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Silica gel doped with cobalt oxide prepared by sol-gel technique; improving dielectric charge storage

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Abstract

The structural, morphology and magnetic properties of Cobalt oxide embedded in silica gel with 10 mol. %, in monolith form, sintered at different temperature ranging from 60 up to 1300˚C, respectively recorded as (SC(60-1300) were prepared by a modified sol gel technique. The sintering temperature effect on the crystallization, surface morphology and magnetic behaviors of the prepared samples will be study,

Phase identification by using surface morphology and X-ray diffraction will be study by using Transmission electron microscope (TEM) and Field emission scanning electron microscope imaging (FESEM). The nano-scale presence and the formation of the α-cristobalyte tetragonal phase of silica gel as well as the doped samples with cobalt oxide crystallinity enhancement were detected using the mentioned techniques. FTIR spectra were being study. The saturation magnetization (M_s), remnant magnetization (M_r) and coercive force (H_c), was founded to be equl to 0.183,0.031 emu/g and 46.7O_e respectively. The A. C. conductivity increased by increasing the temperature .Therefore, by increasing the sintering temperature of the silica gel network doped with$Co_3O_4$are applicant for several electronic and industrial devices with improved dielectric charge storage capacity and strength.

Key Words: Cobalt Oxide, Sol Gel, FTIR, X-ray diffraction, Electrical conductivity.

Introduction

Nano-structure and nanocomposites fabricated from particles embedded in a porous matrix have attracted large interest, where, the structural confinement of the nanoparticles causes tailoring their physical properties [20]. A porous matrix provides, enough nucleation sites for nanoparticle formation and an effective way to avoid their aggregation [7]. Silica gels are the most suitable net - work for this application due to their chemical inertness, large accessible surface area, high porosity, transparency, low thermal expansion coefficient, exceptional mechanical, electrical, optical and magnetic properties [8,9, 16, 19]. Cobalt oxide nanoparticles embedded in a silica matrix by adopting different preparation methods such as sol-gel [17], hydrothermal, grafting, chemical vapor deposition and direct solution impregnation methods [2]. Cobalt silica composite materials have been widely studied for many technological applications such as catalysis, magnetic recording, sensing, gas sensor, and super capacitance [15].Cobalt oxide is an active material having multi oxidation states and silica has a porous structure. So the composite of cobalt oxide and silica would process interesting properties . There are two main approaches for obtaining dispersed nanoparticles in silica matrix namely: (i) focus –synthesis methods which, is based on synthesis of cobalt nanoparticles in pre-prepared silica matrix, such as, impregnation, ion-exchange, and grafting techniques (ii). The sol-gel method has an accurate control of the synthesis condition thus allowing modifying surface chemical properties·One of the advantage of the sol-gel process, the amount of water strongly influences the structure of the final composite· It is well know that the silica – gel is often used with microclimates to control relative Humidity.

The main aim of the present work is to explore the effect of increasing the sintering temperture on the structure of the host material (silica – gelglass) when cobalt nitrate was in corporated in it. Morover the evaluation of the cobaltspecies embedded in silica – gelglass net work, using the X-Ray diffraction, FTIR, TEM and magnetic properties techniques. The AC electrical conductivity will be discussed as a function of frequncy ranging from 42 Hz up to 5 MHz . Where the A.C. conductivity increased by increasing the sintering temperature from 60 up to 1300°C.

Experimental work

Silica-gel net-work containing 10 mol.% of Cobalt oxide (CoO_2) nanocomposite; (SiO_2–10 mol.% CoO_2), abbreviated as S10C(60-1300) were prepared in monolithic form; Sintered by adjusting different temperature ranging from 60 up to 1300°C for 3 hours. Hydrolysis and poly condensation reaction of tetra-ethoxysilane $(CH_3CH_2OH)_4$-Si(TEOS), (TEOS,99.999%, Sigma–Aldrich) as SiO_2 and CoO_2 precursors dissolved in ethanol (CH_3CH_2OH) solutions were carried out. A pre-hydrolysis reaction of silica-gel net-work containing 10 mol.% of Cobalt oxide (CoO_2)solution was obtained by applying at room temperature vigorous stirring with duration of 4

hours by using distilled water (H_2O), in addition ethanol and HCl were used to act as solvent and catalyst, respectively. Before performing as monolith, the mentioned solutions were filtered. The obtained solutions of monolith materials were left in square plastic boxes during gelation process, for obtaining hydrolyzed species within the mixture. Aging process for all samples for 21 days was use 60˚C, where the samples were in the transition states from low viscosity (liquid) to the intermediate viscosity (gel) by evaporating the solvent molecules before drying. So the samples were heated by sintering at 60, 200, 300, 500, 1200 and 1300 ˚C for 3h. Amuffle furnace type Carbolated CWF1200 was used for sample calcinations with heating rate (0.2- 0.4)˚C/ min.

Characterization

The X-ray diffraction (XRD) using monochromatic $CuK_{\alpha 1}$ radiation of wavelength 1.54056Å from a fixed source operated at 45 kV and 9 mA. The crystallite size (G) was calculated from the Scherrer's equation [19];

$$G = K\lambda / D\cos\theta \quad (1)$$

where *K* (= 0.89) is the Scherer constant, *λ* is the wavelength, *D* is the full width (in radians) of the peak at half maximum (FWHM) intensity and θ is the diffracted angle.

The micro structure and the morphology were characterized by using JEOL transmission electron microscope (TEM), model: Jeol 123000 with magnification up to 600 kx, giving a resolution down to 0.2 nm. The chemical structure of samples were measured at room temperature by (FTIR) spectroscopy (VERTEX 80V Bruker range from (4000 – 400) cm^{-1}.The morphology of the prepared samples was depicted by using (FE-SEM). FESEM instrument (FEI, model: Quanta 250 FEG) to determine the crystalline phases. To achieve this, the samples were gold-sputtered using Edwards S150A Sputter coater to make the images more obvious.

For dielectric measurement, we utilized acomputerized LRC bridge (Hioki model 3531 Z Hi Tester), at different frequencies ranging from 42 Hz to 1 MHz The samples used in the measurement were in the form of disk, have 10 mm in diameter and 3 mm thickness, pressed using a pressure of 10 Tons, at room temperature.The permittivity (*ε'*) and AC conductivity (σ_{ac}) were calculated from the output parameters like capacitance (*C*) and loss tangent (*tanδ*) as follows:

$$\varepsilon' = \frac{Cd}{\varepsilon_o A} \quad (1)$$

$$\sigma_{ac} = \omega\varepsilon_o\varepsilon' tan\delta \quad (2)$$

Where ε_o is the permittivity of free space (8.854×10^{-12} F/m), *d* is the sample thickness and *A* is the sample surface area. The measurement uncertainty reported to be ±1% and ±3% in *ε'* and *tanδ*, respectively. The measurement reproducibility was tested by re-measuring *ε'* and *tanδ* after experimenting once again.

