Investigate the effects of synthesizing techniques on the particle size

By Barlin Oemar

Investigate the effects of synthesizing techniques on the particle size reduction and thermal behaviors of carbon-based material from natural wood charcoal

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Abstract. In this research work, a novel investigation of the carbon-based material from natural wood charcoal synthesized with various top-down techniques on the particle size reduction and thermal behaviors. First, three powder samples were synthesized using manually by hand. Second, two powder samples were synthesized using ball milling. The particle size (length and width) were analysed using SEM micrographs. Furthermore, the thermal characterization had been carried out using thermogravimetric analysis. The results showed that the length and width of the ball milled sample are smaller than the manually milled samples (53, 74 and 105 μm) due to the higher energy mill between the ball mill and the charcoal powder. The thermogravimetric analysis showed that the samples takes places in four steps of decomposition. First stage occurred at 49.9-50°C, second at 72.58-83.69 °C, third at 268-319 °C and fourth at 689-738 °C. The ball milled samples exhibited the lower weight loss total (23.3 and 59.12%) than the manually milled samples (29.58, 34.9 and 36.39%) which is affected by the chemical composition (lignin, cellulose and hemicellulose) and inorganic substances present in the samples. As a conclusion, the ball milling gave the optimum results in terms of particle size reduction and thermal behaviors.

1. Introduction

With larger surface area, high conductivity and adsorption capacity, carbon-based material (CBM) and activated-CBM (aCBM) have attracted to more attention on many applications, such as hydrogen storage material [1–3], catalyst support [4] in the water electrolyser system, and supercapacitor[5]. CBM were reported to have light weight, inexpensive cost and the abundant raw material resources in nature[6].

Nowadays, nanostructured materials have been investigated intensively due to their converted properties compared to the bulk materials [7]. Damonte and coworkers said the synthesis of nanostructured materials is an effective, useful and simple processing technique [8]. The nanostructured material scan be synthesized either by bottom-up technique (sol-gel and hydrothermal) or top-down technique (grinding, conventional milling and high energy ball milling). High energy ball milling has been developed as a useful technique, the input energy is capable of producing material[9]. Planetary ball mill (PBM) is commonly used for particle size reducing down to nanometer scale due to their good reproducibility, safe handling and short processing time[10].

In this study, two different top-down synthesizing techniques (conventional milling and high energy ball milling) of the nanostructured materials were applied on CBM obtained from natural wood

charcoal. The effects of different techniques on the particle size reduction was measured using directly analysis of SEM micrographs. Then, the thermal gravimetric analysis was conducted to analyze the thermal behaviors (temperature range and weight loss of CBM).

2. Materials and Methods

2.1. Material and preparation
The raw material of this study is obtained from natural wood charcoal. The charcoal lump was purchased from supermarket in Tainan city, Taiwan, which was produced by Spreading Co., Ltd, Thailand. To attain different particle size, two synthesizing techniques were used to prepare the charcoal samples: manually crushing by hand (MBH) and milling by ball mill (BMM). Three samples were produced using MBH technique, another two used BMM technique. For MBH technique, charcoal lump was crushed by hand in a stainless steel crucible. The crushed samples were sieved with a sieve shaker for five hours. By using three stainless steel sieve (Bunsekifurui Mesh 270, 200 and 140), the samples were formed in different particle sizes (53, 74 and 105 μm). The second technique was employed to obtain fines particles in microscale. Charcoal powder with particle size >105 μm was milled with a planetary ball mill. The PBM (SE-PM 4L) rotated at 300 rpm for 30 hours. To optimize the effect of ball mill to powder ratio (BPR), the PBM was operated in two different BPR (2:1 and 3:1). The used ball mill consist of Z_rO_3 (94.5%), Y_2O_3 (5.1%) and Al_2O_3 (0.25%). The ball milling condition used in this work are shown in table 1.

Table 1. The ball milling condition used for the milled samples.

