P reparation and Characteristics of Activated Carbons from Coffee Residue by Chemical Activation Method
การเตรียมและศึกษาคุณสมบัติของถ่านกัมมันต์ จากกากกาแฟโดยวิธีการกระตุ้นทางเคมี

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บทคัดย่อ*

กากกาแฟซึ่งเป็นสารชีวมวลชนิดหนึ่งสามารถนำมาใช้เป็นวัตถุดิบที่สำคัญในการเตรียมถ่านกัมมันต์
ด้วยวิธีการกระตุ้นทางเคมีได้ การเตรียมถ่านกัมมันต์จากกากกาแฟเตรียมได้ 2 ขั้นตอน ขั้นแรก คือ
กระบวนการคาร์บอไนเซชันตามด้วยกระบวนการกระตุ้นทางเคมี งานวิจัยนี้ได้ศึกษาปัจจัยที่มีผลต่อ
สภาวะการกระตุ้น เพื่อหาสภาวะที่เหมาะสมในการเตรียมถ่านกัมมันต์จากกากกาแฟ ซึ่งได้แก่ ปัจจัย
ดังต่อไปนี้ ชนิดของสารกระตุ้น อัตราส่วนระหว่างสารกระตุ้นต่อวัตถุดิบ อุณหภูมิ และเวลาที่ใช้ใน
การกระตุ้น ถ่านกัมมันต์ที่เตรียมขึ้นจะถูกนำไปตรวจสอบสมบัติทางกายภาพ ซึ่งสามารถทำได้โดย
วิเคราะห์การดูดซับที่สภาวะแก๊สและของเหลว ในการทดสอบการดูดซับที่สถานะแก๊ส ทำโดยนำ
ถ่านกัมมันต์ที่เตรียมขึ้นไปวัดพื้นที่ผิว ปริมาณรูพรุนรวม และขนาดรูพรุนเฉลี่ยด้วยวิธี Nitrogen
Adsorption Isotherm ที่อุณหภูมิ 77 K ด้วยเครื่อง Autosorp (BEL MINISORP) คำนวณโดยใช้
สมการของ BET สำหรับการทดสอบการดูดซับที่สถานะของเหลว ทำโดยนำถ่านกัมมันต์ที่เตรียม
ขึ้นไปวัดค่าการดูดซับไอโอดีนโดยแสดงเป็นค่าไอโอดีนนัมเบอร์ในหน่วยมิลลิกรัมต่อกรัม (mg/g)
จากการทดลอง พบว่า สภาวะที่เหมาะสมสำหรับการเตรียมถ่านกัมมันต์จากกากกาแฟ ได้แก่ การใช้
$ZnCl_2$ เป็นสารกระตุ้น อัตราส่วนระหว่างสารกระตุ้นต่อปริมาณกากกาแฟที่ใช้ คือ 3:1 อุณหภูมิ
และเวลาที่ใช้ในการกระตุ้น คือ 500 °C และ 2 ชั่วโมง ตามลำดับ ที่สภาวะดังกล่าวได้ถ่านกัมมันต์

ที่มีค่าไอโอดีนนัมเบอร์สูงที่สุดเท่ากับ 926 mg/g พื้นที่ผิว – BET เท่ากับ 1,218.18 m^2/g และ
ปริมาตรรูพรุนรวมเท่ากับ 0.86 cm^3/g ส่วนไอโซเทอมของการดูดซับก๊าซไนโตรเจนมีลักษณะเป็น
ไอโซเทอมแบบ **Type I** นอกจากนี้ ได้ศึกษาและเปรียบเทียบสมบัติในการดูดซับ พื้นที่ผิว โครงสร้าง
รูพรุน และค่าไอโอดีนนัมเบอร์ของถ่านกัมมันต์ที่เตรียมขึ้นกับถ่านกัมมันต์ในทางการค้า อย่างไร
ก็ตาม กากกาแฟมีคุณสมบัติเพียงพอที่จะนำมาผลิตเป็นถ่านกัมมันต์ได้ เนื่องจากมีค่าไอโอดีน
นัมเบอร์มากกว่า 600 mg/g และมีร้อยละความชื้นน้อยกว่า 8 ตามมาตรฐานของ มอก.900–2547

