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Characterization of CNT-MnO_2 Nanocomposite by Electrophoretic Deposition as Potential Electrode for Supercapacitor

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Abstract. Energy crisis that occured in Indonesia suggests that energy supply could not offset the high rate request and needs an electric energy saving device which can save high voltage, safety, and unlimited lifetime. The weakness of batteries is durable but has a low power density while the capacitor has a high power density but it doesn't durable. The renewal of this study is CNT-MnO_2 thin film fabrication method using electrophoretic deposition. Electrophoretic deposition is a newest method to deposited CNT using power supply with cheap, and make a good result. The result of FTIR analysis showed that the best CNT-MnO_2 composition is 75:25 and C-C bond is detected in fingerprint area. The result is electrode thin film homogen and characterized by X-ray diffraction (XRD) peaks $2\theta=26{,}63^{o}$ is characterization of graphite, and $2\theta=43{,}97^{o}$ is characterization of diamond Carbon type and measured by Scherrer formula results 52,3 nm material average size .EIS test results its capacitance about 7,86 F. from the data it can be concluded that CNT-MnO_2 potential electrode very promising for further study and has a potential to be a high capacitance, and fast charge supercapacitor which can be applied for electronic devices, energy converter, even electric car.

INTRODUCTION

The energy crisis that occurred in Indonesia towards the end of the 20th century suggests that the energy supply cannot keep pace with the high rate of demand. According to statistical data the Ministry of Energy and Mineral Resources Indonesia in 2012, the demand for energy in Indonesia is about 200 million TOE in 2011, while the energy supply in Indonesia in 2011 only 161 million TOE. Dependence on fossil fuels as an energy source, has three serious threat, they are the depletion of oil reserves, the unstability of price due to greater demand and greenhouse gas pollution resulting from the burning of fossil fuels. Electric energy become one of the best alternatives for the energy supply of the future [1].

Various energy storage devices offered are a battery and a capacitor [2]. The battery has the disadvantage of having a long lifetime, but has a low power density, meaning that the battery lasts longer but can only contain low power [3]. While the capacitor has a large power density, but has a short lifetime. Various problems is it needs storage of electrical energy which has the high power density and long lifetime, the storage media which capable of storing electrical energy with large scale and in the long term, called supercapacitor, which is currently being developed for the development of the development such as military technology, spacecraft, and electric cars [4].

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Supercapacitor contains potential electrodes, celgard separator and charge collecting in the form of a stainless steel foil. Nanocomposite Carbon nanotubes (CNT) is a potential electrode material used for supercapacitors. CNT has excellent mechanical properties and electrical conductivity [5]. Wang et al (2011) reported that manufacture of CNT nanocomposite can be fabricated into a supercapacitor electrode material, because the electrode is an element that stores the electric field in a supercapacitor, the high conductivity, the better the quality of the capacitor [5]. The use of nanocomposite CNT as supercapacitor electrodes will improve the performance of ion mobility to the surface to improve the electronic and electrochemical conductivity.

Various methods have been developed to make the supercapacitor. The examples are double-layer thin filmmethod [5,6] and the doctor blademethod [7], the method of double-layer thin film which produces the electrode is too thick, the microstructure is not homogeneous, so that the capacitance is still low and doctor blade methods which fragile and easilybroke [8]. Thosevarious methods are not able to make a lightweight, tightly bound to the substrateelectrode, and the method is complicated / difficult to implement.

The innovation that can solve the problem of energy storage device, and the weakness of the manufacturing method is to make a supercapacitor electrode by electrophoretic deposition method. Innovation of this study is to make CNTMnO_2 thin film by electrophoretic deposition method. The advantage of this innovation is produced CNT compositenano-sized, with a homogenous microstructure. As well as the addition of MnO_2 materialas doping with has high conductivity, cheaper, non-toxic, and has a high capacitance about 1370 F/g [5]. In this study aims to make a nano-tech supercapacitor electrode material which cheap, and lightweight because it is made into a thin film that is directly attached to the aluminum substrate with high capacity power storage, and durable.

