Activated carbon production from lesser reedmace by using hydrothermal treatment

By

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A Report Submitted in Partial Fulfillment of the Requirements
for the Degree of Bachelor of Engineering (Petrochemical Engineering)
Department of chemical Engineering, Faculty of Engineering,
King Mongkut's Institute of Technology Ladkrabang
Academic Year 2019

การผลิตถ่านกัมมันต์จากต้นธูปฤาษีโดยวิธีการไฮโดนเทอร์มอลทรีทเมนท์

พัชรพล ฉลาดธัญกิจ

ปริญญานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตร
วิศวกรรมศาสตร์บัณฑิต สาขาวิชาวิศวกรรมปิโตรเคมี
ภาควิชาวิศวกรรมเคมี คณะวิศวกรรมศาสตร์
สถาบันเทคโนโลยีพระจอมเกล้าเจ้าคุณทหารลาดกระบัง
ปีการศึกษา 2562

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ABSTRACT

In this project was study of activated carbon production from lesser reedmace by using hydrothermal treatment and activation by impregnated phosphoric acid. Lesser reedmace flower used in this project because higher of %carbon and %fixed carbon.

The results showed that activated carbon production from lesser reedmace flower by using hydrothermal treatment and activation by impregnated phosphoric acid could give high porosity activated carbon. The first step was carbonization by using hydrothermal treatment which was carried out in the temperature and reaction times range of 150-210°C and 60-180 min. It was found that the optimum condition was 210°C and 120 min, the characteristics analysis was 42.14%yield, 2.94%moisture, 12.34%ash, 29.85%volatile matter, 54.84%fixed carbon and 873.1693 mg/g of iodine number. Then choose 4 best one for activation by impregnated phosphoric acid which were impregnated ration 5:1 (phosphoric acid: char) at room temperature. The study variables were impregnation times. The optimum impregnation times was 12 hr, iodine number increase from 873.1693 mg/g to 1118.087 mg/g. The prepared activated carbon from lesser reedmace flower could be the commercial grade, because the accepted standard iodine absorption is more than 600 mg/g.

Keywords: Activated carbon, Hydrothermal treatment, Iodine number, Activation

เรื่อง การผลิตถ่านกัมมันต์จากต้นธูปฤาษีโดยวิธีการไฮโดนเทอร์มอลทรีทเมนท์

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บทคัดย่อ

งานวิจัยนี้เป็นการศึกษากระบวนการผลิตถ่านกัมมันต์จากต้นธูปฤาษีด้วยกระบวนการไฮโดรเทอร์มอลทรีตเมนท์และกระตุ้นโดยวิธีการแช่กรดฟอสฟอริก ในงานวิจัยนี้ใช้ส่วนดอกของต้นธูปฤาษีในการผลิตถ่านกัมมันต์เพราะส่วนดอกของธูปมีร้อยละคาร์บอนและร้อยละคาร์บอนคงตัวสูงที่สุด

ผลการศึกษาแสดงให้เห็นว่าการนำส่วนดอกของต้นธูปฤาษีมาผลิตถ่านกัมมันต์การโดยวิธีการไฮโดรเทอร์มอลทรีทเมนท์และกระตุ้นโดยวิธีการแช่กรดฟอสฟอริกสามารถผลิตถ่านกำมันต์ที่มีรูพรุนสูงได้ เริ่มต้นด้วยการหาอุณหภูมิและเวลาที่เหมาะสมสำหรับกระบวนการไฮโดรเทอร์มอลทรีตเมนท์ในช่วง 150-210 องศาเซลเซียส และ 60-180 นาที โดยสภาวะการดำเนินงานที่เหมาะสมสำหรับวิธีการไฮโดนเทอร์มอลทรีทเมนท์ที่ความดัน 40 บาร์คือ ที่อุณหภูมิ 210 องศาเซลเซียส เวลา 120 นาที การวิเคราห์คุณสมบัติโดยมีร้อยละผลิตภัณฑ์ 42.14 ร้อยละความชื้น 2.94 ร้อยละเถ้า 12.34 ร้อยละสารละเหย 29.85 ร้อยละคาร์บอนคง 54.84 และค่าการดูดซับไอโอดีนเป็น 873.1693 มิลลิกัมต่อกรัม จากนั้นได้นั้นได้เลือกตัวอย่างที่ดีที่สุด 4 ตัวอย่างไปทำการกระตุ้นต่อด้วยวิธีการแช่ด้วยกรดฟอสฟอริกโดยอัตราส่วยโดยมวลของตัวอย่างต่อกรดคือ 1 ต่อ 5 ที่อุณหภูมิห้อง โดยศึกษาตัวแปรที่ใช้คือเวลาในการกระตุ้นด้วยวิธีการแช่กรอฟอสฟอริกในช่วง 6-18 ชั่วโมง ซึ่งเวลาในการกระตุ้นด้วยวิธีการแช่กรอฟอสฟอริกที่เหมาะสมที่สุดคือ 12 ชั่วโมง พบว่าค่าการดูดซับไอโอดีนเพิ่มขึ้นจาก 873.1693 มิลลิกัมต่อกรัมเป็น 1118.087 มิลลิกัมต่อกรัม ถ่านกัมมันต์ที่ได้จากส่วนดอกของต้นธุปฤาษีอยู่ในมาตราฐานเกรดการค้า เพราะให้ค่าการดูดซับไอโอดีนมากกว่า 600 มิลลิกรัมต่อกรัมนั้นเป็นที่ยอมรับโดยทั่วกัน

คำสำคัญ ถ่านกัมมันต์, ไฮโดรเทอร์มอลทรีทเมนท์, ค่าการดูดซับไอโอดีน, การกระตุ้น

ACKNOWLEDGEMENTS

I would like to express the deepest appreciation to advice and assistance from faculty and individuals

Asst. Prof. Dr. Thachanan Samanmulya, thesis advisor that provides advice, guideline, and knowledge in other areas until this research was successful.

Asst. Prof. Dr. Yaneeporn Patcharavorachot and Asst. Prof. Dr. Tanawan Pinarat whose help in reviewing research articles and providing useful research advice and helped me a lot in finalizing in this project.

The family that teaches is an important encouragement and has been a consultant continuously.

All chemical engineering, who gave me encouragement and support. If anything goes wrong in any way, the facilitator would like to accept and apologize

Patcharapol Chaladthunyakij

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CHAPTER I

INTRODUCTION

1.1 Background [1-2]

Separation and purification of gas or liquid mixtures by adsorption has become a major unit operation in the Chemical industries and Petrochemical industries. Adsorbents are also required in varies application, in either liquid or gas phase processes, such as air purification, gas separation and purification, gas separation and purification, wastewater treatment, organic solvent recovery, heterogeneous catalysis, decolorization of organic substances, etc. Activated carbons are popularly used as adsorbents in various technologies due to their highly porous and large adsorption capacity

As a result of environmental requirements in many countries and new areas of application the demand on activated carbon is still growing. Due to the unavailability of the main basic materials like hard coal, wood or coconut shells in many countries other biomass matters were tested for their appropriateness of activated carbon production.

The objective of this experimental work is the conversion of lesser reedmace into activated carbon. Waste biomass is converted thermally in two steps. First the biomass undergoes a hydrothermal treatment process. The gaseous and liquid hydrothermal treatment products can be used alternative fuels. Second, the solid residue, activated carbon, is treated in an activation process in steam atmosphere in order to enhance the char surface area which was analyzed by standard test method for determination of iodine number of activated carbons. The increase of surface area depends on the type of biomass and on the activation parameters.

In the present, Thailand has many of lesser reedmace. Lesser reedmace are well suited to live in an environment of fluctuating water levels. Used lesser reedmace is one of the interesting raw material because lesser reedmace can be found in wetlands throughout the country their seeds germinate quickly in marshy mudflats, making them quick to breed after human or natural disturbances, easy for carbonization and main structures are carbon.

In this study activated carbon was prepared from lesser reedmace flower by hydrothermal and activate by impregnated phosphoric acid . The produced activated carbon were characterized by iodine number. This research work aims to study the

effects of hydrothermal treatment temperatures and times on the pore surface area and yields of resulting product. The potential applications of activated carbon derived cattail flower as an alternative adsorbent were also examined.

1.2 Objectives

1.2.1 To investigate the effect of hydrothermal treatment on properties of activated carbon.

1.2.2 To study optimal conditions for carbonization by hydrothermal treatment and activation by impregnated phosphoric acid on activated carbon from lesser reedmace.

1.3 Scope of work

For the production of activated carbon from lesser reedmace by hydrothermal treatment and activation by phosphoric acid, the suitable condition such as temperatures, carbonization times and activation times of raw material were studied. The essential procedures are following:

1.3.1 Prepare activated carbon from lesser reedmace by hydrothermal treatment process.

1.3.2 Characterization of lesser reedmace.

1.3.3 Carbonization by hydrothermal treatment changing the following parameters

Temperature: 150, 180 and 210 $^{\circ}$C

Time: 60, 120 and 180 minutes.

1.3.4 Production of activated carbon from lesser reedmace by impregnated phosphoric acid changing the following

Impregnation times: 6, 12 and 18 hrs.

1.3.5 Investigation of the properties of the activated carbon (iodine number)

1.3.6 Summarize the results and conclusion

1.4 Expected Outputs

1.4.1 Expected the hydrothermal treatment could be increased the surface area and porosity.

1.4.2 Expected the results of activated carbon property which prepared from lesser reedmace flower could be nearby the commercial grade.

CHAPTER II

LITERATURE REVIEWS

2.1 Activated Carbon [1]

Activated carbon is a diverse and athletic adsorbent. It can be used to control the abundant majority of all the various molecules that environment pollution. These carbons are able to adsorb a large range of various molecules. Activated carbon can be made from various raw materials, different qualities and different shapes and sizes of porous. This is a very important character when the mix of chemicals is unknown, or variable, or perhaps too complex. Reforming these factors to optimize efficiency and lifetime for different applications. Depending on activated carbon operates with a physical adsorption, chemical adsorption or catalytic mechanism and the applications.

