PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON DERIVED FROM RUBBER WOOD

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I declare that this dissertation is of my own research except for quotations and summary that have been cited in references. This dissertation has not been accepted for any degree and is not concurrently submitted in candidature of any degree.

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ABSTRACT

This project aims to prepare activated carbon (AC) from rubber wood (*Hevea brasiliensis*) activated with potassium hydroxide (KOH), followed by the physical and chemical characterization of the AC produced. Rubber wood impregnated with varying KOH concentrations was heated in a two-stage activation method. Rubber wood with higher KOH concentration produced less yields as KOH enhances pyrolytic decomposition. The pH test showed that the ACs prepared are acidic after reflux. Moisture content increases with increasing KOH concentration, averaging 4.71% whereas ash is independent of KOH concentration and averages 4.69%. Flame atomic absorption spectrophotometric analysis revealed the presence of Fe and Pb but the absence of Cd. Both methylene blue adsorption and iodine tests showed the AC adsorptive capacity increases with KOH concentration until 20% KOH corresponding to methylene blue adsorbed of 40mg/g and iodine number of 72.39mg/g before decreasing. Scanning electron microscopy analysis confirmed development of more refined pores with larger internal surface area as compared to untreated rubber wood.

ABSTRAK

Projek ini bertujuan menyediakan karbon teraktif (AC) daripada kayu pokok getah (*Hevea brasiliensis*) yang diaktifkan kalium hidroksida (KOH), diikuti pencirian AC yang terhasil. Kayu pokok getah yang diimpregnasi dengan kepekatan KOH yang berbeza dihasilkan secara pengaktifan dua peringkat. Sampel kayu pokok getah memberikan hasil yang semakin berkurang dengan bertambahnya kepekatan KOH kerana KOH menggalakkan penguraian pirolitik. Ujikaji pH menunjukkan AC yang diperoleh bersifat asid selepas proses refluks. Kandungan kelembapan meningkat dengan kepekatan KOH dengan nilai purata 4.71% bagi semua sampel AC. Kandungan abu tidak bergantung kepada kepekatan KOH dan memberikan nilai purata 4.69% bagi semua sampel AC. Analisis serapan spektrofotometri nyalaan menunjukkan kehadiran unsur Fe dan Pb dalam semua sampel kecuali unsur Cd. Kedua-dua ujikaji penjerapan metilena biru dan nombor iodin menunjukkan peningkatan kebolehan penjerapan dengan meningkatnya kepekatan KOH sehingga 20% KOH, bersamaan dengan metilena biru terjerap 40mg/g dan nombor iodin 72.39mg/g, sebelum kebolehan penjerapan menyusut. Analisis mikroskop elektron pengimbas menunjukkan pembentukan liang dengan luas permukaan lebih besar berbanding sampel kayu pokok getah yang tidak dirawat.

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KOH	Potassium hydroxide
$Na_2S_2O_3$	Sodium thiosulphate
NH_3	Ammonia
H_2SO_4	Sulfuric acid
K_2S	Potassium sulfide
H_3PO_4	Phosphoric acid
SEM	Scanning Electron Microscope
ΔH	Enthalpy change
w/v	Weight over volume
M_x	Concentration of x
V_x	Volume of x

CHAPTER 1

INTRODUCTION

1.1 Project Background

Malaysia has been one of the main rubber producers and exporters in the Asian region in recent decades. As the demand for natural rubber started to decrease and Malaysian rubber plantations began to shift their activities towards other forms of agriculture commodities, rubber trees from these rubber plantations are utilized for many other purposes, some of which are furniture-making, construction and pulp processing. This project currently undertakes the task to utilize rubber wood as a precursor for activated carbon. The preparation of activated carbon from rubber wood is one of the ways of adding values to rubber wood. This project will also contribute towards the on-going research of activated carbon by chemically activating the rubber wood, which is a form of lignocellulosic, with potassium hydroxide (KOH) during the preparation of activated carbon. The justification of using KOH as the activating agent is that there are scarce researches on the preparation of activated carbon from lignocellulosic activated with KOH and it leads to optimal textural and chemical properties of the carbon (Marsh and Yan, 1984; Ahmadpour and Do, 1996). The most common chemical activating agents used in the production of activated carbon from lignocellulosics are zinc chloride, phosphoric acid, aluminium chloride and boric acid

(Huidoboro et al, 2001).

