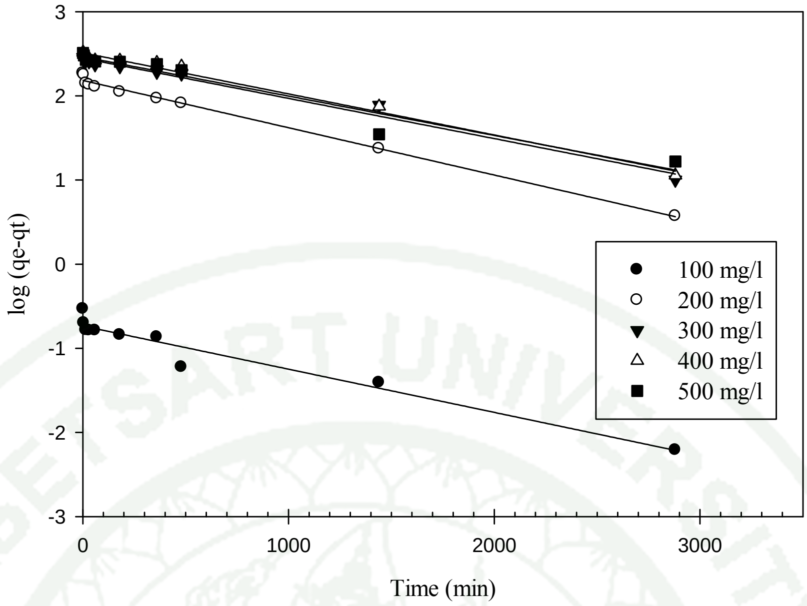

Figure 25 Psudo-first-order kinetics for the adsorption of MB dye on the activated carbon.

Psudo-second-order reaction kinetics model is given as follows;

$$\left(\frac{t}{q_t}\right) = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}(t) \qquad (18)$$

The plot of $\left(\frac{t}{q_t}\right)$ and t of Eq. (12) should give a linear relationship from which q_e and k_2 can be determined from the slope and intercept of the plot which can be seen in Figure 25.

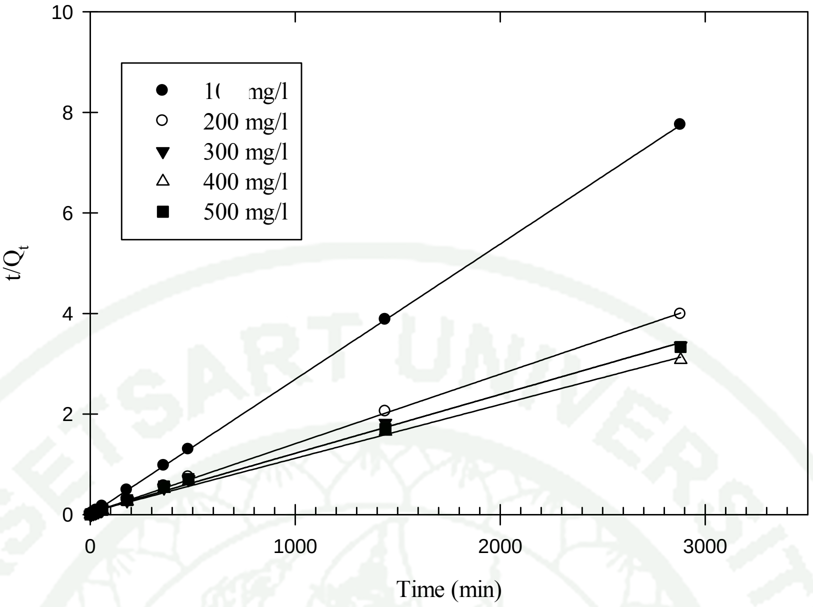

Figure 26 Psudo-second-order kinetics for the adsorption of MB dye on the activated carbon.

Table 8 lists the result of the rate constant studies for different initial dye concentrations by the psudo-first-order and psudo-second-order models. The correlation coefficient, R^2 for the psudo-second-order adsorption model has high value. This fact suggests that the psudo-second-order adsorption mechanism is predominant and that the overall rate of the dye adsorption process appear to be controlled by chemisorptions process.

Table 8 Comparison of the psudo-first-order and second-order adsorption rate constants and calculated and experimental q_e values for different initial dye concentration.