คำสำคัญ: ถ่านกัมมันต์ กากกาแฟ การกระตุ้นทางเคมี

Abstract*

Coffee residue, a biomass, was used as an important source of precursors for the preparation of activated carbon by a chemical activation method. Activated carbon from coffee residue was prepared in a two-step process: carbonization and chemical activation processes. The influence of the activation condition variables such as activation reagents, activation reagent/precursor ratios, activation temperatures and activation times were investigated to find the optimum conditions. The quality of the derived activated carbon is characterized in terms of its physical properties of adsorption using different analytical methods for liquid and gas phase adsorption. For the gas adsorption test, the surface area and total pore volume of the derived activated carbons were measured by Nitrogen Adsorption Isotherms at 77 K (BET method), using an Autosorp (BEL MINISORP). The liquid adsorption test, the adsorption quality of the derived activated carbon, was evaluated in terms of iodine adsorption capacity (Iodine Number) reflecting the surface area. The optimum condition from experimental results was $ZnCl_2$/Coffee weight ratio of 3:1, an activation temperature of 500 $^\circ$C and activation time of 2 hrs. The characteristics of the derived activated carbon produced under optimum conditions were an iodine number of 926 mg/g, BET surface area of 1,218.18 m^2/g, and total pore volume of 0.86 cm^3/g. The nitrogen adsorption isotherm of the derived activated carbon exhibits **Type I.** Furthermore, the commercial activated carbons used to compare the adsorption quality, the surface area, the pore structure and iodine number with coffee residue activated carbon were reported. Activated carbons obtained from coffee residue showed good qualities for use as an activated carbon because iodine numbers

are higher than 600 mg/g and the moisture content (%) is less than 8%, values considered by the Thai Industrial Standards Institute, TISI.900-2004.

Keywords: Activated Carbons, Coffee Residue, Chemical Activation

Introduction

Coffee residue is a biomass waste of significant quantities in instant coffee manufacturing, of which 80,000 tons are produced annually in Thailand (Thailand, Ministry of Finance, Customs Department, 2007). The demand in coffee consumption is increasing each year, creating a considerable problem in waste disposal. Normally coffee residue is sent to the landfill or burned, which results in air pollution. Therefore, to make better use of this biomass waste, it could be recycled and converted into activated carbon, which is an economically valuable product. Activated carbon is porous carbonaceous material with a large surface area, typically produced from coal, wood species and agricultural by-products such as rice husk shells, coconut shells, nut shells, corncobs, sawdust and bagasse, etc. (Antal, et al., 2000; Babu and Chaurasia, 2003; Cheung, Porter, and Mckay, 2002; Salame and Bandosz, 2000).

Basically, there are two different processes for the preparation of activated carbon: carbonization and activation processes. Carbonization consists of thermal decomposition of precursors at 200-400 $^{\circ}$C in the absence of oxygen, eliminating non-carbon and producing a higher grade fixed carbon mass with a rudimentary pore structure called char, followed by an activation processes. The method of activation is a parameter which influences the final properties of activated carbons. Either physical or chemical activation may be used. In physical activation, precursors are activated at high temperatures in the range of 800-1,000 $^{\circ}$C by CO_2 gas or steam. In chemical activation, the most widely used activation reagents are H_2SO_4, H_3PO_4, HNO_3, H_3BO_3, NaOH, KOH, $Ca(OH)_2$, $ZnCl_2$, Na_2CO_3, $CaCl_2$, KSCN, $FeCl_3$, $Ca_3(PO_4)_2$, and NaCl (Girgis, Yunis, and Soliman, 2002; Kirubakaran, Krishnaiah, and Seshadri, 1991; Teng and Yeh, 1998; Zou and Han, 2001). The precursors are impregnated with activation reagent and then heated to temperatures of 500-700 $^{\circ}$C in a nitrogen

atmosphere. The advantages of chemical activation are a lower activation temperature, higher production yield and larger pore size (Kennedy, Vijaya, and Sekaran, 2004; Mohanty, et al., 2005).

The objective of this research was to produce activated carbons from coffee residue by the chemical activation method. The influence of activation condition variables, such as activation reagent, activation reagent/precursors ratio, activation temperature and time was investigated to find the optimum condition. Moreover, the derived activated carbons are then compared with the adsorption quality of commercial activated carbons.

Experimental Section

Materials and Apparatus: Coffee residue, a biomass obtained from a local coffee shop nearby and Nestle Products (Thailand) Inc., was used as the precursor. The commercial activated carbons and activation reagent, $ZnCl_2$, KOH, NaOH, H_3PO_4, H_2SO_4, and NaCl were purchased from Italmar (Thailand) Co., Ltd. Nitrogen gas was purchased from TIG (Thailand). The proximate analysis of coffee residue obtained from the experiment using ASTM methods (American Standard for Testing Materials), moisture content (ASTM Committee on Standards, 1998b), volatile matter content (ASTM Committee on

Standards, 1998d), total ash content (ASTM Committee on Standards, 1998c) and fixed carbon (by difference) is listed in Table 1.

