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Functionality Analysis of Carbon Nanosheet, Oxidized Carbon Nanosheet and Reduced Carbon Nanosheet Oxide by Using Fourier Transform Infra Red and Boehm Titration Method

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Abstract. Functionality analysis of carbon nanosheet (CNS), oxidized carbon nanosheet (o-CNS) and reduced carbon nanosheet oxide (r-CNSO) has been done by using fourier transform infra red (FTIR) and boehm titration methods. The results showed that the functional group differences between CNS, o-CNS and r-CNSO were variated in intensity value for FTIR spectrogram. CNS spectrogram showed a wide peak about 3400 cm^{-1} for hydroxyl group, bond C=C vibration in 1500 cm^{-1} , epoxy and alkoxy in 1200-1100 cm^{-1} . o-CNS spectrogram shows the carboxylic acid about 2300 cm^{-1} . Peak at 1700 cm^{-1} showed for C=C bond of epoxy and alkoxy functional groups were shown in 1250 cm^{-1} and 1125 cm^{-1} . Hydroxyl group of r-CNSO were showed in the 3441 cm^{-1} . The highest intensity of IR absorbance for hydroxyl group can be observed in r-CNSO. Carboxylic functional group in three carbon type was observed in 2337 cm^{-1} . Moderate peak at 1627 cm^{-1} showed C=C group. Epoxy and alkoxy were shown in 1172 cm^{-1} and 1056 cm^{-1} . The Boehm titration method showed that three carbon have differences amounts of funtional groups, except for the carboxylic and lactone groups. The CNS have funtional group concentration for fenolic, lactonic and base were 0.089, 0.069 and 0.082 mmol g^{-1} , respectively. The o-CNS have funtional group concentration for fenolic, lactonic and base were 0.089, 0.078 and 1.146 mmol g^{-1} , respectively. r-CNSO have funtional group concentration for fenolic, lactonic, carboxylic and base were 0.05, 0.1, 0.025 and 0.05 mmol g^{-1} , respectively.

Keywords : carbon nanosheet (CNS), boehm titration methods, o-CNS, r-CNS

1. Introduction

The performance of electrode is highly related with the performance of lithium battery and supercapacitor [1,2]. One of key-factor for the performance of electrode is functionality of electrode material [3]. One of common material used as an electrode is a carbon nanosheet (CNS). CNS can be made of gelam wood bark (GWB) [4]. It showed that the high electronic and ionic conductivity can be achieved after its surface modification [5]. Surface modification has been done by oxidation of the



CNS (CNSO) by Hummer method [6] and followed by the reduction of CNS-oxide by using liquid exfoliation [7] to have reduced carbon nanosheet oxide (rCNSO). The changing in CNS, CNSO and rCNSO functionalities can be detected by using FT-IR method and Boehm titration [8].

2. Methods

2.1 Preparation of Carbon Nanosheet (CNS)

GWB was cleaned by soaking in water for 12 hours and then dried under the sunlight for one day. GWB was cut into small pieces using scissors and put into blender to make powder. The mixture of powder 40 g GWB and 0.08 g KOH in 100 mL water was stirred for 3 minutes, placed in the hydrothermal reactor and heated in the electric baking oven at 200 °C. Under this condition, the pressure inside the reactor reached 20 – 30 bars. After 16 hours in the oven, the reactor was cooled to room temperature and atmospheric pressure. Hydrothermal treatment resulted torrefaction material [9].

Microwave pyrolysis was applied to the mixture of torrefaction material powder 20 g and graphite rod 4 g as microwave absorber in a crucible and microwave furnace. The mixture was put into microwave oven and heated for 20 minutes so that the temperature can reach 800°C. The result of this process was carbon nanosheets (CNS).

2.2 Oxidation of Carbon Nanosheet (CNS) and Reduction of Carbon Nanosheet Oxide (CNSO)

3 g of CNS was mixed with 3 g of NaNO₃ in erlenmeyer, and dissolved with 80 ml of H₂SO₄, followed by homogenizing with shaker for 1 hour. The addition of 9 g KMnO₄ into mixture were done after homogenizing and was shaken for 1 hour, in ice bath for maintained the temperature. The mixture was deployed from ice bath and allowed to reach 35 °C. The mixture was then stirred for 12 hours to form brown paste. The paste was added with 10 mL 32% of H₂O₂ and followed by distilled water as the mixture temperature reached room temperature. The precipitated was washed with distilled water and 3% HCl to neutralized. Finally filter sediment and ultrasound for 30 minutes and oven for 1 hour with the temperature of 120°C.

0.01 g of powdered graphite oxide was added to 10 mL of deionized water and sonicated for 24 hours to make reduce carbon nanosheet oxide (r-CNSO). The sonicated solution was centrifuged at 15.000 rpm for 1 hour. Light brown supernatant was precipitated resulted rCNSO. Liquid part was removed gently from the tube by using pipette.

2.3 Characterization functionality of CNS, o-CNS and r-CNSO

The functionality of CNS, o-CNS and r-CNSO are characterization with FTIR metode and Boehm titration. The Boehm titration method was applied to determine the acid and basic sites in the activated carbons [10]. The total acid sites were neutralized with 0.1 M NaOH solutions, and the basic sites were neutralized with a 0.1 M HCl solution. The reactions between the reagents and acidic oxygenated functional groups on the surface were based on the difference strength of the acid/base. The strength order of acidic and basic groups is as follow: carboxyl > lactone > phenol. The carboxylic and lactonic sites were titrated with a 0.1 M Na₂CO₃ solution, the carboxylic sites were titrated with a 0.1 M NaHCO₃ solution, and the phenolic sites were determined by the difference. For each determination, 50 mL of the solution was added to one gram of activated carbon in a glass container. The container

was inserted into vortex shaker for over one day. A 10 mL sample was titrated with 0.1 M of HCl or NaOH solutions [11].