2.2 Raw materials for the production of activated carbon [2]

The principal properties of manufacture activated carbons depend on the type and properties of the raw material used. Any cheap substance with a high carbon and low ash content can be used as a raw material for the production of activated carbon. In Europe the most essential raw materials used for this purpose are wood, charcoal, peat, peat coke, certain types of hard and brown coal. To produce activated carbon, which should exhibit high adsorption capacity and a large volume of the smallest pores. Source materials that have been studied for the production of activated carbon.

Bagasse	Corncobs and corn	Leather waste	Petroleum ferricyanide
Beet-sugar sludges	Stalk	Lampblack	Potassium ferricyanide
Bones	Distillery waste	Lignite	Petroleum coke
Carbohydrates	Fish	Molasses	Pulp-mill waste
Cereals	Flue dust	Nut shells	Rice hulls
Coal	Fruit pits	Oil shale	Rubber waste
Coconut shells	Graphite	Peat	Sawdust
Coffee beans	Kelp and Seaweed	Polymer scrap	Wood

2.3 Lesser reedmace [3-4]

Lesser reedmace are a common wetland plant. Easily recognizable by their tall leaves and signature brown pistillate spike, they grow along marsh edges in emergent wetlands throughout the world. Although they can cause problems when growing out of control, they are also a necessary keystone species.

They provide shelter for birds and mammals, shade water to cool fish, and their energy-rich rhizomes and shoots are a nutritious food source enjoyed by many animals including humans.

Lesser reedmace are well suited to live in an environment of fluctuating water levels and high fertility. Their seeds germinate quickly in marshy mudflats, making them quick to recolonize after human or natural disturbances. They can grow in a wide range of shallow water depths depending on species, age, and condition of the stand. Maximum water depths are typically 2-3 feet, although greater depths can be tolerated for brief periods. Cattail can even grow as floating mats on the water's surface, helping it to colonize water deeper than it could grow in otherwise. Once a stand of cattails is established, it alters its habitat. New stems and root/rhizome masses grow and accumulate on dead stalks and other organic material. As sediments accumulate, they alter nutrient cycles and prevent light from reaching the substrate, thus excluding other plants. The accumulation of sediments and root mass eliminates shallowly flooded marsh edges. Cattails are also excellent at filtering polluted runoff containing sediment, fertilizer, and heavy metals. By capturing these pollutants, they prevent, or at least delay, them from having larger negative effects in the environment.

The lesser reedmace inflorescence is at the top of stems and begins developing within sheathing leaves in late May or early June. By late June, leaves open and show the two-tiered spike: the top containing tiny male flowers with pollen and the lower, pistillate spikes holding the female flowers, where seeds will develop. Both flower types are densely packed, making it hard to identify individual flowers. When spikes first emerge, they are green but turn brown as they mature then break down as pollen. Later fluffy seeds are shed.

2.4 Adsorption [5]

Adsorption is a phenomenon wherein the local concentration of a substance at the surface of a solid or liquid convert greater than the concentration all over the bulk. Capability of all solid substances to attract to their surface molecules of gases or solutions with which they are in contact. Solids that are used to adsorb gases or dissolved substances are called adsorbents. We thus have the well-known phenomenon of gas molecules concentrating on charcoal or pigments passing from solution in oil to deposit on clay. By contrast, absorption relates to the uniform penetration and dispersal of one item into another. Thus, solute molecules which reduce surface tension will concentrate at an interface between a solid and a solution, and tend to be adsorbed on the solid. If, as happens in some cases, the solute increases surface tension, the concentration at the interface is decrease this is known as negative adsorption. Water has a high surface tension most solutes reduce this, so they are easily taken up by an adsorbent.

2.4.1 The classification of adsorption [6]

Physical adsorption is the result of a relatively weak solid-gas interaction. It is a physical attraction resulting from nonspecific, relatively weak Van der Waal's forces and adsorption energy not exceeding 5-50 kJ/mole. Physically adsorbed molecules may widespread along the surface of the adsorbent and normally are not bound to a specific location on the surface. Being only weakly bound, physical adsorption is simply reversed.

Chemical adsorption can result in a surface complex, a union much stronger than a physical bond with heats of adsorption up to about 200-400 kJ/mole for C-N bondsand 800 kJ/mole for chemical bonds. A chemical bond involves sharing of electrons between the adsorbate and the adsorbent and may be regarded as the formation of a surface compound. Due to the bond strength, chemical adsorption is difficult to reverse.

Table 2.1 Comparison between physisorption and chemisorption [7]

Physical adsorption	**Chemical adsorption**
Low enthalpy (5–50 kJ/mol)	High enthalpy (200–400 kJ/mol)
Reversible process	Irreversible process
Intermolecular forces of attraction are van der Waals forces, hydrogen bonding, etc.	Valence forces of attraction are chemical bond forces
Multi-molecular layers may be formed	Generally, monomolecular layer is formed
Process is take place at low temperature	This process takes place at high temperatures
It is not specific	It is highly specific

2.4.2 Factors affecting adsorption [8]

- Surface area of adsorbent. Larger sizes imply a greater adsorption capacity.
- Particle size of adsorbent. Smaller particle sizes reduce internal diffusional and mass transfer limitation to the penetration of the adsorbate inside the adsorbent. However, wastewater drop across column packed with powdered material is too high for use of this material in packed beds. Addition of powdered adsorbent must be followed by their removal.
- Contact time or residence time. The complete adsorption might need longer residence time.
- Solubility of solute (adsorbed) in liquid (wastewater). Low solubility substance will be more easily removed from water than high solubility substances.
- Non-polar substances will be more easily removed than polar substances since the latter have a greater affinity for water.
- Affinity of the solute for the adsorbent (carbon). The surface of activated carbon is only slightly polar. Hence non-polar substances will be more easily picked up by the carbon than polar ones.
- Number of carbon atoms. For substances in the same homologous series a larger number of carbon atoms is generally associated with a lower polarity and hence a greater potential for being adsorbed.

2.4.3 Adsorption isotherm [9]

Adsorption in a solid-liquid system result in the removal of solute from solution and their concentration at the surface of the solid, to such time as the concentration of the solute remaining in solution is in a dynamic equilibrium with that at the sureface. At this position of equilibrium, there is a set distribution of solute between the liquid and solid phases. The distribution expresses the quantity q_e as a function of C at fixed temperature, the quantity q_e being the amount of solute adsorbed per unit weight of solid adsorpbent, and C the concentration of solute remaining in solution at equilibrium. An expression of this type is termed an adsorption isotherm.

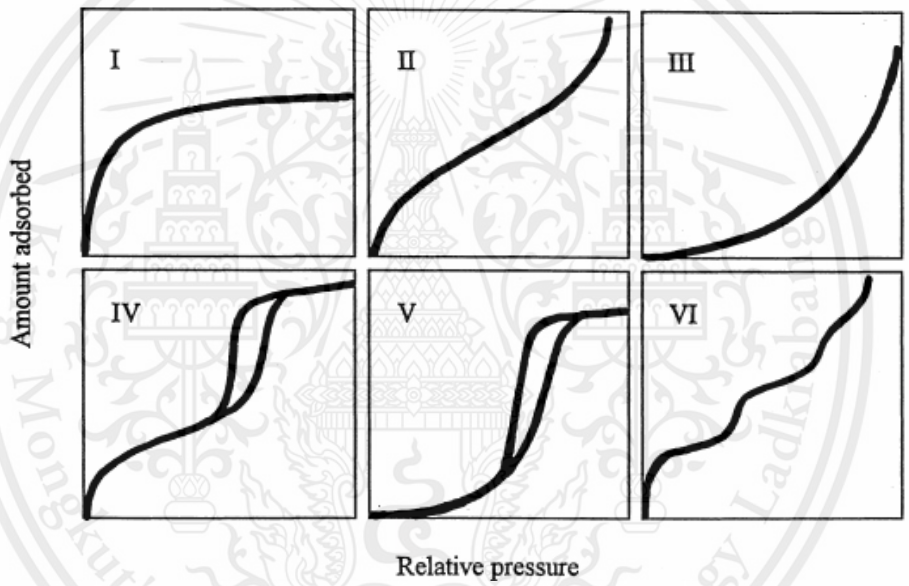

Figure 2.1. The IUPAC classification of adsorption isotherms showing both the adsorption and desorption pathways. Note the hysteresis in types IV and V.

1. Type I isotherm is concave to the p/p^0 axis and approaches a limiting value as p/p°. Type I isotherms are given by microporous solids having relatively small external surfaces the limiting uptake being governed by the accessible micropore volume rather than by the internal surface area.

2. Type II isotherm is the normal form of isotherm is given by a non-porous or macroporous adsorbent. Type II isotherm represents unlimited monolayer-multilayer adsorption. Point B, the beginning of the almost linear middle section of the isotherm,

is often taken to indicate the stage at which monolayer coverage is complete and multilayer adsorption about to begin.

3. Type III isotherm is convex to the p/p^0 axis over its entire range and therefore does not exhibit a Point B. Isotherms of this type are not common; the best known examples are found with water vapor adsorption on pure non-porous carbons. However, there are a number of systems which give isotherms with gradual curvature and an indistinct Point B. In such cases, the adsorbent-adsorbate interaction is weak as compared with the adsorbate-adsorbate interactions.

4. Type IV isotherm are its hysteresis loop, which is associated with capillary condensation taking place in mesopores, and the limiting uptake over a range of high p/p°. The initial part of the Type IV isotherm is attributed to monolayer multilayer adsorption since it follows the same path as the corresponding part of a Type II isotherm obtained with the given adsorptive on the same surface area of the adsorbent in a non-porous form. Type IV isotherms are given by many mesoporous industrial adsorbents.

