Synthesis and Characterization of Cobalt Oxide Nanoparticles by Sol-Gel Method

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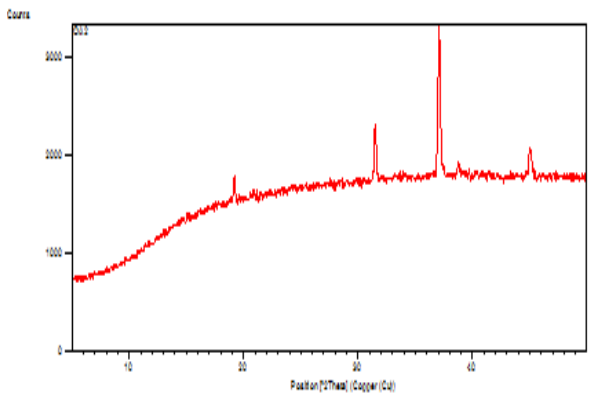
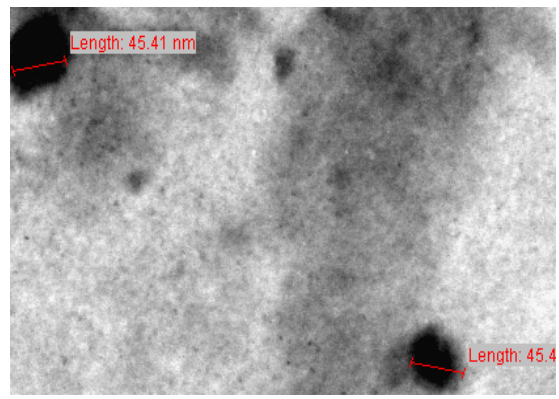
ABSTRACT

Co_3O_4 nanoparticles were prepared by Sol-Gel method using green chemistry. The cobalt oxide nanoparticles thus prepared were characterised by UV-visible, FTIR, XRD, and TEM techniques. FTIR spectroscopy was performed in order to know the synthesis condition. UV-visible spectroscopy was carried out for the optical characterization of metal nanoparticles. The average size of the Co_3O_4 nanoparticles was found to be 45 nm from XRD and TEM techniques.





Keywords: Co_3O_4, nanoparticles, sol-gel method, XRD, TEM.

1. INTRODUCTION

Nanotechnology is the science of production, manipulation and use of materials at subatomic level to produce novel products and processes [1]. In recent years, noble metal oxide nanoparticles have been the subject of focused research due to their unique electronic, optical, mechanical, magnetic and chemical properties that are significantly different from those of bulk counterpart [2-6]. Nanostructured materials have been widely investigated for the fundamental scientific and technological interests in accessing new classes of functional materials with unprecedented properties and applications [7-9]. Co_3O_4 is a very important material extensively used in catalysis, gas sensors, electrochromic films, battery cathodes, heterogeneous catalytic materials and magnetic materials [10, 11]. Co_3O_4 nanoparticles have been synthesized by various methods like sol–gel,

surfactant-mediated synthesis, thermal decomposition, polymer-matrix assisted synthesis and spray-pyrolysis [12, 13]. Co_3O_4 has a cubic spinel crystal structure in which the Co^{2+} ions occupy the tetrahedral sites and the Co^{3+} ions the octahedral site [14]. The Co^{3+} ions at the octahedral sites are diamagnetic in the octahedral crystal field. The Co^{2+} ions at the tetrahedral sites form an antiferromagneti sublattice with a diamond structure. The cobalt spinel compounds can act as efficient catalysts in a lot of heterogeneous chemical processes [15–18]. Nanoparticles of Co_3O_4 are promising materials for electronic devices [19], gas sensors [20], magnetic materials [21], electrochromic devices [22] electrochemical systems [23] and high-temperature solar selective absorbers [24].

In continuations to our earlier study [25-29], here we have reported the green synthesis of Co_3O_4 nanoparticles using sol-gel technique and its structural and optical characterization using UV-Visible, XRD, FT-IR and TEM techniques.

2. EXPERIMENTAL

2.1. Chemicals and Apparatus

All chemicals used in experiment were of analytical grade. The chemicals used in the synthesis were $Co(NO_3)_2.6H_2O$ and oxalic acid. All the solutions were prepared in double distilled water.

