Enhancement of Electrical Conductivity of Bismuth Oxide/Activated Carbon Composite

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Abstract

This study aims to synthesize bismuth oxide/activated carbon composites composed of rice husks for battery anodes and to determine the effect of bismuth nitrate pentahydrate mole variations on the characteristics of the resulting composites. The bismuth oxide/activated carbon composite synthesis was carried out using bismuth nitrate pentahydrate, sodium sulfate, and sodium hydroxide precursors which were mixed with rice husk-based activated carbon. A variation was made for the mole of bismuth nitrate pentahydrate used, while the compositions of activated carbon and other precursors were made fixed. The composites were synthesized using the hydrothermal method at a temperature of 110°C for 5 hours. The results showed that bismuth oxide was successfully formed as a composite in the 8 mmol variation with a composite electrical conductivity value of 2.40 x 10⁻³ S.m⁻¹.

Keywords: activated carbon rice husk, bismuth oxide, composite, hydrothermal, battery anode
1. Introduction

Batteries are considered the most effective and practical technology for the power supply of electronic devices due to their flexible design and long usage time. Battery performs as a result of oxidation-reduction electrochemical reactions in the battery cells, i.e. the transfer of conductive electrons from the negative electrode (anode) to the positive electrode (cathode) to produce electricity and a potential difference.

One indicator of battery work is the electrochemical cycling performance which depends on the anode materials the battery comprises. Yao and Cojocaru reported materials with large energy capacity and a flexible structure would allow for the repeated insertion and release of lithium ions to carry off a long life cycle [1]. In addition, good anode material must have characteristics such as good charge/ion conductivity ($> 10^3 \text{ Sm}^{-1}$), high coulomb output ($\text{Ah}^\circ \text{g}^{-1}$), good efficiency as a reducing agent, good electrical conductivity, stability, easy manufacturing process, and low in cost [2].

One material that can be used as an anode material with a large specific capacity is bismuth oxide [3-7]. Li et al reported [8] bismuth oxide (Bi$_2$O$_3$) has a fairly high volumetric capacity of around 3765 mAhcm$^{-3}$, good electrical and optical properties with a bandgap of 2.8 eV, and a potential difference of 1.75-2.25 V, and is non-toxic. In addition, bismuth is environmentally friendly and relatively low in price compared to noble metals [9, 10]. Based on these advantages, Bi$_2$O$_3$ is considered as a promising anode material for lithium-ion batteries with high energy density [11, 12]. However, the problem with using bismuth oxide as an anode is that it has a low electrical conductivity value. This, nonetheless, can be overcome by combining other materials that have better electrical conductivity values, one of which is activated carbon from rice husks.

Rice husk-based activated carbon has been widely studied and utilized for energy applications [13]. Yu et al [14] reported the electrochemical performance of rice husk based activated carbon significantly improved compared to that of the non-activated carbon. In addition, research conducted by Wang et al showed activated carbon from rice husks has a electrical conductivity value of as high as 21.6 S$m^{-1}$ [15], as well as a large surface area of 300 m$^2$g$^{-1}$ to 3500 m$^2$g$^{-1}$ [16]. Activated carbon also has a volumetric capacity of 1770 mAhcm$^{-3}$ and a potential difference of 0.2 V [17] which can be expected to be able to increase the electrical conductivity of bismuth oxide as a battery anode. It was reported that the combination of a metal or metal oxide with carbon has a great influence on the dynamic diffusion of ions and the performance of electrochemical properties [18-20]. For example, carbon-coated Bi$_2$O$_3$
nanocomposite (Bi$_2$O$_3$@C) prepared by redox reaction was applied as anode material for Sodium ion battery (SIB) [21]. This Bi$_2$O$_3$@C nanocomposite showed excellent cycle stability after 100 cycles with a discharge capacity of 421 mAh$^{-1}$ at a current density of 1500 mA g$^{-1}$ [7]. Carbon can be used as a conductive support and form an active ingredient to prevent particle agglomeration [22]. Carbon acts as a framework to retain volume changes, increase the electrical conductivity and avoid particle agglomeration in Li ions extraction/intercalation [23]. When the particles (bismuth oxide) are agglomerated, the particles are not well distributed so that the diffusion of Li ions is disrupted and enable to eliminate electrical connectivity among particles thereby blocking the access of Li ions to the active particles [24].

