

Summary and Conclusions:-

In this work studies on the structural, electrical and the effect of γ - irradiation on the $\text{ZnCo}_{0.9}\text{M}_{0.1}\text{MnO}_4$ [$\text{M} = \text{Cr}$ and Fe] and $\text{ZnM}_{0.9}\text{Cr}_{0.1}\text{MnO}_4$ [$\text{M} = \text{Ni}$ and V] ferrites have been carried out . The microstructure, phases, electrical (DC and AC) properties of the prepared samples are reported to provide more information to understand the correlation between the physical properties of the magnetite and their chemical composition.

Therefore, the work carried out on this thesis presented in five main chapters.

The first chapter, includes the general introduction, which contains literature survey about the investigated samples and the aim of work.

The second chapter, shows the experimental methods including the preparation of $\text{ZnCo}_{0.9}\text{Cr}_{0.1}\text{MnO}_4$, $\text{ZnNi}_{0.9}\text{Cr}_{0.1}\text{MnO}_4$, $\text{ZnCo}_{0.9}\text{Fe}_{0.1}\text{MnO}_4$ and $\text{ZnV}_{0.9}\text{Cr}_{0.1}\text{MnO}_4$ spinels by convential ceramic method . This chapter includes also the techniques used in the investigation of samples (XRD, FT-IR, DTA and SEM) and in measuring the density, electrical properties and gamma- irradiation.

The third chapter, describes the theoretical and calculation methods used for analyzing the results (DC-, AC- conductivity) , dielectrical constant (ϵ') , dielectrical loss (ϵ'') parameters .

The fourth chapter, shows the thermal stability and characterization of the investigated samples where the results were reported and discussed.

The fifth chapter, deals with the study of DC - and AC - conductivity of $\text{ZnCo}_{0.9}\text{Cr}_{0.1}\text{MnO}_4$, $\text{ZnNi}_{0.9}\text{Cr}_{0.1}\text{MnO}_4$, $\text{ZnCo}_{0.9}\text{Fe}_{0.1}\text{MnO}_4$ and

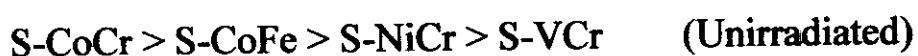
$\text{ZnV}_{0.9}\text{Cr}_{0.1}\text{MnO}_4$ samples at a temperature range from room temperature to 773 °K and frequency of (100Hz- 1MHz) . It includes also the results of the relative permittivity (ϵ') and the dielectrical loss (ϵ'') of the samples under investigation. The measurements were carried out at a temperature rang from room temperature to 773°K and frequency of (100Hz- 1MHz).

The main conclusions are the following:-

- (1) DTA – thermograms show that all samples exhibit thermal peaks at temperatures depend on the sample composition. These peaks may be due to either some phase transition occurring in reacting material or due to some dissociation of some products formed during the thermal treatment.
- (2) IR-spectra indicated that the IR absorption bands are slightly shifted after γ – irradiation.
- (3) IR-spectra of different samples are discussed on the basis that the observed changes in IR peaks can be attributed to the peculiar cation distribution revealed by XRD in the studied samples.
- (4) SEM analysis reveals that the morphology of the sample depends on their chemical composition and irradiated samples show to a some extent a surface morphology differs from that of unirradiated one. Moreover, the pores sizes became smaller after γ –irradiation process.
- (5) SEM of S-CoCr, S*-CoCr samples seems to consist of two different crystallites, one is almost of a rod shape morphology and the others are spherical particles. In case of S-NiCr, S*-NiCr agglomerated ferrite particles consisting of spherical and plate crystallites are observed. SEM of S-CoFe, S*-CoFe sample, reveals that the surface morphology is homogeneous where the particles look alike. SEM analysis of S-VCr, S*-VCr sample, reveals

that the individual particles are always 2 μm spheres that are often agglomerates.

