# PREPARATION AND CHARACTERIZATION OF MAGNESIUM FERRITE ($MgFe_2O_4$) NANOPARTICLES

Toe,[1] Yi Yi Myint,[2] Kyaw Soe[3]

## Abstract

The materials with porous structure and high surface area are very popular for several applications in nanotechnology in the current years. The present research deals with a study on the synthesis and characterization of (spinels) nanoparticles ($MgFe_2O_4$). Two types of $MgFe_2O_4$ nanoparticles were synthesized by sol-gel method using two different acids such as acetic acid and formic acid. The metal nitrates such as magnesium nitrate and ferric nitrate were used as oxidizers. The gels were dried in the oven at temperature of 105°C to obtain constant weights. The resulting samples were thermally treated in the muffle furnace at temperature of 400 ºC for four hours to get required $MgFe_2O_4$ nanoparticles. The prepared samples were characterized by X-ray powder diffraction (XRD), Fourier transform Infrared spectroscopy (FTIR) and Energy Dispersive X-ray fluorescence (EDXRF).

**Keywords:** Magnesium ferrite, nanoparticles, spinels, sol-gel method

## Introduction

Gradually, primary energy resources such as fossil fuel, coal and natural gas are depleting, while the global energy consumption is increasing. Solar energy, wind energy, biomass, tidal and geothermal sources is emerging as an answer to the energy starved planet. These renewable energy resources which are freely available in nature are non-polluting and help in reducing global $CO_2$ emissions. Out of the available sources of energy, solar energy is the cleanest and the most abundant. Second part of the series challenges in the quest for clean energies is focused on different solar technologies and materials that can be used to make an efficient photovoltaic cell. Available photovoltaic cells can be broadly classified into first, second and third generation solar cells. First generation cells are basically silicon based crystalline cells while second generation cells are thin film based and third generation cells comprise new emerging technologies. Solar cells used for power generation must possess certain characteristics like high efficiency, low cost of materials, simple fabrication technique, ease of solar panel installation and long term stability. Unfortunately, there is not yet a device that can simultaneously meet all the above requirements. First and second generation solar cells have high efficiency and stability except for the amorphous silicon solar cells. However, they also possess some disadvantages. Majority of these solar cells employ high efficiency silicon based materials which are expensive. The scarcity of indium which is used in copper indium gallium silicon (CIGS) solar cells is a potential challenge for the widespread use of these cells. The toxicity of cadmium and the low earth abundance of tellurium (CdTe) solar cells.

In the recent years, researchers have focused on the development of cost-effective and feasible non-silicon solar cell technologies. Polycrystalline ferrites are optimal structural materials in high frequency circuits, owing to their excellent electrical and magnetic properties. Moreover, they are more stable than other competing materials and able to fulfill a range of applications in radio frequency circuits, operator devices (Schmid, 2010).

[1] Lecturer, Department of Chemistry, University of Magway
[2] Dr, Professor and Head, Department of Chemistry, University of Mandalay
3 Dr, Lecturer, Department of Chemistry, University of Magway

## Materials and Methods

All chemicals were analytical grade. Magnesium nitrate ($Mg(NO_3)_2.6H_2O$), iron nitrate ($Fe(NO_3)_3.9H_2O$), formic acid($CH_2O_2$), and acetic acid($C_2H_4O_2$) were Merck product with a purity of 99.99%. Ethylene glycol was product from Applichem, Germany. All solutions were prepared using distilled water during preparation procedures. Various conventional and modern instrumental techniques were used throughout the experimental procedure. These include X-ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FT-IR) and Energy Dispersive X-ray Fluorescence (EDXRF).

### Preparation of Magnesium Ferrite ($MgFe_2O_4$) Nanoparticles by Sol-Gel Method

Magnesium nitrate, iron nitrate, acetic acid, formic acid, ethylene glycol and distilled water were used as starting materials to synthesize two types of $MgFe_2O_4$ nanoparticles.