Furthermore, synthesis of bismuth oxide/carbon composites as anode material has been undertaken previously. Demir et al [7] reported bismuth oxide nanoparticles incorporated in carbon nanofiber produced from organic compounds exhibited excellent rate capability and superior capacities values in sodium ion batteries. Moreover, self standing Bi$_2$O$_3$ nanoparticles/carbon nanofiber hybrid films was successfully produced as a binder free anode for sodium batteries [5]. Previous research conducted Aprialdi et al [25] on the synthesis and characterization of bismuth oxide/activated carbon composites for battery anodes using the hydrothermal method with variations in the weight ratio of activated carbon to bismuth nitrate pentahydrate at 2: 1, 1: 1, and 1: 2 has resulted in the electrical conductivity of each variation of $0.59 \times 10^{-5}$ Sm$^{-1}$, $1.24 \times 10^{-5}$ Sm$^{-1}$, and $0.51 \times 10^{-5}$ Sm$^{-1}$, respectively. However, XRD results showed that bismuth oxide was not fully formed in the composite, and SEM results showed that the distribution of carbon was more dominant than bismuth [25].

Based on this background, it is apparent to fabricate a bismuth oxide/activated carbon composite with more precise composition and method. In the present study, bismuth oxide / activated carbon composite was synthesized using the hydrothermal method. The hydrothermal method was chosen because of the relatively low use of temperature, safe reaction process, no requirement for reducing agents [26], and the homogeneity as well as the purity of resulting material purity is better [27]. More importantly, it is reported to have successfully formed bismuth oxide with higher electrical conductivity values [28]. The combination of rice husk activated carbon with bismuth oxide is expected to generate improved electrical conductivity in the resulting composite.
2. Method

2.1 Materials

The materials used in this study were crystals of Bi(NO$_3$)$_3$·5H$_2$O (Sigma Aldrich), distilled water, H$_3$PO$_4$ 60% (v/v), Na$_2$SO$_4$ powder, and NaOH crystals (Merck), and activated carbon (CA) made from rice husks.

2.2 Procedures

2.2.1 Synthesis of Rice Husk Based Activated Carbon

The making of rice husk activated carbon was carried out through the carbonization and activation stages. At the carbonization stage, cleaned rice husks were charred by the method of pyrolysis at a temperature of 300°C for 10 minutes. A total of 5 grams of the carbon product from the pyrolysis was then activated with 125 mL H$_3$PO$_4$ 60% (v/v) and heated in a microwave with 400 watts of power for 5 minutes. The activated carbon was then washed with distilled water until the pH was constant. The activated carbon that had been washed was then dried using an oven at 105°C for 1 hour. The activated carbon produced was then crushed and sieved with a 100 mesh size [29].

2.2.2 Synthesis of Bismuth Oxide/Activated Carbon Composite

A total of 8 mmol of Bi(NO$_3$)$_3$·5H$_2$O was added with 12 mmol of Na$_2$SO$_4$, then dissolved in 40 mL of distilled water, and stirred using a magnetic stirrer (IKA RH Basic KT/C) at 1500 rpm for 45 minutes. The mixture was then added with 40 ml of 72 mmol of NaOH [27]. The next step was the addition of 0.5 grams of activated carbon rice husk into the mixture. The mixture was then fed to a hydrothermal reactor (Figure 1) and heated at 110°C for 5 hours. The resulting mixture was cooled and then filtered. The filtered precipitate was dried using an oven (fisher scientific) at 110°C for 60 minutes. After that, it was sieved with a size of 100 mesh. The same procedure was applied for the manufacture of bismuth oxide/activated carbon composites with 24 mmol and 32 mmol variations.

