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Preparation of ZnO-CuO composite photocatalyst using the sonochemical method

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Abstract. This research aims to synthesize ZnO-CuO composites to be applied as a catalyst material that acts on visible light. The preparation process of ZnO-CuO composite using sonochemistry method. Percursor used in this research is Zn $(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ and $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ with a ratio of 1: 1, 2: 1, 3: 1, 4: 1, 5: 1 and variations in the exposure time used 70, 90, 105, 120, 135 minutes. The result of the FTIR spectral characterization shows that the formation of new peak is ZnO-CuO composite wave at 400-600 nm area, with UV-DRS Obtained ZnO band gap value of ~ 3.25 eV and ZnO-CuO 2: 1 ~ 2.75 eV.

1. Introduction

The application of photocatalyst can be used to generate electricity, as reported in previous research that the CuO as the electrode on the PV-EC cell [1]. The applications of the environmental aspects are, ZnO photocatalyst is used as a sensitizer and a starter on the degradation of humic acid in peat swamp water. In photodegradation of humic acid mineralized into more simple products [2].

Semiconducting zinc oxide (ZnO) is the best alternative choice after TiO_2 in its application as a semiconductor photocatalyst [3]. As a basic consideration is the semiconductor ZnO resistant to corrosion, which has an energy gap ($E_g = 3.2$ to 3.5 eV), and cheaper than TiO_2 . So that ZnO can be activated by UV radiation (ultraviolet), which resulted the efficiency of ZnO photocatalytic lower in visible light. Increasing of the photocatalytic activity of ZnO in visible light can be done by reducing the energy band gap ZnO by the addition of another semiconductor which having a narrow band gap. The incorporation of ZnO has been done with other metal oxides and sulfides such as TiO_2 , SnO_2 , WO_3 , NiO, Fe_2O_3 , and CuO [4-8]

Preparation of ZnO-CuO nano-composites can be done by several methods such as chemical synthesis method solids, coprecipitation, sol gel, and sonochemical (sonochemistry). Sonochemical method (Ultrasonic Irradiation) is used to synthesize ZnO-CuO Nanocomposite because it has several advantages such as the synthesis of an inexpensive, fast, easy to control the size, the reaction temperature is low and the resulting particles have a high homogeneity. Sonochemical synthesis of nanomaterials using the method performed by using ultrasonic waves [9-12].

Research will be carried out to produce ZnO-CuO composites having a high photocatalytic efficiency in the area of visible light, compared to the semiconductor ZnO and CuO.



2. Tools and Materials

2.1. Tool

The tools used in this research are Spectrophotometer UV/Vis, Box photocatalyst consisting of a UV lamp and a magnetic stirrer brands Selecta Multimatic, Ultrasonic (45 Hz) brand of Ultrasonic Cleaner 968, X-Ray diffraction (XRD), Fourier Transform Infrared (FTIR), centrifuges, Oven brands Xu France Etuves, 225 Furnance (Neycraft), analytical Balance (Kern), and glassware: flask, beaker, a pipette and stir bar.

2.2. Materials

Materials research in this study are zinc acetate dihydrate $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ (Merck), $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (Merck), KOH (Merck), ethanol, Cetyltrimethylammonium bromide (CTAB, Merck), ZnO (Merck), CuO (Merck) and distilled water.

3. Experimental

3.1. Synthesis of ZnO-CuO catalyst

Mixed 0.160 g Zn $(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ 0.2497 g, 0.5611 g and 0.1093 g CTAB KOH diluted with distilled water to a volume of 30 mL solution. Then stirred for 15 minutes. The mixture is heated at a temperature 50°C and ultrasonic at a frequency of 45 Hz for one hour. The mixture was further centrifuged at a speed of 5000 rpm for 10 minutes, the filtrate was discarded. The sediment was washed with 30 mL of distilled water and 20 mL of absolute ethanol. The precipitate was oven-dried at 120°C temperature for 2 hours. Calcined at 500°C temperature for 5 hours, by raising the temperature gradually from 200 for 30 minutes, 300 for 45 minutes, and a temperature of 400 for 60 minutes. The same procedure was performed by varying the composition ratio of precursors, namely $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O} : \text{CuSO}_4$ (2: 1, 3: 1, 4: 1, 5: 1) [13].

3.2. Characterization of ZnO-CuO Catalyst

Characterization by using UV-DRS performed to calculate the value of band gap of ZnO-CuO catalysts. Later in the characterization by FTIR instrument which aims to determine the location of the peak of the information contained in the catalyst oxide ZnO-CuO generated.

