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### RESEARCH ARTICLE

#### SUGARCANE JUICE BASED SOL-GEL METHOD FOR MN SUBSTITUTED MAGNESIUM FERRITES.

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#### Abstract

Manganese substituted magnesium ferrites having stoichiometric composition  $Mg_{1-x}Mn_xFe_2O_4$  with  $x$  ranging from 0.0 to 1.0 were prepared sugarcane juice based Sol-gel method. X-ray diffraction was used to characterize the structure and phases present respectively. It was found that of all ferrite compositions at  $800^\circ C$  led to cubes of the ferrite grains and that there was single phase spinel structure. The X-ray diffraction patterns revealed the presence of (311) peak as the most intense one. The crystallite size was found to be within the range of 24-48 nm. Activation energy of resultant samples decreases from  $0.45 \pm 0.02$  eV ( $x = 0.0$ ) to  $0.35 \pm 0.02$  eV ( $x = 1.0$ ).

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#### Introduction:-

Nanoscale crystalline mixed metal oxides are synthesized by several mechanical and chemical methods. The main objective is to reduce the costs of chemical synthesis and to produce materials for technological applications [1–5]. Among the several methods, high random distribution of the metal ions or complexes are based on the use of a matrix are fix (solution or gel). This increases the chemical homogeneity and reduces the segregation of a particular metal during the processing and crystallization of the mixed metal oxides [6–7]. In these chemical methods, the metal ions are immobilized on an atomic scale, which allows obtaining oxides at lower temperatures than solid state reaction. Due to the immobilization, the metal ions react with each other with a minimum diffusion; consequently, it is possible to synthesize homogeneous and stoichiometric oxides [8–9]. Sol–gel is one of the best techniques with these characteristics [10]. In these techniques, the particle size can be controlled by the formation and aggregation of the particles in the starting sol, which is dependent on the concentration of reagents, temperature, pH, electro negativity of the metals, solvent etc. [11–15]. Besides the versatility and simplicity water is the most polar solvent, the aqueous solution–gel method can offer a better chemical homogeneity compared to other solvents. Therefore, aqueous solution–gel synthesis method was tested to produce cheap nanocrystalline powders.

This new technique is based on the use of sugarcane juice as the chelating agent, and it stands out by its simplicity, low processing cost and reduces environmental pollution, using sugarcane juice and metal nitrates nano-crystalline mixed metal oxides are synthesized. The process is similar to previous work [16]. In this paper, the chemical route is similar but sugarcane juice is used as the chelating agent for the synthesis of nanoscale Mn Substituted  $MgFe_2O_4$  for the study of electrical resistivity.

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**Material and Methods:-**

Manganese substituted magnesium ferrite  $Mg_{(1-x)}Mn_xFe_2O_4$  (where  $x=0.0, 0.25, 0.50, 0.75$  and  $1.0$ ) samples were prepared sugarcane juice based sol-gel method [19]. The A.R. Grade chemicals used in the synthesis of materials were  $Mg(NO_3)_2 \cdot 6H_2O$ ,  $Mn(NO_3)_2 \cdot 4H_2O$ ,  $Fe(NO_3)_3 \cdot H_2O$  and  $NH_3$ . The metal nitrates were dissolved in minimum quantity of deionised water and equal quantities of sugarcane juice were added in the metal nitrate solution. In beaker sugarcane juice solutions of Manganese nitrate, magnesium nitrate and cobalt nitrate were mixed in required stoichiometric ratio. The ammonia solution (1:1) was added drop by drop until the pH of the solution reached a value of 9.5. The resulting solution was stirred for 3h to maintain the homogeneity. The gel is formed when solution were heated on hot plate at  $100^\circ C$  after words the gel is further heated at  $150^\circ C$  to convert into flabby mass. This flabby mass was annealed at the temperature of  $800^\circ C$  for 6 h in a Box furnace.

In order to investigate the grain size and phase X-ray diffraction (XRD) analysis was performed (Philips PW-1710 X-ray diffractometer with  $CuK\alpha$  radiation) [17]. The morphology of the material was studied by SEM (SEM Model JEOL-JSM 6360) [18].