2.2.3 Characterization of Bismuth Oxide/Activated Carbon Composite

The resulting bismuth oxide/activated carbon composite material was characterized by Fourier Transform Infra-Red (FTIR) (Shimadzu IRAffinity-1) analysis to determine the functional groups of the composite materials, carried out at wavenumbers 4000 to 400 cm$^{-1}$ with a frequency rate of 0.25 cm$^{-1}$ at room temperature. Products were also analyzed using X-
Ray Diffraction (XRD) (Shimadzu 7000) to identify the crystal structures. Measurements were made for $2\theta$ with Cu-Kα radiation ($\lambda = 0.15406$ nm). The surface morphology and distribution of bismuth oxide and activated carbon in the composites were characterized using SEM Jeol JED 6510LA with magnifications of 100x and 5000x, supported by mapping and EDX. Characterization using TGA-DTG with Mettler Toledo TGA/DSC 3+ was done to determine the thermal stability of the composite materials. The sample analysis was carried out at a temperature range of 40-800°C with a heating rate of 4°C/m in a nitrogen atmosphere. Characterization with GSA (Tristar II 3020) was done to determine pore size and distribution. The samples were analyzed using nitrogen gas flow (N₂). Finally, the products were characterized using the HIOKI 3532-50 LCR meter to determine the electrical conductivity values of the composites. The samples were prepped as pellets with a diameter of 1.5 cm and a thickness of 2-5 mm for the analysis.

3. Results and Discussion

3.1 Activated Carbon

Activated carbon synthesis was carried out according to the procedure reported by Santoso [29]. The process was carried out in two stages, namely the pyrolysis of rice husks to carbon and the carbon activation using H₃PO₄ acid. The pyrolysis process of rice husks produced carbon, tar, and permanent gases such as CO₂, CO, CH₂, H₂, and others [30]. The product of this carbonization was blackened carbon.

The pyrolyzed carbon was activated using phosphoric acid by heating it using microwave radiation. The activation process aimed to enlarge and open carbon pores and increase the surface area of the carbon. Phosphoric acid can remove impurities contained in carbon from the carbonization process (rice husks) such as ketones, alcohol, acids, aldehydes [31, 32]. The activated carbon sample obtained was a fine black powder with uniform size. The synthesis scheme of the activated carbon from rice husks can be seen in Figure 2.

3.2 Bismuth Oxide/Activated Carbon Composites

The synthesis of bismuth oxide/activated carbon composites with mole variation in the bismuth nitrate pentahydrate used was initiated by reacting the Bi(NO₃)₃·5H₂O, Na₂SO₄, and NaOH precursors. The solution was later added with rice husk activated carbon, homogenized,
and heated in a hydrothermal reactor. The reaction mechanism for the formation of bismuth oxide according to Wu et al. [27] can be seen as follow:

\[
2\text{Bi(NO}_3\text{)}_3.5\text{H}_2\text{O} + \text{Na}_2\text{SO}_4 \rightarrow \text{Bi}_2\text{(OH)}_2\text{SO}_4 + 2\text{NaNO}_3 + 4\text{HNO}_3 + 7\text{H}_2\text{O} \quad (1)
\]

\[
\text{Bi}_2\text{(OH)}_2\text{SO}_4 + 2\text{NaOH} + \text{H}_2\text{O} \rightarrow 2\text{Bi(OH)}_3 + \text{Na}_2\text{SO}_4 \quad (2)
\]

\[
2\text{Bi(OH)}_3 \rightarrow \text{Bi}_2\text{O}_3 + 3\text{H}_2\text{O} \quad (3)
\]

The resulting composite from the 8 mmol variation was blackish gray powder with a smooth dark gray texture. In the 24 mmol variation, the resulting composite was a light gray fine powder and at 32 mmol the resulting composite was gray-colored ash with a texture resembling an agglomerate as shown in Figure 3. The gray color was produced from the combination of pale yellow from the bismuth oxide and black from the activated carbon.

