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Optical characteristics of nano-forsterite powders with natural silica sand as source of Si

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ABSTRACT

Nano forsterite powders have been successfully synthesized via ultrasonic assistant for the starting materials and followed by solid reaction and calcination steps. The synthesis products were greenish powders with forsterite as the dominating phase as revealed by XRD data analysis. The formation of forsterite was also confirmed by FTIR spectrometry and supported by SEM images. The phase composition after calcination at 950 °C for 4 h was 93.9 wt% forsterite and 6.1 wt% periclase. The forsterite crystal was nanometric according to the XRD data, while the density of the powder was 2.88 g/cm^3. UV–Vis spectrum of the powder showed relatively strong absorption peaks particularly at 635, 660, 670, 680, 695 and 705 nm which can be associated with the greenish color of the product and is different from previous studies. Therefore, it can be concluded that high purity greenish forsterite can be formed at 950 °C calcination temperature as previously reported, except that the raw materials were subjected to ultrasonic treatment.

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1. Introduction

Forsterite with chemical formula Mg_2SiO_4 is recognized a mineral phase of crystalline magnesium silicate, named after the German scientist Johann Forster. Forsterite is predominately in a molar ratio 1:2 consists of the anion SiO_4^{4-} and the cation Mg^{2+}. It also exhibits excellent insulation properties and good chemical stability even though at high temperatures. Forsterite is insulating materials with very low dielectric constant ($\varepsilon_r' \approx 10.5$) [1] in our prior study. All of these characteristics, make it relevant material for engineers to use them, in the fabrication of numerous technical components such as dielectric substrates [2–4], biomaterials [5,6], composite materials [6,7] and optical devices [8–11]. Here, we continue to produce forsterite powders on a nanometric scale to keep are eminent as the vital materials in nanotechnology at low-temperature calcinating.

In previous studies, several methods have been used to develop high purity nanometric scale forsterite phases such as wet method [12,13] and mechanical activation [14,15]. The phase evolution of pure forsterite is formed at relatively low combustion temperatures (<1000 °C). It was also reported that all of the nanoforsterite powder from combustion is white. However, there is a difference in the color of nano-forsterite powder in this study when given ultrasonic treatment. So the aim of this study is how to explain the greenish nano-forsterite products in terms of optical properties.

2. Method/experiment

The route to synthesize the nano forsterite scale begins with an ultrasonic process of both MgO (Merck) and amorphous silica powder for 30 min so that the starting material particles do not cluster. The next process is the same as when using a solid reaction method with a mechanical activation treatment. All raw materials plus 3% PVA are milled at 150 rpm for 1 h, then the mixed powders were gradually heated up to 950 °C in the furnace for 4 h holding time.

The products were characterized by Fourier-transform infrared spectroscopy (FTIR) to elucidate group bonds, X-ray diffraction (XRD) with Cu Ka radiation to identify phases, scanning electron microscopy (SEM) with energy–dispersive X-ray fluorescence (EDX) to reveal the microstructure and UV–Visible to explain optical properties.

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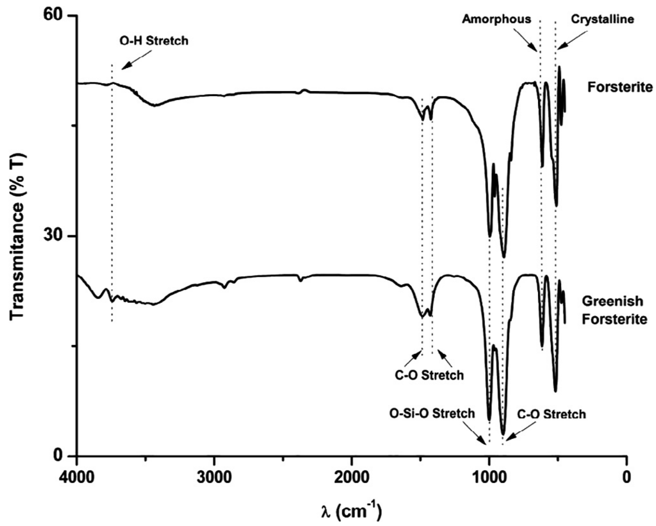

Fig. 1. FTIR patterns of the greenish forsterite and forsterite powder at room temperature.