### Preparation of $MgFe_2O_4$ Nanoparticles using Acetic Acid by Sol-Gel Method

Magnesium nitrate and iron nitrate were dissolved in distilled water to obtain solution (I). Acetic acid was dissolved in distilled water and solution (II) was obtained. The solutions (I) and (II) were mixed as molar ratio of acetic acid and metal ion was 1:2 and then 5 mL of ethylene glycol was added to the mixture solution. The mixture solution was stirred with magnetic stirrer and heated at 70 ºC-80 ºC for 7 h to get $MgFe_2O_4$ gels. The gel was dried at 105 ºC for 4 h and ground with mortar and pestle to get dried $MgFe_2O_4$ powder. The prepared $MgFe_2O_4$ particles was calcined at 400 ºC for 4 h to obtain $MgFe_2O_4$ nanoparticles (MF1) (Sajjadi, 2005).

### Preparation of $MgFe_2O_4$ Nanoparticles using Formic Acid by Sol-Gel Method

Magnesium nitrate and iron nitrate were dissolved in distilled water to obtain solution (I). Formic acid was dissolved in distilled water and solution (II) was obtained. The solution (I) and (II) were mixed as molar ratio of formic acid and metal ion was 1:2 and then 5 mL of ethylene glycol was added to the mixture solution. The mixture solution was stirred with magnetic stirrer and heated at 70 ºC- 80ºC for 7 h to get $MgFe_2O_4$ gels. The gel was dried at 105 ºC for 4 h and ground with mortar and pestle to get dry $MgFe_2O_4$ powder. The prepared $MgFe_2O_4$ particles was calcined at 400 ºC for 4 h to obtain $MgFe_2O_4$ nanoparticles (MF2) (Sajjadi, 2005).

### Characterizations of $MgFe_2O_4$Nanoparticles

Crystal structure and phase analysis were performed by X-ray diffraction (XRD) using Rigaku, D-Max 2200, Japan in Universities' Research Centre, Yangon. The elemental compositions of the two prepared samples were confirmed by using EDXRF 700 spectrometer in Department of Chemistry, Monywa University. The chemical bondings of crystals were studied by Fourier Transform Infrared (FT IR) spectroscopy in Department of Chemistry, Monywa University.

### Determination of Crystalline Size and Interplanar Spacing

The crystallite size of $MgFe_2O_4$ can be calculated by using Debye-Scherrer formula (Leroy an Harold, 1950),

$$D = \frac{0.9\lambda}{\beta cos\theta} \quad \text{and}$$

interplanar spacing was computed by Bragg's equation,

$$d = \frac{\lambda}{2sin\theta}$$

Where, λ = the wave length of X-rays (1.54056Åfor Cu/K-alpha 1)

θ = the diffraction angle

β = full width at half maximum in radian

D =average crystal size

d = interplanar spacing

## Results and Discussion

### EDXRF Analysis of Magnesium Ferrite ($MgFe_2O_4$) Nanoparticles

The relative abundance of elements in magnesium ferrite synthesized with acetic acid (MF1) is shown in Table 1 and Figure 1. From the data, elements contained in MF1 were iron (64.555%) and magnesium (34.047%) and some of trace elements.

Elemental composition of magnesium ferrite MF2 synthesized with formic acid is shown in Table 2 and Figure 2. According to the experimental results, main elemental components were iron (70.886%) and magnesium (27.886%), and some trace elements.

**Table 1 Elemental Composition in Magnesium Ferrite ($MgFe_2O_4$) Nanoparticles MF3**

| No | Elements | Amount (%) |
|---|---|---|
| 1 | Fe | 64.555 |
| 2 | Mg | 34.047 |
| 3 | Si | 0.761 |
| 4 | S | 0.332 |
| 5 | Mn | 0.157 |
| 6 | Ca | 0.084 |
| 7 | Cr | 0.034 |
| 8 | Cu | 0.030 |