3.3 Characteristics of Bismuth/Oxide Composites

3.3.1 Structure

Figure 4 shows the FTIR spectra of activated carbon, pure bismuth oxide, and the composites made in this study. The rice husk activated carbon has a small absorption at a wavenumber of about 3400 cm\(^{-1}\) denoting the absorption of the OH group, 1642 cm\(^{-1}\) referring to the C=C group, and 1095 cm\(^{-1}\) indicating the presence of a CO group [33, 34].

Similar absorption was also shown by each of the composite samples, namely the C=C group in the absorption area around 1642-1645 cm\(^{-1}\) and the C-O group seen in the absorption area around 1100 cm\(^{-1}\). Meanwhile, the pure bismuth oxide spectra present the Bi-O-Bi group absorption peak in the 829 cm\(^{-1}\) area [35-37], and the Bi-O group at a wavenumber of about 1380 cm\(^{-1}\) [38].

The three samples of bismuth oxide/activated carbon composites have almost the same spectra as the two raw materials combine, showing the presence of a fairly sharp peak in the area around 828-830 cm\(^{-1}\) indicating the Bi-O-Bi group and in the of 1375-1384 cm\(^{-1}\), which indicates the Bi-O group. Thus, it can be concluded that bismuth oxide was successfully formed in each of the composite samples. Additionally, the three composite samples were also observed to have a wide O-H absorption at the wavenumber of about 3400 cm\(^{-1}\).

Complete data regarding the functional groups of bismuth oxide/activated carbon composites, bismuth oxide (Bi\(_2\)O\(_3\)), and rice husk activated carbon from the FTIR test results are shown in Table 1.
3.3.2 Crystal Structure

XRD results, as presented in Figure 5, show that the diffractogram of the 8 mmol Bi/CA composite sample had the greatest match with the α-Bi₂O₃ diffractogram and slightly matches the β- and γ-Bi₂O₃ diffractogram. The 8 mmol Bi/CA composite had high peak values at 2 theta (2θ) of 27.430°, 33.090°, 33.296°, and 46.246°. The 2 theta (2θ) values were almost the same as the α-Bi₂O₃ peak values at 27.377°, 33.039°, 46.305° with miller indices (120), (121), (200), and (041), respectively based on JCPDF No. 41-1449, a strong suggestion that the Bi/CA 8 mmol composite contained bismuth oxide crystals with α-Bi₂O₃ (monoclinic) structure which was more dominant than β-Bi₂O₃ and γ-Bi₂O₃ in which β-Bi₂O₃ peak value is shown at 2 theta (2θ) 27.799 with miller index (201) (JCPDF No. 76-0147) while the presence of γ-Bi₂O₃ is indicated by (2θ) values at 27.455° and 32.607° with miller indices (310) and (321) respectively (JCPDF No. 45-1344).

Furthermore, the 24 mmol Bi/CA composite diffractogram showed that the composite had a non-crystalline (amorphous) structure. This indicates that the Bi/CA 24 mmol composite had not borne bismuth oxide. The diffractogram of the 32 mmol composite had its highest peaks at 2 theta (2θ) of 28.900°, 29.473°, and 30.592° with the miller indices (012), (130), (012) based on JCPDF No. 44-0314, showing no compatibility with the α-β-and γ-Bi₂O₃ diffractograms but showed a match with the diffractogram peaks of bismuth nitrate pentahydrate. These peaks are also thought to be the peaks of the by-product, Bi₂O(OH)₂SO₄ [27], which was formed during the reaction (reaction 1). However, the 32 mmol Bi/CA composite diffractogram had small peaks at 27.421° and 33.209° with miller indices (120) and (200), respectively which indicated a good match with the α-Bi₂O₃. This implies that the 32 mmol Bi/CA composite contained more bismuth nitrate pentahydrate precursor and Bi₂O(OH)₂SO₄ by-product than bismuth oxide. Qualitatively, the 32 mmol Bi/CA composite diffractogram had several discrete peaks indicating that the 32 mmol composite had a crystalline structure. Meanwhile, the diffractogram of activated carbon shows that this material had an amorphous structure. This is in accordance with the research reported by Wazir et al. [39].

