

## Microwave synthesis and characterization of non-stoichiometric $Mg_{0.1551}Fe_{1.8966}O_3$

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### ABSTRACT

**Purpose:** of this study is clearly different synthesis method (microwave method) of non-stoichiometric magnesium iron oxide using in broad area such as high frequency magnets, data storage systems and especially cancer treatment.

**Design/methodology/approach:** Analytically magnesium oxide and iron (III) oxide weighed an appropriate molar ratio and homogenized in an agate mortar. The mixture placed into a porcelain crucible to heat in microwave oven. After the material was exposed to microwave irradiation, it was taken from the oven, and then homogenized again. Then, final product was ready to analyse. Powder X-ray Diffractometer (XRD) was used to determine crystal structure of the product. FTIR spectrum was taken to support the functional groups. Thermo gravimetric-differential thermal analysis (TG/DTA) was carried out to identify thermal character. Morphological properties and semi-quantitative analyse of the sample was performed by Scanning electron microscope/ Electron dispersive (SEM/EDX).

**Findings:** The XRD patterns show that the product is  $Mg_{0.1551}Fe_{1.8966}O_3$  (ICSD 51171). The first time synthesized material is crystallized in hexagonal system with unit cell parameters  $a=5.0490$ ,  $b=5.0490$ ,  $c=13.7890$  Å and space group R-3c. The other supporting methods confirm the crystal structure. Non-stoichiometric  $Mg_{0.1551}Fe_{1.8966}O_3$  (ICSD 51171) was synthesized by microwave method completely different from literature for the first time. The characterization was mainly based on powder X-ray diffraction pattern. Also, thermal behaviour and morphology were determined.

**Research limitations/implications:** The synthesis method has some disadvantages such as low repeatability, non-uniform product etc. We tried to minimize these negative aspects in our research and succeeded.

**Practical implications:** The compound can be used to reduce side-effects of drugs for cancer treatment, and find broad usage area in that treatment. For this reason, magnesium iron oxide has practical applications.

**Originality/value:** The originality of the paper is first time microwave synthesis of  $Mg_{0.1551}Fe_{1.8966}O_3$ . In the literature, the magnesium iron oxide was synthesized so many times by different conventional routes. But, as far as we know cheap, clean and short-term synthesis route has not been used before.

**Keywords:** Powder metallurgy; Microwave synthesis; Magnesium iron oxide

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### MATERIALS

## 1. Introduction

Chemotherapeutic drugs, not being wide specificity, are major problem in cancer therapy for tumour cells. The critical side-effects of large doses of drug need to be injected for achieving efficient concentration in the tumour. For this reason, targeted drug delivery is tried to reduce strategies for treatment of tumour using molecular and magnetic systems [1]. Iron oxides have been used with high oxidation state to change the effects of drugs. But in some cases, high oxidation state can be unfavourable. The reduction of stages, minerals having less oxidative stages should be chosen [2]. Magnesium (Mg) is the most abundant intracellular divalent cation and the fourth most abundant mineral in human body. Mg plays major roles in many physiological and psychological functions [3-5]. In this study, our goal is clearly different synthesis method (microwave method) of non-stoichiometric magnesium iron oxide using in broad area such as high frequency magnets, data storage systems and especially cancer treatment.

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## 2. Materials and method

### 2.1. Synthesis of magnesium iron oxide

Analytically magnesium oxide and iron (III) oxide weighed an appropriate molar ratio and homogenized in an agate mortar. The mixture placed into a porcelain crucible to heat in microwave oven. After the material was exposed to microwave irradiation, it was taken from the oven, and then homogenized again. Then, final product was ready to analyse.

### 2.2. Analyse of structure

X-ray powder diffraction analysis (XRD) was performed using PANalytical X'Pert PRO Diffractometer (XRD) with Cu K $\alpha$  (1.5406 Å, 45 kV and 30 mA) radiation. FTIR spectrum was taken on a Perkin Elmer Spectrum 100 FTIR Spectrometer from 4000 to 650 cm<sup>-1</sup>.

Thermo gravimetric-differential thermal analysis (TG/DTA) was carried out by Perkin Elmer Diamond TG/DTA. Morphological properties and semi-quantitative analyse of the sample were realized by ZEISS Supra 40 VP. Siemens V12 domestic microwave oven was used.

## 3. Results and discussion

The XRD pattern of the sample is shown in Figure 1. The microwave synthesis of Mg<sub>0.1551</sub>Fe<sub>1.8966</sub>O<sub>3</sub> (ICSD 51171) was achieved with this research. The material is crystallized in hexagonal system with unit cell parameters a=5.0490, b=5.0490, c=13.7890 Å and space group R-3c. The other supporting methods confirm the crystal structure. Non-stoichiometric Mg<sub>0.1551</sub>Fe<sub>1.8966</sub>O<sub>3</sub> (ICSD 51171) was synthesized by microwave method completely different from literature for the first time.