4. Results and Discussion

4.1. Characterization

4.1.1. Catalyst Characterization by Fourier Transform Infra Red (FTIR).

Characterization of ZnO-CuO use *Fourier Transform Infrared*(FTIR) aims to analyze the absorption bands of the vibrations of functional groups ZnO. The working principle of FTIR is based on the amount of absorption of light by a sample. If a sample is passed by infrared radiation, the molecules will absorb energy and there is a transition between the basic vibrational level (ground state) and the excited vibrational levels (excited state) [14].

FTIR analysis was conducted to see the vibrations of functional groups ZnO-CuO nanoparticles. Measurements were taken at a wavelength range of $400\text{-}4000\text{ cm}^{-1}$. From this test result chart % Transmittance (% T) of the wavenumber (cm^{-1}). FTIR will generate absorption peaks associated with the frequency of vibration of the atoms making up the bonds of a material.

In this research, FTIR analysis done catalyst sample of pure ZnO, CuO purely as a comparison standard for catalyst samples are synthesized ZnO-CuO. FTIR spectra of ZnO-CuO catalysts can be seen in Figure 1

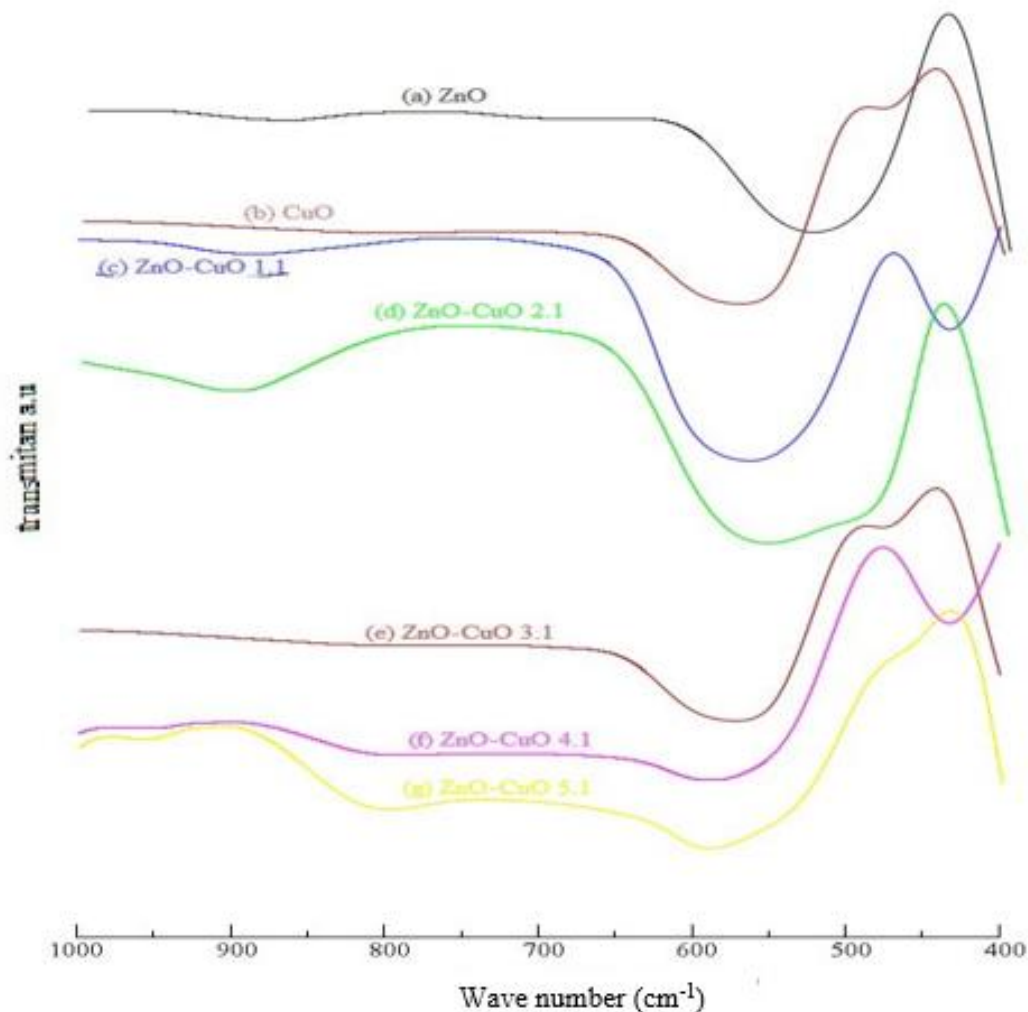


Figure 1. FTIR spectra of (a) ZnO, (b) CuO, (c) ZnO-CuO 1.1, (d) ZnO-CuO 2.1, (e) The ZnO-CuO 3.1, (f) the ZnO-CuO 4.1, (g) ZnO-CuO 5.1

