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Adsorption Characteristics and Pore Structure of Activated Carbons Prepared from Sorghum Distillery Residue

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Sorghum distillery residue (SDR), one type of fermentation waste, was used as the precursor to prepare activated carbon (AC) using the method of physics activation with steam– or CO_2–activation. The yield and iodine value of sorghum distillery residue activated carbon (SDRAC) was 15.74 to 29.24% and 113 to 684 mg/g using CO_2–activation, as well as 10.63 to 26.07% and 548 to 770 mg/g using steam–activation. As the iodine value of SDRAC was above 700 mg/g, the methylene blue adsorption value was 143.56 to 344.87 mg/g, the BET specific surface area was 502.7 to 530.1 m^2/g, the average pore diameter ranged from 2.89 to 3.18 nm and the true density was 1.86 to 1.97 g/cm^3. The nitrogen adsorption–desorption isotherms of the SDRAC were classified as Type Ⅳ, indicating the presence of microporous and mesoporous structures, according to the Bruauer, Deming, Deming and Teller (BDDT) Classification, and were H3 type hysteresis loops for most of the mesoporous structures in accordance with the International Union of Pure and Applied Chemistry (IUPAC). SDR, with a suitable activation condition, can serve as an excellent precursor for preparing AC because the iodine value and the BET specific surface area of the resulting SDRAC can be reached at the commercial value. SDRAC is able to be applied to liquid absorption, such as water purification, due to its mesoporous structure.

Key words: Sorghum Distillery Residue (SDR), Precursor, Physics Activation, Activated Carbon (AC), Mesoporous Structure

INTRODUCTION

Sorghum distillery residue (SDR), one residual product brewed from sorghum liquor, generally belongs to fermentation wastes in food industries. According to the Environmental Protection Administration, Executive Yuan in Taiwan (2012) the annual output for SDR is about 131 000 MT, which is in accordance with the statistics report for industrial waste, and the industrial wastes for SDR production is ranked within 20 in Taiwan. The daily output of SDR is about 150 to 300 tons in Kinmen County, one of the important production areas for sorghum liquor in Taiwan, and this increases year by year (Su *et al*., 2010). SDR can be used as an organic feed, combined with herbage in animal husbandry (Yang *et al*., 2003) because it contains a great deal of protein, starch and other nutritious substances (Ma *et al*., 2006). However, the growth of a microorganism, such as *Escherichia coli*, *Salmonella* or *Staphylococcus aureaus*, can produce aflatoxin which is a toxic compound produced by a mould fungus in agricultural crops, if the SDR has not been carefully stored (Bustamante *et al*., 2008). Research development of SDR, includes soil improvement and crop nutrition (Bustamante *et al*., 2007; Esperanza *et al*., 2007), compost materials (Bustamante *et al*., 2009; Paradelo *et al*., 2013), biotransformation using fungus (Perdih *et al*., 1991), extracting antioxidants (Lafka *et al*., 2007; Aliakbarian *et al*., 2012), functional application of extracts (Dong *et al*., 2010; Lin, 2008), production of ethanol biomass (Su *et al*., 2010) and feed improvement (Lee and Pan, 2003; Anandan *et al*., 2012; Nguyen Thi, 2012; Wang, 2012), but these reports have only been in the research stage. SDR has not been developed nor has it become a high value–added product. Moreover, an environmental protection problem has recently been proposed in Taiwan. The wastes, e.g. SDR, as well as the resources will help social and economic development if they can be reused and/or regenerated, especial for Kinmen County because Kinmen is an island and the utilization of SDR could solve the waste problem in that local area.

In general, common raw materials (precursor) are fibrous materials and/or carbonaceous matter (Hsieh, 1998), such as coconut shells, peanut shells, rice hulls, hard cores, coffee beans, bagasses and cotton seed hulls, which can become activated carbon (AC) by using a suitable pyrolysis and/or activation method. AC has a very special pore structure, usually represented by pore volume and pore size (Lu, 1994). Based on the distribution of pores in different diameters, pore structure and a high specific surface area, the specific surface area of AC is

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usually 500 to 1500 m^2/g (Huang, 2002) or to 2000 m^2/g (Rodríguez–Reinoso and Molina–sabio, 1992). It is more applicable to adsorbing organic pollutants because of its high specific surface area (Kienle and Bader, 1990; Wu and Tseng, 2000). A trend toward the demand of AC has increased year–by–year in Taiwan, according to the import and export data from the Directorate General of Customs (2013). The average imported amount, especially over the last ten years, was about 14,079 tons of AC. Furthermore, the "Resource Recovery Program" and the "Full Waste Classification and Zero Waste Group Action Project" are promoted by the Environmental Protection Administration (EPA) in Taiwan. AC prepared from waste is part of their project; therefore, many studies have been reported lately (Kim *et al*., 2001; Zhang *et al*., 2004; Peng, 2006; Amuda *et al*., 2007; Tseng *et al*., 2007; Aworn *et al*., 2008 and Wu *et al*., 2010). To promote the value–added and the wide application of fermentation wastes – SDR in food industries, as well as to increase the demand for AC in Taiwan, it is important to find a means of resource reutilization for SDR that can be used as a precursor for preparing AC.

