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Evaluation of Ball-Milling Process for the Production of Carbon Particles from Rice Straw Waste

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ABSTRACT

The purpose of this study is to evaluate the ball-milling process for the production of carbon particles from rice straw waste. In the experimental method, carbon particles are prepared by adding heat treatment to the rice straw waste at the temperature of 250ºC. The heated rice straw waste is grinded using a conventional grinding method and put into the ball-milling process. Physicochemical properties of carbon particles are evaluated before and after the ball-milling process. The results indicate that sizes of carbon particles decrease significantly from micro (after the conventional grinding process) to submicron range (after additional ball milling process). Almost no changes in the chemical properties of carbon particles are observed. This result implies that the ball-milling process is effective to decrease the size of the prepared carbon particles. The collision phenomenon combined with shear stress among the stainless steel balls is the main reason for breaking carbon particles, making the particle sizes smaller.

Keywords: Activated carbon particles, ball-milling process, rice straw waste

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INTRODUCTION

One of the best and popular methods for reducing particle size, known as a top-down method, is a ball-milling process. This process has been widely used for various materials, such as organo-vermiculite (Wang et al., 2011), Bi_3NbO_7 ceramics (Zhou, Wang, & Yao, 2007), nanocrystalline ZrO_2 (Chadwick, Pooley, Rammutla, Savin, & Rougier, 2003), Pb $(Zr_{0.52}Ti_{0.48})O_3$ (Kong, Zhu, & Tan, 2000), magnetic nanoparticles (Chakka, Altuncevahir, Jin, Li, & Liu, 2006), WO_3/TiO_2 photocatalyst (Shifu, Lei, Shen, & Gengyu, 2005), and carbon nanotubes (Pierard, et al., 2001). This method is effective to reduce particle size down to submicron and further to nanometre range. Although the experimental procedures have been well-documented, reports on the use of ball-milling process to prepare carbon from rice straw waste are relatively rare.

Previous studies (Permatasari, Sucahya, & Nandiyanto, 2016; Nandiyanto et al., 2016a, 2016b, 2017a, 2017b, 2017c; Nandiyanto, Zaen, Oktiani, Abdullah, & Danuwijaya, 2018; Nandiyanto, Zaen, & Oktiani, in press), developed a method for the production of particles with controllable physicochemical properties. Control of sizes from nano to submicrometre range was also discussed.

Based on the previous works, the objective of this study is to evaluate the impact of ball-milling process on the changes of physicochemical properties of carbon particles gained from rice straw waste. To support the objective of this study, the physical appearance of the carbon particles prepared via the ball-milling process is analysed using a scanning electron microscope (SEM), whereas the chemical properties are evaluated using an X-Ray Diffraction (XRD).

Why Rice Straw Waste is an Excellent Candidate for Carbon Source?

Rice straw is used as a candidate for producing carbon particles (Karyasa, 2016; Permatasari et al., 2016; and Nandiyanto et al., 2016a). It offers many advantages, reducing serious environmental problems gained from rice straw waste. A large amount of rice straw increases as the production of rice continues. Although this abundant rice straw as by-products can be minimised by disposing and burning in the rice field, the burning process is not effective since the secondary product is still persisted (Nandiyanto, Zaen, & Oktiani, 2017d). Thus, understanding how to solve or reuse this material is inevitable.

Rice straw is rich in organic nutrients (such as cellulose (32-47%), hemicellulose (19-27%), lignin (5-24%), and ash (13-20%) that can be converted into carbon material (Nandiyanto et al., 2018). This is another main reason for the selection of rice straw waste as one the excellent candidates for carbon sources. Previous studies have reported the preparation of carbon from rice straw using chemical activation, pyrolysis, and temperature treatment. This information has been reviewed by Ioannidou and Zabaniotou (2007). Although their method is effective, there is no information how to reduce the carbon particle size using the ball-milling process. In fact, reducing particle size has a great impact on improving material performance, such as surface area.

Illustration for the Production of Carbon from Rice Straw Waste

Figure 1 shows an illustration mechanism for converting rice straw waste into carbon particles. The process involves two steps. The first is heat treatment, which is an effective method to convert an organic component of rice straw waste into carbon. The next step is ball-milling process, in which this method is used to decrease the particle size of the heated rice straw waste. Detailed information about the preparation of carbon from rice straw is described in the previous report (Nandiyanto, et al., 2017c).

