Title	Characterization of Magnesium Ferrite, $MgFe_2O_4$
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Abstract

Magnesium ferrite, $MgFe_2O_4$, was prepared by solid state reaction method. Structural and vibrational characteristics of the sample were studied by X-ray diffraction (XRD) and Fourier Transform Infrared (FTIR) spectroscopic methods. Morphological features of the sample were investigated by Scanning Electron Microscope (SEM). Simultaneous Thermogravimetric and Differential Thermal Analysis (TG-DTA) measurement was carried out to investigate the high temperature phases of the sample in the temperature range 303 K – 873 K (30°C - 600°C). Electrical conductivities and dielectric constants of the pelletized sample were investigated in the temperature range 300 K - 973 K (27°C - 700°C). It was found that the sample is a thermally high resistance material.

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Introduction

Ferrites have been the emerging focus of recent scientific research and technological point of view [1, 3, 12]. Ferrites have proved to be good in microwave applications because of their low cost, high resistivity and low eddy current losses. Spinel ferrites with the general formula AFe_2O_4 (A = Mg, Mn, Co, Ni, or Zn) are very important magnetic materials because of their interesting magnetic and electrical properties with chemical and thermal stabilities [4 - 6]. Magnesium ferrite ($MgFe_2O_4$) is one of the most important ferrites. It has a cubic structure of normal spinel-type and is a soft magnetic *n*-type semiconducting material, which finds a number of applications in heterogeneous catalysis, adsorption, sensors, and in magnetic technologies [7 - 9]. The excellent combination of electric and dielectric properties of $MgFe_2O_4$ ferrites can be used to fulfill the future demand for high-frequency applications [10]. In this work, Magnesium ferrite ($MgFe_2O_4$) was prepared by solid state reaction method and characterized by X-ray diffraction (XRD), Fourier Transform Infrared (FTIR) Spectroscopy, Scanning Electron Microscopy (SEM), and Thermogravimetric and Differential Thermal Analysis (TG-DTA). Electrical conductivities and dielectric constants of the sample with temperatures were also studied.

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Materials and Method

Preparation of Magnesium Ferrite, $MgFe_2O_4$

Magnesium spinel ferrite, $MgFe_2O_4$, was prepared by solid state reaction method. The starting materials of Analar (AR) grade Magnesium Oxide (MgO) and Ferric Oxide (Fe_2O_3) were used to prepare the sample and as shown in Fig 1(a) and (b). Firstly, the starting materials were weighed with stoichiometric composition. Next, the mixture of the starting materials was ground by an agate motor for 3 h to be homogeneous and to obtain fine grain powders. The powder was then annealed at 1100°C for 22 h in the vacuum chamber (160 mmHg) by using thermal resistive heating coil that controlled DELTA A SERIES temperature controller. The K-type thermocouple was used as the temperature sensor to read-out the real temperature of the sample. Finally, the candidate material of $MgFe_2O_4$ spinel ferrite was obtained. Photographs of experimental setup of sample preparation system, temperature controller and $MgFe_2O_4$ spinel ferrite are shown in Fig 1(c) - (e). Fig 1(f) shows the flow diagram of sample preparation procedure.