5. Type V isotherm is uncommon; it is related to the Type III isotherm in that the adsorbent-adsorbate interaction is weak, but is obtained with certain porous adsorbents.

6. Type VI isotherm represents stepwise multilayer adsorption on a uniform non-porous surface. The step height now represents the monolayer capacity for each adsorbed layer and, in the simplest case, remains nearly constant for two or three adsorbed layers. Amongst the best examples of Type VI isotherms are those obtained with argon or krypton on graphitized carbon blacks at liquid nitrogen temperature.

Langmuir adsorption model **[10]**

The Langmuir adsorption isotherm is based on the following assumptions.

1. The adsorbent surface consists of a certain number of active sites, at each of which only one molecule may be adsorbed.
2. No lateral interaction between the adsorbed molecules, thus the heat of adsorption is constant and independent of coverage.
3. The adsorbed molecule remains at the site of adsorption until it is desorbed.

4. At maximum adsorption, only a monolayer is formed: molecules of adsorbate do not deposit on the other, already adsorbed, molecules of adsorbate, only on the free surface of adsorbent.

Assign to

$$\boldsymbol{\theta = q/q_m}$$

Where $\boldsymbol{\theta}$ is the degree of filling of sites.

q is the amount adsorbed adsorbate per unit weight of adsorbent at equilibrium (mg/g)

q_m is the maximum solute concentration in the adsorbent (mg/g)

The rates of adsorption

$$r_a = k_a C(1-\theta)$$

Where r_a is rates of adsorption.

k_a is adsorption constant.

C is equilibrium concentration of adsorbate in solution.

The rates of desorption.

$$r_d = k_d \theta$$

Where r_d is the rates of desorption.

k_a is desorption constant.

At equilibrium

$$k_a C_x (1-\theta) = k_d \theta$$

$$\theta = k_a C_x / (k_d + k_a C_x)$$

Define $K_A = k_a/k_d$ = equilibrium constant

Lead to

$$q = \frac{q_m K_A C}{1+K_A C}$$

Can be arranged to the linear from as follows;

$$\frac{C}{q} = \frac{1}{K_A q_m} + \frac{C}{q_m}$$

or

$$\frac{1}{q} = \frac{1}{q_m} + \frac{1}{(K_A q_m)C}$$

q_m and K_A can be determined from slope and y-intercept of graph

2.5 The Determination of the Specific Surface Area using the BET Method [11]

The specific surface area is an important parameter affecting the course of the chemical reactions occurring in coal during its pyrolysis, combustion, hydrogenation, and other processes. The adsorption characteristics of the fine-grained (<0.071 mm) samples of activated carbons were measured performing low-temperature argon adsorption, the BET method. The BET equation does not predict surface areas of microporous carbons. However, estimation of surface areas of different porous materials is important for practice. To evaluate specific surface areas of different carbonaceous materials, nitrogen, argon, and carbon dioxide are most often used. In this work, argon at 77 K is used as the adsorbate to monitor the surface evolution properties of the tested carbon materials as the treatment progresses. Its spherical shape

$$\frac{P}{V(P_0-P)} = \frac{1}{v_m c} + \frac{(c-1)P}{v_m c P_0}$$

Where V = volume of gas adsorbed (cm^3)

P/P_0 = relative pressure

V_m = the quantity of gas adsorbed for monolayer coverage of surface (cm^3)

C = BET constant

$$S = \frac{V_m N_{AV}}{22400} a$$

Where S = porous surface area (m^2)

N_{AV} = Avogado Number (6.02×10^{23} molecules mol^{-1})

a = is area by unit of molecular gases (cm^2 $molecule^{-1}$)

2.6 Iodine number [12]

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

Calculate the value of X/M as follows:

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

Where: X/M = iodine absorbed per gram of carbon, mg/g.

S = sodium thiosulfate, mL, and

M = carbon used, g.

$$DF = (1 + H) / F$$

Where: DF = dilution factor.

I = iodine, ml.

H = 5 % hydrochloric acid used, ml. and

F = filtrate, ml.

Carbon dosage may be estimated as follows:

$$M = [A – (DF)\ (C)\ (126.93)\ (50)] / E$$

Where: M = carbon, g.

A = (N2) (12693.0)

DF = dilution factor.

C = residual iodine, and

E = estimated iodine number of the carbon.

2.7 Conversion of biomass by hydrothermal treatment. [13]

Technologies for the conversion of waste biomass into bio-based chemicals and biofuels are hydrothermal (HT) processes, which use subcritical and supercritical water as processing. Fig. 2.2 presents phase diagram of water. Subcritical water is pressurized water at below the critical and temperatures above its boiling point. System properties become more sensitive to pressure and temperature changes. By increasing temperature, the density of the liquid phase decreases, the density of the vapor increases and becomes more like at critical point. Water above the critical point is called supercritical water and has properties between liquid phase and gas phase. Generally, HT processes is divided for four main processes: carbonization (HTC), aqueous phase reforming (APR), liquefaction (HTL), and gasification (HTG). In these processes water has the role of reactant, solvent, and also a catalyst. The main advantage over other processing methods includes ability to use wet biomass without prior dewatering and enables production of versatile chemicals and fuels in gaseous, liquid, or solid state.

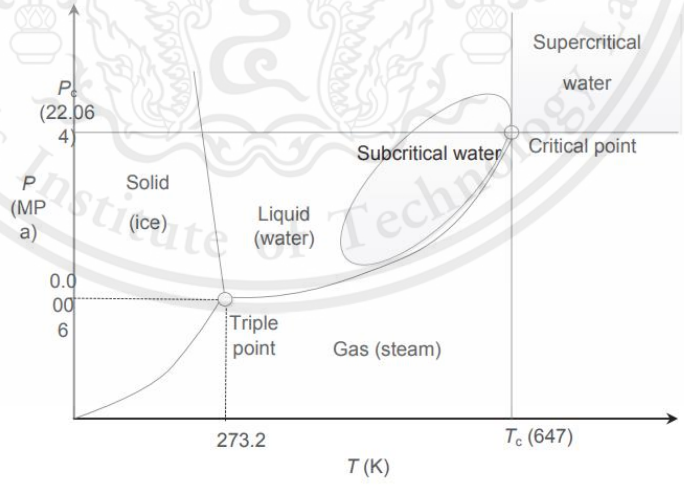

Figure 2.2. Phase diagram of water with area of application for hydrothermal treatment; (T_c - critical temperature, P_c - critical pressure).

Hydrothermal (HT) reactions appear in water media at higher temperatures and pressures (sub- and supercritical water) where hot compressed water can act as a solvent, as a reactant, or as a catalyst. The advantage of hydrothermal reaction processes over conventional ones is that wet biomass usually contains around 70 wt% or more water which can be more economically converted to chemicals and bio-based fuels. The four main HT reactions of biomass conversion into desired energy products and different operating parameters are presented in Fig 2.3 and Table 2

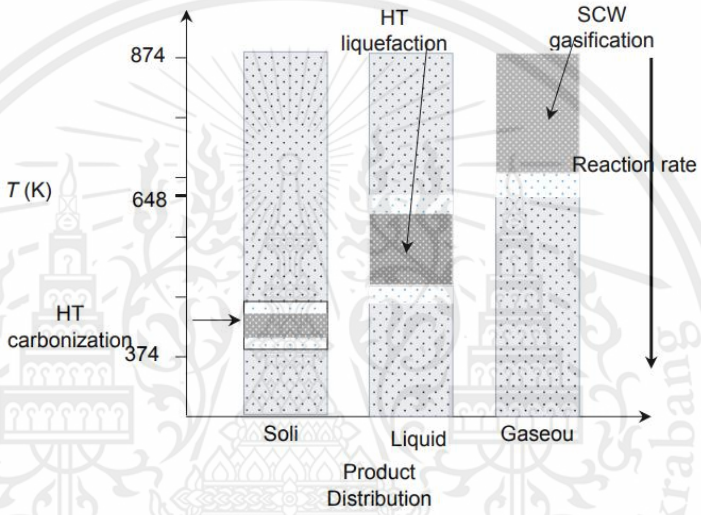

Figure 2.3 Hydrothermal reactions for biomass conversion e products are controlled by changing process parameters.

Fig. 2.3 presents reaction rate as a strong function of temperature. Depending on the reaction temperature and pressure it may vary considerably; from some seconds to several hours. Different products are obtained by fine-tuning process parameters. As demonstrated in Fig. 2.3, hydrothermal carbonization is performed at mild temperatures, usually up to 524 K. Solid products with a high content of carbon are obtained. Such products have high energy contents and are suitable chemicals for different applications. Hydrothermal liquefaction is demonstrated as a process that is typically carried out at temperatures between 524 K and 648 K. The product is highly viscous liquid - pyrolysis oil which can be applied as a pure chemical or can be added to diesel fuel. It is also demonstrated that at highest temperatures, above 648 K, supercritical water gasification is performed. Gaseous products with a high content of

hydrogen and carbon monoxide are obtained. CO_2 and other hydrocarbons are present in lower concentrations. This gas could be used as a source of energy or as "syngas" for synthesis of higher value chemicals. The operating conditions of biomass treatment by hot compressed water effect both, product distribution and characterization. The physicochemical properties of the target products are influenced by hydrothermal temperature and residence time

Table 2.2 Main hydrothermal reactions and different working conditions for obtaining highly valued product.

