



WILLIAMSON-HALL PLOTTING STUDIES OF MgO AND Fe DOPED MgO NANOPARTICLES BY GREEN METHOD

L. Umaralikhan* and M. Jamal Mohamed Jaffar

PG and Research Department of Physics, Jamal Mohamed College, Tiruchirappalli, Tamil Nadu-620 020, India.

***Corresponding Author: L. Umaralikhan**

PG and Research Department of Physics, Jamal Mohamed College, Tiruchirappalli, Tamil Nadu-620 020, India.

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ABSTRACT

The synthesis of MgO and Fe (%1, %2) doped MgO nanoparticles prepared by green method using *Pisidium guajava* leaf extract. MgO and Fe doped MgO nanoparticle sizes were calculated from the X-ray powder diffraction studies. When nanoparticles are not perfect crystals, microstrain contributes to the line broadening of diffraction peaks. In the present work, we show how to calculate directly the micro-strain for MgO and Fe (%1, %2) doped MgO nanoparticles using the Williamson-Hall plotting method. In addition, FESEM image shows the synthesized nanoparticles spherical shape.

KEYWORD: MgO, Fe doped MgO, X-ray broadening, FESEM.

INTRODUCTION

Nanoparticles have been versatile applications in the field of science and technology. It has been enormous attention due to their exceedingly small size and large surface-to-volume ratio, which leads to major change in their properties compared to mass of the same element constitution.^[1] Due to this reason synthesis and characterization of nanoparticles have been very important.

MgO nanoparticles have been attracted considerable attention because of their fundamental properties and applications in many areas of physics, chemistry, and materials science. MgO nanoparticles are used as catalysis antibacterial agent, biodiesel synthesis, and optoelectronic devices.^[2]

A perfect crystal would extend infinitely in all directions but no crystals are perfect due to their finite size. This deviation from perfect crystallinity leads to a broadening of diffraction peaks. The two main properties extracted from peak width analysis are the crystallite size and lattice strain. The crystallite size is a measure of the size of coherently diffracting domains. The crystallite size of the particles is generally not the same as the particle size due to the formation of polycrystalline aggregates. The lattice strain is a measure of the distribution of lattice constants arising from crystal imperfections such as lattice dislocations. Other sources of strain include the grain boundary triple junction, contact or sinter stresses, stacking faults and coherency stresses.^[3]

In literature metal oxide nanoparticles were synthesized by various methods such as chemical and physical methods^[4,5], and also green methods.^[2] Among this, Green method have been suggested as possible eco-friendly alternative methods over chemical and physical methods.

In present work, MgO^[2] and Fe (%1, %2) doped MgO NPs synthesized by *Pisidium guajava* leaf extract. The comparative evaluation of average particle size of pure and Fe doped MgO NPs obtained from powder XRD procedures has been reported. In addition, strain of the nanoparticles calculated using Williamson-Hall plotting method. To the best of our knowledge, this is the first report on the phytosynthesized MgO and Fe doped MgO NPs examined by broadening analysis.

MATERIALS AND METHODS

Sample preparation

The 10 g of fresh cut leaves of *Pisidium guajava* leaf (PGL) was added with 100 ml of double distilled water and boiled at 80 °C for 20 min. The obtained extraction was filtered using Whatman No. 1 filter paper and the filtrate was collected in 250 ml Erlenmeyer flask and stored at room temperature for further usage.

The following high purity chemicals such as Magnesium nitrate hexahydrate ($\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) and Iron (III) nitrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$), were used as the precursors without further purification.

The synthesis of MgO NPs methodology was published in our earlier paper.^[2]

The preparation of Fe (1%) doped MgO NPs (%1 FMO), 0.001 M aqueous Iron (III) nitrate solution was added into 0.099 M of aqueous Magnesium nitrate hexahydrate solution. This solution was mixed with 100 ml of PGL extract; we get the brown color homogeneous mixture solution.

Similarly, The preparation of Fe (2%) doped MgO NPs (%2 FMO), 0.002 M aqueous Iron (III) nitrate solution was added into 0.098 M of aqueous Magnesium nitrate hexahydrate solution. This solution was mixed with 100 ml of PGL extract; we get the brown color homogeneous mixture solution.

Both solution was stirred constantly at a temperature of 80 °C for 4–6h. Further the precipitate was calcined at 700 °C for 5 h.