The objectives of this study were to use SDR as a precursor for preparing AC using physics activation with steam– or CO_2–activation. The characterizations of sorghum distillery residue activated carbon (SDRAC) were investigated, including yield, iodine value, methylene blue adsorption value, BET specific surface area and porosity characteristics. Hopefully, the results can be used as a reference for resource reutilization from fermentation waste and can also be used as an example to food industries of how to implement the sustainable reuse of resources.

MATERIALS AND METHODS

Specimen and its basic properties

Precursor

Sorghum distillery residue (SDR) as a precursor was obtained from Kinmen Kaoliang Liquor INC. The SDR was washed and had a moisture content of less than 15.0% after being air–dried at ambient temperatures.

Ash

According to the Chinese National Standards (CNS) 5581 (1980) Method of Testing for Total Ash Content of Activated Carbon, the ash content of SDR was measured. The formula used is as follows: ash (%) = [(the weight of crucible with precursor ash – the weight of crucible) / (the weight of crucible with precursor – the weight of crucible)] * 100

Element analysis

The carbon (C), hydrogen (H), nitrogen (N), oxygen (O) and sulfur (S) contents of the SDR were determined using an elemental analyzer (Elemental Vario CHNS/O Analyzer, EA, Germany).

Chemical composition analysis

The chemical composition analysis of SDR were included the contents of holocellulose and lignin and ethanol–toluene extractives. The holocellulose of the SDR was measured according to the CNS 4713 (2005) Method of Test for Holocellulose Content of Pulpwood and Other Fibrous Materials. The formula for holocellulose (%) = (the absolute dried weight of holocellulose / the absolute dried weight of precursor) ×100

The lignin content of SDR was tested by the CNS 2721 (2010) Method of Testing for Determination of Acid–Insoluble Lignin in Pulp and the CNS 12108 (1987) Method of Testing for Acid–Soluble Lignin (Klason lignin) in Wood and Pulp. The formula for acid–insoluble lignin (%) = (the absolute dried weight of lignin / the absolute dried weight of precursor) ×100. The formula for acid–insoluble lignin (%) is $(B \times V \times 100) / (1000 \times W)$ where B is the concentration of acid–soluble lignin (g/L); V is the total volume of solution (mL) and W is the absolute dried weight of the specimen. Furthermore, $B = (A \times D) / 110$ where A is the absorbance and D is the diluted fold. Lignin content (%) = acid–insoluble lignin + acid–soluble lignin.

The ethanol–toluene extractives of SDR were measured according to the CNS 4713 (2005) Method of Testing for Ethanol–Toluene Extractives in Wood. The formula for ethanol–toluene extractives (%) = (the absolute dried weight of extractives / the absolute dried weight of precursor) ×100.

Thermogravimetric analysis

The SDR was dried in a convection oven at 40 ± 3°C for 3 h to prepare it for evaluation of thermal behavior, pyrolysis temperature and char using thermogravimetric analysis (TGA). Throughout this experiment, TGA was carried out using a Perkin–Elmer TGA 1, fully supported by computer–controlled software options from Perkin–Elmer Thermal Analysis Systems, for control and data handling. The dried SDR, approximately 5–10 mg in weight, was placed into an open experimental sample pan suspended from a sensitive microbalance. A furnace surrounded the specimen to provide accurate heating from 50 to 850°C, while measurements were taking place. All runs were purged in a nitrogen atmosphere at a flow rate of 100 mL/min, with a heating rate of 10°C/min. The TGA curve for SDR was analyzed. The TGA results obtained were supported by the derivative curve – the differential thermogravimetric (DTG) curve.

Preparation of SDRAC

CO_2–activation

The precursor was dried in an oven at 105°C for 24 h. For the first step–carbonization, the resulting specimen was loaded in a crucible, which was placed inside an upright high–temperature activation furnace (inner diameter, 26 cm; inner height, 40 cm) and was heated under a nitrogen (N_2 gas) flow rate of 200 mL/min for carbonization at a rate of 10°C/min. N_2 gas was added to make the container oxygen free. The carbonization temperature was set at 750, 800, 850 and 900°C. The second step–activation inserted the activation gas–CO_2 (purity of 99.99%) flow rate at 200 mL/min. The activation temperature was set at 750, 800, 850 and 950°C, and then, maintained at the desired temperature for 60 min (activation duration). In the third step, the sorghum distillery residue activated carbon (SDRAC) was cooled by N_2

Table 1. Abbreviation of each SDRAC[1)]

Activation method	Activation temperature (°C)	Activation duration (min)	Flow rate (mL/min)	Abbreviation [2)]of SDRAC
CO_2–activation	750	60	200	T750–60–200–CO_2
	800			T800–60–200–CO_2
	850			T850–60–200–CO_2
	900			T900–60–200–CO_2
Steam–Activation	750	60	90	T750–60–090–H_2O
			120	T750–60–120–H_2O
		90	90	T750–90–090–H_2O
	800	60	60	T800–60–060–H_2O
			90	T800–60–090–H_2O
			120	T800–60–120–H_2O
			150	T800–60–150–H_2O
		90	90	T800–90–090–H_2O
	850	60	90	T850–60–090–H_2O
			120	T850–60–120–H_2O
		90	90	T850–90–090–H_2O