2.2. Synthesis of Co_3O_4 Nanoparticles

Metal salt solution was mixed in double distilled water with continuous stirring for 1 hour. In another beaker oxalic acid solution was prepared in double distilled water and continuous stirring was done for half an hour. The oxalic acid was then mixed into the metal salt solution drop wise with continuous stirring for three hours. The resultant light pink coloured precipitates thus obtained were washed with double distilled water and then dried at 100°C in oven for 5 hours. Finally, these were put into the muffle furnace at 600°C for 2 hours. Black colour Co_3O_4 nanoparticles were thus obtained.

2.3. Characterization techniques

The size, structure, morphology and magnetic properties of as prepared metal nanoparticles were characterised by FTIR (Thermo-USA, FTIR-380) in the wavelength range 400-4000 $cm^{-1,}$ UV-Visible spectroscopy (Shimadzu 1800) in the wavelength range 200-1000 cm^{-1}, XRD (Rikagu mini-2 using Cuα1, λ=0.15406 nm radiations) and Transmission Electron Microscopy (TEM).

3. RESULTS AND DISCUSSION

The average particle size was calculated from XRD data using Scherrer's equation. Particle morphology of the sample was investigated by a TEM. FTIR spectroscopy was performed in order

to know the synthesis condition and UV-visible spectroscopy was carried out for the optical study of metal nanoparticles. Figure 1 shows XRD pattern of the Co_3O_4 nanoparticles. The X-ray diffraction pattern revealed major peaks at 2θ values of 19.22 (111), 31.48 (220), 37.053 (311), 38.78 (222), 44.99 (400) corresponding to the simple cubic Co_3O_4 nanoparticles as confirmed by JCPDS card file 073-1701. Average particle size of the Co_3O_4 nanoparticles was found to be 44.89 nm using Scherrer's formula.

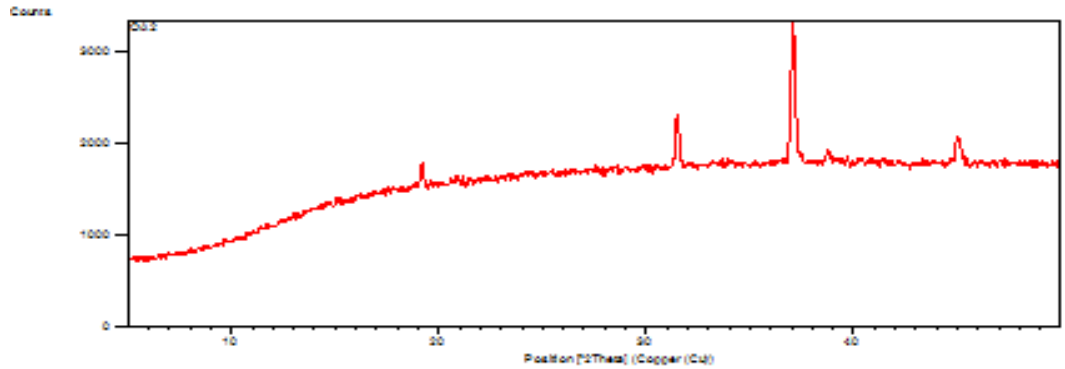


Fig. 1. XRD pattern of Co_3O_4 nanoparticles.

Figure 2 shows the TEM images of the Co_3O_4 nanoparticles. The microstructural characterization studies were conducted to determine the size of nanoparticles and examine the homogeneity and size distribution. It can be seen from the Figure 2 that there is a uniform distribution of particle size with mean particle size 45.4 nm which is in agreement with the XRD result.