Table 1: The proximate analysis of coffee residue used as raw material

Component	Quantities
Moisture (%wt)	19.0
Total Ash (%wt)	15.0
Volatile Matter (%wt)	23.0
Fixed Carbon (%wt)	43.0

Preparation of Activated Carbons: Activated carbons from coffee residue were prepared in a two-step process: carbonization and chemical activation processes. In the carbonization process, coffee residue was first washed with distilled water to remove the water-soluble impurities and then dried at 110 °C for 12 hrs. Then the precursor was heated to 400 °C to remove the moisture, non-carbon species and other volatile impurities at the rate of 10 °C/min for about 4 hrs. in N_2 to ensure an oxygen free atmosphere inside the reactor, then cooled to room temperature at the same rate. The resulting material is called chars (a fixed carbon mass with rudimentary pore structure). The chars were then subjected to chemical activation. In the chemical activation process, 50.0g of the carbonized carbon (W_1) were mixed with 50 mL of distilled water and an amount of

activation agent, depending on the ratios of activation reagent/carbonized carbons desired (1:1, 2:1 3:1 and 4:1, by weight (W_2)) for each activation reagent ($ZnCl_2$, KOH, NaOH, H_3PO_4, and H_2SO_4). The mixture was stirred to homogenize it at 85 °C for 4 hrs. This slurry mixture was dried at 110 °C for 12 hrs. Activation was conducted at one of three different temperatures, 500, 600, and 700 °C, respectively under nitrogen flow at a heating rate of 10 °C/min, and held at the final temperature for one of three different times, 2, 3 and 4 hrs, respectively. After activation, the derived activated carbons were removed from the reactor, weighed (W_3) and rinsed with hot and cold water several times to remove other inorganic matter until the pH become neutral, filtered and finally dried at 110 °C for 24 hrs, and then weighed again (W_4).

Production Yield: The yield of activated carbons was defined as the ratio of the dry weight of the activated carbon product to that of the original coffee residue, shown by Equation 1

$$\% \text{ yield} = \left(\frac{W_4}{W_1} \right) x100 \quad \underline{\quad}(1)$$

Activation Reagent Recovery (ARR): ARR is defined as the amount of activation reagent remaining with the product before washing and recovery from the washing solution, shown by Equation 2

$$\%ARR = \frac{(W_3 - W_4)}{W_2} x100 \quad \underline{\quad}(2)$$

Physical Characterization:

In the gas adsorption test, the surface area and pore volume of the derived activated carbons were measured by Nitrogen Adsorption Isotherms at 77K (BET method), using an Autosorp (BEL MINISORP). The BET equation (after Brunauer, Emmett and Teller) is used to calculate specific surface areas, expressed as Equation 3. (Rouquerol, Rouquerol, and Sing, 1999). The cross-sectional area of nitrogen molecules at 77K was considered to be 0.162 nm^2, using the adsorption points at the relative pressures $\left(\dfrac{P}{P_0} \right)$ 0.05-0.35. An adsorption isotherm was plotted between volume of gas adsorbed and relative pressure.

$$\frac{P}{V(P^0 - P)} = \frac{1}{n_m C} + \frac{C-1}{n_m C}\left(\frac{P}{P^0} \right) \quad \underline{\quad}(3)$$

$$C = \exp^{\left[\frac{\Delta H_A - \Delta H_L}{RT} \right]}$$

Where P is the equilibrium pressure, P^0 the saturated vapor pressure, V the adsorbed volume at pressure P, n_m the amount of adsorption by activated carbons (mono layer *mol/g*) and C the constant which is related to adsorption energy.

Before measurement, the samples were heated to 423K and out gassed at this temperature to evacuate all of the gas until a constant vacuum of 10^{-5} torr was reached, and then cooled to a temperature of 77K, the temperature of liquid nitrogen. At this temperature, nitrogen will be adsorbed on the surface of the samples.

In the liquid adsorption test, the adsorption quality of the derived activated carbon was evaluated in terms of the iodine adsorption capacity of the surface area. The higher the amount of iodine adsorbed, the better the quality of activated carbons. The amount of iodine adsorbed (*mg*) per 1 *gram* of activated carbon at an iodine residual concentration of 0.02 *mol/L* was reported as the iodine number (I_2 No., *mg/g*). The iodine adsorption test was followed by ASTMD 4607-94 (ASTM Committee on Standards, 1998a). The iodine number was calculated employing a three-point adsorption isotherm. A standard iodine solution was treated with three different weights of activated carbon. The iodine residual concentration was determined using the iodometry method by titration with standard $Na_2S_2O_3$ solution. The adsorption isotherm was plotted from the resulting data.

