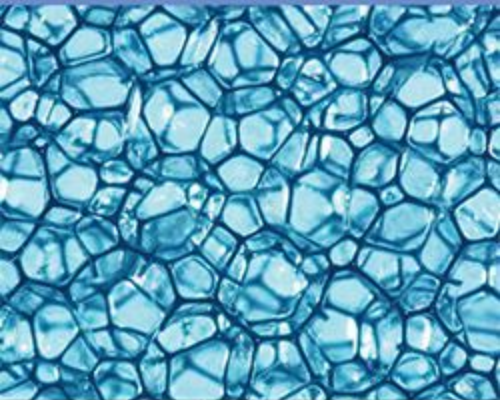


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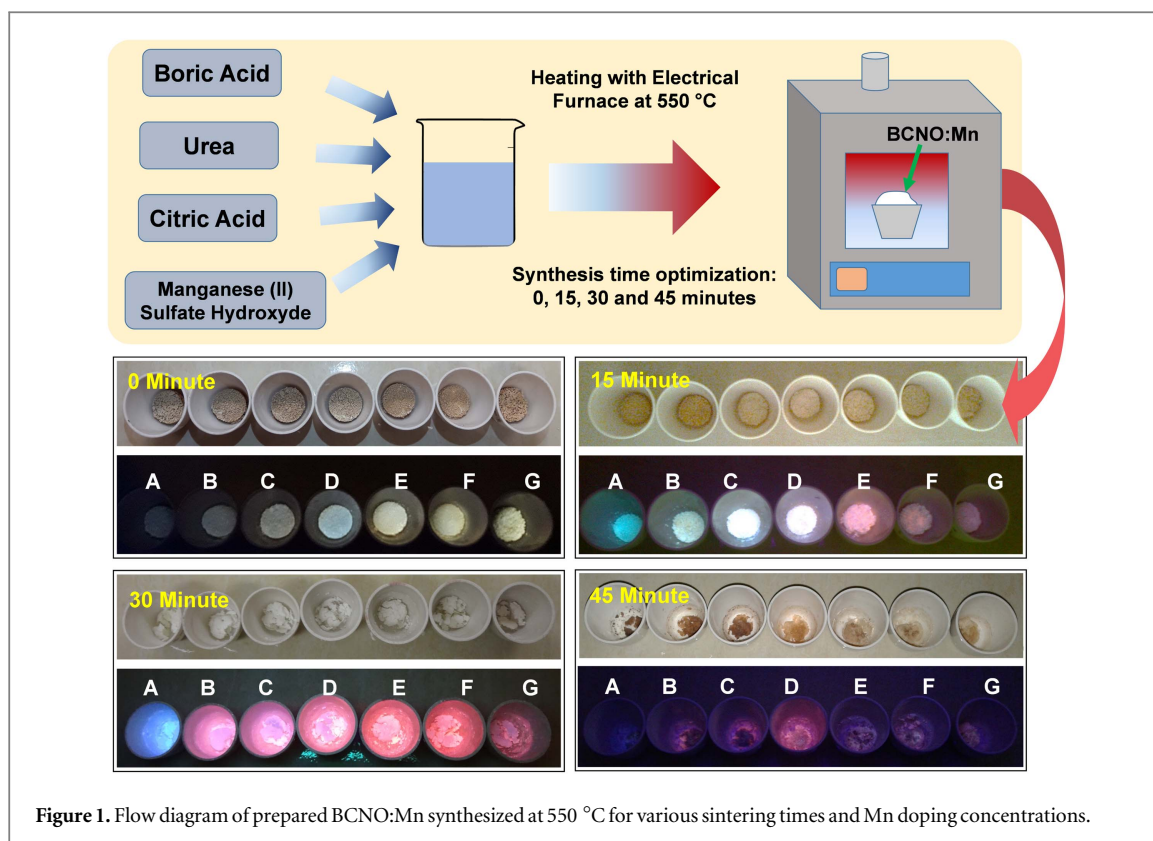
Keywords: BCNO:Mn phosphor materials, red photoluminescence, concentration quenching effect, sintering time optimization

Abstract

The effect of sintering time to the transient nature and optimization of red photoluminescence manganese-doped boron carbon oxynitride (BCNO:Mn) phosphor was investigated. The BCNO:Mn samples were synthesized using a facile urea-assisted combustion route involving boric acid, citric acid, manganese salt and urea. The optimized intensity of the dual peak emission at 420 nm (blue emission) and 630 nm (red emission) in the photoluminescence (PL) spectrum could be achieved by controlling the sintering time of the BCNO:Mn. The BCNO:Mn samples in high-crystalline form was found to be in a cubic and hexagonal structure. Based on the PL analysis, it is suggested that the BCNO:Mn symmetric band at 630 nm can be attributed to the ${}^4T_1({}^4G) \rightarrow {}^6A_1({}^6S)$ transition absorption of Mn^{2+} ions into the hexagonal structure. Microstructure analysis showed an irregular and agglomerated shape of the BCNO:Mn sample.

