

EFFECT OF pH CONDITION ON THE PRODUCTION OF WELL-DISPERSED CARBON NANOPARTICLES FROM RICE HUSKS

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Abstract

Researches on the preparation of carbon nanoparticles have been well-documented, however, information regarding the reports on how to make them well-dispersed in a facile way is still lack. Therefore, the aim of this study was to investigate the simple method to produce well-dispersed carbon nanoparticles in the aqueous solution. In this study, carbon nanoparticles were produced using rice husk. Then, the produced carbon nanoparticles were dispersed in the aqueous solution by manipulating pH solution. To ensure the effect of pH solution on dispersing carbon, we varied pH in the range of between 2 and 12. The results showed that carbon nanoparticles can be produced by burning and grinding rice straw ash. And, the dispersion process was successful in a certain range of pH. The pH can affect the formation of well-dispersed carbon nanoparticles through the formation of OH functionalization on the surface of the particle. This is confirmed by the observation that low pH can create less dispersion, whereas high pH can bring advantages in the formation of well-dispersed particles. This study brings new ideas for informing a facile way in the dispersing process of carbon nanoparticles and can be used for further development in the dispersing process.

Keywords: Carbon, Nanoparticles, pH, Rice husks, Well-dispersed.

1. Introduction

Carbon has been well-known due to its uses in certain functions, including photosynthesis of aquatic plants [1], as anode material for sodium-ion batteries [2], tracking the flow of organic matter in swamps [3], and as carbon supercapacitors [4]. Many methods have been reported along with information for possible various raw materials [5-9]. However, information regarding the reports on how to make them well-dispersed in a facile way is still lack. Therefore, the aim of this study was to investigate the simple method to produce well-dispersed carbon nanoparticles in the aqueous solution.

In this study, carbon nanoparticles were produced using rice husk. Although reports on the utilization of rice husk have been well-published [10-13], this study was focused on the strategies on producing well-dispersed carbon nanoparticles in an aqueous solution. In short, the produced carbon nanoparticles that were from burning rice husk were dispersed in the aqueous solution by manipulating pH solution. To ensure the effect of pH solution on dispersing carbon, we varied pH in the range of between 2 and 12.

2. Experimental Method

The method used in this study was divided into two stages: (i) producing well-dispersed carbon nanoparticles from rice husks and (ii) the well-dispersed carbon nanoparticles.

In short, carbon particles were produced by burning rice husks (Jaya Makmur Farm Shop, Bandung, Indonesia) at 200 °C in an electrical furnace. The produced particles were then grinded and saw-milled, using apparatus used in our previous study [14-16], to get carbon powder. The carbon powder then was diluted into 30 mL of water. To manipulate the pH solution, the suspension was mixed and dropped by adding a few drops of acid solution (phosphoric acid (H_3PO_4 ; Bratachem, Indonesia)) or alkaline solution (sodium hydroxide (NaOH; Bratachem, Indonesia)) at room temperature.

In general, to make a solution with $pH < 7$ (acid condition), we added H_3PO_4 solution into a carbon solution, whereas to make a solution with $pH > 7$ (basic condition), we added NaOH solution. To ensure the pH condition mixed homogenously, we put the suspension into a shaker (Vortex Shaker 3000, Wiggins Co., Ltd., China) at a speed of 1500 rpm for 5 minutes.

To remove undispersed particles, the solution was centrifuged (TG16WS High-Speed Benchtop Centrifuge, Zhengzhou Hepo International Trading, Co., Ltd., China; 11,000 rpm for 5 minutes). Several analyses were carried out to characterize carbon microparticles from the result of burning rice husks and well-dispersed carbon nanoparticles in several pH conditions.

The Fourier Transform Infrared Spectroscopy (FTIR-4600, Jasco Corp., Japan) was used to analyse the chemical composition of carbon made from the burning of rice husks. X-Ray Diffraction (XRD; PANalytical X'Pert PRO; Philips Corp., The Netherlands) was used to examine the constituent compounds of the burning carbon.

Scanning Electron Microscope (SEM) (JSM-6360LA; JEOL Ltd., Japan) was used to characterize the morphology of well-dispersed carbon nanoparticles

produced at a certain pH. Then, Energy Dispersive Spectroscopy (EDX-7000/8000; Shimadzu Scientific Instruments Inc., Japan) was used to check the content and percentage of its elemental content in the sample. In the SEM and EDS analyses, a drop of well-dispersed carbon nanoparticles was placed into the wafer silicon and dry in room temperature.