Results and Discussion

Effect of Activation Reagents

The effects of five activation reagents ($ZnCl_2$, KOH, NaOH, H_3PO_4, and H_2SO_4) on the characteristics of the coffee residue activated carbon are listed in Table 2. This Table presents five samples of coffee residue activated carbon produced using the same activation conditions (activation temperature, 500 $^{\circ}C$; activation time 2 hrs., and activation reagent/coffee residue ratio 1:1). The results show that activated carbon made using $ZnCl_2$ has the highest BET surface area of 511.55 m^2/g, total pore volume of 0.28 cm^3/g, average pore diameters of 2.20 nm and an iodine number of 655 mg/g. These results could be because $ZnCl_2$ plays an important role in retarding tar and other liquids which seal off pores during the activation process (Lua and Yang, 2005). The structure and composition of the carbons contained in coffee residue was not suitable for KOH, NaOH, H_3PO_4, H_2SO_4 activation. Therefore $ZnCl_2$ was chosen as the activation reagent for studying the next parameters.

Figure 1 shows the nitrogen adsorption isotherm of the derived activated carbon using five different activation reagents. The isotherm exhibits a *Type I* isotherm which results in a relatively homogeneous microporous carbon.

Table 2: The effects of five activation reagents on the characteristics of the derived activated carbons

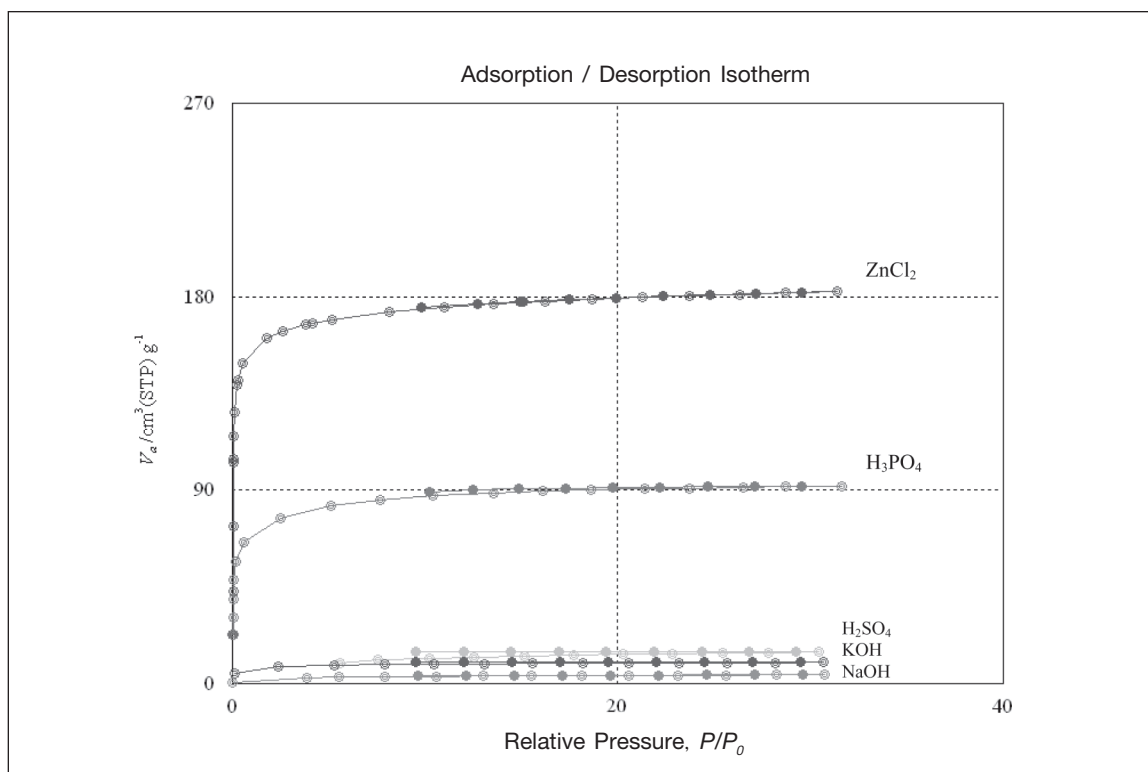| Activation Reagents	BET Surface Area (m^2/g)	Total Pore Volume (cm^3/g)	Average Pore Diameters (nm)	Iodine Number (mg/g)
$ZnCl_2$	511.55	0.28	2.20	655
H_3PO_4	259.53	0.14	2.19	320
H_2SO_4	44.84	0.02	2.04	180
KOH	28.34	0.02	2.04	124
NaOH	27.15	0.02	2.02	212

Figure 1: Nitrogen adsorption isotherm of the derived activated carbons

Effect of Activation Temperatures

The effect of activation temperatures on BET surface area, total pore volume, iodine number, production yield, and activation reagent recovery are shown in Table 3.

