ORIGINAL ARTICLE

Impact of variation in synthesis parameter on dielectric properties of Ni-Zn spinel ferrite.

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ABSTRACT

Nickel – Zinc ferrite Ni_{0.65}Zn_{0.35}Fe₂O₄ was prepared by the sol -gel auto combustion method using L-ascorbic acid as fuel. The ferrite samples were synthesized with different pH values 7, 8, 9 and 10.These synthesized powder has been characterized by XRD and effect of pH values on the dielectric properties of the ferrite sample were studied. XRD pattern confirmed that samples were crystalline in nature. The dielectric properties of all the samples such as dielectric constant ($\dot{\epsilon}$), dielectric loss ($\dot{\epsilon}'$) and dielectric tangent(tan δ) were obtained by measuring the capacitance of the nanoparticles of the pellet form at room temperature using LCR-Q meter bridge in the frequency range 10 KHz-5 MHz. The dielectric constant, dielectric loss and dielectric tangent all decrease with frequency for all the values of pH. The dielectric constant decreases as pH increases from 7 to 10.

Keywords: Sol-gel auto combustion, XRD, dielectric properties.

INTRODUCTION

Ferromagnetic material are especially ferrites, are magnetic ceramic consisting of iron oxides and metal oxides having potential applications for making many electrical, electronic and other devices such as permanent magnets, memory devices, microwave devices etc.[1].

Properties of spinel ferrites are extremely sensitive to synthesis methods and synthesis conditions. Magnetic nanoparticles of spinel ferrites have been the focus of an increasing the amount of the recent literature, which has chronicled research in to the both synthesis and applications. In the recent years, it is found that the properties of nano ferrites are interesting, unusual and superior to their bulk counterparts. In addition, the nano size spinel ferrites find applications in magnetic drug delivery, hyperthermia, as catalyst, as sensor etc.[2].Nickel ferrite is a unique ferrites has inverse spinel structure and possesses good electrical ,dialectical and magnetic properties and in nano size it exhibits enhanced magnetic as well as electrical properties.

The high surface-to-volume ratio of nanoparticles causes the surface energy to dominate in various properties. Significant enhancement in chemical activity in catalyst field is well known [3].

The nickel zinc ferrites properties are also dependent on the methodology adopted for their synthesis, preparative conditions like pH of solution, sintering temperature and time, chemical composition, grain size. In the present work we have prepared the nickel zinc ferrite powder by sol gel auto combustion method. The main objective is to study the effect values of variation of pH on dielectric properties

METHODOLOGY

Material required

Nickel Nitrate, Zinc Nitrate, Iron Nitrate, L-Ascorbic acid, Ammonia solution

Preparation of NZF nanoparticles

Sol –gel auto combustion method was employed to synthesize Ni-Zn ferrite having composition Ni_{0.65}Zn0.35Fe2O4.Metal nitrates of constituent ions i.e. nickel nitrate, zinc nitrate, and ferric nitrate of AR grade supplied by Merck were used as a raw material'sascorbic acid was used as fuel. The metal nitrate to fuel ratio was taken to be 1:3.All nitrates and L- ascorbic acid were first dissolved in minimum amount of double distilled water and stirred for 30 minutes and then mixed together. This initial solution was highly acidic. Ammonia was added to adjust different pH (7, 8, 9, and 10).The mixed solution was kept onto a hot plate with continuous stirring at 750 to get a dense sticky gel. After gel formation, the temperature was increased to 110 °C for the dehydration process. The temperature was then increased rapidly and when it reached approximately to 120 °C, large amount of gases

 (CO_2, H_2O, N_2) were liberated, and a dark brown ferrite power was produced through the combustion process. Finally the burnt power was calcined at 500 °C for 6 hrs. with rate of 5 °C/min to obtain the Ni-Zn ferrite nanoparticles

RESULTS AND DISCUSSION

X-RAY DIFRACTION STUDY

Lattice parameter (a), X-ray density (d_x), Unit cell volume (V), and Crystallite size (t) were calculated from XRD data for all samples [4-7]. Particle size of the nanoparticles were about 22, 24, 28 and32 nm respectively. The average crystallite size is increased as the pH increases. Due to rapid combustion rate and high flame temperature with increasing pH, higher pH produces larger crystallite size and good crystallinity There is slight changes were observed for lattice constant which could be associated with the pH solution. The X-ray density depends on molecular weights of samples and lattice constant, therefore same nature as that of lattice constant was observed for X-ray density values with increase in pH of samples.

