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Synthesis and Structural Properties of Homogenous Nanoporous Lithium Substituted Magnesium Ferrite

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ABSTRACT

Nanotechnology progress in Lithium substituted Magnesium Ferrite offers the opportunity to study the role of nano-particles and nanopores in various applications like humidity sensing by physisorption of water vapors on magnesium ferrite and hydroelectric cell based on water dissociation by nanoporous ferrite. The nanoparticles synthesized by various researchers were not homogeneous as these involve very less calcinations time. In the present work, lithium is substituted in different proportions in Magnesium Ferrite. Synthesis of magnesium ferrite at low temperature generated very homogenous nanopores in microstructure. The nanoparticles of ferrite were prepared by solid state reaction method. The synthesized ferrites were characterized by X-ray diffraction (XRD) and scanning electron microscope (SEM) and resemble the structure of ferrite.

Keywords: Lithium substituted Magnesium Ferrite, Octahedral sites, Nanopores, Physisorption.

I. INTRODUCTION

The reserves of non renewable sources of energy like fossil fuels are close at vanishing stage. As per the report on climate change 2014 by International Panel on the climate change, the world must increase the use of green energy sources [1]. Present researchers are focusing more and more on renewable and clean sources of energy. In the past, a lot of work is done on thermoelectric materials, perovskite solar cell, production of hydrogen for fuel cells, silicon solar cell and organic solar cell [2-6]. In 2014, the renewable energy in a global electricity generation has approached a share of 22.8% standing third largest contributor just behind coal and gas [7]. A lot of efforts have been done to use water or water vapors as electrical source, but a little progress is done in this direction. Graphene oxide based cell of area 0.1 cm^2 at 70% humidity can produce a maximum power of 2μW [8]. Jumping water droplet from super hydrophobic surface can generate picowatt power [9]. Using different combinations of electrodes in cement matrix has been explored as a battery. This produces a very low current of 0.1 mA out of $7\times7\times4$ cm^3 cement electrolyte mould [10].

Lithium substituted magnesium ferrite can be used as Hydroelectric Cell (HEC) which generates 82 mA short circuit current and 920 mV emf with a maximum power output of 741 mW using water by Kotnala et al [11]. They have invented a revolutionary hydroelectric cell based upon nanoporous lithium substituted magnesium ferrite.

Nanomaterials like spinel ferrites play an important role in chemical and physical applications due to high surface energies. They are important magnetic materials and have low cost. Spinel ferrites have the formula AB_2O_4, where A-site is occupied by divalent cations which are tetrahedrally coordinated and B-site is occupied by trivalent ion Fe^{3+} and are octahedrally coordinated. These ferrites are widely used in electronic and magnetic devices because of their low magnetic losses and high magnetic permeability. They can be prepared by various methods as discussed below.

In co-precipitation method 2.0 mol of $Fe(NO_3).9H_2O$ and 1.0 mol of $Mg(NO_3)_2.6H_2O$ are dissolved in 20 ml of distilled water with a pH value 9 to 10 by adding 0.1 mol NaOH to give brown precipitates. To get pH 7 precipitates are filtered and washed repeatedly. Then the precipitates are dried and calcined to get desired ferrite [12]. In combustion method polyethylene glycol, urea and oxalyl dihydrazide are used to prepare $MgFe_2O_4$ nano particles. By using hydrazine hydrate and diethyl oxalate ODH required for synthesis is prepared. PEG or urea act as fuel and metal nitrates act as oxidizers [13]. In sol gel method, 20 ml of distilled water 2.0 mol of $Fe(NO_3)_3.9H_2O$ and 1.0 mol of $Mg(NO_3)_2.6H_2O$ are dissolved and 2.22 ml of citric acid is added. The aqueous mixture is magnetically stirred at 60 °C. To adjust solution pH 7, ammonium hydroxide is added resulting the mixture to form sol then it is stirred for 8 hours to turn in to gel. At 100 °C gel is dried for 4 h and calcined for 3h at 500 °C to make the ferrite [14]. But in all of these methods calcination time is less, so homogenous nanoporous material is not formed.