Results and discussion

XRD results

Figure 1 (a-f) displays the effect of increasing the sintering temperature on the X-ray diffraction patterns of silica doped with cobalt oxide at different temperature (60, 200, 300, 500, 1200and1300) °C. From figure 1(a-d), we can detected abroad peak between 2θ˚ equal (18˚ and 25˚) corresponding to α-cristobalite tetragonal phase of silica gel. At temperature 500° C, the small peaks at 2θ = (32° and39°), appeared, indicating that the only detected phase was Co_3O_4 according to(JCPDS card #78-1970) is . From fig. 1(e-f), the XRD patterns of samples heat treated at 1200°C and 1300°C it is illustrates that the peaks at 2θ° values equal to (21.8°, 28.3°, 31.4° and 36°) which, indicated the formation of a cobalt silicate Co_2SiO_4 phase (JCPDS card #72-1508). However with an increase in temperature, the following small peaks according to 2θ° equal (42.5°,44.7°,46.9°, 48.6°,52.1°,54.1°,57°,60.3°,61.9°,64.9°,67° and 68.6°) of CO_3O_4 appeared, respectively. By using Sheerer s formula D= 0.94 λ / β cosθ, the size of CO_3O_4 nanoparticles embedded in silica – gel is 14.5, 27 and 29 nm for the sintering temperature at 500 ° C, 1200° C and 1300°C, respectively[14].

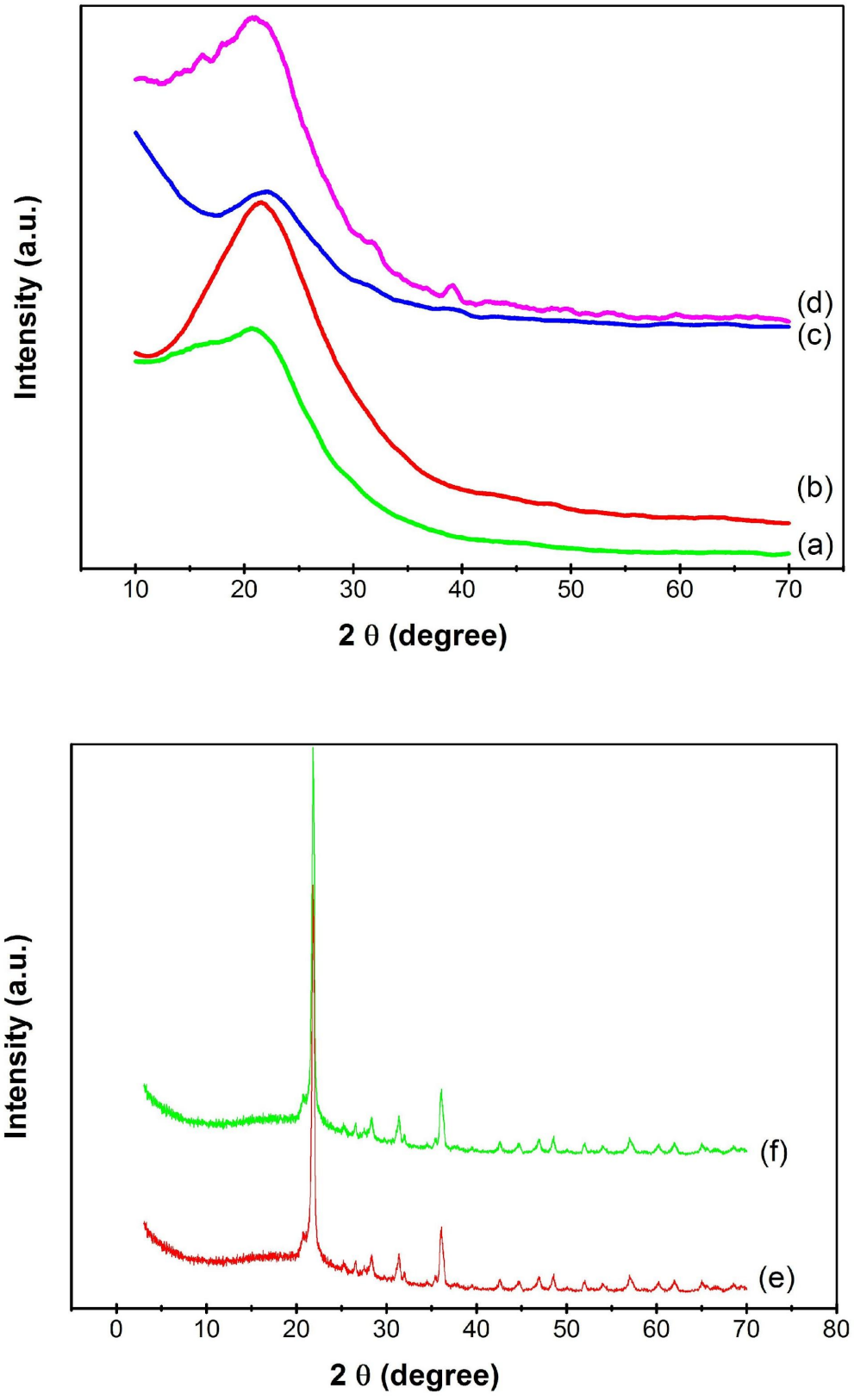


Figure 1(a-f) : The XRD patterns of the (a) S10C(60), (b) S10C(200),(c) S10C(300),(d) S10C(500),(e) S10C(1200) and (f) S10C (1300), respectively.

FTIR results

Figure: 2(a-f) show the FTIR spectra of Co/SiO_2nano composite at different temperature (60,200,300,500, 1200 and 1300°C). It is obvious that the absorption bands at 3413 Cm^{-1} appeared in all figures ascribed to the stretching