Characterization Techniques

The synthesized NPs were characterized by X-ray diffractometer (model: X'PERT PRO PANalytical). The diffraction pattern was recorded in the range of 25°–80° for the synthesized NPs samples, where the monochromatic wavelength of 1.54 Å was used. The surface morphology of the product was examined by Field Emission Scanning Electron Microscopy (Carl Zeiss Ultra 55 FESEM) with EDAX (model: Inca).

RESULTS AND DISCUSSION

The XRD pattern of %1 FMO and %2 FMO NPs are shown in Fig. 1. The observed d-spacing of %1 FMO and %2 FMO NPs for the corresponding diffraction planes of (111), (200), (220), (311) and (222) are well oriented with the JCPDS card no: 4-829 and belong to a space group *fm-3m* (225).^[5] The obtained diffraction patterns reveal that the synthesized nanoparticles are of cubic structure. The lower angle shift in diffraction peaks for (%1, %2) FMO NPs, which is may be due to Fe³⁺ ion doping effects. FMO NPs intensity increased as

compared to that of MgO NPs. This may be incorporation of Fe³⁺ ions in MgO matrix (see Fig. 2).

The micro-strain the breadth of the Bragg peak is a combination of both instrument and sample-dependent effects. To decouple these contributions, it is necessary to collect a diffraction pattern from the line broadening of a standard material such as silicon to determine the instrumental broadening. The instrument-corrected broadening $\beta_{hkl}^{[6]}$ can be represented by.

$$\beta_{hkl} = [\beta_{hkl}^2 \text{ measured} - \beta_{hkl}^2 \text{ instrumental}]^{1/2} \quad (1)$$

Synthesized MgO and FMO NPs size were determined by the X-ray line broadening method using the Scherrer's equation,

$$D = k\lambda / (\beta \cos \theta) \quad (2)$$

where D-is the size in nanometers, λ is the wavelength of the radiation (1.5406 Å for CuK α), k is a constant (0.94), β D-is the peak width at half-maximum intensity and θ is the peak position. The average crystallite size 50, 34 and 28 nm for pure MgO, %1 FMO and %2 FMO NPs respectively. The strain-induced broadening β_{ϵ} is given by the Wilson formula $\beta_{\epsilon} = 4\epsilon \tan \theta$, where ϵ is the root mean square value of the micro-strain. Assuming that the particles size and strain contributing to the line broadening and independent of each other and both have a Cauchy like profile, the observed line width is simply the sum of these two, i.e., $\beta_{hkl} = (k\lambda / D \cos \theta) + 4\epsilon \tan \theta$, which becomes as.

$$\beta_{hkl} \cos \theta = (k\lambda / D) + 4\epsilon \sin \theta \quad (3)$$

When plotting the Williamson-Hall equation between $\beta \cos \theta$ Vs. $4 \sin \theta$, the slope of the line is the strain ϵ . Figures 3 (a-b) show the plots of Williamson-Hall equation for MgO and FMO NPs samples. As compared to the MgO NPs, the strain decreases in the %1 FMO and %2 FMO NPs. This is due to the relaxation of the strain in the respective unit cells. As the result, particle size decreases with increases of doping percentage. This size reduction mainly due to the distortion in the host lattice by the foreign impurities (Fe³⁺ ion).

The calculated strain values and size are compared with the pure MgO NPs given in Table 1.

Table 1: Strain and size comparison of MgO, %1 FMO and %2 FMO.

Samples	JCPDS Card NO	Average Crystallite size D (nm)	Strain
MgO [2]	89-7746	50	0.00348
%1 FMO	4-829	34	0.001153
%2 FMO	4-829	28	0.00065