1. Introduction

Boron carbon oxynitride (BCNO) materials are nitride semiconductors with an energy gap of >3 eV and have been widely developed due to their potential applications in lighting, bio-imaging, optoelectronic and luminescent devices [1–4]. Firstly, Ogi *et al* successfully developed a new type of boron nitride based phosphor, by preparing nitrogen and oxygen co-doped boron nitride (BCNO) phosphor through a urea combustion method [1]. BCNO phosphor has an emission spectrum in the range of visible light with higher quantum value results under UV light or blue LED exposure [1–4]. Until now, a great deal of research has been done that was focused on the control of the emission spectrum and quantum yield value of BCNO materials. For example, Wang *et al* [5] succeeded in optimizing the emission properties of BCNO materials from blue (387 nm) to dark yellow (571 nm) by changing the molar ratio of the carbon (C/B) or nitrogen (N/B) source to boron sources. Meanwhile, Kaihatsu *et al* [6] and Nuryadin *et al* [7] have examined the effects of different carbon sources, such as poly(ethyleneimine) and poly(ethylene glycol) with various molecular weights (MW) or citric acid, on the emission spectrum optimization of BCNO materials. In addition, Nuryadin *et al* [8], and Zhang *et al* [9] have induced red emission of BCNO materials with emission peaks at 581 nm to 620 nm using chemical and physical optimization. However, the induced red emission of BCNO still had low PL properties and quantum yields. Very recently, our group has synthesized a bright red emitting boron carbon oxynitride by doping with manganese (BCNO:Mn) with the emission peak at 600–650 nm [10]. The crystal structure, chemical composition, morphology and Mn^{2+} ion concentration influenced the photoluminescence properties of the BCNO. These parameters strongly depend on the preparation method and the experimental conditions, especially sintering time. Therefore, further research on the effect of sintering time on PL properties of BCNO:Mn is crucial in order to optimize PL performance and to understand the transient nature of the BCNO:Mn.



In this paper, we report the effect of sintering time to the transient nature and optimization of red photoluminescence manganese-doped boron carbon oxynitride (BCNO:Mn) phosphor synthesized by urea-assisted combustion route. By controlling the sintering time and doping concentration, optimization of the red emitting BCNO:Mn could be achieved. In addition, the crystal structure, photoluminescence (PL) spectra, decay curve, morphology and chemical composition of the synthesized BCNO:Mn have been investigated systematically. We believe that the optimization and understanding of BCNO:Mn formation can provide meaningful information for its application in optoelectronics, semiconductor lighting or panel displays.

2. Methods

2.1. Materials and synthesis

The urea-assisted combustion method is an important technique for the synthesis and processing of ceramics, metal oxides, catalysts, nanomaterial and composites. A flow diagram of the BCNO:Mn synthesis with different Mn^{2+} doping concentrations using the urea-assisted combustion method is shown in figure 1. The BCNO:Mn samples were synthesized using boric acid (H_3BO_3), urea ($(NH_2)_2CO$), citric acid ($C_6H_8O_7$) and manganese (II) sulfate monohydrate ($MnSO_4 \cdot H_2O$) as boron, nitrogen, carbon and manganese sources, respectively. The mass ratios of the boron, nitrogen and carbon sources were maintained constant at 5, 20 and 0.1, respectively. Furthermore, the manganese atom concentration was tuned by varying the Mn/B molar ratio from 0 to 2.84% mol/mol (table 1). Precursors were prepared by dissolving all raw materials in hot water under high speed stirring until becoming clear solutions. The prepared precursors were heated in an electrical furnace at 550 °C under variation of sintering time from 0, 15, 30 and 45 min. All samples were cooled naturally and powdered using mortar grinding for further analysis.

2.2. Materials characterization

The structures were characterized by x-ray diffraction (XRD, Philips Analytical PW 1710 BASED, PANalytical, the Netherlands) using $Cu-K\alpha$ as the x-ray source ($\lambda = 1.5 \text{ \AA}$). The particle morphology was observed by scanning electron microscopy (SEM; JCM-6000 NeoScope Benchtop, JEOL, Japan). The room-temperature photoluminescence (PL) spectra, excitation (PLE) spectra and luminescence decay curves (lifetime) of all BCNO:Mn phosphor samples were characterized using a Cary Eclipse spectrofluorophotometer (Agilent Ltd, Australia). The Fourier transform infrared (FT-infrared, Bruker Optics, Ettlingen, Germany) spectra with an Alpha Platinum ATR sampling module were used to determine the chemical bonding of the prepared samples.