3.3.3 Morphology and Particle Distribution of Bismuth Oxide

SEM images of bismuth oxide/activated carbon composites with 8 mmol, 24 mmol, and 32 mmol variations are shown in Figure 6. Figure 6 shows that the 8 mmol variation of the Bi/CA composites had a rod-like particle shape with a length of 5.14-19.16 μm and a width of 0.83-1.66 μm. The rod-like particle shape marked the presence of bismuth oxide in the
composite which is in agreement with study reported by Wu et al. [27]. Meanwhile, the Bi/CA composite with 24 mmol variation showed irregular particle shape, clustered like clouds. Furthermore, the Bi/CA composite with 32 mmol variation showed that the particles had irregular shapes and had formed agglomerations with particle sizes of about 1.35-3.37 μm. This sample also appeared to have a less hollow or porous surface which was theorized to originate from the pores that were covered by the dominant elements in the composite. The SEM mapping images of the three composite samples are shown in Figure 7.

Figure 7 illustrates the distribution of the bismuth (Bi), carbon (C), oxygen (O), and Si elements in the Bi/CA composites in the 8 mmol, 24 mmol, 32 mmol variations, respectively marked by red, green, yellowish-green and blue. Figure 7 shows that the 8 mmol variation of the composites had Bi and C elements that appeared almost balanced and evenly distributed across the surface of the material. In the 24 mmol variation it can be seen that the material surface was more dominated by the distribution of Bi elements, whereas in the 32 mmol variation the distribution of the Bi and O elements dominated the sample surface and caused the C element to be less visible on the sample surface.

3.3.4 Thermal Stability of Composites

TGA-DTG characterization was used to determine the decomposition process and thermal stability of the composite materials. The TGA-DTG characterization results of each mole variation of bismuth nitrate pentahydrate are shown in Figure 8.

Based on the results of the TGA and DTG analysis in Figure 8, the 8 mmol and 24 mmol samples exhibit five stages of the decomposition process, while the 32 mmol sample showed four stages of decomposition. The first stage of decomposition occurred at a temperature range of 750-1900°C indicating the evaporation of water content adsorbed onto the surface of the activated carbon [34]. The weight loss at this stage also indicated the rapid evaporation of organic volatiles under an oxygen atmosphere [40].

Stage II occurred in a temperature range of 2250-3200°C which was assumed to be the process of burning of cellulose and hemicellulose that were present in the activated carbon [40], in addition to the removal of residues [41] such as NaNO₃, NaOH, and SO₄²⁻. The weight reduction for stage II of the 32 mmol variation was bigger when compared to the weight loss graph for stage II for the 8 and 24 mmol variations. This indicated that more residues were decomposed in the 32 mmol variation as it contained the reaction byproduct, Bi₂O(OH)₂SO₄, and also the Bi(NO₃)₃.5H₂O precursor as suggested by the XRD results shown in Figure 5.
Stage III occurred in a temperature range of 330°-500°C, showing a significant reduction in weight for each composite. The weight of the 8 mmol variation sample was lost from 94.86% to 81.51%, amounting to a weight loss of around 13.35%. The 24 mmol variation experienced a weight loss percentage of about 8.42% whilst the 32 mmol variation showed a significant reduction in weight from 91.98% to 85.29%, equating to 6.69%. The weight loss at this stage was interpreted as the phase transformation from the precursor material to bismuth oxide [41] which occurred endothermically.

The stage IV of decomposition occurred in the range of 500°-610°C, indicating the formation of monoclinic alpha bismuth oxide (α-Bi₂O₃) and the transformation from the monoclinic structure to the body-centered cubic (γ-Bi₂O₃) structure [42]. Lastly, stage V occurred in the range of 640°-700°C denoting the transformation of γ-Bi₂O₃ to α-Bi₂O₃, as temperatures above 600°C the thermal behavior of bismuth oxide is common [43].

