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Synthesis and Characteristics of Nanoparticle NiO onto Activated Carbon from Cinnamon Bark Waste

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Abstract:
Cinnamon bark waste was converted into activated carbon as matrix for immobilization ofnanoparticle nickel oxide (NiO). The NiO/activated carbon was prepared by a combination of nanoparticle and activated carbon with ratio 1:2 using hydrothermal process. The functional groups of composite were obtained at 3636 cm^{-1} assigned OH bond and 501 cm^{-1} attributed to Ni-O bond. The XRD analysis showed that the composite has high crystalline phase. The SEM images showed spherical shape and blocked particles. The nitrogen adsorption desorption isotherm shows Type V indicating a combination of the meso and macropores. The result of this study proved that the NiO particle was successfully loaded onto the activated carbon from cinnamon bark waste.

Keywords: *Cinnamon bark, nickel oxide, activated carbon, composite, hydrothermal.*

1. Introduction

Activated carbon is a porous material containing of 85-95% carbonaceous compound [1]. In general, activated carbon was prepared by chemical physical activation under inert gas such as nitrogen. Pyrolysis process was carried at high temperature in range of 500-1000^{o}C [2]. However, preparation using hydrothermal process may decrease a temperature pyrolysis in the range of 250-400^{o}C [3].Cinnamon bark (Cinnamon burmanii) is agricultural by-product containing cellulose, hemicellulose and lignin including essential oil such as eugenol oil [4]. It is one ofthe potential commodities to be developed. The cinnamon bark has a distinctive odor, usually it used as a food flavouring.

In Indonesia, cinnamon production is quite large resulting in a lot of cinnamon bark waste. Therefore, this research was conducted to utilize cinnamon bark waste into activated carbon. However, the resulted activated carbon form cinnamon shows low thermal stability. It was good challenge to improve the activated carbon properties by the combination with metal oxide such as nickel oxide to form composite material. Composites are combination of materials containing different phase with more desirable properties [5].

The goal of this study is to synthesize of NiO/activated carbon composite from cinnamon waste and study its characterization.
Characterizations were carried out by the FTIR, XRD , SEM-EDX and GSA.

2. Experimental

2.1. Materials

Cinnamon bark waste was obtained at open area around essential oil industry, Yogyakarta, Indonesia. Chemical used in this study such as phosphoric acid (H3PO4), nitric acid (HNO3), nickel oxide and ethanol are from MERCK.

2.2. Synthesis of activated carbon

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The cinnamon bark waste was cut into small size in the in the range of 0.1 - 0.3 mm. It was washed and dried in to oven for overnight. The dried sample was impregnated with phosphoric acid 20% and refluxed at 85^{o}C in water bath for 5 h. The impregnated sample was washed to reduce the chemical content and neutralize to pH 6-7, then it was placed into the reactor for pyrolysis process at 350^{o}C for 10 h. Resulted activated carbon was immersed with 1M nitric acid for 5 hours, washed with distilled water and then dried into oven at 110^{o}C.

2.3. Preparation of NiO/activated carbon
Approximately, 50 g of NiO was dissolved in distilled water and ethanol (1:1). The mixture was rigorously stirred and added 100 g of activated carbon. The mixture was placed into hydrothermal reactor and heated into a graphite furnace at 300oC for 2 h. The resulted composite was washed with distilled water to reduce the ash content, and then it was dried in oven at 110 for 4 h.

2.4. Material characterization

The X-ray diffraction (XRD) technique was conducted to observe the crystallinity of the NiO/activated carbon [6]. The vibrational mode analysis of the composite was employed by the Fourier transform infrared (FTIR). The surface morphology and elemental analysis of sample were carried out by the SEM-EDX. The surface area of the composite was obtained by the gas sorption analyzer (GSA) at relative pressure in the range of P/Po = 0.05-0.3 [7].

3. Result and discussion

3.1. Surface morphology of NiO/activated carbon composite

The SEM-EDX analysis of the composite prepared by the hydrothermal process is shown in Fig. 1. The spherical NiO with block like particles was observed at on the surface of composite. It can be inferred that the composite of NiO/activated carbon with highly pores structure which are closed by the fraction of particles. The EDX analysis showed the elemental compositions of the composite consisting of nickel (45.7 %), oxygen (31.6 %), carbon (15.3 %), phosphate (2.0 %), Chloride (1.9 %) and impurities elements. The composite showed the highest elements which related to the loading nickel onto the activated carbon [8]. In addition, the high oxygen contain reveals the high oxygen functional groups on the surface composite.

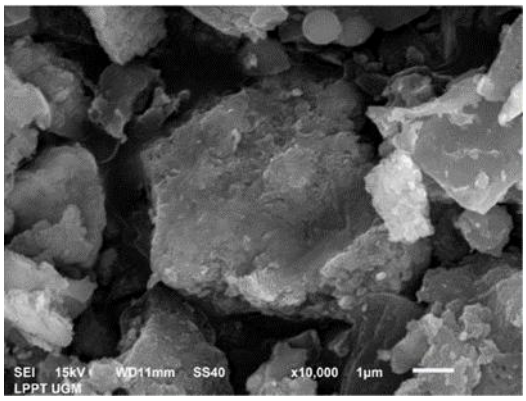

Figure 1. Scanning electron micrograph of the NiO/activated carbon composite

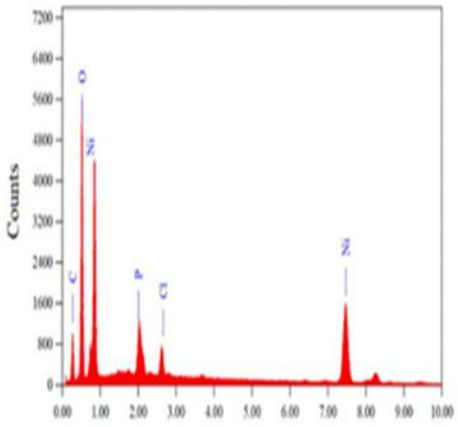

Figure 2. EDX Spectrum of the NiO/activated carbon composite

3.2. Structure morphology of NiO/activated carbon composite

The XRD patterns of NiO/AC composites are show Fig. 3. There are five strong intensity peaks at 2θ

