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INVESTIGATIONS ON SYNTHESIS, STRUCTURAL AND ELECTRICAL PROPERTIES OF MgO NANOPARTICLES BY SOL-GEL METHOD

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Magnesium oxide (MgO) nanoparticles were synthesized by sol-gel method. The synthesized samples have been characterized by X-Ray diffraction (XRD), scanning electron microscopy (SEM) and dielectric studies. SEM and XRD characterization studies confirmed that MgO particles thus obtained have hierarchical structures with high purity, and the particle sizes. The dielectric properties of MgO nanoparticles were studied in the different frequency range of 50Hz-5MHz at different temperatures. The frequency dependence of the dielectric constant and dielectric loss is found to decrease with increase in frequency at different temperatures. In addition, the electronic properties like valence electron plasma energy, average energy gap or Penn gap, Fermi energy and electronic polarizability of the MgO nanoparticles are also calculated.

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1. Introduction

Nanotechnology is the term used to cover the design, construction and utilization of functional structures with at least one characteristic dimension measured in nanometres. Such materials and systems can be designed to show new and significantly improved physical, chemical and biological properties, phenomena and processes as a outcome of the limited size of their constituent particles or molecules. During the last few years, synthesis of nanostructured metal oxide materials has concerned the researchers due to its potential applications [1]. In current years, researchers have focused more on the synthesis of MgO nanoparticles due to its novel applications in advanced technologies [2]. Metal oxides are very significant technological materials to be used in electronic and photonic devices [3]. The magnesium oxide (MgO) is a very suitable candidate for insulation applications due to its low heat capacity and high melting point [4]. Recently, it was reported that MgO has a good bactericidal show in aqueous environments due to the formation of super-oxide [5]. Various properties of MgO, such as catalytic behaviour, can be further enhanced if it is used as nanosized particles compared to micron-sized particles. Therefore, the formation of MgO nanostructures with a small crystallite size of less than 100 nm and homogeneous morphology has involved much attention due to their unique physicochemical properties including high surface area-to-volume ratio. It is extensively accepted that the properties of MgO nanostructures depend strongly on the synthesis methods and the processing conditions. Much effort was devoted to synthesize MgO nanostructures using various methods such as precipitation [6], solvothermal [7], chemical vapour deposition [8], electrochemical [9], sonochemical [10], microwave [11], electron spinning [12], combustion [13], template [14] and carbothermic reduction [15]. Each method has its own advantages and disadvantages. An important issue regarding synthesis and preparation of nanostructured MgO is controlling the parameters in order to obtain a more uniform size as well as morphology of the nanoparticles. In the present investigation, report the synthesis and characterization of MgO nanoparticles. The MgO nanoparticles subjected to study are characterized by X-ray diffraction, scanning electron microscopy (SEM) and dielectric studies. The scope of the present work is to study the dielectric

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properties of MgO nanoparticles as a function of the frequency and the temperatures. The electronic properties such as valence electron plasma energy, average energy gap or Penn gap, Fermi energy and electronic polarizability of the MgO nanoparticles required for the device application is also determined.

2. Experimental method

Magnesium nitrate, sodium hydroxide, ammonia solution, doubly distilled water were used for synthesis. The chemicals were employed without further purification Magnesium nitrate was dissolved in 100 mL of doubly distilled water. Sodium hydroxide was added drop by drop under sonication. The mixture was then ultrasonicated for 3 hours at room temperature to form magnesium hydroxide without any agglomeration. The magnesium hydroxide precipitate formed was filtered by washing it with doubly distilled water. It was followed by the addition of ethanol and the obtained sample was dried at 110^{o}C for one day. Calcinations were carried out at the temperature of 500^{o}C for 3 hours to obtain the purest form of MgO with hierarchical structure.