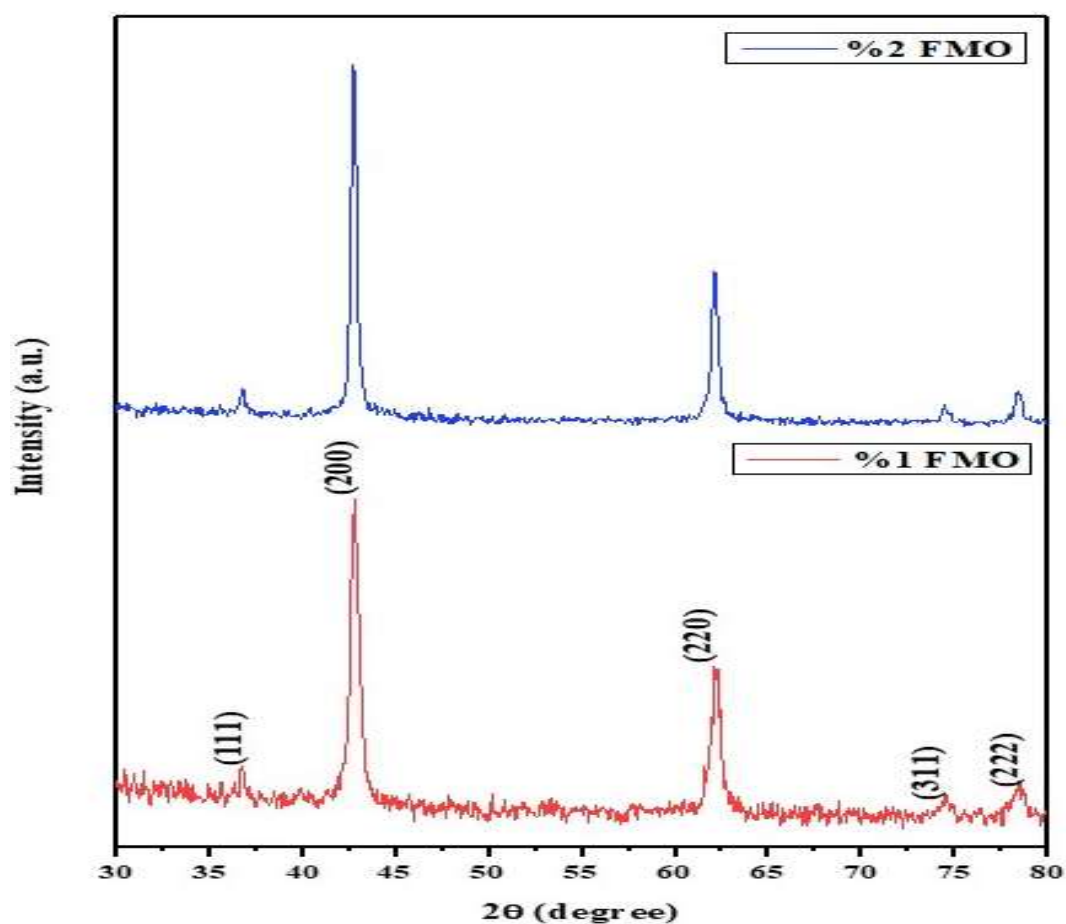


Figure 1: X-ray powder diffraction patterns of %1 FMO and %2 FMO NPs.