3. Results and Discussion

3.1. Physicochemical properties of carbon particles prepared from rice husks

FTIR analysis was carried out to characterize the chemical composition of the sample [17]. Figure 1 shows the results of FTIR analysis of carbon in the form of a wavelength spectra of % transmittance. From these spectra, several peaks can be detected, which indicate the existence of bonds between atoms of carbon obtained.

Detected peaks indicate chemical bonds from carbon microparticles from burning. The peak at 3325.33 cm^{-1} indicates the presence of NH bonds in the sample. Another peak at 1609.19 cm^{-1} was detected, in which, the peak was a CO bond, confirming that the sample contained carbon.

The peak at 1063.25 cm^{-1} indicates the C-F bond. The value of FTIR wavelength at 798.25 cm^{-1} is a C-Cl bond. The peak at 594.47 cm^{-1} is a C-Br bond [17]. The results of the analysis showed that the sample contains a carbon bond.

To examine the composition of the prepared carbon particles, XRD analysis was conducted (see Fig. 2). Figure 2 shows the results of XRD analysis of the prepared carbon particles, in which, the form of carbon was obtained in the spectra of 27° . This peak indicates the content of graphite in the sample, which is in a good agreement with previous studies [16, 18].

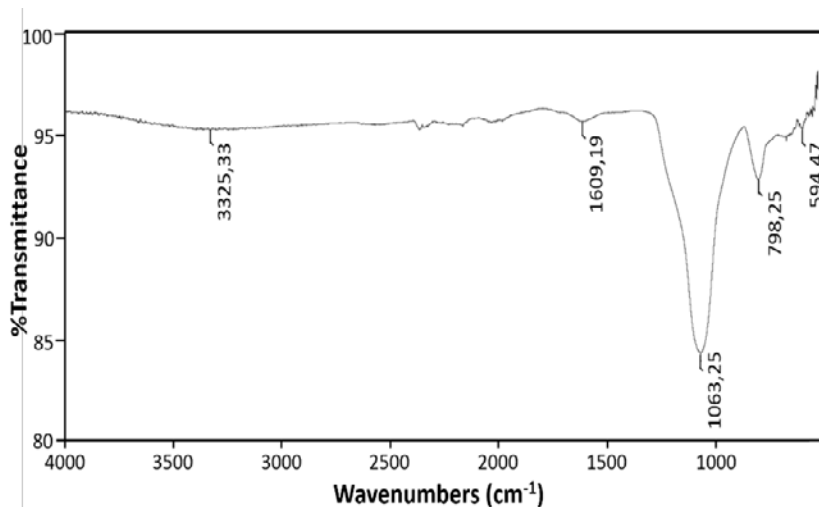


Fig. 1. FTIR analysis result of carbon microparticles from rice husks.

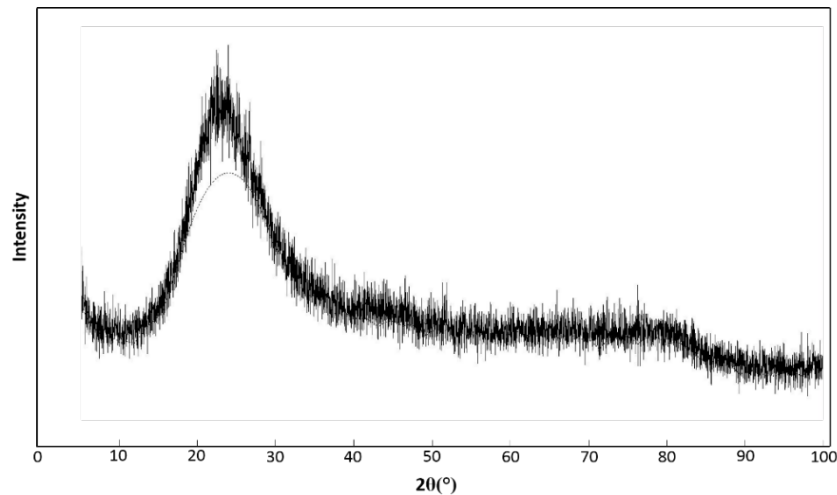


Fig. 2. XRD analysis result of carbon from rice husks.