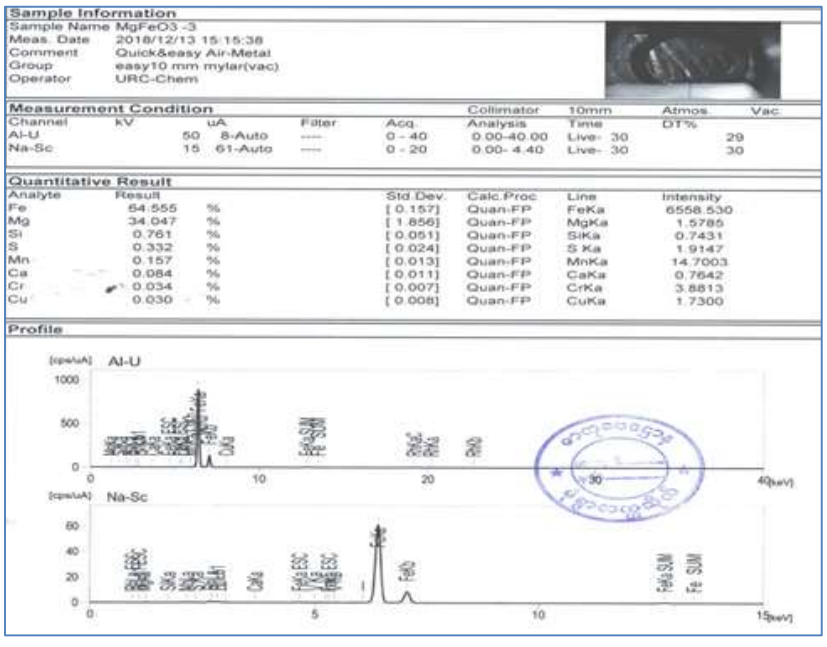


**Figure 1** EDXRF spectrum of MF1 nanoparticles

**Table 2 Elemental Composition in Magnesium Ferrite ($MgFe_2O_4$) Nanoparticles MF2**

| No | Element | Amount (%) |
|---|---|---|
| 1 | Fe | 70.886 |
| 2 | Mg | 27.886 |
| 3 | Si | 0.806 |
| 4 | S | 0.348 |
| 5 | Cr | 0.075 |

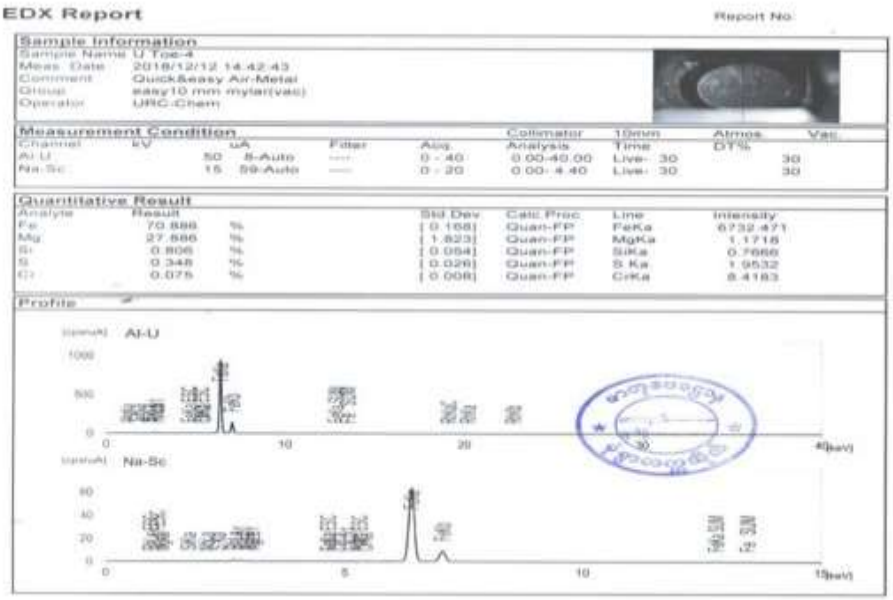


**Figure 2** EDXRF spectrum of MF4 nanoparticles

## FT IR Analysis of Magnesium ferrite ($MgFe_2O_4$) Nanoparticles

Magnesium ferrites (MF1 and MF2) were analyzed by FT-IR spectrophotometer. The characteristic features of FT IR spectra of MF1 and MF2 are shown in Figures 3 and 4, respectively. According to the FT IR spectral data as shown in Table 3, the stretching vibration of metal-oxygen bond of MF1 and MF2 were 523 $cm^{-1}$ and 530 $cm^{-1}$, respectively. The region 520-630 $cm^{-1}$ corresponds to stretching vibrations of metal ions in the tetrahedral sites (Kaur and Kaur, 2014). Anam *et al.* (2017) reported the stretching vibration of of Mg-O bond occured between 650-530 $cm^{-1}$.

