Preparing the carbon-based material with different milling

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Preparing the carbon-based material with different milling settings to change the morphology and crystalline structure

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Abstract. Carbon-based material (CBM) were prepared from charcoal natural wood as material. It was milled by a planetary ball mill with varied weight ratio of ball mill in range from 2:1 – 3:1 and milling time at 2 hours, 18 hours and 30 hours. Characterization of surface morphology and crystalline were conducted by using Scanning Electron Microscope (SEM) and X-ray Diffraction (XRD). The result showed that the surface morphology of carbon obtained were irregular particle size and dissimilar shape. The weight ratio and milling time affect to the particle size and crystalline structure of natural wood carbon. The minimum particle size obtained is 1.73 µm from 108.7 µm. The smallest particle size obtained was resulted with 18 hours of milling time. The lower crystalline percentage is 4 % (weight ratio 3:1, 18 hours) and the highest is 5.3 % (weight ratio 3:1, 12 hours).

1. Introduction

Carbon-based material (CBM) is one of the prospective porous material for hydrogen storage [1]–[3] . The larger surface area and high adsorption capacity and desorption performance are noble properties of CBM [4]. Other properties are light weight, low-cost and can be produced from plentiful materials in nature such as empty fruit bunch [5], corncob [6] and coffee bean wastes [7].

For many years, most studies have only addressed the issue on carbonization and activated carbon process and examining the effects on the activated carbon properties [8], [9]. Only few researchers started to do the research on the characterization of surface morphology, particle size distribution and crystalline structure of CBM. Whereas these characterization has become an important issue in fabrication of carbon material for some applications.

This aims of this study is to examine the surface morphology, particle size distribution and crystalline structure of carbon from natural wood with varied in weight ratio 2 : 1 and 3 : 1. A planetary ball mill was used to reduce the particle size with milling time of ball milling machine also varied in 12, 18 and 30 hours.

2. Research Method

2.1. Material and preparation

The charcoal was used as carbon-based material. The material was obtained from Spreading International Co.,Ltd products (Bangkok, Thailand). It consisted of charcoal lump made from natural wood were purchased from market in Tainan city, Taiwan. For preparation purposes of material, the

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charcoal lump was milled by a plannetary ball mill machine (Model SE-PM 4L) with 5.01 mm of ball mill diameter. Ball milling method (BMM) was employed to obtain fines particles of charcoal in microscale. The ball mill consists of Z_rO_3 (94.5 %), Y_2O_3 (5.1 %) and Al_2O_3 (0.25 %). The machine was operated for 12, 18 and 30 hours with the constant revolution speed of 300 rpm at room temperature.The weight of ball mill and charcoal were varied in different ratio (2 : 1 and 3 : 1). The weight of natural wood carbon are 35 to 45 gram and 15 gram for ball mill. The particle size of carbon used for a planetary ball milling machine has range > 105 µm.

2.2. Characterization of carbon-based material

The form fine powder of charcoal milled planetary ball mill were analyzed by using the FE-SEM (JEOL JSM-6701F) for observing the surface morphology. Particle size distribution were analyzed by direct observation of SEM micrographs. Crystalline structure were characterized by D2 XRD (Bruker D2 Phaser) from Bruker AXS company equipped with Cu radiation, 20° to 80° of 2Theta with 0.0326 of step size and λ= 1.54184 (°A). The voltage supplied was 30 kV with a current of 10 mA

3. Results and discussion

3.1. SEM micrographs of natural wood carbon

The surface morphology of natural wood carbon varied in different weight ratio of ball mill and natural wood carbon were found in figure 1 (weight ratio 2 : 1) and figure 3 (weight ratio 3 : 1).
SEM micropraph confirmed that the surface morphology of carbon were dissimilar shape and irregular particle size. Particle size distribution and performance descriptive statistics were produced after milling process by using a planetary ball mill are shown in table 1 and figure 2. The average particle size were reduced are 6.67 µm (12 hours), 6.59 µm (18 hours) and 4.79 µm (30 hours) from 170.5 µm (>105 µm). The minimum particle size obtained are 4.97 µm (12 hours), 1.73 µm (18 hours) and 2.42 µm (30 hours) from 108.7 µm (>105 µm). The smallest particle size obtained was resulted with 18 hours of milling time.