of O-H mode of H-bonded and at 1620 Cm^{-1} is assigned to the bending of O-H mode of water molecules. The presence of this band at all temperature indicates that water exists in the structure conformation. The narrow peak at 1383Cm^{-1}indicate that the presence of nitrate ions in the nano-composite. This band is ascribed to the asymmetric stretching of N-O bonds in the NO_3^- group. The intensity of this band is decrease with increasing temperature because the cobalt nitrate is thermally decomposed, and probably evolves to the atmosphere in the form of nitrogen oxides. The band at 2925Cm^{-1}corresponds to the stretching vibrations of hydroxyl groups. The strong absorption band at 1094Cm^{-1} corresponding to the asymmetric stretching vibration of the Si-O-Si in the SiO_2 bond structure. The band at 794Cm^{-1} is agreeing well the symmetric stretching vibration of the Si-O-Si. The bond at 467Cm^{-1} is assigned to the bending vibration of Si-O-Si chains. The weak intensity band at 876Cm^{-1} is assigned to either Si-OH and /or $Si-O^-$ stretching vibrations. The appearance of $Si-O^-$ groups is related to the interaction between Co^{+2} ions and the chain of alternate silicon and oxygen atoms [1]. It can be noticed that, at high temperature the band intensity decreases, due to the calcinations of the samples due to conversion of $Si(OH)_4$ to SiO_2. The absorption band at 620 Cm^{-1} is due to the presence of Co(III) –O bond in Co_3O_4, also this band is assigned to Co-O stretching in Si-O-Co matrix. The band at 450Cm^{-1} associated with Co-O stretching is appeared in all different temperature. The presence of Si-O-Si bands as confirmed from IR spectra together with the results obtained by XRD analysis implying that the formed phases Co_3O_4 and $CoSiO_3$ are mostly embedded in an amorphous silica matrix.

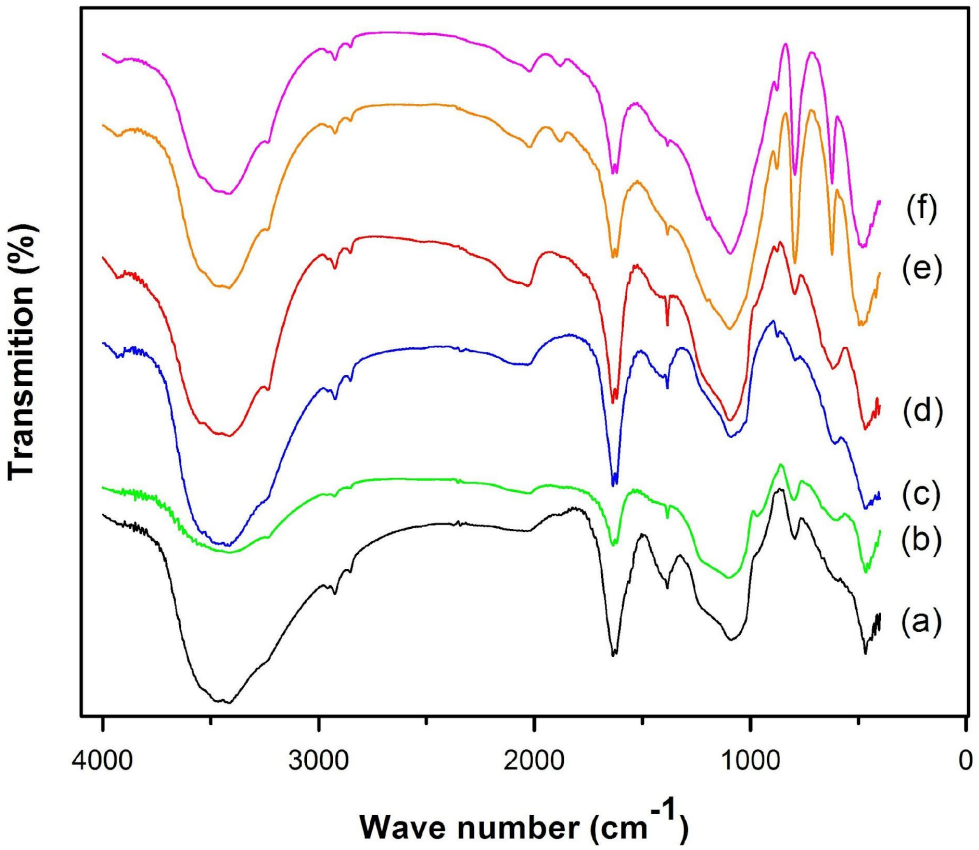
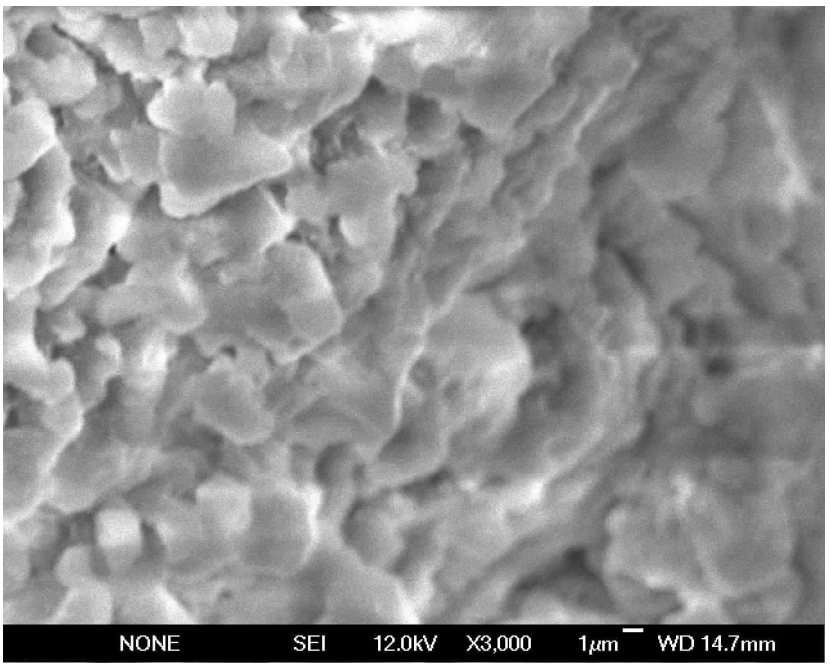


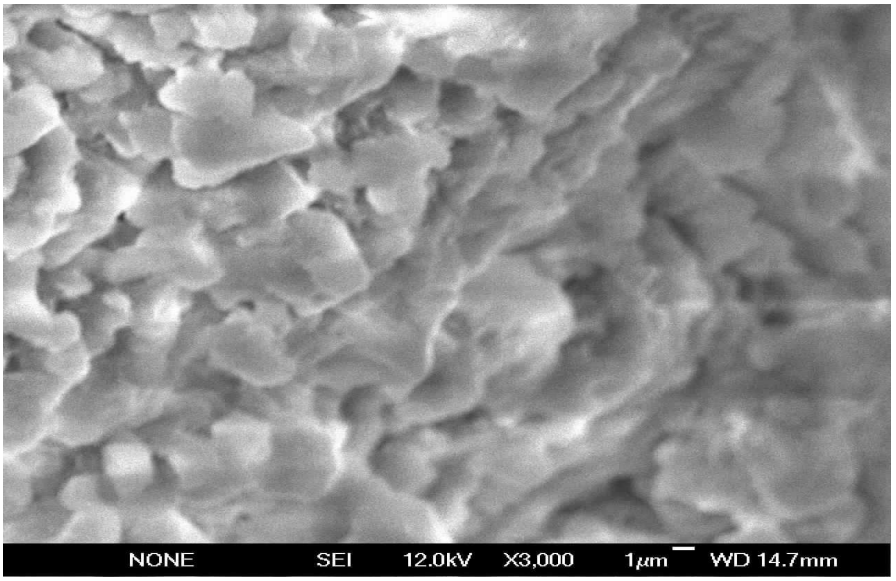
Figure 2(a-f): FTIR spectra of the (a) S10C(60), (b) S10C(200),(c) S10C(300),(d) S10C(500),(e) S10C(1200) and (f)S10C (1300), respectively.

