PREPARATION AND CHARACTERIZATION OF YTTRIUM BARIUM COPPER OXIDE (YBCO) SUPERCONDUCTOR WITH ADDITION OF COBALT OXIDE (Co₃O₄)

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Abstract
This study was carried out to investigate the electrical properties of YBCO sample as superconductor and the effect of addition of Co₃O₄ on the superconducting properties of YBCO superconductor. The YBCO sample was prepared by solid state reaction route. The samples were prepared by each with weight percentage of cobalt oxide of x= 0.00, x= 0.01, x= 0.02 and x= 0.03. Electrical Conduction by Multimeter, Fourier Transform Infrared (FTIR), Critical temperature (Tc) measurement, X-ray Diffraction (XRD), and Scanning Electron Microscopy (SEM) were conducted for analysis. Multimeter showed all samples were in electric conduction, FTIR showed that carbonyl compound in the sample was removed after calcinations. Tc measurement showed that the critical temperature of sample of x= 0.02 was increased compared to sample of x= 0.00. XRD showed all samples have orthorhombic structure and SEM showed that the grain size was increased as increased the cobalt addition in YBCO superconductor. Besides, the EDX also showed the composition of elements YBCO were tally with chemicals used for pure YBCO and addition cobalt oxide into YBCO superconductor.

Keywords: Critical temperature (Tc), solid state, YBCO superconductor

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Introduction
Yttrium barium copper oxide (YBCO) superconductor is most currently in research because the preparation is simple, reliable and low cost (Tang et al., 2016, Wei et al. 2016). It also gives higher critical temperature (Tc) that also provides high current applications (Malik et al., 2016). The addition of nanomaterial in high critical temperature will enhance the superconductor properties due to its easy process (Salama et al., 2016, Dadras et al., 2017). Besides, the addition of nanosized material also can improve the critical current density (Jc) on pinning centres of superconducting matrix (Kumar et al., 2016). Today, many researchers reveal the superconductors in different type of materials that can increase the critical temperature, Tc of superconductors such as the addition 15% of silver, Ag which has metallic properties on YBCO superconductors (Malik et al., 2016), the addition of cerium oxide, CeO₂ with 1% increased the superconductor properties on bismuth strontium calcium copper oxide (BSCCO) (Bartuneck and Smrckova, 2010) and the addition of 2% YBCO on BSCCO sample (Arlina et al., 2015). Element substitution on sample plays an important role in enhancing superconductivity properties (Salama et al., 2016). However, the determination of the addition Co₃O₄ into YBCO sample is still rare and less of study was conducted. Hence, this study will determine the capability of Co₃O₄ in YBCO superconductors.
Methods

The chemicals used in this study were yttrium oxide (Y$_2$O$_3$), barium carbonate (BaCO$_3$), copper (II) oxide (CuO) with cobalt oxide (Co$_3$O$_4$), 1 M hydrochloric acid (HCl) to acetone of 99% purity and distilled water. The yttrium barium copper oxide (YBCO) with the addition of Co$_3$O$_4$ samples was prepared by mixing and reacting the stoichiometric amounts of the corresponding oxide. For the method, the yttrium barium copper oxide (YBCO) was prepared according to ratio of 1:2:3 or YBCO (123). Superconductor powders with nominal starting composition of YBa$_2$Cu$_3$O$_y$ (Co$_3$O$_4$)$_x$ (x = 0–0.05 wt %) were prepared based on the stated ratio, x is representing the percentage of cobalt oxide that was added. The samples of YBCO were prepared by using solid state reaction route which all the powder compounds were mixed in mortar and ground with pestle for approximately one hour to make all compounds homogenized. Next, the samples were calcined at 900 °C for 24 hours in order to remove CO$_2$. Then, after a slow cooling process in the tube furnace, the calcined powder samples were grounded again. To get homogenous mixture, a second calcination was performed at 900 °C for 24 hours. After slow cooling process at room temperature, the samples were grounded again and pressed into a pellet form under pressure about 60 kN cm$^{-2}$. The pellet samples were sintered again at 920 °C for 24 hours in the tube furnace. Next, the YBCO samples were examined by multimeter to determine the presence of electric conduction, Fourier Transform Infrared (FTIR) spectrometer to proof the presence of carbonyl group before and after calcination and $T_c$ measurement to obtain the critical temperature of YBCO samples (Schuetze, 2004) with or without Co$_3$O$_4$. The X-Ray diffraction was used to determine the superconducting phase in YBCO samples by using Bragg’s Law formula. Lastly, SEM was used to observe the microstructure surface and indirectly determine the percentage of elements in the sample by energy-dispersive x-ray spectroscopy (EDX).

