Laser Raman micro spectroscopy and high frequency dielectric properties of silica-xerogel loaded with different concentrations of α-Fe₂O₃

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Abstract

Sol-gel derived nano-composites silica-gel doped with different iron oxide concentrations at (3.5, 11, 33 and 37 mol. %) sintered at constant temperature at 1170°C, are illustrated in this work. Structure, surface morphology, high frequency dielectric and magnetic trends of the mentioned nano-composites analyzed and investigated through XRD, UV-Vis, TEM, FESEM and Magnetic hysteresis curves. Raman micro-spectroscopy gives rise to samples homogenous distribution. XRD illustrate that the Hematite (α-Fe₂O₃) is the optimum phase rather than the Maghemite (γ-Fe₂O₃) and Magnetite (δ-Fe₂O₃). Big amount of Hematite (α-Fe₂O₃) crystalline nano-particles are expected at 37 mol. % of Fe₂O₃. XRD confirmed that the average crystallite size increased from (25-31) nm with increasing the concentration of (α-Fe₂O₃) from 3.5 up to 37 mol%. The A.C. conductivity σ_ac increased by increasing the α-Fe₂O₃ content, according to the presence of oxygen vacancy and the crystalline size increases. The decreasing behavior in magnetization value (M_s) is related to the changes of the crystallite size, magnetic iron oxide particles and concentration. All analysis explains Hematite is extremely stable at ambient conditions and at high temperatures, so it is the better for particular applications.

Keywords: iron oxide; sol gel; laser Raman micro spectroscopy; Impedance analyzer (KEYSIGHT-E4991B).

1. Introduction

In modern research, transition metal oxide nanoparticles attracted a large attention because of their electrical and optical properties making them suitable for application in different kinds of opto-electronic devices [1], also have been at the heart of many dramatic advances in the material science [2], for these potential technological applications. Iron oxide has different phases, namely hematite (α-Fe₂O₃) anti ferromagnetic, maghemite (γ-Fe₂O₃) ferromagnetic and magnetite (δ-Fe₂O₃). Hematite (α-Fe₂O₃) is the most stable iron oxide under ambient conditions. It is a low cost non-toxic environment friendly material easily available in nature [3]. α-Fe₂O₃ shows antiferromagnetic (weakly ferromagnetic) at room temperature shows
Paramagnetic above temperature of $948\text{K}$. $\alpha$-$\text{Fe}_2\text{O}_3$ displays wide ranges of applications such as, catalysis, gas sensors, solar cells, magnetic resonance imaging, pigments, and spin electronic devices. Silica nanoparticles have gained importance in recent years because of their applications in various areas and easy synthesis process. Surface modification opens up the door for its future application in the field of biotechnology and medicine such as for cancer treatment, dental filling composites and drug delivery. The silica gels are suitable for this application due to their high porosity, inertness chemical, large accessible surface area and transparency, also Silica have also been used as cell markers, catalytic substrates, absorbents, and matrix fillers. $\text{Fe}_2\text{O}_3/\text{SiO}_2$, nano-composites have interesting properties and applications in catalysis, sensing and magnetism. The conventional materials change their thermal, optical, magnetic properties in nano form corresponding to surface area increased and quantum effect. Recently, the magnetic nano particles (ferrite nano materials) have much attention and show interesting properties in many fields such as data storage, optical fiber, biomedical and medical fields etc. Sol gel is mainly interesting method due to its low cost, high purity, short preparation time, and homogenous solution of doping element and magnify the excellent polycrystalline samples. Information about A.C. conductivity can be gained by studying the influences of frequency and temperature on dielectric properties of different nano-composites. The silica xerogel beads embedded with $\text{Fe}_2\text{O}_3$ nanoparticles used for more column stability to remove the product gases from the reactants without clogging the column unlike in the powder samples. The $\text{Fe}_2\text{O}_3$ nanoparticles immobilized on $\text{SiO}_2$ support is explored, anticipating that the $\text{Fe}_2\text{O}_3$ - $\text{SiO}_2$ interactions may prevent self-agglomeration of $\text{Fe}_2\text{O}_3$ nanoparticles.