FESEM and TEM results

The FESEM images of the prepared powder samples S10C(60) and (b) S10C (1300), respectively revealed the modification of the surface morphology smoothed the microscopic hill-like structures and the new surface structures are created at lower temperature Fig.3(a).This surface evolution is a competition between the two studied temperature and Co atom diffusion inside the silica matrix. Then when the investigated samples are subjected to higher temperature of preparation at 1300°C the surface roughness decreases Fig, 3(b). The decrease in surface roughness suggests the existence of such surface mentioned smoothening properties. This may be due to the induced viscous, high density, compacting structure, volume diffusion, or surface diffusion at higher temperature which can all result in smoothening of the surface.



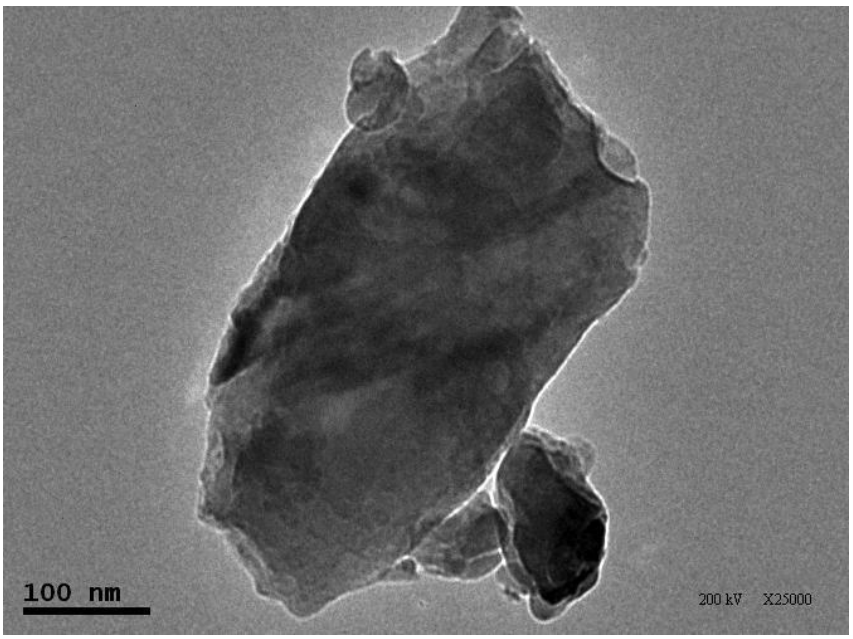
(a)



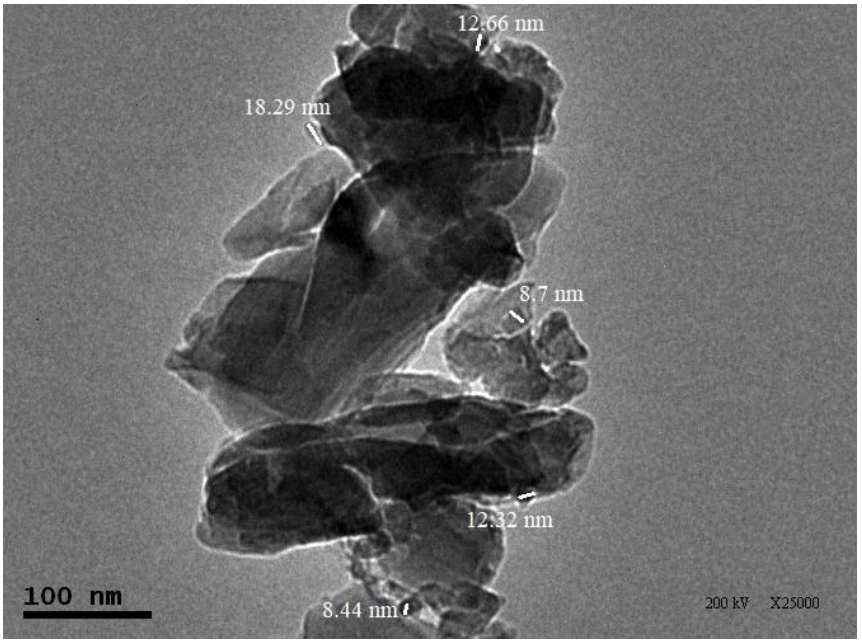
(b)

Figure3 (a, b): The FESEM images of the (a) S10C (60) and (b) S10C (1300), respectively.

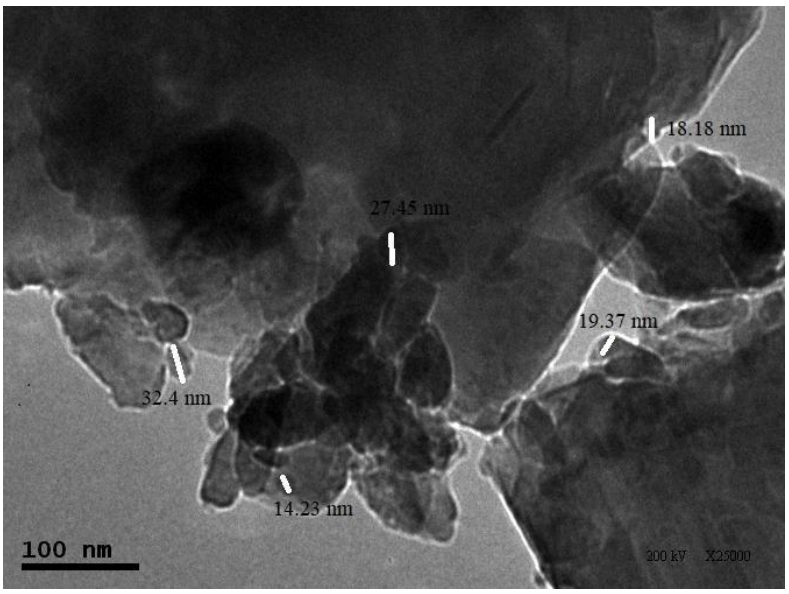
To verify the micro-structure of silica gel doped with 10 mol. % of Cobalt oxide Co_3O_4 sintered at different temperature at 60, 500, 1200 and 1300°C, respectively transmission electron microscope (TEM) micrographs are displayed in Fig.4 (a-d). They confirm the presence of the nano-structure phase. From the figures, it can be seen that a remarkable uniform dispersion of Co_3O_4 nano-particles inside the silica matrix is present showing a better dispersion of smaller cobalt particles over the silica framework. In all samples the cobalt particle sizes is strongly dependent on increasing temperature, where the crystallite sizes were increased from about 12.1 up to 25.6 nm as shown in Table (1). However, at 60°C the sample shows an amorphous phase at such lower sintering temperature. These data seem to be in agreement with the presence of small Co_3O_4 nano-particles sizes as derived and obtained from the X-ray diffraction patterns[3].