Table 1. Performance descriptive statistics of natural wood carbon with weight ratio 2 : 1

Milling time (h)	Mean	Standard Deviation	Sum	Minimum	Median	Maximum
Before Milled	170.5	51.53	1534.6	108.7	161.9	287.7
12	6.76	1.86	40.49	4.97	6.1	9.81
18	6.59	4.84	46.08	1.73	6.06	16.46
30	4.79	3.45	47.81	2.42	3.71	13.97

Table 2 shows the average of particle size for the weight ratio 3 : 1. The size of particle size are 5.05 µm (12 hours), 5.77 µm (18 hours) and 5.77 µm (30 hours). The minimum particle size obtained are 1.07 µm (12 hours), 1.79 µm (18 hours) and 1.46 µm (30 hours) from 108.7 µm (>105 µm). The smallest particle size obtained was resulted with 12 hours of milling time. The analysis did not show any significant difference the effect of milling time on the average and minimum of particle size milled. These results offer invaluable evidence for using the ball milling method. The ball milling method is one of the method that can be used to make particle size reduction. The particle size were smaller than before crushing with a plannetary ball mill machine. Size reduction of particle occurred due to the effect of deformation and destruction of ball mill and carbon. The higher of weight ratio, the smaller particle size resulted. The longer of milling time with the same weight ratio, the smaller of particle size milled.

Table 2. Performance descriptive statistics of natural wood carbon with weight ratio 3 : 1

Milling time (h)	Mean	Standard Deviation	Sum	Minimum	Median	Maximum
Before Milled	170.5	51.53	1534.6	108.7	161.9	287.7
12	5.05	2.92	50.48	1.07	5.325	10.21
18	5.77	3.29	46.14	1.79	5.73	11.65
30	5.77	3.29	57.61	1.46	4.865	15.84