1.2 Objectives

This research attempts to utilize rubber wood as a source of activated carbon for the purpose of preparation and characterization. This dissertation addresses two main objectives:

1.2.1 To produce activated carbon from rubber wood (*Hevea brasiliensis*) by a two-stage activation process with potassium hydroxide (KOH) as the activating agent.

1.2.2 To physically and chemically characterize the activated carbon prepared.

1.3 Scope of Research

This research will be using rubber wood (*Hevea brasiliensis*) as the precursor of activated carbon, in which the activated carbon will be prepared through a two-step activation process in a self-generated atmosphere. Potassium hydroxide (KOH) is the activating agent used in the impregnation of rubber wood samples. The concentration of KOH solution will be varied for the impregnation of rubber wood. Therefore, the main parameter to be studied is the impregnation effect of different KOH concentrations on the activated carbon prepared.

CHAPTER 2

LITERATURE REVIEW

2.1 Activated Carbon

Activated carbon includes a wide range of amorphous carbon-based materials prepared to exhibit a high degree of porosity and an extended interparticulate surface area (Bansal *et al*, 1988). These qualities impart activated carbon with excellent adsorbent characteristics that make carbon very useful for a wide variety of processes, including filtration, purification, deodorization, decolourization, purification and separation of chemicals from gases and liquids.

Activated carbons are extremely versatile adsorbents of industrial significance and are used in a wide range of applications which are concerned principally with the removal of undesired species by adsorption from liquids or gases, in order to effect purification or the recovery of chemical constituents. They also find use as catalysts or catalyst supports (Patrick, 1995; Jin *et al,* 1996; Mochida *et al,* 1997). The strong market position held by the activated carbon adsorbents relates to their unique properties and low cost compared with that of possible competitive inorganic and shape is usually obtained in sharp contrast to almost constant pore size in zeolites. This makes activated carbons more versatile adsorbing materials. Porous adsorbing carbons are associated with new processes for development of these materials, mechanisms, material characterization and newer applications. More than 30% of the carbon research over the world revolves around activated carbons; newer precursors, methods of activation and applications (Manocha, 2003). A schematic model of activated carbon is illustrated in Figure 2.1.