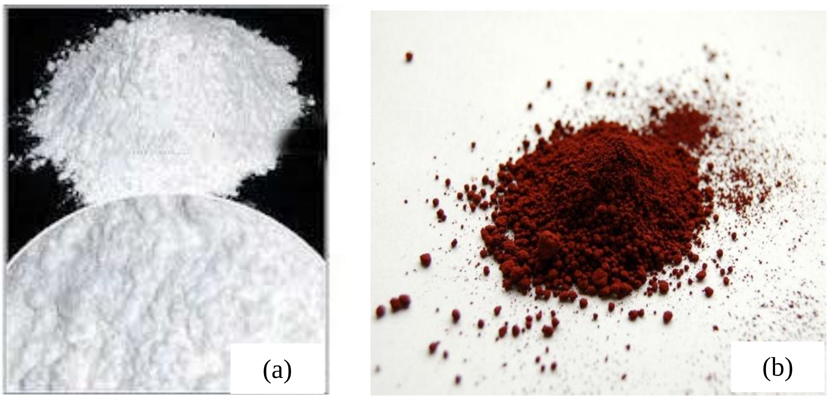

Fig 1 Photographs of the starting materials of (a) MgO and (b) Fe_2O_3

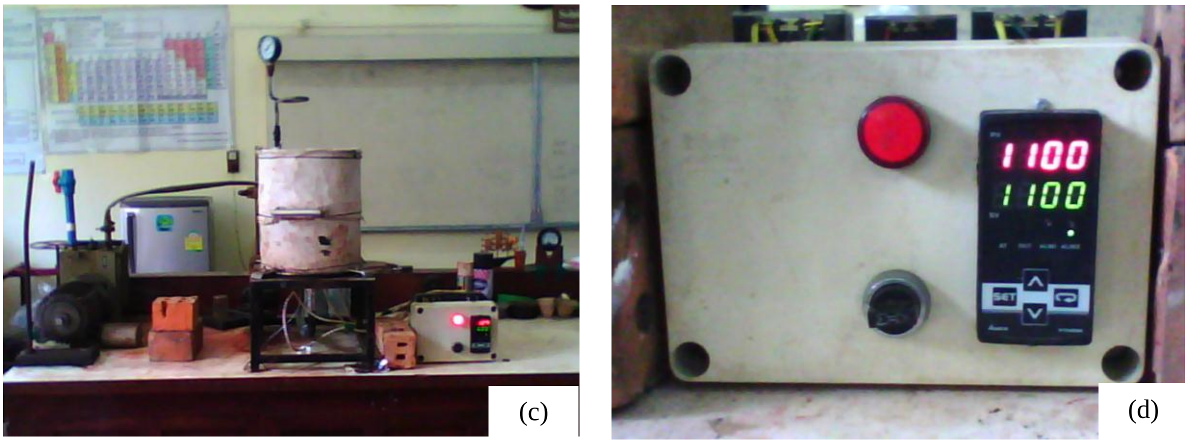

Fig 1(c) experiment setup of sample preparation system and (d) temperature controller