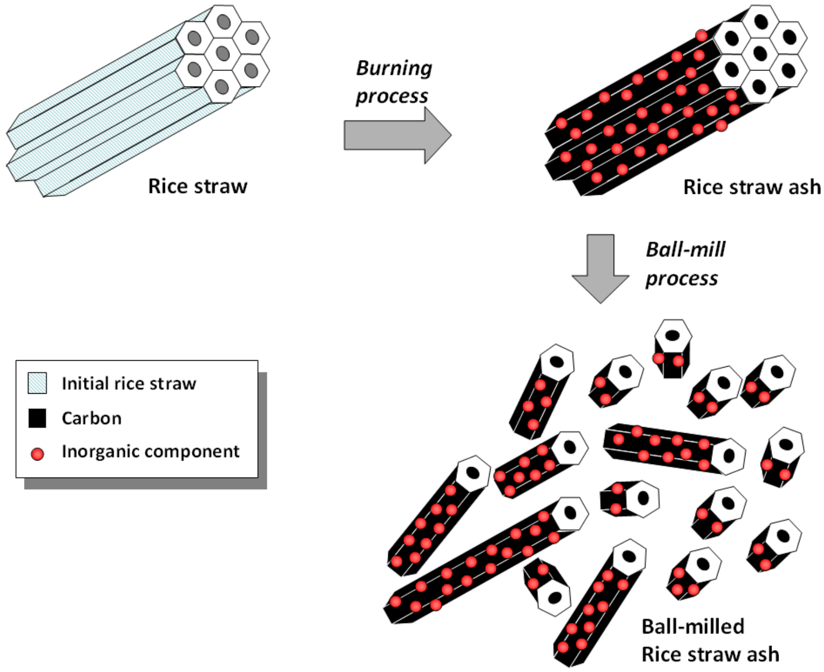

Figure 1. Schematic illustration of the formation of carbon porous particles from rice straw waste

Heat Treatment Process for Converting Rice Straw Waste into Carbon Material

Figure 2 shows the effect of additional heat treatment on the rice straw waste based on the previous studies. To confirm this impact, Nandiyanto et al. (2017c) reported the thermal analysis during the additional heat treatment on the rice straw waste. The analysis showed the detection of mass reduction during the heating process with existence of several exothermic and endothermic reactions. They concluded that some phenomena happened during the heating process (Nandiyanto et al., 2016a, 2017c):

(i) Evaporation of physically adsorbed water at temperature of 20-100°C;

(ii) Carbonisation from cellulose and hemicellulose degradation process at temperature of 250-320°C;

(iii) Carbonisation from lignin degradation process at temperature of 330-470°C;

(iv) Carbon degradation due to oxidation at temperature of higher than 500°C.

According to above results, the effective temperature used in the heat treatment process of rice straw to produce carbon material is 250°C. This temperature is considered optimum to obtain carbon in the highest volume. If the temperature applied is higher than the optimum temperature condition, it can create two disadvantages; one is wasting thermal energy and the other is oxidation process that can deter carbon volume. Although some processes show excess temperature can increase the physicochemical properties of the prepared carbon due to its impact on activating carbon, the present study will not focus on the activation process.

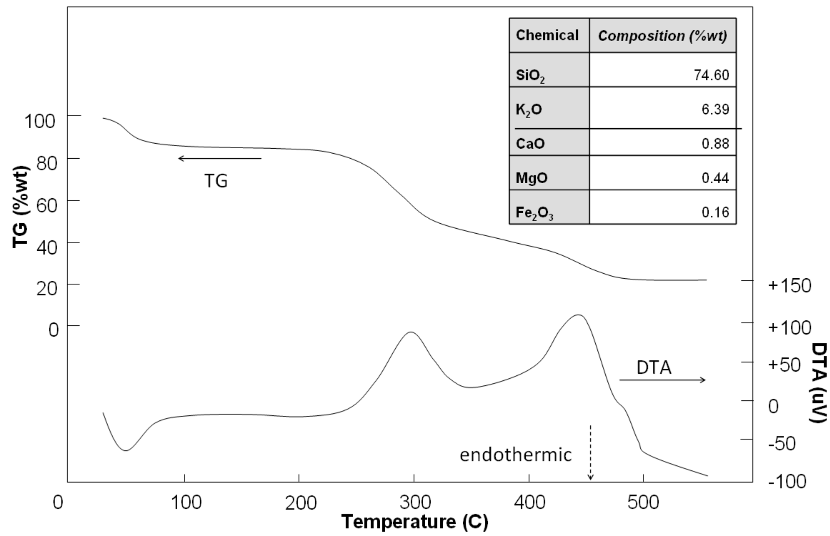

Figure 2. Thermal analysis results of rice straw waste. The insert table shows the chemical composition of rice straw. From Nandiyanto et al., 2016b

Technical Information about Ball-milling Process

When applying heat treatment to the rice straw waste, the prepared carbon is a replication of the initial size of the rice straw waste. Therefore, to create smaller particles, the rice straw waste must be sliced very thinly. However, cutting rice straw waste into smaller size is difficult since the rice straw is relatively pliant. Thus, some researchers add mechanical milling process after the burning rice straw, such as ball-milling process, for creating smaller carbon particles.