3. Results and discussion

3.1 Structural characterization

In order to determine the size and to study the structural properties of the synthesized MgO nanoparticles, the powder XRD analysis was performed. Structural identification of MgO nanoparticles were carried out with X-ray diffraction in the range of angle 2θ between 10º to 90º. Fig. 1 shows the XRD patterns for MgO nanoparticles, which were nanocrystalline in nature. The crystalline structure of nano MgO was determined by XRD analysis. The existence of strong and sharp diffraction peaks (1 1 1), (2 0 0) and (2 2 0) planes respectively indicated the formation of MgO. The average particle size of the nanomaterial was determined using the following Debye - Scherrer equation and it was found to be 20 nm.

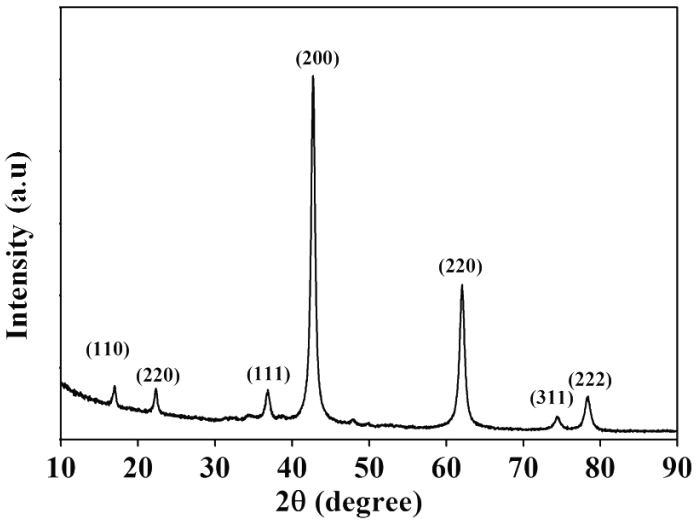

Fig.1.XRD spectrum of MgO nanoparticles

3.2 Surface morphology

Scanning electron microscope (SEM) was used for the morphological study of MgO nanoparticles. Fig. 2 shows the SEM images of the MgO nanoparticles. The MgO nanoparticles

formed were highly agglomerated. From the SEM images the particle sizes of the pure MgO nanocrystals were found to be in the range 20-50 nm. It is also clear that the synthesized MgO nanoparticle is very porous increases with pores and open voids.