In the present study our aim is to prepare and study Lithium substituted Magnesium Ferrite with different ratios of lithium in it with homogenous nanopores formation.

II. EXPERIMENTAL

In this work lithium substituted magnesium ferrite was prepared by solid state reaction method. Four types of samples with different ratios were prepared by using the formula $Li_xMg_{1-x}Fe_2O_4$ where x=0.1 to 0.3. Highly pure powders of Li_2CO_3, $MgCO_3$ and Fe_2O_3 were taken in different molar ratios x: 1-x: 1.0 where x= 0.1 to 0.3. The precursor powders were mixed and grinded for 2.5 h in mortar and pestle to get fine powder. The powder obtained after grinding was calcined for 10 h in air at 800 °C and then it is grinded for 30 min with PVA and circular pellets of diameter 35 mm were formed. These pellets were sintered at temperature 1000 °C for 6h in air. Pellet formed has porous microstructure and is analyzed by SEM at accelerating voltage of 10kV.

III. RESULTS AND DISCUSSION

XRD analysis of lithium substituted magnesium ferrite

The XRD for all the three samples of lithium substituted magnesium ferrite is shown in the below figures. By using Scherer's equation the crystalline size (D) was measured

$$D= 0.9\lambda /\beta\cos\theta$$

Where β is equal to full width, λ is X-ray wavelength and θ is Bragg angle. After considering all the major peaks the average crystalline size is 28.7, 29.5 and 30.3 for samples 1, 2 and 3 respectively. Analysis of XRD revealed that the structures of synthesized samples are composed of diffraction peaks that agree with international standard diffraction data card. The most intense reflections obtained at 2θ are at about 30.3, 35.5, 43.3, 53.9, 57.4, 63.1 and 67.6 degree are assigned to (220), (311), (400), (422), (511), 440 and (533) planes of cubic spiel ferrite structure.

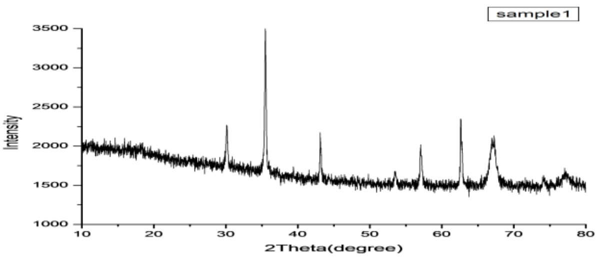

Figure 1. Lithium substituted magnesium ferrite sample1 XRD plot

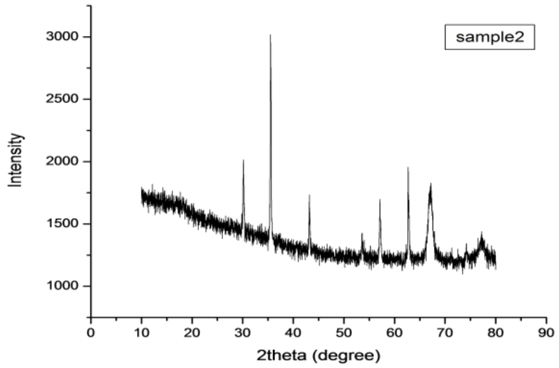

Figure 2. Lithium substituted magnesium ferrite sample2 XRD plot

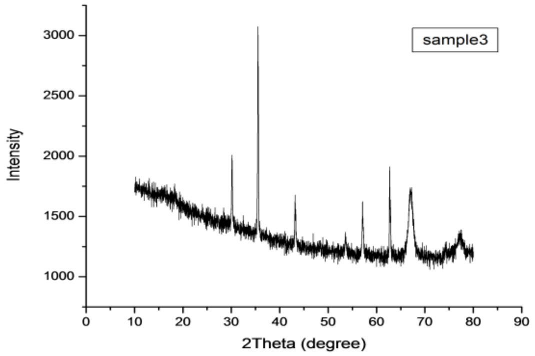

Figure 3. Lithium substituted magnesium ferrite sample3 XRD plot

SEM studies of lithium substituted magnesium ferrite

The SEM images of lithium substituted magnesium ferrite are obtained from EV018 Zeiss installed at SAIF, AIIMS New Delhi equipped with digital imaging and 35 mm photography system.