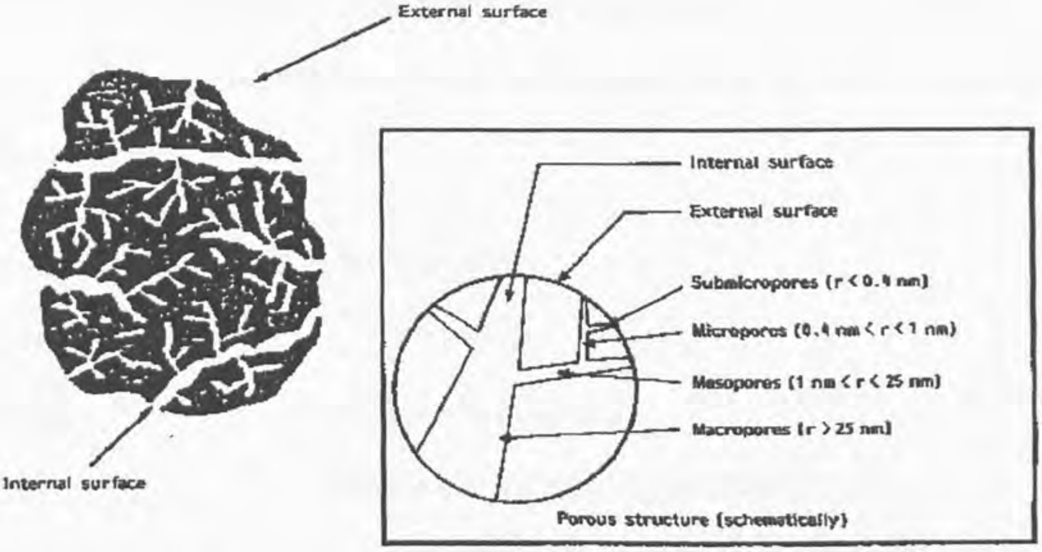


Figure 2.1 Schematic model of activated carbon (Henning and Schäfer, 2004).

For comparison, a given type or sample of activated carbon is usually quantified based on four primary criteria: *total surface area*, *carbon density*, *particle size distribution*, and *adsorptive capacity*. All of these factors influence adsorption rate and capacity. *Adsorptive capacity* is characterized by the effectiveness of activated carbon in removing a given contaminant. For comparison, several standard compounds are used for these measurements. For example, the commonly-used 'iodine number' describes the carbon's capacity to adsorb low-molecular-weight substances, while the 'molasses number' characterizes a carbon's capacity for more complex compounds (Manocha, 2003).

2.2 Pore Sizes

The size of individual pores can vary greatly in size and shape for different adsorbents and even within the same adsorbent. Pores are usually characterized in terms of their width, meaning the diameter of a cylindrical pore or the distance between two sizes of a slit-shaped pore. Dubinin (1960) proposed a classification of pores presented in Table 2.1 which was later adopted by the International Union of Pure and Applied Chemistry (IUPAC, 1972).

Table 2.1 Pore Classifications by Pore Width

Pore Classification	Pore Width
Micropore	less than ~20 Å (2 nm)
Mesopore	between ~20 and 500 Å (2 and 50 nm)
Macropore	more than 500 Å (50 nm)

The basis for the pore classifications presented in Table 2.1 is that each size range corresponds to different adsorption effects, as observed in an adsorption isotherm. The interaction potential in micropores is much greater than that in larger pores due to the closeness of the pore walls, resulting in an enhanced adsorption potential. An adsorbate molecule within a micropore is held there by adsorption forces originating from approximately ten nearest surface atoms. The forces on adsorbate molecules are a function of distance between adsorbate and adsorbent atoms (pore size) and polarity (permanent or induced) of the adsorbate and adsorbent atoms (Marsh, 1987). Capillary condensation takes place within mesopores, resulting in a hysteresis loop in the adsorption isotherm. The pores are so wide in the macropore range that it is nearly impossible to map out the isotherm in detail because the relative pressures of the adsorbate (P/P_0) would be so close to unity. Mercury is typically used to examine macropore structure, due to its low vapor pressure. Figure 2.2 shows the relative structure of macropore and micropore within an impregnated activated carbon.

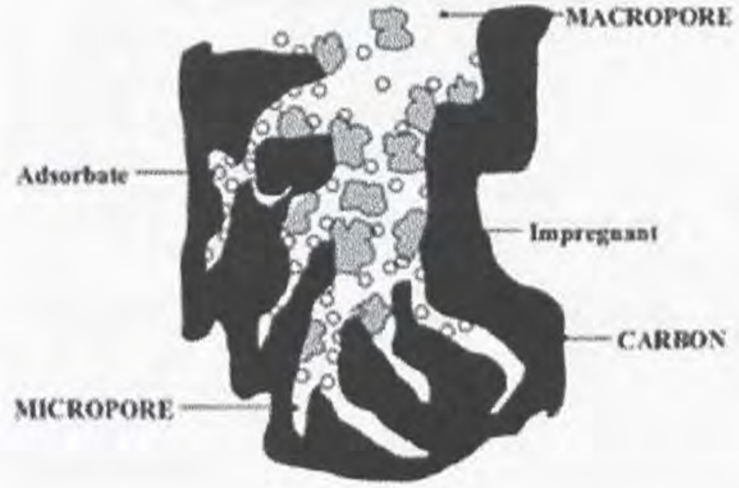
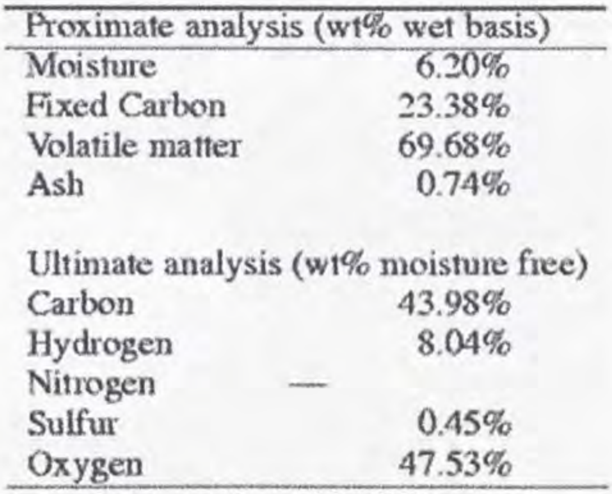