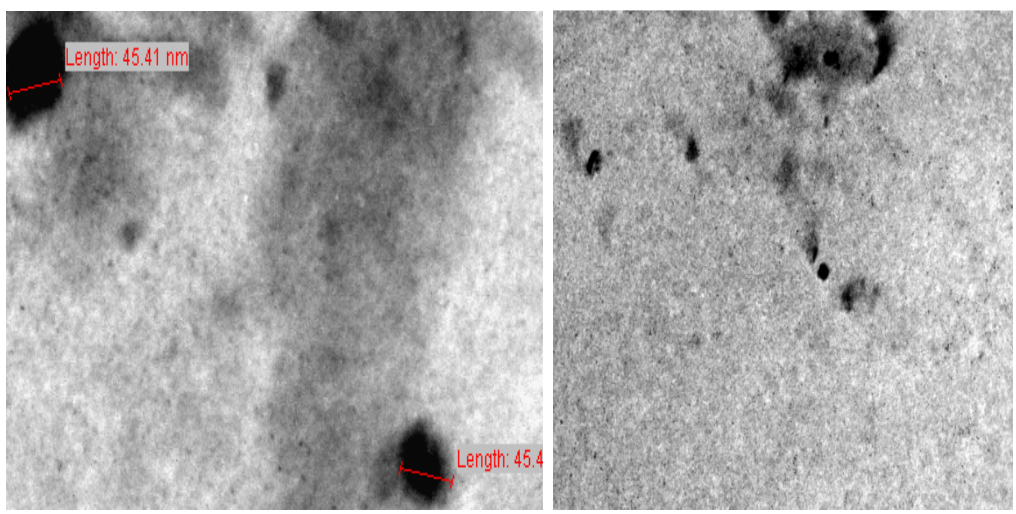


Fig. 2. TEM images of Co_3O_4 nanoparticles.

Figure 3 shows FTIR spectra of Co_3O_4 nanoparticles synthesized by sol-gel technique. FTIR spectroscopy was carried out in order to ascertain the purity and nature of metal or metal oxide nanoparticles. FT-IR spectrum of Co_3O_4 nanoparticles showed significant absorption peaks at 567 and 661 cm^{-1}. The absorption band at 567 cm^{-1} was assigned to Co-O stretching vibration mode [30] and 661 cm^{-1} was assigned to the bridging vibration of O-Co-O bond [31]. The absorption peak at 2926.01 cm^{-1} may be due to $–CH_3$ stretching vibrations. The absorption peaks at 2856.58, 2308.79, 1222.89 cm^{-1} may be due to $–CH_2$ stretching, =CH stretching and –CH stretching vibrations.