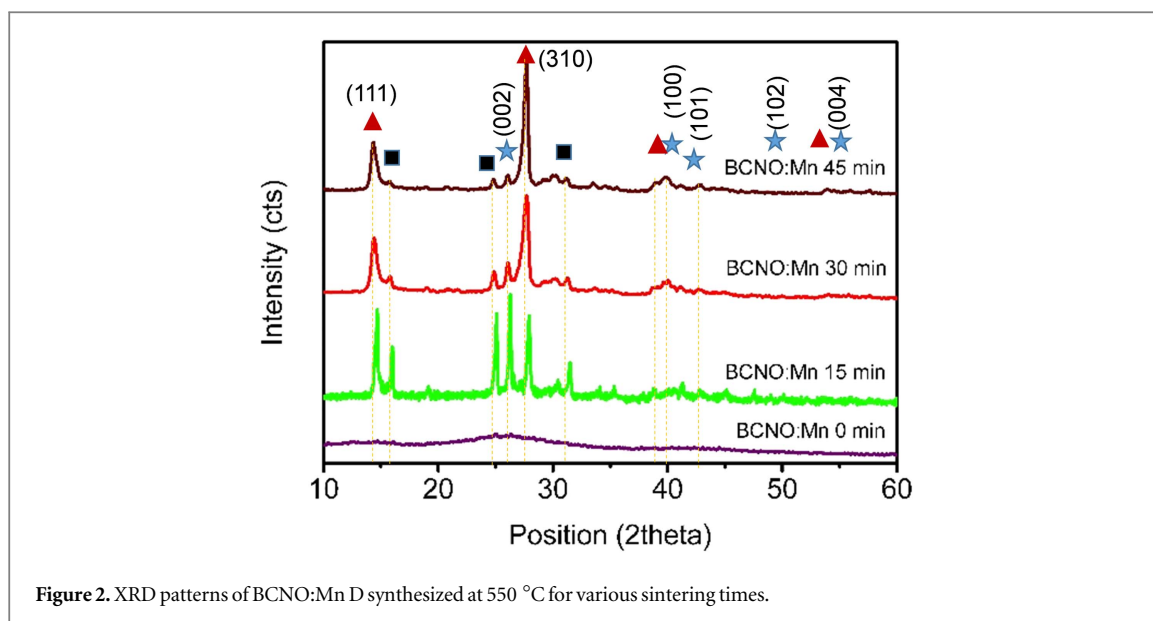


Figure 2. XRD patterns of BCNO:Mn D synthesized at 550 °C for various sintering times.