From Table 3, Figures 2 and 3, twelve samples (A-L) were prepared using $ZnCl_2$ as the activation reagent ($ZnCl_2$ gave the best result in our previous experiment). The activation time was 2hrs, The ratio of activation reagent/coffee residue varied from 1:1 to 4:1 and three activation temperatures 500, 600, and 700 $^{\circ}$C were compared.

It can be seen from Table 3, Figures 2 and 3 that the higher activation temperatures resulted in a lower BET surface area and iodine number. This is explained by the change of porosity and surface areas of activated carbon. When the activation occurs at high temperatures, it results in the contraction and realignment of carbon porous structures. (Mohanty, et al., 2005; Timur, et al., 2006; Tsai, et al., 2001)

It can be seen from Figures 4 and 5 that the production yield and activation reagent recovery decreased with an increase in the activation temperature. The production yield decreased with increasing activation temperatures due to the weight loss, including loss of the remaining volatile matter and the burn-off of the carbon (Gonzalez-Serrano, et al., 2004). In addition, the activation reagent recovery values decreased with increasing activation temperatures; this might be due to the evaporation and bond dissociation of $ZnCl_2$ at higher activation temperatures (Onal, et al., 2006). Thus, the activation temperature of 500 $^{\circ}$C was chosen as an optimum for activation of coffee residue.

Table 3: Properties of activated carbon from coffee residue by chemical activation with $ZnCl_2$

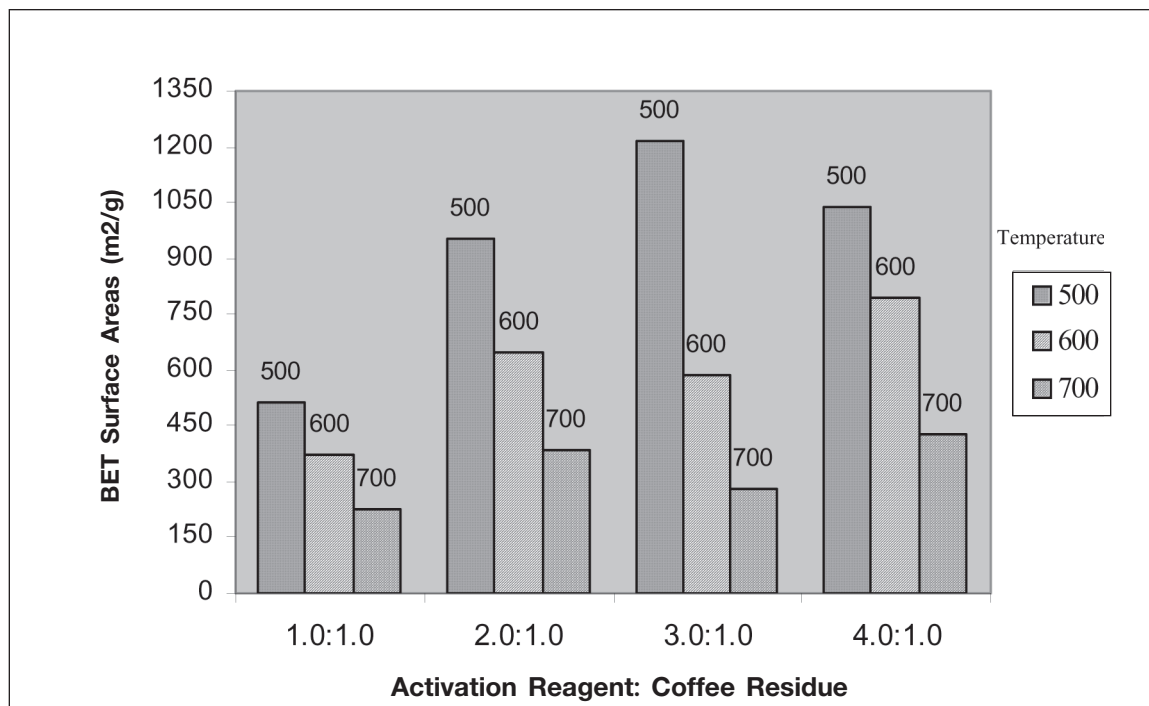| Samples	Activation Conditions			BET Surface Area (m^2/g)	Total Pore Volume, V_{total}, (cm^3/g)	Iodine Number (mg/g)	Yield (wt%)	Activation Reagent Recovery (wt%)
	$ZnCl_2$: Coffee Residue	Temp (°C)	Time (hr)					
A	1 : 1	500	2	511.55	0.28	655	31.37	68.32
B	1 : 1	600	2	374.61	0.21	404	28.87	27.58
C	1 : 1	700	2	224.35	0.17	292	19.94	3.64
D	2 : 1	500	2	953.46	0.67	767	22.85	71.97
E	2 : 1	600	2	649.72	0.34	515	19.63	32.43
F	2 : 1	700	2	386.24	0.23	487	15.87	2.97
G	3 : 1	500	2	1,218.18	0.86	926	37.66	72.43
H	3 : 1	600	2	585.79	0.29	487	32.00	36.06
I	3 : 1	700	2	279.13	0.19	348	19.21	1.75
J	4 : 1	500	2	1,039.08	0.72	878	25.63	79.36
K	4 : 1	600	2	793.64	0.59	627	18.96	38.43
L	4 : 1	700	2	426.87	0.29	459	13.45	5.23
M	3 : 1	500	3	1,228.54	0.81	906	21.52	64.55
N	3 : 1	500	4	843.56	0.62	739	10.96	33.67