3.2. Effect of pH on dispersion of carbon

Physical observation of the carbon nanoparticles solution with different pH conditions is shown in Fig. 3. The number panelled in the picture shows the pH value of each solution in the bottle. At pH = 2, the solution is colourless and clearer than the other bottles. At pH = 6, the solution is colourless but more turbid than the solution at pH = 2. The solution with pH = 6 remains denser than the carbon nanoparticles solution at pH = 2.

The higher the pH of the solution, the more turbid for the colour of the solution can be obtained. This confirmed the existence of more carbon nanoparticles dispersed into the solution [13].

Figure 4 shows the results of the SEM test of well-dispersed carbon nanoparticles with pH varies, with a range of between 2 and 12. At pH = 2 (see Fig. 4(a)), particles were aggregating each other. This result is different from samples at pH = 8, 10, and 12. At pH = 8 (see Fig. 4(b)), the carbon observed still forms bulk, but it started to split into smaller particles.

Likewise, with samples at pH = 10 and 12 (see Figs. 4(c) and (d), respectively), more carbon nanoparticles were obtained. This is in a good correlation with our hypothetical analysis that increasing pH to basic condition results in the breakage of carbon particles into smaller. The higher the pH, the more silica component to be dispersed. Indeed, since silica can reach 50% [11], carbon particles break up can be obtained [12].

EDS analysis is shown in Figs. 5 to 8. The analysis shows that all samples contain carbon (C), oxygen (O), and silicon (Si). Different samples were tested for pH = 2, 8, 10, and 12, corresponding to Figs. 5 to 8, respectively. However, since the sample was analysed on the surface of the silicon wafer, EDS analysis cannot be used for determining the elemental content in the particle. EDS just confirmed that our samples are relatively pure because other components such as Nitrogen (N), Potassium (K), Magnesium (Mg) have less amount and can be neglected.



Fig. 3. Photograph image of carbon nanoparticles solution at various pH from 2 to 12.

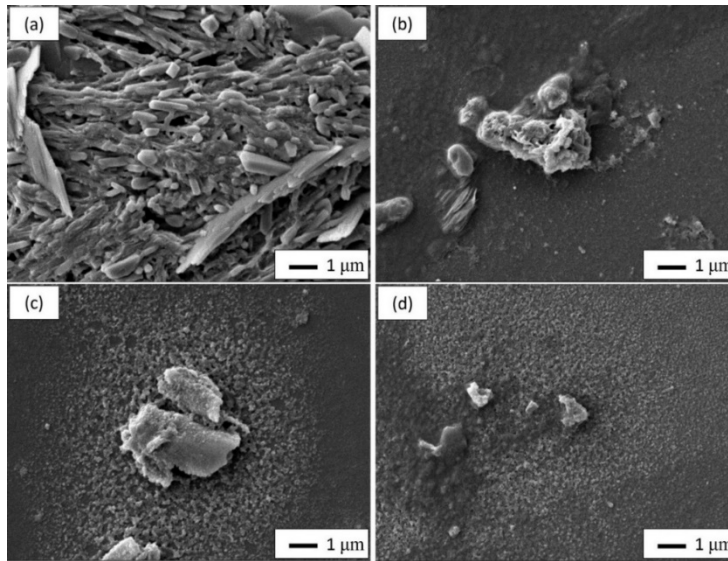


Fig. 4. SEM analysis images of the well-dispersed carbon nanoparticles at (a) pH = 2, (b) pH = 8, (c) pH = 10, and (d) pH = 12.

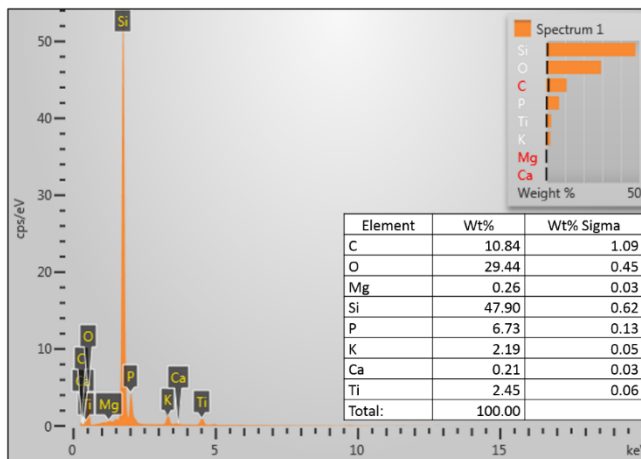


Fig. 5. EDS analysis result of the well-dispersed carbon nanoparticles at pH = 2.