Hydrothermal reactions	Temperature (°C)	Pressure (bars)	Reaction time	Main product
HT carbonization	150 - 280	20 – 40	Several hours	Hydrochar
HT liquefaction				
Low temperature	280 - 370	100 - 250	Few seconds	Increasing bio-oil yield
High temperature	300 - 600	100 - 250	Few seconds	Improved to transport fuel by increasing C/H ratio
HT gasification				
Near-critical	300 - 500	Various	Few seconds	Methane
Supercritical	500 - 800	Various	Few seconds	Syngas H_2 with minor, CO_2, C_1- C_4 gasses
Aqueous phase reforming	220 - 250	15 - 50	Several hours	H_2 and CO_2 with minor C_1

Hydrochar [14]

Hydrochar is produced at a relatively low temperature of 180-260°C through hydrothermal carbonization in subcritical water. Though biochar, torrefied wood and hydrochar looks similar there are significant difference between them in terms of how they are produced, product motivation as well as physical and chemical properties. Biochar is produced in dry oxygen starved ambience, while hydrochar is produced in

subcritical water at lower temperature. Biochar is used primarily for soil remediation and as an agent for carbon sequestration while hydrocarbon has value added industrial use. Hydrothermal carbonization being a faster process required much shorter residence time than dry torrefaction.

2.8 Activation [14]

Activation is a process that targets for increasing surface area and pore of activated carbon which can enhance the ability of adsorption. The type and degree of activation process affect its physical and chemical properties as follows:

2.8.1 Physical activation [15]

Physical activation processes with stream, nitrogen or carbon dioxide are used for mild oxidation of the carbonaceous matter. The process is usually carried out in two stage, carbonization and activation stage. Raw material is primary process in physical carbonization by pyrolyzed at temperature range of 600-900°C in absence air and consequently physical activation process by carbonized with oxidizing atmosphere at temperature usually range from 600-1200°C

2.8.2 Chemical activation [16]

Chemical activation usually uses chemical agents to impregnate raw material at range of temperature 450-900°C. The activated carbons obtained by chemical activation are with high surface area and with well-developed microporosity, which can be controlled and maintained narrow. In chemical activation processes, all chemical agents used are dehydrating agents that influence pyrolytic decomposition and inhibit formation or tar, thus, enhancing the yield of carbon.

A typical chemical composition of lignocellulosic materials could be taken as 48% C, 6% H and 45% O, the inorganic matter being a minor component [6]. The corresponding atomic ratios are: O/C = 0.73, H/C = 1.5 and H/O = 2.07. Since the transformation in char requires the removal of O and H, the degree of conversion to carbon (carbonization yield) may vary widely as a function of the amount of carbon being removed with O and H. A conventional carbonization process leads to a yield around 20–30%, lower than the expected value (up to 48%) if only water is the result

of the removal of H and O. This means that a chemical activation, where the activating agent is a dehydrating compound, will mean not only an increase in yield but also a change in the thermal degradation of the precursor, with the subsequent change in the evolution of porosity.

Several authors have studied the carbonization of lignocellulosic materials and it was found that the main degradation of the precursor takes place in the 200–350 ˚C temperature range, with evolution of H_2O, CO, CO_2, CH_4, aldehydes, etc. Distillation of heavier hydrocarbons (tar) takes place in the range 350–500 ˚C, and above 500 ◦C, there is little weight loss, thus indicating that the basic structure is already formed. Consequently, if chemical activation is used, one could expect that the activity of the reagent is almost complete at this temperature, this being the main reason why chemical activation is commonly carried out at 450–600 ◦C. Since at this temperature the carbonization is not complete, the chemical composition of the activated carbon obtained (after the elimination of the chemical by washing) is between that of the precursor and the char.

There is a contraction of the lignocellulosic precursor along the carbonization process. Studies carried out with almond shells show that the weight loss of around 75% is accompanied by a contraction of around 30%. This change in dimensions during carbonization are more important in chemical activation since the reagent is incorporated into the interior of the particles and it may inhibit the expected contraction with increasing temperature. This means that the reagent may act as template for the creation of microporosity. Of the many reagent proposed for chemical activation (zinc chloride, phosphoric acid, aluminium chloride, magnesium chloride, potassium hydroxide, sodium hydroxide, etc.) those more commonly used are $ZnCl_2$, H_3PO_4 and KOH

2.9 Related research

Nadhem K. Hamadi [17] presented the removal of pesticide from wastewater under different batch experimental conditions, using a car tire derived activated carbon was investigated. The pesticide utilized in the study was Paraquat dichloride (1,1-dimethyl-4,4-bipyridyl dichloride), which is a well known herbicide. The adsorbent was produced from the pyrolysis and activation of used tires (TAC). The performances

of this adsorbent and a commercial activated carbon F300 (CAC) have been compared. It was determined that the adsorption of Paraquat was weakly pH dependent. The effects of particle size, carbon dosage, temperature and the initial concentration of the Paraquat were studied. Further experiments investigating the regeneration capabilities of the tire-supplied carbon were performed. The regenerated carbons that were washed with basic pH solution were found to have the best sorption capacity recovery. It was found that the rate of sorption of Paraquat onto the carbon is very fast with almost 90% of the maximum possible adsorption taking place in the first 5 min. Nevertheless, the batch sorption kinetics was fitted for a first-order reversible reaction, a pseudo-first-order reaction and a pseudo-second-order reaction. The pseudo-second-order chemical reaction model appears to provide the best correlation. The applicability of the Langmuir isotherm for the present system has been evaluated at different temperatures. The isotherms show that the sorption capacity of CAC decreases with temperature and the dominant mechanism of CAC adsorption is physical sorption.

Monica Puccini [18] presented the hydrothermal carbonization (HTC) is a thermo-chemical process for recovery and valorization of biomass and organic waste that uses sub-critical liquid water as a reaction medium for converting feedstock into valueadded products. HTC treatment produces a carbonaceous solid, named hydrochar, with attractive characteristics that can be used in a variety of applications, such as activated carbon or soil amendment. In this study, activated carbons were prepared by KOH chemical activation of hydrochars obtained from hydrothermal carbonization of green waste and municipal solid waste. The effects of precursors and different KOH/char mass ratios on physical characteristics of the activated carbon were investigated. Methylene blue and iodine numbers were determined to evaluate the adsorption properties of obtained activated carbons. The results have shown a significant increase of porosity and a change in hydrochar structure by KOH activation method. The amount of mesopores and micropores increase with increasing KOH/char ratio. In particular, the sample by treatment of hydrochar from green waste activated with a 2:1 KOH/char weight ratio presented good mesoporosity, with a methylene blue number of 385 mg/g, and a well-developed micropore structure, with iodine number of 747 mg/g. Therefore, the application of KOH activated hydrochar as activated carbon appears to be promising.

CHAPTER III

RESEARCH METHODOLGY

3.1 Raw material

This research was experimental in laboratory to find out condition in producing activated carbon from lesser reedmace by carbonized by hydrothermal treatment and activation by impregnated with phosphoric acid. The pictures of lesser reedmace and char are shown in Figure 3.1 respectively

Figure 3.1 The powder of lesser reedmace flower.

Figure 3.2 The char of lesser reedmace flower after carbonization by hydrothermal treatment.

3.1.1 Sample preparation of the lesser reedmace

3.1.1.1) Bring lesser reedmace flower, then was dried at 105 degrees Celsius for 24 hours.

3.1.1.2) Then take the lesser reedmace flower to grind it with a biomass blender to have a smaller particle size.

3.1.1.3) Take each powder of lesser reedmace flower through a sieve of smaller than 150μm, and collect in an air seal bag.

3.1.2 Analysis of the basic components of the lesser reedmace

3.1.2.1 Moisture content

1) Weigh the fresh lesser reedmace flower before drying. Record the weight. Then put in the oven at 70 degrees Celsius to expel the moisture from each part.

2) Weigh the samples every 6 hours in order to record the weight change from drying.

3) Drying the sample until the weight of lesser reedmace flower is constant or there is a difference of the weight measured 3 times, the difference must not be more than 0.003-0.005 grams and then calculate the percentage of moisture.

3.1.2.2 TGA analysis

3.1.2.3 C, H, N, S analysis

3.2 Chemicals

1. Distillation water.
2. Hydrochloric Acid, concentrated.
3. Sodium Thiosulfate, ($Na_2S_2O_3 \cdot 5H_2O$).
4. Standard iodine solution 0.1 N.
5. Potassium Iodide.
6. Potassium Iodate, primary standard.
7. Starch.
8. Sodium Carbonate.
9. Phosphoric acid.

3.3 Apparatus

1. High pressure auto clave
2. Oven: 0-200 °C, WT binder, Germany.
3. Desicator.
4. Laboratory test sieve
5. Shaker
6. Crucible.
7. Spectrophotometer
8. C.H.N Analyzer
9. Analytical Balance, accuracy 60.0001 g.
10. Burette, 10-mL capacity or 5-mL precision burette.
11. Flasks, Erlenmeyer 250-mL capacity with ground glass stoppers.
12. Flask, Erlenmeyer wide-mouthed, 250-mL capacity.
13. Beakers, assorted sizes.
14. Bottles, amber, for storage of iodine and thiosulfate solutions.
15. Funnels, 100-mm top inside diameter.
16. Filter Paper.
17. Pipets, volumetric type, 5.0, 10.0, 25.0, 50.0, and 100.0-mL capacity.
18. Volumetric Flasks, 1 L.
19. Graduated Cylinders, 100 mL and 500 ml.

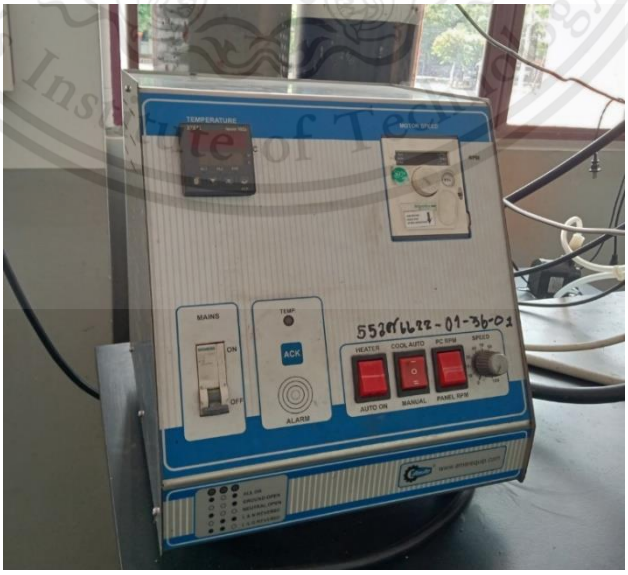

Figure 3.3 Temperature and pressure controller.