[1)] SDR: Sorghum distillery residue; SDRAC: Sorghum distillery residue activated carbon
[2)] T (Activation temperature) – Activation duration – Flow rate – Activating agent

gas to a normal temperature and taken out. The aforesaid preparation conditions refer to (Chang *et al*., 1998; Kim *et al*., 2001; Zhang *et al*., 2004; Amuda *et al*., 2007; Tseng *et al*., 2007; Aworn *et al*., 2008; Huang *et al*., 2010; Wu *et al*., 2010; Liou, 2011 and Peng *et al*., 2012).

Steam–activation

The method of steam–activation was the same as the aforesaid preparation for the precursor and first step–carbonization. The second step–activation inserted the activation gas–steam, which was heated from deionized water with a flow rate set at 60, 90, 120 and 150 mL/h. The activation temperature was set at 750, 800, 850 and 950°C with activation duration of 60 and 90 min. The third step was also the same as the method of CO_2–activation. The steam–activation conditions refer to (Kim *et al*., 2001; Zhang *et al*., 2004; Amuda *et al*., 2007; Tseng *et al*., 2007; Aworn *et al*., 2008; Huang *et al*., 2010; Wu *et al*., 2010; Liou, 2011; Peng *et al*., 2012 and Lin *et al*., 2014).

The resulting SDRAC code was an activation temperature–activation duration–flow rate–CO_2–activation (CO_2) or a steam–activation (H_2O), such as: T750–60–200–CO_2 for SDRAC that was prepared with the carbonization temperature of 750°C with activation duration of 60 min and a flow rate of 200 L/h with CO_2. The abbreviation for each SDRAC is shown in Table 1.

Characterization of SDRAC

Yield

The yield for SDRAC (based on a dry basis) was calculated using the following equation:

Yield (%) = (the absolute dried weight of SDRAC / the absolute dried weight of precursor) × 100.

Iodine value

The diameter of iodine molecules was 0.56 nm (Hsieh and Teng, 1999). It acts as an absorption index for micropores with pore size diameters less than 2 nm, which have been classified by the International Union of Pure and Applied Chemistry (IUPAC). The iodine value was determined according to the Japanese Industrial Standard (JIS) K 1474 (1991) Test Methods for Activated Carbon. The formula for iodine adsorption capacity is: $I = [(10 - K \times f) \times 12.69 \times 5] / M$. The abbreviations for the formula are I: iodine adsorption capacity (mg/g); K: the volume of titrated sodium thiosulfate (mL); f: the ratio of 0.1 N sodium thiosulfate to 0.1 N iodine solution and M: the weight of absolute dried SDRAC (0.5 g).

Methylene blue adsorption value

To determine the methylene blue (MB) adsorption value, SDRAC (1 mg) was added to a 25 mL aqueous solution containing 1 g/L of MB and shaken at room temperature (30°C). When the aqueous solution became colorless, MB was repeatedly added to the flask to assure equilibrium adsorption of the MB. After filtration, the concentration of residual MB was determined using a UV–vis spectrophotometer (CECIL, CE3041) at a wavelength of 664 nm. This method was based on that used by Wu *et al*. (2010).

True density

The SDRAC was crushed and run through a sieve to obtain particles with a size between 40 to 60 mesh and then dried in an oven at 105°C for 24 h. By using the Ultrapycnometer 1000 (Quantachrome Instrument Co. Ltd.) in the Divisions of Forest Utilization in the Taiwan Forestry Research Institute, the distribution of the density for various SDRAC specimens was investigated. The experimental details of the density refer to (Hwang *et al*., 2013). Three replicas of each SDRAC were investi-

gated.

Porosity measurements

The pore structure characteristics of SDRAC were measured by nitrogen adsorption–desorption isotherms at 77 K using a Micromeritics ASAP 2020 at a relative pressure (*P/Po*) ranging from 10^{-2} to 1. The BET specific surface area (S_{BET}) was determined using the standard BET equation applied to a relative pressure range from 0.06 to 0.2. A t–plot and Barret–Joyner–Halenda (BJH) (Barrett *et al*., 1951) analysis software were used to calculate the external surface area (Sex), micropore volume (Vmi) and pore size distribution. The total pore volume (Vtot) was estimated as the liquid volume of nitrogen at a high relative pressure of 0.98, and the average pore diameter (D) was calculated using the equation $(4Vtot / S_{BET}) \times 10^3$ (Hu and Srinivasan, 1999).

Microscopic observation

The specimens, including SDR and SDRAC with various preparation conditions, for microscopic observation were oven–dried. An area of each specimen was sputter coated with gold–palladium and then observed using a Scanning Electron Microscope (SEM, HITACH S–2400 types) at 500 and 1000 X magnification.