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19,43˚; and 25,40˚ show the characteristics of amorphous structure of activated carbon, and the spesific peaks of NiO/activated carbon were obtained at 2θ = 33,24˚; 38,71˚; 52,13 and 59,12˚ consisting of face center cubic (FCC) [9]. The hkl values of each other of 2θ are (110), (111), (020) and (202).TheNiO characteristic peak gradually turns to be sharper and higher with the increase of NiO content [10].

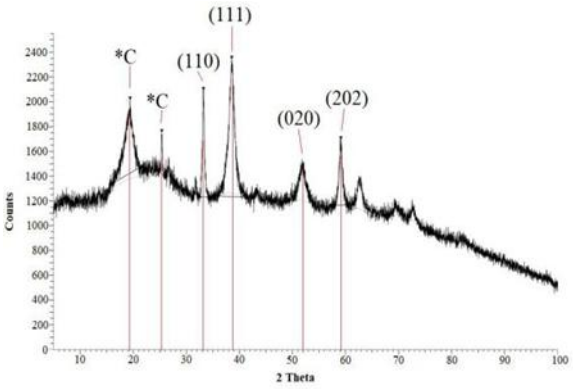

Figure 3. Diffractogram of the NiO/activatedcarbon composite

3.3. Functional groups of NiO/activated carbon composite

The functional of the NiO/activated carbon composite was determined by the vibrational mode of FTIR as shown Fig 5. The wave number of the composite was investigated between 4000-400cm^{-1}. The peak at 3636 cm^{-1} is only fond in the composite which is allocated to the OH stretching vibration. The peaks at 2050 which are presence in activated carbon and NiO/activated are ascribed to the vibration of C≡N triple bond [11]. The peak at 1600 cm^{-1} is ascribed to the NiO molecule in the NiO/activated carbon. The band at 1030 cm^{-1} is atributed to the C-O bonding. The band at 501 cm^{-1} is related to the presence of the Ni-O bonding [12].

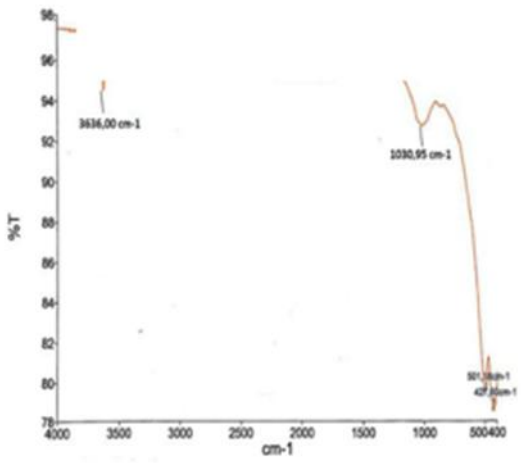

Figure 4. FTIR Spectra of the activated carbon from cinnamon waste

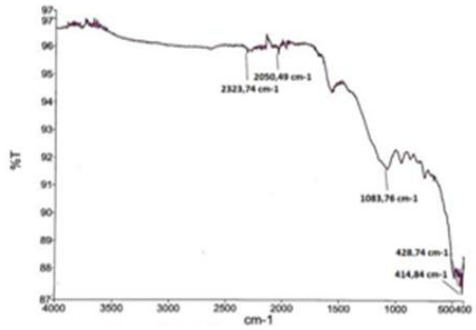

Figure 5. FTIR Spectra of the NiO/activated carbon composite

3.4. Surface area of NiO/activated carbon composite

The BET surface area of the composite is shown in the Figure 6. The nitrogen adsorption desorption isotherm shows type V with wide hysteresis loop indicating the presence of meso and macroporosity [13,14]. The BET surface area of the NiO/activated carbon prepared from the cinnamon waste is 17.9 m^{2}g^{-1}.

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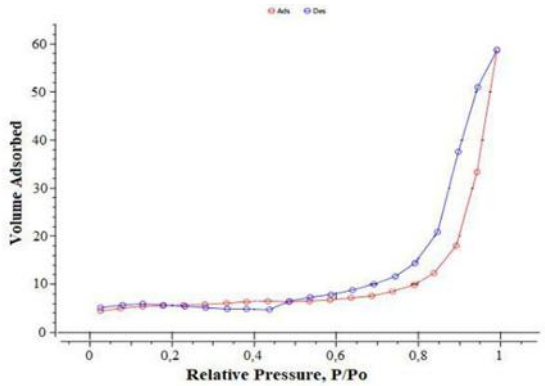

Figure 6. Nitrogen adsorption desorption isotherm of the NiO/Activated Carbon Composite

4. Conclusion

Characteristics of the NiO/activated carbon composite was investigated by the analysis equitments. The surface morphology of the composite shows the pores of composite which was closed by fraction of particles. The nitrogen adsorption desorption showed hysteresis loop relating to the meso- and macropores. The nickel was loaded onto activated carbon to form a composite of NiO/activated carbon. The result of this study exhibited that the composite was succsefully obtained from the waste of cinnamon waste.

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