Figure 2.2 Structure of macropore, micropore within an impregnated activated carbon (http://buildingprotection.sbccom.army.mil/index.htm).

2.3 Rubber Wood

Rubber wood (*Hevea brasiliensis*) is one of the main plantation crops in South East Asia with an estimated plantation area of 1.82 million hectares in Malaysia alone which accounts for 20% of global plantation. The rubber trees are cut after their latex yielding period of around 25 years and the wood would be utilized for many downstream processing. It is estimated that a gross yield of rubber wood per hectare would be around 180m^3 (Hong & Sim., 1994) and in the process of converting raw logs into the sawn timber the yield is estimated to be only around 20% (Hong & Sim., 1994) generating large amounts of residual biomass. Table 2.2 summarizes the proximate and ultimate analysis of rubber wood sawdust.

Table 2.2 Proximate and ultimate analysis of rubber wood sawdust



Proximate analysis (wt% wet basis)	
Moisture	6.20%
Fixed Carbon	23.38%
Volatile matter	69.68%
Ash	0.74%
Ultimate analysis (wt% moisture free)	
Carbon	43.98%
Hydrogen	8.04%
Nitrogen	—
Sulfur	0.45%
Oxygen	47.53%

*Oxygen estimated by difference

Source: Srinivasakannan & Mohd. Zailani, 2004.

2.3.1 Rubber Wood Properties

Fresh, sawn rubber wood (as shown in Photo 2.1) is white to creamy in colour, sometimes with a pinkish tinge, and has a fairly straight grain. It turns yellowish after seasoning. Heartwood and sapwood are not distinguishable. Pores are large and scattered and show radially and tangentially as brown lines.

The air-dry density ranges from 560 to 650 kg per cubic meter, depending on clone, age, site and plantation management (Killmann & Hong, 2000). Fresh rubber wood has an initial moisture content of 60 to 80 percent (Killmann, 1992). The timber tends to develop seasoning defects such as cupping, twisting, bowing and checking, particularly in heartwood from the centre of the stem. A brown discoloration of the timber is sometimes observed after kiln seasoning.

Photo 2.1 Fresh rubber wood has a creamy homogeneous colour; heartwood and sapwood are not distinguishable (Killmann, W.)

The timber has good overall qualities for woodworking and machining - for sawing, boring, turning, nailing and gluing. However, remaining latex can clog the saw teeth, and they have to be cleaned frequently. Tension wood can lead to fuzzy grain when machined. Rubber wood is easy to mould with feed speeds above 20 m per minute (Gloeckner, 1990; Rumboll, 1990). Fingerjointing is often applied to achieve larger dimensions. Rubber wood can be steam-bent with good results. When properly sanded it takes finishes well, and it can easily be stained to resemble walnut, cherry, oak or other woods, depending on consumer demand. In strength and mechanical properties it is comparable to traditional timbers used for furniture making and woodworking. Table 2.3 illustrates the physical and mechanical properties of rubber wood.