3.3.5 Electrical Conductivity

Figure 9 shows the relationship graph between the log conductivity and the log frequency of the composites. The x-axis (log frequency) shows the number of electrons flowing to generate electric current, while the y-axis (log conductivity) signifies the sample's ability to conduct electric current. The relationship between the two properties is linear, meaning that the greater the frequency value, the greater the conductivity value. The electrical conductivity value of the sample was determined from the value of the intercept of the graph plateau line equation of each composite sample. The plateau area is an area with stable values of y (log conductivity) (no/very small increase) respective to the increase in the value of x (log frequency). The conductivity value of the sample was obtained from the antilog of the graph's intercept value.

The electrical conductivity values obtained from the EIS test on the bismuth oxide, rice husk activated carbon (CA), and bismuth oxide/activated carbon (Bi/CA) composite samples with varying bismuth nitrate pentahydrate moles are shown in Table 2. Based on Table 2, the electrical conductivity value of the rice husk activated carbon was higher than the electrical conductivity value of pure bismuth oxide. The conductivities of the composites were seen to have increased relative to the pure bismuth oxide and activated carbon.

The electrical conductivity values of the Bi/CA composites from the highest to the lowest were shown by the 32 mmol, 8 mmol, and 24 mmol variations, respectively. The 32 mmol composite sample had a high electrical conductivity value because of the large amount of bismuth nitrate pentahydrate in the composite as shown in the XRD results (Figure 5). In
addition, the 32 mmol Bi/CA composite exhibited agglomerations that also affected the electrical conductivity value, because samples with large crystal sizes have a good level of atomic order that increased the mobility of electrons in the sample [44, 45].

The electrical conductivity value of the 8 mmol variation composite was higher than the 24 mmol variation. This is because in the 8 mmol Bi/CA composite, between the precursor material for the formation of bismuth oxide and the added activated carbon, not one was more dominant than the other. In other terms, the bismuth oxide formed in the composite, as denoted in the FTIR (Figure 4) and XRD (Figure 5) results, were evenly distributed throughout the surface of the material as seen in the SEM mapping image shown in Figure 7. The even distribution of bismuth oxide would allow for more freedom in the mobility of electrons in the material, thereby increasing the electrical conductivity value [46, 47]. The 24 mmol composite had the smallest electrical conductivity value because bismuth oxide had not been successfully formed and its crystal structure was amorphous. Overall, the three composite samples had electrical conductivity values in the range $10^{-8}$ S.m$^{-1}$ to $10^{-3}$ S.m$^{-1}$, indicating that the materials had semiconductor properties [48].

3.3.6 Porosity, pore surface area, and pore size distribution

Characterization of composite samples using a Gas Sorption Analyzer (GSA) was aimed to determine the surface area, pore size, pore volume, and pore distribution of the materials. The BET charts of the rice husk activated and the 8 mmol; 24 mmol and 32 mmol Bi/AC composites can be seen in Figure 10.

Figure 10 is a graph showing the amount of adsorption and desorption of nitrogen gas (N$_2$) with respect to relative pressure (p/p0). The graph shows that the three composite samples had a similar isotherm pattern, namely, type IV isotherm characterized by the hysteresis loop. The hysteresis pattern indicated that the samples are materials that exhibit pores because they were able to absorb N$_2$ gas molecules. The GSA results of the rice husk activated carbon, and the 8 mmol, 24 mmol, and 32 mmol Bi/AC composites are shown in Table 3.

Table 3 shows that the three composite samples had mesoporous sized pores because the pore radius ranged from 3-50 nm [49]. Subhan et al reported [50] on the fabrication and characterization of Li$_4$Ti$_5$O$_{12}$ for battery anode materials; porosity will affect the electrical conductivity value of the sample. Samples with high porosity, i.e. samples that have many pores with small pore size, will tend to have a low electrical conductivity value because a large number of pores will cause a greater resistance, thereby reducing the electrical conductivity of the sample [51, 52].
This is in line with the results of the characterization of the 32 mmol Bi/AC composite which demonstrated the largest pore size of 15.509 nm as well as a small pore volume and surface area of 0.012 cm³/g and 3.110 m²/g. This depicted that the 32 mmol composite sample had a small number of pores or small porosity, which when analysed with an LCR meter, the measured resistance was low and the sample electrical conductivity was high, as per the theory mentioned. In addition, material density also affects the electrical conductivity value of the sample. The higher the density of the material, the level of the order of the atoms in the crystal also increases and will consequently increase the value of electron mobility [44, 45]. If associated with the characterization results in Table 3, the material density increased in order from the 8 mmol, then 24 mmol to the 32 mmol variations. This can be seen from the value of the total volume of each composite sample which decreased as the number of moles of bismuth nitrate pentahydrate increased. The decrease in pore volume was probably due to the bismuth adsorbed into the pore of the activated carbon. The more bismuth particles, the more these particles will occupy the activated carbon pores. As a result, the originally large pore volume becomes smaller. Thus, more bismuth was adsorbed into the pore causing the pore volume to decrease and caused the material to be clumpy.