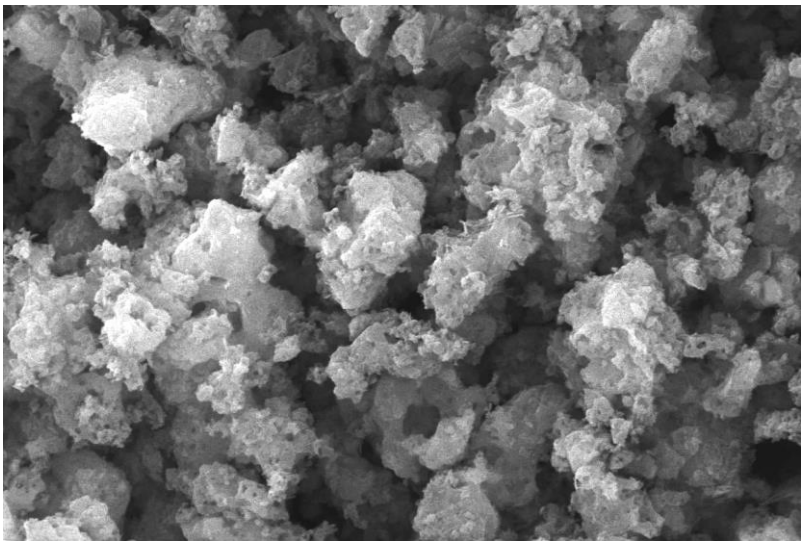

Fig.2 SEM micrograph of MgO nanoparticles

3.3 Dielectric properties

In the dielectric constant and the dielectric loss of the MgO nanoparticles were studied at different temperatures using the HIOKI 3532 LCR HITESTER instrument in the frequency region of 50 Hz to 5 MHz. The dielectric constant was measured as a function of the frequency at different temperatures as shown in Fig.3, while the corresponding dielectric losses are depicted in Fig.4. Fig. 3 shows the plot of the dielectric constant (ε_r) versus applied frequency. It is observed (Fig.3) that the dielectric constant decreases exponentially with increasing frequency and then attains almost a constant value in the high frequency region. This also indicates that the value of the dielectric constant increases with an increase in the temperatures. The net polarization present in the material is due to ionic, electronic, dipolar and space charge polarizations [16]. The large value of the dielectric constant is due to the fact that MgO nanoparticles act as a nanodipole under electric fields. The small-sized particles necessitate a large number of particles per unit volume, resulting in an increase of the dipole moment per unit volume, and a high dielectric constant. The dielectric loss studied as a function of frequency at different temperatures is shown in Fig.4. These curves suggest that the dielectric loss is strongly dependent on the frequency of the applied field, similar to that of the dielectric constant. The dielectric loss decreases with an increase in the frequency at almost all temperatures, but appears to achieve saturation in the higher frequency range at all the temperatures. In the low frequency region, high energy loss is observed, which may be due to the dielectric polarization, space-charge and movement of electrons in rotational fashion at low frequency range.

