

SYNTHESIS AND CHARACTERIZATION OF CrO-ZnO NANOCOMPOSITES**R. Ramesh¹, M. Vaitheeswari² M.J. Uma³, K.U. Madhu⁴**

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ABSTRACT

Nanotechnology has come to mean a range of highly promising disciplines in science and technology. The essence of nanoscience and nanotechnology is the ability to understand, fabricate and engineer materials, devices and systems in the nanometer scale. In our present work, we have reported the preparation and characterization of CrO-ZnO nanocomposites by solvothermal method. Microwave heating which allows a considerable reduction of the time, as been used in organic chemistry for several decades. The lattice parameter values of CrO-ZnO nanocomposites were determined by PXRD measurements. The PXRD and AC conductivity analysis were reported.

Keywords : CrO-ZnO, nanocomposites, solvothermal, PXRD, AC Electrical parameters

I. Introduction :

A nanocomposite combines two or more materials – of which at least one is a nanomaterial with different physical and chemical properties. Nanocomposite materials are designed to exhibit properties that exceed, sometimes drastically, the capabilities of the sum of their constituent parts. Nanocomposites are made by embedding materials called the reinforcing phase into another material called the matrix phase. Either one or both phases can be nanomaterials. Nanocomposites are currently being used in a number of fields and new applications are being continuously developed. Applications for nanocomposites include:

- Thin-film capacitors for computer chips
- Solid polymer electrolytes for batteries.
- Automotive engine parts and fuel tanks
- Impellers and blades
- Oxygen and gas barriers
- Food packaging

Nanocomposites are those composites in which one phase has nanoscale morphology like nanoparticles, nanotubes, or lamellar nanostructure. They have multiphases, so are multiphase materials, at least of the phases should have dimensions in the range of 10–100 nm. To overcome the limitation of different engineering materials now-a-days, nanocomposites are emerged to provide beneficial alternatives. Nanocomposites can be classified on the basis of their dispersed matrix and dispersed phase materials [1].

Nanocomposites are materials that have a solid structure in which the distance between the phases is leastwise formed of a dimension with nanoscale size and general form of an inorganic matrix set in the organic phase, or vice versa, from an organic matrix set in the inorganic phase. Zinc oxide is an inorganic compound with the formula ZnO. ZnO is a white powder that is insoluble in water and it is widely used as an additive in numerous materials and products including rubbers, plastics, ceramics, glass, cement lubricants paints, orient, adhesives, sea lands, pigments, foods, batteries, ferrimarcetes, and first-aid tapes. Although it occurs naturally as the mineral zincite most zinc oxide is produced synthetically [2].

Zinc oxide forms cement like products when mixed with a strong aqueous solution of zinc chloride and these are best described as zinc hydroxyl chlorides [3]. This cement is used in density. When ointment containing ZnO and water are melted and exposed to ultraviolet light, hydrogen peroxide is produced [4]. ZnO crystallizes in two forms, hexagonal wurtzite and cubic zinc blende [5]. The high heat capacity and heat conductivity low thermal expansion and heigh melting temperature of ZnO are beneficial for ceramics [6]. ZnO exhibits a very long-lived optical phonon E2 (low) with a lifetime as high as 133 ps at 10k[7]. In this thesis, we have attempted to investigate the structural of pure zinc, chromium and chromium zinc oxide nanocomposites. Simple microwave assisted solvothermal method was used for the preparation of samples. The prepared samples were characterized by PXRD and AC conductivity measurements. The results were reported and discussed here. The solvothermal method is analogous to the hydrothermal method, except the organic solvents, instead of water, are used in the synthetic procedure. Nuclei formation and growth are the two key steps in the solvothermal synthesis, which can be manipulated by varying some experimental factors, and therefore controlling the properties of the crystals [8].