Fig 1(e) Photograph of $MgFe_2O_4$ ferrite

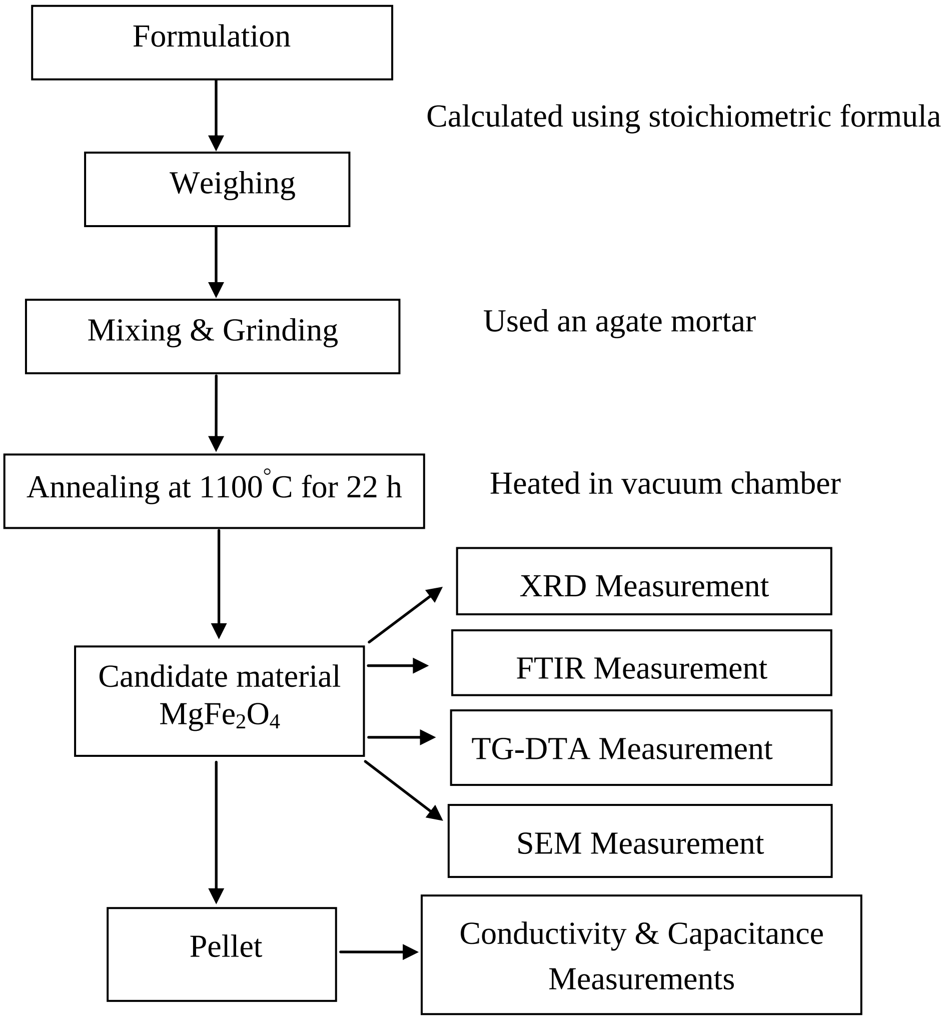

Fig 1(f) Flow diagram of sample preparation procedure

Measurements

Structure analysis of $MgFe_2O_4$ ferrite was investigated by RIGAKU MULTIFLEX X-ray diffractometer using Ni-filter with CuK_α radiation, λ = 1.54056 Å. FTIR transmission spectrum of the sample with Potassium Bromide, KBr pellet method was recorded on PC controlled FTIR-8400 (SHIMADZU) Spectrophotometer. Morphological features of $MgFe_2O_4$ spinel powder were investigated by using JEOL JSM-5610LV SEM with the accelerating voltage of 15 kV, the beam current of 50 mA and 10000 times photo magnification. SHIMADZU DTG-60H Thermal Analyzer was used to investigate the high temperature phases of the sample. Electrical resistances and capacitances of the $MgFe_2O_4$ pallet (1.20 cm in diameter and 0.26 cm in thickness) were observed in the temperature range 300 K – 973 K using CAHO SR-T903 Temperature Controller. Electrical resistances and capacitances of the sample were measured by using FLUKE 45 Dual Display digital multimeter and FUKE DM6013A Digital CAPACITANCE METER. K-type thermocouple was used as the temperature sensor. 300 W heating coil with stainless steel covered was used as the heating element. Photograph showing the experimental setup of electrical resistances and capacitances measurements are shown in Fig 2.

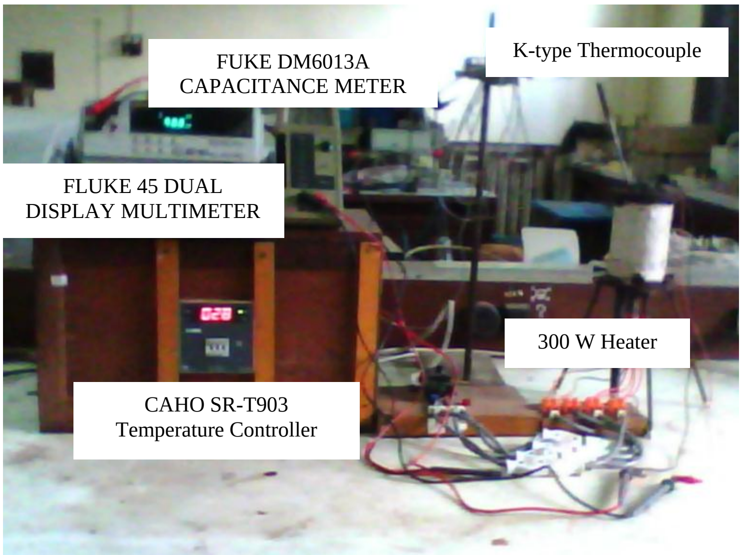

Fig 2 Photograph of the experimental setup of electrical resistance and capacitance measurement

Results and Discussion

Powder X-ray diffraction pattern of $MgFe_2O_4$ ferrite is shown in Fig 3. XRD analysis reveals that the sample belongs to cubic structure and the unit cell parameters of the grown crystal are $a = b = c$ = 6.4355 Å. The crystallite size is obtained as 51.86 nm using the line at 35.75° or (311) of the strongest intensity (I = 100%).