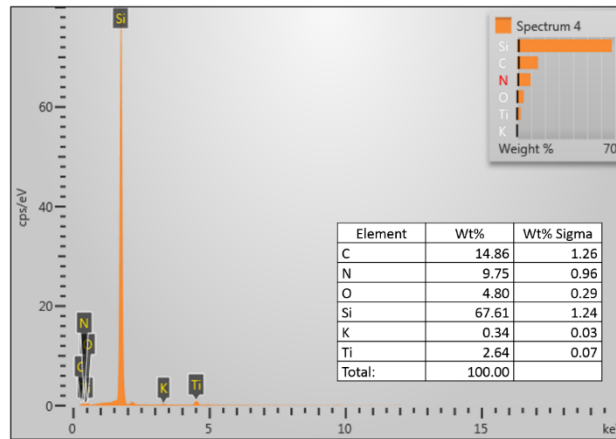


Fig. 6. EDS analysis result of the well-dispersed carbon nanoparticles at pH = 8.

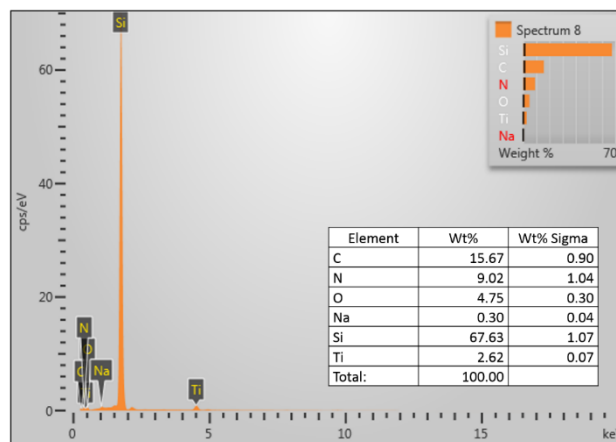


Fig. 7. EDS analysis result of the well-dispersed carbon nanoparticles at pH = 10.

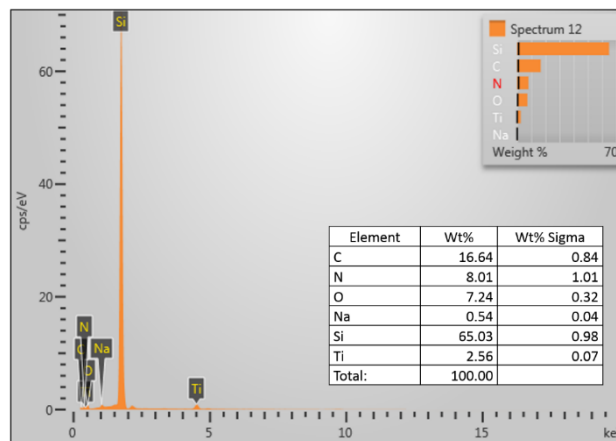


Fig. 8. EDS analysis result of the well-dispersed carbon nanoparticles at pH = 12.

3.3. Proposal mechanism in the formation of well-dispersed carbon nanoparticles

The results showed that carbon nanoparticles can be formed from rice husk and pH can affect the dispersion of carbon in the aqueous solution. The higher the pH of the solution will result in a more well-dispersed carbon nanoparticle.

Figure 9 shows an illustration of breaking carbon nanoparticles due to the addition of acid and basic condition. When acid and basic conditions were applied, it will create the existence of H^+ and OH^- ions. In the initial state, carbons bind to one another (as shown in Fig. 4(a)).

When H^+ is added to carbon, carbon still binds to each other. H^+ cannot bind carbon to the solution, making the size of carbon to be relatively unchanged. When OH^- is added, OH^- can bind carbon element, disperse silica component, and break particle. OH^- incorporates with silica can cover and coat the carbon element on the surface of carbon particles. Indeed, this makes the more OH^- added to lead the more nanoparticles formed [13]. However, in this study, we will not focus on the silica extraction since it has been discussed in our previous report [19].

Detailed information regarding the possible breakage of carbon is shown in Fig. 10. When OH^- is added to the carbon suspension, OH^- will be bound to carbon element. This OH^- bonding causes the carbon-carbon bonding to be cut off. The broken and separated carbon is then re-structuring with other broken carbons to form carbon nanoparticles. During their breakage phenomena, OH^- still interacts with carbon to form C-OH [12]. Thus, it can be concluded that OH can cover and coat the formed carbon particles.