2. Preparation Techniques:

The preparation of pure CrO nano particles 1:3 molecular ratio of chromium acetate and urea were taken and dissolved seperately in 100 ml Ethylene Glycol. The stirring condition of chromium acetate, urea solution was added and the resultant solution were kept in a microwave oven. The microwave oven irradiation were carried out until the solvent gets evaporated completely and the colloidal precipitate were formed. This colloidal precipitate were washed several times with double distilled water and then with acetone to remove any organic impurities if present the washed samples were dried in atmospheric air and collected as the yield. The collected samples were annealed at 600°C for one hour. The same procedure was carried out by the preparation of ZnO nanoparticles by using the materials were zinc acetate, urea and ethylene glycol as solvent.

The weight of the substance were measured using the relation,

$$W = \frac{MXV}{1000}$$

Where,

M - Mass of the reactants

X – Concentration of the Substance

V – Volume of the solvent

The yield percentage were calculated by using the relation,

$$\text{Yield percentage} = \frac{\text{product of mass}}{\text{sum of mass of reactant}} \times 100$$

3. Result And Discussions :

Reaction time, colour and yield percentage

The reaction time, colour and yield percentage of the 3 samples were noted and presented in Table

1. The photographs of the prepared samples were given in the figure 1.

Table 1: The reaction time, yield and colour

Name of the sample	Reaction time (min)	Colour	Yield percentage
/CrO	19	Black	33.7
ZnO	32	White	19.4
CrO-ZnO	13	Ash	23.9



CrO nanoparticles



ZnO nanoparticles



CrO-ZnO nanocomposite

Figure 1: Photographs of the samples

PXRD Analysis

PXRD patterns recorded in the present study for CrO, ZnO and CrO- ZnO nanocomposites and the xrd graphs were shown in figure 2 - 4. In the fig 2, the PXRD pattern shows sharp and intense peaks which confirm the formation of CrO nanoparticles. In the fig 3 the PXRD pattern confirms ZnO nanoparticles which are crystalline in nature and has single phase. The PXRD pattern observed data were indexed by matching with the data available for CrO nanocrystal in the JCPDS

file (38-1479) and the PXRD pattern data were indexed by matching with the data observed for ZnO nanocrystal in the JCPDS file (36-1451).