SEM pictograph of sample1, sample2 and sample3 are shown in the figures given below. The images show that synthesized pellets have uniform particle size as is depicted in table and show increase in traces of lithium in the samples from sample1 to sample3. Table 1 shows the comparison of particle size as obtained from XRD (using Scherer's formula) and SEM pictograph.

Table 1. Comparison of Particle size by XRD and SEM

S. No.	Sample name	Average particle size (nm) from XRD	Average particle size (nm) from SEM
1	Sample1	28.7	255
2	Sample2	29.5	246
3	Sample3	30.3	252

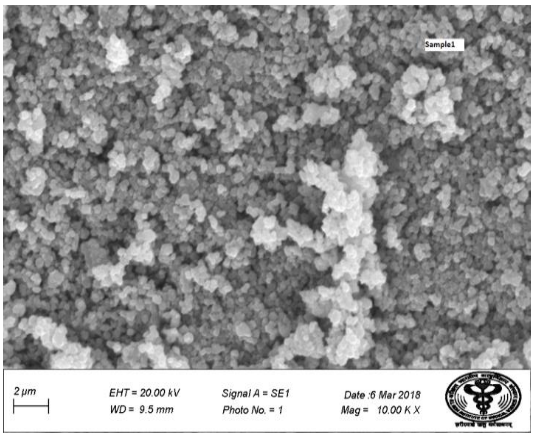

Figure 4. SEM Image of Sample1

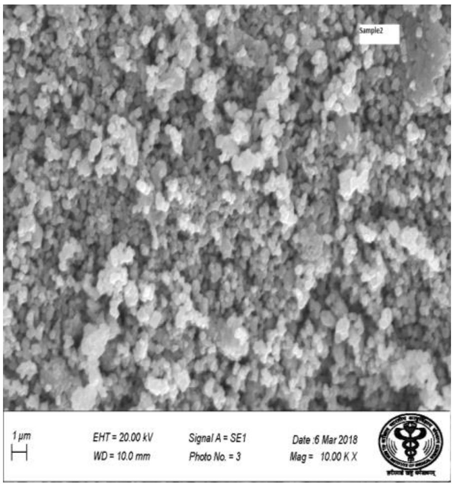

Figure 5. SEM Image of Sample2

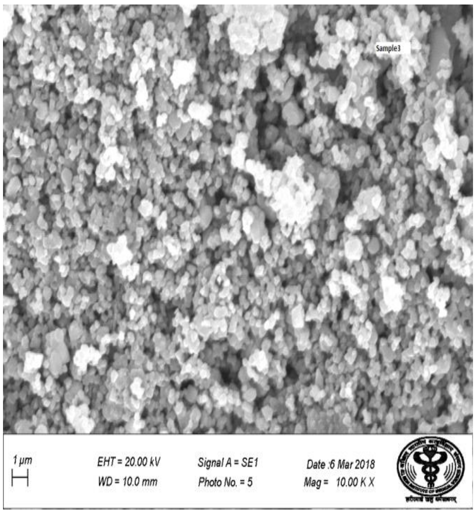

Figure 6. SEM Image of Sample3

IV. CONCLUSION

Lithium substituted magnesium ferrites in different ratios of lithium were synthesized successfully with homogenous particle sizes 28.7, 29.5 and 30.3 by using solid state reaction method. The XRD shows that the synthesized ferrite is not pure $MgFe_2O_4$ and it shows the existence of lithium in it. Studies from SEM shows particle sizes 255, 246 and 252 nm of the samples with traces of lithium. SEM images clearly show homogenous nanopores in the samples. The synthesized ferrites can be used in various applications like hydroelectric cell, humidity sensors and other sensing equipments.

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