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# Characterization of $CuCrO_2$ Material Produced Using Sol-Gel Method

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**ABSTRACT**

This study aimed to analyze $CuCrO_2$ material produced using the sol-gel method. The characterization used is X-ray Diffraction (XRD) and Scanning Electron Micoscope-Energy Dispersive Spectroscopy (SEM-EDS). The sample was made by weighing the raw material and measuring the solution with a measuring cup according to stoichiometric calculations, then dissolving it, burning it, grinding it, calcining it, sintering it, forming it into pellets, and re-sintering it. The XRD results show that the main phase of the sample is $CuCrO_2$ and impurities ($CuCr_2O_4$). From the SEM results, the average particle size is 155.948µm.

**Keyword:** Delafossite, $CuCrO_2$, sol-gel method.

**ABSTRAK**

Penelitian ini bertujuan untuk menganalisis material $CuCrO_2$ dengan menggunakan metode sol-gel. Karakterisasi yang digunakan adalah X-ray Diffraction (XRD) dan Scanning Electron Micoscope-Energy Dispersive Spectroscopy (SEM-EDS). Pembuatan sampel dilakukan dengan cara menimbang bahan baku dan mengukur larutan dengan gelas ukur sesuai perhitungan stoikiometri, kemudian dilarutkan, dibakar, digiling, dikalsinasi, disinter, dibentuk menjadi pelet, dan disinter kembali. Hasil XRD menunjukkan bahwa fasa utama dari sampel adalah $CuCrO_2$ dan pengotor ($CuCr_2O_4$). Dari hasil SEM rata-rata ukuran partikel adalah 155,948µm.

**Kata kunci:** *Delafossite*, $CuCrO_2$, Metode *sol-gel.*

## 1. Introduction

Related studies on delafossite oxides are of interest in examining the properties of TCO and their applications as photocatalysts for hydrogen evolution [1] – [4]. $AMO_2$ delafossite compounds derived from the mineral $CuFeO_2$ are quite attractive materials because of their ability to be stabilized with a large number of A and M cations and over a wide range of off-stoichiometric values, leading to different physical properties [5] – [7].

Delafossite oxide $CuRO_2$ (R¼ trivalent cations) is one of several systems with triangular antiferromagnetic sub-lattices. $CuRO_2$ has a layered structure with space group R-3M, which is seen as an alternative arrangement of edge-to-edge RO 6 octahedral ($RO_2$) layers and a Cu layer. The magnetic properties of these layered compounds have attracted much attention since geometric frustration in the magnetic triangular *sublattice* at the R site leads to interesting properties such as field-induced multistep magnetization changes [8]. Research by Amami et al. [9] produces Al-doped $CuCrO_2$ material that dilutes its magnetization and screws up order antiferromagnetic. In a study conducted by Luo et al. regarding the effect of Ni doping on $CuCrO_2$ material, it turns out that adding Ni doping can increase characteristic ferroelectric and magnetization against $CuCrO_2$ [10] material.

Based on the above explanation, this study aimed to synthesize $CuCrO_2$ materials using the sol-gel method to obtain unique properties on the characteristics of $CuCrO_2$ materials.

## 2. Method

The materials used are $Cr_2O_3$, $Cu(NO_3)$, $CO(NH_2)_2$, aquadest and 65% $HNO_3$ solution. The materials were weighed based on the compositions given in Table 1.

Table 1. Material Composition

| Ingredients | Mass |
|---|---|
| $Cr_2O_3$ | 1.52 g |
| Aquadest | 10 ml |
| $HNO_3$ 65% | 4.17 ml |
| $Cu(NO_3)$ | 4.83 g |
| $CO(NH_2)_2$ | 2.40 g |

The $Cr_2O_3$, $Cu(NO_3)$, and $CO(NH_2)_2$ were dissolved in 65% $HNO_3$ solution using a magnetic stirrer on a hotplate at 210 rpm. Next, the sample was heated at 90°C for 2 hours; after 2 hours, the magnetic stirrer was taken and heated for 1 hour at 200°C. Then, process the sample has turned into a gel and is then burned by inserting the sample into the furnace for 3 hours at 300°C. After this process, the sample will become powder. Next, the materials were mixed into the mortar and crushed manually for 3 hours, then calcined in a furnace with a temperature of 780°C for 3 hours, then crushed again for 3 hours. Next, the sample powder is pressed (350 MPa ) into a pellet with a diameter of 10 mm and a thickness of 2 mm. Finally, the pellets were sintered again at 1000°C for 3 hours.

Furthermore, the pellets were characterized using X-ray Diffractometer (XRD) and Scanning Electron Microscopy/Energy Dispersive Spectroscopy SEM/EDS

## 3. Results and Discussion

### *3.1 XRD Analysis*

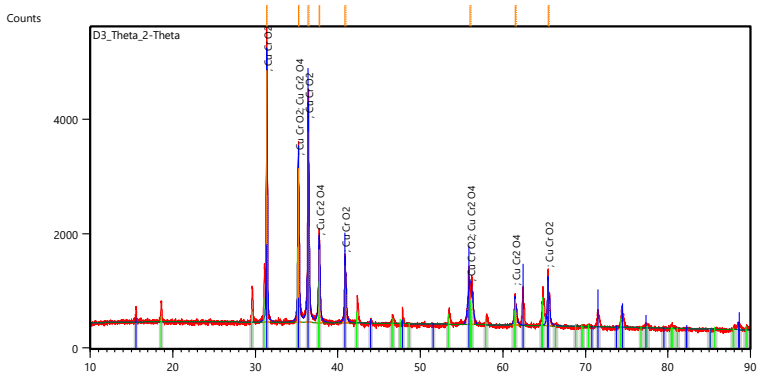


Figure 1. XRD pattern of $CuCrO_2$

Figure 1 shows the XRD results on the $CuCrO_2$ sample. XRD test was carried out to determine the formed phases. From the diffraction pattern in Figure 1, the $CuCrO_2$ phase is obtained as the main phase and $CuCr_2O_4$ phase as the impurity phase. The percentage fraction $CuCrO_2$ phase was 68%, and $CuCr_2O_4$ was 32% phase, which was analyzed using the HighScore Plus software with the database, namely COD 2021.