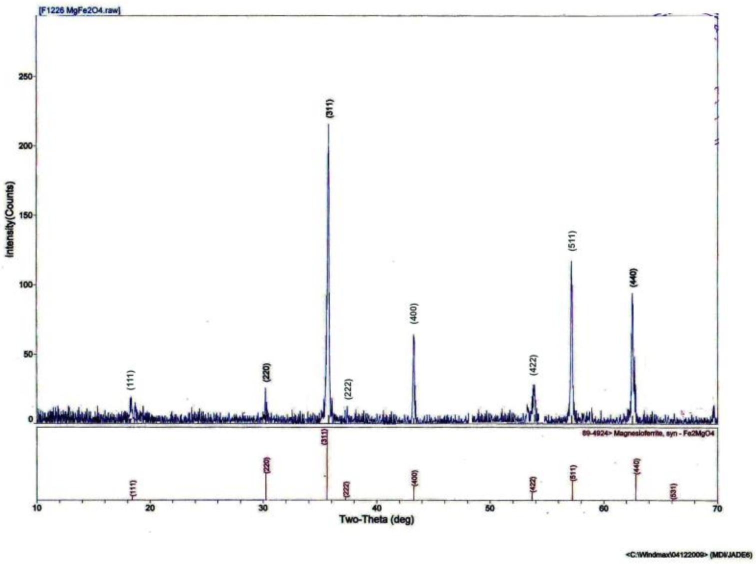

Fig 3 XRD pattern of $MgFe_2O_4$ ferrite

Most of the spectroscopic information deals with the internal modes of isolated vibrating group, there is a growing interest compounds which do not contain such group. The vibrational frequencies (wavenumbers) of Magnesium Ferrite, $MgFe_2O_4$, are mainly 406 cm^{-1} and 433 cm^{-1} for B atoms on octahedral sites and 565 cm^{-1} and 581 cm^{-1} for A atoms on tetrahedral sites [2,11]. Moreover, the wavenumbers of the tetrahedral sites atoms are assigned as ν_1-mode and the octahedral sites atoms are also assigned as ν_2-mode. FTIR transmission spectrum of Magnesium Ferrite, $MgFe_2O_4$, with KBr pellet method is shown in Fig 4. Vibrational characteristics and mode assignments of molecules are tabulated in Table 1.

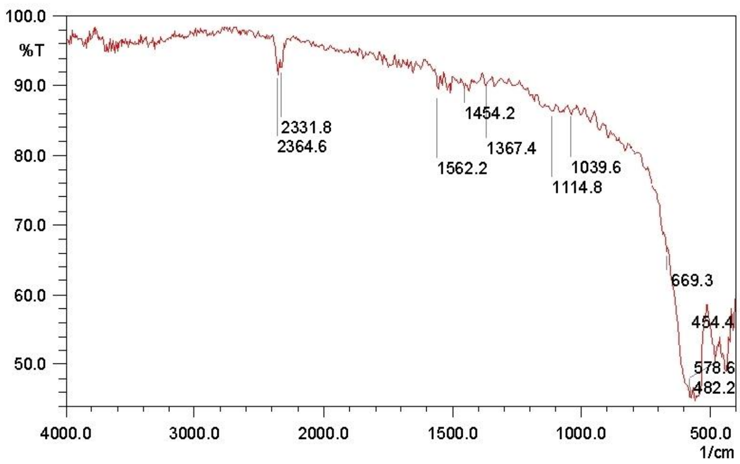

Fig 4 FTIR transmission spectrum of $MgFe_2O_4$ spinel

Table 1 Wavenumbers and vibrational mode assignments of $MgFe_2O_4$ spinel

Line No	Wavenumber (cm^{-1})	Vibrational Characteristics and Mode Assignments	Molecules
1	454, 482	Stretching vibration, ν_2-mode	Octahedral site B atoms in $MgFe_2O_4$
2	579	Stretching vibration, ν_1-mode	Tetrahedral site A atoms in $MgFe_2O_4$
3	669	ν-internal mode	Octahedral site B atoms in $MgFe_2O_4$
4	1040, 1115, 1367, 1454, 1562	Combination band	$MgFe_2O_4$
5	2332, 2365	Bending, ν_2-mode	CO_2