Sample	Weight ratio	Total ball mill weight (gr)	Charcoal powder weight (gr)	Milling time (h)	Milling speed (rpm)	Ball mill diameter (mm)
S1-3	2:1	30.02	15.01	30	300	5.01
S3-3	3:1	45.03	15.01	30	300	5.01

2.2. Material characterizations
The crushed and milled powder samples were characterized using the FE-SEM (JEOL JSM-6701F) for observing the surface morphology. Particle size reduction analysis was obtained from the SEM micrographs. Thermogravimetric analysis of the carbon-based material produced from natural wood charcoal was performed using a thermogravimetric analyzer (TGA 8000, Perkin-Elmer) with 50 ml/min nitrogen flowrate and 10 °C/min heating rate until 600 °C. Approximately, 5-10 mg of samples were placed in platinum plate in a TGA furnace. The samples were dried at 100°C for 1 hours to remove the water vapor before placing in platinum plate. The nitrogen gas was used to stabilized the TGA furnace and bring out the generated gas when the samples were warmed up. The thermal cracking temperature and the residual ratio of charcoal with different weight percentages during the TGA analysis were measured.

3. Results and Discussion

3.1. Particle size reduction
The average particle size of the crushed by hand samples (53, 74 and 105 μm) and milled by ball mill samples (S1-3 and S3-3) are shown in Table 2. It shows a comparison between the obtained values such as length and width as the effect of various synthesizing technique i.e. manually crushing by hand and ball milling. The average particle size of the milled samples were lower than the manually crushed samples. The average particle size were reduced from >105μm to 4-5μm (weight ratio 2:1) and 12-13μm (weight ratio 3:1). The ball mill technique reduced the particle size around 95.2% (S1-3) and 88.6% (S3-3). As can see in figure 1, the crushed by hand samples showed the higher of the obtained length and width than the ball milled samples. It is clearly observed that the ball mill technique can reduced the samples to the smallest length and width size. According to the previous studies [11], comparing the different weight ratio indicated that in spite of having milling speed and time, the total weight of ball mill has a significant effect on milling efficiency. The frequency of collisions in a

planetary ball mill can be obtained from the number of ball mill and milling speed. The overall kinetic energy received by charcoal powder is affected by the frequency of collision, energy of each impact and weight of charcoal powder. The milled sample with higher weight ratio will has the higher number of collision per powder particle. Based on the weight ratio in Table 1, the number of collision (S3-3) is 1.5 times more than of S1-3. The higher weight ratio, the more number of ball mill. Presence of more than one ball mill in a vial results in a decrease in collision energy of each ball mill. The average particle size of the S1-3 sample obtained is smaller than S3-3 as a result of the increasing of collision energy of each ball mill. Particle breakage may take place by the collision between entering balls to the charcoal powder, the balls existing already in the charcoal powder and the balls to the refractory container wall. The collision between newly entering balls to the charcoal and the balls existing already in the charcoal which would be the most efficient source of the particle size reduction[12].

Table 2. The average particle size of the natural wood charcoal samples with different synthesizing techniques.

| Sample code	Synthesizing technique	Average particle size (µm)		Standard deviation (µm)	
		Length	Width	Length	Width
53 µm	Crushing by hand (manually)	27.24	25.70	9.42	8.42
74 µm	Crushing by hand (manually)	24.91	26.24	9.99	8.15
105 µm	Crushing by hand (manually)	84.47	80.04	43.44	32.63
S1-3	Ball mill (weight ratio 2:1, 30 hours)	5.41	4.98	3.16	2.37
S3-3	Ball mill (weight ratio 3:1, 30 hours)	12.16	13.62	4.77	4.77
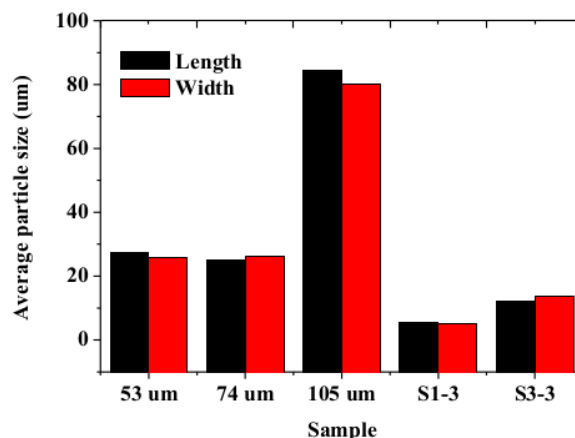
Figure 1. The average length and width of the synthesized samples.