Table 2. Crystallite size of $CuCrO_2$

| Peak | Bobs. [°2Th] | B std. [°2Th] | Peak post. [°2Th] | B struct. [°2Th] | Crystallite size [Å] |
|---|---|---|---|---|---|
| 1 | 0.049 | 0.008 | 31.397 | 0.041 | 2013 |
| 2 | 0.098 | 0.008 | 35.220 | 0.09 | 926 |
| 3 | 0.098 | 0.008 | 36.423 | 0.09 | 929 |
| 4 | 0.118 | 0.008 | 37.746 | 0.11 | 763 |
| 5 | 0.197 | 0.008 | 40.910 | 0.189 | 449 |
| 6 | 0.63 | 0.008 | 56.061 | 0.622 | 145 |
| 7 | 0.315 | 0.008 | 61.519 | 0.307 | 301 |
| 8 | 0.315 | 0.008 | 65.521 | 0.307 | 308 |
| Average | | | | | 729.25 |

Crystallite size value was calculated using the Scherer equation using HighScore plus calculator from the XRD testing data by selecting the eight highest peaks. The average size obtained crystals on $CuCrO_2$ samples was 729.25Å.

*3.2 SEM Analysis*

Characterization SEM is used to analyze the surface morphology of the $CuCrO_2$, as seen in Figure 2.

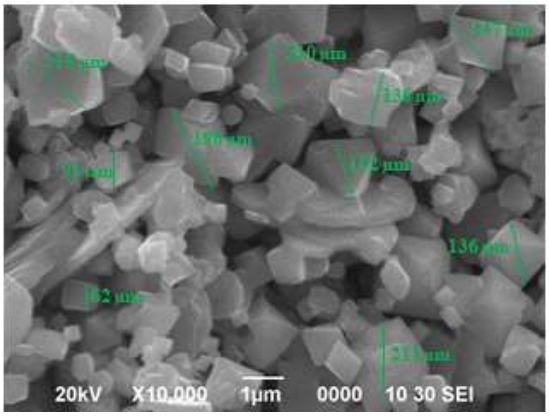


Figure 2. SEM results of $CuCrO_2$ samples with a magnification of 10,000 times

As seen in Figure 2, the sample has a hexagonal shape, which is distributed evenly with different sizes. The calculation of the particle size was conducted using ImageJ software, in which the average size of the sample is 155.948 µm

*3.3 SEM-EDS Analysis*

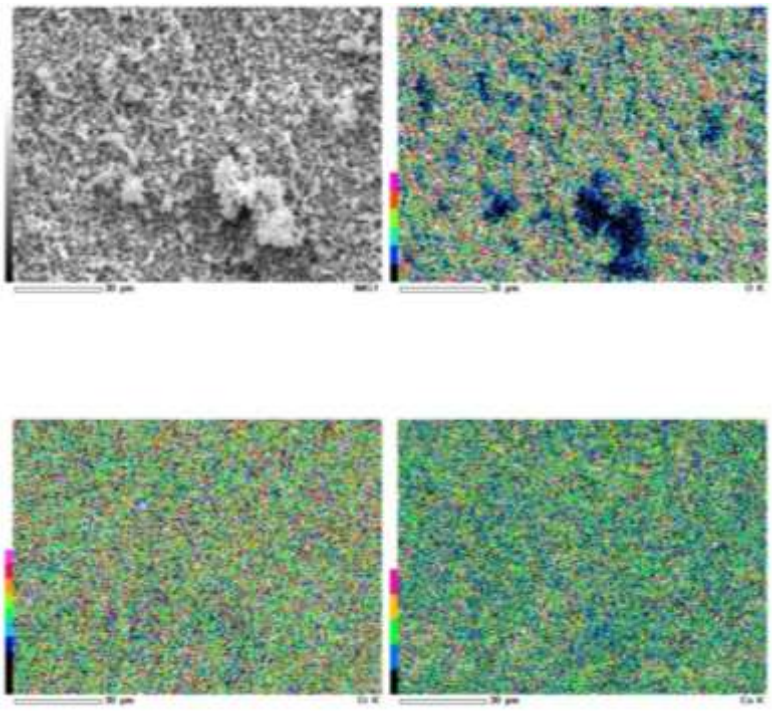

Figure 3. The elemental mapping of $CuCrO_2$ samples with 1000X magnification

As shown in Figure 3, the percentage of Cu and Cr mass elements was 39.47% and 42.28%, respectively, which is sufficiently high and tends to spread equally. In contrast, elemental O was 15.24% with a low mass percentage but distributed evenly.

## 4. Conclusion

The $CuCrO_2$ has been produced successfully using the sol-gel method. The XRD pattern shows that the dominant phase is the $CuCrO_2$ phase even though there are some impurity peaks. Particle average size on SEM testing is 155.948 µm, and seen clearly on mappings SEM-EDS testing that patterns on Cu and Cr elements are distributed evenly.

## 5. Acknowledgments

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