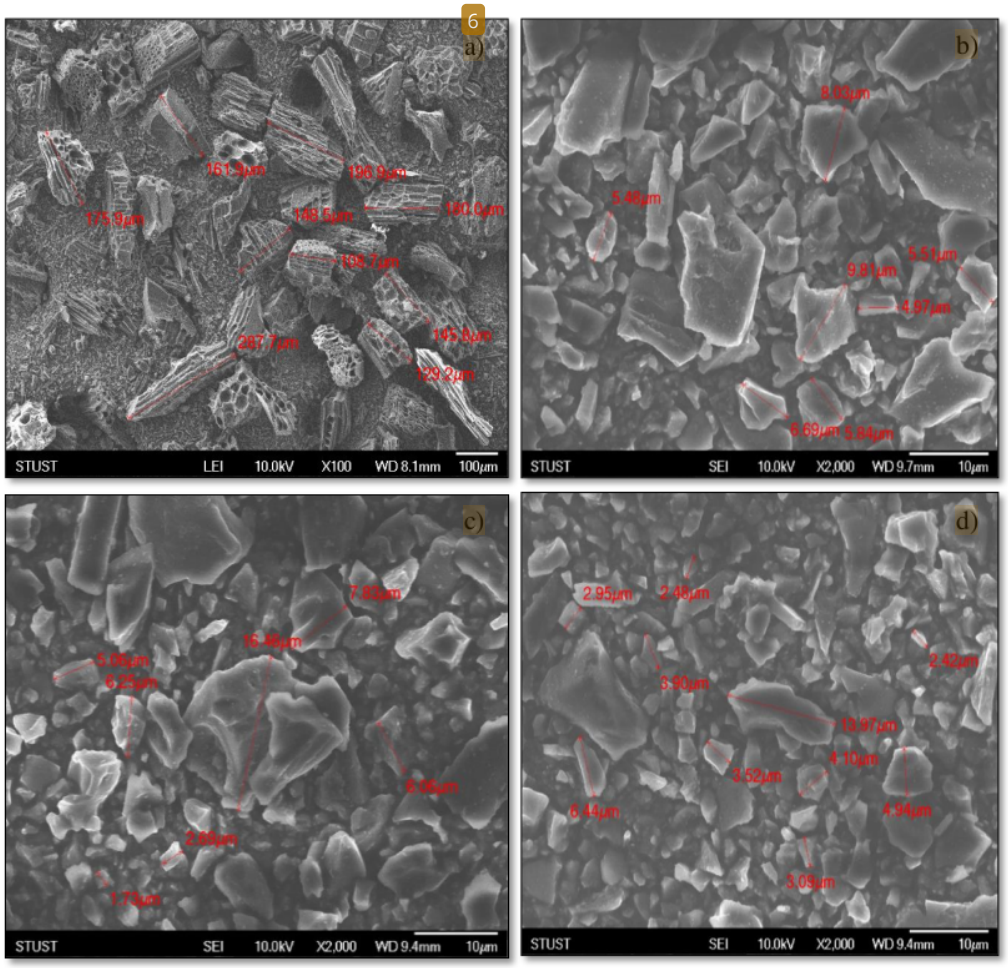

Figure 1. SEM micrographs of natural wood carbon with weight ratio 2 : 1, a). Before milled, b). 12 hours, c). 18 hours, d). 30 hours

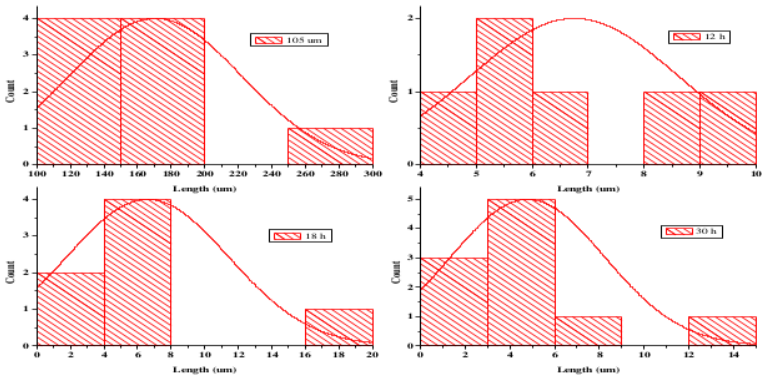

Figure 2. Particle size distribution of natural wood carbon with weight ratio 2 : 1

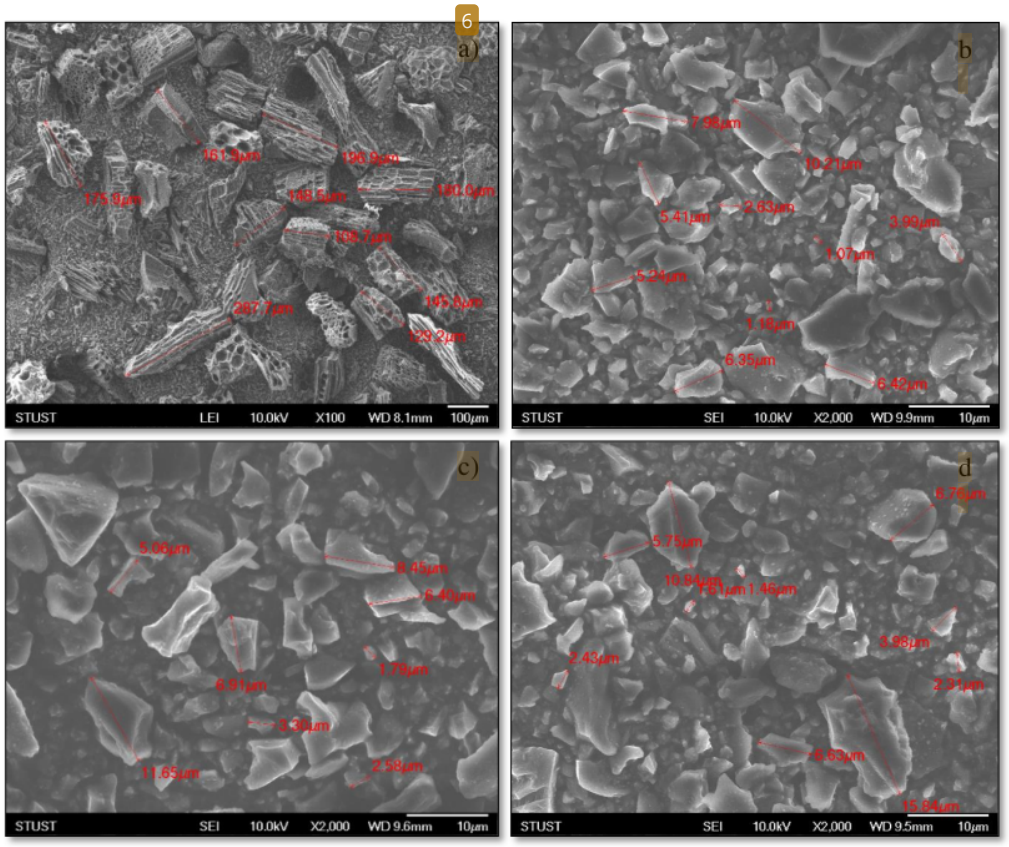

Figure 3. SEM micrographs of natural wood carbon with weight ratio 3 : 1, a). Before milled, b). 12 hours, c). 18 hours, d). 30 hours