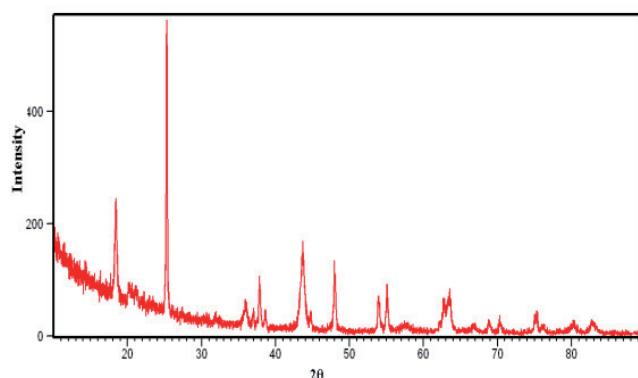
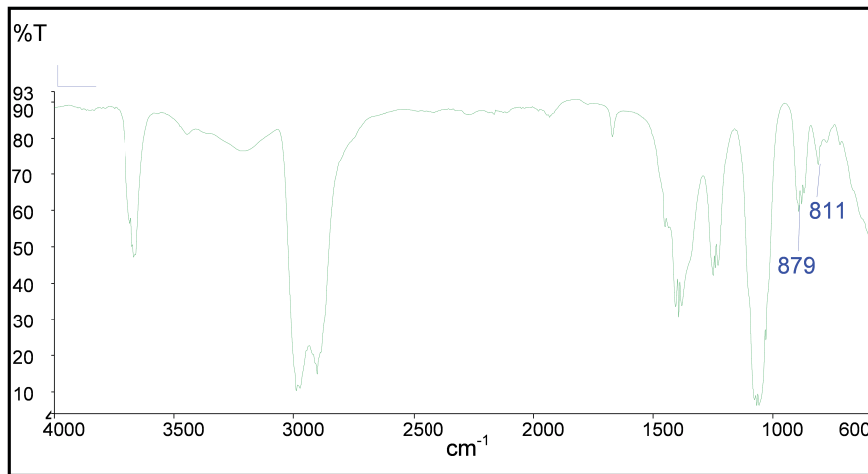
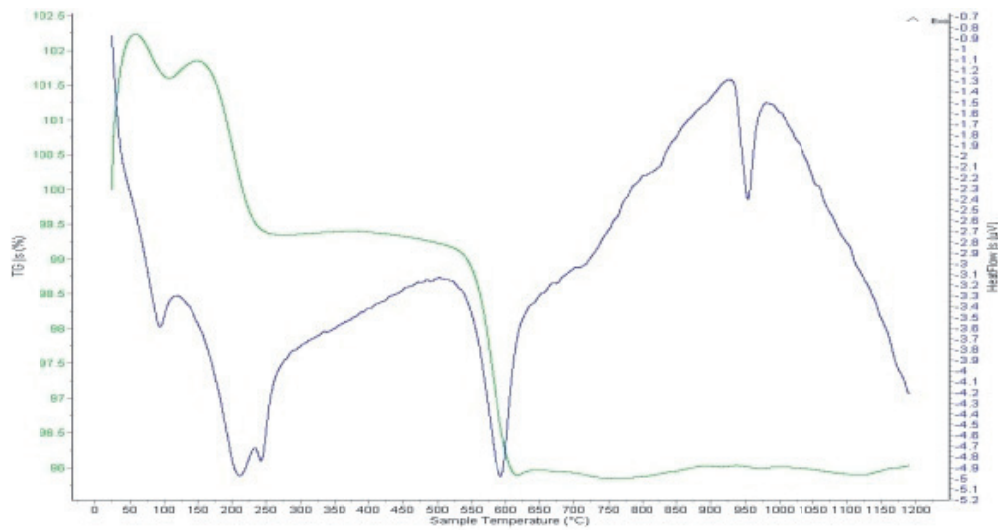
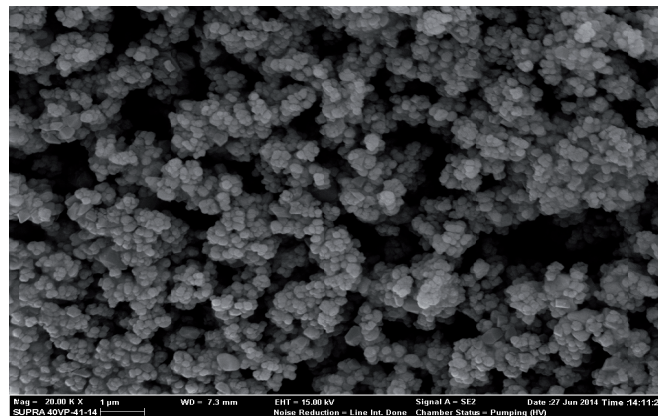