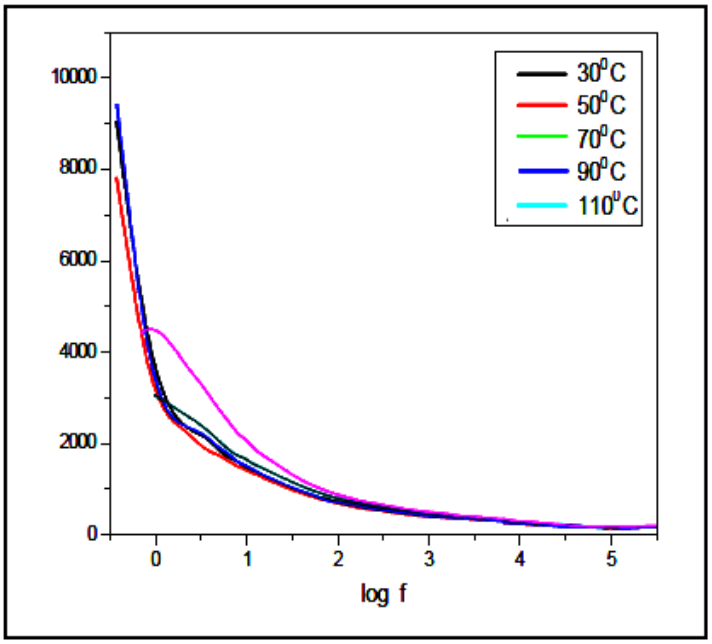

Fig.3. Dielectric constant of MgO nanoparticles, as a function of frequency

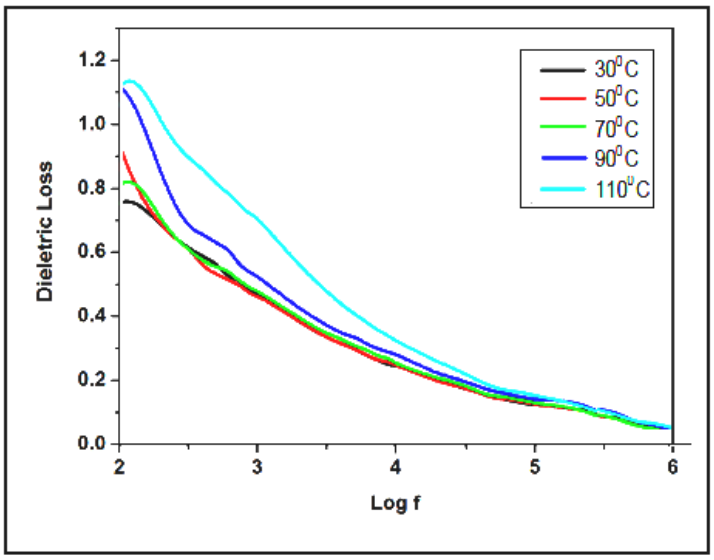

Fig.4. Dielectric loss of MgO nanoparticles, as a function of frequency

In the proposed relation, only one parameter viz, the high frequency dielectric constant is required as input, to evaluate electronic properties like valence electron plasma energy, average energy gap or Penn gap, Fermi energy and electronic polarizability of the MgO nanoparticles. The theoretical calculations show that the high frequency dielectric constant is explicitly dependent on the valence electron Plasma energy, an average energy gap referred to as the Penn gap and Fermi energy. The Penn gap is determined by fitting the dielectric constant with the Plasmon energy [17]. The valence electron plasma energy, $\hbar\omega_p$, is calculated using the relation [18],

$$\hbar\omega_P = 28.8\left(\frac{Z\rho}{M}\right)^{1/2} \quad (1)$$

According to the Penn model [19], the average energy gap for the MgO nanoparticles is given by

$$E_P = \frac{\hbar\omega_P}{(\varepsilon_\infty - 1)^{1/2}} \tag{2}$$

where $\hbar\omega_P$ is the valence electron plasmon energy and the Fermi energy [17] given by

$$E_F = 0.2948(\hbar\omega_P)^{4/3} \tag{3}$$

Then we obtained the electronic polarizability α, using a relation [19, 20],

$$\alpha = \left[\frac{(\hbar\omega_P)^2 S_0}{(\hbar\omega_P)^2 S_0 + 3E_P^2}\right] \times \frac{M}{\rho} \times 0.396 \times 10^{-24} cm^3 \tag{4}$$

where S_0 is a constant given by

$$S_0 = 1 - \left[\frac{E_P}{4E_F}\right] + \frac{1}{3}\left[\frac{E_P}{4E_F}\right]^2 \tag{5}$$

The value of α obtained from equation (4) closely matches with that obtained using the Clausius-Mossotti relation,

$$\alpha = \frac{3}{4}\frac{M}{\pi N_a \rho}\left[\frac{\varepsilon_\infty - 1}{\varepsilon_\infty + 2}\right] \tag{6}$$

Considering that the polarizability is highly sensitive to the band gap [21], the following empirical relationship is also used to calculate α,

$$\alpha = \left[1 - \frac{\sqrt{E_g}}{4.06}\right] \times \frac{M}{\rho} \times 0.396 \times 10^{-24} cm^3 \tag{7}$$

where E_g is the band gap value determined through the UV absorption spectrum. The high frequency dielectric constant of the materials is a very important parameter for calculating the physical or electronic properties of materials. All the above parameters as estimated are shown in Table 1.

Table.1 Electronic properties of the MgO nanoparticles

Parameters	**Value**
Plasma energy ($h\omega_p$)	24.34 eV
Penn gap (E_p)	6.51 eV
Fermi Energy (E_F)	20.57 eV
Electronic polarizability (using the Penn analysis)	3.28 x 10^{-24} cm^3
Electronic polarizability (using the Clausius-Mossotti relation)	3.32 x 10^{-24} cm^3
Electronic polarizability (using band gap)	3.18 x 10^{-24} cm^3

4. Conclusion

MgO nanocrystallites were synthesized by sol-gel method. The XRD studies show that, MgO nanoparticles prepared are in nanocrystalline range and also diffraction peaks are found. The size and morphology of the MgO nanoparticles were characterized using scanning electron microscopy (SEM). The dielectric constant and dielectric loss of the MgO nanoparticles are measured in the frequency range of 50Hz-5MHz at different temperatures. The dielectric studies reveal that both the dielectric constant and dielectric loss decrease with an increase in frequency. Electronic properties such as plasma energy, Penn gap, Fermi energy and electronic polarizability of the MgO nanoparticles, which are required for device application, have been calculated.

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