C_0 (mg/l)	First-order kinetics model			Second-order kinetics model		
	q_e (mg/g)	k_{s1} (l/min)	R^2	q_e (mg/g)	k_{s1} (l/min)	R^2
100	0.18	0.0012	0.948	370.37	0.0729	1
200	152.44	0.0014	0.993	714.29	6.9E-05	0.999
300	279.90	0.0012	0.985	833.33	3.36E-05	0.997
400	324.12	0.0012	0.988	909.09	2.44E-05	0.997
500	291.41	0.0012	0.951	909.09	2.41E-05	0.997

CONCLUSION AND RECOMMENDATION

Conclusion

The results of the preparation of activated carbon derived from jatropha seed coat can be concluded as follows:

1. Activated carbon synthesized by chemical and physico-chemical activation gave higher surface area while physical activation producedthe worst iodine adsorption property, indicating the worst surface area.

2. Increasing the activation temperature from 600 to 900 °C increased the BET surface area and micropore volume for both chemical and physico-chemical activations. The greatest surface area obtained at activation temperature of 900 °C for chemical and physico-chemical activations were 1400.23 m^2/g and 1397.11 m^2/g, respectively.

3. Increasing the activation time from 1 h to 2 h continuously rose the BET surface area and micropore volume. However**,** at 3 h which was too high activation time will result in the carbon burn off of carbon structure and the widening of micropores to meso- and macropores.

4. The methylene blue adsorption on jatropha seed coat activated carbon synthesized from by physico-chemical activation at the final temperature of 900 °C for 2 h can be described by monolayer Langmuir type isotherm. The maximum monolayer adsorption capacity was 909.09 mg/g. The kinetic studies showed that it followed pseudo-second-order model.

Recommendation

Further study in gas adsorption should be done as the guideline for commercial application in the future**.**

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APPENDICES

Appendix A

Standard test method for compositional analysis by thermogravimetry

Standard test method for compositional analysis by thermogravimetry Accordance with ASTM E1131-03

1. Apparatus

The essential equipment required to provide the minimum thermogravimetric analyzer capability for this method includes:

1.1 A thermobalance, composed of (a) a furnace to provide uniform controlled heating or a specimen to a constant temperature or at a constant rate within the 25 to 1000 °C temperature range of this test method; (b) a temperature sensor to provide an indication of the specimen/furnace temperature to ± 1 °C; (c) an electrobalance to continuously measure the specimen mass with minimum capacity of 30 mg and a sensitivity of ±1 µg; and (d) a means of sustaining the specimen/container under atmosphere control with a purge rate of 10 to 100 ± 5 ml/min.

1.2 A temperature controller, capable of executing a specific temperature program by operating the furnace between selected temperature limits at a rate of temperature change between 10 and 100 °C/min constant to within ±1% for a minimum of 100 minutes.

1.3 A recording device, either analog or digital, capable of recording and displaying the change in mass (TGA curve) with a Y-axis resolution of 5 µg and temperature or time with an X-axis resolution of 1 K or 0.1 min.

1.4 Containers (pans, crucibles, and so forth), which are inert to the specimen and which will remain dimensionally stable within the temperature limits of this method.

2. Procedure

2.1 Establish the inert (nitrogen) and reactive (air) gases at a flow rates of 100 mL/min.

2.2 Swich the purge gas to the inert (nitrogen) gas.

2.3 Zero the recorder and tare the balance. It is recommended that this be done in a range at least on recorder setting more sensitive than that to be used in the final weighing.

2.4 Open the apparatus to expose the specimen holder.

2.5 Carefully place 250 µm activated carbon in the specimen holder. Typically, a sample mass of 10 to 30 mg shall be used and enclose the specimen holder.

2.6 Record the initial mass. If the apparatus in use has provisions for direct measurements, adjust to read 100%.

2.7 Initial the heating program within the desired temperature range. See Table 1 for suggested heating rates and temperature ranges. Record the specimen mass change continuously over the temperature interval.

Table A1 Compositional analysis parameters.