4. Conclusion

Befitting compositions in the synthesis of bismuth oxide/activated carbon composites were able to produce composites with better electrical conductivity values than their precursors. The results showed that bismuth oxide was successfully formed in the 8 mmol variation with electrical conductivity value of 2.40x10⁻³ S.m⁻¹. The highest electrical conductivity value was produced by the variation of 32 mmol, though the bismuth oxide formed was very small based on the XRD results. Its high electrical conductivity value was thought to be derived from the electrical conductivity of bismuth nitrate pentahydrate precursor present. This investigation is expected to give recommendation on the use of activated carbon and bismuth oxide in the form of composite as potential candidate for battery anode by paying attention to the factors influencing the formation of composites especially the composite precursor composition factor. In addition, this investigation is expected to give a scientific contribution.

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TABLE CAPTIONS

Table 1. Functional groups observed in the samples
Table 2. Electrical conductivity of pure bismuth oxide, activated carbon, bismuth nitrate pentahydrate, 8mmol Bi/CA, 24 mmol Bi/CA, 32 mmol Bi/CA
Table 3. Surface area, pore volume and average pore size

FIGURES CAPTIONS

Figure 1. (a) a hydrothermal reactor, (b) reactor scheme
Figure 2. Activated Carbon Synthesis Scheme
Figure 3. Bi/CA synthesized composites using (a) 8 mmol, (b) 24 mmol, (c) 32 mmol bismuth nitrate pentahydrate
Figure 4. FTIR spectra of pure bismuth oxide [37], rice husk activated carbon [29], and the bismuth oxide/activated carbon composite samples
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Figure 6. SEM images of Bi/CA composites synthesized using (a) 8 mmol (b) 24 mmol (c) 32 mmol bismuth nitrate pentahydrate
Figure 7. SEM mapping of Bi/CA composites synthesized using (a) 8 mmol (b) 24 mmol (c) 32 mmol bismuth nitrate pentahydrate
Figure 8. Thermal stability of composites analyzed using TGA-DTG
Figure 9. The relationship graph between the log conductivity and the log frequency of the composites
Figure 10. N₂ adsorption/desorption isotherm graphs of (a) 8 mmol; (b) 24 mmol; (c) 32 mmol Bi/AC composites
### Table 1.

<table>
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<tr>
<th>Functional groups</th>
<th>Activate carbon Bi-oxid</th>
<th>Bi/CA 8 mmol</th>
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### Table 2.

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<th>Electrical conductivity</th>
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<td>Pure Bi₂O₃</td>
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<tr>
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<tr>
<td>Bismuth Nitrate Pentahydrate</td>
<td>1.70 x 10⁻¹ S.m⁻¹</td>
</tr>
<tr>
<td>8 mmol Bi/CA</td>
<td>2.40 x 10⁻³ S.m⁻¹</td>
</tr>
<tr>
<td>24 mmol Bi/CA</td>
<td>4.91 x 10⁻⁴ S.m⁻¹</td>
</tr>
<tr>
<td>32 mmol Bi/CA</td>
<td>15.54 x 10⁻³ S.m⁻¹</td>
</tr>
</tbody>
</table>