In short, illustration of the ball-milling process is shown in Figure 3. The ball-milling process is conducted by adding material and balls (as a material destroyer) into the milling vessel (see Figure 3(a)). During the ball-milling process (See Figure 3(b)), the balls inside the ball-milling machine cylinder vessel are rotated. During rotation, balls move and create shear stress. Then, when the ball reaches a certain point, it falls due gravitational force. The falling ball then overwrites other balls, creating collision process. Indeed, a combination between the "shear stress" and the "collision process" gives mechanical force and leads to the creation of particles with smaller sizes.

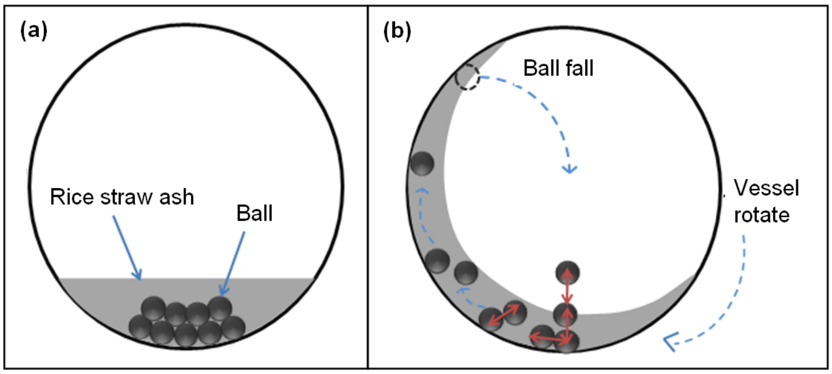

Figure 3. (a) The illustration of ball-mill processing in the initial condition and (b) after rotation process/ during ball-milling process

EXPERIMENTAL METHOD

The material used for the preparation of carbon material was rice straw waste gained from rice field in Kebumen, Indonesia. In the experimental procedure, the carbon particles were prepared using several steps: (i) heat treatment process; and (ii) ball-milling process.

Heat Treatment

Rice straw waste was washed, cut into sizes of about 1 cm, and dried at 100°C. The dried rice straw was then heat-treated at 250ºC in an electrical furnace. The heat-treated rice straw was grinded using a conventional grinding method to form carbon particles.

Ball-milling Process

The grinded carbon particles are put into a 1.42 L of batch-typed ball-milling vessel equipped with stainless steel balls (diameter of 8 mm). Ball-mill vessel is made from stainless steel with dimensions of 25 and 8.50 cm for length and diameter respectively. Ball-milling process is conducted at a room temperature with rotation speed of 70 rpm for 30 minutes. Illustration of the ball-milling process is shown in Figure 3.

Characterisation

The physical appearances of the produced carbon particles are analysed using an electron microscope (SEM; JSM-6360LA; JEOL Ltd., Japan). The chemical properties of the produced carbon particles are characterised using an X-ray diffraction (XRD; PANalytical X'PertPRO; Philips Corp., Netherland).

RESULTS AND DISCUSSION

Figure 4 shows the SEM analysis images of the prepared carbon particles before and after the ball-milling process. The differences in shape and size are observed. Carbon particles before ball-milling process have a homogeneous shape that is rectangular with a length of between

1800 and 3750 nm (Figure 4(a)). The size of the carbon particles is identical to the size of the initial rice straw waste, confirming the carbon particles are the replication of rice straw waste. Then, applying ball-milling process to the carbon resulted in the decreases in the particle size, in which the particles have more diverse and irregular shape with a length of between 125 and 625 nm (Figure 4(b)).

Based on the above results, we can conclude that the conventional grinding process is effective to create micrometer-sized particles. However, this method has limitations due to ineffectiveness of the production of particles with sizes of submicron or further nanometre range. The additional ball-milling process is decrease the particle size down to hundreds of nanometres. Differences in shape and size gainded from above methods (i.e. conventional grinding method and ball-milling process) are due to the existence of shear stress and collisions among stainless steel balls during the ball-milling process.