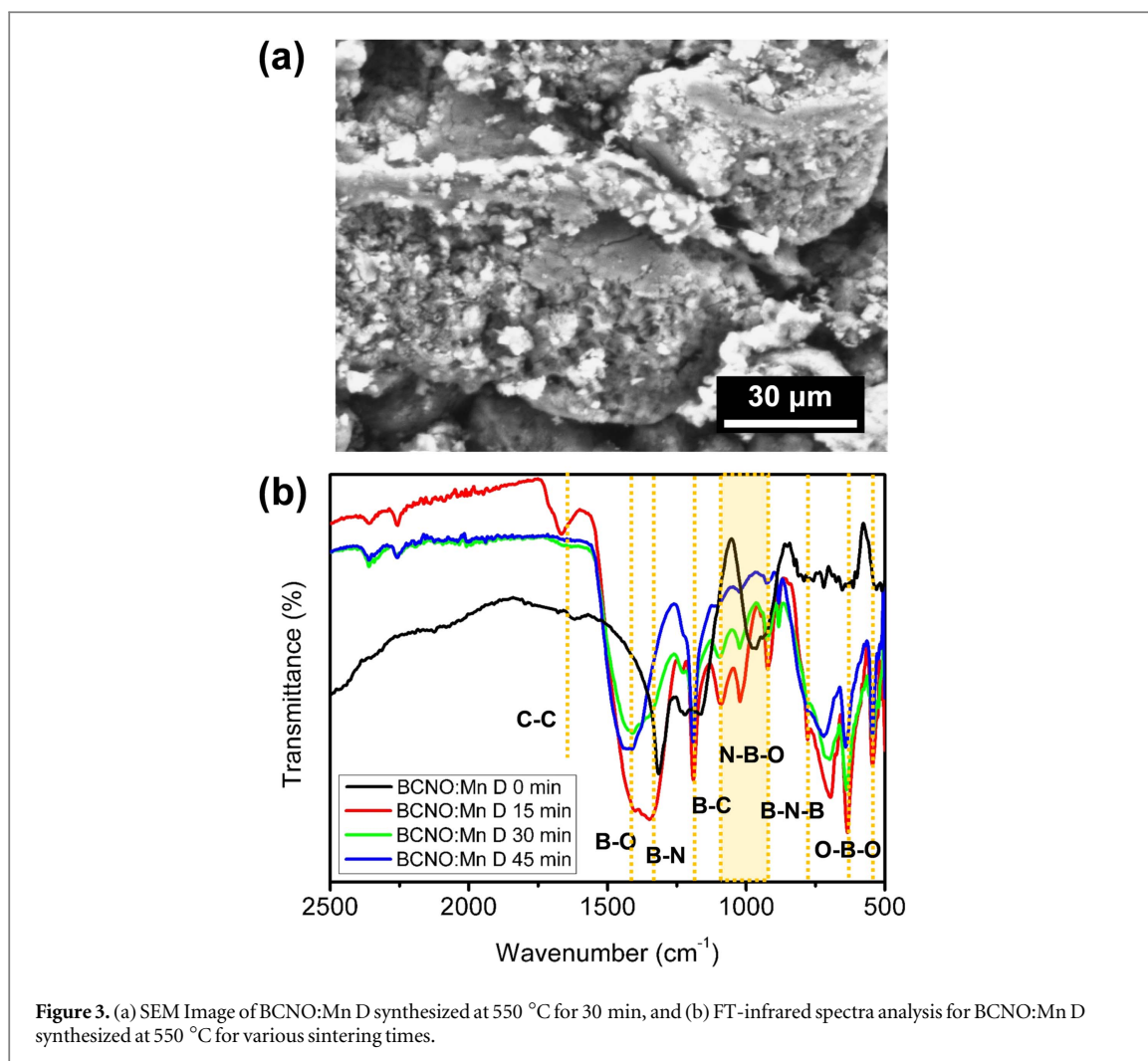
Table 1. The chemical composition of precursor for BCNO:Mn phosphor.

Sample	H ₃ BO ₃ (g)	Urea (g)	Citric Acid (g)	Mn/B (%) mol/mol)
A				0%
B				0.18%
C				0.36%
D	0.5	2	0.01	0.71%
E				1.42%
F				2.13%
G				2.84%