Component	Start temperature (°C)	Rate (°C/min)	Final temperature (°C)	Hold (min)	Gas
Moisture	25	10	110 (X)	15	N_2
Volatile	110	10	950 (Y)	15	N_2
Fixed carbon	950	0	950	0	Air
Ash	950	0	950 (Z)	15	Air

2.8 Switch to the inert purge gas.

2.9 Calculate and report the sample composition

3. Calculation

3.1Moisture is represented by a mass loss measured between the starting temperature and Temperature X. Temperature X should be taken in the center of first mass loss plateau. The moisture content may be determined by the following equation:

$$V = \frac{W - R}{W} \times 100 \qquad \text{(A. 1-1)}$$

where: V = Moisture content, as received basis (%)

W = original specimen mass (mg)

R = mass measured at temperature X (mg)

3.2 Volatile matter content can be determined using the following equation:

$$O = \frac{R - S}{W} \times 100 \qquad \text{(A. 1-2)}$$

where: O = volatile content, as received basis (%)

R = mass measured at temperature X (mg)

S = mass measured at temperature Y (mg)

W = original specimen mass (mg)

3.3 Fixed carbon content nay be calculated by the following equation:

$$C = \frac{S - T}{W} \times 100 \qquad \text{(A. 1-3)}$$

where: C = Fixed carbon content, as received basis (%)

S = mass measured at temperature Y (mg)

W = mass measured at temperature Z (mg)

W = original specimen mass (mg)

3.4 The ash content may be calculated by the following equation:

$$A = \frac{T}{W} \times 100 \qquad \text{(A. 1-4)}$$

where: A = Ash content, as received basis (%)

T = mass measured at temperature Z (mg)

W = original specimen mass (mg)

Appendix B

Iodine number calculation

Iodine number calculation accordance with ASTM D4607-94

1. Preparation of Solution

1.1 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.

1.2 Sodium thiosulfate (0.100N)

Dissolve 24.820 g of sodium thiosulfate in approximately 75 ± 25 ml of boiled distilled water. Add 0.1 ± 0.01 g of sodium carbonate to minimize bacterial decomposition of the thiosulfate solution. Then, transfer the mixture to 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.

1.3 Standard iodine solution (0.100 ± 0.001 N)

Weigh 12.700 g of iodine and 19.100g 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 of volumetric flask and fill to the mark with distilled water. It is important that the standard iodine solution has an iodine-to-iodine weight ratio of 1.5 to 1. Store the solution in an amber bottle.

1.4 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 desiccator. Dissolve 3.5667 ± 0.1 mg of the dried potassium iodate in about 100 ml of distilled water. Quantitatively transfer to a 1 l of volumetric flask and fill to the mark with distilled water. Mix thoroughly and store in a glass-stoppered bottle.

1.5 Starch solution

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

2. Standardization of solutions

2.1 Standardization of 0.100 N sodium thiosulfate

Pipet 25.0 ml of potassium iodate (KIO_3) solution from 1.4 into a 250 ml titration (or wide-mouthed Erlenmeyer) flask. Add 2.00 ± 0.01 g of potassium iodide (KI) to the flask and then shake 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 and continue the titration drop wise until on drop produces a colorless solution. Determine sodium thiosulfate normality as follows:

$$N_1 = (P \times R)/S \qquad \text{(B. 1-1)}$$

where; N_1 = sodium thiosulfate, N

P = potassium iodate, ml

R = potassium iodate, N

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 of 0.003 N.

2.2 Standardization of 0.100 ± 0.001 N iodine solution

Pipet 25.0 ml of iodine solution (1.3) into a 250 ml wide mouthed Erlenmeyer flask. Titrate with standardized sodium thiosulfate until the iodine solution is a light yellow color. Add a few drops of starch indicator and continue titration until one drop produces a colorless solution. Determine the iodine solution normality as follows:

$$N_2 = (S \times N_2)/I \qquad \text{(B. 1-2)}$$

where; N_2 = iodine, N
S = sodium thiosulfate, ml
N_1 = sodium thiosulfate, N
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 of 0.003 N. The iodine solution concentration must be 0.100 ± 0.001 N. If this requirement is not met, the repetition has to be done.

3. Procedure

3.1 Grind a sample 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.

3.2 Dry the grounded carbon from 3.1 at the temperature of 110 °C for 3 h. Cool the dried carbon to room temperature in a desiccator.

3.3 Weight three appropriate amounts of dry carbon to the nearest milligram. Transfer each weighed sample of carbon to a clean, a 250 ml Erlenmeyer flask equipped with stopper.

3.4 Pipet 10.0 ml of 5 wt% hydrochloric acid solution into each flask containing carbon. Stopper each flask and gently swirl until the carbon is completely wetted. Put the stopper off to vent the flasks, place it on hotplate in a fume hood, and bring the contents to a boil for 30 ± 2 s to remove any sulfur which may interfere with the testing results. Remove the flasks from the hotplate and cool to room temperature.