METHOD

The materials used in this study was purified CNT, HNO_3 p.a (Merck), $KMnO_4$ pa (Merck), aluminum foil, ethanol p.a (Merck), distilled water (deionized water). The equipment used are spray pyrolysis system to produce CNT, glassware, reflux set equipment, a set of electrophoretic deposition, UV-Vis Spectroscopy (UV, Milton Roy - Spectronic 3000), SEM (type JEOL JSM-35C series), XRD (Philip Analytical X-Ray B. V), FTIR (FTIR spectrophotometer Shimadzu 8201PC), and Impedance Test.

This study uses a variable not fixed is the coating of CNTs on a alumunium foil substrate, deposition process timeusing the electrophoretic deposition method. Variables assessed in this study is the size of CNTs on an aluminum foil substrate, supercapacitor electrode CNT thin filmcapacitance and impedance.

Research procedure is performed through three stages, they are: synthesis and purification of CNT material, the manufacture of a mixture of CNT-MnO_2, manufacture CNT-MnO_2 thin film on the aluminum foil by electrophoretic deposition method, material characterization, testing, and manufacturing of prototype supercapacitor.

CNT synthesis is done using spray pyrolysis method by mixing 0.6 g of Ferrocene and 10 mL of Benzene were subsequently dispray into a quartz tube that was placed in the furnace at a temperature of 900^{o}C. Fig. 1 shows a spray pyrolysis system used to produce CNT.

CNT is generated in the form of powder and sieved at 175 mesh, then purified by washing using HNO3 acid 65% as much as 50mL and reflux for 7 hours. CNTs are purified and then put in an oven and calcined using the furnace at a temperature of 80 ° C for 1 hour. The next step is mixing the composition of CNT/MnO2, preparation nano MnO2 of sol gel method of KMnO4, with variations of CNT:MnO2 25:75, 50:50, 75:25 The results of three samples test performed FT-IR

Series of electrophoretic deposition aims to deposed CNT-MnO_2 into aluminum foil. The equipment used is aluminum foil and stainless steel foil plate, a power supply, and a solution of CNT-MnO_2. The specification of this power supply has a voltage of 50 volts, the current of 5 A, and the power of 250 Watts. The circuit schematic as shfown Fig. 2.

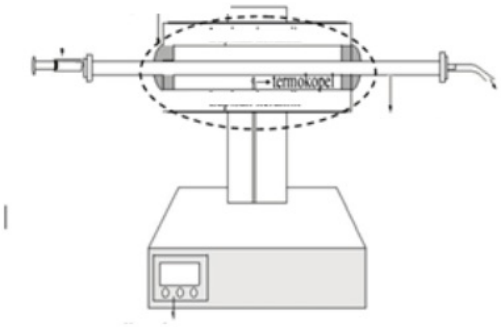

FIGURE 1. Spray Pyrolisis System used for CNT Production

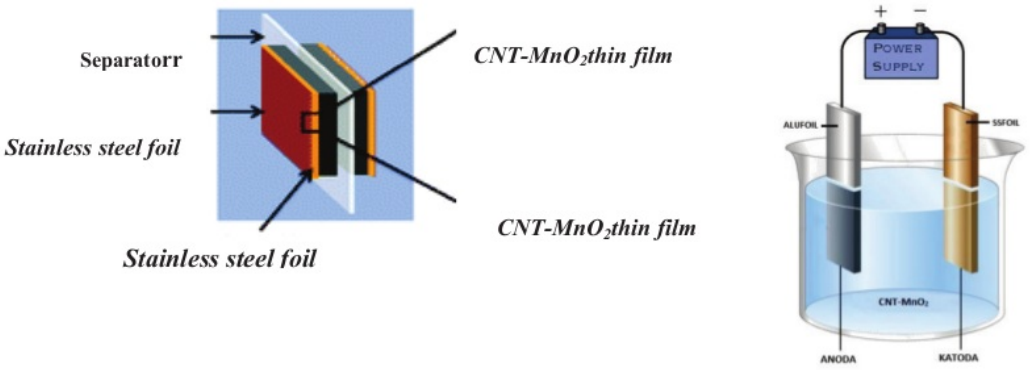

FIGURE 2. Electrophoretic Deposition Method Visualisation

RESULTS AND DISCUSSION

Figure 3, Figure 4, Figure 5, Figure 6 and Figure 7 shows the testing of each successive FT-IR spectra IR onCNT material, the material MnO_2 IR spectra, IR spectra at CNT:MnO_2material 25:75, the IR spectra CNT:MnO_2material 50:50, and the IR spectra at CNT:MnO_2material 75:25 which aims to determine the transitionalvibration bond in CNT material against MnO_2:

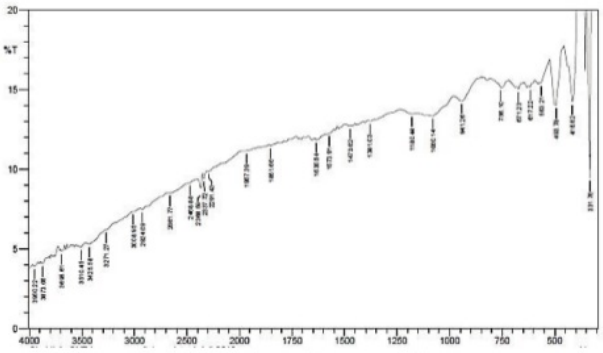

FIGURE 3. IR Spectra of CNT

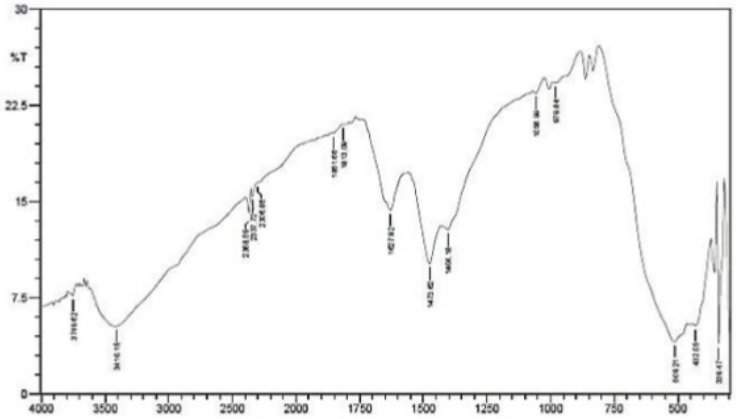

FIGURE 4. IR Spectra of MnO_2

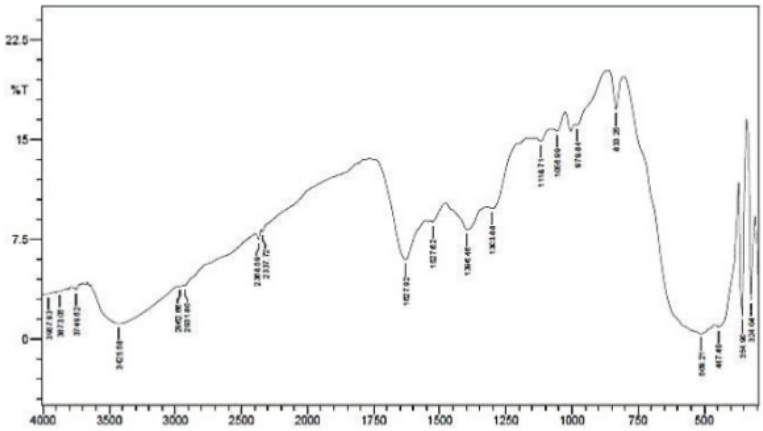

FIGURE 5. IR Spectra of CNT:MnO_2 25:75

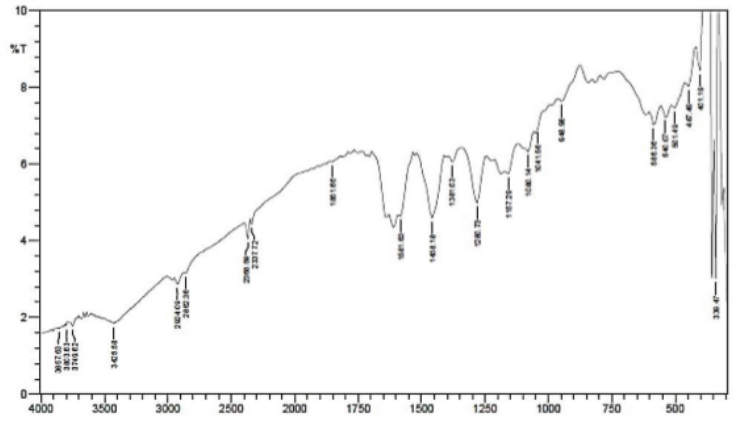

FIGURE 6. IR Spectra of CNT:MnO_2 50:50

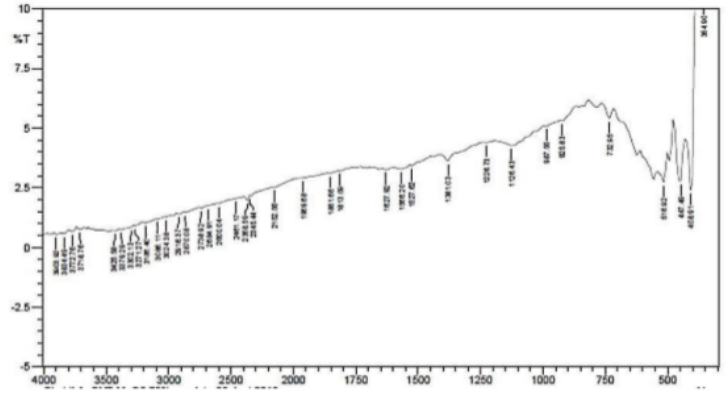

FIGURE 7. IR Spectra of CNT:MnO_2 75:25

Uptake of specific wavelengths of CNT compounds is shown in Figure 4 can be observed at the wavelength of about 1725 cm^{-1}, in addition to the vibrations of the compound CHX group can also be observed in the area of 1584 and 1200 cm^{-1}(Kouklin et al, 2004). Uptake wavelength addressed in Fig. 5 obtained absorption wavelengths of specific compounds of MnO_2 can be observed in the area around 922 cm^{-1}, in addition to the vibrations of the compound Mn-O can also be observed in the area of 829 and 520 cm^{-1} [9]. Then analyzed respectively in Fig. 5, Fig. 6 and Fig. 7 respectively show the IR spectra for CNT and MnO_2. In the IR spectra for MnO_2 produce absorption at a wavelength of 979.84 and 509.21 cm^{-1}, which can be for a Mn-O bond vibrations in MnO_2 compounds. In the IR spectra for 25% CNT/MnO_2 as shown in Fig 5, the visible absorption at a wavelength of 979.84 and 509.21$cm^{-1}$is