Statistical analysis

The test results are represented by a mean (standard deviation), and the test groups are compared by Duncan's Analysis. If the ρ value is smaller than 0.05, meaning a significant difference among the test groups, it is represented by different superscript upper case letters.

RESULTS AND DISCUSSION

Basic properties of SDR

Ash and element content

The ash and element analysis of SDR are not expressed in a table. The SDR ash was 6.05 (0.50)%. The C content of the materials (including waste, residual substance, etc.) can be regarded as one of the precursors for preparing AC (Lu, 2001). The C content of SDR was 45.52 (0.12)%, and the other contents were 3.26 (0.04)% N, 7.24 (0.09)% H and about 43.98(0.12)% O. The results were the same as those published by Diao *et al*. (2002) which were 43.43% C, 0.95% N, 7.10% H and 48.52% O. Compared to other precursors, the C content of a walnut shell was about 46.60% (Kim *et al*., 2001), the C content of corn stick was about 46.60% (Zhang *et al*., 2004), the C contents of a corn cob, mushroom stalk and bagasse pith were from 41.60–46.90% (Tseng *et al*., 2007). Lin *et al*. (2002) also reported that the average carbon content of nine sorts of conifers was 48.21%. The aforesaid results and references suggest that SDR could be a potential precursor for preparing AC.

Chemical compositions

The chemical compositions of holocellulose, lignin and ethanol–toluene extractives of SDR were 47.30 (0.24)%, 21.95 (0.39)% and 13.12 (0.52)%, respectively (results not shown in Table). Su *et al*. (2010) reported that for evaluating SDR as biomass materials, the composition percent of cellulose, hemicellulose and acid–soluble lignin of SDR were 18.2%, 21.6% and 20.6%, respectively. The ethanol–toluene extractives of SDR were higher than for other waste such as recycled cartons which were from 0.26 to 0.27% (Huang *et al*., 2010) or betel palm stems which were from 2.53 to 12.28% (Huang, 2005; Wu, 2011). The ethanol–toluene extractives of SDR had a higher content because the shell of sorghum contained higher tannin (Awika and Rooney, 2004). Lin *et al*. (2006) reported that the higher the ethanol–toluene extractives, the higher the thermal behavior exhibited. In other words, for the precursor with a higher content of ethanol–toluene extractives it is possible to have higher thermal–resistant components. Liou and Duh (2009) also reported that a higher content of ethanol–toluene extractives and lignin influenced the activation degree of AC.

Thermogravimetric analysis

TGA is a common technique for measuring the weight change of a material as a function of temperature or time (Coats and Redfern, 1963; Cornfield *et al*., 1993; Pasek and McIntyre 1993; Pasek, 1995; Helsen *et al*., 1997; Hsu *et al*., 2000). To comprehend the thermal behavior (pyrolysis, thermal decomposition), the SDR was measured by TGA, after being air–dried at ambient temperatures. Decomposition profiles were obtained while heating at a rate of 10°C/min in nitrogen in temperatures between 50°C (initial temperature) and 850°C (end temperature). The relationships between temperature and weight loss for the TGA curve and the derivative weight (DTG curve) for SDR are shown in Fig. 1. As expected, the SDR typically showed a gradual weight loss. The curves from TGA and DTG analysis revealed that the peaks of pyrolysis temperatures were 264.47, 412.63,

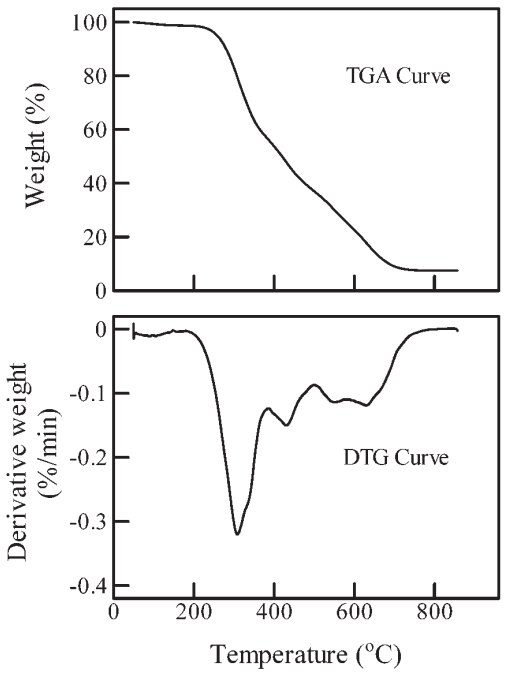

Fig. 1. TGA and DTG curves for SDR in nitrogen at 10°C/min.
Note SDR: see the Table 1

523.31 and 611.71˚C, with a corresponding weight loss of 42.62, 62.00, 73.68 and 91.38%. The amount of char for SDR, at a temperature of 850˚C, was 7.54%. Saunders and Allcorn (1972) reported that the pyrolysis temperature for cellulose and hemicellulose was 200–260˚C, and for lignin the temperature was about 400–450˚C; therefore, the results infer that at a high temperature (over 500˚C), the pyrolysis temperature was related to the ethanol–toluene extractives of SDR due to tannin.