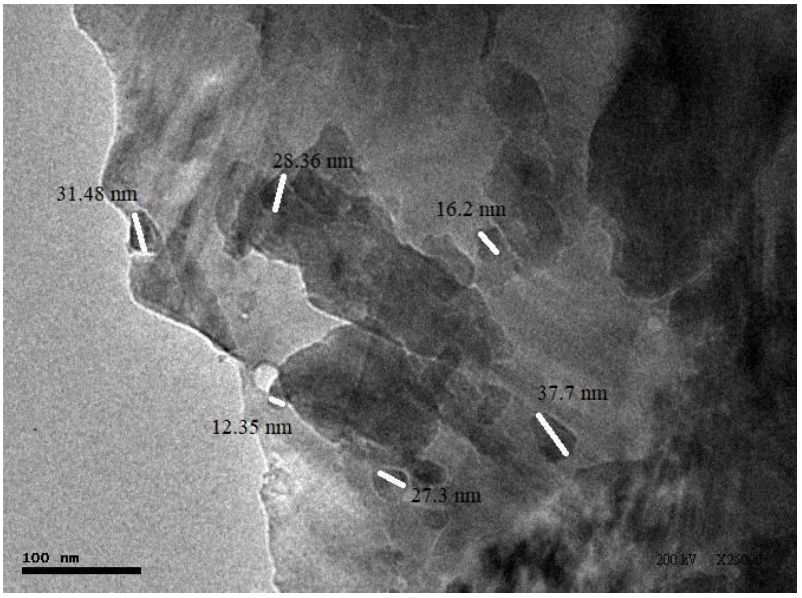
a)60°C)



(b)500°C



c)1200°C**)**



(d) 1300˚C

Figure4 (a-d): The TEM micrographs of the (a) S10C (60), (b) S10C (500), (c) S10C (1200), and (d) S10C (1300), respectively.

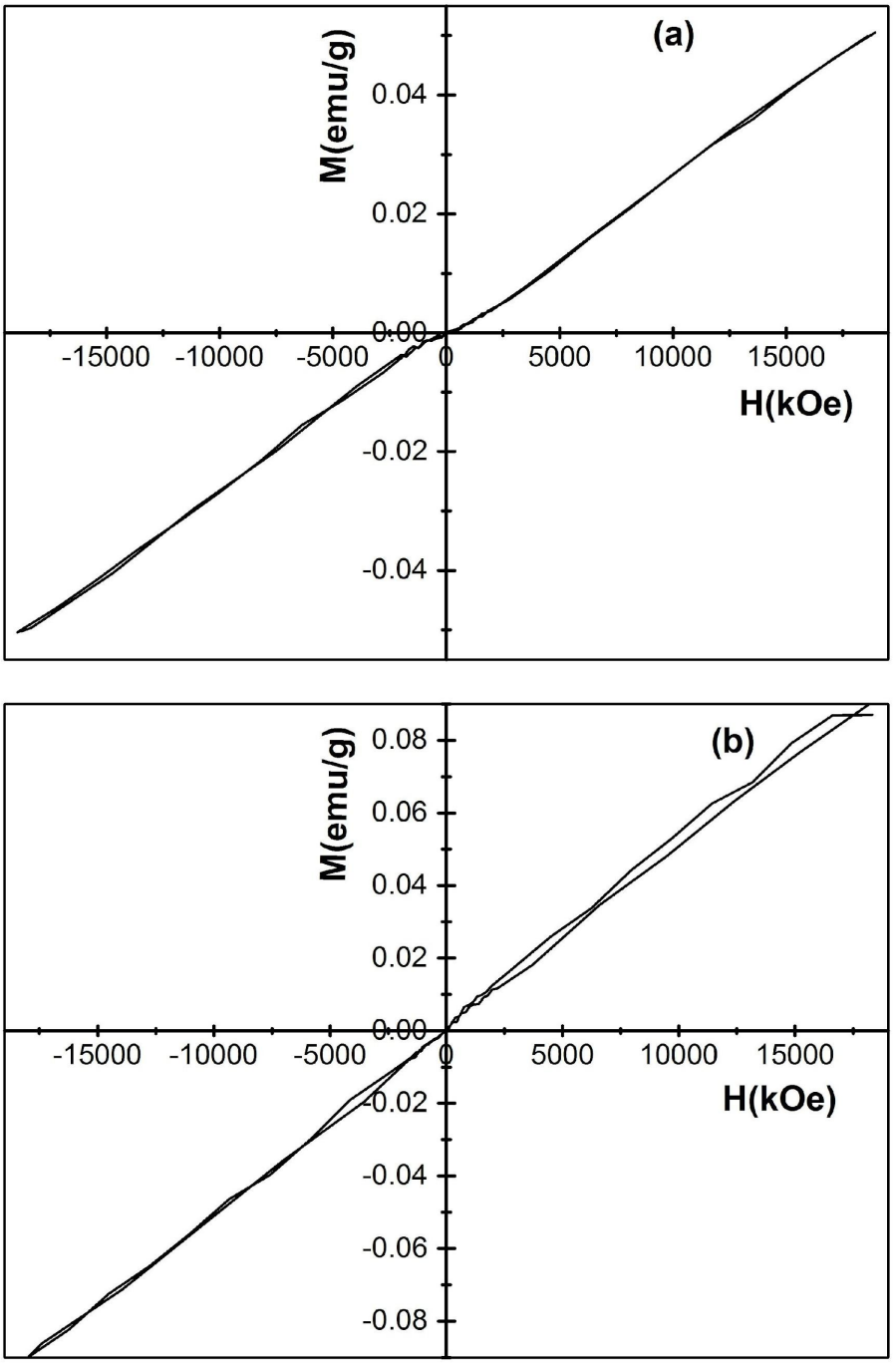
Table (1) Particle size

Temperature °C	TEM	XRD
500	12.1	14.5
1200	21	27
1300	25.6	29

Magnetic Results

The magnetic hysteresis loops of Co_3O_4/SiO_2nano-composites annealed at (60,300,500 and 1300°C) are presented in fig. (5:a-d).As witnessed in samples which annealed at low temperatures 60 and 300°C,fig. (5:a,b) have very weak magnetization. The hysteresis curves tend to be linear and hardly saturated up to magnetic fields of 20kOe, thus favoring paramagnetic properties [13].These unusual features may be attributed to large lattice

strain of the samples. At temperature 500°C, fig. (5: c), the hysteresis loop shows the presence of curvature shape which indicates Co ferromagnetic nature[18]. At higher annealing temperature, 1300°C, fig. (5:d),it has been demonstrated that the magnetization of Co_3O_4/SiO_2nano-particles have reached the saturation. The corresponding saturation magnetization (M_s), Coercively (H_c) and remnant magnetization (M_r) were noted to be 0.1813emu/g, 46.7Oe and 0.031emu/g, respectively.



