Microwave Synthesis of BCNO/SiO₂ Nanocomposite Material

I D Faryuni¹*, F Ramdhani¹, J Sampurno¹, B W Nuryadin², F A Noor³ and F Iskandar³

¹ Department of Physics, Faculty of Mathematics and Natural Sciences, Tanjungpura University, Pontianak, Indonesia
² Department of Physics, Faculty Sciences and Technology, UIN Sunan Gunung Djati Bandung, Cibiru-Bandung, Indonesia
³ Physics of Electronic Materials Research Division, Faculty of Mathematics and Natural Sciences, Institut Teknologi Bandung, Bandung 40132, Indonesia

*irfana@physics.untan.ac.id

Abstract. In the present work, we report the preparation of BCNO/SiO₂ phosphor synthesized using a microwave-assisted method. This method allows a lower temperature and a shorter reaction time than simple heating (furnace). The phosphors were prepared from precursors containing, boric acid, urea, citric acid and SiO₂ nanoparticles. To These precursors, silica nanoparticles were added at various concentrations from 0 to 5 %wt. The emission wavelength produced by the phosphor was varied by varying the fraction mass of the silica that were added to the precursors. The results showed that higher photoluminescence (PL) intensity was produced by the BCNO/SiO₂ with 3 %wt silica addition. The novelty of this research is using microwave heating for BCNO/SiO₂ synthesis, which is usually conducted using a simple heating method.

Keywords: BCNO SiO₂, microwave synthesis

1. Introduction

Boron carbon oxynitride (BCNO) is a phosphoric material without rare earth metal, composed of boron (B), carbon (C), nitrogen (NO) and oxygen (O). BCNO can be an alternative fluorescent material for lighting applications[1]. BCNO can be prepared by several techniques, such as simple heating (furnace) [2][3][4], hydrothermal synthesis [5], electrospinning [6] and microwave heating[7]. Microwave-assisted BCNO synthesis was reported by Iwasaki[8]. We have reported previously that BCNO/SiO₂ was successfully synthesized using a simple heating process [9].

In the present study, we investigated the utilization of microwave heating for synthesis of BCNO/SiO₂ nanoparticles, which has not been reported on previously. Using boric acid, urea and citric acid as raw materials, and the addition of fumed silica with microwave-assisted method allow a shorter reaction time and lower reaction temperature than electric-furnace heating.

2. Materials and synthesis

A schematic flow chart for the BCNO/SiO₂ synthesis via microwave heating method is shown in Fig.1. Compound of BCNO/SiO₂ was prepared using a microwave-assisted method. First, urea [(NH₂)₂CO with mw of 60.07g/mol], boric acid [H₃BO₃ with mw of 61.83 g/mol] and citric acid [C₆H₈O₇ with mw of 192.124 g/mol] were used as the nitrogen sources, boron sources and carbon sources, respectively. The additional material consisted of SiO₂ nanoparticles (particle size of 40 nm) (Wacker
Chemicals Fumed Silica Co. Ltd. China). Precursor solutions were prepared by dissolving the urea, boric acid, citric acid and nanoparticle silica in 15 ml distilled water as the solvent, followed by stirring in a magnetic stirrer to obtain homogeneous solutions. The precursors were then heated in a commercial microwave with a maximum oven output of 1000 Watt for 120s.

![Microwave-assisted synthesis of BCNO/SiO₂ phosphor.](image)

**Figure 1.** Microwave-assisted synthesis of BCNO/SiO₂ phosphor.

### 2.1 Measurement and Characterization
The photoluminescence (PL) spectrum of each sample was measured at room temperature using Cary Eclipse spectrofluorophotometer (Agilent, Australia). All PL analyses were performed with 365 nm excitation at room temperature.

### 3. Discussion
To investigate the effect of silica addition (0-5wt%) on the PL spectra, the microwave and the time were set constant at 120 second. Figure 2 shows the emission spectra of BCNO/SiO₂ and the PL spectra of the samples under excitation at 365 nm. The PL spectra of the sample with no silica addition has an emission peak at 488.029 nm with a PL intensity of 119.175 a.u., which is a greenish blue emission. The PL spectra of the sample with 3wt% silica addition has an emission peak at 461.059 nm with a PL intensity of 381.671 a.u, which is a blue emission. The PL spectra of the sample with 5 wt% silica addition has an emission peak at 486.059 nm with a PL intensity of 194.587 a.u, which is a greenish blue emission.

![PL Spectra of BCNO/SiO₂ fabricated with microwave assisted method.](image)

**Figure 2.** PL Spectra of BCNO/SiO₂ fabricated with microwave assisted method.
The PL spectra variation depends on the silica addition. The sample with 3wt% silica addition shows the highest intensity. The intensity is 3-fold larger than that of samples without SiO$_2$ nanoparticles. The PL spectra of the sample that is synthesized using the microwave method with 3 wt% silica has a higher intensity than the PL spectra of the sample with 0 wt% and 5 wt% silica. This phenomenon is consistent with our previous observations [8]. In our previous research, the PL spectra of the sample with 3 wt% silica that was synthesized using a simple heating method also has a higher intensity than thePL spectra of the sample with 0 wt% and 5 wt% silica. In our previous research the carbon source was PEG MW 20000, while in this report the carbon source is citric acid. The thermal properties of SiO$_2$ are suspected to be an important factor affecting the luminescence spectra and the uniformity of BCNO/SiO$_2$. In a crystallized nanocomposite, which comprised 3wt% SiO$_2$, more homogeneous photoluminescence is produced and more distributed heat happen. These phenomena are caused by SiO$_2$ which spreads to the entire of sample body and related with the nitrogen composition. On the other hand, while the fraction of SiO$_2$ was increased to 5wt%, the agglomeration of SiO$_2$ displacement happens. As the consequence, the luminescent centers were distributed with not homogeneous, the nitrogen composition in the powder decreased and finally, in order to obtain the best luminescence properties, we believe that 3wt% is the optimum SiO$_2$ fraction for a BCNO/SiO$_2$ nanocomposite.

4. Conclusions
In this study, we have successfully synthesized BCNO/SiO$_2$ from boric acid, urea, citric acid and SiO$_2$ nanoparticles using a microwave-assisted method. It is found that the emission wavelength changes depending on silica addition. Samples with 3wt% SiO$_2$ shows the highest PL intensity, as previously reported for samples with 3wt% synthesized using a simple heating method. Shorter heating period and lower reaction temperature makes possible using microwave-assisted method, it could lead to efficient energy use.

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