Characterization of SDRAC

Yield and iodine value

As shown in Table 2, the yield from SDRAC was from 15.74 to 29.23% using CO_2–activation with a 200 mL/min flow rate at activation temperatures of 750, 800, 850 and 950˚C and an activation duration of 60 min. Using steam–activation with a flow rate from 60 to 150 mL/min with the same ranges in activation temperature and duration, the yield of SDRAC was from 10.63 to 26.07%. The results revealed that the difference in the yield was significant (5%) in accordance with Duncan's multiple range tests, except for the SDRAC prepared at an activation temperature of 800˚C for 60 min of activation duration with a flow rate from 90 to 150 mL/min. No matter what kind of activation method was used, results were dependent on the activation temperature and duration, and the influence of the activation temperature was greater than that of the activation duration (Chang *et al*., 1998 and 2003; Zhang *et al*., 2004; Tseng *et al*., 2007; Huang *et al*., 2010; Wu *et al*., 2010; Peng *et al*., 2010). This is because the activation temperature influences the precursor by means of the pyrolytic decomposition decreasing the volatile compounds and tar, as well as increasing carbonaceous gasification (Teng and Hus, 1999). The results also show that the yield of SDRAC by CO_2–activation was higher than that by steam–activation, indicating that the steam–activation method produced oxygen that resulted in much more carbonaceous surface oxidization (Zhou *et al*., 2003).

The iodine value of SDRAC using CO_2–activation was 113.2–684.5 mg/g (Table 2), which is lower than the value using steam–activation, 548.7–761.8 mg/g. The iodine value of SDRAC prepared with steam at 90 mL/min at 900˚C for 60 min was the highest, 761.8 mg/g. According to Duncan's analysis, when using steam–activation there was an obvious difference in the iodine value of SDRAC due to the increase in activation temperature. The results in Table 2 also show that the higher the flow rate of steam, the greater the decrease in iodine value, such as the flow rate at 150 mL/min. This indicates that a higher flow rate results in expansion on micropores or a burn–off on the carbonaceous surface due to uneven activation; that is, the micropores' neighbor is ruined and its pore scale is expanded (Zhou *et al*., 2003; Aworn *et al*., 2008).

The diameter of iodine molecules is 0.56 nm (Hsieh and Teng, 1999). The iodine value can be an indicator of micropore amounts of AC (Chen, 2003). Micropores with pore sizes less than 2 nm in diameter have been classified by IUPAC. The iodine value increases as the amount of AC micropores increases and the absorption of AC improves. The iodine standard value for commercial AC is 600–1000 mg/g (Wu and Tseng, 1999). In order to evaluate the absorption and porosity of SDRAC in the following experiments, including the MB adsorption value, true density, nitrogen adsorption–desorption isotherms and pore size distribution, SDRAC specimens are determined to meet the commercial standard iodine value, and their yield must be more than 17%. The selected SDRAC specimens were prepared by using steam–activation with a flow rate from 90 to 150 mL/min at an activation temperature of 800 and 850˚C. Abbreviations for the specimens were T850–60–90–H_2O, T800–60–90–H_2O, T800–60–120–H_2O and T800–60–150–H_2O (see Table 1).

Table 2. Yield and iodine value of SDRAC[1)] prepared under various conditions

Specimen [2)]	Yield (%)	Iodine value (mg/g)
T750–60–200–CO_2	29.23 (0.02) [g 3)]	113.2 (00.00) [a]
T800–60–200–CO_2	26.56 (0.44) [f]	406.3 (00.27) [b]
T850–60–200–CO_2	23.90 (0.03) [e]	502.6 (00.00) [c]
T900–60–200–CO_2	15.74 (0.24) [b]	684.5 (00.00) [d]
T750–60–090–H_2O	26.07 (0.10) [f]	548.7 (07.25) [c]
T800–60–090–H_2O	19.53 (0.49) [cd]	703.6 (00.00) [d]
T850–60–090–H_2O	17.67 (0.31) [c]	724.1 (06.87) [de]
T900–60–090–H_2O	14.08 (0.37) [a]	761.8 (06.85) [f]
T750–90–090–H_2O	22.15 (0.49) [de]	648.8 (05.64) [d]
T800–90–090–H_2O	14.18 (0.23) [b]	712.0 (05.89) [d]
T850–90–090–H_2O	12.26 (0.31) [ab]	732.9 (05.96) [de]
T750–60–120–H_2O	21.33 (0.63) [d]	678.1 (00.64) [d]
T800–60–120–H_2O	18.31 (0.12) [c]	770.7 (07.40) [f]
T850–60–120–H_2O	10.63 (0.12) [a]	732.9 (05.92) [de]
T800–60–060–H_2O	21.25 (0.46) [d]	686.7 (14.11) [d]
T800–60–090–H_2O	19.53 (0.49) [c]	703.6 (00.00) [d]
T800–60–120–H_2O	18.31 (0.12) [c]	770.7 (07.40) [f]
T800–60–150–H_2O	17.61 (0.29) [c]	705.9 (06.57) [d]