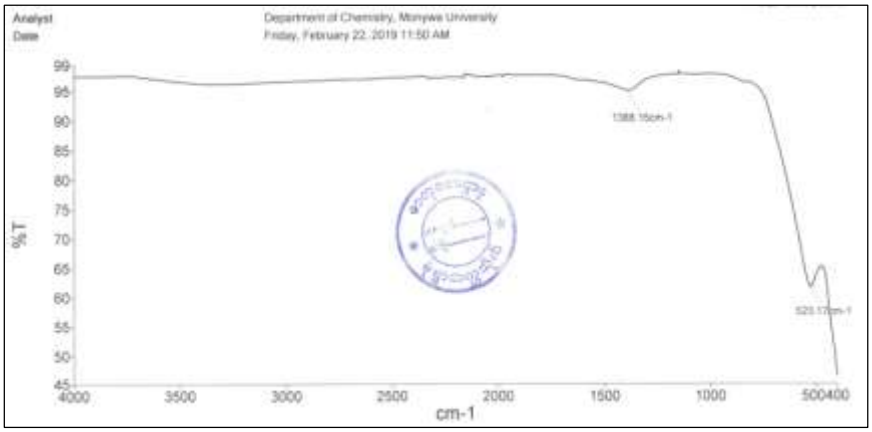


**Figure 3** FT IR spectrum of MF1 nanoparticles

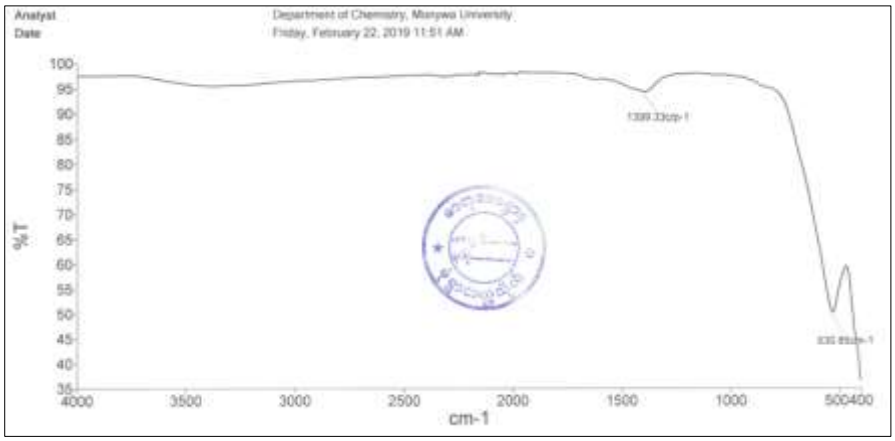


**Figure 4** FT IR spectrum of MF2 nanoparticles

**Table 3 FT IR Spectra Data for $MgFe_2O_4$ Nanoparticles**

| Sample | Wave number ($cm^{-1}$) | Remark | *Reported value |
|---|---|---|---|
| MF1 | 523 | stretching vibrations of metal ions in the tetrahedral sites | 520-630 $cm^{-1}$ |
| MF2 | 530 | stretching vibrations of metal ions in the tetrahedral sites | 520-630 $cm^{-1}$ |

* Kaur and Kaur (2014)

## XRD Analysis of Magnesium Ferrite ($MgFe_2O_4$) Nanoparticles

Figures 5 and 6 show XRD diffractograms of magnesium ferrites MF1 and MF2, respectively. The XRD patterns were compared with the standard powder diffraction pattern. The major planes correspond to (111), (220), (311), (222), (400), (422) and (511) were found to be matched with the library data which confirmed the presence of magnesium ferrite. All the observed peaks and Miller indices of the prepared magnesium ferrite were in agreement with the reported values (Arulmurugan *et al*., 2005; Spiers *et a*l., 2004) for $MgFe_2O_4$. Tables 4 and 5 show the phase identification of prepared magnesium ferrite samples, MF1 and MF2, and their crystallite sizes respectively. Peak locations (2θ) and Miller indices are also shown in these tables. Only single phase of magnesium ferrite was observed in both MF 1 and MF 2. From their data, chemical formulae of these nanoparticles were magnesium ferrites (spinels). The crystallite sizes were calculated using Bragg angle, 2θ(degree) and β, radian by Debye-Scherrer formula, $D = 0.9\ \lambda/\beta \cos\theta$. According to the calculated data, the average crystallite size of MF 2 (magnesium ferrite using formic acid) was slightly higher (28.58 nm) than MF 1 (magnesium ferrite using acetic acid) (15.25 nm ). These values were in the range of nanosize (1-100 nm). The difference in crystallite size was due to different preparation conditions for ferrite synthesis.