TABLE 2.3 Physical and mechanical properties of rubber wood.

Property	Rubber wood (at 15% moisture content)
Density	460-650 kg/m^3
Modulus of rupture (MOR)	66 N/mm^2
Modulus of elasticity (MOE)	9 240 N/mm^2
Compression parallel to grain	32 N/mm^2
Compression perpendicular to grain	5 N/mm^2
Shear	11 N/mm^2
Hardness (Janka)	4 350 N

Source: Lee *et al,* (1982), rubber wood; Soerianegara and Lehmmens (1993).

Fresh rubber wood contains 1.0-2.3% free sugars and 7.5-10.2% starch. Because of the relatively high content of free sugars and the absence of extractives, the wood is non-durable and is easily attacked by fungi and insects. The content of free carbohydrates has also created other problems, e.g. for the setting of cement in cement-bonded panels manufactured in Malaysia. This problem was resolved by open-air storage of the chips, which reduced the sugar and starch to 0.2 and 1 %, respectively (Killmann, 1992).

2.4 Processing of Activated Carbon

Activated carbons are manufactured by the pyrolysis of carbonaceous materials of vegetable origin, such as wood, coal, peat, fruit stones, and shells or synthetic polymers such as viscose rayon, polyacrylonitrile (PAN) or phenolics or hard coal, followed by activation of the chars obtained from them. The pyrolysis of any carbonaceous material in absence of air involves decomposition of organic molecules, evolution of tarry and gaseous products, and finally in a solid porous carbon mass. The porous carbons obtained so contain predominantly macropores and practically inactive materials with specific surface area of the order of several square meters per gram (Bansal, 1988). An adsorbent with a highly developed porosity and a correspondingly large surface area is obtained only by activating the carbonized material either by physical or chemical activation. The processing of activated carbon basically involves selection of raw material, carbonization and activation (Manocha, 2003).

2.4.1 Raw Materials

For the selection of an appropriate raw material for preparation of porous carbon, several factors are taken into consideration. Industrially, inexpensive material with high carbon and low inorganic (i.e. low ash) content is preferred as raw material for the production of activated carbon. High density of the precursor and sufficient volatile content are of considerable importance. Evolution of volatiles during pyrolysis results in porous char, essential for making activated carbons, while high density contributes to enhanced structural strength of the carbon, essential to withstand excessive particle crumble during use (Manocha, 2003).

Raw materials used for preparation of activated carbons vary with their application. Conventional raw materials in order of their importance as porous carbon production capacity, characteristics and market are: wood, coal, lignite, coconut shell, peat and others (Manocha, 2003).

Amongst woods, pine is by far the precursor for the largest production of activated carbon and forms about 50% of precursor, while all other woods contribute about 40% (Manocha, 2003). Biological wastes and woods have also been investigated and have been found to be promising precursors for porous carbons.

In the last two decades, extensive studies have been made on the selection and pyrolysis behaviour of new synthetic precursors to develop active carbons with very high adsorption capacities and controlled pore size distributions for specific energy applications. These precursors are polymer-based fibrous materials. The examples are viscose rayon, polyacrylonitrile, saran, phenolic, PFA etc. (Patrick, 1995; Jin *et al*, 1996; Mays, 1999; Inagaki, 2000).

2.4.2 Carbonization

During carbonization, most of the non-carbon elements, hydrogen and oxygen are first removed in gaseous form by pyrolytic decomposition of the starting materials, and the free atoms of elementary carbon are grouped into organized crystallographic formations known as elementary graphite crystallites. The mutual arrangement of the crystallite is irregular, so that free interstices exist between them. Thus carbonization involves thermal decomposition of carbonaceous material, eliminating non-carbon species producing a fixed carbon mass and rudimentary pore structure (Derbyshier *et al*, 1995). The process is usually carried out at temperature below 800°C in a continuous stream of an inert atmosphere.

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