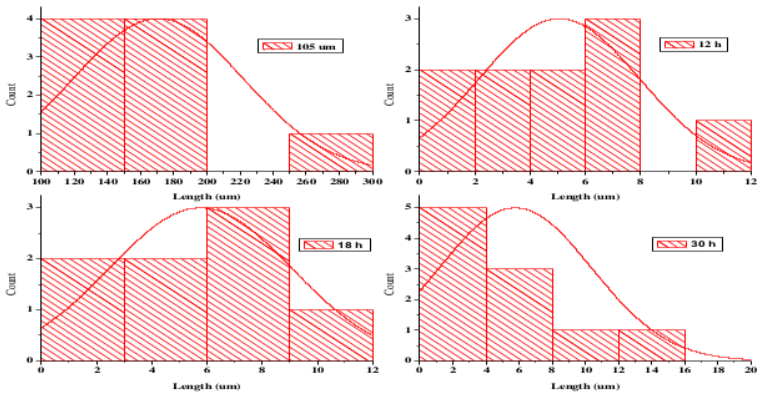

Figure 4. Particle size distribution of natural wood carbon with weight ratio 3 : 1

3.2. XRD pattern of of natural wood carbon

The peak intensity of crystalline structure of natural wood carbon was studied by milling time varied in 12, 18 and 30 hours. The peak intensity of carbon was determined by XRD technique are presented in figure 5 (weight ratio 2 : 1) and figure 6 (weight ratio 3 : 1). Figure 5 shows that the intensity is slowly decreased. The lower peak intensity is in carbon made in 12 hours and the higher peak intensity is in 30 hours.

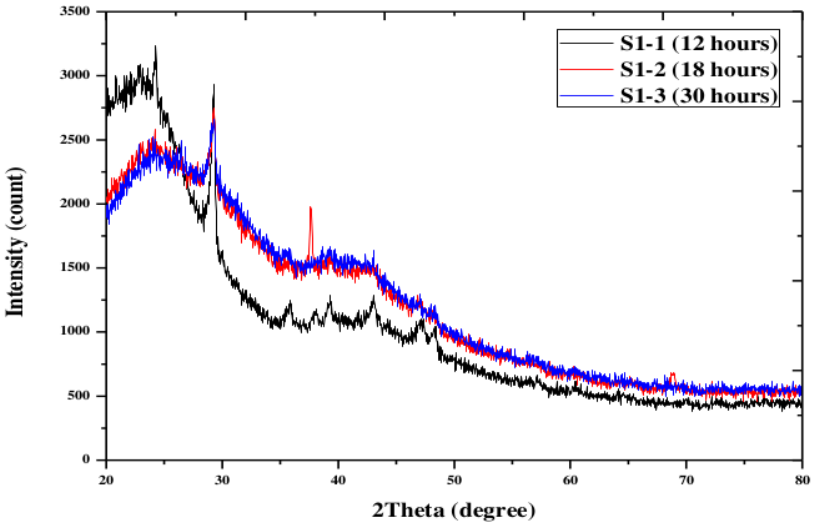

Figure 5. XRD patterns of natural wood carbon with weight ratio 2 : 1 at different milling time

Figure 6 depicts the intensity on different milling time. As seen in the figure 6, the trend of peak intensity of carbon is slowly decresed. The higher and lower peak intensity are carbon made in 30 hours and 12 hours of milling time respectively.