Figure 2: Effect of activation temperature on BET surface areas of the derived activated carbons at four different activation reagents to coffee residue ratios (activation time, 2 hrs.)
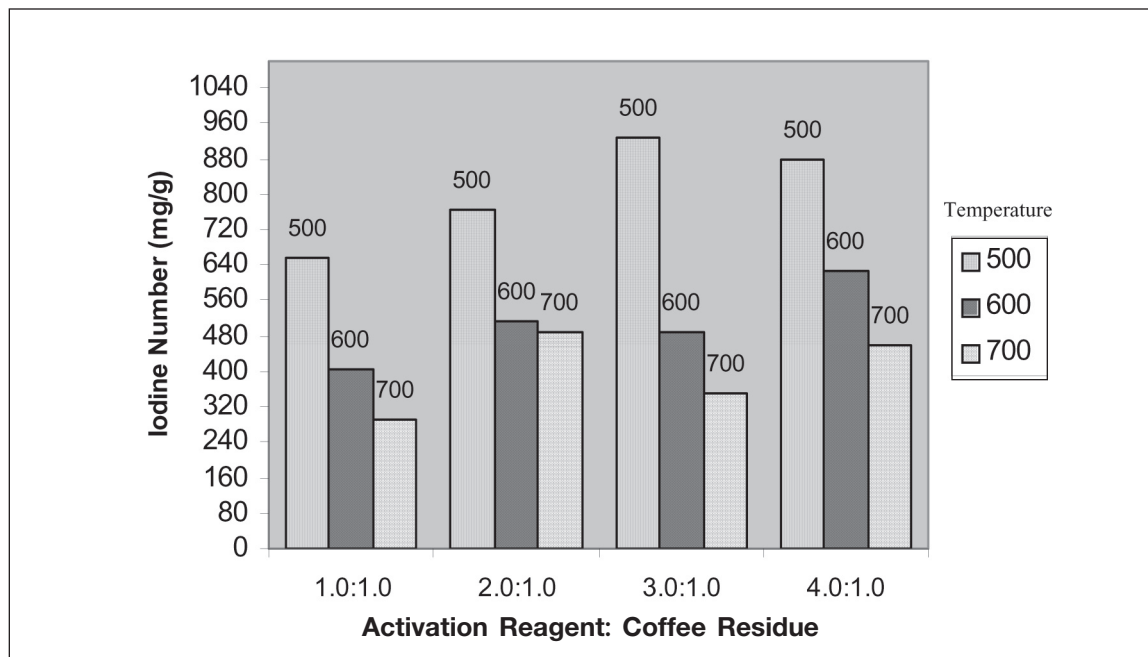
Figure 3: Effect of activation temperature on iodine number of the derived activated carbons at four different activation reagents to coffee residue ratios (activation time, 2hrs.)
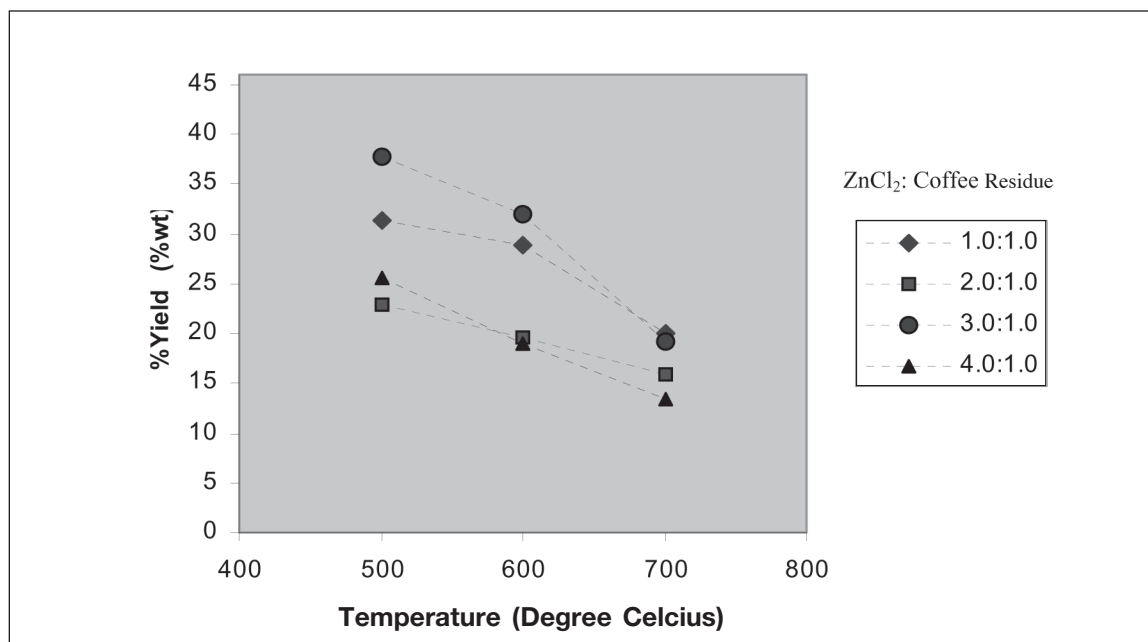