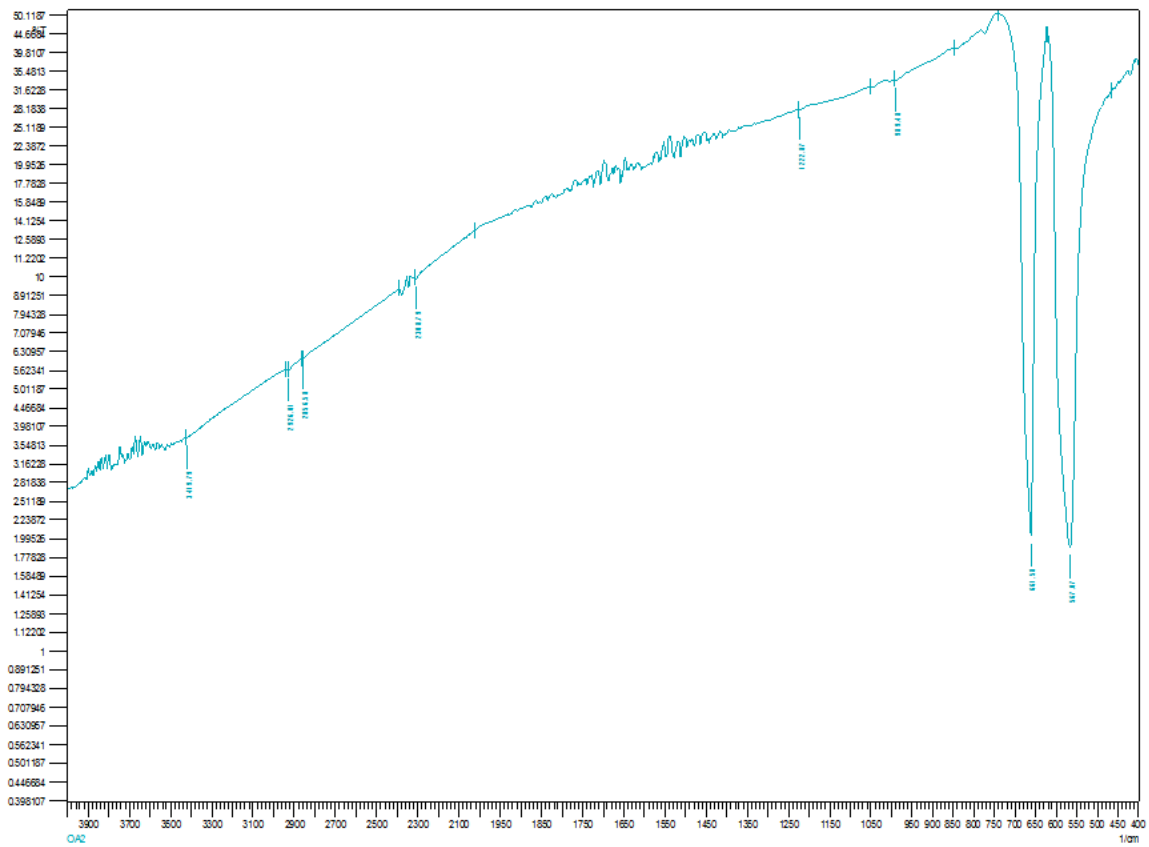


Fig. 3. FTIR spectra of Co_3O_4 nanoparticles.

The optical characterisation of the sample was recorded on UV-Visible absorption spectrophotometer. Figure 4 shows UV-Visible spectra of Co_3O_4 nanoparticles as a function of wavelength. The UV-Visible absorption spectroscopy of Co_3O_4 nanoparticles shows a absorption peak at about 339.60 nm.

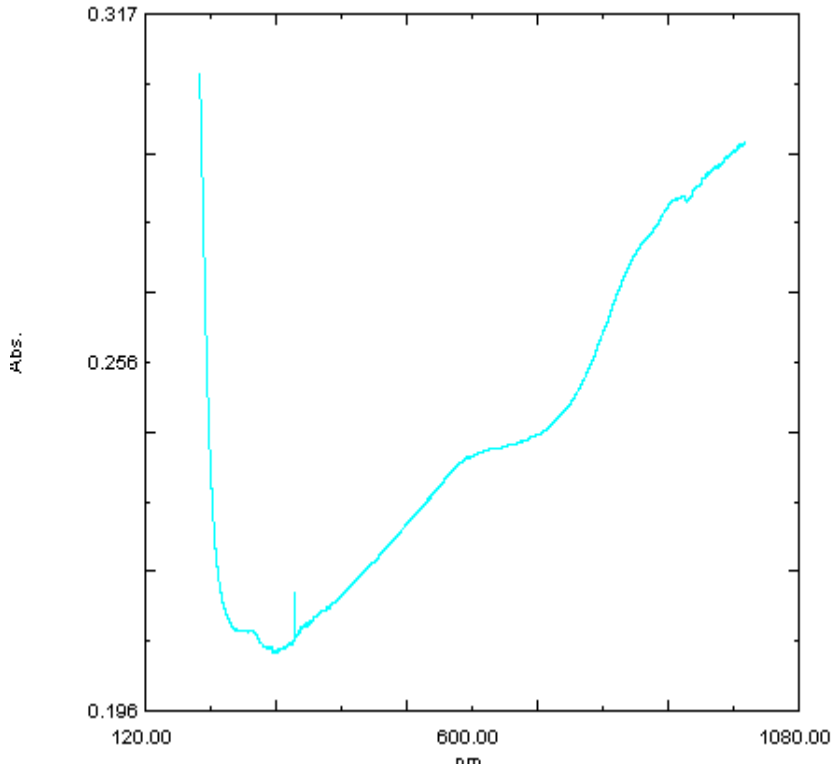


Fig. 4. UV-Visible spectra of Co_3O_4 nanoparticles.

4. CONCLUSION

Co_3O_4 nanoparticles with an average size of 45 nm were prepared by sol-gel technique using green chemistry. The cobalt oxide nanoparticles were characterized by using XRD, TEM, UV-visible. The XRD confirms the simple cubic crystal structure of the Co_3O_4. The optical absorption spectrum of cobalt oxide nanoparticles was studied by UV-Visible spectroscopy. The mean particle size determined by TEM is in close agreement with the XRD. The present synthesis method of nanoparticles by using green chemistry can be applied for large scale industrial production of Co_3O_4 nanoparticles thus protecting our environment from the use of harmful chemicals.

REFERENCES

[1] Porter Alan L, Youtie Jan, Shapira Philip, Schoeneck David J. *Refining search terms for nanotechnology,* J Nanopart Res 2008; 10:715–728.