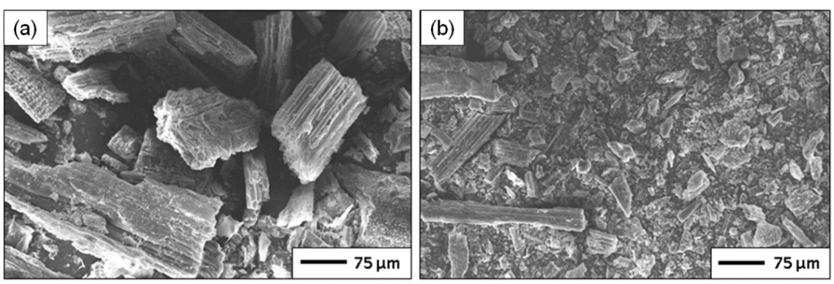

Figure 4. SEM images of carbon particles from rice straw waste (a) before and (b) after ball-milling process

In order understand the phenomenon that occurs during the ball-milling process, Figure 5 illustrates the size destruction mechanism during ball-milling process. In this figure, collision between balls is focused, while the shear stress is assumed to have less impact than the ball collision.

The collision between two stainless steel balls pressures the carbon to break into smaller parts. Since the colliding process continuously occurs, the carbon faces continuous collision, resulting in more size destruction to happen. Indeed, the final particle sizes become smaller.

The breaking characteristics of carbon particles depend on the position of the carbon material when the collision between the stainless steel balls takes place. If the collision occurs at the center of the carbon material, the carbon would break in the middle parts, permitting formation of two equal lengths and some smaller irregular parts of carbon particles (see Figure 5(a)). Indeed, when the collision occurs at the edge of the carbon material, the edge of the carbon turns into smaller irregular parts (see Figure 5(b)). If the collision occurs in the vertical position of the activated carbon, the carbon graphite breaks into some irregular parts (see Figure 5(c)). When the collission process continously occurs, the final particles have smaller sizes with various shapes. Indeed, the sizes and shapes of ball-milled carbon are different compared with that of originated carbon, as shown in Figure 4(b)).

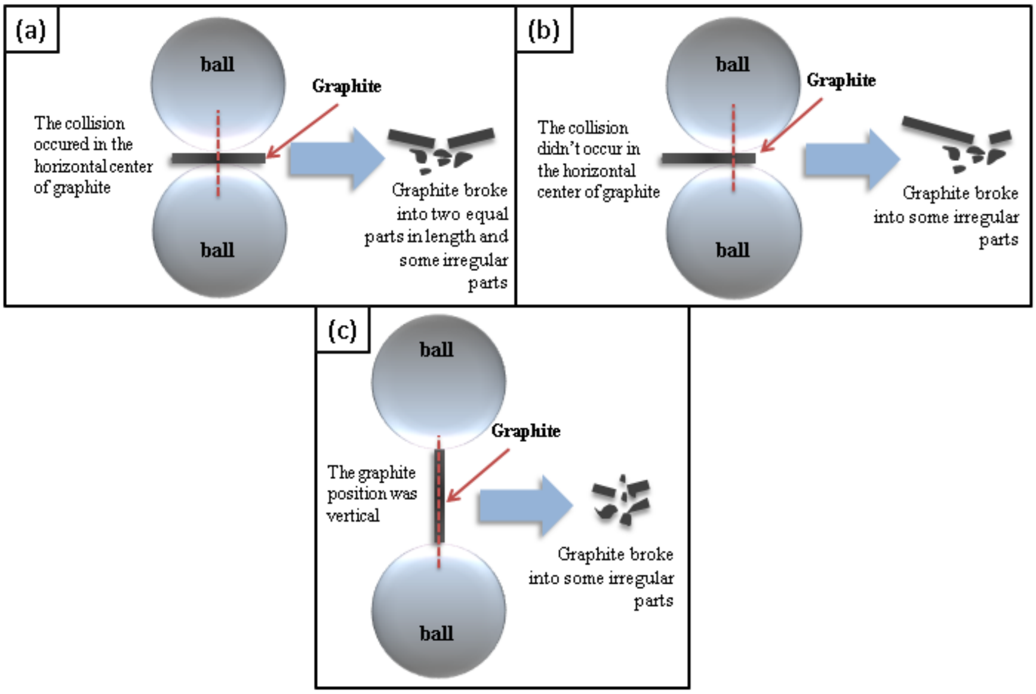

Figure 5. The process of rupture of graphite due to the collision of stainless steel balls in the ball milling process. (a) The collision occurring in the horizontal center, (b) does not occur in the horizontal center, and (c) when the graphite position is vertical

Figure 6 shows XRD analysis results of the prepared carbon particles. Three samples were analysed. The first sample was heat-treated rice straw waste. The next sample undergone additional conventional grinding process on the heated rice straw waste. Finally, the third sample undergone additional conventional grinding with ball-milling processes on the heated rice straw waste. All samples were tested based on the Joint Committee Powder Diffraction System (JCPDS) for graphite (JCPDS No. 75-1821), silica (JCPDS No. 27-1402), and calcium magnesium silicate ($CaMgSi_2O_6$) (JCPDS No. 11-854).