3. Result and discussions

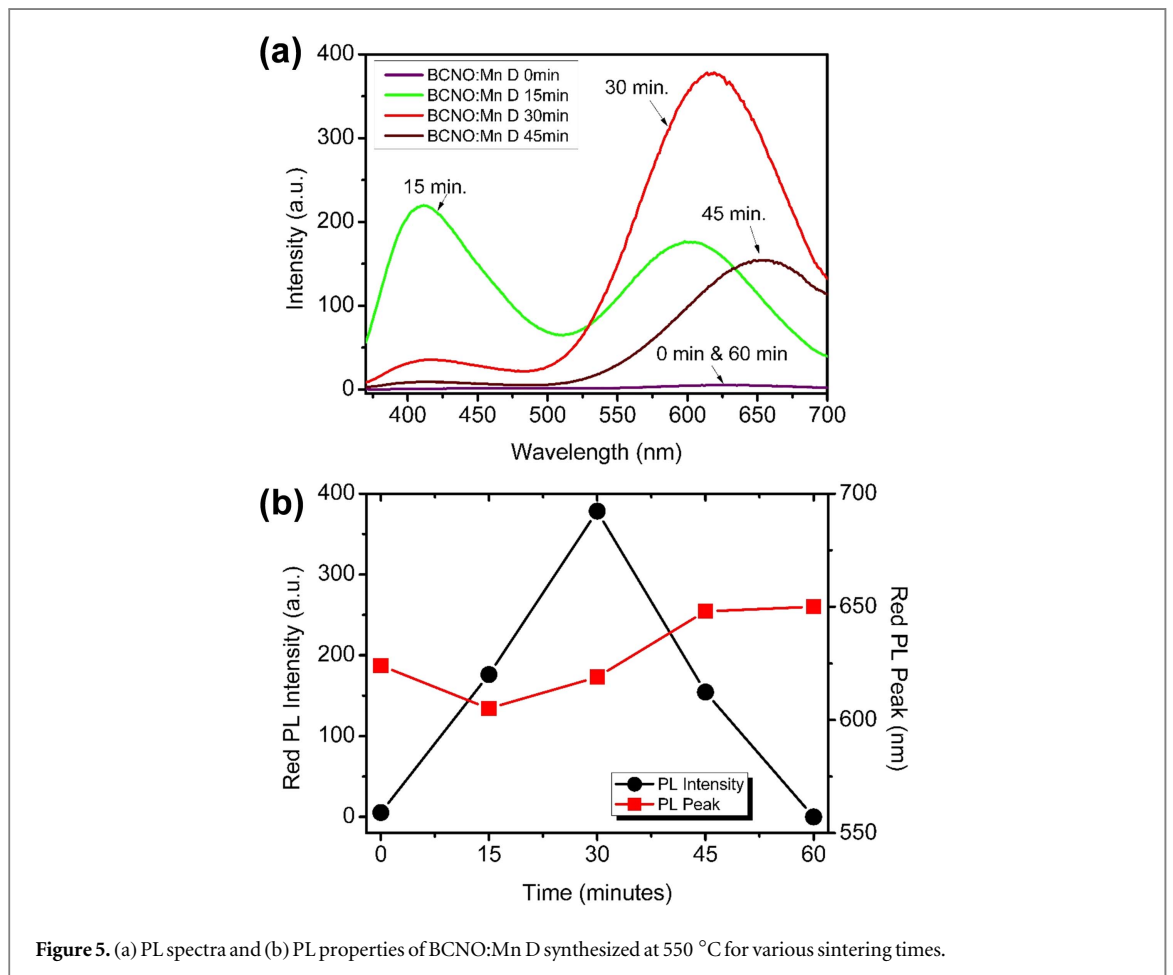
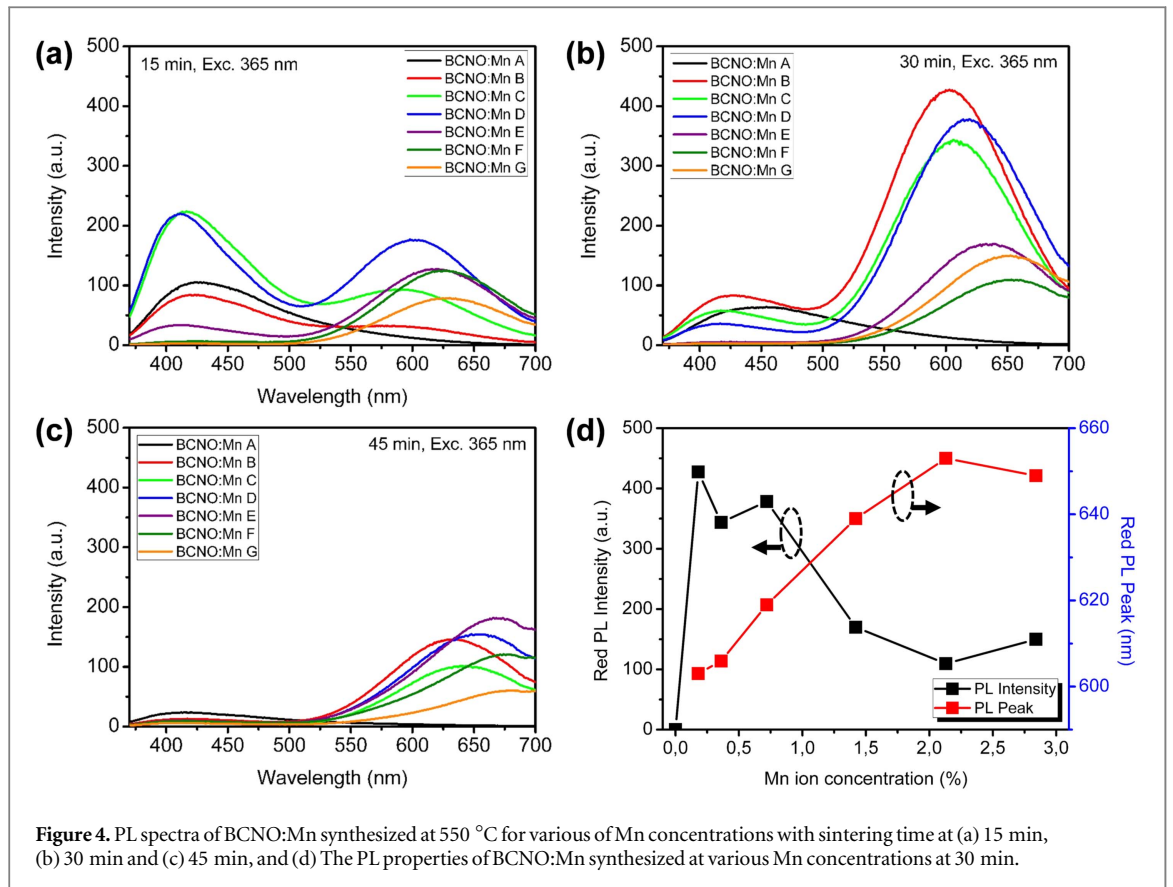
To determine the effect of sintering time on the crystal structure and phase purity, we characterized the BCNO:Mn D by x-ray diffractometry (XRD). Figure 2 shows the XRD patterns of the BCNO:Mn synthesized at 550 °C for various sintering times. The XRD patterns of the BCNO:Mn indicates the existence of pristine *h*-BN (blue star), boron oxide (red triangle) and graphitic carbon (black square). The formation of pristine *h*-BN (JCPDS no. 74-1978) [11], indicated by two board peaks at 26.27° and 41.27°, yielded optimal intensity at 15 min, after which the intensity decreased with the increase of sintering time. The graphitic carbon peaks (JCPDS no. 75-1681) [12, 13] at 25.04° and 31.48° appear as imperfect decomposition of urea and become lower with the increase of sintering time. Besides that, the distinct and sharp peaks at 14.64°, 27.91° and 41.27° correspond to the crystal structure of *c*-B₂O₃ (JCPDS no. 06-0297) [14]. Observation showed a change of the structure of *h*-BN into *c*-B₂O₃ caused by the oxidation process in crystalline *h*-BN along with the increase in sintering time of the BCNO. Some researchers have shown that carbon and oxygen atoms can behave as impurity atoms in the crystal lattice of the BCNO. Thus, an increased sintering time causes chemical decomposition and the crystal structure becomes more perfect.