3. Results and Discussions

Surface modification has been done by the oxidation of the CNS and followed by the reduction of o-CNS to get r-CNSO. The characterization steps were proceed by using FTIR and boehm titration to analyzed functional groups that featuring CNS, o-CNS and also r-CNSO. Preparation of CNS, o-CNS and r-CNSO can be visualized as discoloration of carbon. Figure 1 show visible color changes from green to black in the form of slurry.. The changing of color is an indicator for making such carbons [12]. In general, the color of the solution of carbon change from greenish solution (figure 1a) gradually, to blackish that suggests the phase formation in o-CNS was occurred (figure 1b).

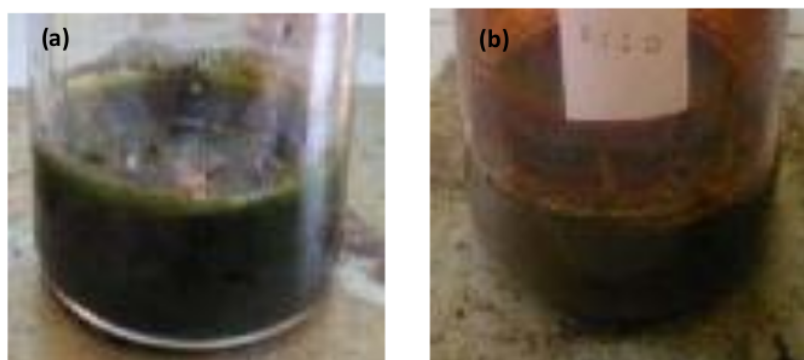


Figure 1. (a) Greenish solution after the addition of KMnO_4 , initial condition (b) the black, final condition, the formation of CNSO

Black solid slurry was dried at $120\text{ }^\circ\text{C}$ produced has the texture of soft, fragile and very hygroscopic consistency. Preparation of r-CNSO was done by using sonicator for o-CNS within 1 hour. This step was done because o-CNS reduced so that the product while have difference functional groups from the other.

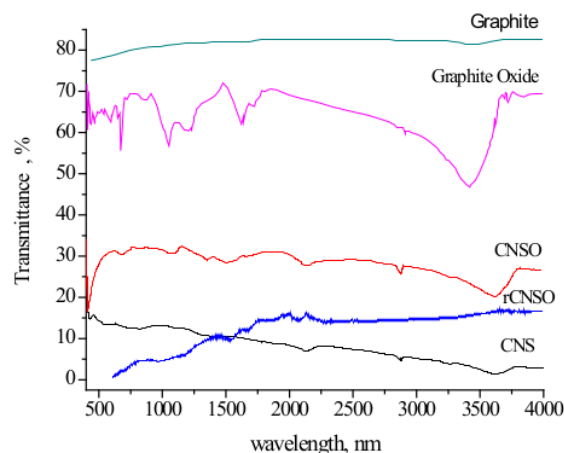


Figure 2. Spectrogram FTIR of CNS, o-CNS and r-CNSO

The result shows that the differentiation of functional group between CNS, o-CNS and r-CNSO is in intensity of FTIR spectrogram. Figure 2 showed that spectrum CNS, o-CNS and r-CNS. At which the CNS shows a wide peak in area 3400 cm^{-1} for hydroxyl group, bond C=C vibration in area 1500 cm^{-1} , epoxy and alkoxy in area $1200\text{-}1100\text{ cm}^{-1}$. Spectrogram of o-CNS showed that the carboxylic acid appear in 2300 cm^{-1} ; C=C in 1700 cm^{-1} . Epoxy and alkoxy are shown in 1250 cm^{-1} and 1125 cm^{-1} . The r-CNSO shows a hydroxyl group in the 3441 cm^{-1} . The hydroxy group is in highest intensity at r-CNSO. Carboxylic acid can be shown in 2337 cm^{-1} . Moderate peak at 1627 cm^{-1} is for C=C bond. Epoxy and alkoxy were shown in 1172 cm^{-1} and 1056 cm^{-1} .

Table 1. Functional group concentration

Samples	Functional group concentration, mmol g^{-1}			
	Base sites	Carboxylic	Phenolic	Lactonic
CNS	0.082	0.003	0.089	0.069
o-CNS	1.146	0.003	0.089	0.078
r-CNSO	0.050	0.025	0.005	0.100

The Boehm titration method showed that three carbon have differences amounts of functional groups, except for the carboxylic and lactone groups. The CNS have functional group concentration of phenolic is 0.089 mmol g^{-1} , lactonic is 0.069 mmol g^{-1} and base is 0.082 mmol g^{-1} . The o-CNS have functional group concentration of phenolic is 0.089 mmol g^{-1} , lactonic 0.078 mmol g^{-1} , and base is 1.146 mmol g^{-1} . r-CNSO have functional group concentration of phenolic is 0.05 mmol g^{-1} , lactonic is 0.1 mmol g^{-1} , carboxylic is 0.025 mmol g^{-1} and base is 0.05 mmol g^{-1} as shown in Table 1. It can be shown that the

oxidation treatment gives significantly the addition of base and lactonic groups concentration. There are no change in concentrations of carboxylic and phenolic. The concentration of functional groups decrease after reduction treatment except for lactonic. This can be interpreted that the treatment applied runs properly. This character appears with the additional oxygen bonded onto the surface of carbon in the form of C–O polar functional group that is responsible for increasing of the surface energy [13].

4. Conclusions

The conclusion of this research is that all carbons have the differentiation each other. The spectrograms show that the functionality of carbocilyc function for their CNS and o-CNS, while r-CNSO showed the difference functionality of each other.

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