Figure 3.4 High pressure autoclave.

Figure 3.5 Nitrogen tank.

3.4 Hydrothermal treatment

The variation of temperature and time for carbonization of lesser reedmace were studied at 150,180 and 210 °C. 10 g of lesser reedmace char with particle size of 1.18-2.36 mm was loaded into the auto clave. Then the auto clave was passed 100 mL/min with N_2 flow through from the top to the bottom. Then the reactor was heated to the desired temperature to vary at 150, 180 and 210 °C. The superheated steam was charged continuously on, vary for 60,120 and 180 min of reaction time. Finally, the product was characterized by iodine number.

The effect of carbonization temperature

Three different temperatures were studied for carbonization by using hydrothermal treatment. They were 150, 180 and 210 °C. 10 g of lesser reedmace powder was loaded into a high pressure autoclave reactor. Then nitrogen was feed into high pressure autoclave reactor until pressure in high pressure autoclave reactor up to 40 bars. Then the high pressure autoclave reactor was heated to the desired temperature at 150, 180 and 210°C. The superheated steam was charge continuously with lesser reedmace powder, for each temperature for 60, 120 and 180 min of reaction time. Finally, the product was characterized by iodine number.

The effect of carbonization time

Different carbonization times of 1, 2 and 3 hrs. were studied for carbonization. 10 g of lesser reedmace was loaded into the high pressure auto clave. The N_2 were charged up to 40 bars. Then the sample in high pressure auto clave was heated with super heated steam. Secondary, the auto clave was heated to the desired temperature at 150, 180 and 210°C. Then operate for 1, 2 and 3 hr. The super heated steam were operated continuously. Finally, the product was characterized by iodine number.

3.5 Activation

In the activation step. Activation by impregnated with phosphoric acid were impregnated ration 5:1 (phosphoric acid: char) at room temperature. Studied variables in this process were impregnation time of 6, 12 and 18 hr.

3.6 Iodine number determination

3.6.1 Preparation of Solutions

3.6.1.1 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.

3.6.1.2 Sodium Thiosulfate (0.1 N)

Dissolve 24.820 g of sodium thiosulfate in approximately 75 mL of boiled distilled water. Add 0.10 g of sodium carbonate to minimize bacterial decomposition of the thiosulfate solution. Then 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.

3.6.1.3 Standard Iodine Solution (0.1 N)

Weigh 12.7 g of iodine and 19.1 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. Then adding tiny increments of water 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 completely dissolved. Whereupon 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-toiodine weight ratio of 1.5 to 1. Store the solution in an amber bottle.

3.6.1.4 Starch Solution

Mix 1.0 to 1.5 g of starch with 5 to 10 mL of cold water to make a paste. Add an additional 25 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.

3.6.2 Procedure

3.6.2.1) The procedure applies to either powdered or granular activated carbon. When granular carbon is to be tested, grind a representative sample of carbon until 60 wt % and 95 wt % or more will pass through a 100-mesh screen Carbon received in the powdered form may need additional grinding to meet the particle size requirement given above

3.6.2.2) Dry the ground carbon and cool the dry carbon to room temperature in a desiccator.

3.6.2.3) Determination of iodine number requires an estimation of three carbon dosages. 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 then stopper the flasks.

3.6.2.4) Pipet 10.0 mL of 5 wt % hydrochloric acid solution into each flask containing carbon. Stopper each flask and dial slowly until the carbon is entirely wetted. Unloose 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 slowly for 30 s to remove any sulfur which may meddle with the test results. Remove the flasks from the hot plate and cool to room temperature.

3.6.2.5) Pipet 100.0 mL of 0.100 N iodine solution into each flask. Standardize the iodine solution just previous to use. Pour 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 s. Quickly filter each mixture by gravity through one sheet of filter paper into a beaker. Filtration equipment must be prepared in advance so no delay is encountered in filtering the samples.

3.6.2.6) For each filtrate, use the first 20 to 30 mL to rinse a pipette. 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 weak yellow. Add 2 mL of the starch solution and titration with sodium thiosulfate until one drop produces a colorless solution. Record the volume of sodium thiosulfate used.

Chapter IV

RESULTS AND DISCUSSION

4.1 Properties of lesser reedmace

Lesser reedmace was preliminarily analyzed the %Fixed carbon as shown in Table 4.1. The C, H, N composition of the lesser reedmace was measured by a C H N analyzer, the results was show in Table 4.2. Lesser reedmace flower used in this research because highest %C and %fixed carbon.

Table 4.1 %Fixed carbon of lesser reedmace

Type	Fixed carbon (%)
Flower	21.381
Leaf	20.543
Stem	19.785
Root	19.467

Table 4.2 Elemental analysis of lesser reedmace

Type	C (%)	H (%)	N (%)
Flower	45.612	5.873	1.576
Leaf	41.881	5.745	1.631
Stem	36.142	4.752	4.752
Root	34.065	4.991	1.383

4.2 Carbonization

The powder of lesser reedmace flower was carbonized in order to prepare for activation by impregnation with phosphoric acid. The studied condition was temperature of 150, 180 and 210°C. Each temperature was carried out at the time of 60, 120 and 180 min. The final products of this step are called hydrochar. The proximate analysis of hydrochar are shown in Table 4.3-4.4 and Figure 4.1-4.6

Table 4.3 Characteristics of lesser reedmace from carbonize by hydrothermal treatment at different temperatures and reaction times

		Lesser reedmaceOn dry basis				
Temperature (°C)	Time (min)	Yield (%wt)	Moisture (%wt)	Ash (%wt)	Volatile matter (%wt)	Fixed matter (%wt)
150	60	47.24	3.12	9.12	44.51	43.25
	120	46.11	2.99	9.98	39.12	47.91
	180	45.02	3.02	10.56	36.64	48.78
180	60	44.19	3.15	10.64	32.67	53.54
	120	43.88	2.97	10.65	32.98	53.40
	180	43.56	3.03	11.12	31.15	54.70
210	60	43.24	3.14	11.89	30.85	54.12
	120	42.14	2.94	12.34	29.85	54.87
	180	42.04	2.96	13.23	28.52	55.29

4.2.1 The effect of temperatures and time on %yield, %moisture, %ash, %volatile matter and %fixed carbon for carbonization by hydrothermal treatment.

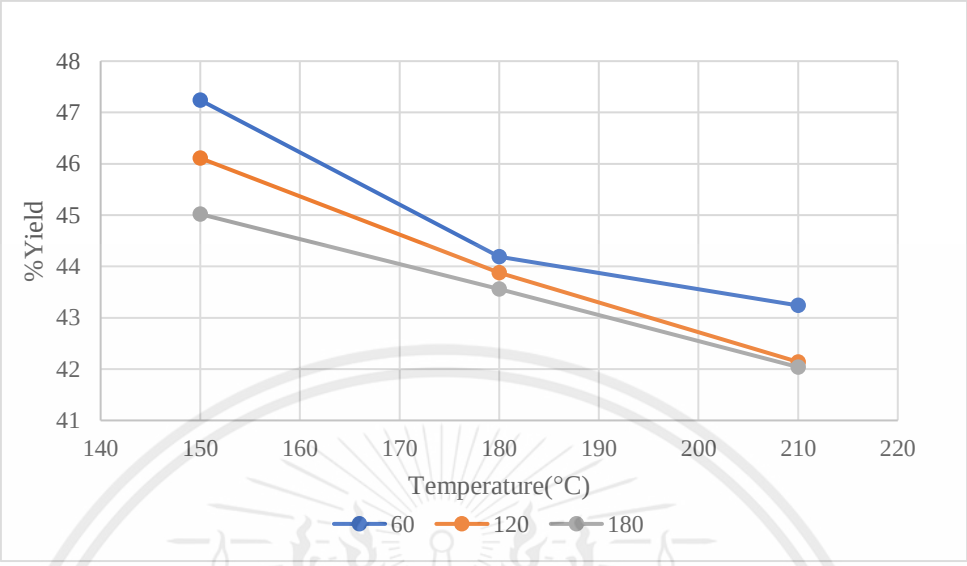

Figure 4.1 Effect of carbonization temperature and time on % yield.

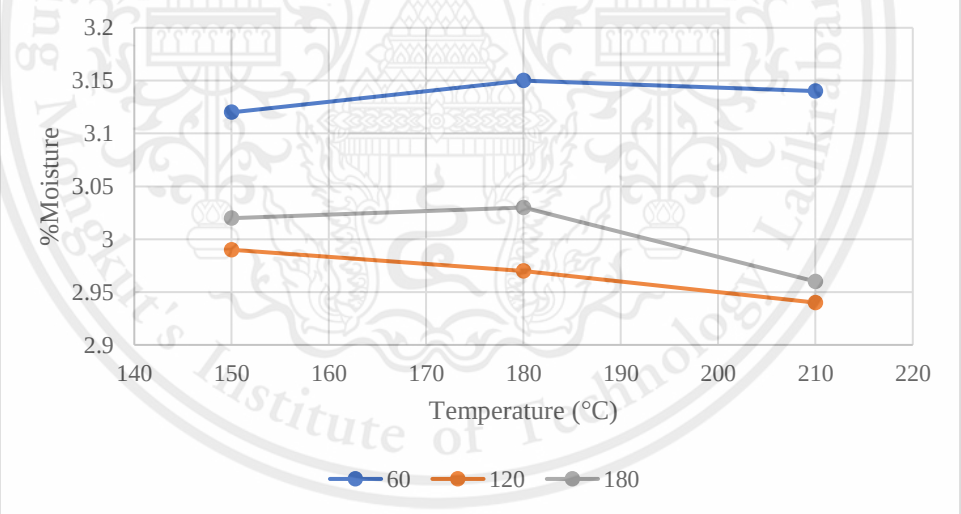

Figure 4.2 Effect of carbonization temperature and time on % Moisture.