3.2. Thermal behaviors

Thermal analysis has been widely used to get thermal behaviors of materials. The thermal behaviors of material can be obtained using thermogravimetric analysis (TGA). The TGA is a well proven thermal analysis method for measuring mass changes in physical and chemical properties as a function of temperature or time. In this study, thermal behaviors are measured as a function of increasing temperature with constant heating rate. Changes in mass of materials attributable to various thermal process. These thermal process are included absorption, desorption, vaporization, oxidation, reduction and decomposition. The quasi-static TGA type are usedto analyse the thermal behaviors due to the samples are heated with a constant weight at each of a series of increasing temperature.

3.2.1. Thermogravimetric (TG/DTG) curveof natural wood charcoal

Thermogramsobatained from the TG/DTG analysis as a function of temperature for the crushed by hand (53, 74 and 105 μm) and milled by ball mill samples(S1-3 and S3-3) are shown in figure 2 and 3. As can be seen in figure 2 and 3, all the samples showed the linearly decreased curves. Temperature stage and weight loss of each sample are explained in sect 3.2.2 and 3.2.3.
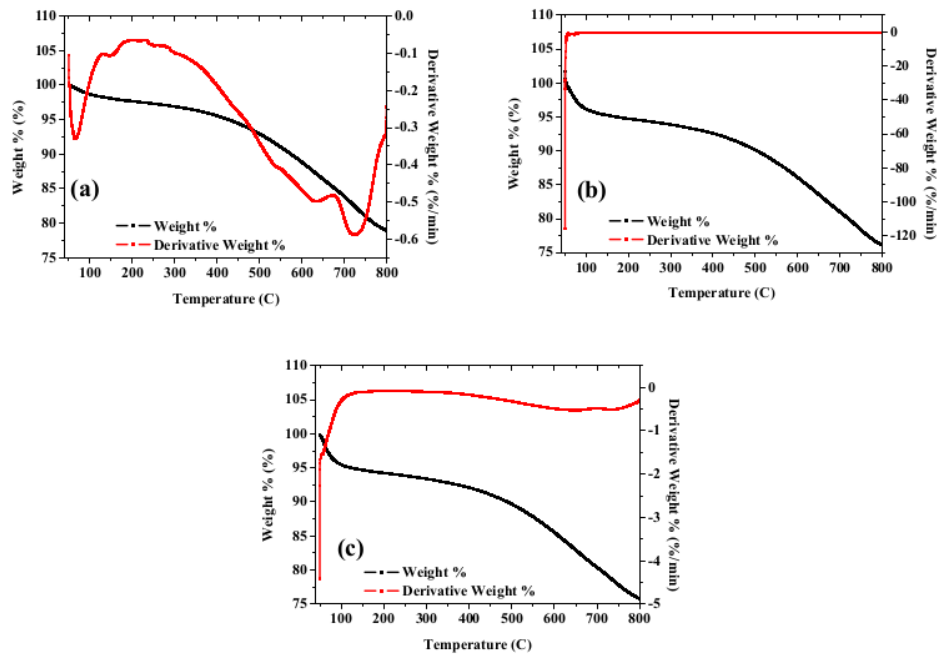
Figure 2. TG/DTG curves of the crushed by hand samples; (a). 53 μm, (b). 74 μm, (c). 105 μm.
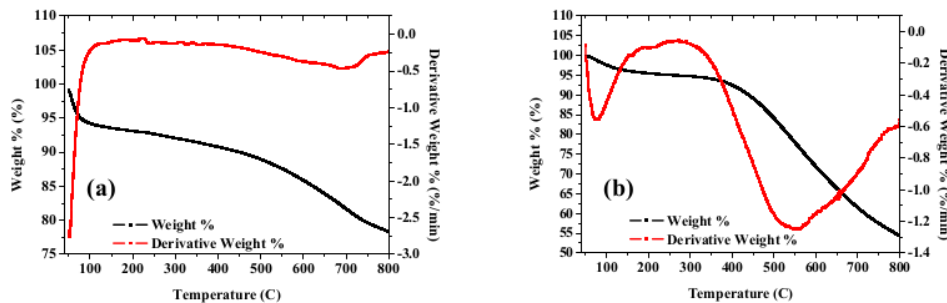
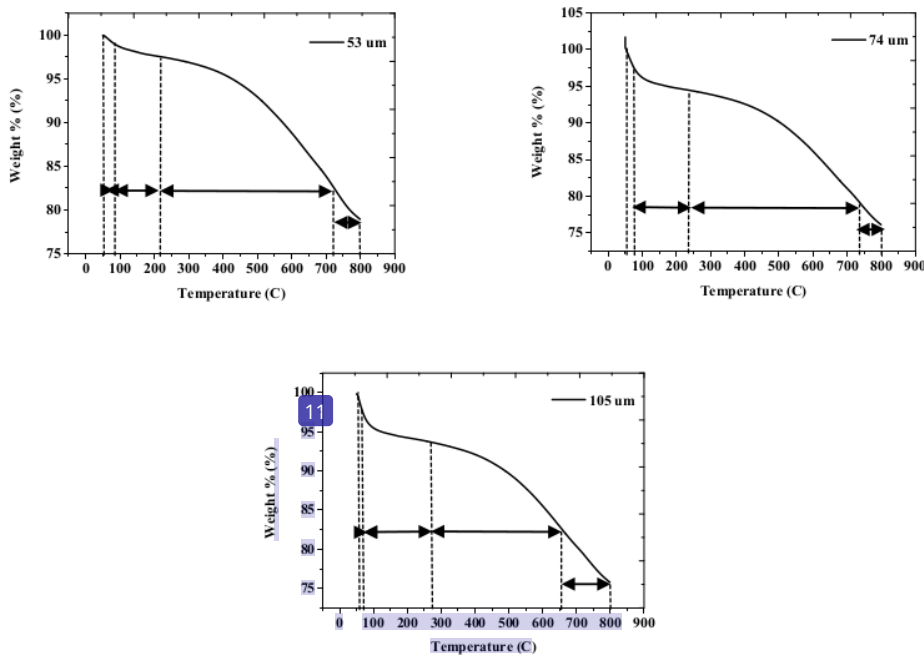**Figure 3**. TG/DTG curves of the milled by ball mill samples; (a). S1-3, (b). S3-3.