Figure 4: Effect of activation temperature on production yield of the derived activated carbons at four different activation reagents to coffee residue ratios (activation time, 2hrs.)
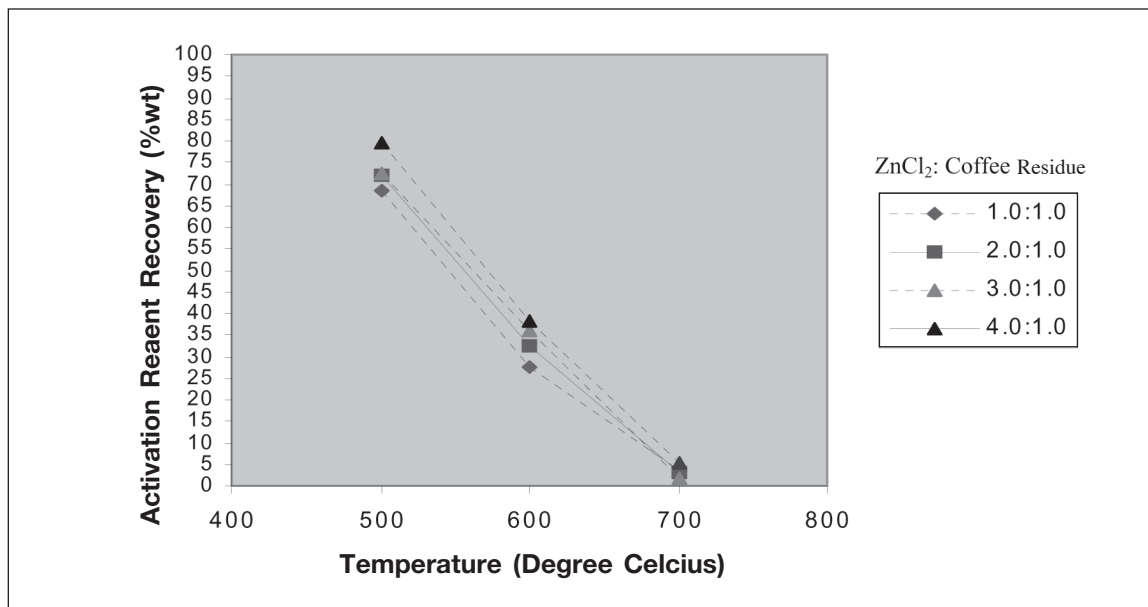
Figure 5: Effect of activation temperature on activation reagent recovery of the derived activated carbons at four different activation reagents to coffee residue ratios (activation time, 2hrs.)

Effect of Activation Reagent/Coffee Residue Ratios

The effect of activation reagent/coffee residue ratios on BET surface area, total pore volume, average pore diameters and the iodine number are listed in Table 4.

Table 4 shows the four samples prepared using $ZnCl_2$ as an activation reagent, with an activation time of 2 hrs., and an increase in the ratio of activation reagent/coffee residue from 1 to 4 at the same activation temperature of 500 $^{\circ}$C. Experimental results show that the BET surface areas, total pore volume, average pore diameters and iodine numbers

increased at first based on an increase in the ratio of activation reagent to coffee residue, but reached their maximum at 3:1, and thereafter decreased with further increases in the ratio from 3:1 to 4:1. The increase can be explained as corresponding to the change in porosity and surface areas of activated carbons, where activation with high ratios produces a development of the activated carbon with higher surface areas and porosity resulting from the opening of blocked pores and widening of existing pores than those obtained at lower ratios. However, the decrease in the ratio of 4:1 was possibly due to some of the pores being sealed off as a result of excessive

amounts of activation reagent. Since a maximum BET surface area of 1,218.18 m^2/g, a total pore volume of 0.86 cm^3/g, an average 2.82 nm for pore diameters and an iodine number of 926 mg/g were attained for a 3:1 activation reagent/coffee residue ratio, the ratio of 3:1 was found to be optimum and chosen to study for the next parameters.