3.5 Pipet 100.0 ml of 0.100 N iodine solution into each flask. Immediately stopper the flasks, and shake vigorously for 30 ± 1 s. Subsequently filter each mixture through one sheet of filter paper into the beaker.

3.6 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 titration with sodium thiosulfate until one drop produces a colorless solution. Record the volume of sodium thiosulfate used.

4. Calculation

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

$$A = (N_2)(12693.0) \qquad \text{(B. 1-3)}$$

where; N_2= iodine, N

$$B = (N_1)(126.93) \qquad \text{(B. 1-4)}$$

where; N_1 = sodium thiosulfate, N

$$DF = (I+H)/F \tag{B. 1-5}$$

where; DF = dilution factor

I = iodine, ml

H = 5% hydrochloric acid used, ml

F = filtrate, ml

4.2 Calculate the value of X/M as follows:

$$X/M = [A-(DF)(B)(S)]/M \tag{B. 1-6}$$

where; X/M = iodine adsorbed per gram of carbon, mg/g

S = sodium thiosulfate, ml

M = carbon used, g

4.3 Calculate the value of C as follows:

$$C = (N_1 \cdot S)/F \tag{B. 1-7}$$

where; C = residual filtrate, N

N_1= sodium thiosulfate, N

F = filtrate, ml

4.4 Using logarithmic paper, plot X/M (as the ordinate) versus C (as the abscissa) for each of three carbon dosages (see fig. 1). Calculate the least squares fit for the three points and plot. The iodine number is the X/M the residual iodine concentration (C) of 0.02 N.

Appendix C

Methylene blue adsorption performance

Methylene blue adsorption performance accordance with JIS K 1474-1991

1. Methylene blue preparation

Dry the methylene blue at the temperature of 110 °C for 2 h and then keep it into the dessicator. Dissolve 500 mg of the dried methylene blue in about 100 ml of DI water. Quantitatively transfer to a 1 L volumetric flask and fill to the mark with DI water. Repeat it again by changing the weight of methylene blue to 100, 200, 300 and 400 mg.

2. Standardization of methylene blue

2.1 Dry the methylene blue at the temperature of 110 °C for 2 h and then keep it into the dessicator. Dissolve 500 mg of the dried methylene blue in about 100 ml of DI water. Quantitatively transfer to a 1 L volumetric flask and fill to the mark with DI water.

2.2 Take 10 ml of the solution and dilute in 50 ml volumetric flask with DI water. The methylene blue having concentration of 100 mg/l will be obtained. Repeat it again by changing the concentration of methylene blue solution into 200, 300, 400 and 500 respectively.

2.2 Take 1 ml of the diluted methylene blue solution into one mark volumetric flask 100 ml and add DI water up to the marked line. The methylene blue with concentration of 1, 2, 3, 4 and 5 mg/l respectively will be obtained.

2.3 Take some of the methylene blue solution into an adsorption cell of 10 mm in optical path length and using a photometer, measure the absorbance tat 665 nm in wavelength.

3. Procedure

3.1 Grind a sample of carbon until all of them will pass through a 60-mesh screen.

3.2 Dry the ground carbon from 3.1. Cool the dried carbon to room temperature in a desiccator.

3.3 Weight amounts of dried carbon to the nearest milligram. Transfer each weighed sample of carbon to a clean, a 250 ml Erlenmeyer flask with stopper.

3.4 Add 200 ml of 100, 200, 300, 400 and 500 mg/l methylene blue and put on the stopper. Shake at room temperature using a water bath and shaker for 48 h.

3.4 Take 1ml of methylene blue out at the time of 1 min, 5min, 15 min, 30 min, 1 h, 3 h, 6 h, 8 h, 24 h and 48 h and then separate activated carbon and solution using centrifuge.

3.5 Pipet 0.1 ml of methylene blue solution and dilute to 10 ml.

3.6 Take some of the methylene blue solution into an adsorption cell of 10 mm in optical path length and using a photometer, measure the absorbance at 665 nm in wavelength.

3.7 Plot the relationship between concentration of standard methylene blue (x-axis) and absorbance (y-axis). The linear equation will be given.

4. Calculation

4.1 The methylene blue adsorption amount (mg/g) will be calculated using the following equation:

$$q = \frac{(A - C)x0.02}{S} \qquad \text{(C. 1-1)}$$

where; q = methyelne blue adsorption amount (mg/g)

A = initial concentration of methylene blue (mg/l)

C = remaining concentration of methylene blue (mg/l)

S = mass of sample (g)

0.02 = volume of methylene blue (l)

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