SEM micrograph of $MgFe_2O_4$ sample is shown in Fig 5. Morphological feature of the grain shape is found as the spherical shape and the grain sizes are obtained as 0.10 μm – 0.40 μm. Most of the grains are uniform. It seems that the sample is smooth, spongy and pores are seen in the micrograph. The primary mechanism parameters that influence sensing behavior. Among them, small grain size plays an important role since it was shown that decrease at the grain size to the nanometer level lead to many interesting and new properties. Thus, the spinel such as $MgFe_2O_4$ is suitable for aggressive environments or humidity sensing materials.

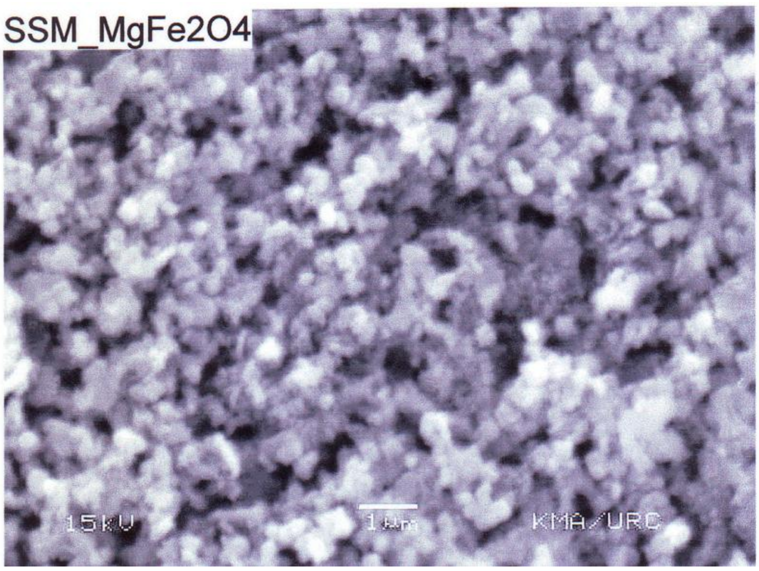

Fig 5 SEM micrograph of $MgFe_2O_4$ ferrite

TG-DTA thermograms of the sample were observed by SHIMADZU DTG-60H Thermal Analyzer in the temperature range 303 K – 873 K (30°C to 600°C) and as shown in Fig 6. One endothermic reaction peak is found in the DTA curve at 411.15°C and it indicates that the sublimation of the sample. In the observed temperature range, any phase changes not occurred in the sample because most of the cubic structure type materials are thermal stability and optically isotropic materials. Also the TGA thermogram shows that the mass variation (weight loss) of the sample. The mass variation of the sample is obtained as 3.051% loss of its starting amount.