Fig. 1. XRD pattern of Mg<sub>0.1551</sub>Fe<sub>1.8966</sub>O<sub>3</sub>

FTIR spectra of the product are presented in Figure 2. The peak at 879 cm<sup>-1</sup> belongs to Fe-O vibration frequency. The peak at 811 cm<sup>-1</sup> belongs to Mg-O vibration frequency [6].

TG/DTA curves of magnesium iron oxide were given in Figure 3. The analysis was done in the 0-1200°C range. Because of no major weight loss in the range, the material is so stable to heat.

In Figure 4, SEM micrograph of the sample is displayed. Homogeneous view of the sample shows the high quality of crystallization and point out purity of the sample.

Fig. 2. FTIR spectrum of  $\text{Mg}_{0.1551}\text{Fe}_{1.8966}\text{O}_3$ Fig. 3. TG/DTA curves of  $\text{Mg}_{0.1551}\text{Fe}_{1.8966}\text{O}_3$ Fig. 4. SEM micrograph of  $\text{Mg}_{0.1551}\text{Fe}_{1.8966}\text{O}_3$

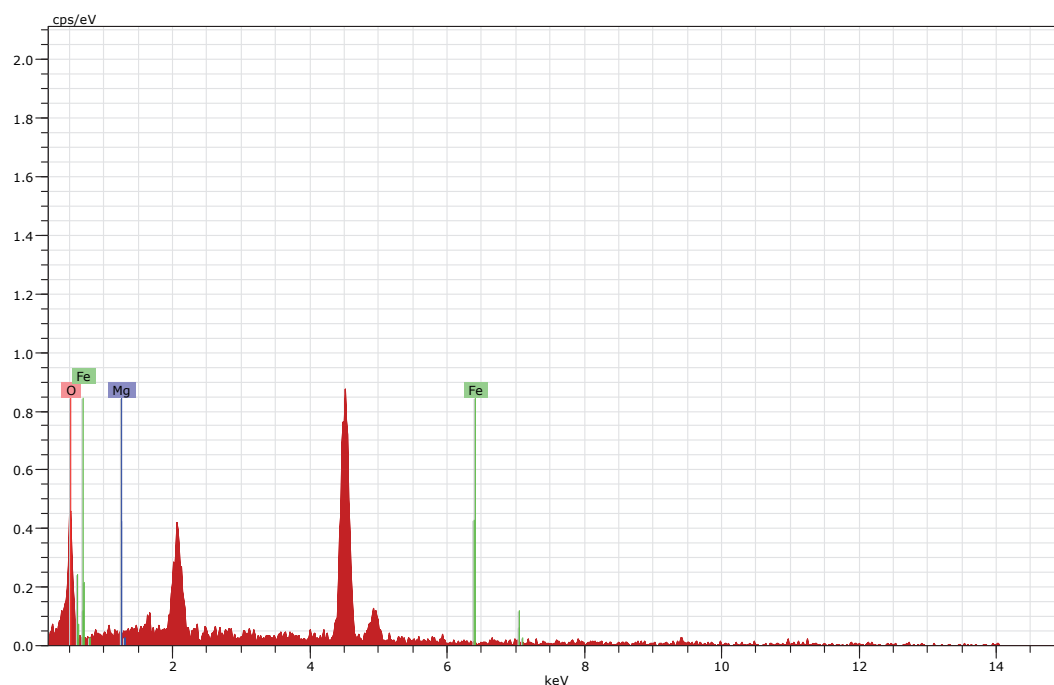


Fig. 5. EDX result of  $\text{Mg}_{0.1551}\text{Fe}_{1.8966}\text{O}_3$

The results of EDX analyse is exhibited in Figure 5. Green, pink and blue lines correspond with Fe, O, and Mg, respectively. The small peaks near 2, 4.3 and 4.5 keV become involved with platinum used covering the sample. The results are taken into account; the calculated combination ratio of the elements is coherent to the XRD data.

#### 4. Conclusions

In conclusion, non-stoichiometric  $\text{Mg}_{0.1551}\text{Fe}_{1.8966}\text{O}_3$  (ICSD 51171) was synthesized by microwave method completely different from literature for the first time. The characterization was mainly based on powder X-ray diffraction pattern. Also, thermal behaviour, morphology and EDX results were supported to the structure.

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#### Additional information

This paper will be presented in IMSP'2014.

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