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Effect of Chemically Treated Ferric Nitrate: its Dielectric Studies of Sisal Fiber Composite

KEYWORDS: Fe2O3, sintering method, dielectric measurement

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ABSTRACT *Iron oxide synthesized through sintering route. The present research work deals with ferrite composite prepared using chemical reactions. Ferric nitrates and ammonium chloride doped with sisal fiber has been prepared. The comparative studies of ferric oxide were examined through dielectric measurement.*

1. Introduction

Iron oxides are chemical compounds composed of iron and oxygen. All together, there are sixteen known iron oxides and oxyhydroxides. [1] Iron (III) oxide or ferric oxide is the inorganic compound with the formula Fe2O3. The steelmaking by-products such as dust and mill scale, very rich in iron (≈ 72% Fe), are currently produced in large quantities and represent a potential of almost 5 million tons in the world [2]. Powder metallurgy comprises a set of processes of forming having for common denominator a raw material in a powder form. The reduced iron powder is the most widely used material in powder metallurgy industry.

LiFeO2 has various crystalline structures such as α- LiFeO2, β-LiFeO2, γ-LiFeO2, Layered LiFeO2, Corrugated LiFeO2, Goethite type LiFeO2 etc. The crystalline structure of LiFeO2 depends mainly on the preparation methods. Many researches prepared LiFeO2 with different structures. V.R. Galakhov et al. prepared α-LiFeO2 with Fm-3m space group by using solid state reaction and M. Tabuchi et al. prepared α-LiFeO2 with Fm3m space group by hydrothermal synthesis [3-5]. Similarly, β-LiFeO2, γ- LiFeO2 and layered LiFeO2 are prepared by hydrothermal synthesis and other methods. Corrugated LiFeO2 and Goethite type LiFeO2 are prepared by ion exchange method. In comparison with the conventional solid phase synthesis methods, hydrothermal method is one of the simplest and best methods to prepare lithium based cathode materials [6-9]. In case of the electrical properties of the oxides, grain boundaries play an important role. The measurement of conductivity and permittivity shows dispersion behavior which offers an opportunity to gain some information of ionic migration process. Considering the significance, the electrical conductivity studies on various lithium-based oxides such as LiCoO2, LiCeO2, LiSmO2, Li2SnO3, Li2MnO3, LiMn2O4, and Li2V2O5, and others have been reported in the literature [10-13]. However, to the best of our knowledge, there are meager reports on electrical and dielectric properties of LiFeO2. A detailed study on the temperature and frequency depended electrical properties is necessary to understand the conduction mechanism in LiFeO2 for effective utilization as cathode material in the fabrication of lithium ion batteries.

In the oxides of iron a-Fe203 is the most stable compound. For the non-existence of Fe++ ions a-Fe203 has higher electrical resistivity than other oxides of iron such as Fe304, FeO, and ferrites. It has been reported, however, that at the temperature above 1200°C there is the possibility of the appearance of Fe++ ions in a-Fe203. When the oxides contain ferrous ions, the hopping of electrons between ferrous and ferric ions gives rise to higher conductivity. Thus for samples possessing both the conductive and less-conductive phases the Maxwell-Wagner interfacial polarizations are observed. With the surface modified by the use of mild reducing condition of sintering Hirbon reported the interfacial polarization in the sintered compacts of a-Fe203. On the other hand, in a-Fe203 containing other ions of different valences such as Ti" ions polarizations due to permanent dipoles of Fe++_Fe+++ induced by Ti+4 ions were observed at very low temperature [14-18]

2. Material and method

2.1Chemical treatment of fiber

Ferric Nitrate (Fe (NO3)3.9H2O) and ammonium chloride (NH4Cl) was taken in the ratio 10:4 in 500 ml of distilled water. The mixture was stirred till a homogenous solution was obtained. In this mixture 10g of sisal fiber was added and then 1:1 solution of NH4OH (liquid ammonia) was added to it then left the solution for one hour. Again the mixture thus obtained was dried and then annealed in muffle furnace at 1000oC and kept it at that temperature for 15 min.

The reaction may take place in this way

2Fe (NO3)3.9H2O +3NH4Cl+3NH4OH+ Fiber→ Fe2O3: fiber + 6NH4NO3 + 3HOCl + 18H2O

When ferric nitrate reacts with ammonium chloride and ammonium hydroxide along with sisal fiber at 1000oC with ammonium nitrate and HO-Cl (hypoclorous acid) decomposed at such high temperature and only ferric oxide is left.

2.2 Nature and structure of sample after firing

The material formed was found to in solid crystals in physical appearance. The sample appeared in powder form and it is like Cole in color.

3. Result and discussion

The electrical properties of the insulating material Fe2O3 composite were measured by impedance analyzer these dielectric measurement of Fe2O3 composite doped with sisal fiber shown in fig 1-3 and 4-6. In the Fig 1 represents

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the graph between frequency and tanδ and fig 2 shows the comparison between frequency and ε.