3.2.2. Temperature stage of natural wood charcoal

Figure 4and 5shows the temperature range curve of the manually crushed and ball milled samples per each stage. The decomposition behavior of both the manually crushed and milled samples takes place in four stages. It is observed that the initial and final temperature for all samples are about 50and 799°C, respectively. The S3-3 and 74 μm samples showed the lowest and the highest temperature range in fourth stage. The third stage exposed the longer of temperature range. The S3-3 sample occurred the fastest of temperature range at the fourth stage. The 53 μm sample had the latest

temperature range at the fourth stage. Further discussion about the weight loss for each stage is in sect. 3.2.3. According to the previous studies, the decomposition behavior of material takes in four steps [13].The detailed temperature range for each stage are presented in Table 3.

Table 3. Temperature range of the natural wood charcoal samples.

Sample	Temperature range (^{o}C)			
	First stage	Second stage	Third stage	Fourth stage
53 µm	50-83.69	83.69-204	204-724	724-798.92
74 µm	50-60.93	60.93-240	240-738	738-798.94
105 µm	50-57.64	57.64-268	268-649	649-798.96
S1-3	49.5-49.79	49.79-319	319-689	689-799.03
S3-3	49.9-72.58	72.58-264	264-555	555-799.11

Figure 4. Temperature stage curves of the crushed by hand samples.
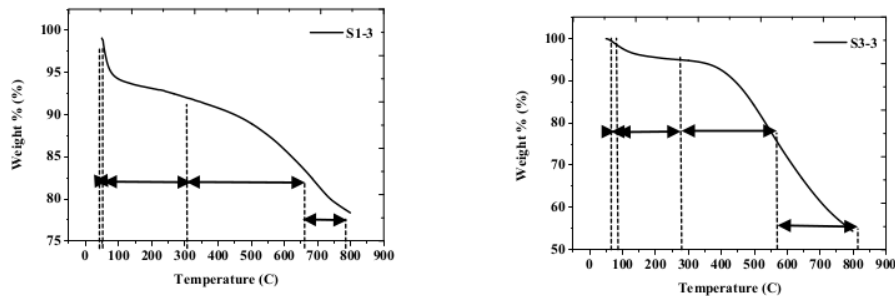
Figure 5. Temperature stage curves of the milled by ball millsamples.

Biomass has three major components i.e. hemicellulose, cellulose and lignin. Hemicelluloses has linear polymer structure with short side chain. Cellulose is constructed of semicrystalline arrays chains. As for lignin, it is a complicated structure of phenolic polymer encasing the polysaccharides of the cell walls that produces strong and durable composite materials[14,15]. Hemicelluloses decomposes at between 200 to 350°C, cellulose from 305 to 375°C. Lignin occurs from temperature as low as 150 up to 900°C since it is more thermally stable in contrast to cellulose and hemicelluloses[16].

3.3. Weight loss of natural wood charcoal

Figure 6 shows the weight changes of the manually crushing and ball milling samples as a function of decomposition stage. The weight loss of the manually crushing samples as the effects of the increasing temperature for the first stage were 0.97% (53 µm53 µm), 2.68% (74 µm) and 0.86% (105 µm). The highest and the lowest weight loss were 74 and 105 µm samples, respectively. The ball milled samples (S1-3 and S3-3) were 0.09 and 0.87% (first), 9.68 and 17.62% (second), 9.7 and 17.7% (third) and 3.83 and 22.93% (fourth). The weight loss total were 23.3 and 59.12%. The S3-3 samples gave the highest weight loss. The weight losses are found to be relevant to the composition of cellulose, hemicelluloses and lignin fractions in biomass. The first stage involves the loss of moisture content in the sample. The second stage is related to the release of volatiles resulting from the decomposition of hemicellulose. The third stage is characterized by decomposition of cellulose and lignin [17].
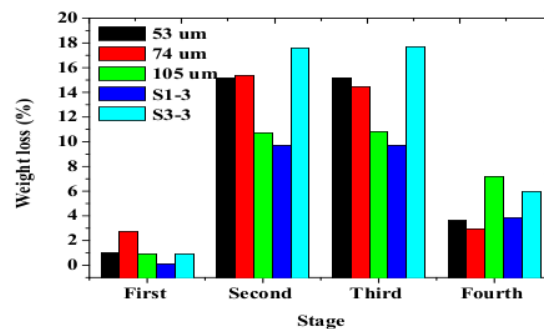

Figure 6. Weight loss of the natural wood charcoal samples.

4. Conclusions

A study on carbon-based material produced from natural wood charcoal was held to investigate the effects of nanostructured material synthesizing techniques on the particle size reduction and the thermal behaviors. The results showed a significant effect of the synthesizing techniques on the particle size reduction and the thermal behaviors. The milled by ball mill samples (S1-3 and S3-3) developed smaller particle size reduction (5.41 and 12.16 µm in length and 4.98 and 13.62 µm in width). According to the results, it is concluded that preparing by ball mill techniques gives the optimum in particle size reduction. The characterization of thermal behaviors showed temperature range for decomposition stages. The samples occurred in four stages. The weight loss results indicated decomposition of the samples were affected by the composition of samples (cellulose, hemicelluloses and lignin). This study can be useful for the synthesizing of nanostructured materials.

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