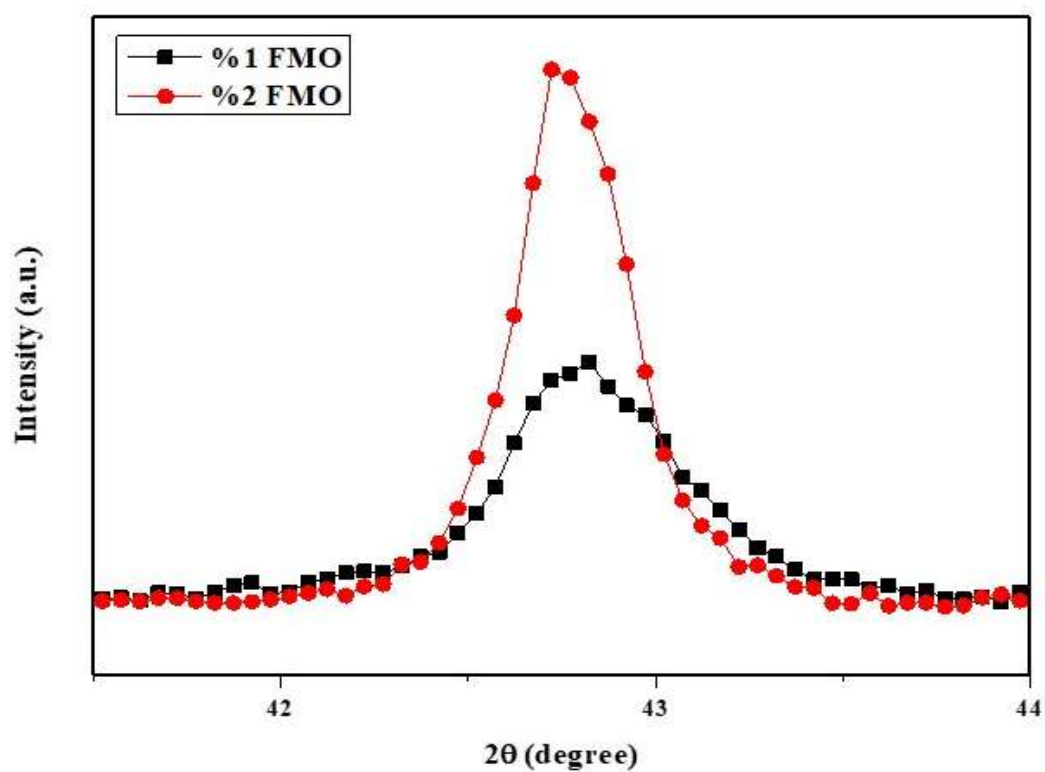
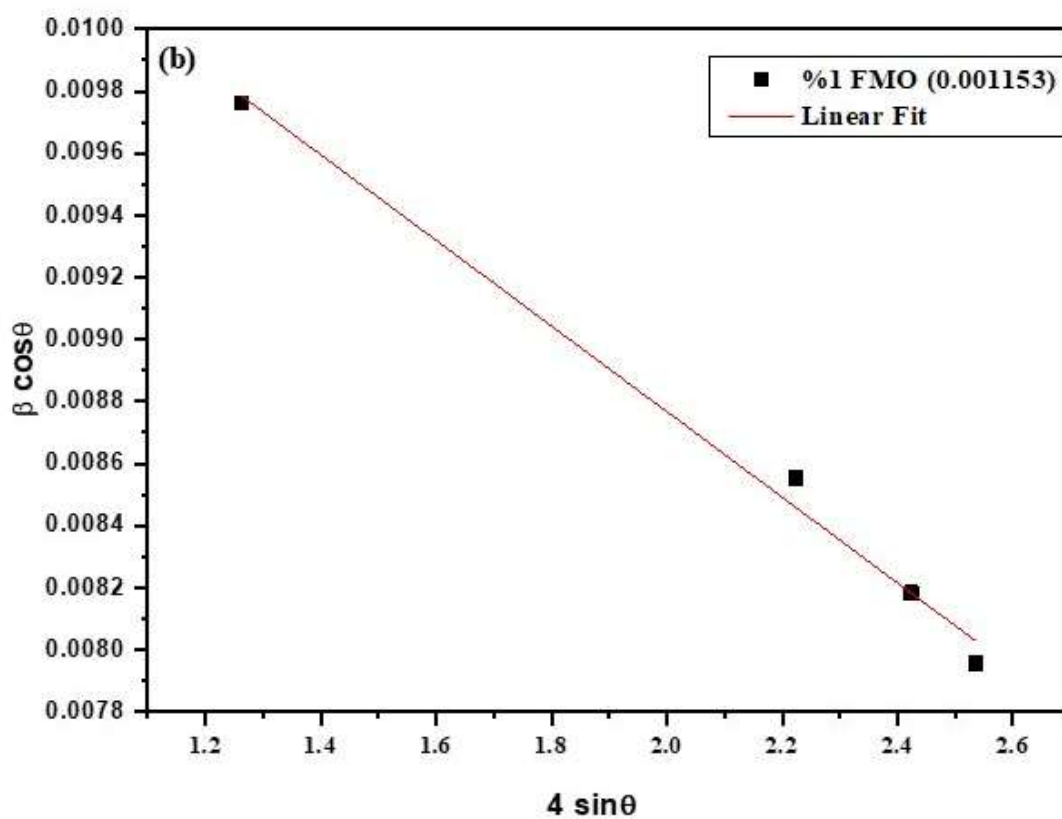