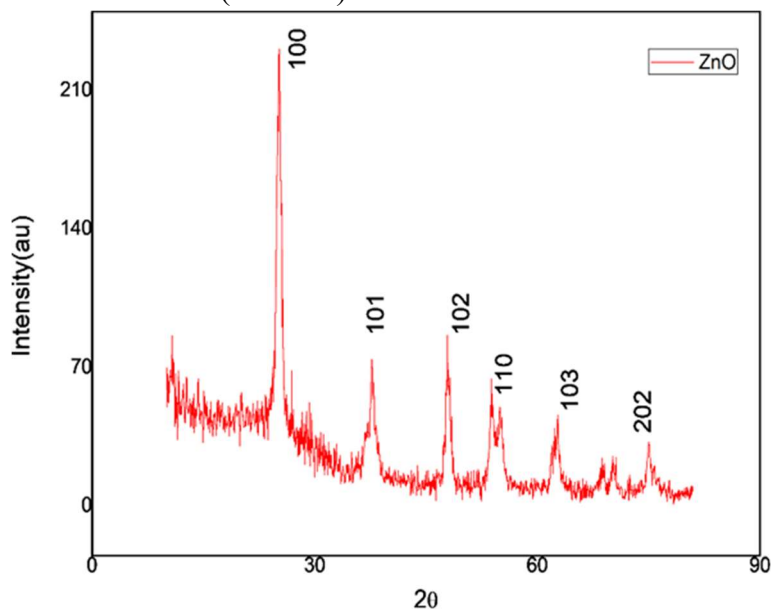


Figure: 2 PXRD pattern for ZnO nanoparticles

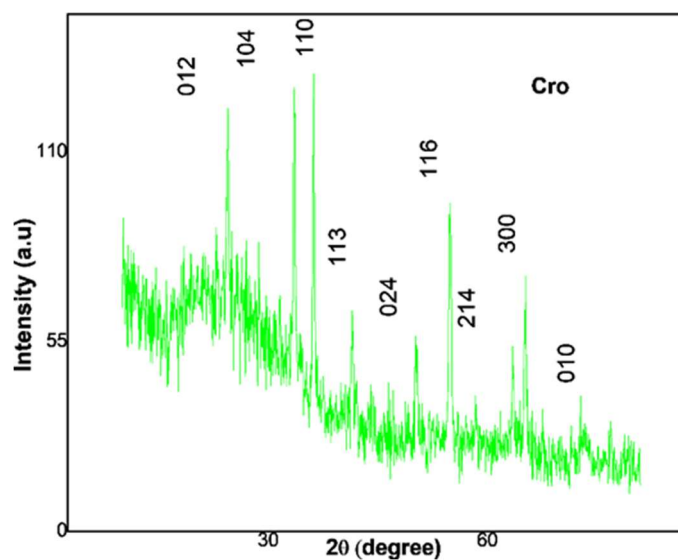


Figure 3 PXRD pattern for CrO nanoparticles

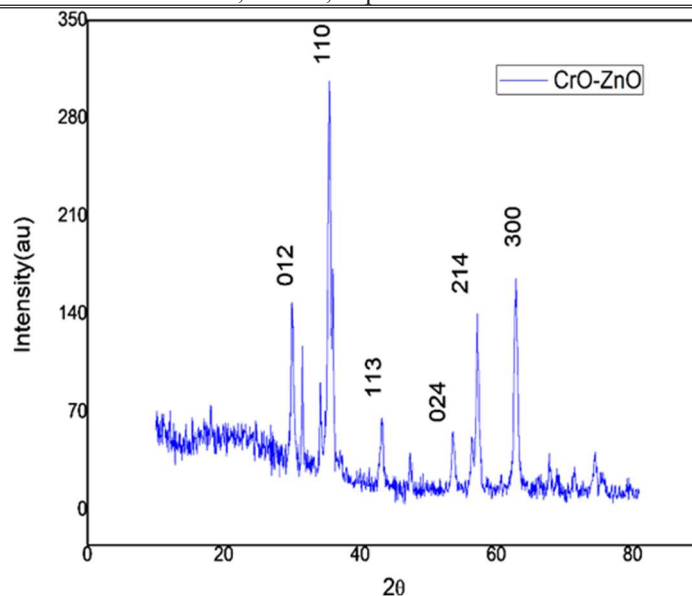


Figure 4: PXRD pattern for CrO-ZnO nanocomposites

From the PXRD pattern of the average crystallite size was determined and tabulated in the Table 2. The average crystallite size was determined by Scherer formula

Table 2 : Particle size of the sample

Name of the sample	Particle size (nm)
CrO	52.8
ZnO	27.7
CrO-ZnO	27.1

4.1 AC CONDUCTIVITY ANALYSIS

In the present work AC conductivity of the prepared samples were taken in the frequencies 100Hz, 1KHz, 10KHz, 100KHz and 1MHz in the temperature 50-150°C. Dielectric constant, dielectric loss and dielectric conductivity with different frequencies varies at different temperatures of CrO, ZnO and CrO-ZnO nanocomposites were shown in the figures (5 - 13). In the figures show the AC electric conductivity is found to be high for higher frequencies at different temperatures which confirms small polaron hopping in the present sample. As the frequencies of the applied field increases hoping of charge carriers also increases thereby increasing the conductivity. AC conductivity of the prepared samples were increases with increasing temperature and frequencies. Dielectric constant and dielectric loss values of the samples were increases with increase in temperature and decrease with increase in frequencies. The result was conforming at that the prepared samples have semiconductive properties. So, the prepared samples were the good semiconductor and have good electrical properties.