The properties of $\text{Fe}_2\text{O}_3/\text{SiO}_2$ materials in fact, depend on the nature of the interaction between silica xero-gel and iron oxide. Increasing temperature and/or iron concentrations the prepared samples are assumed to cause changes in the bond lengths and/or bond angles of the structural silicate units within network. In this work, Silica doped with iron-oxide in different molar ratios 3.5, 11, 33 and 37 mol.%, were synthesized with the sol gel process sintered at $1170^\circ\text{C}$. The prepared nano-composite materials were characterized with laser Raman micro spectroscopy, X-Ray Diffract meter (XRD). The prepared samples compatibility was investigated by SEM. The dielectric parameters of the samples were studied for first time in a wide range of frequencies (1 MHz - 1 GHz). Finally, many efforts will be done to find the possibility to use the studied nano-composite prepared samples as promising candidates for sensors and magnetic devices.
2. Experimental

A composite of silica gel and different concentration of iron oxide was prepared by modified sol-gel process. By mixing tetra-ethoxy-saline, \((\text{CH}_3\text{CH}_2\text{O})_4\text{Si} \) (TEOS) (Aldrich, 98%), ethyl alcohol \((\text{CH}_3\text{CH}_2\text{OH})\), distilled water and HCL with molar ratios 0.28: 0.174: 0.028: 0.0823 respectively.\(^{19}\)

An aqueous solution of iron nitrate \((\text{Fe(NO}_3)_3-9\text{H}_2\text{O})\) was introduced in initial stage of the process with different concentrations (3.5, 11, 33 and 37 mol %) to doped the silica giving a nano-composites. These solutions were stirring for one hour. The clear sol was then poured into a Teflon beaker and allowed to gel in air. The gel was dried in an oven for one week by slowly raising the temperature up to 100\(^\circ\)C \(^{20}\), and then kept for 24 h at 150\(^\circ\)C. Heating steps between 50\(^\circ\)C and 1170\(^\circ\)C at each temperature for 30 min respectively. After the treatment at higher temperature the samples changed and looked like ceramics. The overall process can be written as:

\[
\text{Si(O-CH}_2\text{CH}_3)_4 + 4\text{H}_2\text{O} \rightarrow \text{Si(OH)}_4 + 4\text{C}_2\text{H}_5\text{OH}
\]

\[
\text{Si(OH)}_4 \rightarrow \text{SiO}_2 + 2\text{H}_2\text{O}
\]

3. Characterization techniques:

The X-ray diffraction (XRD) patterns for the prepared samples are recorded with a Philips X-ray diffract meter using mono chromatic CuK\(_\alpha\) radiation of wavelength 1.54056\(\text{Å}\) from a fixed source operated at 45 kV and 9 mA. Absorption spectra (200-1800 nm) were measured using a The Model V-570 UV/VIS/NIR spectrophotometer. The instrument specified by resolution 0.1 nm and wavelength accuracy ±0.3 nm (at a spectral bandwidth of 0.5 nm). The Raman single point measurement using Alpha 300 RA/S from Witec-Ulm Germany is used with 532 nm laser and 100X Ziss objective. The morphology of the prepared samples was depicted by using high resolution field emission gun quanta FEG 250 scanning electron microscope (FE-SEM). The FE-SEM gives information on the samples surface morphology. Dielectric properties were measured in a wide range of frequencies between 1 MHz and 1 GHz using network impedance analyzer (KEYSIGHT-E4991B).

4. Results and discussion

X-Ray diffraction (XRD) characterization:

The composition and crystalline phase purity of silica gel doped with different iron oxide concentration at (3.5,11,33, and 37 mol %) sintered at 1170\(^\circ\)C were examined by XRD
pattern in the 2θ range of 10-80 degrees are shown in Figure (1:a→d). In all the cases a hump due to amorphous silica phase is visible at 20 ~ 22 degrees (mineral name: crystobalite, tetragonal crystal system, ICSD card No. 47219) [21]. Also it is clearly seen that many diffraction peaks detected at 2θ⁰ = 22º, 26º, 28.4º, 31.9º, and 36.5º corresponding to α-Fe₂O₃ (mineral name: Hematite, Rombohedral phase, JCPDS card No. (98-017-3024)) indexed as (111),(120),(121),(031) and (201) respectively. Moreover, the figure illustrates some distinct characteristic peaks at 23.8º,32.8º,35.2º,40.2º,49.1º,53º and 62.5º, which are ascribed to α-Fe₂O₃ with preferred orientations (111),(122),(200),(132),(142),(204) and (026). Also, other very weak peaks are observed at 57.9º, 63.3º, 71.7º and 75.3º attributed to γ-Fe₂O₃ (mineral name maghemite, spinel structure with disordered vacancies, cubic crystal system) and Ε-Fe₂O₃ with preferred orientations as (035), (330),(235) and (401), respectively. By increasing the Fe₂O₃ content on the host silica gel, it has been reported that the peaks intensity increased and shifted to higher 2θ° at 37 mol % of Fe₂O₃ suggesting a growing of crystal domain size[25]. Moreover, it can be noticed that the principal tetragonal α-crystobalite peak is observed at 2θ°= 22º index at (111) increased and changed by increasing the Fe₂O₃ concentration due to the rearrangement acting in the structure as a result of doping with iron oxide. The obtained data revealed that the hematite α-Fe₂O₃ is the major phase and a very minor component of γ-Fe₂O₃ and Ε-Fe₂O₃ in all the samples, pointing to high purity and succession of the nano-composite samples preparation. It has been noticed that the annealing at high temperature 1170°C and increasing the concentration gives rise to the crystallization into the more stable α-Fe₂O₃. The average crystalline sizes calculated using Scherer’s equation [26] from the principle peak were found to be equal to 25,26, 27 and 31 nm for 3.5,11,33 and37 mol%, respectively. One can conclude that the Hematite (α-Fe₂O₃) phase is the dominant phase in all samples. Lattice parameters of the synthesized of α-Fe₂O₃,Ε-Fe₂O₃, and γ-Fe₂O₃ nanoparticles are tabulated in Table 1.
Figure 1. The XRD of silica gel doped with different concentration of Fe$_2$O$_3$ (a) 3.5,(b) 11, (c) 33and(d) 37mol%, respectively sintered at 1170°C.

Table 1: Crystallographic data obtained from XRD of α-Fe$_2$O$_3$ ε- Fe$_2$O$_3$, and γ-Fe$_2$O$_3$

<table>
<thead>
<tr>
<th>property</th>
<th>Hematite</th>
<th>Magnetite</th>
<th>Maghemite</th>
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<tr>
<td>Molecular formula</td>
<td>α-Fe$_2$O$_3$</td>
<td>ε- Fe$_2$O$_3$</td>
<td>γ-Fe$_2$O$_3$</td>
</tr>
<tr>
<td>Crystallographic</td>
<td>Rhombohedral</td>
<td>Cubic</td>
<td>Cubic</td>
</tr>
<tr>
<td>Lattice parameter(nm)</td>
<td>a=0.508 ,b=0.878 ,c=0.947</td>
<td>a=0.939 ,b=0.939 ,c=0.939</td>
<td>a= 0.928 ,b=0.928 ,c=0.928</td>
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<tr>
<td>Relative percentage</td>
<td>44.6%</td>
<td>1.4%</td>
<td>1.3%</td>
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Applications

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<th>Applications</th>
<th>Catalysts, pigments, gas sensors</th>
<th>Solar energy conversion, drug delivery, environmental catalysis, recording devices</th>
<th>Magnetic recording, high frequency switch modes, electromagnetic absorbers</th>
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Crystalline size of α-Fe₂O₃

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<tr>
<th>mol%</th>
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<th>11 mol%</th>
<th>26 nm</th>
<th>33 mol%</th>
<th>27 nm</th>
<th>37 mol%</th>
<th>31 nm</th>
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<tbody>
<tr>
<td>3.5</td>
<td>25 nm</td>
<td>11 mol%</td>
<td>26 nm</td>
<td>33 mol%</td>
<td>27 nm</td>
<td>37 mol%</td>
<td>31 nm</td>
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<tr>
<td>11</td>
<td>26 nm</td>
<td>11 mol%</td>
<td>26 nm</td>
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<td>31 nm</td>
<td>37 mol%</td>
<td>31 nm</td>
<td>37 mol%</td>
<td>31 nm</td>
<td>37 mol%</td>
<td>31 nm</td>
</tr>
</tbody>
</table>

TEM and SEM analysis:

Transmission electron microscopy (TEM) of the α-Fe₂O₃ (3.5 mol%)/SiO₂ sample annealed at 1170°C are shown in figure (2). From the figure it is clearly exhibit that the SiO₂ particles are spherical with particle size in range ~ 30-50 nm. The small dots could be noticed, which may be due to α-Fe₂O₃ in form of spherical. The crystalline nano particle size of Fe₂O₃ is ~ 24 nm. This indicates the uniform dispersion of Fe₂O₃ grains on silica matrix.