Table 4.3 and Figure 4.1 show that the % yields of various carbonization condition decrease with the increased temperature and time, because at high temperature and increase time, the carbon in lesser reedmace is diffused out in form of volatile, so the % yield in the products decrease. Table 4.3 and Figure 4.2 show that %moisture of various condition tend decrease with the increased temperature and time because some liquid was removed in high temperature and times.

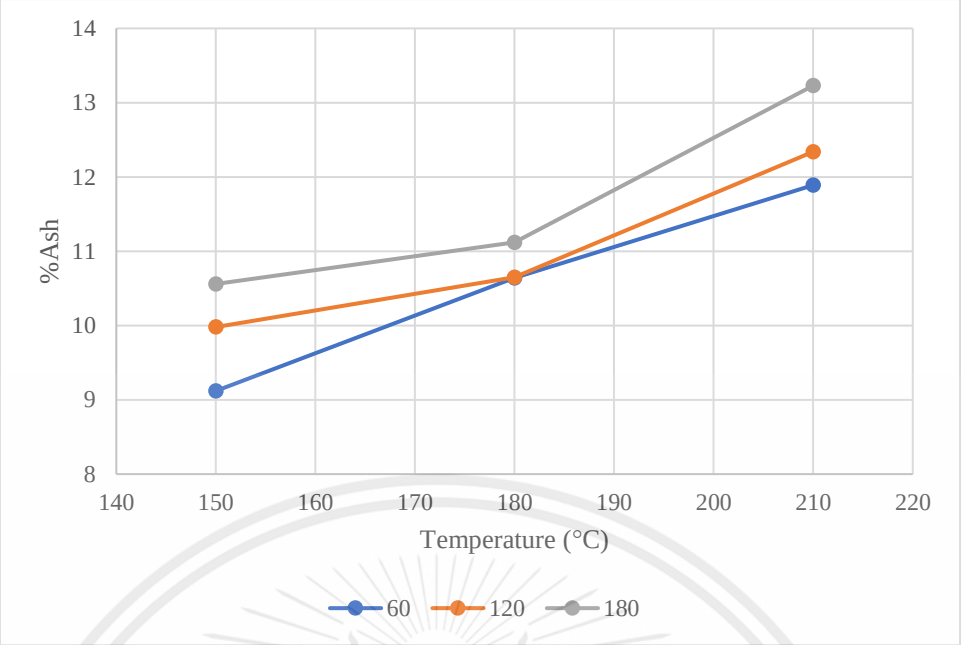

Figure 4.3 Effect of carbonization temperature and carbonization time on % Ash.

The change of % ash at 150 ˚C for 60 min is small (9.12%) shown in Table 4.3 and Figure 4.3, because at low temperature and reaction time, only some the volatile matter was removed. So, the other components remained in the residue was still high and resulted the lower % ash. When the increased temperature to 210 ˚C, % ash increase from 9.12% to 11.89%, because of some hydrocarbon react to itself and change into ash.

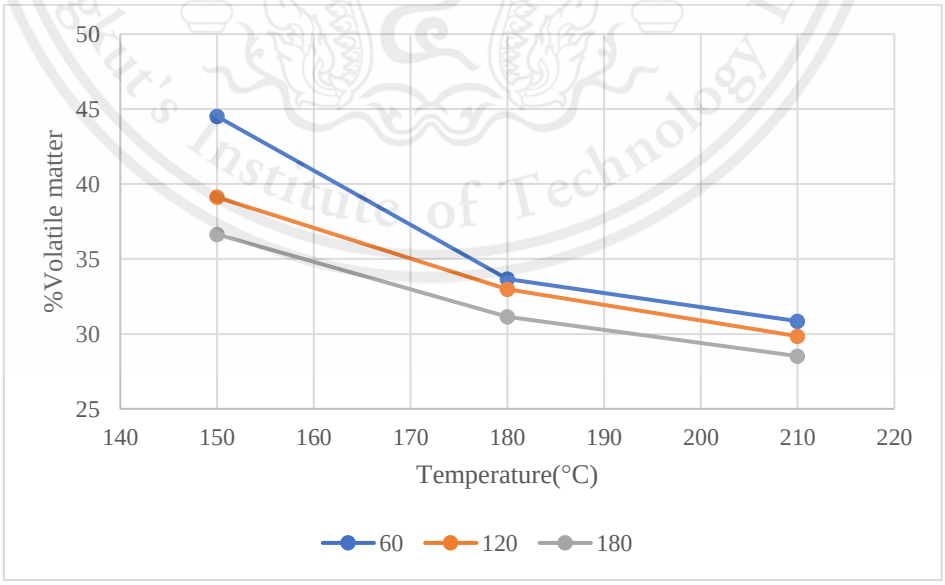

Figure 4.4 Effect of carbonization temperature and carbonization time on % Volatile matter.

Table 4.3 and Figure 4.4 show that the % volatile matter of various carbonization condition from 150-210 ˚C at 60 min decrease largely from 44.51% to 28.52%, because at low temperature, only the volatile is removed and the carbon in lesser reedmace was diffused out in the form of volatile. When increased temperature to 210 ˚C, % volatile decreased to 28.52%, because some hydrocarbon react extremely and change in ash.

The decrease of volatile matter in the time of 60 min is faster than at 120 and 180 min because in the first times range, only volatile at the surface was removed. When time increase above 120 min, volatile in the interior particle would be removed, but with more difficulty than volatile at the surface.

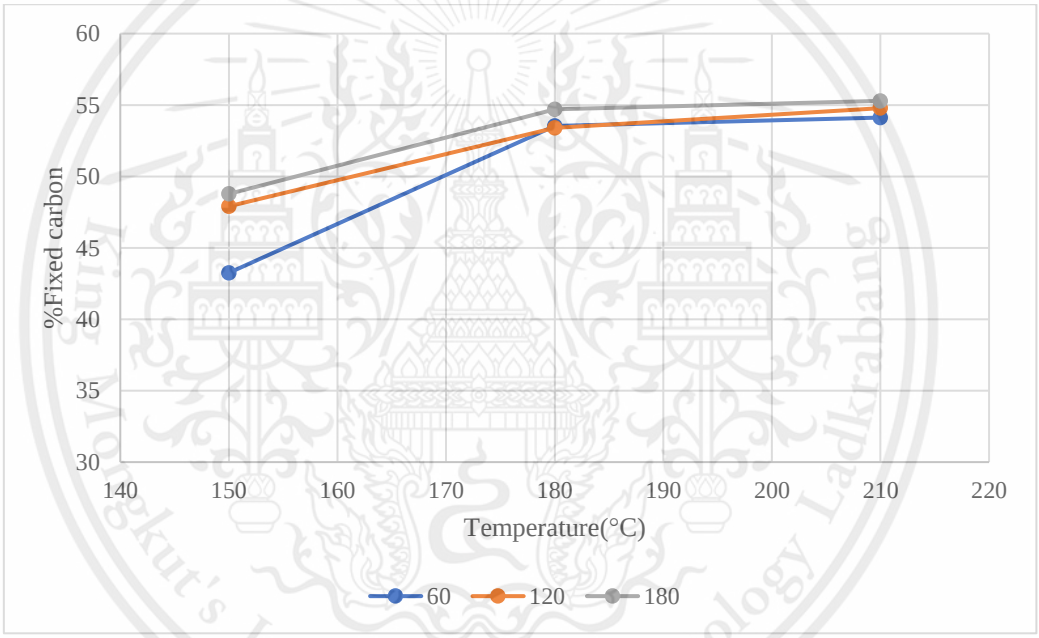

Figure 4.5 Effect of carbonization temperature and carbonization time on % Fixed carbon.

Table 4.3 and Figure 4.5, show that the % fixed carbon increase largely from 43.25% to 54.12% at 60 min. The carbon in lesser reedmace was diffused out in the form of volatile and change finally into ash. The increase of %fixed carbon in the time at 60 min is faster than 120 and 180 min because in the first time, only volatile at the surface is removed. When time increase higher, carbon in the interior particle would be remove and the last changed to ash.

Table 4.4 Iodine number after carbonization by hydrothermal treatment.

No.	Temperature (°C)	Time (min)	IA (mg/g)
1	150	60	778.4892
2		120	799.4925
3		180	797.9849
4	180	60	808.578
5		120	829.2456
6		180	816.1008
7	210	60	810.2701
8		120	873.1693
9		180	757.1363

4.2.2 The effect of temperature and times on Iodine number after carbonization by hydrothermal treatment.

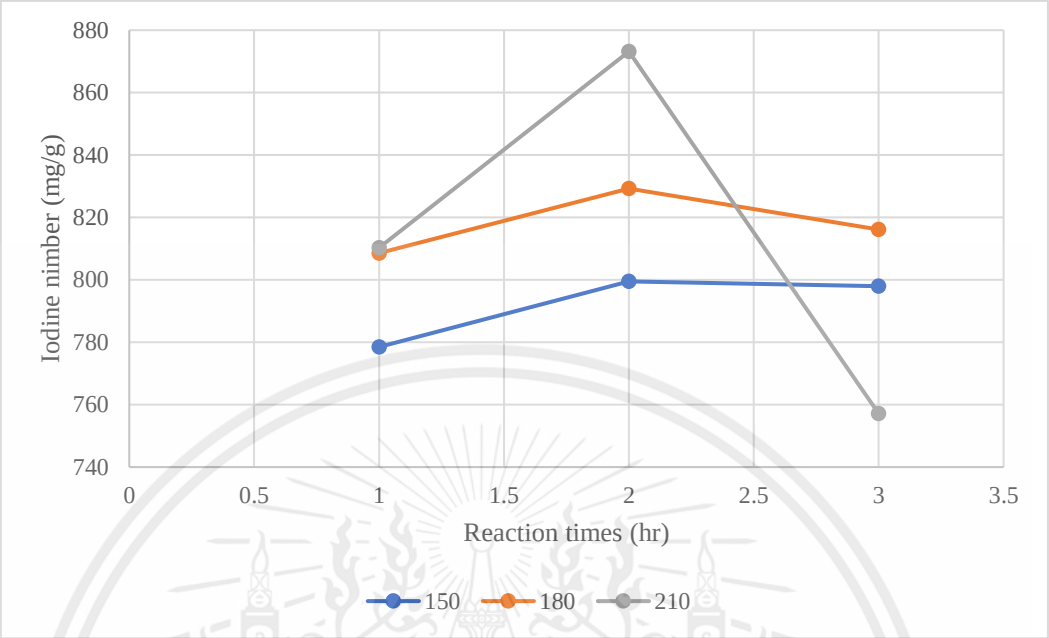

Figure 4.6 Effect of carbonization temperature and carbonization times on Iodine number after carbonization by hydrothermal treatment.