[1)] and [2)] See Table 1; [3)] Mean (standard deviation) with the different superscripts is significantly different ($\rho < 0.05$) by Duncan's multiple range tests

Methylene blue adsorption value and true density

The MB adsorption value is an indicator of larger micropore and smaller mesopore amounts of AC (Sun and Jiang, 2010) because the diameter of MB molecules is 1.3 nm (Tseng *et al*., 2003). The iodine value denotes the amount of micropores, whereas the MB adsorption value can be used to estimate the capability of AC to absorb large organic molecules (Heschel and Klose, 1995). In other words, the MB adsorption value increases as the amount of AC micropores and mesopores increases and the absorption of AC improves. As shown in Table 3, the MB adsorption value ranged from 143.56 to 344.87 mg/g for the SDRAC prepared by using steam–activation with a flow rate from 90 to 150 mL/min at an activation tem-

perature of 800 and 850°C. According to Duncan's multiple range tests, the difference in the MB adsorption value was insignificant (5%) for T800–60–90–H_2O and T800–60–120–H_2O, but it was significant for T800–60–90–H_2O and T850–60–90–H_2O. The tendencies of aforesaid results are the same as those of the iodine value; that is, the MB adsorption value is influenced by the activation temperature which results in a better activation and pore formation, and it is influenced by the steam's flow rate which expands and/or ruins the pore (Aworn *et al*., 2008), such as: T800–60–150–H_2O. The highest MB adsorption value (344.87 mg/g) was obtained at an activation temperature of 850°C (T850–60–90–H_2O), but the MB adsorption value of the T800–60–150–H_2O was insignificant.

AC is frequently used in adsorptive separation processes. True density is the density of the solid material, excluding the volume of any open and closed pores. Therefore, in general a larger true density is needed if AC is to be considered as a material of liquid absorption, such as water purification (Salvador and Jiménez, 1999). Table 3 also shows that the true densities of the selected SDRAC specimens were between 1.86 to 1.97 g/cm^3. The highest value was for specimen T850–60–90–H_2O (1.97 g/cm^3); the lowest value was for T800–60–150–H2 (1.86 g/cm^3). Lin and Hwang (2006) reported that the higher the activation temperature, the greater the true density. These results are the same as results from other studies: 1.35–1.96 g/cm^3 (Lo and Wang, 2007); 1.42–1.88 g/cm^3 (Lan *et al*., 2008); 1.68–1.82 g/cm^3 (Lin *et al*., 2010), but are less than for others: 2.13–2.06 g/cm^3 (Huang *et al*., 2010); 2.01–2.87 g/cm^3 (Wu *et al*., 2010); 2.11–2.17 g/cm^3 (Lin *et al*., 2014).

Table 3. True density and methylene blue adsorption value of SDRAC[1)] prepared under various conditions

Specimen [2)]	methylene blue adsorption value (mg/g)	True density (g/cm^3)
T800–60–090–H_2O	143.56 (16.06) [a 3)]	1.92 (0.00) [a]
T800–60–120–H_2O	245.43 (16.94) [a]	1.96 (0.00) [a]
T800–60–150–H_2O	334.37 (22.32) [b]	1.86 (0.00) [a]
T850–60–090–H_2O	344.87 (13.57) [b]	1.97 (0.00) [a]

[1)] and [2)] See Table 1; [3)] See Table 2

Porosity and microscopic observation

The S_{BET} and pore characteristics of selected SDRAC specimens are shown in Table 4. The S_{BET}, Sex (from t–plot and BJH analysis) and Vtot increased as the activation temperature and flow rate of steam increased. T800–60–120 exhibited the highest values, with a S_{BET}, Sex and Vtot of 530 m^2/g, 162 m^2/g and 0.41 cm^3/g. The best S_{BET} and porosity can be obtained using an activation duration of 60 min. However, with a higher flow rate (150 mL/min), the S_{BET} decreased from 530.1 to 502.7 m^2/g, the Sex decreased from 162.00 to 144.39 m^2/g and the Vtot decreased from 0.41 to 0.36 cm^2/g. We deduced that an increase in the flow rate resulted in a collapse of the internal walls of the micropores or the combination of micropores, creating large pores (becoming mesopores), and thus, decreasing the S_{BET} and Vtot of the SDRAC. Commercial AC exhibits S_{BET} values ranging from 500 to 2000 m^2/g (Rodríguez–Reinoso and Molina–Sabio, 1992). The results obtained demonstrated that the S_{BET} of SDRAC was in the range of commercial AC. Wang (2004) reported that as the surface areas increased, the adsorption became better, so that the surface area could be an adsorption indicator of AC.