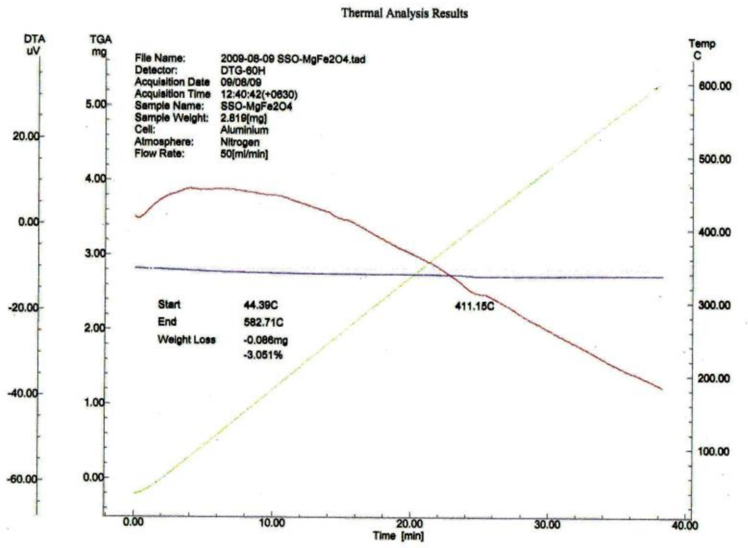

Fig 6 TG-DTA thermograms of $MgFe_2O_4$ ferrite

Electrical conductivity of a spinel sample obeys an Arrhenius expression $\sigma = \sigma_0 \exp(-E_a / kT)$, where σ is the conductivity, σ_0 is the pre-exponential factor, E_a is the activation energy for electrical conduction, k is the Boltzmann constant and T is the absolute temperature. Arrhenius plot of the ln σ versus reciprocal temperature of $MgFe_2O_4$ in the temperature range 300 K - 973 K is shown in Fig 7. Electrical conductivities of the sample are increased with increasing temperatures due to the interstitial diffusion of oxygen in the sample. Arrhenius expression of the electrical conductivity (σ) can be written as the form:

$$\begin{aligned}\sigma &= \sigma_0 \exp(-E_a / kT)\\ \ln(\sigma) &= (-E_a / kT) + \ln(\sigma_0)\\ &= (-E_a / k)(1/T) + \ln(\sigma_0)\end{aligned}$$

The activation energy E_a can be evaluated by using the slope of the ln(σ) versus 1000/T graph and obtained as 0.34 eV. The electrical conductivities of the sample at 300 K (starting temperature) and 973 K (final temperature) are obtained as 6.31 x 10^{-9} S cm^{-1} and 5.17 x 10^{-5} S cm^{-1} respectively. The experimental data are tabulated in Table 2. As presented in Table 2, the electrical conductivity of the sample at T ≥ 823 K is obtained as

~ 10^{-5} S cm^{-1} that shows the superionic conductivity of the sample. Thus, the sample is exhibited as a superionic conductor or fast ion conductor at high temperature (T ≥ 823 K).

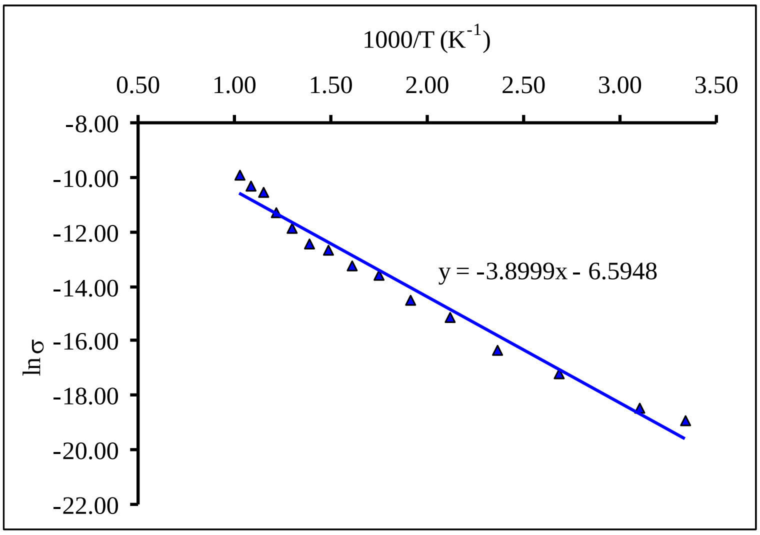

Fig 7 Arrhenius plot of the ln σ versus reciprocal temperature of $MgFe_2O_4$ ferrite

In the present work, variation of dielectric constants with temperatures of $MgFe_2O_4$ in the temperature range 300 K – 973 K is shown in Fig 8. It is found that the dielectric constants of the sample are increased with increasing temperatures. The slope of the (ε_r – T) curve is found to the linear and that indicates any phase changes characters are not occurred in the sample due to thermal agitation. It can be said that the sample is a thermally stability or thermally inert material. Experimental data of electrical conductivity and dielectric constant measurement of $MgFe_2O_4$ ferrite are tabulated in Table 2.