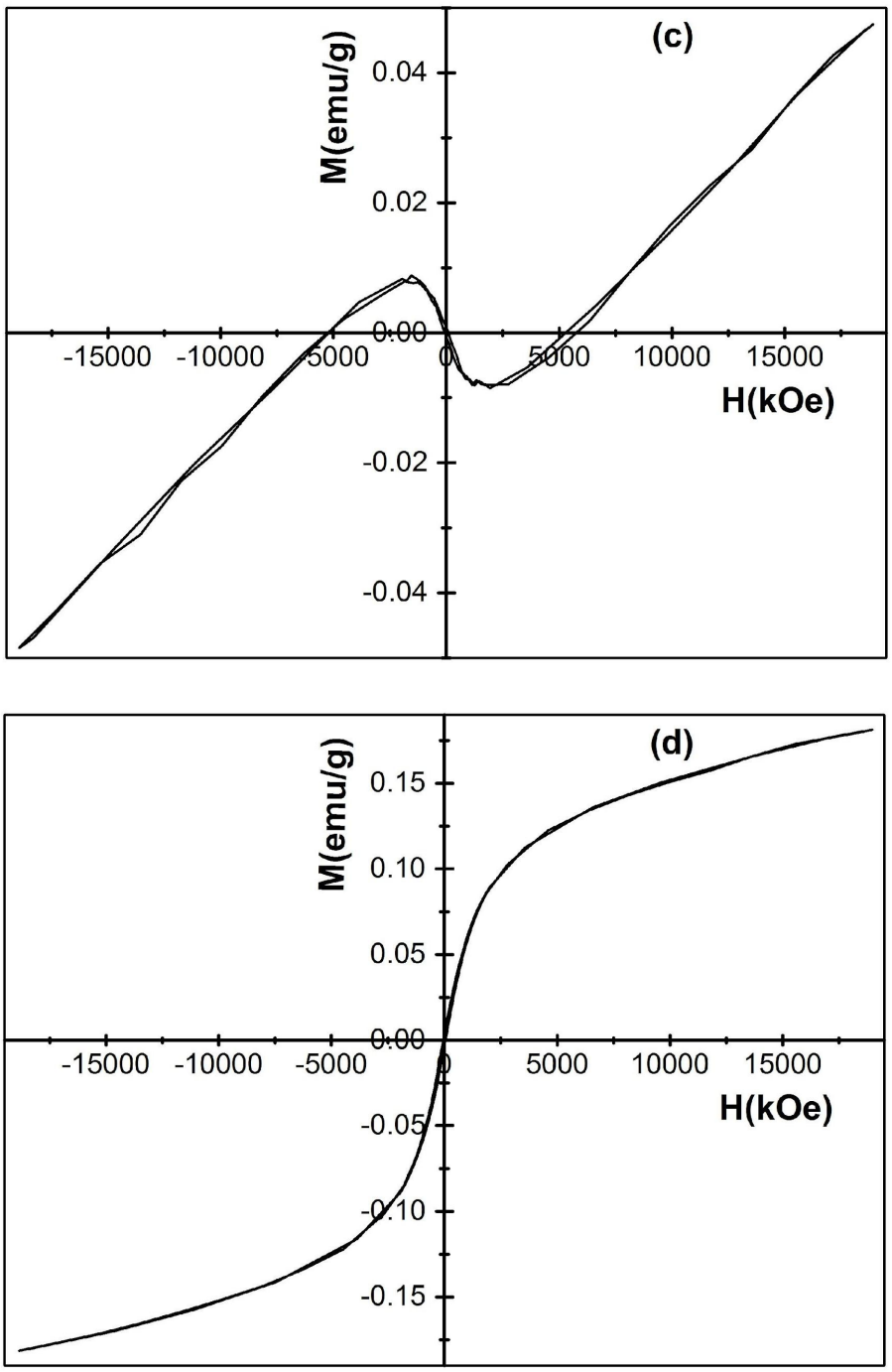


Figure 5: Magnetic hysteresis curves for (a) S10C(60) , (b) S10C(300) ,(c)S10C(500), and(d) S10C(1300), respectively.

Dielectric Results

The dielectric properties of the samples Co_3O_4/SiO_2 nanocomposite at different temperature (60, 300, 500, 1200 and 1300)°C where investigated at frequency range (42 Hz up to 1 MHz)as displayed in figs. (6-8). Figure (6) shows the permittivity (ε`) as a function of Ac frequency. At low frequency the permittivity was found to decrease rapidly then remains mainly constant at bigger frequencies value. The obtained behavior could be according to the dipoles inability to rotate rapidly for leading to a lag between the applied field and that of the oscillating dipole frequency. The large values of (ε`) at low frequencies are according to the interfacial polarization which is always characterized the materials exhibited more than one phase. Interfacial polarization results from the collection of charges at the interfaces between the prepared samples and the electrodes. When these charges are move by the application of an external electric field, the motion will be slowly in the prepared samples, causing space charges to be present, which causes distort the macroscopic field and act as polarization [6].At

elevated frequencies; the charge carriers have no chance to reach electrodes. The permivity (ε`) charge carrier's value show a little bit dependence on the applied electric field. Moreover, it can be noticed that ε`increases by increasing the sintering temperature for all frequencies. This increase is usually associated with the decrease in bond energies.[5]The increase in the dielectric permittivity is attributed to the increase in charge carrier density with increasing Co_3O_4content.

Figure (7) shows variation of dielectric loss (ε``) as a function of frequency. At lower frequencies, ε`` exhibits very large values. This may be due to the motion of the free charge in the matrix and to the motion of polar groups at Co_3O_4/SiO_2nano-composite.While, at the higher applied frequencies, ε``is low and constant due to the polarized orientation and chain motion cannot maintain phase with the rapidly oscillating electric field[12]. There are relaxation regions in figs. (6&7), about frequency range (4×10^3 - 7×10^3) Hz which associated with the hopping conduction due to the mobile charge carriers over long distances [4].