From the results of the FTIR spectra Figure 1 shows that the catalyst synthesis of ZnO-CuO 2:1 shows that formed a new absorption band showing the stretching vibration of the metal oxide composite ZnO-CuO, while the comparison of the other isn't formed Composite ZnO-CuO but only CuO stretching vibration peaks are formed. This is because period of relative molecular greater than Cu Zn resulting in the peak of CuO. In the range of 400-600 nm wave number is stretching vibration area absorption of the metal oxide [15].

4.1.2. Band Gap Analysis of ZnO and ZnO-CuO 2: 1 with UV-DRS

Band gap analysis was performed using UV-DRS to determine the value of the band gap. The band gap is a gap between the valence band is full of electrons with an empty conduction band electrons. Values in the semiconductor band gap is very important because it affects the performance of the semiconductor in a stream of electrons and holes [16-21].

In this research, the analysis by using UV-DRS is only carried out on samples of pure ZnO-CuO and ZnO catalyst is synthesized ZnO-CuO 2:1 a sample ZnO-CuO catalysts maximum seen from the results Phototransformation LAS. Analysis of samples obtained from the relationship% Reflectance on wavelength. The data obtained will be processed to obtain estimates of the value of the band gap of each sample. which the results we got can be determined how the pure ZnO influenced with catalyst

synthesis ZnO-CuO 2:1 against the tire energy gap of each sample. Graph band gap of ZnO and ZnO-CuO 2:1 shown in Figure 2