Tables 6 and 7 show lattice constants calculated from peak locations and Miller indices for MF1 and MF2 nanoparticles. From the data, lattice parameters of MF1 and MF2 were a= b= c= 0.836 nm and a = b=c= 0.839 nm, respectively. The crystal structures were indexed as face centered cubic having all odd or all even Miller indices. The volume of unit cells of prepared samples MF1 and MF2 were computed from their lattice parameters and found to be 506.17Å and 511.45Å, respectively.

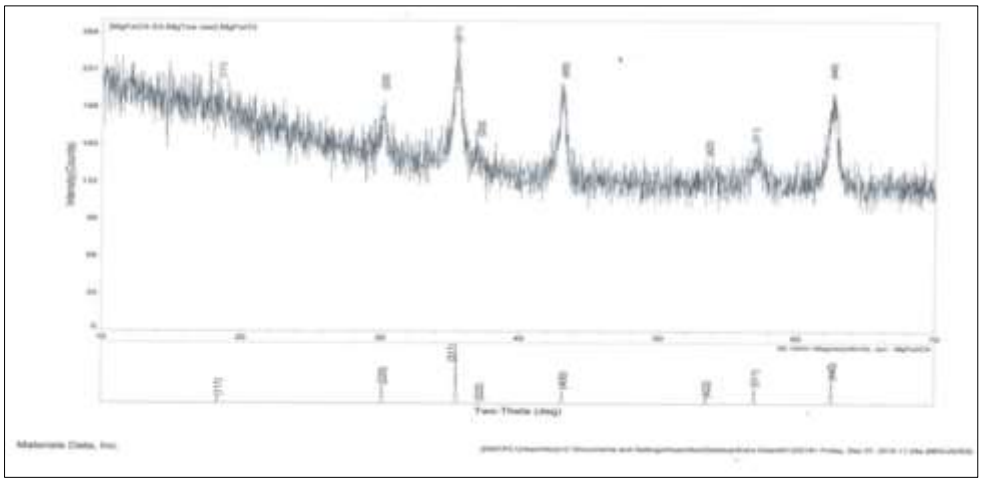

**Figure 5** XRD diffractogram of MF1 nanoparticles

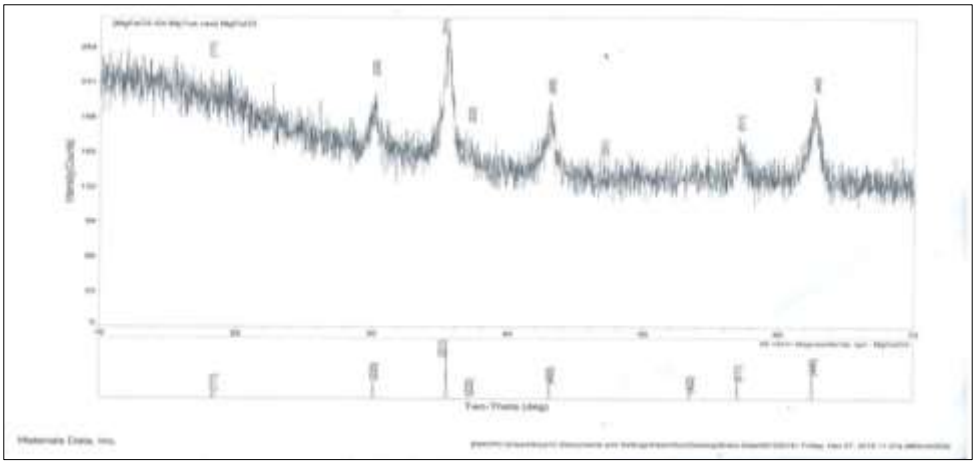

**Figure 6** XRD diffractogram of MF2 nanoparticles

**Table 4 Phase Identification and Crystallite Size of MF 1 Nanoparticles**

| No | Bragg angle,2θ ( degree) | Miller indices (hkl) | Interplanar spacing,d(nm ) | Phase ID | (β) radian | Crystallite size D (nm) |
|---|---|---|---|---|---|---|
| 1 | 18.553 | 1 1 1 | 0.478 | $MgFe_2O_4$ | 0.01216 | 11.55 |
| 2 | 30.210 | 2 2 0 | 0.296 | $MgFe_2O_4$ | 0.00751 | 19.26 |
| 3 | 35.422 | 3 1 1 | 0.253 | $MgFe_2O_4$ | 0.01021 | 14.29 |
| 4 | 37.155 | 2 2 2 | 0.242 | $MgFe_2O_4$ | 0.01019 | 14.44 |
| 5 | 43.138 | 4 0 0 | 0.210 | $MgFe_2O_4$ | 0.00899 | 16.71 |
| 6 | 53.556 | 4 2 2 | 0.171 | $MgFe_2O_4$ | 0.00981 | 15.94 |
| 7 | 56.907 | 5 1 1 | 0.162 | $MgFe_2O_4$ | 0.01178 | 13.46 |
| 8 | 62.516 | 4 4 0 | 0.148 | $MgFe_2O_4$ | 0.01006 | 16.31 |

Range of crystallite size = 11.55 – 16.71 nm