The value of volume of unit cell depends upon lattice constant so it also have same natures as that of lattice constant with increase in pH of samples All these obtained values are tabulated in Table 1.

DIELECTRIC PROPERTIES

The dielectric properties of NZP for different pH values (pH-7, 8, 9 and 10) of prepared samples were measured using LCR-Q meter.

Dielectric constant (έ) was calculated by using relation [8]

$$\dot{\varepsilon} = \frac{Cd}{\varepsilon_{0A}}$$

Dielectric loss (ϵ'') was calculated by using relation [9];

 $\tilde{\epsilon} = \dot{\epsilon} \tan \delta$

Dielectric loss tangent (tan δ) was calculated by using relation [10];

$$\tan \delta = \dot{\epsilon} / \ddot{\epsilon}$$

Where C is the capacitance of the pellet in farad is the thickness, A is the cross –sectional area of the flat surface of the pellet and ϵ_0 is the constant of permittivity for free space.

Table 1: Lattice parameter (a), X-ray density (d_x) , Unit cell volume (V) and particle size (t) of Ni_{0.65}Zn_{0.35}Fe₂O₄ nanoparticles (pH-7, 8, 9 and 10).

'pH' maintain at	A(Å)	d _x (gm/cm ³)	$V(Å^3)$	Particle size(t)
pH-7	8.3640	5.3748	585.557	22
pH-8	8.3678	5.3671	586.334	24
pH-9	8.3603	5.3825	584.759	28
pH-10	8.3667	5.3690	586.103	32



Fig. 1: Variation of dielectric constant ($\dot{\epsilon}$) with frequency at room temperature of Ni_{0.65}Zn_{0.35}Fe₂O₄ nanoparticles for (pH=7 to 10).



Fig.2: Variation of dielectric loss ($\dot{\epsilon}'$) with frequency at room temperature of Ni_{0.65}Zn_{0.35}Fe₂O₄ nanoparticles for (pH=7 to 10).



Fig.3: Variation of dielectric loss tangent (tan δ) with frequency at room temperature of Ni_{0.65}Zn_{0.35}Fe₂O₄ nanoparticles for (pH=7 to 10).

Dielectric dispersion in ferrites can be explained on the basis of space charge polarization, which is a result of the presence of higher conductivity phases (grains) in the insulating matrix (grain boundaries) of a dielectric causing localized accumulation of charge under the influence of an electric field. In order to improve its high frequency performance (such as low dielectric constant and loss). The variation of dielectric constant of the pellets with different pH is shown in Fig1.The dielectric constant decreases with increasing pH value; at the same time, the dielectric loss also decreases as shown in Fig.2.The results may be due to high pH value suppressed the grain growth and caused the grain size to decrease, so that the proportion of the grain boundary was enhanced and this also contributed to the decrease of the dielectric constant. Normally, there is a strong correlation between conduction mechanism and the dielectric behaviour of ferrites. This can be explained by the fact that an increase in frequency induces either weakening or disappearance of ionic polarization, and consequently decreases the dielectric constant values. For the ferrite materials, the dielectric properties will be affected by many factors such as the content of the individual crystalline phase, formation of the phases, level of porosity and degree of densification [11, 12]. In fact, all these factors could rely on the variation of pH values in the solution. [13]. Higher porosity and lower density result in lower dielectric constant. However, it is also observed that all the samples show no significant frequency dependent phenomenon, i.e. the permittivity maintains an almost constant value at higher frequency. It is observed that the dielectric constant and dielectric loss decreases with increase in the pH value, which can be ascribed to the formed higher porosity results in lower dielectric constant and dielectric loss. The variation of dielectric loss tangent (tan δ) of the pellets with different pH is shown in Fig.3. Low dielectric loss tangent is desirable for reducing heat generation at high frequency.

CONCLUSION

Nanocrystalline Ni-Zn ferrite has been synthesized at different pH via Sol-gel auto combustion method, which is characterized on nanosized shape etc. by X-Ray diffraction. The particle size is in the range of 22-32 nm. The prepared Ni-Zn samples annealed at temperature 500 °C for 2 hr is formed a good crystalline cubic phase spinel structure. The dielectric constant, dielectric loss and dielectric loss tangent all decreases with frequency for all the values of pH. The dielectric constant and other related parameters decreases as pH increases from 7 to 10. The saturation magnetization obtained from M-H plot show decreases with increase in pH.

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