**Results and discussion:-**

The XRD data of these ferrite samples agreed very closely with the standard values given in the JCPDS Data Cards ( $MgFe_2O_4$ -36-0398 and  $MnFe_2O_4$ -74-2403) thus confirming the spinel phase Fig.1. The phase identification of the final products of the various compositions of  $Mg_{1-x}Mn_xFe_2O_4$  oxides were made on Philips PW 1710 with  $Cu K\alpha$  radiation ( $\lambda=1.5405\text{\AA}$ ). The X-ray diffraction patterns of the investigated mixed Mg-Mn ferrite revealed that all the samples had a single phase cubic structure. Lattice parameters varied between 8.33 and  $8.36^\circ\text{\AA}$  with increasing Mn content. This increase may be related to the replacement of  $Mg^{2+}$  ion (0.065nm) with larger  $Mn^{2+}$  ion with ionic radius (0.0745 nm). Average particle size was determined from the broadening of X-ray diffraction peaks using the Scherrer equation and found to be in the range 24 to 48nm.(Table no.1)[19].

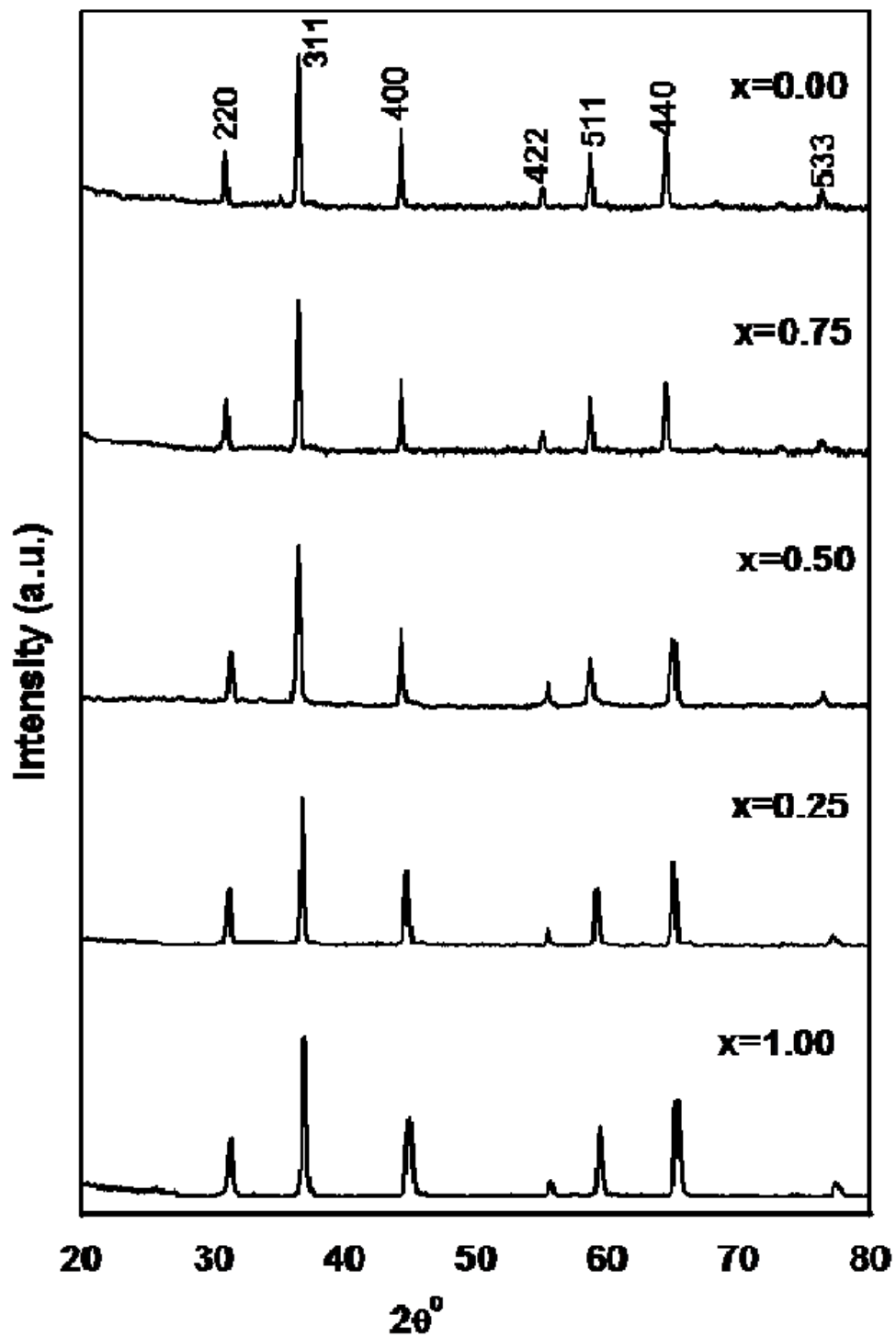
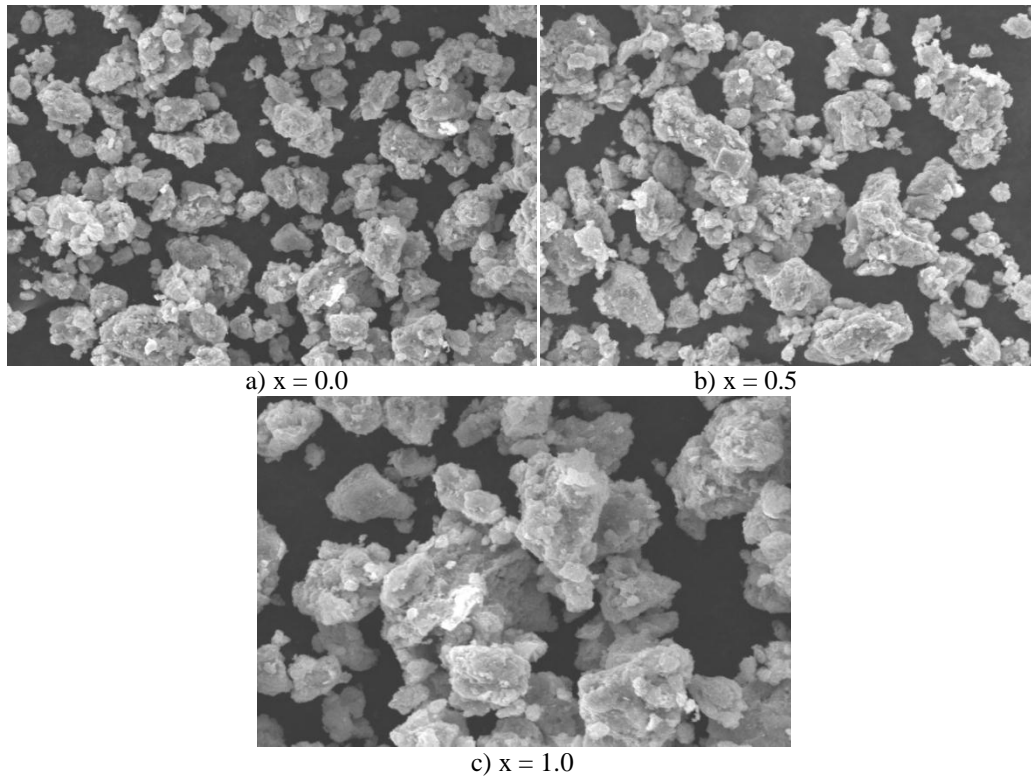


Fig.1:- XRD patterns of the  $\text{Mg}_{1-x}\text{Mn}_x\text{Fe}_2\text{O}_4$  ( $0.0 \leq x \leq 1.0$ )

**Table No.1:-**Lattice constant, Activation Energy and particle size for  $Mg_{1-x}Mn_xFe_2O_4$ .

Composition(x)	Lattice constant(a)	Activation Energy(eV)	Particle size(nm)
0.0	8.332	0.45	25
0.25	8.346	0.25	31
0.50	8.359	0.34	39
0.75	8.365	0.45	41
1.00	8.351	0.35	49

The morphological features were observed with a scanning electron microscope (SEM: model JEOL-JSM 6360).The scanning electron micrographs of all samples are shown in fig.2. The grain size was calculated from SEM micrographs, the average grain size was found to be 5  $\mu$ m. It appears that increase in the grain size with increase in Mn content.