thought to be vibrati3 absorption compound Mn-O bond, while the 833.25cm^{-1} area is a Mn-O bond vibrations of MnO_2. Furthermore, the IR spectra for 50% CNT/MnO_2, Mn-O bond vibration of the Manganese Dioxide did not appear and shift the wavelength of vibrations of Mn-O bond compounds into 948.98 and 501.49 cm^{-1}. IR spectra for 75% CNT/MnO_2 shows the vibration of Mn-O bond compound at a wavelength of 925.83 and 516.92 cm^{-1}, but as the IR spectra for 50% CNT/MnO_2, Mn-O bond vibrations of$MnO_2$is not visible. Overall if seen IR spectra for CNT, it is not found absorption at specific wavelengths for Mn-O bond. Meanwhile, as described above it is clear the specific absorption wavelength for vibration bonding alloy Mn-O on the CNT/MnO_2. This proves that the composite CNT/MnO_2 allegedly been formed in each mixture. Chen et al 2007 reported by the addition of MnO_2composition

on the CNT will fill the pores of carbon, it can improve the cycling unstability and the capacitance of a supercapacitor that is 146F/g, so that the selected CNT material composition: MnO2 is 75:25.

Further mixing the nanocomposite powders on Na_2SO_4 and sonifikasi done by electrophoretic deposition, electrode test results of electrophoretic analysis using SEM-EDS, XRD, and capacitance test.