Figure 2. TEM micrograph of silica gel doped with 3.5 mol% of α-Fe₂O₃, sintered at 1170°C.
High resolution Surface electron microscopy (FESEM) images of $\alpha$-Fe$_2$O$_3$ (37mol%)/SiO$_2$ nano-composite sintering at 1170°C is shown in figure(3). The mean grain size is in the nano-scale range, even at higher iron concentration calculated by the XRD. The SEM micro-graph shows a uniform grain size distribution, a fine monolith size and homogeneous microstructure appeared as shown in figure.3. In the graph, we can observe large particles were distribution over the bulk gel, which consists diffraction lines appear in the XRD patterns, whose peak positions are assigned to $\alpha$-Fe$_2$O$_3$ phase.

Figure 3. FESEM image of silica gel doped with 37 mol% of $\alpha$-Fe$_2$O$_3$, sintered at 1170°C.

UV-vis spectroscopy:

To estimate optical properties of the $\alpha$-Fe$_2$O$_3$ (3.5→37)mol%/SiO$_2$ sintered at 1170°C, the UV-vis absorption spectra in wavelength range (200-800)nm are showed in figure(4). The figure illustrated the $\alpha$-Fe$_2$O$_3$ have two absorption peaks in wave length ranges of the ultraviolet and visible light, respectively. The first region ranged from 238 to 353nm is assigned to the transfer spectra between metal and silica matrix, and the second region ranged from 500 to 575nm is the finger print region of hematite. Moreover, the absorption strength of the $\alpha$-Fe$_2$O$_3$ / SiO$_2$ increases with the increase of Fe$_2$O$_3$ content. These facts may be attributed to the transition in crystal field and the charge transfer process, which increased with increasing Fe$_2$O$_3$. Furthermore, the band gap energies of the $\alpha$-Fe$_2$O$_3$ / SiO$_2$ could be estimated by the following equation\[^{29}\]

$$E_g=1240 / \lambda_g \quad (1)$$

Where $E_g$ is the band gap energies of the $\alpha$-Fe$_2$O$_3$ /SiO$_2$ nanocomposite, $\lambda_g$ is the wavelength at the overlap of the vertical and horizontal portions on the band edge.
As seen in figure (4), the values of $\lambda_g$ is shifted to higher wavelength with increasing $\alpha$-Fe$_2$O$_3$ concentration, which shifts from 550 to 575 nm corresponding to band gap energy from 2.25 eV to 2.15 eV respectively. This result illustrated that the value of band gap of nanocomposite decreased with increasing $\alpha$-Fe$_2$O$_3$ nanoparticle, also the reduction in the band gap is due to increase in the grain size.$^{[30]}$

![Absorbance graph](image)

**Figure 4.** UV-vis Absorbance of silica gel doped with different concentration of $\alpha$-Fe$_2$O$_3$ (3.5→37) mol%/SiO$_2$, sintered at 1170°C.

**Raman spectrum**

Raman spectroscopy for silica gel doped with different concentration 3.5, 11 33 and 37 mol% of Fe$_2$O$_3$ sintered at 1170°C, is observed in Fig(5), respectively. The most pronounced Raman bands for the prepared samples are given and assigned in Table (1). The low intense peak appeared at about 1070 Cm$^{-1}$ is according to Si-O-Si stretching vibrating bond asymmetric. The small peaks at 820 Cm$^{-1}$ are attributed to the Si-O-Si symmetric stretching vibrating mode. The Raman spectra at 496 and 609 Cm$^{-1}$ corresponding to the presence of Si-O rings for 4 and 3-fold, respectively.