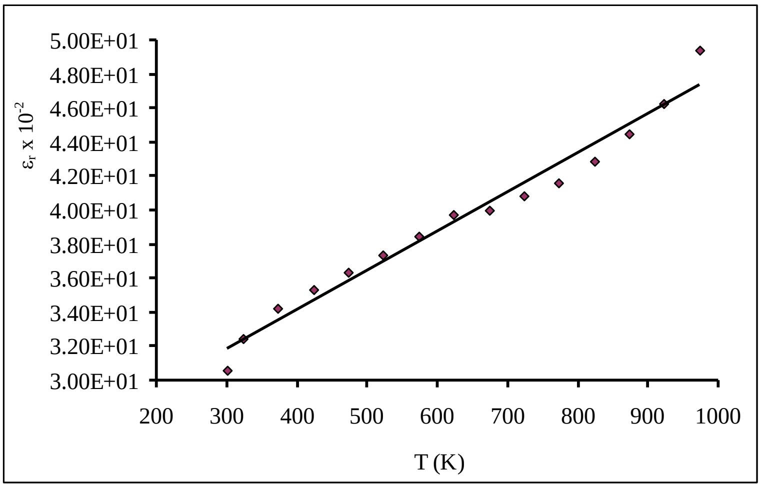

Fig 8 Plot of the variation of dielectric constants with temperatures of $MgFe_2O_4$ ferrite

Table 2 Experimental data of electrical conductivity and dielectric constant measurement of $MgFe_2O_4$ ferrite

T (K)	1000/T (K^{-1})	R (Ω)	σ (S cm^{-1})	C (F)	$\varepsilon_r \times 10^{-2}$
300	3.33	36700000	6.31E-09	1.1700E-09	3.0611E+01
323	3.10	24000000	9.65E-09	1.2400E-09	3.2442E+01
373	2.68	6650000	34.80E-09	1.3100E-09	3.4274E+01
423	2.36	2780000	83.30E-09	1.3500E-09	3.5320E+01
473	2.11	830000	279.00E-09	1.3900E-09	3.6367E+01
523	1.91	453000	511.00E-09	1.4300E-09	3.7413E+01
573	1.75	180000	1290.00E-09	1.4700E-09	3.8460E+01
623	1.61	130000	1780.00E-09	1.5200E-09	3.9768E+01
673	1.49	73800	3140.00E-09	1.5300E-09	4.0030E+01
723	1.38	56900	4070.00E-09	1.5600E-09	4.0815E+01
773	1.29	32500	7130.00E-09	1.5900E-09	4.1599E+01
823	1.22	18700	12400.00E-09	1.6400E-09	4.2908E+01
873	1.15	8850	26200.00E-09	1.7000E-09	4.4477E+01
923	1.08	6680	34700.00E-09	1.7700E-09	4.6309E+01
973	1.03	4480	51700.00E-09	1.8900E-09	4.9448E+01

Conclusion

Magnesium ferrite, $MgFe_2O_4$, has been successfully prepared by solid state reaction method. Structural, vibrational and thermal characteristics of the sample were studied by means of XRD, FTIR and TG-DTA methods. XRD pattern reveals that $MgFe_2O_4$ belongs to cubic crystal structure and the lattice parameters and crystallite size are obtained as $a = b = c = 6.4355$ Å and 51.86 nm. The appearances of the diffraction lines demonstrate that the sample is single phase nanosized crystalline material. FTIR spectrum shows that the phase confirmation of the sample with molecular vibration. The surface morphological feature of the grain shape is found as the spherical shape and the sizes are obtained as 0.10 μm – 0.40 μm. TG - DTA thermograms indicate that high temperature phases of the sample. According to TG-DTA thermograms, one endothermic reaction peak is found in the

DTA thermogram at 411.15°C and it indicates the sublimation of the sample. TGA thermogram shows that the mass variation of the sample is 3.051% of its starting amount due to the sublimation of the sample. The electrical conductivities of the sample are increased with increasing temperatures. Some of the electrical conductivities at 300 K and 973 K are obtained as 6.31×10^{-9} S cm^{-1} and 5.17×10^{-5} S cm^{-1} respectively. The activation energy is obtained as 0.34 eV. It was found that the sample is a superionic conductor or fast ion conductor in the high temperatures ($T \geq 823$ K). The dielectric constants of the sample are increased with increasing temperatures. In the (ε_r versus T) graph, any phase changes characteristics are not found. It is the structural stability of the sample. $MgFe_2O_4$ ferrite can be used as a high thermal resistive or thermal insulating material and it is considered as catalysis, the transformer cores, high density magnetic recording, high frequency devices and humidity sensors.

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