The influence of increasing the sintering temperature on the variation of AC conductivity (σ_{Ac}) as a function of frequency for Co/SiO_2nano-compositesat various temperature (60, 300, 500, 1200 and 1300˚C) is illustrated in Fig. (8).It is obvious that (σ_{Ac}) increases with the increase in frequency as well as with the increase in temperature. The increasing of (σ_{Ac})with Ac frequency is according to bound carriers engaged in the sample ,Also, the increases of (σ_{Ac}) may be due to, two factors: Firstly, at high frequency, the electric energy activates the charge carriers to leap between nanoparticles. Secondly, the dielectric relaxation of polarization of nanoparticles increases which, leads to increase of Ac conductivity (σ_{Ac}) [11]. By increasing the temperature, the excitation of electron from one conducting cluster to another, caused an increase in the A.C. conductivity. Also, at higher frequencies, the hopping of electrons became more enhanced which interpreted that, the conductivity is relatively higher for all the samples [10]. The maximum value of (σ_{ac}), at higher temperature doped with 10 mol % of Co_3O_4. Finally, the σ_{ac} of the investigated films reveals that CBH is the most probable conduction mechanism for Co_3O_4 doped prepared samples. Based on the obtained results, the Co_3O_4 embedded in silica gel and by increasing its sintering temperature can be used to enhance the dielectric properties of the prepared samples. Therefore, by increasing the sintering temperature of the silica gel network doped with $Co_3O_4$are a candidate for several electronic and industrial devices with improved dielectric charge storage capacity and strength.

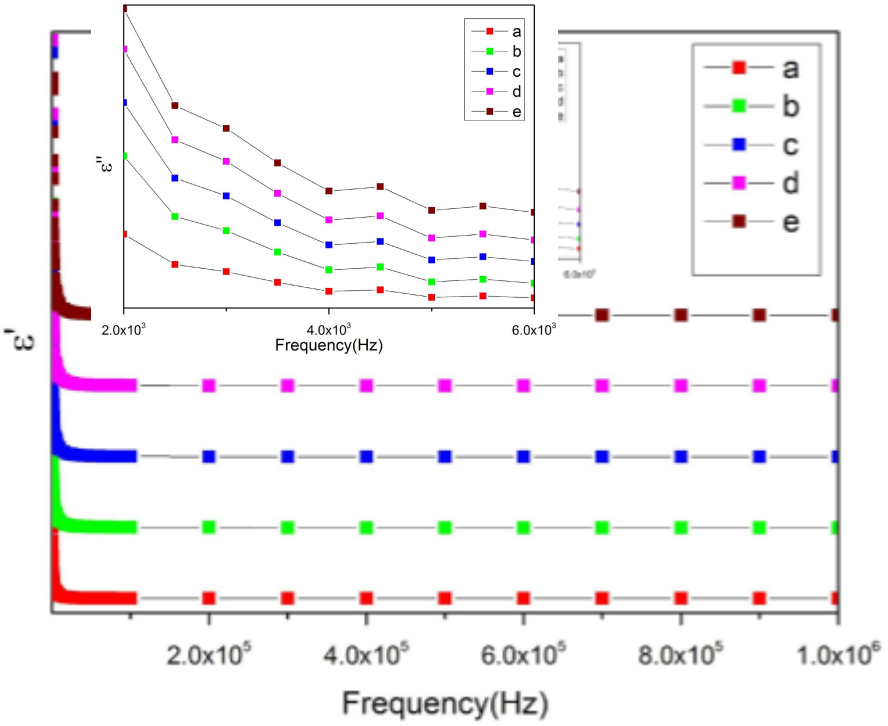


Figure 6: The RT ε`(ω) as a function of frequency for (a) S10C(60) , (b) S10C(300),(c)S10C(500), (d) S10C(1200), and(e) S10C(1300) , respectively, in the frequency range (42-10^6) Hz.

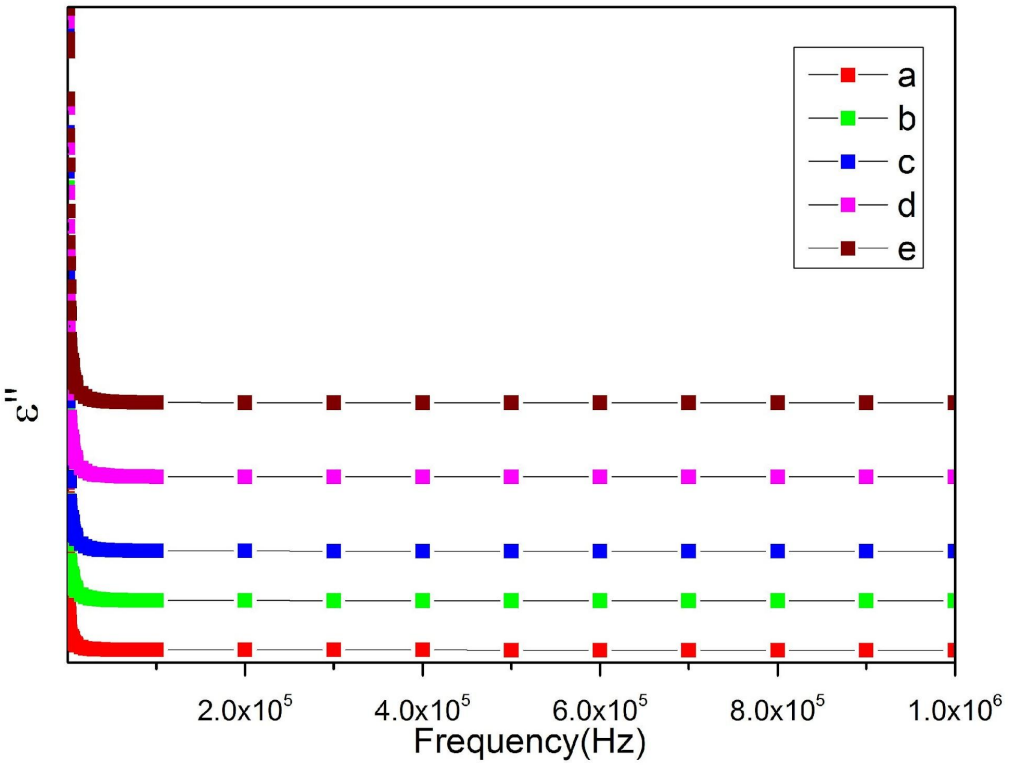


Figure7: The RT ε``(ω) as a function of frequency for (a) S10C(60) , (b) S10C(300) ,(c)S10C(500), (d) S10C(1200), and(e) S10C(1300) , respectively, in the frequency range (42-10⁶) Hz.