The morphology and the chemical bonding of the BCNO:Mn were measured using SEM and FTIR characterization. Figure 3(a) shows an SEM image of the prepared BCNO:Mn D synthesized at 550 °C. The image shows randomly agglomerated particles and irregular shapes with an average particle size of $\pm 5 \mu\text{m}$. This particle size is typical for phosphor powders used in lighting applications [7, 15, 16]. Figure 3(b) shows the typical FT-infrared spectra of BCNO:Mn material synthesized at 550 °C for various of sintering times in the range of 500–2500 cm⁻¹. The B–N stretching mode at $\sim 1400 \text{ cm}^{-1}$, the B–C vibration peak at 1196 cm⁻¹, and the B–N–B vibration peak at $\sim 790 \text{ cm}^{-1}$ was observed in all of the BCNO:Mn samples [17, 18]. The absorption peaks at 560 cm⁻¹, 650 cm⁻¹, and 1500 cm⁻¹ correspond to B–O vibration bonds [19, 20]. The N–B–O absorption band is evident from $\sim 900 \text{ cm}^{-1}$ to 1100 cm⁻¹ [9, 21]. In addition, the intensity of the N–B–O bonds increased with increasing sintering time, and the B–O absorption peak became clearer and its intensity increased caused by the decomposition and/or oxidation process as well as changes in the crystal structure of the BCNO:



Mn. From the FT-infrared spectra, it can be concluded that the sintering time was involved in the chemical bonding and composition of the BCNO:Mn samples.

The photoluminescence (PL) spectra of the BCNO:Mn synthesized at 550 °C with various of sintering times and Mn²⁺ doping concentrations under UV light excitation at 365 nm, are shown in the figure 4. The spectra show that the PL properties of the BCNO:Mn exhibiting blue (~425 nm) and red emission (~630 nm) varied with different sintering times and Mn²⁺ concentrations. Previous research shows that blue emission of BCNO phosphor produced by the crystal structure of *h*-BN doped by oxygen and carbon [5–7]. Meanwhile, red emission was significantly appearing after BCNO material doped with Mn²⁺ ions. Nuryadin *et al* (2017) have shown that the red emission in BCNO:Mn²⁺ phosphor appears due to the associated Mn²⁺ ions in the *h*-BN hexagonal structure, and forms a new energy level in the BCNO electronic structure [10]. The BCNO:Mn synthesized for 0 min had a very low emission intensity. The emission intensity of the BCNO:Mn synthesized for 15 min showed an increase in comparison with the sample synthesized for 0 min, figure 4(a). Meanwhile, the BCNO:Mn synthesized for 30 min showed the highest red emission intensity (λ_{red} peak at 630 nm) compared with the prepared samples synthesized for 15 min and 45 min, figures 4(b)–(c). The decrease or increases in emission intensity can be ascribed to the crystal structure, chemical decomposition and doping quenching of the BCNO:Mn [7, 10, 22]. Observation showed that the PL intensity increased due to the increase in Mn²⁺ concentration that functions as a luminescence center of the red emission of the BCNO, as shown in figure 4(d). When the Mn²⁺ concentration increased, it caused the distance between the Mn²⁺ ions to become shorter and the energy transfer rates to get smaller. This facilitates the energy transfer between Mn²⁺ ions so that the energy transfer rate between the Mn²⁺ ions is equal to the emission of the Mn²⁺ ions and the emission intensity reaches its maximum value (radiative decays). At very high Mn²⁺ concentrations, the transfer energy between the Mn²⁺ ions is very tight and the energy transfer between the Mn²⁺ ions exceeds the emission levels. In other words, the Mn²⁺ ions will not be able to emit light, even though energy transfer occurs. Therefore, the excitation energy is absorbed by the crystal lattice transfer energy (non-radiative decays) and causes the emission intensity to decrease, known as the concentration quenching effect [10, 23, 24].