The appearance of the peak at 413 Cm$^{-1}$ could is attributed to Si-O-Si, bending oxygen mode. Band with sharp intensity located at 228 Cm$^{-1}$ is the fingerprint of the Raman spectrum
of α-cristobalite. These results are compatible with that obtained from XRD patterns and previous studies. The observed difference is the sharpness of the appeared peaks of samples, which may be due to the increase of the degree of crystallinity as indicated by XRD. Also, from the table (2), shows the assigned bands corresponding to α-Fe₂O₃ (hematite) phase[^31].

Table (2): Raman bands assignment of silica gel doped with different concentration of Fe₂O₃: 3.5, 11, 33 and 37 mol%, respectively sintered at 1170°C

<table>
<thead>
<tr>
<th>Raman bands</th>
<th>The assignment</th>
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<tr>
<td>SiO₂/Fe₂O₃</td>
<td>3.5 mol%</td>
</tr>
<tr>
<td>228 Cm⁻¹</td>
<td>223</td>
</tr>
<tr>
<td></td>
<td>292</td>
</tr>
<tr>
<td>430 Cm⁻¹</td>
<td>430</td>
</tr>
<tr>
<td></td>
<td>404</td>
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<tr>
<td>496 Cm⁻¹</td>
<td>496</td>
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<tr>
<td>609 Cm⁻¹</td>
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<tr>
<td>820 Cm⁻¹</td>
<td>820</td>
</tr>
<tr>
<td>1070 Cm⁻¹</td>
<td>1070</td>
</tr>
<tr>
<td></td>
<td>1311</td>
</tr>
</tbody>
</table>
Figure 5. Raman spectroscopy spectra of silica gel doped with different concentration of α-Fe$_2$O$_3$ (a) 3.5, (b) 11, (c) 33 and (d) 37 mol%, respectively sintered at 1170°C

Magnetic measurements:

The magnetic properties of α-Fe$_2$O$_3$ / SiO$_2$ nanoparticles using different concentration (3.5, 11, 33 and 37 mol%) of Fe$_2$O$_3$ sintered at 1170°C were drawn in Figure (6). The saturation magnetization (M$_s$), remnant magnetization (M$_r$) and the value of coactivity (H$_c$) are presented in Table (3). The values of the saturation magnetization (M$_s$) decreases from 0.813 emu/g to 0.0397 emu/g with increasing α-Fe$_2$O$_3$ from 3.5 to 33 mol%, respectively. This decreasing behavior in magnetization value is related to the changes of the crystallite size, magnetic iron oxide particles and concentration.\[32\] The results shows that the (M$_s$) increases up to 0.0659 emu/g for α-Fe$_2$O$_3$ (37 mol%)/SiO$_2$. This is attributed to the larger particle size of Fe$_2$O$_3$ embedded in silica matrix. The high (H$_c$) of the sample obtained at (37 mol%) of α-Fe$_2$O$_3$ may be attributed to the magneto static dipole interactions and / or the shape anisotropy in the high crystalline complex nanostructures in this work.\[33\] When α-Fe$_2$O$_3$ nano-particles embedded in silica, the Si-O-Fe bond has been formed. The magnetic moment of iron ions of α-Fe$_2$O$_3$ nanoparticles disappeared through the Si-O-Fe connection and as a result, the magnetization decreased.
Figure 6. Magnetic hysteresis curves of silica gel doped with different concentration of $\alpha$-Fe$_2$O$_3$ (a) 3.5, (b) 11, (c) 33 and (d) 37 mol%, sintered at 1170 °C

Table (3) The $(H_C, (M_r, (M_S)$ and $(M_r/M_S)$ of silica gel doped with different concentration of $\alpha$-Fe$_2$O$_3$ (a) 3.5, (b) 11, (c) 33 and (d) 37 mol%, sintered at 1170°C

<table>
<thead>
<tr>
<th>Sample</th>
<th>$(H_C)$ (kOe)</th>
<th>$M_r$ (emu/g)</th>
<th>$M_S$ (emu/g)</th>
<th>$M_r/M_S$</th>
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<tr>
<td>3.5</td>
<td>0.0387</td>
<td>0.2325</td>
<td>0.8138</td>
<td>0.2856</td>
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<tr>
<td>11</td>
<td>0.4641</td>
<td>0.03056</td>
<td>0.0458</td>
<td>0.6672</td>
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<tr>
<td>33</td>
<td>0.5477</td>
<td>0.02125</td>
<td>0.0397</td>
<td>0.5343</td>
</tr>
<tr>
<td>37</td>
<td>1.367</td>
<td>0.01593</td>
<td>0.0659</td>
<td>0.2417</td>
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</table>
Dielectric and Conductivity properties:

The AC conductivity $\sigma_{ac}$ of $\alpha$-Fe$_2$O$_3$ / SiO$_2$ nanoparticles using different concentration (3.5, 11, 33 and 37 mol%) of Fe$_2$O$_3$ sintered at 1170°C in frequency range (1MHz-1GHz) illustrated in figure(7). The figure shows that $\sigma_{ac}$ increases by increasing frequency. The observed conductivity in the lower frequency can be attributed to the weakly localized carriers which drift over large distances. At higher frequency the mean displacement of these carriers is reduced to show proximity in the conductivity $^{[34]}$. Also it can be noticed that, $\sigma_{ac}$ increased by increasing the Fe$_2$O$_3$ content, this may be due to the increasing in the mobility of the free charge carriers in silica matrix $^{[35]}$. The lower values of $\sigma_{ac}$ for Fe$_2$O$_3$ (3.5 mol%)/SiO$_2$ should be assigned to its particles size (25 nm), which leads to a decrease of the free ions number $^{[36]}$. However, the higher values of $\sigma_{ac}$ at higher iron oxide concentration (37 mol%) with particle size (31 nm)$^{[37]}$. Hence, it may be understood here that the particle size plays an important role in conductivity $^{[38]}$.

The dielectric constant ($\varepsilon'$) and Dielectric loss ($\varepsilon''$) of $\alpha$-Fe$_2$O$_3$/SiO$_2$ nano particles sintered at 1170°C were also studied in the frequency range (1MHz-1GHz) in figures (8, 9). It was observed that the ($\varepsilon'$) and ($\varepsilon''$) showed the same decreasing trend with increasing frequency. This observed decrease might be according to the charge carriers scattering and the electric filed fast variation accompanied with the frequency, which leading to random orientation of the dipole moments. It can be noticed that, ($\varepsilon'$) and ($\varepsilon''$) detect an increasing behavior by increasing $\alpha$-Fe$_2$O$_3$ content. This increase in dielectric constant may be attributed to the decrease of the distance between grains $^{[39]}$. The dielectric properties of ferrites are dependent upon several factors: including the method of preparation, chemical composition, grain structure and particle size$^{[40]}$. So the observed dielectric increasing behavior of our samples may be due to the increase in the particle size (25-31) nm of Fe$_2$O$_3$ concentration (3.5-37mol%).
Fig. 7: The $\sigma_{ac}$ variation against frequency of silica gel doped with different concentration of $\alpha$-$\text{Fe}_2\text{O}_3$ (a) 3.5, (b) 11, (c) 33 and (d) 37 mol%, sintered at 1170°C in frequency range (10$^6$-10$^8$) Hz.

Fig. 8: The $\varepsilon'(\omega)$ variation against frequency of silica gel doped with different concentration of $\alpha$-$\text{Fe}_2\text{O}_3$ (a) 3.5, (b) 11, (c) 33 and (d) 37 mol%, sintered at 1170°C in frequency range (10$^6$-10$^8$) Hz.
Figure 9. The $\varepsilon''(\omega)$ variation against frequency of silica gel doped with different concentration of $\alpha$-Fe$_2$O$_3$ (a) 3.5, (b) 11, (c) 33 and (d) 37 mol%, sintered at 1170°C in frequency range ($10^6$-$10^8$) Hz.