### Table 3.

<table>
<thead>
<tr>
<th>Sample</th>
<th>S_BET (m²/g)</th>
<th>V_total (cm³/g)</th>
<th>Average pore size (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rice husk activated carbon</td>
<td>18.837</td>
<td>0.014</td>
<td>3.090</td>
</tr>
<tr>
<td>8 mmol Bi/AC composite</td>
<td>24.221</td>
<td>0.051</td>
<td>8.473</td>
</tr>
<tr>
<td>24 mmol Bi/AC composite</td>
<td>8.102</td>
<td>0.026</td>
<td>13.055</td>
</tr>
<tr>
<td>32 mmol Bi/AC composite</td>
<td>3.113</td>
<td>0.012</td>
<td>15.509</td>
</tr>
</tbody>
</table>
LIST OF FIGURES

Figure 1
Figure 4

[Graph showing vibration modes and wavenumber ranges for different compounds]
Figure 5

* : α-Bi₂O₃
▼ : β-Bi₂O₃
□ : α-Bi₂O₃
● : pure BNP

Intensity (a.u)

2 theta (degree)

Rice husk-activated carbon
Composite of Bi/CA 32 mmol
γ-Bi₂O₃
Composite of Bi/CA 8 mmol
α-Bi₂O₃
β-Bi₂O₃
Composite of Bi/CA 24 mmol
pure BNP
Figure 7
Figure 8

(a)

(b)

(c)
Figure 9

The graph illustrates the relationship between log frequency and log conductivity for different composite Bi/CA concentrations: 8 mmol, 24 mmol, and 32 mmol. The equations for the linear fits are as follows:

- Composite Bi/CA 8 mmol: \( y = 0.1393x - 3.8084 \)
- Composite Bi/CA 24 mmol: \( y = 0.2062x - 5.3614 \)
- Composite Bi/CA 32 mmol: \( y = 0.1375x - 4.9007 \)
Figure 10

(a) 

(b) 

(c)
Biographies

**Yayuk Astuti** is a lecturer in Chemistry at Faculty of Sciences and Mathematics, Universitas Diponegoro, Indonesia. She obtained her bachelor's degree in chemistry at the Department of Chemistry, FMIPA Universitas Diponegoro, Indonesia in October 2005. In less than a year of graduation, Yayuk Astuti was accepted as a lecturer in the same University. Doctoral degree was obtained through an accelerated program at Newcastle University, UK in early 2014 (master leads to PhD). Currently, her research concern is material chemistry, especially synthesis, modification, characterization and application. Some of the materials that have been developed are carbon, nanodiamond and bismuth oxide for adsorbents, battery electrodes, drug carriers and photocatalysts.

**Roshana Mei** was an undergraduate student at Fakultas Sains dan Matematika, Universitas Diponegoro, Indonesia in 2016. She received her bachelor’s degree in Chemistry in 2020. Her research project was synthesis of bismuth oxide/activated carbon composite for battery anode.

**Arnelli** has been a Chemistry lecturer at Fakultas Sains dan Matematika, Universitas Diponegoro, Indonesia since 1989. She studied undergraduate at Andalas University from 1979 to 1986 in Pure Chemistry and got master degree at Bandung Institute of Technology (ITB) in 1989 in the same field, especially chemical physics. Her research interests are surfactant and its application, activated carbon and its modification for adsorbent.

**Adi Darmawan** is a lecturer at Chemistry Department, Faculty of Sciences and Mathematics, Universitas Diponegoro, Indonesia. He received his bachelor’s degree at the same University he works in 1996 and his master degree was received from Departmen of Chemistry, Gadjah Mada University, Indonesia in 2003. In 2014 he received his doctorate degree from School of Chemical Engineering, The University of Queensland, Australia. His research interest is inorganic materials especially on silica and carbon for membrane separation.

**Hendri Widiyandari** has been a lecturer at Physics Department Sebelas Maret University, Indonesia, since 2018. Before that, she had been a lecturer at the Department of Physics, Diponegoro University, Indonesia, from 1999 until 2017. She received her Ph.D. degree in Chemical Engineering from Hiroshima University, Japan. Her research interest is in the synthesis and characterization of functional materials for energy storage and conversion. One of the research interests is the development of active materials for Li-ion batteries.