Average crystallite size = 15.25 nm

**Table 5 Phase Identification and Crystallite Size of MF 2 Nanoparticles**

| No | Bragg angle,2θ (degree) | Miller indices (hkl) | Interplanar spacing, d(nm) | Phase ID | (β) radian | Crystallite size D (nm) |
|---|---|---|---|---|---|---|
| 1 | 18.199 | 1 1 1 | 0.487 | $MgFe_2O_4$ | 0.00399 | 35.55 |
| 2 | 30.180 | 2 2 0 | 0.296 | $MgFe_2O_4$ | 0.00957 | 15.07 |
| 3 | 35.585 | 3 1 1 | 0.252 | $MgFe_2O_4$ | 0.01012 | 14.44 |
| 4 | 37.223 | 2 2 2 | 0.241 | $MgFe_2O_4$ | 0.00191 | 77.03 |
| 5 | 43.115 | 4 0 0 | 0.021 | $MgFe_2O_4$ | 0.0115 | 13.46 |
| 6 | 47.032 | 3 3 1 | 0.193 | $MgFe_2O_4$ | 0.00412 | 37.47 |
| 7 | 57.072 | 5 1 1 | 0.161 | $MgFe_2O_4$ | 0.00816 | 19.53 |
| 8 | 62.728 | 4 4 0 | 0.148 | $MgFe_2O_4$ | 0.01009 | 16.12 |

Range of crystallite size = 13.46 – 77.03 nm
Average crystallite size = 28.58 nm

**Table 6 Lattice Constants from Peak Locations and Miller Indices for MF1**

| No | Bragg angle (2θ) | Miller indices (hkl) | Inter planar spacingd (nm) | a-Axis (nm) | b-Axis (nm) | c-Axis (nm) |
|---|---|---|---|---|---|---|
| 1 | 18.553 | 1 1 1 | 0.478 | 0.828 | 0.828 | 0.828 |
| 2 | 30.210 | 2 2 0 | 0.296 | 0.836 | 0.836 | 0.836 |
| 3 | 35.422 | 3 1 1 | 0.253 | 0.840 | 0.840 | 0.840 |
| 4 | 37.155 | 2 2 2 | 0.242 | 0.838 | 0.838 | 0.838 |
| 5 | 43.138 | 4 0 0 | 0.210 | 0.838 | 0.838 | 0.838 |
| 6 | 53.556 | 4 2 2 | 0.171 | 0.838 | 0.838 | 0.838 |