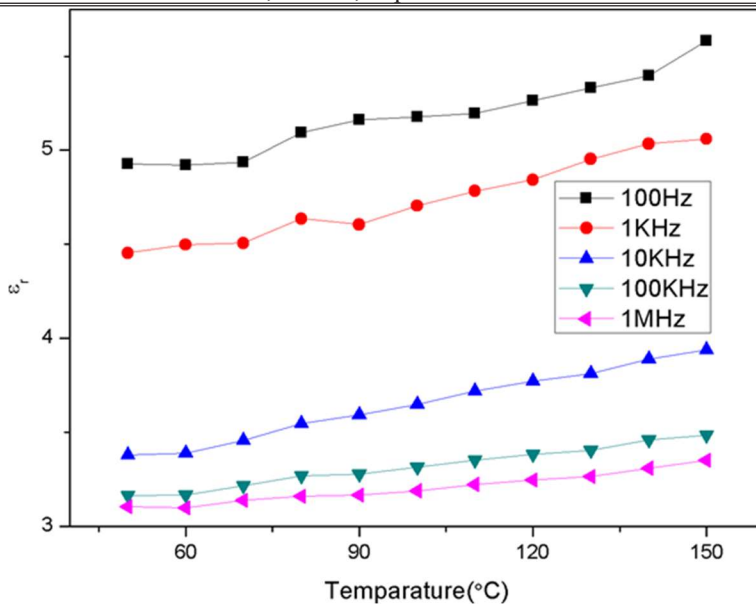


Fig 5 Dielectric constant pattern for ZnO nanoparticles

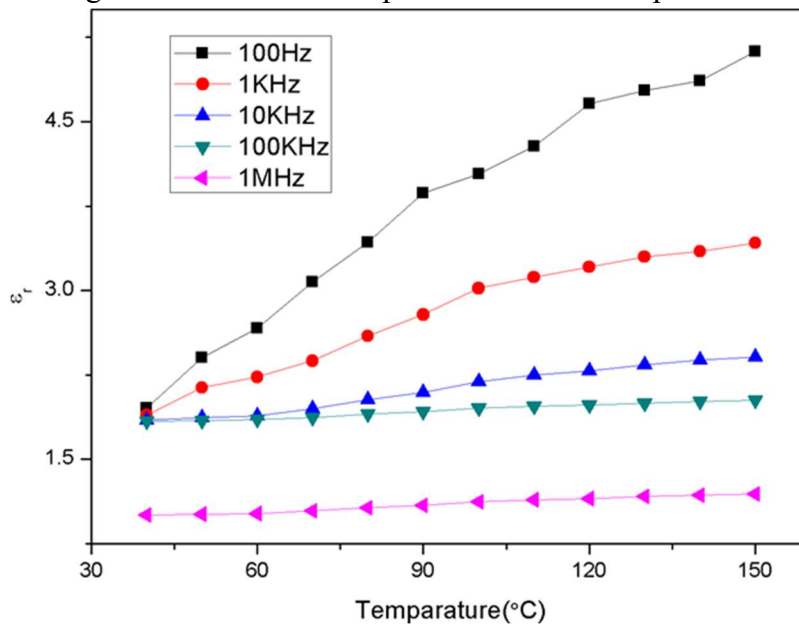


Fig 6 Dielectric constant pattern for CrO nanoparticles

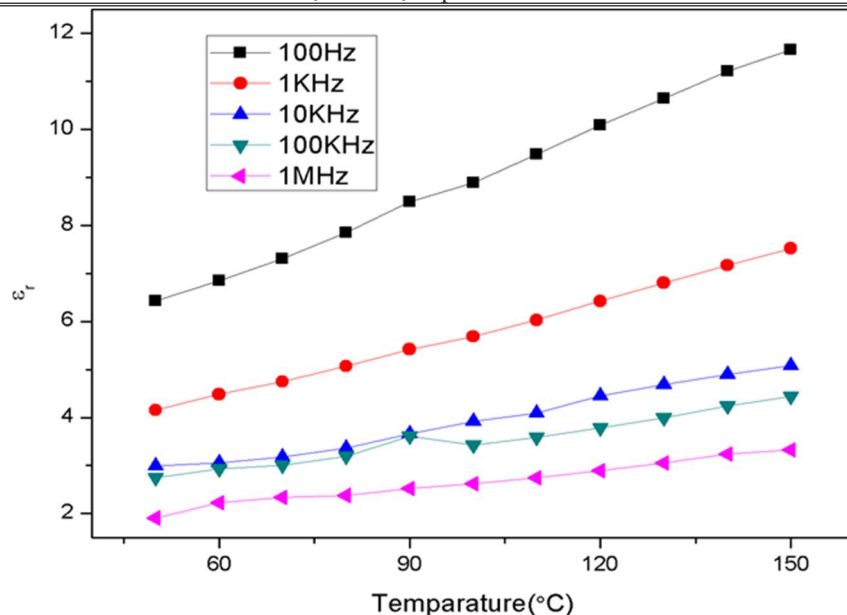


Fig 7 Dielectric constant pattern for CrO-ZnO nanocomposite

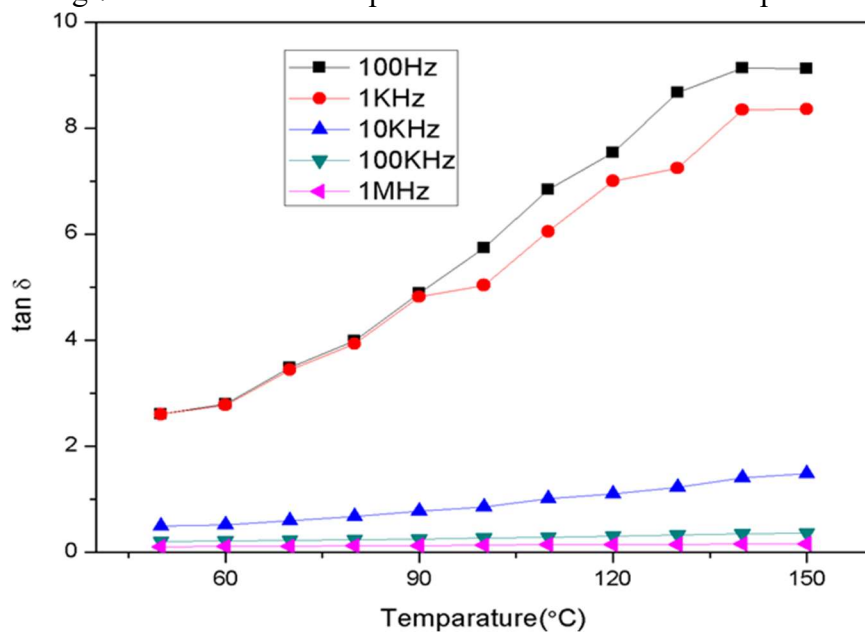


Fig 8 Dielectric loss pattern for ZnO nanoparticle

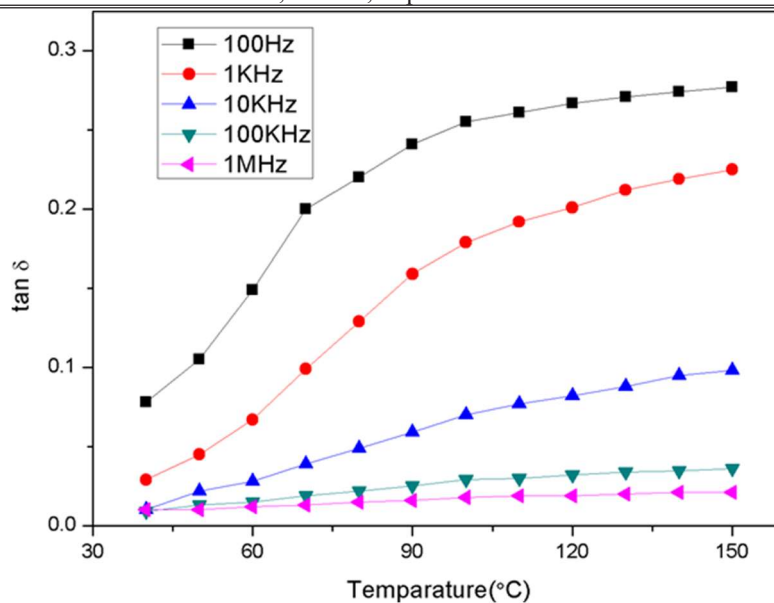


Fig 9 Dielectric loss pattern for CrO nanoparticle

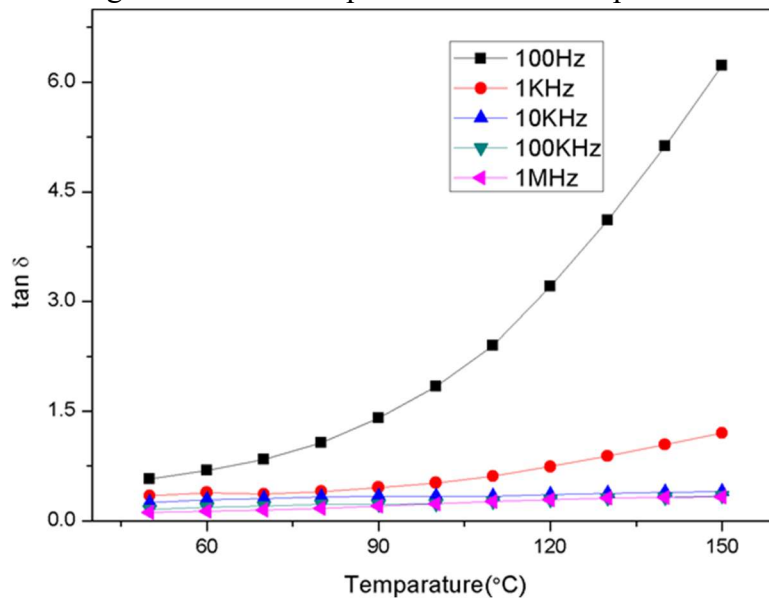


Fig 10 Dielectric loss pattern for CrO-Zno nanocomposite