Figure 4.6 showed the effect of temperature and times on iodine number. At the temperature was 210°C, when reaction times increase from 2 hr to 3 hr, hydrocarbon in lesser reedmace was diffuse out in the form of volatile matter and then change to ash, the micropores would be broken to mesopores and macropores. While the condition of hydrothermal treatment was 210°C and 2 hr, iodine adsorption capacity increase largely because it seems that the superheated steam diffuse in to pore and produce the minimize pore size, therefore a number of pores were produced at this temperature and times. The experimented results show that the optimum temperature and time is 210°C and 2 hr since it has maximum iodine adsorption and total surface area. Thus choose 4 best one for activation by impregnated phosphoric acid.

4.3 Activation

The hydrochar from the hydrothermal treatment at 150°C for 120 min, 180°C for 120 min, 180°C for 180 min and 210°C for 120 min was prepared for being the raw material to produce activated carbon. It was crushed and sieved to particle sizes less than 150 µm. The hydrochar was activated by impregnated with phosphoric acid

Table 4.5 Iodine number after activation by impregnated with phosphoric acid.

No.	Carbonization Temperature (°C)	Carbonization Time (min)	Impregnation Time (hrs.)	Iodine number (mg/g)
1	150	120	6	830.145
			12	849.126
			18	841.661
2	180	120	6	868.758
			12	913.892
			18	910.068
3	180	180	6	895.661
			12	920.746
			18	917.106
4	210	120	6	1100.225
			12	1118.087
			18	1087.051

4.3.1 The effect of impregnation times on Iodine number after activation by impregnated with phosphoric acid.

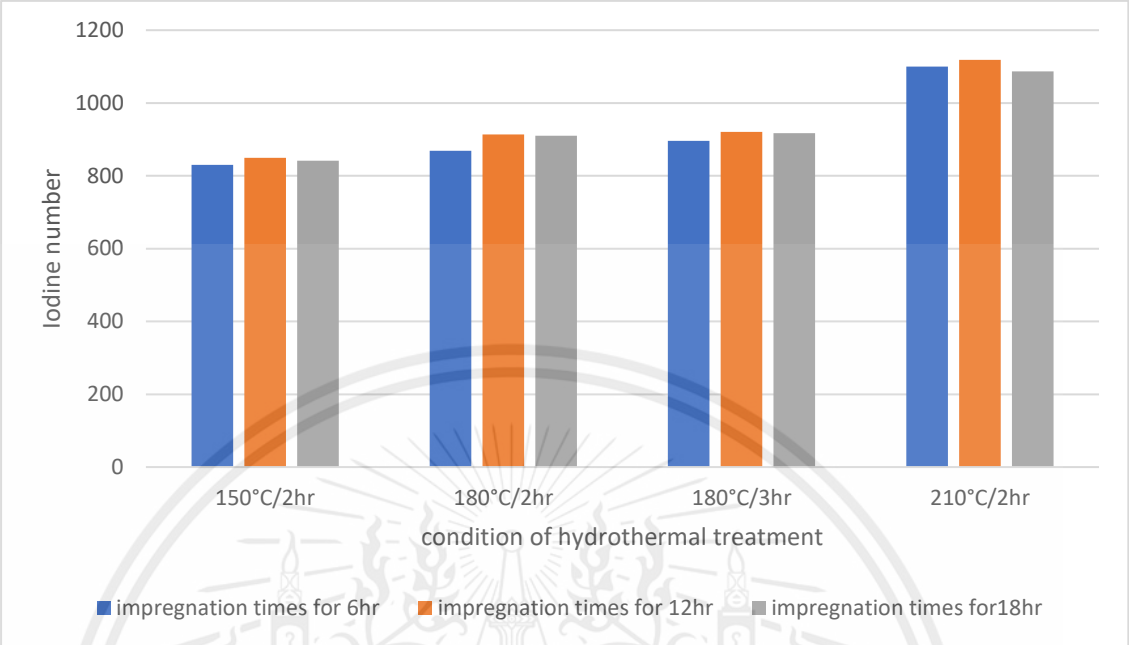

Figure 4.7 Effect of impregnation times on Iodine number after activation by impregnated with phosphoric acid.

Figure 4.7 shown that the optimum impregnation times was 12 hr, at condition of carbonization by hydrothermal treatment at 210°C/2hr and impregnation times was 12 hr, iodine number increased from 873.1693 mg/g to 1118.087 mg/g. Impregnation times increased, iodine absorption capacity decreased dramatically because small pore structure was destroyed, resulting in larger pores. As a result, the ability to absorb iodine decreased. So the high suitable impregnation time (impregnation times for 18 hr) were unfavorable for the porous structure of the char. The porous structure cracked, and the pores might be partially blocked as a result of the softening and melting of the material constituents, leading to a poor surface property.

Chapter V

CONCLUSION AND RECOMENTATION

The experimental result showed the possibility to produce the activated carbon from lesser reedmace flower. In this research, carbonization by hydrothermal treatment and activation were proposed to produce activated carbon from lesser reedmace flower. In the carbonization step, the studied variables were temperature and time at the range of 150-210°C and 60-180 min respectively. The optimum condition of this step was 210°C and reaction time of 120 minutes with 42.14% yield, 2.94% moisture, 12.34% ash, 29.85% volatile matter, 54.84% fixed carbon and iodine number 873.1693 mg/g.

In the activation step,2 method was used. Activation by impregnation phosphoric acid were impregnated ration 5:1 (phosphoric acid: char) at room temperature. Studied variables in this process were impregnation time of 6, 12 and 18 hr. From the results of Activation by impregnated phosphoric acid, it was found that the suitable impregnation time was 12 hr, iodine number increase from 873.169 mg/g to 1118.087 mg/g.

It can be concluded that lesser reedmace flower, which most people think that it is useless, was converted to activated carbon and can be used in adsorption application. It was also proved that carbonization by hydrothermal treatment and activation by impregnation phosphoric acid is suitable.

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Appendix

Appendix A

Characteristics of lesser reedmace flower

Appendix A1

Table A1 %Element of lesser reedmace by CHNS method

Sample	%Element			
	Nitrogen	**Carbon**	**Hydrogen**	**Sulphur**
Stem	0.9138	36.1418	4.7521	0
Root	1.3831	34.0651	4.9908	0
Leave	1.6315	41.8812	5.7449	0
Flower	1.5759	45.6121	5.8735	0

Appendix A2 TGA analysis of flower of lesser reedmace.

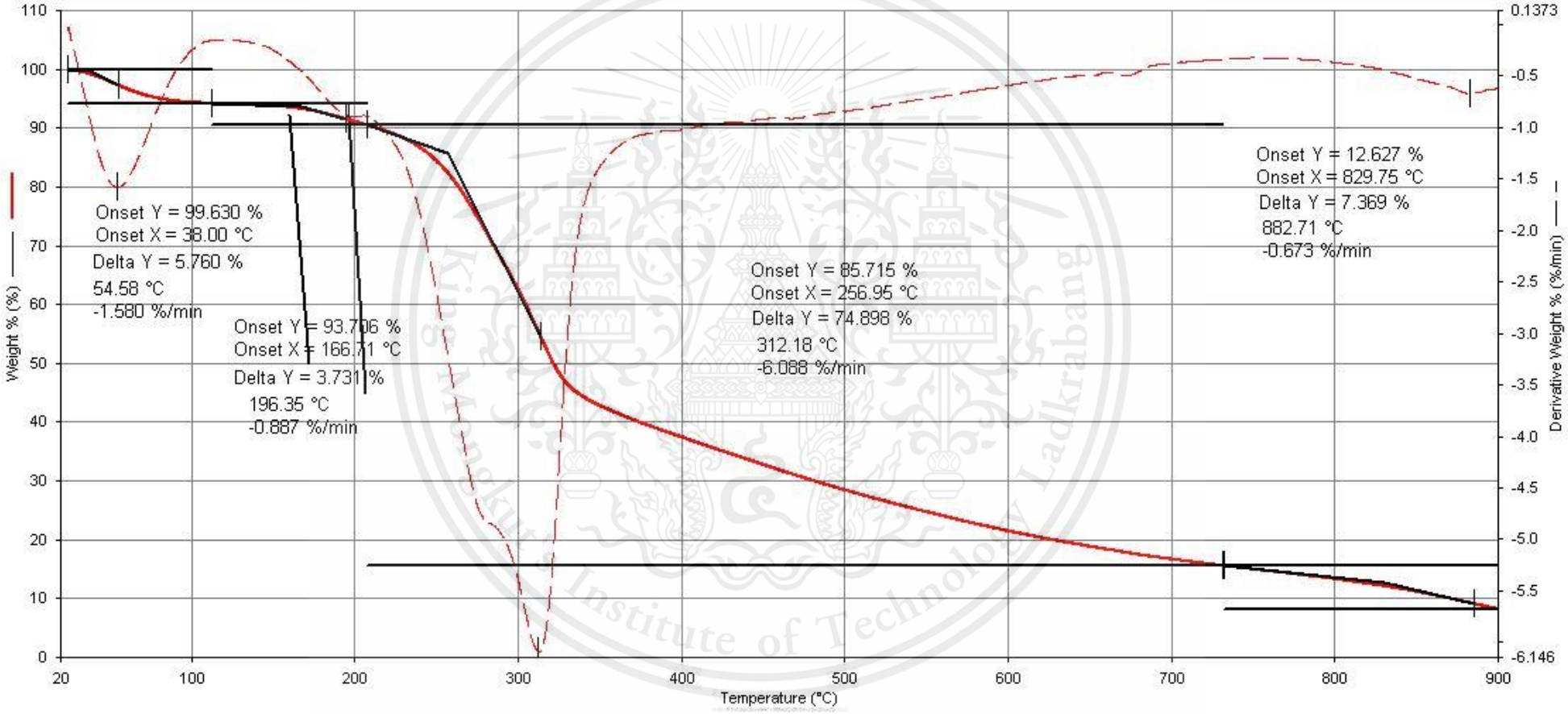

Figure A2. TGA analysis of flower of lesser reedmace.