In addition, the main reason for the possible dispersion process is relating to the Archimedes law (see Fig. 11). When the carbon materials are broken to form smaller particles, it will correlate to the existence of less weight. According to Archimedes' law, gravity is inversely proportional to its buoyant force. If the weight is lowered, the value of the buoyant force will be greater [20, 21]. In this study, the more carbon nanoparticles are formed, so that the buoyant force of carbon is bigger. Consequently, carbon nanoparticles can float in solution, in which, this is known as a well-dispersed condition [22].

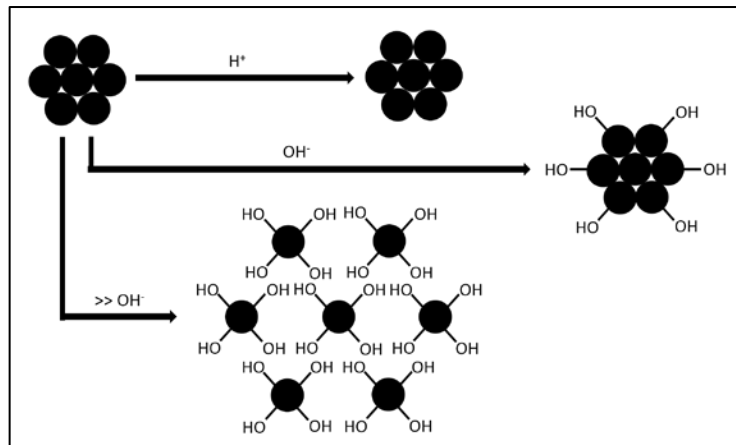


Fig. 9. Illustration of carbon-atomic bond.

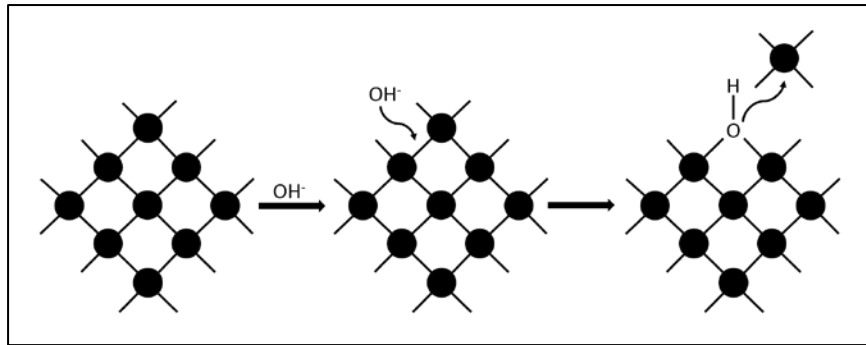


Fig. 10. Carbon nanoparticles formation.

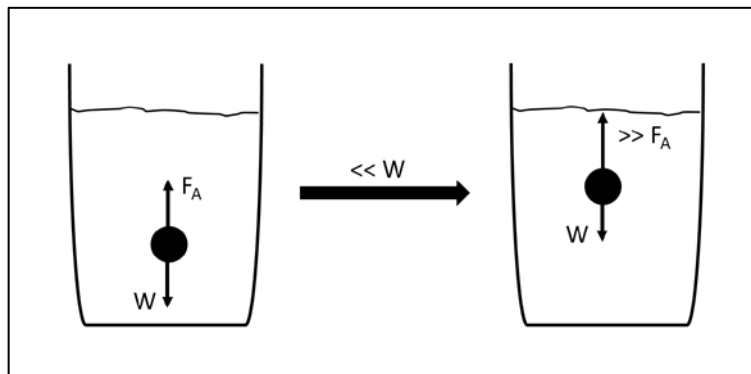


Fig. 11. Illustration of the correlation between well-dispersed carbon nanoparticles and Archimedes' law.

4. Conclusions

The method to get well-dispersed carbon nanoparticles by varying pH conditions has been well explained. Well-dispersed carbon nanoparticles can be produced from carbon microparticles by burning of rice husks at 200 °C, grinding, and adding the dispersion process with basic solutions. The higher pH of the solution results in the more breakdown of carbon into smaller particles. Indeed, this will create more well-dispersed particles to be obtained.

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