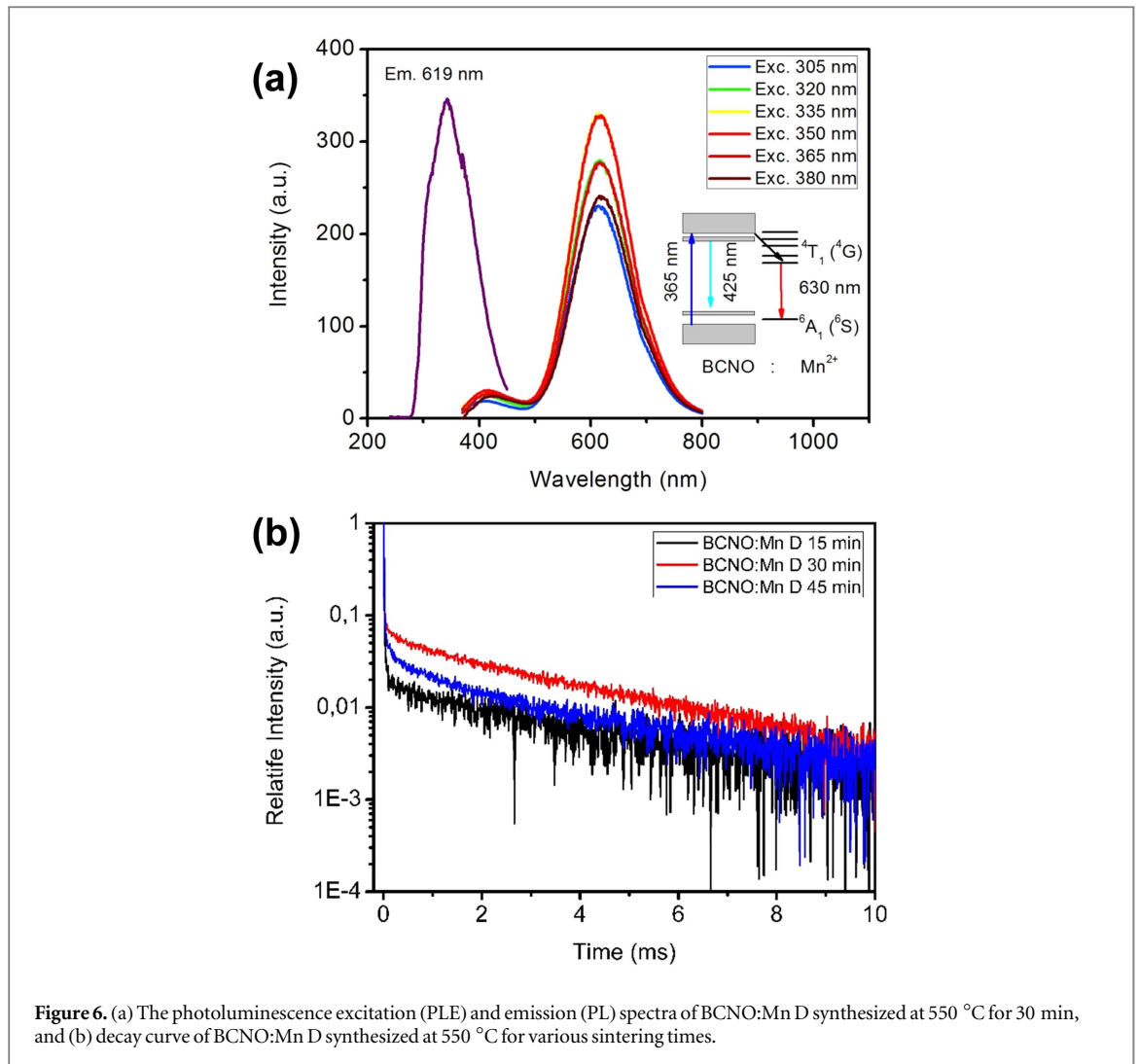


Figure 6. (a) The photoluminescence excitation (PLE) and emission (PL) spectra of BCNO:Mn D synthesized at 550 °C for 30 min, and (b) decay curve of BCNO:Mn D synthesized at 550 °C for various sintering times.