Results and Discussion

Table 1 shows the presence of electrical conduction of pellet samples by using Multimeter. The sample was analyzed using FTIR to proof the presence of carbonyl, C=O group in the compound that comes from chemical of barium carbonate, BaCO$_3$ before and after the calcination process at 900 °C. Based on the result obtained, before the calcination, there is an absorption band of carbonyl group at 1420.50 cm$^{-1}$ wavenumber. However, after the calcination process at 900 °C, there was no band appeared. This result showed that the carbon dioxide was removed at high temperature. The calcination process was needed to help the compound turn to metal oxides rather than metal carbonate. Thus the calcination process is important in order the sample to become a superconductor. Figure 1 showed the spectrum of FTIR before and after calcination process.

<table>
<thead>
<tr>
<th>Sample (wt %)</th>
<th>Electric Conduction</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>Yes</td>
</tr>
<tr>
<td>x= 0.00</td>
<td>/</td>
</tr>
<tr>
<td>x= 0.01</td>
<td>/</td>
</tr>
<tr>
<td>x= 0.02</td>
<td>/</td>
</tr>
<tr>
<td>x= 0.03</td>
<td>/</td>
</tr>
<tr>
<td>x= 0.04</td>
<td>/</td>
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</tbody>
</table>
Figure 1 Spectra of FTIR before and after calcination process.

Figure 2 and 3, shows the critical temperature, $T_c$ for added sample with $x = 0.02$ wt% and non-added sample, $x = 0.00$ wt% sample at 58 K and 87 K. The addition of cobalt oxide into YBCO superconductor sample increased the critical temperature value. There are several causes that can increase the $T_c$ of superconductor such as cooling rate during calcinations and final sintering. The longer the time for cooling rate, the higher possibilities for the sample to increase the $T_c$ (Montevecchi and Indekeu, 2000).

Figure 2 Critical Temperature, $T_c$ of YBCO non-added sample ($x = 0.00$ wt%)
Figure 3 Critical Temperature, $T_c$ of YBCO with Co$_3$O$_4$ added sample (x = 0.02 wt%).

Figure 4 shows the XRD results for sample x = 0.00 – 0.03. Y123, single phase was clearly observed in all samples. However, as the Co$_3$O$_4$ was added with different percentage values, certain peaks became more intense from x= 0.0 to x= 0.02 and slightly decreased when x= 0.03. The x = 0.00 XRD pattern was used as a reference. The intensity increased from x= 0.00 to x= 0.03 due to the fact that the addition of magnetic material, which cobalt oxide will improve the properties of material of YBCO (Lavakumar, 2017). The Miller indices were used to determine the lattice parameters ($a$, $b$, $c$ parameters). The lattice parameters of the unit cell for all samples were summarized in Table 2. Based on the table, the lattice parameters for all samples remained and showed that the structure of all samples were orthorhombic geometry.

SEM micrograph of cobalt oxide added to YBCO samples were shown in Figure 5 (a), (b), (c) and (d) with magnification of 2000 X. This micrograph clearly seen that the grain size were increased as the increased percentage weight of cobalt oxide from the pure YBCO sample. The micrograph showed the superconducting grain was well connected. As the x= 0.01 percentage weight of cobalt was added to YBCO sample, there were pores anticipate between the regions of well-connected grains. The pores between the grains also were increased with the increasing of cobalt percentage weight when x= 0.02 and x= 0.03. EDX were used to assure the composition of elements in the samples. The spectra of EDX were shown in Figure 6 (a) and (b).
Figure 4 XRD pattern of YBCO with Co$_3$O$_4$ added sample from x= 0.00 wt% to x=0.03 wt%.

Table 2 Lattice parameters of the unit cell.

<table>
<thead>
<tr>
<th>% Co$_3$O$_4$</th>
<th>Lattice parameters (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>a</td>
</tr>
<tr>
<td>x= 0.00</td>
<td>3.817</td>
</tr>
<tr>
<td>x= 0.01</td>
<td>3.818</td>
</tr>
<tr>
<td>x= 0.02</td>
<td>3.818</td>
</tr>
<tr>
<td>x= 0.03</td>
<td>3.818</td>
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</tbody>
</table>
Figure 5 SEM micrograph of YBCO sample with Co₃O₄ weight percentage of (a) x= 0.00, (b) x= 0.01, (c) x= 0.02 and (d) x= 0.03 with magnification of 2000 X.
Figure 6 EDX spectrum of YBCO sample with (a) x= 0.00 and (b) x= 0.02

Conclusion
Superconducting ceramic material of YBCO with a ratio of 1:2:3 compositions were analyzed. The critical temperature ($T_c$) measurement shows that the addition of cobalt oxide to YBCO superconductor was increased from 58 K of x= 0.00 wt% to 87 K of x= 0.02 wt%. The XRD of YBCO samples proved that the sintered material consist of Y123, single phase. All samples showed the structure of YBCO orthorhombic structure. SEM characterization showed that the grain size increased as increasing of the weight percentage of cobalt oxide.

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