Appendix A3 FTIR spectra of the lesser reedmace

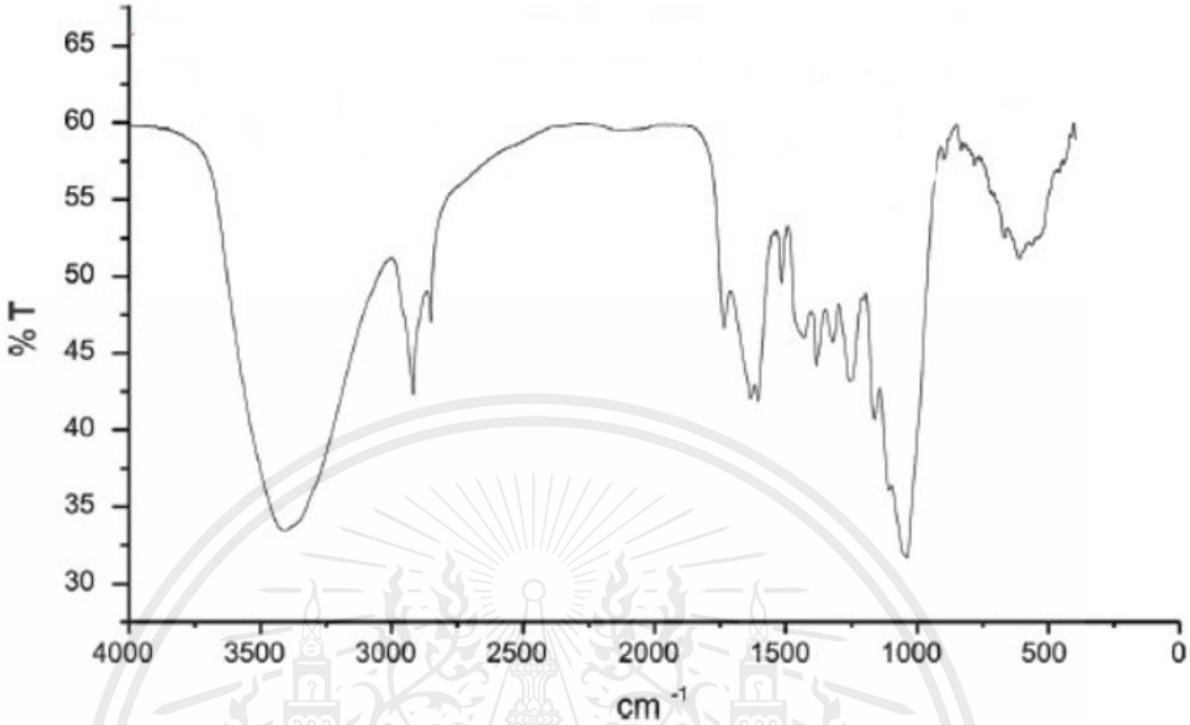

Figure A3. FTIR spectra of the lesser reedmace

In order to examine the mechanism of adsorption process on flower of lesser reedmace, different functional groups found in the sorbent material are the key factors. To confirm the functional groups involved in pesticides uptake in the current adsorbent, the fresh and pesticide loaded adsorbent were mixed with spectroscopic grade KBr and made in the form of pellet. The pellet formed was scanned in the spectral range of 4000-100 cm^{-1}. The FT-IR spectra, Figure A3, of flower of lesser reedmace pesticides uptake show a number of different absorption peaks, indicating the complex nature of adsorbent. As can be seen from the FT-IR spectra, the vibration bands of the adsorbent are due to the presence of hydrogen bonded OH stretch (alcohol) at 3410.5 cm^{-1}, alkyl C-H stretching at 2918.5 cm^{-1} and alkenyl C-H stretch at 2850.5 cm^{-1}. In addition, the absorption peaks at 1735, 1606 and 1039.5 cm^{-1} are due to C=O, C=C and C-O functionalities, respectively. As shown in Figure A3, the major absorption peaks of the adsorbent including fingerprint region, after pesticides uptake have undergone a change in their absorption bands intensities and frequencies confirming the participation of these functional groups in the adsorption of pesticides.

Appendix B

Standard test method for chemical analysis of hydrochar and

Iodine number calculation

Appendix B.1

Standard test method for chemical analysis of hydrochar (ASTM D 1762-84)

This test method covers the determination of moisture, volatile matter, and ash in charcoal made from lesser reedmace flkower. The sample is ground in a specified manner and the moisture determined as loss in a drying oven at 105°C. Volatile matter is determined as the residue after burning to constant weight 750°C.

Procedure (for Activated carbon passing A No.150 Sieve)

1) Make duplicate determinations.

2) ***Moisture*** Heat the muffle furnace to 750°C and place previously ignited porcelain crucible and covers in the furnace for 10 min. Cool the crucibles in a desiccator for 1 hr. Weight the crucibles and add to each approximately 1 g, weighed to the nearest 0.1 mg, of the ground sample. Place the samples in the oven at 105°C for 2 hr. Place the dried samples in a desiccator for 1 hr and weight.

3) ***Volatile matter*** Heat the muffle furnace to 950°C. Preheat the crucibles used for the moisture determination, with lids in place and containing the sample, as follows: with the furnace door oven ,for 2 min on the outer ledge of the furnace (300°C) and then for 3 min on the edge of the furnace (500°C). Then move the samples to the rare of the furnace for 6 min with the muffle door closed. Watch the samples through a small peep-hole in the muffle door. If sparking occurs, results will be in error. Cool the samples in a desiccator for 1 hr and weigh.

4) ***Ash*** Place the lids and the uncovered crucible used for the matter determination, and containing the sample in the muffle furnace at 750°C for 6 hr. Cool the crucibles with lids in place in a desiccator for 1 hr and weight. Repeat burning of the sample until a fully 1 hr. period of heating results in a loss of less than 0.0005 g.

Calculation

1.Calculate the percentage of moisture in the sample as follows:

Calculated the moisture content as follows:

$$\text{Moisture, \%} = [(A-B) / A] \times 100$$

where:

A = grams of air-dry sample used, and

B = grams of sample after drying at 105°C.

2. Calculate the percentage of volatile matter content in the sample as follows:

$$\text{Volatile matter, \%} = [(B-C) / B] \times 100$$

where:

C = grams of sample after drying at 950°C.

3.Calculate the percentage of ash in the sample as follows:

$$\text{Ash, \%} = (D / B) \times 100$$

where

D = gram of residue.

Appendix B.2

Iodine number calculation

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.

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

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

$$A = (N_2)\ (12693.0)$$

Where : N2 = iodine, N

$$B = (N_1)\ (126.93)$$

Where : N1 = sodium thiosulfate, N

$$DF = (I + H) / F$$

Where : DF = dilution factor,

I = iodine, mL

H = 5 % hydrochloric acid used, mL, and F = filtrate, ml.

F = filtrate, ml.

3) Calculate the value of X/M as follows:

$$X/M = [A – (DF) (B) (S)] / M$$

Where : X/M = iodine absorbed per gram of carbon, mg/g,

S = sodium thiosulfate, mL, and

M = carbon used, g.

4) Calculate the value of C as follows:

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

Where: C = residual filtrate, N,

N_1 = sodium thiosulfate, N, and

F = filtrate, ml.

5) Using logarithmic paper, plot X/M (as the ordinate) versus C (as the abscissa) for each of the three carbon dosages. 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:

$$M = [A – (DF) (C) (126.93) (50)] / E$$

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.

Appendix C

Properties of commercial activated carbons

Appendix C

Table C.1 Properties of commercial activated carbons limit by TIS 900-1989

Types	Size	%Moisture	BD (cm^3/g)	IA (mg)
Powdered activated carbon	<150µm	-	0.20.0.75	>600
Granular activated carbon	>150µm	<8	>0.36	>600

Table C.2 Properties of commercial powdered activated carbon

Trade names	Raw material	Ash (%)	BD (g/cm^3)	S_{BET} (m^2/g)	MB (mg/g)	IA (mg/g)
UDP CHEMICAL (Thailand)						
MDP 1-7325	Coconut shell	3	0.40-0.55	1200-1300	230	1050
PN3	-	8	0.46	-	-	800
Taiko PKW	Sawdust	2	-	-	-	-
Shirasagi	Sawdust	3	-	-	-	-
KW	-	3-5	0.37-0.40	-	-	900
Nuchar SA	-	3-5	0.37-0.40	-	-	900
PITTSBURGH ACTIVATED CARBONS						
RB	Bituminous	23	-	1250-1400	-	1200
RC	Bituminous	21	-	1100-1300	-	1100
BL	Bituminous	8.5	-	1000-1100	300	1000
C	Bituminous	14	-	1000-1100	-	900
GW	Bituminous	12	-	1000-1100	180	700