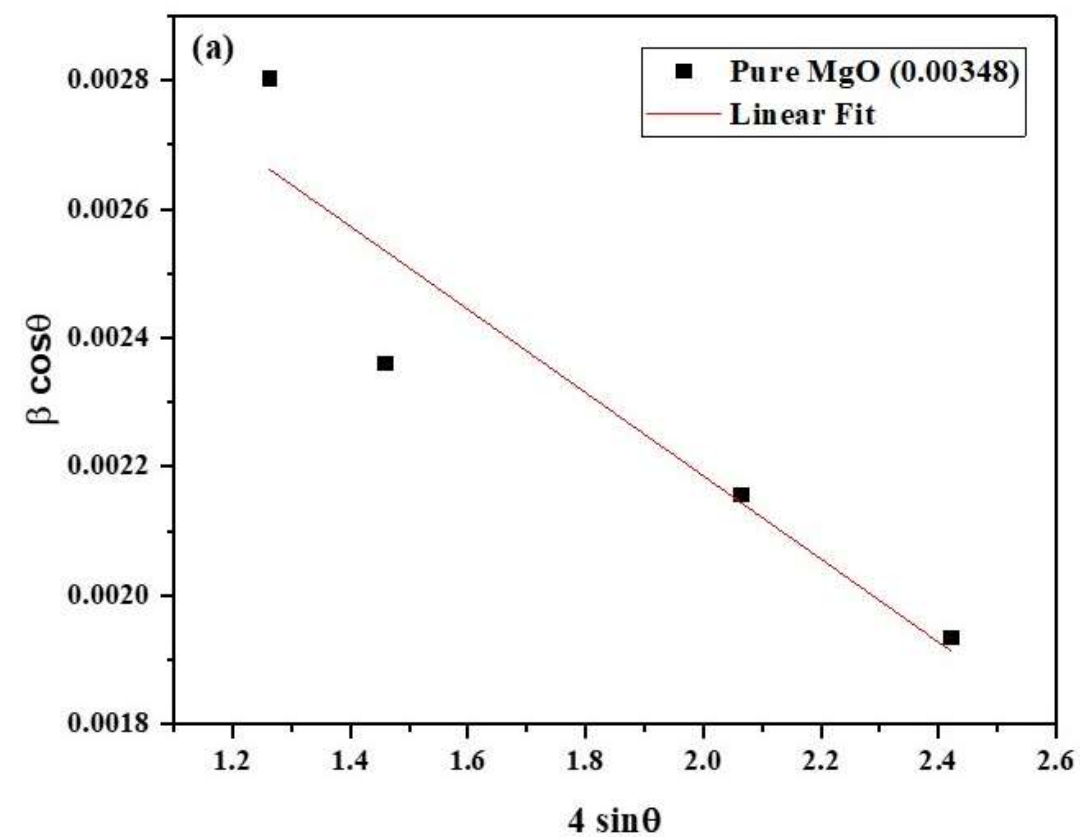