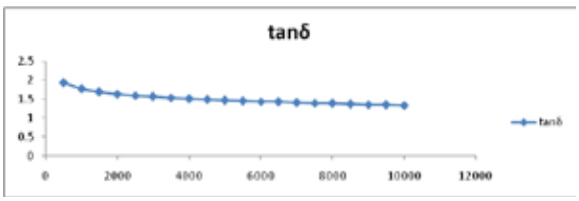

Fig 1

The dielectric constant ε and loss tanδ of Fe2O3 at room temperature 30oC are measured to be 1.9 to 1.3 respectively and are found to decrease with the increase in the frequency. The value of ε and tanδ are found to different with each other. In fig 1 the variation of dielectric constant at different frequencies with room temperature 30oC for fe2O3 is shows that it decreases considerably with increase in frequency.

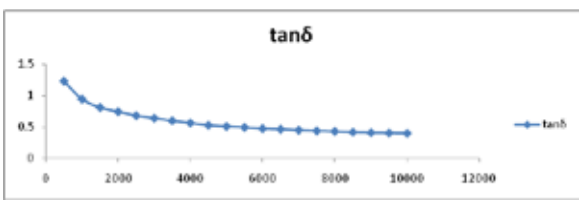

Fig 2

This dielectric dispersion is attributed to the Maxwell and Wagner type of interfacial polarization in agreement with Koop's phenomenological theory [19]. Since polarization decreases with increasing frequency and reaches constant values, a decrease in dielectric constant with frequency is observed.

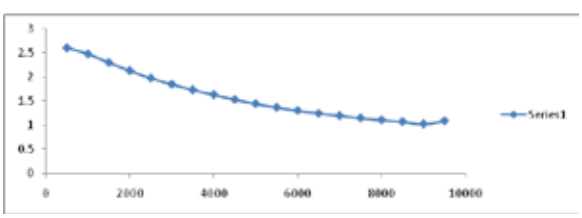

Fig 3

At lower frequencies, dielectric loss tanδ is large and it decreases with increasing frequency. The tanδ is the energy dissipation in the dielectric system, which is proportional to the imaginary part of the dielectric constant. An increase in loss factor at higher frequencies may be due to the series resistance of the electrodes, leads, etc [20-22].

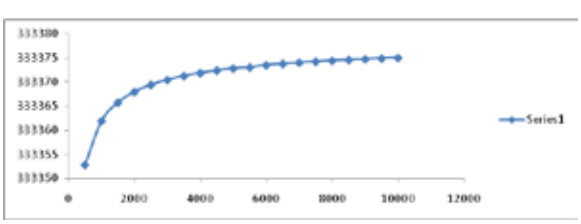

Fig 4

While in fig 4, 5 & 6 is a comparison plot of variation of dielectric constant with different frequency at constant temperature shows an increase considerably with increase in frequency. It was observed from these figures that the dielectric constant increases continuously with increase in frequency for all the samples, followed with a frequency independent behavior. This is a normal behavior for high density, fine chemically homogeneous ferromagnetic material.

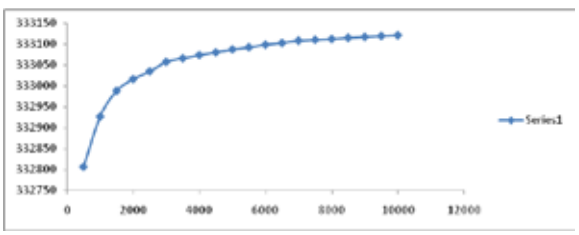

Fig 5

The dielectric properties of ferrites are dependent upon several factors, including the method of preparation, chemical composition and grain structure, size and natural fiber (sisal fiber) and it's contains cellulose, hemicelluloses, lignin and pectin. The observed dielectric behavior of our samples may also be due to the particle size effect and is also in concurrence with observation made by other Investigators [23-24]. The effect of electrical homogeneity fine grain structure and shape of the ferrite samples affect the dielectric properties [25]. It also presence the α impurities contributes towards the change in dielectric properties. The dielectric constant of 25-30 at 10 KHz and an increasing trend is observed at higher frequencies. This is normal behavior for high density fine chemically homogenous ferromagnetic material [26].

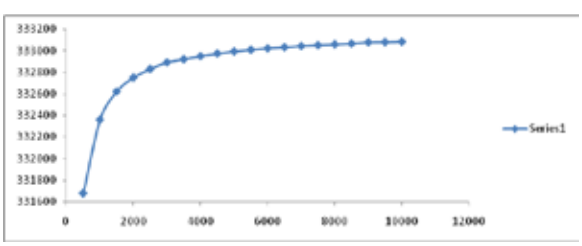

Fig 6

The electronic exchange between ferrous and ferric ions didn't follow the path of frequency which is externally applied alternating beyond the critical frequency value. The dielectric behavior of samples which shown in graphs is because of its particle size effect [27]. If ε remains unaffected with increase in temperature then frequency also remains the same, which means that the local carriers are immobile. It is understood that the local charge carriers are immobile and the thermal activation in negligible. This further suggests that the samples possess a high chemical homogeneity and fine grain distribution. The presence of moisture, cellulose, hemicelluloses, lignin and pectin is also indicated by the electrical conductivity results. It is reported the presence of moisture plays an important role in the formation and stabilization of Fe2O3 [28].

Conclusion

The effect of chemical homogeneity, fine grain structure, particle size and shape of the ferrite samples are understood to affect the properties of dielectric behavior. The presence of moisture, cellulose, hemicelluloses, lignin and pectin also contribute towards changes in dielectric properties.

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