The percentage of Vmi/Vtot showed that SDRAC has some micropores from 41.02 to 44.44%. Physical activation has pore–drilling and expansion effects at 800°C producing multiple micropores (Lua and Guo, 2000; Yun *et al*., 2001). The average pore diameters of SDRAC were from 2.69 to 3.18 nm. When the activation temperature rises, as the expansion effect is greater than the pore–drilling effect, multiple mesopores or some macropores are produced (Walker and Almagro, 1995). Therefore, the average pore diameter for SDRAC: T850–60–90–H_2O was 3.14 nm, larger than that for T800–60–90–H_2O which was 2.89 nm. At a higher flow rate of steam, the average pore diameter of SDRAC T800–60–120–H_2O was greater than that of T850–60–90–H_2O, but it became lower for T800–60–150–H_2O, and the S_{BET}, Sex and Vtot were decreased as well. This indicates that a higher flow rate can result in expansion of micropores to mesopores, but the burn–off on the carbonaceous surface is affected by over–activation (Aworn *et al*., 2008; Lin *et al*., 2014).

Figure 2 shows the nitrogen adsorption–desorption isotherms of the selected SDRAC specimens. When the relative pressure *P/Po* was near 0, the quantity of N2 adsorbed from 4 types of SDRAC specimens was the

Table 4. Porosity of SDRAC[1)] prepared under various conditions

Specimen [2)]	S_{BET} [3)] (m^2/g)	t–plot		Vtot [6)] (m^3/g)	Vmi / Vtot (%)	D [7)] (nm)
		Sex [4)] (m^2/g)	Vmi [5)] (m^3/g)			
T800–60–090–H_2O	511.6	154.24	0.16	0.36	44.44	2.89
T800–60–120–H_2O	530.1	162.00	0.17	0.41	41.46	3.18
T800–60–150–H_2O	502.7	144.39	0.16	0.36	44.44	2.96
T850–60–090–H_2O	516.9	155.20	0.16	0.39	41.02	3.14

[1)] and [2)] See Table 1; [3)] S_{BET}: specific surface area; [4)] Sex: external surface; [5)] Vmi: micropore volum; [6)] Vtot: total pore volum; [7)] D: average pore diameter

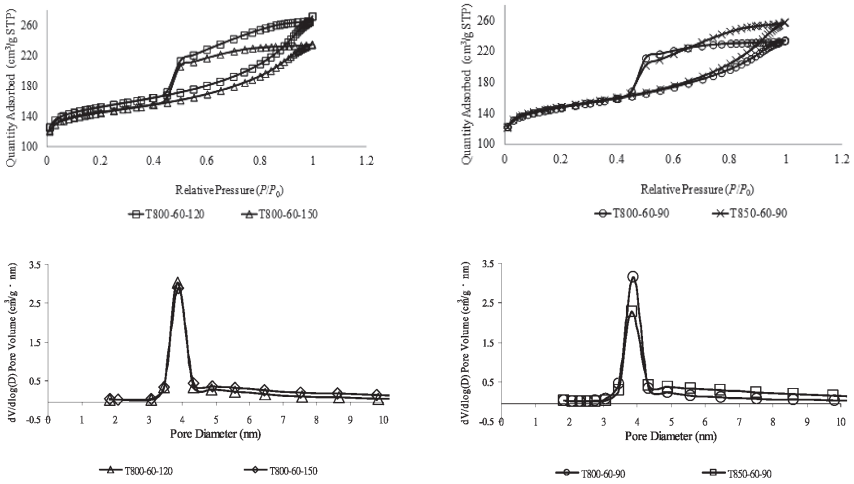

Fig. 2. Nitrogen adsorption–desorption isotherms and Pore size distribution of SDRAC[1)] prepared under various conditions.
Note [1)] See Table 1

same, about 120 cm^3/g. The isotherms of T850–60–90–H_2O, T800–60–90–H_2O, T800–60–120–H_2O and T800–60–150–H_2O all exhibited a well–defined plateau and hysteresis loop, and both the hysteresis loop and adsorption "tail" were obvious at a high relative pressure (*P/Po*). According to the Bruauer, Deming, Deming and Teller (BDDT) classification, these SDRACs were Type IV, indicating the micro–mesopore content of the adsorbents, and were H3 type hysteresis loops with the most of mesoporous structures, in accordance with the IUPAC. The isotherms of the SDRAC also displayed a slight slope with a hysteresis loop, typical of Type Ⅳ isotherms and associated with capillary condensation. This loop is of the H3 type (Sing *et al*., 1985) and is common in microporous carbons in which mesopores are also present (Gregg and Sing, 1982). The pore size distribution of the SDRAC is also shown in Fig. 2. The range of the mesopores drastically increased from 2 to 4 nm, indicating the creation of micropores to produce mesopores. The formation of mesopores is known to be enhanced by an increase in the activation temperature or the flow rate of steam (Zhou, 2003; Aworn *et al*., 2008). The selected SDRAC specimens resulted in an obvious peak with a pore diameter of about 4.0 nm (Fig. 2), indicating that they contained many mesoporous structures. The aforesaid results had the same tendency as seen in the results of the average pore diameters of SDRAC, from 2.69 to 3.18 nm (Table 4). Kunio *et al*. (2001) reported that AC with an optimal mesoporous structure could be effective for adsorbing organic pollutants. This indicates that SDRAC with lots of mesoporous structures can be expected to be a liquid absorption material.