- (6) SEM results indicate that the mean grain sizes of investigated samples lie between 3.7 and 2.4 μm .
- (7) SEM indicates that the average grain size and/or the microstructure of investigated ferrites depends on the chemical composition.
- (8) XRD showed that no trace of an impurity phase of starting materials is observable in the present patterns, indicating the formation of single-phase cubic Spinel compounds.
- (9) XRD indicated that octahedral bond length more strongly influences the lattice parameter than does the tetrahedral bond length. The change in bond distances has also introduced a change in tetrahedral and octahedral volumes in the unit cell.
- (10) The grain size has been determined via scanning electron microscopy (SEM) and the crystallite size using X-ray diffraction. The X-ray measurements showed smaller sizes than the SEM studies; this is due to the presence of non-crystalline material at the particle surface. Both techniques showed that the mean grain sizes of investigated samples lie between 3.7 and 2.4 μm .
- (11) The electrical properties of the investigated samples are found to be dependent on the distribution of the iron and the divalent metal ions among the octahedral and tetrahedral sites of the Spinel lattice.
- (12) The DC-conductivity obtained at a temperature range between 300 – 773 °K showed that the electrical conductivity increases according to the following order:



(13) The observed change in DC electrical conductivity with the change in sample composition may be attributed to a large extent to the change occurring in the M^{2+}/M^{3+} ratio in B sites.

(14) The value of activation energy ranges from 0.02 to 1.25 eV.

The observed change in activation energy with the change in the sample composition is based on the changes in ionic distances in the Spinel ferrite crystal structure.

(15) The temperature dependence of DC- electrical conductivity for γ -irradiated samples showed similar behavior to that of the unirradiated ones.

(16) The γ -irradiated samples showed an increase in the activation energy values and a decrease in the electrical conductivity as compared with that found for the unirradiated ones.

(17) The AC- conductivity of all the investigated samples showed almost similar behavior in which the conductivity increased with increasing temperature. This may be attributed to the increase in the drift mobility and hopping frequency of charge carriers with increasing temperature.

(18) The AC- conductivity of all the samples shows a semi-conducting trend, as commonly seen in most ferrites. The AC- conductivity, in most cases, $\sigma_{a.c}$ is found to be slightly frequency dependent at lower frequencies, but increase at the higher frequency. The dispersion of the AC conductivity with frequency was also found to decrease with increasing the temperature. The activation energy is also found to decrease with increasing frequency for the studied ferrites.

(19) The difference between the activation energies of $\sigma_{d.c}$ and $\sigma_{a.c}$ may be attributed to the effective drop of the electric field within the bulk due

Summary and Conclusions

to the presence of space charge accumulations at the electrodes which were noticed in DC –measurements.

- (20) The relaxation intensity ($\Delta \epsilon'$) is slightly effected at lower frequencies, but increases at higher frequencies with increasing the temperature for all compositions.
- (21) It was found that the dielectric constant value (ϵ') decrease with increasing the testing frequency.
- (22) The (ϵ') values for irradiated samples are higher than that of unirradiated ones. The observed increase in (ϵ') values by irradiation may be attributed to a gradual formation of more charge carriers or easily orientable dipolar molecules that are capable of conducting the electric current.
- (23) The dielectric loss factor (ϵ'') increases with increasing temperature at all frequencies. The increase in (ϵ'') with temperature in these samples is due to the relaxation of the dipole molecules in cooperation with the resulting drop in the relaxation time.
- (24) Irradiation effect of all γ -irradiated samples showed the same behavior as that of unirradiated ones ,but with a slight decrease in the values of both the dielectrical constant and the dielectrical loss than that found for unirradiated ones .
- (25) Dependence of σ , ϵ' and ϵ'' on composition of the sample at room temperature and frequency of 10^3 Hz showed the following order:

$$S-CoCr > S-CoFe > S-NiCr > S-VCr$$