In addition, the red emission optimization of BCNO:Mn material remains consistent with the effect of sintering time observation on changes in the crystal structure and chemical composition due to decomposition and oxidation processes. For the synthesis time at 15 min, the BCNO crystals have been formed with predominant *h*-BN crystals so that the Mn^{2+} ions are associated with the hexagonal structure and produce the red emission energy level. However, due to the presence of undecomposed carbon impurities that produces non-radiative energy levels, so the red luminance intensity is low. For the time synthesis at 30 min, the presence of carbon impurities was reduced by thermal decomposition so that the intensity of the red emission was increased significantly. Meanwhile, for the time of synthesis of 45 min and 60 min, the process of decomposition and oxidation dominant place in the structure of *h*-BN, being B_2O_3 , so that the Mn^{2+} ion is not in a hexagonal structure and cause the intensity of red emission decreased (even disappear) from BCNO: Mn^{2+} , as shown at figure 5.

Figure 6(a) shows the photoluminescence excitation (PLE) and emission (PL) spectra of BCNO:Mn D synthesized at 550 °C for 30 min. The excitation spectra monitored at 630 nm show that the BCNO:Mn had an excitation wavelength in the near-UV light region, 300 nm to 420 nm. Excitation peaks of the BCNO:Mn D samples were observed at 350 nm and 370 nm. It is suggested that the peaks of 350 nm and 370 nm can be attributed to the electronic band transition (excitation) of the *h*-BN structure doped by oxygen, carbon, and crystal surface defects [7, 23, 25]. Thus, this red photoluminescence of BCNO:Mn phosphors can be well excited by near-UV light and blue LED. The decay curve profile related to the BCNO:Mn for various sintering times was estimated by monitoring the excitation peaks at 365 nm and the emission at 630 nm in accordance with the ${}^4T_1({}^4G) \rightarrow {}^6A_1({}^6S)$ triplet emission band of the most intense of the Mn^{2+} ions, as can clearly be seen in figure 6(b). The decay curve measurements of the ${}^4T_1({}^4G) \rightarrow {}^6A_1({}^6S)$ triplet transition fit with a double exponential profile for all prepared samples. Therefore, the lifetime of the 4T_1 state can be obtained by fitting the experimental decay curve with the double exponential function given below,

$$I_t = I_{01}e^{-\frac{t}{\tau_1}} + I_{02}e^{-\frac{t}{\tau_2}}$$

where, I_t , I_{01} and I_{02} are the emission intensity at t and $t = 0$ respectively, τ is the lifetime of the excited state [26, 27]. The lifetime values of the 4T_1 level of the BCNO:Mn were obtained by calculating the first exponential constant of the decay curves. The experimental lifetime (τ) values of the 4T_1 level were found to be 2.8 ms, 4.1 ms and 3.6 ms for the BCNO:Mn synthesized at 15 min, 30 min, and 45 min, respectively. The discrepancy in the experimental and calculated lifetime values is caused by non-radiative decays like multi-phonon relaxation and quenching effects caused by the Mn^{2+} ions.

4. Conclusions

In summary, the effect of sintering time of red emission manganese-doped boron carbon oxynitride (BCNO: Mn) phosphor on its PL properties was reported. The BCNO:Mn in high-crystalline form was found to be in a cubic and hexagonal structure. Microstructure analysis showed an irregular and agglomerated shape of the BCNO:Mn sample. The FTIR spectra showed that sintering time affects N-B-O, B-N, and B-O chemical bonding of BCNO:Mn. The optimum intensity of dual-peak emission at 420 nm and 630 nm in the photoluminescence (PL) spectrum could be optimized by controlling the sintering time of the BCNO:Mn. It is suggested that the BCNO:Mn symmetric band at 630 nm can be attributed to the ${}^4T_1({}^4G) \rightarrow {}^6A_1({}^6S)$ triplet transition absorption of the Mn^{2+} ions into the hexagonal structure.

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