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journal homepage: [www.elsevier.com/locate/jalcom](http://www.elsevier.com/locate/jalcom)Structural, optical and electrical properties of sol–gel prepared mesoporous  $\text{Co}_3\text{O}_4/\text{SiO}_2$  nanocompositesGomaa A.M. Ali<sup>a,b</sup>, Osama A. Fouad<sup>c</sup>, Salah A. Makhlof<sup>d,e,\*</sup><sup>a</sup>Chemistry Department, Faculty of Science, Al-Azhar University, Assiut branch, Assiut 71524, Egypt<sup>b</sup>Faculty of Industrial Science and Technology, University Malaysia Pahang (UMP), 26300 Kuantan, Pahang, Malaysia<sup>c</sup>Central Metallurgical Research and Development Institute, CMRDI, P.O. Box 87, Helwan 11421, Egypt<sup>d</sup>Physics Department, Faculty of Science, Assiut University, Assiut 71516, Egypt<sup>e</sup>Deanship of Scientific Research, Al Imam Mohammad Ibn Saud Islamic University (IMSIU), Riyadh 11463, Saudi Arabia

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## ABSTRACT

Structures, optical and electrical properties of  $\text{Co}_3\text{O}_4/\text{SiO}_2$  nanocomposites are reported. Well crystalline  $\text{Co}_3\text{O}_4$  nanoparticles embedded in an amorphous  $\text{SiO}_2$  matrix is formed, and confirmed by XRD and FTIR measurements upon calcination of gel precursors up to 800 °C. The obtained nanocomposites have high surface area  $\sim 126\text{--}312\text{ m}^2\text{ g}^{-1}$ , and the  $\text{Co}_3\text{O}_4$  particle size was  $\sim 7\text{--}15\text{ nm}$ . The optical properties of the  $\text{Co}_3\text{O}_4/\text{SiO}_2$  nanocomposites indicate the presence of two energy gaps; both of them are smaller than those reported for the  $\text{Co}_3\text{O}_4$  bulk phase. The first is varied from 1.32 to 1.44 eV and the second one is varied from 1.76 to 1.87 eV depending on the particles size. DC conductivity was measured in the temperatures range 300–673 K. The activation energy for DC conduction varies with particle size. The conduction mechanism was suggested to be through small polarons and variable range hopping mechanisms, at high and low temperatures respectively.

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## 1. Introduction

The interest in nanocomposite materials fabrication arises from the combination of the unique properties of the guest phases and host matrices. Nanocomposites can have different functionalities depending on the nature of the matrix, and size and distribution of the dispersed materials. Recently, synthesis of nanoscale  $\text{Co}_3\text{O}_4$  has received extensive attention because of its thermal stability, chemical and magnetic properties [1,2]. Cobalt oxide/silica nanocomposites have been used as catalytic [3–5], gas and humidity sensing [6–9], optical [10,11], magnetic [12,13] and electrical [14,15] materials in various technological applications.

Synthesis routes play a crucial role in preparing the target product and determining its properties [16]. Various methodologies and routes have been used for synthesis and fabrication of these materials such as sol–gel [3,6,16–18], citrate-gel-Pechini [9], surface organic modification [4] and wet impregnation [5] techniques. The advantages of the sol–gel process are good composition, size control and homogeneity, low processing temperature and low cost [3,16].

Optical absorption studies on the multi-valence spinel tricobalt tetraoxide,  $\text{Co}_3\text{O}_4$ , has shown that it exhibits multiple band gap

energies 2.28 eV ( $\text{O}^{2-} \rightarrow \text{Co}^{2+}$ ) and 1.57 eV ( $\text{O}^{2-} \rightarrow \text{Co}^{3+}$ ) [14,19]. The distribution of cations on spinel  $\text{Co}_3\text{O}_4$  is shown to be  $\text{Co}^{2+}[\text{Co}^{2+3}\text{O}_4]^{2-}$ : where the cations inside parentheses are octahedral and those outside are tetrahedrally coordinated with oxygen ions [20]. Moreover, the electrical conduction in these p-type semiconducting materials normally occurs by the hopping of small polarons between two different valency states of the cobalt ions [15]. The basic conduction mechanism is due to transfer of electrons between cobalt ions in different valence states. The current carriers are highly localized in the matrix. The strong electron–lattice interaction results in the formation of small polarons which were described by the small–polaron theory [21].

In this paper, we report on sol–gel synthesis of  $\text{Co}_3\text{O}_4/\text{SiO}_2$  nanocomposites. Chemical composition, phase analysis, porosity, particle size, pore size distribution and specific surface area results are discussed and compared. The optical and electrical properties; and the conduction mechanism of these nanocomposites were also investigated.

## 2. Experimental procedures and techniques

## 2.1. Sample preparation

$\text{Co}_3\text{O}_4/\text{SiO}_2$  nanocomposites having different weight content of Co varied from 0 to 50 wt% were prepared by sol–gel method. Co–gelation process starting from alcoholic solution of cobalt nitrate and TOES was performed. The route is similar to that described in our previous work [16]. The formed gel was centrifuged, dried at 80 °C for 24 h and calcined at 400 °C to obtain  $\text{Co}_3\text{O}_4$  crystallites embedded in

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