Figure 2: X-ray powder diffraction patterns of the doping-induced peak shift for %1 FMO and %2 FMO NPs.



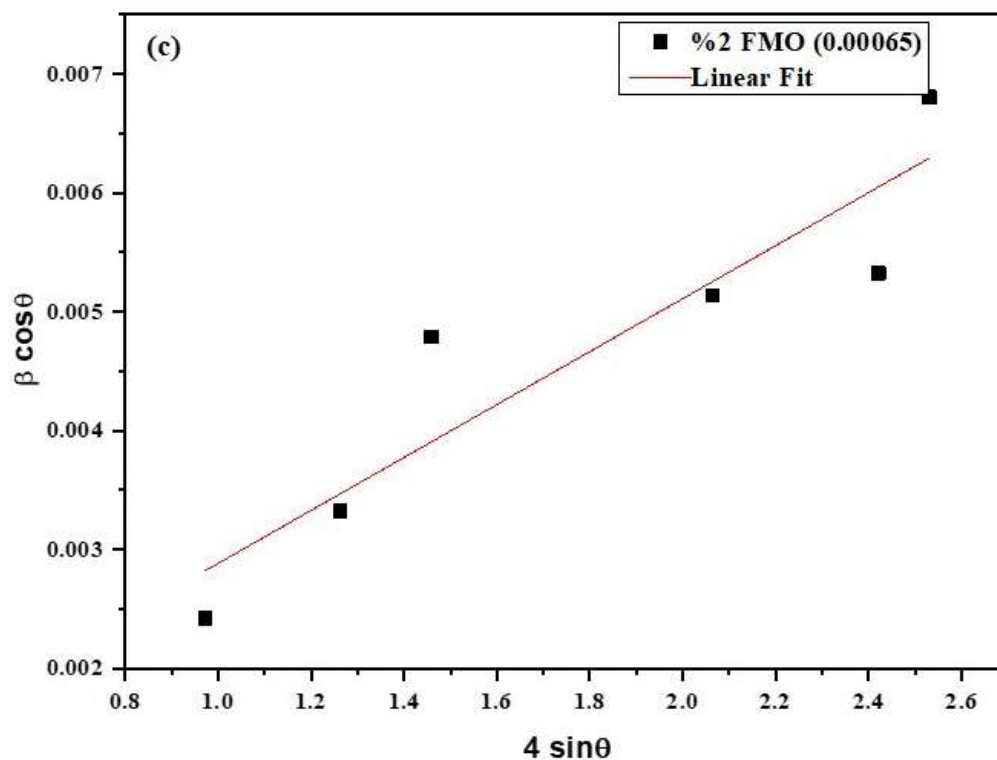
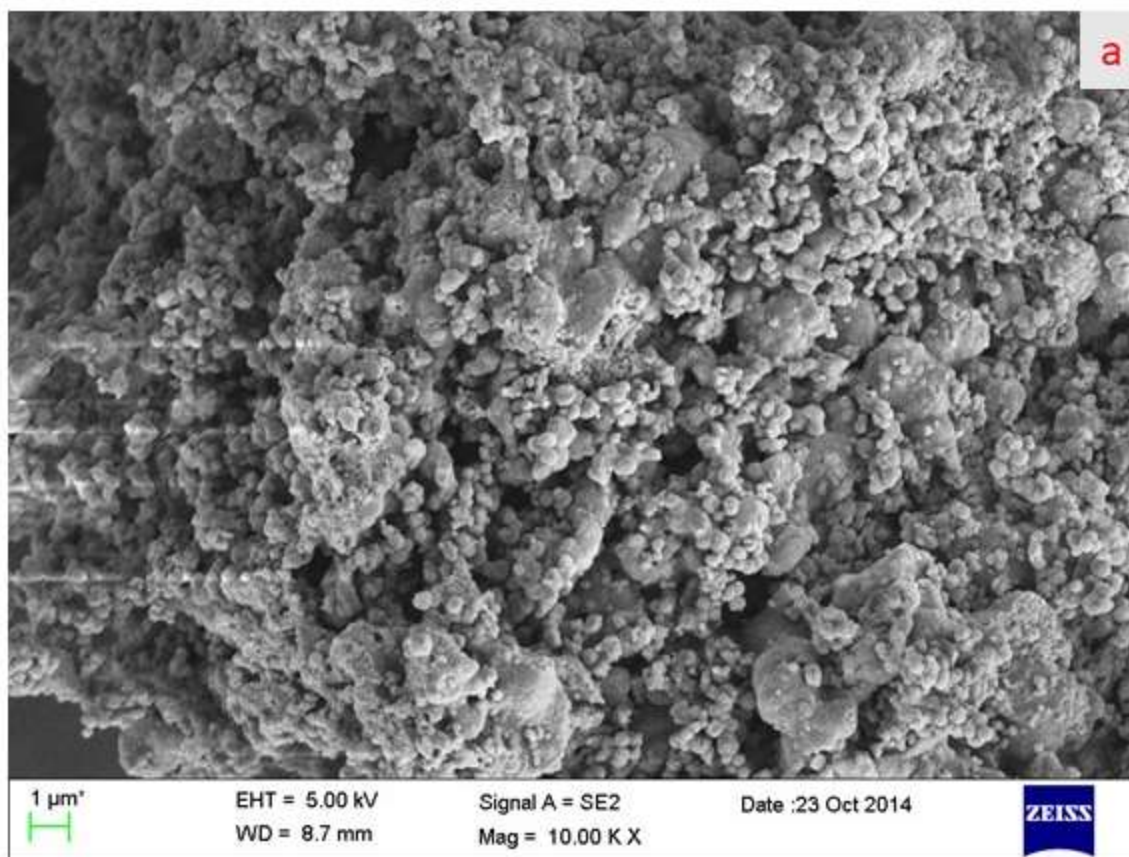


Figure 3: The Williamson-Hall plots of (a) MgO, (b) %1 FMO and (c) %2 FMO NPs. The solid lines show the theoretical fit.