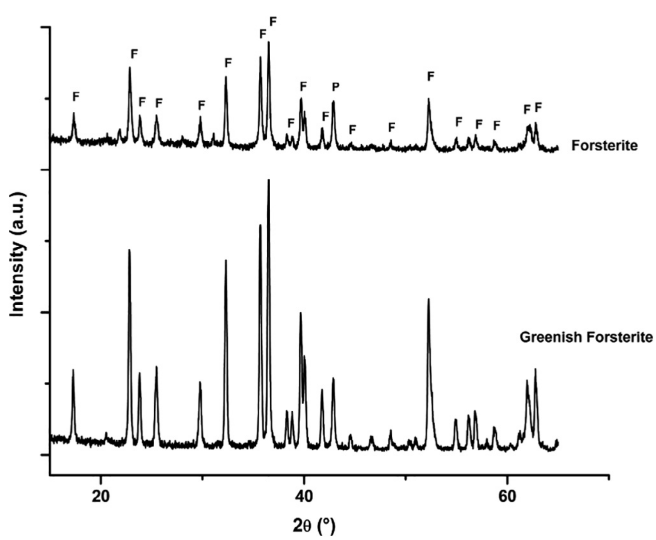

Fig. 2. X-Ray Diffraction Patterns (CuK_α radiation) of forsterite and greenish forsterit.

3. Result and discussion

Fig. 1 illustrates the FTIR spectrum of forsterite powder fired at 1150 °C and nano-greenish forsterite powder fired at 950 °C by the Perkin Elmer Frontier FTIR spectroscopy. Two spectrums are used to follow the formation of crystalline and amorphous forsterites, Si O Si bonds and OH bands. FTIR analysis shows that the intense bands of forsterite concerned to the characteristic of peaks that appeared in the range 830–880 cm^{-1} and 1010–1030 cm^{-1}, identical results were also reported by Mathur [17]. A few bands in the range of 500–505 cm^{-1} were associated with MgO stretching mode. Some characteristics bands are also traced related to the SiO_4 stretching mode (in the range of 980–1000 cm^{-1} and 860–890 cm^{-1}) and SiO_4 bending modes (600–615 cm^{-1}). These peaks justify the formation of forsterite as it is shown in XRD pattern of the samples. Thus, the FTIR test is able to explain the chemical bonds or molecular structure formed in the samples.

XRD patterns of the samples showed in Fig. 2 that collected by X'Pert3 Powder Panalytical. For both greenish forsterite and forsterite, the primary identified phase was forsterite (Mg_2SiO_4, PDF# 34-0189) and the secondary phase was periclase (MgO, PDF# 45-0946). Another secondary phase, namely protoenstatite (MgSiO3, PDF# 74-816) and low-cristobalite (SiO2, PDF# 82-1232) were present only for forsterite [1]. Thus, the composition phases of greenish forsterite are simpler than forsterite. Moreover, the mean crystallite size was predicted from the peak broadening in an XRD diffraction pattern [16]. The crystallite size of greenish forsterite is nanometric, which indicated from the peak width of the diffraction pattern that relatively broad, ca > 0.2° 2θ. The SEM image of the associated powder (Fig. 3a) also revealed the presence of numerous grains, which indicates that the greenish forsterite sample contains nanopowders.

A quantitative analysis of the greenish forsterite showed that forsterite was dominant, i.e. 93.9 wt%, while the secondary phases periclase was found to be of 6.1 wt%. Furthermore, the lattice parameter values of greenish forsterite in our powder were a = 4.749 Å, b = 10.188 Å, and c = 5.975 Å (by the Rietveld-based Rietica software), which are in good agreement with the prior study [1]. The largest difference is on the color of product, which conforms to the optical properties as shown in Fig. 4.

Typical SEM images are shown in Fig. 3 a representative high-magnification image reveals that the surface morphologies of the greenish-forsterite powder relatively as a uniform in shape and size. The shape is orthorhombic and the grain size is about several hundred nanometers (Fig. 3a). Furthermore, the crystallite size calculated by using software entitled as 'Image J 1.47 T' from SEM micrograph, the mean particle size is about 128(6) nm. Meanwhile,