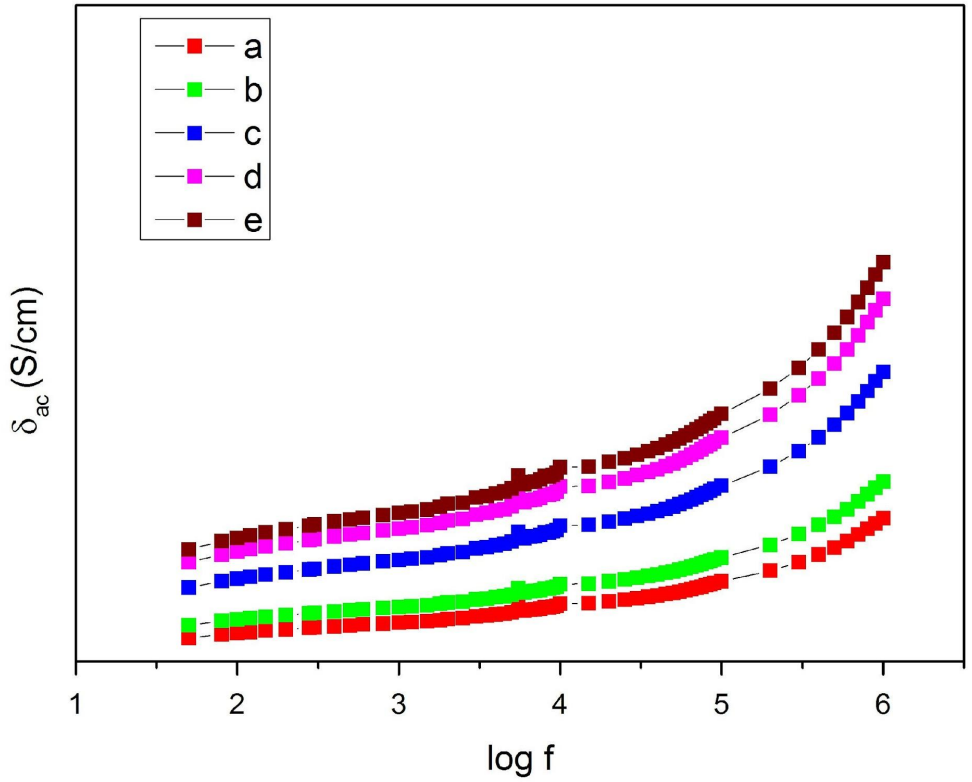


Figure8: The RT σ_{ac} as a function of frequency for(a) S10C(60) , (b) S10C(300) ,(c)S10C(500), (d) S10C(1200), and(e) S10C(1300) , respectively, in the frequency range (42-10⁶) Hz.

Conclusion:

The structural, morphology and magnetic properties of Cobalt oxide embedded in silica gel with 10 mol. %, annealed at different temperature were succefully prepared by a modified sol gel technique. Phase identification by using surface morphology and X-ray diffraction was studied by using Transmission electron microscope (TEM) and Field emission scanning electron microscope imaging (FESEM). The nano-scale presence and the formation of the α-cristobalyte tetragonal phase of silica gel as well as the doped samples with cobalt oxide crystallinity enhancement were detected using the mentioned techniques. The molecular structure of cobalt oxide imbedded in silica gel at different temperature was studied by FTIR spectra. It was observed that the band at 450 cm^{-1} assigned to CoO_2 was present in all different temperature. TEM and XRD investigations of the samples revealed that highly dispersed cobalt nanoparticles embedded in silica matrix. The spherical cobalt nanoparticles were measured giving an average particle size of 12.1,21 and 25.6 nm at temperature 500,1200 and 1300˚C respectively. The saturation magnetization M_s, remanent magnetization M_r and coercive force H_c, was founded to be equal to 0.183, 0.031 emu/g and 46.7O_e respectively. The current study could help in understanding the parameters affecting the dispersion and phase formation of cobalt species in silica matrix which indeed affect the activity of Co/SiO_2nano-composite as a catalyst in different processes and reactions. The A. C. conductivity increased by increasing the sintering temperature from 60 up to 1300˚C.Therefore, by increasing the sintering temperature of the silica gel network doped with Co_3O_4 can be considered as candidate for several electronic and industrial devices with improved dielectric charge storage capacity and strength.

Figure captions:

Figure 1(a-f) : The XRD patterns of the (a) S10C(60), (b) S10C(200),(c) S10C(300),(d) S10C(500),(e) S10C(1200) and (f) S10C (1300), respectively.

Figure 2(a-f): FTIR spectra of the (a) S10C(60), (b) S10C(200),(c) S10C(300),(d) S10C(500),(e) S10C(1200) and (f)S10C (1300), respectively.

Figure 3 (a , b): The FESEM images of the (a) S10C (60) and (b) S10C (1300), respectively.

Figure 4 (a-d): The TEM micrographs of the (a) S10C(60) , (b) S10C(500), (c) S10C(1200), and(d) S10C(1300), respectively.

Figure 5: Magnetic hysteresis curves for (a) S10C(60) , (b) S10C(300) ,(c)S10C(500), and(d) S10C(1300), respectively.

Figure 6: The RT ε`(ω) as a function of frequency for (a) S10C(60) , (b) S10C(300),(c)S10C(500), (d) S10C(1200), and(e) S10C(1300) , respectively, in the frequency range (42-10^6) Hz.

Figure 7: The RT ε``(ω) as a function of frequency for (a) S10C(60) , (b) S10C(300) ,(c)S10C(500), (d) S10C(1200), and(e) S10C(1300) , respectively, in the frequency range (42-10^6) Hz.

Figure 8: The RT σ_{ac} as a function of frequency for(a) S10C(60) , (b) S10C(300) ,(c)S10C(500), (d) S10C(1200), and(e) S10C(1300) , respectively, in the frequency range (42-10^6) Hz.

Table caption:

Table(1) The particle size

Ethics approval: The manuscript was approved by the Ethical Committee of Ain Shams University.

Consent to participate: Informed consents were obtained from Ain Shams University.

Availability of data and material: All data and material are available if required.

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Authors' contributions:

- **Reham Kamal Abd El Hamid,** Sharing in the data analysis, drawing the fingers, drafting the manuscript and preparing for the publication, and manuscript submission.
- **Inas Kamal Battisha,** Sharing in the data analysis, drafting the manuscript and preparing for the publication.
- **Naglaa Ahmad Mohamed Shahin,** Sharing in the data analysis, drawing the fingers, drafting the manuscript and preparing for the publication,

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