The results show all samples contain graphite, silica, and $CaMgSi_2O_6$. The content of silica and $CaMgSi_2O_6$ is probably derived from the initial rice straw as the previous described in our previous reports (Nandiyanto et al., 2016b) using atomic absorption spectrophotometry and gravimetric analyses. No additional pattern appeared in all samples, confirming no chemical reaction during the grinding and ball-milling process. However, the intensities of graphite, silica, and $CaMgSi_2O_6$ change during grinding and ball-milling process. When adding grinding and ball-milling processes, intensity of graphite decreases followed by the increasing intensities of silica and $CaMgSi_2O_6$.

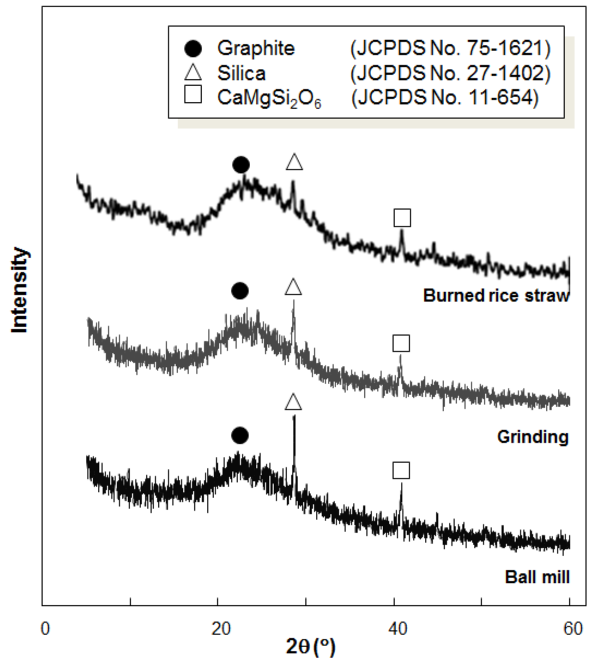

Figure 6. XRD analysis result of burnt rice straw, activated carbon by grinding, and activated carbon by ball milling

To confirm the analysis of the chemical component during the grinding and ball-milling process, component analysis based on pattern area in the XRD result was conducted (See Table 1). The result shows that graphite composition decreases from about 90 to about 84%. The decrease in graphite composition reached more than 6%. This decrease of the graphite composition was followed by the increases in the silica and $CaMgSi_2O_6$ of 4.91 and 1.38% respectively.

Two hypotheses can explain the change in the composition. The first hypothesis is due to the possibility of carbon attached to the balls and the ball-milling apparatus. However, this hypothesis cannot be accepted clearly. If the carbon is attached to the balls and the ball-milling apparatus, this should be followed by a decrease in yield and this will not be composition. The second hypothesis is formulated based on the existence of oxidation process during the ball-milling process. However, the present ball-milling process was conducted in batch process. No circulation of air happens inside the ball-milling process. Further, the process is fixed at room temperature, deterring the possibility of oxidation process in this temperature range.

In addition, the use of batch-typed ball-milling process is the best, compared with that of continuous-typed ball-milling process. The existence of oxygen can be minimised. Indeed, this will deter the possibility of the oxidation process as discussed in the second hypothesis. However, based on both hypotheses, further analyses must be conducted and will be done in our future work.

Table 1
Percentage (%) of intensity from rice straw prepared by burning, grinding, and ball-milling process

Component	Rice straw waste		
	Burned	Grinded	Ball-milled
Graphite	90,45%	88,36%	84,16%
Silica	7,54%	8,41%	12,45%
$CaMgSi_2O_6$	2,01%	3,23%	3,39%

CONCLUSION

The results show the ball-milling process is effective in producing smaller activated carbon particles. By applying the ball-milling process, the size of the carbon particles decreases from micrometer to sub-micrometre range. Interestingly, the particle sizes can be decreased to 125 nm. In addition, there are no different material compositions between samples with grinding and ball-milling process. This confirms no chemical reaction during the grinding and ball-milling process. The main reason for this successful production of sub-micrometre particles when applying ball-milling process is due to the existence of shear stress and collision phenomena among stainless steel balls and carbon material.

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