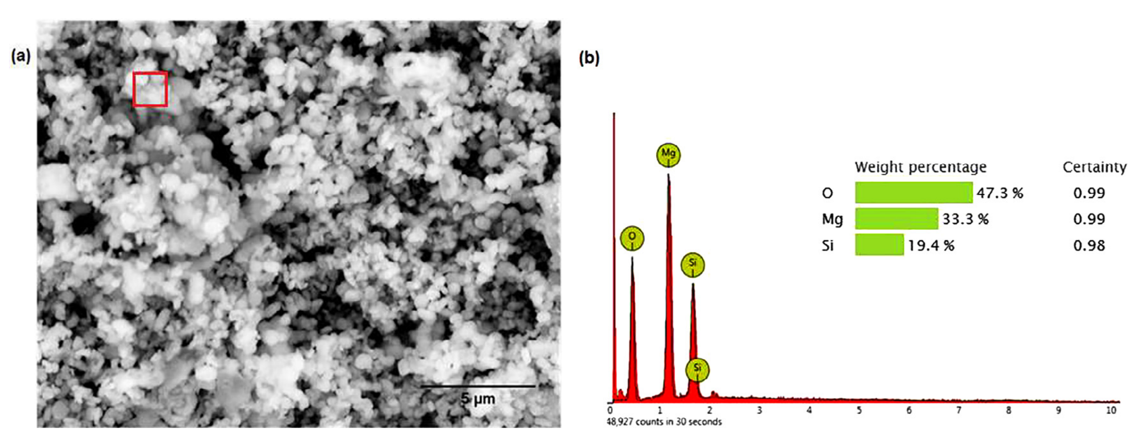

Fig. 3. (a) SEM image with spot and; (b) EDX on greenish forsterite.

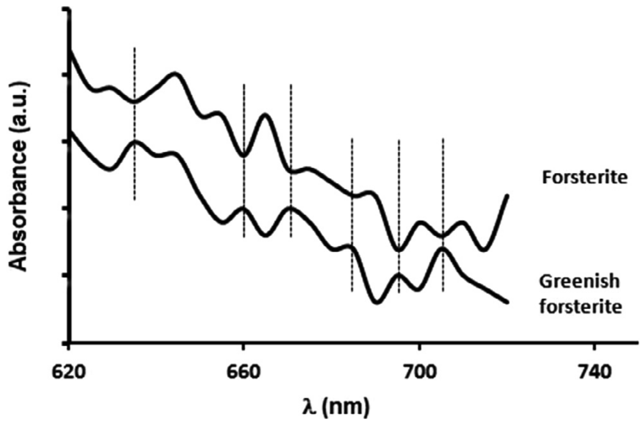

Fig. 4. UV–Vis patterns of samples for visible spectra at 620–720 wavelengths.

EDX analysis (Fig. 3b) reveals that the ratio of weight percentage of Mg/Si is 1.7(1) that similar to the previous result [1]. Generally, it can be seen that the characteristic morphology of greenish forsterite powders shows crystallite with orthorhombic structures, this is in accordance with the values of the lattice parameters in the crystallographic analysis.

Fig. 4 shows the Uv–Vis spectrum of the samples by Genesys 10 s UV–Vis Spectrophotometer. There are 6 peaks of greenish forsterite at wavelength of 635, 660, 670, 680, 695 and 705 nm, meanwhile at the same wavelength in the forsterite spectrum occur the opposite peaks. This means that forsterite nano-greenish cannot emit these six wavelengths at room temperature, or it can be interpreted that the color spectrum at these wavelengths is absorbed by the nano-greenish forsterite. So that ultrasonic treatment for the starting material in the synthesis process produces forsterite powder with a greenish color.

4. Conclusion

In summary, nano-greenish forsterite Mg_2SiO_4 powders with uniform size have been synthesized via ultrasonic assistant for starting material before mixing through solid state method. The UV–Visible properties of the greenish forsterite were observed, the absorption peak between 620 and 720 nm is indicated to understand that way that the color of product is different with previous studies, although it has a phase with an equivalent high level of purity. Some wavelengths (635, 660, 670, 680, 695 and 705 nm) are detected as the absorption peak in the sample which means that the wavelength is not reflected so that the forsterite is not white but is slightly greenish. The synthesis route of nano-scale forsterite assistant by an ultrasonic process will offer great opportunity to produce the nano-greenish forsterite.

CRediT authorship contribution statement

U. Nurbaiti: . : Conceptualization, Methodology. **W.S.W. Prayitno:** Data curation, Visualization. **Khumaedi:** Investigation, Writing - original draft. **S. Pratapa:** Supervision, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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