[2] Mazur M. *Electrochemically prepared silver nanoflakes and nanowires*. Electrochemistry Communications 2004; 6:400-403.

[3] Feng L, Zhang C, Gao G, Cui D. *Facile synthesis of hollow Cu_2O octahedral and spherical nanocrystals and their morphology-dependent photo catalytic properties*.
Nanoscale Research Letters 2012; 7:276-280.

[4] Pal A, Shah, Devi S. *Preparation of silver, gold and silver-gold bimetallic nanoparticles in w/o micro emulsion containing TritonX-100*. Colloids and surfaces A: Physicochemical and Engineering Aspects 2007; 302:483-487.

[5] Rosemery M J, Thalappil, Pradeep. *Solvothermal synthesis of silver nanoparticles from thiolates*. Colloids and Surfaces A: Physicochemical and Engineering Aspects A 2003; 268:81-84.

[6] Xie Y, Ye R, Liu H. *Synthesis of silver nanoparticles in reverse micelles stabilized by natural biosurfactant*. Colloids and Surfaces A: Physicochem. Eng. Aspects 2006; 97:175-178.

[7] Lina Yu, Xie Ting, Cheng Baochang, Geng Baoyou, Zhang Lide. Chem Phys Lett 2003; 380:521-525.

[8] Manigandan R, Suresh R, Giribabu K, Vijayalakshmi L, Stephen A ,Narayanan V. Adv Mater Res 2012; 584:263-266.

[9] Amekura H, Umeda N, Takeda Y, Lu J ,Kishimoto N. Appl Phys Lett 2004; 85(6).

[10] Shiloms M I, Pshenichnikov A F, Morozov K I, Shurubor I Yu, J Magn Magn Mater 1990; 85:40-46.

[11] Bergemann C, Muller-Schulte D, Oster J, Brassard L A and Lubbe A S. J Magn Mater 1999; 194:45-52.

[12] Gruttner C, Teller J. J Magn Magn Mater 1999; 194:8-15.

[13] Guo L, Huang J, Li X Y, Yang S H**.** Phys Chem

[14] Mocuta C, Barbier A, Renaud G. Appl. Surf. Sci 2002; 56:162.

[15] Fornasari G, Gusi S, Trifiro F, Vaccari A. Ind. Eng. Chem. Res 1987; 26:1500.

[16] Natile M M, Glisenti A. Chem. Mater 2002; 14:3090.

[17] Fierro G, Ferraris G, Dragone R, Lo Jacono M, Faticanti M. Catal. Today 2006; 116:38.

[18] Wojciechowska M, Przystajko W, Zielinski M. Catal. Today 2007; 119:338.

[19] Cheng C S, Serizawa M, Sakata H, Hirayama T. Mater.Chem. Phys 1998; 53:225.

[20] Ando M, Kobayashi T, Iijima S, Haruta M. J. Mater. Chem 1997; 7:1779.

[21] Verelst M T, Ely O, Amiens C, Snoeck E, Lecante P, Mosset A, Respaud M, Brotom J M, Chaudret B. Chem. Mater 1999; 11:2702.

[22] Maruyama T, Arai S. J. Electrochem. Soc 1996; 143:1383.

[23] Wang G X, Chen Y, Konstantinov K, Yao J, Ahn J H, Liu H K, Dou S X. J. Alloys Compd 2002; 340:L5.

[24] Smith G B, Ignatiev A, Zajac G. J. Appl. Phys 1980; 51:4186.

[25] Rani R, Kumar H, Salar R K,Singh S. Purewal Int. J. Pharmaceutical Res. Development 2014; 06(01):72-78.

[26] Kumar H, Rani R. Int. J. Engg. & Innovative Tech 2013

[27] Kumar H, Rani R. Int. Letters of Chem., Phys. & Astronomy 2013; 14:26-36.

[28] Kumar H, Rani R, Salar R K Res. J. of Chem. Sciences 2011; 1(9)**:**1-7.

[29] Kumar H, Rani R, Purva Mimaansha 2011; 2(2):1-16.

[30] Estepa L, Daudon M. Biospectroscopy 1997; 3:347-369.

[31] Wu S H, Chen D H**,** J Colloid Interface Sci. 2003; 259:282-286.