Conclusion

In the present work, the silica-gel glasses doped with different concentrations of Fe$_2$O$_3$, and their structural, dielectric, magnetic and morphological trends have been evaluated. The Silica-gel doped with different concentrations of Fe$_2$O$_3$ (3.5, 11, 33 and 37 mol. %) were prepared by sol gel process. The used process give rise to hematite $\alpha$-Fe$_2$O$_3$/SiO$_2$ phase annealed at 1170°C with homogenous distribution of the nanoparticles. In addition the X-ray measurements detect the hematite phase presence. Moreover, the iron oxide embedded in silica gel host material is always in nano-structure range. The SEM measurements confirm the nano-scale presence; it is enhanced by high $\alpha$-Fe$_2$O$_3$ concentration in the mentioned prepared samples. The Laser Raman is used to clarify the structural group's presence. The dielectric properties will be studied in frequency range (1MHz-1GHz) for first time. The obtained data revealed that the A.C. conductivity $\sigma_{ac}$ increased by increasing the Fe$_2$O$_3$ content at (3.5, 11, 33 and 37% mol. %), the dielectric permittivity increases. Finally, the values of the saturation
magnetization ($M_s$) decreases from 0.813 emu/g to 0.0397 emu/g with increasing $\alpha$-Fe$_2$O$_3$ from 3.5 to 33mol%, respectively. This decreasing behavior in magnetization value is related to the changes of the crystallite size, magnetic iron oxide particles and concentration. The all results supplied a new strategy to design and prepared the high – temperature photonic crystals, thermal protects and magnetic materials.

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- R.K. Abd El-Hamid, Sharing in the data analysis, drawing the figures, sharing in the manuscript drafting, preparing it for the publication, and manuscript submission.
- N.A.M. Shahin, Sharing in the data analysis, drawing the figures, sharing in the manuscript drafting, preparing it for the publication.

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الملخص العربي

التحليل الطيفي الدقيق بالليزر Raman وخصائص العزل الكهربائي عالية التردد للسيليكا xerogel محملة بتركيزات مختلفة من α-Fe₂O₃

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في هذا البحث تم تحضير مركبات SiO₂/3O₂Fe-α محملة بتركيزات مختلفة من اكسيد الحديد (Fe₂O₃/SiO₂) وتستمخها عند درجة حراره (1170°C) وتسخينها عند درجة حراره (1117°C). اظهرت نتائج حيود الاشعه السينيه وجود α-cristobalite بلورات سيليكا رباعية كما اظهرت وجود مراحل الحديد المختلفة وهي مع قليل من (γ-Fe₂O₃) و Maghemite (ξ-Fe₂O₃), Magnetite (ρ-Fe₂O₃). وبزيادة تركيز الحديد ظهرت قمم ضيقة وحاده دليل على زيادة بلورية العينه بزيادة نسبة الحديد. عند درجات الحراره المنخفضه تكون مراحل الحديد الموجودة هي (γ-Fe₂O₃) و Maghemite و Hematite. وحسب الحجم الجزئي للمركبات وجد انها تزيد من 25 إلى 31 نانومتر بزيادة نسبة الحديد. وباستخدام الميكروسكوب الاكتروني التالف (SEM) وجد ان متوسط الحجم الجزيئي للعينات 24 نانومتر مما يتفق مع نتائج Raman. بينما صور الميكروسكوب (TEM) الاكتروني الماسح وجد ان توزيع الجزيئات دون اي تكتلات ولها شكل بلورى رباعى -cristobalite α من خلال قياسات Raman للعينات المحضره وجد α-cristobalite و 1 MHz - 1 GHz XRD مما يتفق مع نتائج التوصيلية الكهربيه للمركبات في مدى تردد (1 MHz - 1 GHz). ودراسة الخصائص الكهربائيه للمركبات في مدى تردد (1 MHz - 1 GHz) مما يتكافئ مع زيادة مراحل الشحنات عبر العينه. أما تأثير تركيز الحديد في زيادة حركة حاملات الشحن داخل سلسلة السيليكا مما يؤدي إلى زيادة التوصيلية الكهربائيه وقياس خصائص العزل عند الترددات المنخفضه يكون في قيم 'ε' و 'ε'. وزيادة التركيز تؤدي إلى تقل قيمتهما وذلك لعدم قدرة حاملات الشحن على تبع زيادة في المجال الكهربائي. كما وجد ان قيم 'ε' و 'ε' تقل بزيادة تركيز اكسيد الحديد. وبدراسة نتائج زيادة نسبة الحديد على الخصائص المغناطيسيه للعينات يتمكح ان المغناطيسيه تقل بزيادة نسبة الحديد. ومن نتائج القياسات وجد ان ال Hematite هو أكثر انتهاج الحديد استقرارا في الظروف المحيطة وكذلك تحت تأثير درجات الحراره العالية لذلك يفضل استخدامه في معظم التطبيقات.