A microscope observation for the SDR and the selected SDRAC specimens is shown in Fig. 3. The 500 and 1000 times SEM micrograph of SDR indicated that there was no pore with a rugged surface (shown by the top figure on the left and right sides of Fig. 3). The images of SDRAC show the presence of uneven honeycomb–like groups of pores on the surface T850–60–90–H_2O (the second line figure on the left side) with some

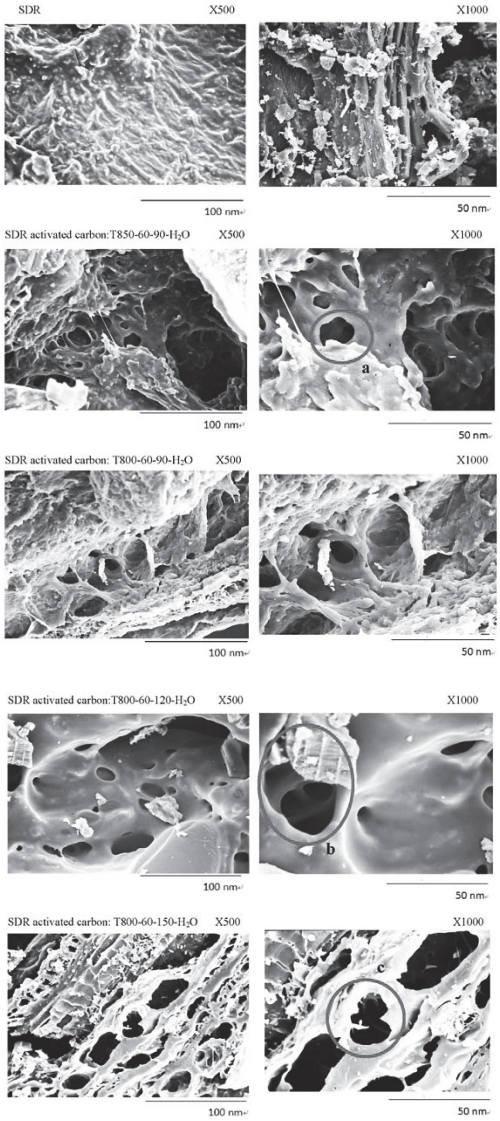

Fig. 3. SEM photographs of SDR[1)] and SDRAC[2)] prepared under various conditions
Legends: a: a pore inside with a pore; b: microporous and mesoporous structures; c: the neighbor of micropores is ruined and its pore scale is expanded
Note [1)] and [2)] See Table 1

carbonaceous skeleton from the plant capillary structure of the raw material, SDR. It indicates that the pores are formed after SDR has been activated with steam, such as the pores formed from T800–60–90–H_2O (the third line figure on the left side). Compared to T850–60–90–H_2O, the pore formed (Fig. 3. a, ×1000) was larger than that of T800–60–90–H_2O (the third line figure on the right side, ×1000), indicating the activation temperature effect on the pore formed (Chang *et al*., 1998 and 2003; Zhang *et al*. 2004). These were the same results as those from the iodine value and MB adsorption value on Tables 2 and 3. With the increase in the steam flow rate on the surface of specimen T800–60–120–H_2O we could see lots of mesopores and some micropores (the fourth line figure on the left side and Fig. 3. b, ×1000). However, a higher flow rate resulted in the micropore's neighbor being ruined and becoming a mesopore (Zhou *et al*., 2003; Aworn *et al*., 2008), as shown on the fifth line figure on the left side and Fig. 3. c, ×1000. These observations were the same as the results for the iodine value (Table 2) and the average pore diameter from Table 4.

CONCLUSIONS

SDR was used as the precursor to prepare SDRAC using the method of physics activation with steam– or CO_2–activation. SDR exhibited a high carbon content of 45.52%, indicating that this precursor was suitable for preparing AC. The chemical compositions of holocellulose, lignin and ethanol–toluene extractives for SDR were 47.30, 21.95 and 13.12%, indicating the precursor with a higher content of ethanol–toluene extractives had higher thermal–resistant components. The yield of SDRAC was 15.74 to 29.24% using CO_2–activation and 10.63 to 26.07% using steam–activation. The iodine value of SDRAC was 548.7 to 770.7 mg/g using steam–activation, which is higher than that (113.2 to 684.5 mg/g) using CO_2–activation. The MB adsorption value, the true density, the BET specific surface area and the average pore diameter of the selected SDRAC specimens (above 700 mg/g of iodine value and over 17% yield) were 143.56–344.87 mg/g, 1.86–1.97 g/cm^3, 502.7–530.1 m^2/g and 2.89–3.18 nm. The nitrogen adsorption–desorption isotherm of all SDRAC was classified as Type IV, indicating the presence of micro– and mesoporous structures and was an H3 type hysteresis loop with most mesoporous structures. SDR, with a suitable activation condition, can serve as an excellent precursor for preparing AC because the iodine value and the BET specific surface area of the resulting SDRAC can reach a commercial value. Hence, the results indicate that the SDRAC prepared in this study features a lot of mesoporous structures, and the SDRAC can be used as an adsorbent of liquid absorption, such as water purification.

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