Table 4: The effects of four activation reagent/coffee residue ratios on the characteristics of the derived activated carbon

$ZnCl_2$: Coffee Residue	BET Surface Area (m^2/g)	Total Pore Volume (cm^3/g)	Average Pore Diameters (nm)	Iodine Number (mg/g)
1:1	511.55	0.28	2.20	655
2:1	953.46	0.67	2.80	767
3:1	1,218.18	0.86	2.82	926
4:1	1,039.08	0.72	2.78	878

Effect of Activation Time

As shown in Table 3, three samples (G, M and N) were prepared using $ZnCl_2$ as activation reagent with a ratio of activation reagent/coffee residue of 3:1, an activation temperature of 500 $^\circ$C and variations of the activation time of 2, 3 and 4 hrs. From previous experiments, the above activation reagent, ratio of activation reagent/coffee residue and activation temperature was found to be optimum. It was observed that BET surface areas first increase with activation time and achieve their maximum at 3 hrs. but thereafter decrease. At the activation time of 3 hrs. BET surface areas were observed with a maximum of 1,228.54 m^2/g, and decreased to 843.56 m^2/g with a further increase of the activation time from 3 to 4 hrs. In addition, the total pore volume and iodine number decreased with an increase in activation time. This indicated that a prolonged activation time resulted in a decreased adsorption capacity. This decrease was due to some of the pores being sealed off and the collapse of pore walls (Sun, et al., 1997). Increasing the activation time has an effect on production yield similar to that of increasing activation temperatures. Furthermore, activation reagent recovery values decrease with the greater activation times. This might be due to the evaporation of $ZnCl_2$ at extended activation times. Therefore, it can be concluded that the shorter activation time of 2 hrs., produces activated carbons with the highest quality characteristics.

Comparison with Commercial Activated Carbon

The activated carbon produced from coffee residue under optimum conditions are compared to the commercial activated carbon in term of BET surface areas, total pore volume and iodine number in Table 5. From the experiment, the activated carbon produced from coffee residue presented more BET surface areas (1,218.18 vs.1,189.56 m^2/g), higher total pore volume (0.86 vs. 0.84 cm^3/g) and larger average pore diameter (2.82 vs. 2.79 nm), but lower iodine number (926 vs. 1,012 mg/g) than the commercial activated carbon.

Nevertheless, activated carbon obtained from coffee residue reflects a high grade for use as an activated carbon because iodine numbers are above 600 mg/g and the moisture content (%) is less than 8%, which are the values considered by the Thai Industrial Standards Institute, TISI-900 (Thai Industrial Standards Institute, 2004).

Table 5: Characteristics of the activated carbon produced from coffee residue compared to commercial activated carbon

Activated Carbons	Moisture (%wt)	BET Surface Area (m^2/g)	Total Pore Volume (cm^3/g)	Average Pore Diameters (nm)	Iodine Number (mg/g)
Activated Carbons Produced from Coffee Residue ($ZnCl_2$/3:1/500°C/2h)	6.0	1,218.18	0.86	2.82	926
Commercial Activated Carbons	4.0	1,189.56	0.84	2.79	1,012

Conclusions

In this study, coffee residue has been shown to be a feasible source for preparation of activated carbon by a chemical activation method. Chemical activator $ZnCl_2$ was chosen as the most effective activation reagent for activation of coffee residue for producing the best result in BET surface areas, total pore volume and iodine number. The nitrogen adsorption isotherm of the derived activated carbons exhibits a ***Type I*** isotherm, which results in a relatively homogeneous microporous carbon. It was found that a maximum BET surface area of 1218.18 m^2/g, total pore volume of 0.86 cm^3/g, average pore diameter of 2.82 nm and iodine number of 926 mg/g were attained from a 3:1 ratio of activation reagent to coffee residue, based on an activation temperature of 500 °C and an activation time of 2 hrs. Based on this study, the activation of **$ZnCl_2$-3:1-500 °C-2hrs.** seems to be the optimum combination for the

preparation of activated carbon from coffee residue.

Furthermore, compared with commercial activated carbon, those produced from coffee residue under optimum conditions produced more BET surface area, greater total pore volume and average pore diameter, but less iodine number. However the derived activated carbon showed superior quality for use as an activated carbon because iodine numbers are greater than 600 mg/g and the moisture content (%) is less than 8%, which are the values recommended by the Thai Industrial Standards Institute, TISI.

Acknowledgement

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