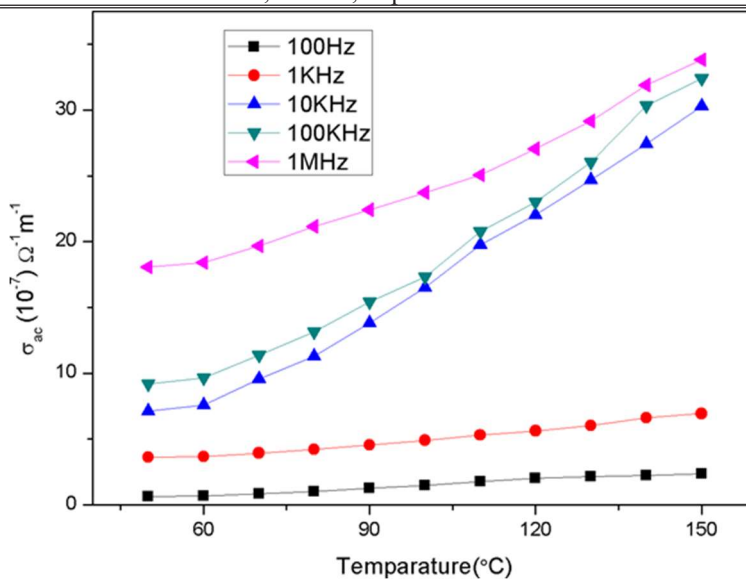


Fig 4.11 AC conductivity pattern for ZnO nanoparticle

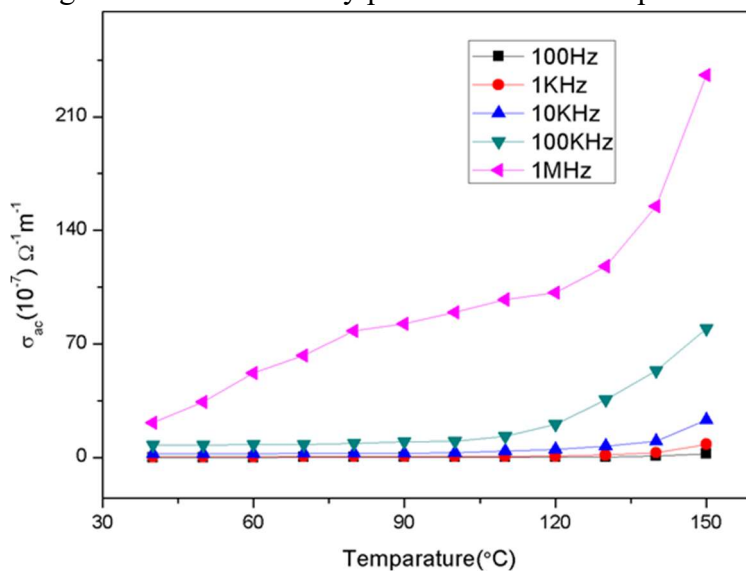


Fig 4.12 AC conductivity pattern for CrO nanoparticle

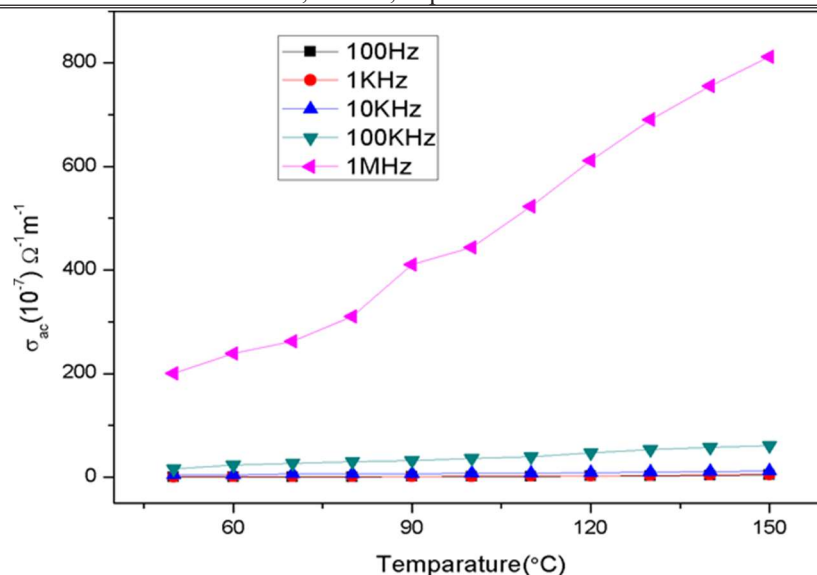


Fig 4.13 AC conductivity pattern for CrO-ZnO nanocomposite

CONCLUSION

Nanotechnology has been widely studied for its potential to advance the field of biotechnology and medical research. The phenomena that take place at dimensions in the nanometre scale are utilised in the design, characterisation, production and application of materials, structure, devices and systems. In the present study, we have successfully synthesized CrO-ZnO nanocomposites by using a simple microwave assisted solvothermal method with a domestic microwave oven. The prepared samples were calcinated and characterized by powder x-ray diffraction (PXRD) carried out to characterize the prepared sample structurally. The AC Conductivity studies were carried out for the prepared pure chromium oxide nanoparticles. Results obtained in the present study indicates that the method adopted is found to be an effective and economical one for preparing the CrO-ZnO nanocomposites with reduced size and homogeneity. AC conductivity of the prepared samples were increases with increasing temperature and frequencies. Dielectric constant and dielectric loss values of the samples were increases with increase in temperature and decrease with increase in frequencies. The result was conforming at that the prepared samples have semi conductive properties. So, the prepared samples were the good semiconductor and have good electrical properties.

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