**Table 7 Lattice Constants from Peak Locations and Miller Indices for MF2**

| No | Bragg angle (2θ) | Miller indices (hkl) | Inter planar spacing d(nm) | a-Axis (nm) | b-Axis (nm) | c-Axis (nm) |
|---|---|---|---|---|---|---|
| 1 | 18.199 | 1 1 1 | 0.487 | 0.844 | 0.844 | 0.844 |
| 2 | 30.180 | 2 2 0 | 0.296 | 0.837 | 0.837 | 0.837 |
| 3 | 35.585 | 3 1 1 | 0.252 | 0.836 | 0.836 | 0.836 |
| 4 | 37.223 | 2 2 2 | 0.241 | 0.836 | 0.836 | 0.836 |
| 5 | 43.115 | 4 0 0 | 0.021 | 0.839 | 0.839 | 0.839 |
| 6 | 47.032 | 3 3 1 | 0.193 | 0.842 | 0.842 | 0.842 |

## Conclusion

In this study, two types of magnesium ferrite ($MgFe_2O_4$) nanoparticles were synthesized from magnesium nitrate and ferric nitrate by sol-gel method with different acids such as acetic acid and formic acid. MF1 synthesized with acetic acid and MF2 synthesized with formic acid were characterized by modern sophisticated methods such as EDXRF, XRD and FTIR. From EDXRF data, the amount of magnesium and iron were found to be 34.047 % and 64.555 % for MF1 and 27.886 % and 70.886 % for MF2. Prepared magnesium ferrite samples were confirmed by the presence of the stretching band of metal-oxygen bonds appeared between 520-630 $cm^{-1}$ in FT IR spectra. The XRD reults revealed the impurity free nanocrystalline spinel MgFe2O4 by showing no impurity peaks except magnesium ferrite.From the XRD results, the average crystallite sizes of

vibration prepared nanoparticles for MF1 and MF2 were found to be 15.25 nm and 28.58 nm, respectively, which were in the range of nanosize. The prepared magnesium ferrites were indexed as face centered cubic structure with equal lengths of 0.836 nm and0.839 nm for MF1 and MF 2, respectively. Thus, the sol-gel method is well suited for the synthesis of nano-sized spinel $MgFe_2O_4$.

## Acknowledgements

I am extremely grateful to Dr Khin Maung Oo, Rector, University of Magway and Professor and Head, Dr Thidar Aung, Department of Chemistry, Magway University for their provision and suggestions of the research facilities.

## References

Anam, A., Abad, A., Mohd, A. and Shams, U. (2017). "Microwave- Assisted MgO NP Catalyzed One- Pot Multicomponent Synthesis of Polysubstituted Steroidal Pyridines". *New Journal of Chemistry,* vol. 42(1), pp. 184-197

Arulmurugan,R., Jeyadevan, B.,Vaidyanathan,G. and Sendhilnathan,S.(2005). "Effect of Zinc Substitution on CoZn and Mn-Zn ferrite Nanoparticles Prepared by Coprecipitation",. *J. Magn. Magn. Mater*., vol., 288, pp. 470-477

Kaur,N. and Kaur, M. ( 2014). "Comparative Studies on Impact of Synthesis Methods on Structural and Magnetic Properties of Magnesium Ferrite Nanoparticles".*Processing and Application of Ceramics,* vol. 8 (3), pp.137–143

Leroy, A. and Harold, P. K. (1950). "Determination of Crystallite Size with the X-Ray Spectrometer". *Journal of Applied Physics*, **21** (1), 137-142

Sajjadi, S. P. (2005). "Sol-gel Process and its Application in Nanotechnology". *Journal of Polymer Engineering and Technology,* vol.23 (1), pp.38-41

Schmid, G.. (2010). *Nanoparticles: From Theory to Application*. Weinheim: 2nd, Wiley-VCH, 517-522

Spiers, H., Parkin,I.P., Pankhurst,Q.A., Affleck,L., Green, M., Caruana, D.J. ,Kuznetsov, M.V. ,Yao, J.,Vaughan, G., Terry, A. and Kvick, A.(2004). "Self Propagating High Temperature Synthesis of Magnesium Zinc Ferrites $Mg_xZn_{1-x}Fe_2O_4$: Thermal Imaging and Time Resolved X-ray Diffraction Experiments". *J. Mater. Chem*., vol. 14, pp. 1104 -1111