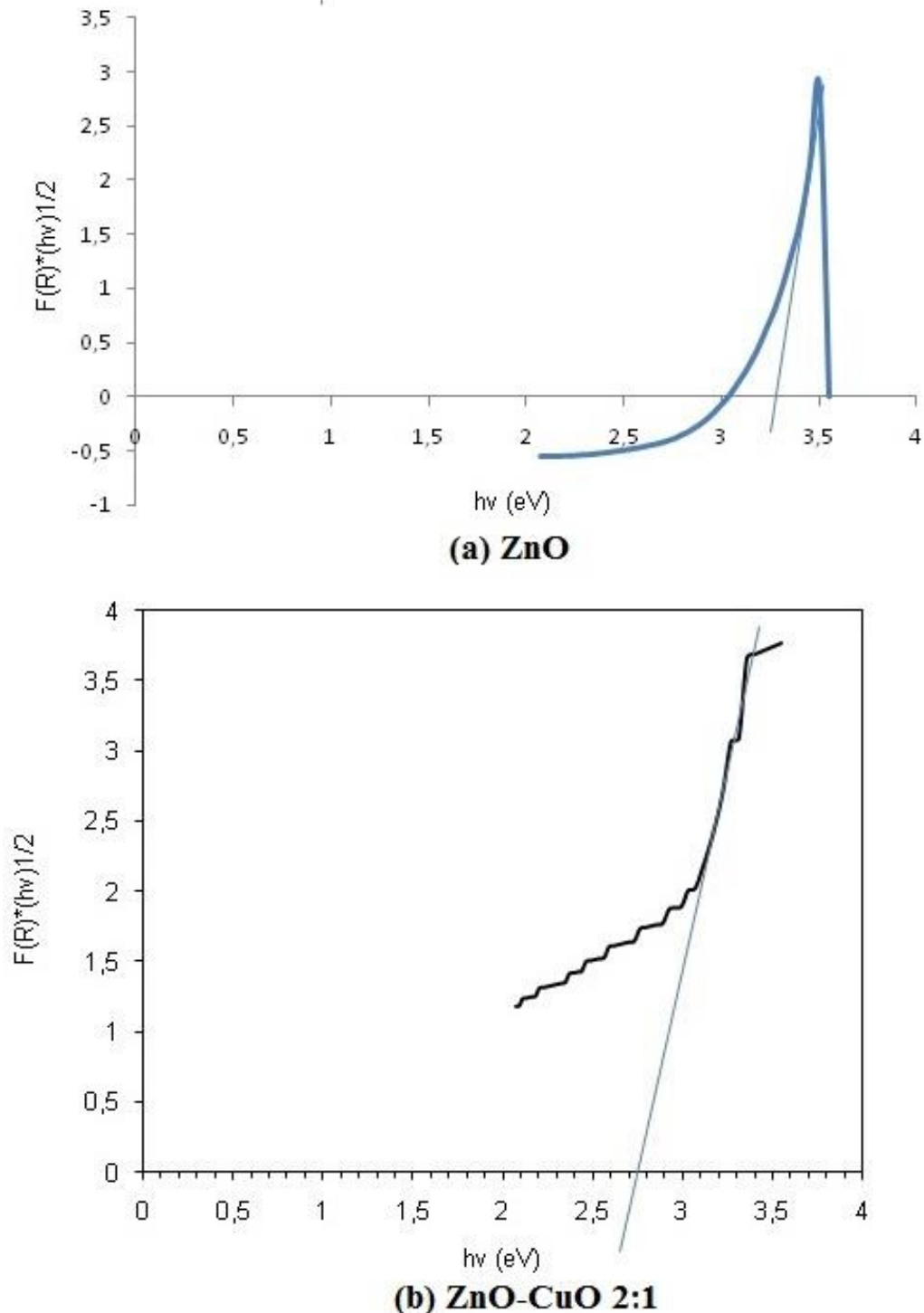


Figure 2. Graph band gap value (a) ZnO, (b) ZnO-CuO 2: 1 by using UV-DRS.

The value of the band gap of the semiconductor can be calculated using Kubelka-Munk theory. The value of the band gap of ZnO and ZnO-CuO 2: 1 calculated from spectral reflectance Based on Figure 2, visible band gap value obtained for ZnO \sim 3.25 eV and ZnO-CuO 2: 1 \sim 2.75 eV. This shows that with the addition of another semiconductor is CuO on ZnO catalyst can lower the value of the band

gap. small band gap value obtained on ZnO-CuO 2: 1. This makes the photocatalytic activity more effective than ZnO, CuO and ZnO-CuO composite other comparisons.

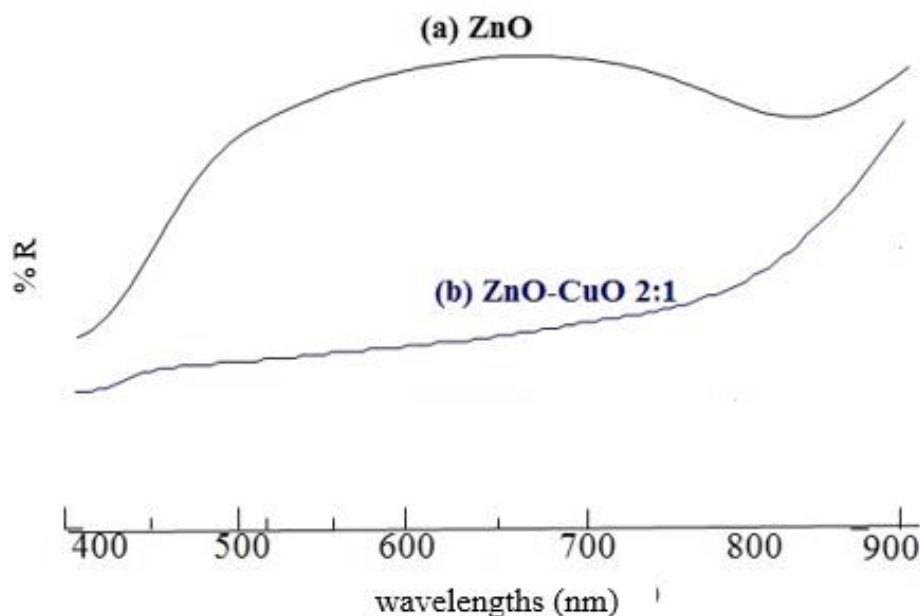


Figure 3. Absorbance Spectra Catalyst namely: (a) CuO, (b) ZnO-CuO 2: 1 and ZnO of Spectrophotometer UV-DRS

From figure 3 can be seen that the Merger ZnO with CuO having a narrow band gap can reduce the band gap ZnO itself so that the absorption of ZnO shifted to the visible light region.

4.2. Conclusions

Based on this result, FTIR spectral characterization shows that the formation of new peak is ZnO-CuO composite wave at 400-600 nm area, with UV-DRS Obtained ZnO band gap value of ~ 3.25 eV and ZnO-CuO 2: 1 ~ 2.75 eV.

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