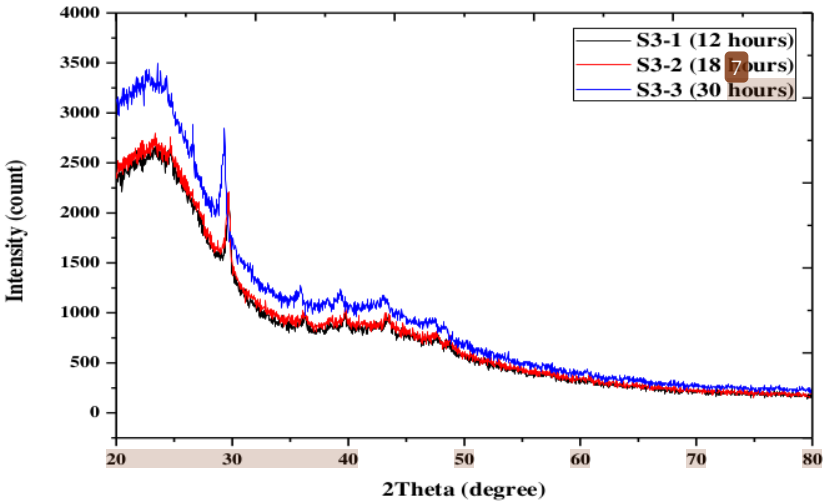

Figure 6. XRD patterns of natural wood carbon with weight ratio 3 : 1 at different milling time

3.3. Crystalline structure of natural wood carbon

The crystalline percentage of natural wood carbon for different weight of ratio and milling time are presented in figure 7 and figure 8. The crystalline percentage designates the structure of carbon. The lower of crystalline percentage indicates the most amorphous and porous structure. In this study, the crystalline percentage of weight ratio 3:1 is higher than 2:1. The higher and the lower of crystalline percentage are 5.3 % and 4.1 %. Based on the crystalline percentage, the natural wood carbon milled with weight ratio 2 : 1 is more amorphous and porous structure. The milling time has no high effect on the crystalline percentage for weight ratio 2 : 1.

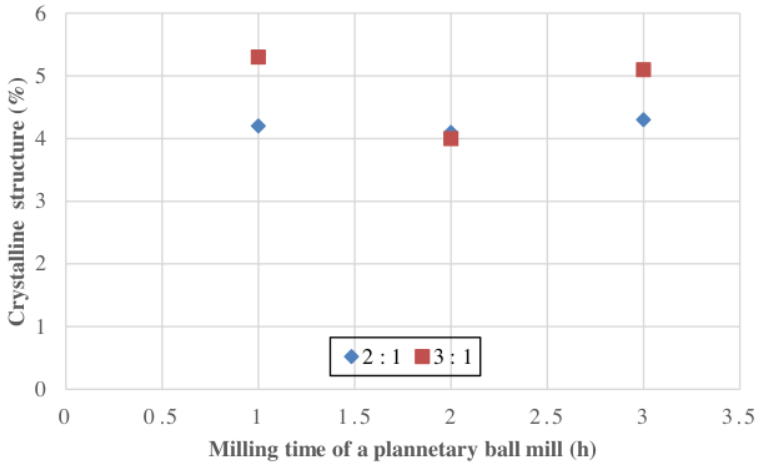

Figure 7. Crystalline structure of natural wood carbon at different of weight ratio

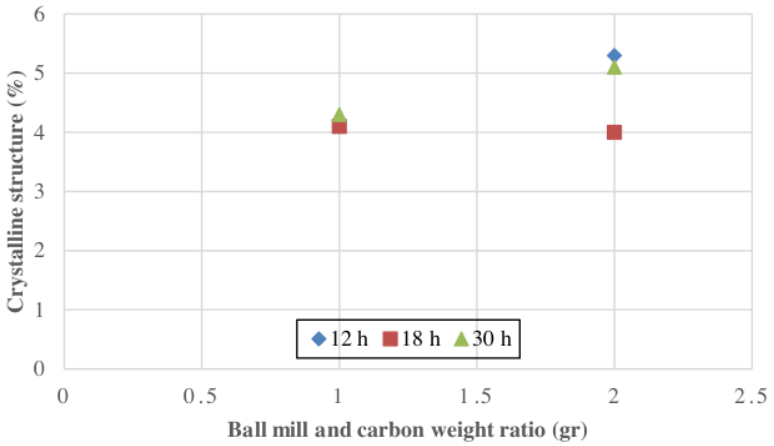

Figure 8. Crystalline structure of natural wood carbon at different of milling time

4. Conclusions

In this study, we investigated the natural wood carbon milled by ball mill machine. The particle size decreased after milling process. The particle size decreases with the higher of weight ratio. The higher of weight ratio, the smaller particle size formed. The longer of milling time with the same weight ratio, the smaller of particle size refined. The crystalline structure increases with the higher of weight ratio.

Acknowledgements

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