**Fig.2:-**SEM micrographs of the  $Mg_{1-x}Mn_xFe_2O_4$  ferrites (a) 0.0 (b) 0.5 (c) 1.0

The electrical properties indicated that resistivity decreases with increase in temperature obeying Willson’s law.

$$\rho = \rho_0 \exp (\Delta E / K T) \text{ ----- (1)}$$

Where,  $\Delta E$  = Activation energy,  $K$  = Boltzmann constant &  $T$  = Absolute temperature. The variation of resistivity ( $\rho$ ) as a function of temperature ( $10^3/T$ ) is shown in Fig.3. The activation energies are calculated from the slopes, from these values it is evident that the lower activation energy in the ferromagnetic region is attributed to the phase transition or impurity phases, while the change in activation energy is attributed to the change in conduction mechanism [20].The change in activation energy for different compositions is attributed to the hopping of electrons [21]. An increasing Mn content (at A- site) the Mg ion concentration (at A- site) will decrease. Consequently ( $\rho$ ) decreases with increase in Mn content. Another reason for decreases in ( $\rho$ ) on increase in Mn ion substitution is that Mn is less resistive than Mg.

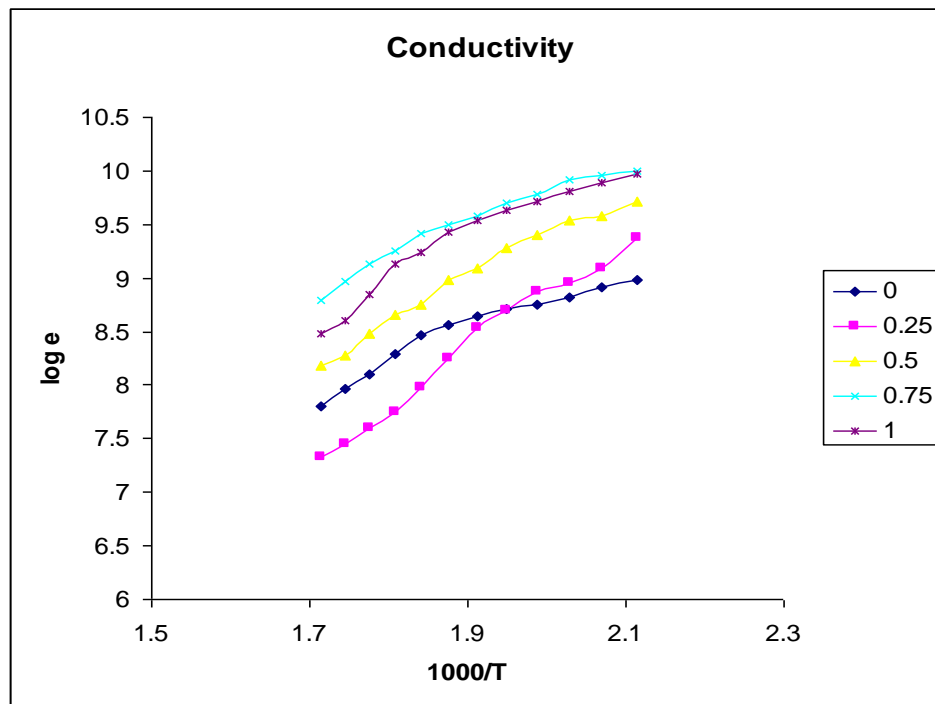


Fig.3:-Log  $\rho$  versus  $1000/T$  of samples of the  $Mg_{1-x}Mn_xFe_2O_4$  system.

### Conclusion:-

The ferrites  $Mg_{1-x}Mn_xFe_2O_4$  have been prepared by sugarcane juice based Sol-gel method in appropriate molar proportions. This new root stands out by its simplicity, low processing cost and reduces environmental pollution. The spinel structure was confirmed by X-ray diffraction. The particle size was determined from the broadening of X-ray diffraction peaks. SEM was no very good agreement with each other indicating that, there was no agglomeration and the size distribution of the prepared particles is uniform. The SEM showed that the particles synthesized sugarcane juice based sol-gel technique posses much smaller size. The conductivity graph shows that resistivity decreases with increasing in temperature, indicating that the samples are semiconducting in nature.

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