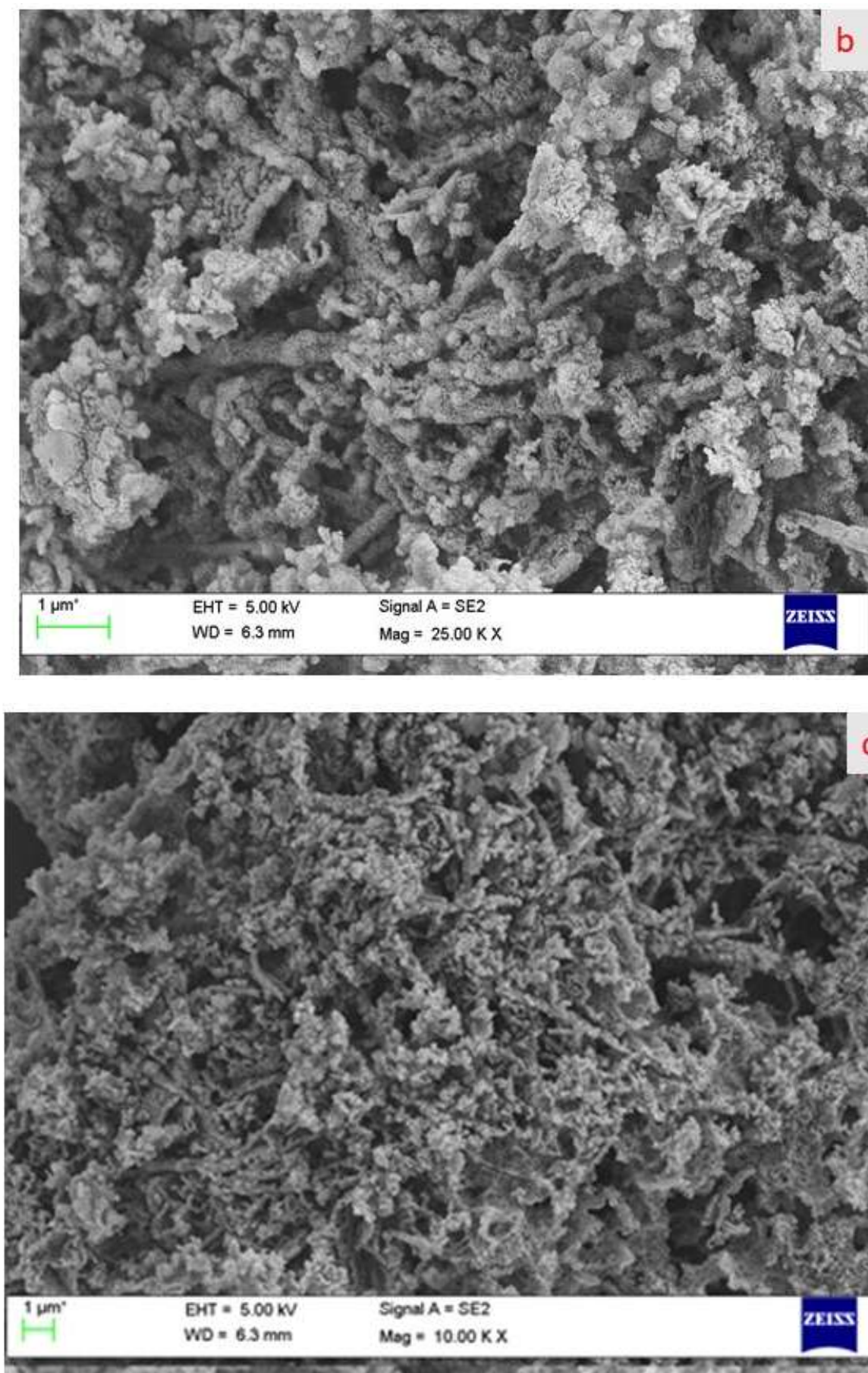


Figure 4: SEM image of (a) MgO (b) %1 FMO and (c) %2 FMO NPs.

Figure 4 displays a representative FESEM image of the as-prepared nanoparticles. It was clearly observed from SEM pictures shows hollow sphered morphology with porous and large agglomerates of very fine particles. The SEM results reveal that the powder was porous and agglomerated with poly-crystalline nanoparticles.

CONCLUSION

The MgO and Fe(%1, %2) doped MgO NPs were synthesized by green method using PGL leaf extract. Synthesized samples were calcinated at 700 °C. The XRD patterns confirmed the cubic structure and particle size. The MgO, %1 FMO and %2 FMO NPs average particle sizes were 50, 34 and 28 nm respectively. Fe doped MgO NPs sizes were decreases as compared to pure MgO NPs, this size reduction mainly due to the distortion in the host MgO lattice by Fe^{3+} ion. The line broadening analysis of MgO and Fe (%1, %2) doped MgO were done by W-H method. The results showed, the crystallite size and lattice strains decreases with increases of Fe doping concentration. From FESEM image

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