Morphology CNT:MnO_2, shown by SEM on Figure 8 on the preparation done by coating platinum on the first electrode layer, the data obtained:

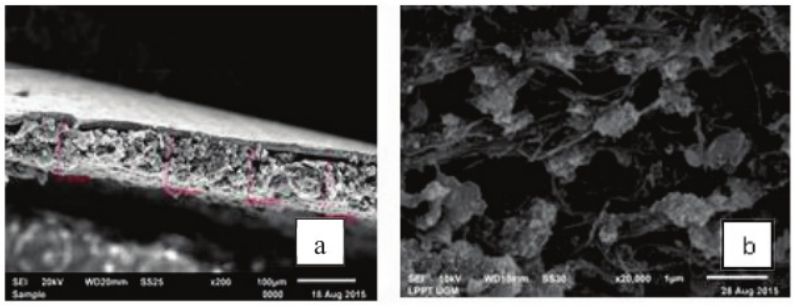

FIGURE 8. SEM image CNT:MnO_2 75:25 with Magnifying of (a) 200x (b) 20,000x

The Resultof material morphology is shown in Figure 8.a can be seen that coating thickness about 800nm, and is shown in Fig. 7.b size of the crystal morphology of MnO_2 into the sidelines during CNT range of 40nm-50nm. The reaction Mn^{2+} is oxidized by $KMnO_4$ will form MnO_2 particle along SDS surfactant chains attached to the tube wall of CNT, and are firmly anchored in the Aluminum Foilsubstrate.

Du (2006) reported coating process of CNT on the stainless steel foilelectrode using electrophoretic technique, the mobilization of ions occur into the positive pole called anode, so that the material can be deposited at the anode with aluminum as redox reactions and morphology becomes uniform. In this study, CNT material has been modified by the addition of MnO_2, which serves to increase the electrode capacitance and cycling stability [5].

Testing XRD is to determine the type of crystals that exist in layers of CNT: MnO_2 on the stainless steel foil.

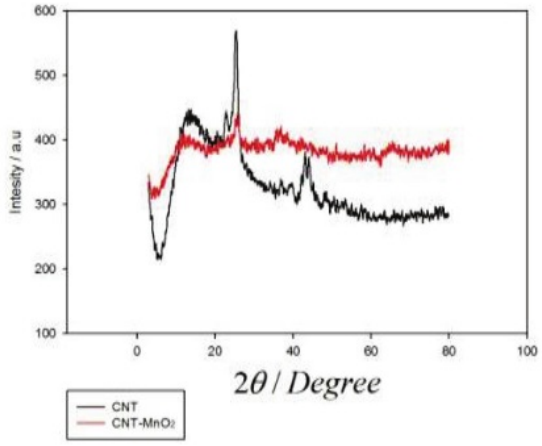

FIGURE 9. XRD Difractogram Analysis of CNT/MnO_2

The diffractogram data is then processed using JCPDS database and using the peak fitting software Origin Pro. 8. From the data diffractogram shows the results of the analysis of X-Ray Diffraction on CNT-MnO_2 material. Data from the first peak with the highest intensity is observed each at $2\theta = 26{,}63^o$ which is characteristic of the type of Carbon graphite, and at $2\theta = 43.97^o$ which is characteristic of the type of diamond Carbon. Data from the second peak with the highest intensity observed at $2\theta = 26{,}25^o$. Peak is a characteristic of MnO_2 manganitetype. It can be concluded that crystal CNT: MnO_2obtained in the positive material detected CNT and MnO_2. Measuring the size of CNT:MnO_2crystalline done using Schrerrer equation, so we get an average crystalline size of CNT: MnO_2 about 52.3 nm.

Tests conducted to determine the capacitance in the supercapacitors capacitance. Results obtained CNT/MnO_2 made prototypes double layer supercapacitors with aluminum foil current collectors and Cel gard separator. Electrolyte solution made with 1M Na_2SO_4 shows the value of specific capacitance of 7.86 F. Currently, the market has a capacitance capacitors <2 F in this study was obtained energy storage has a huge power savings, durable, and has a characterization cycling stability is quickly filling [3].

2 CONCLUSION

Based on the results, it can be concluded that the CNT:MnO_2 have been successfully synthesized with an average size of 40-50nm. CNT:MnO_2 has successfully coated on the SS Foil substrate perfectly using electrophoretic deposition method. CNT capacitance measurement results: MnO_2 shows the value of specific capacitance of 7.86 F / g. These results indicate that the CNT-MnO_2 electrode potential is very promising for further investigation and potentially a supercapacitor that has a high capacity and fast charge can be applied to electronic equipment, energy converters, even electric cars.

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