INTERNATIONAL JOURNAL OF NATURAL AND APPLIED SCIENCES (IJNAS) VOL. 1, No. 1: pp. 81 – 83, 1 TABLE, 4 FIGS.

Synthesis and characterization of Co_3O_4 spinel.

B. I. Ita[1]

ABSTRACT

Co_3O_4 spinel has been obtained from cobalt (II) acetylacetonate by heating at 600°C in a blended solvent containing methanol and ethyleneglycol via a sol-gel technique. The Co_3O_4 has been found to possess a cubic structure having unit-cell parameters of a = b = c = 8.0773A°. The mean-particle size of the Co_3O_4 is 32nm and the Braunner-Emmett-Teller (BET) surface area obtained by nitrogen adsorption was 80m^2/g.

Keywords: Co_3O_4, particle size, cubic, sol-gel technique.

INTRODUCTION

Materials science is nowadays very much concerned with the synthesis of nanopolycrystalline spherical inorganic oxidic materials because these materials have great potentials for use in science and technology (Mahesh *et al*., 1992). One important problem faced by materials scientists and engineers is to be able to synthesize these oxidic materials with controlled microstructure to give the desired properties. It has been established that a particular material may have different magnetic, electrical and even structural properties, etc depending on its particle size which is a function of a particular method of preparation, starting material, temperature, etc. (Ita, 2000). Several methods of preparing oxides such as Co_3O_4 from various starting materials are available (Cansell *et al*., 1999). Co_3O_4 has been used as redox catalyst for processes in the gas phase by Antolini (1997). It has also been used as a precursor for the preparation of compounds like $Li_xCo_{3-x}O_4$ (Antolini, 1997; Rao, 1999), which is used for lithium battery. The present paper describes the synthesis and characterization of Co_3O_4 by sol-gel route from cobalt (II) acetylacetonate.

EXPERIMENTAL

Ethyleneglycol (10ml) was added to 0.07M (18g) cobalt (II) acetylacetonate solution in methanol (10ml). The mixture was stirred magnetically for 30min. to obtain a sol. The sol was dried at 140°C for 45min. for gelation to occur. The gel obtained was further dried for another 20min. after which the dried gel (xerogel) was then decomposed at 300°C for 20h. in a muffle furnace to give an amorphous powder of Co_3O_4. The powder was calcined at 600°C in a ceramic boat kept in a muffle furnace to produce nanocrystalline Co_3O_4. The chemical route of the synthesis can be represented as:

$$[CH_3COCH_2COCH_3]Co \xrightarrow[\text{Ethyleneglycol}]{\text{Methanol}} \text{Sol} \xrightarrow{140^oC} \text{Gel} \xrightarrow{300^oC} Co_3O_4.$$

The synthesized Co_3O_4 was characterized using X-ray diffraction (XRD) method. A Seifert 3000TT X-ray powder diffractometer with CuKα radiation (θ-θ geometry) was employed for the XRD measurement. The morphology and particle size were analyzed by employing a Leica S 440 i scanning electron microscopy (SEM) and electron diffraction (ED) images analyzed using a JEOL 3010 microscope. The Brunauer-Emmett-Teller (BET) surface area was obtained with the help of micromeritics Accusorb 2100E instrument.

RESULTS AND DISCUSSION

The XRD spectrum of the synthesized Co_3O_4 spinel is as recorded in Fig. 1. This spectrum is consistent with JCPDS file number 9-418. The appearance of peaks indicating the Miller indices shows the crystalline nature of the prepared Co_3O_4. This shows that the oxide powder contains crystallites which are not amorphous. The XRD spectrum of an amorphous material, do not show peaks. This crystalline nature of the oxide is further confirmed from the ED image of Co_3O_4 (Fig. 2) as white spots are observed. Again, amorphous oxides do not exhibit white spots on ED images. The presence of white spots signifies that the crystallites are able to scatter the incoming radiation, the spots representing the points occupied by the crystallites. Amorphous materials do not have such crystallites and therefore do not scatter radiation and no white spots are observed.

Lattice parameter refinement using the d-values and Miller indices from XRD, by the popular proszki computer program reveals that the prepared Co_3O_4 is cubic with a = b = c = 8.0773A°. The SEM (Fig. 3) reveals that the prepared Co_3O_4 has near-spherical particles less than

Manuscript received by the Editor October 22, 2004; Revised manuscript accepted September 8, 2005.
[1]Department of Pure and Applied Chemistry, University of Calabar, P.M.B. 1115, Calabar, Cross River State, NIGERIA.

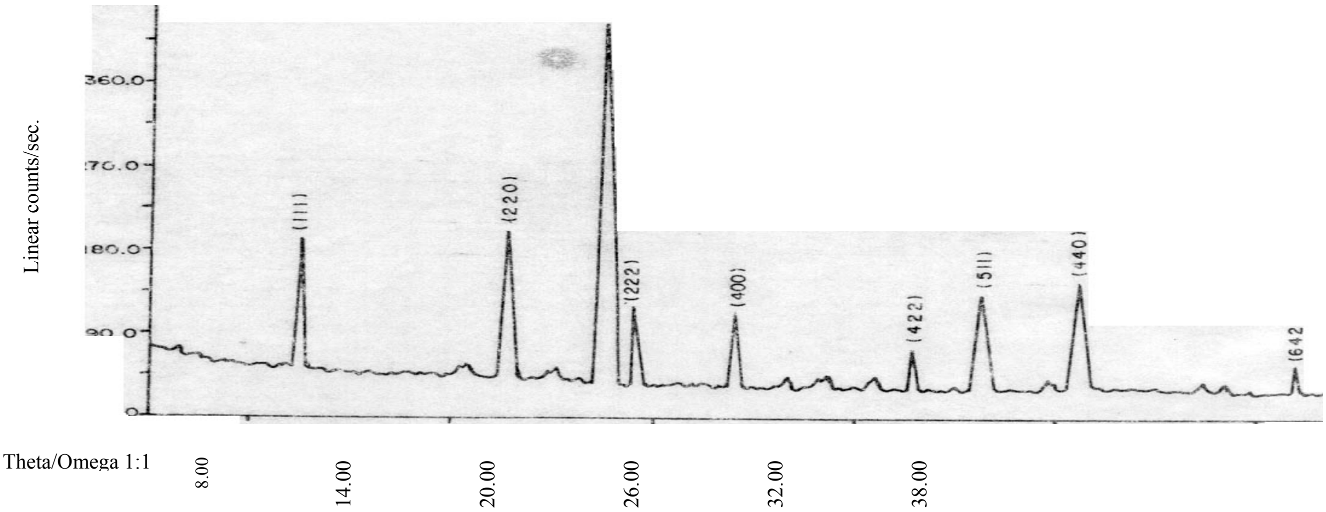

Fig. 1. The XRD spectrum of Co_3O_4 synthesized via sol-gel route

about 100nm. The mean-size of the crystallites is obtained as 32nm from the TEM (Fig.4) while the BET surface area was $80m^2/g$. Thus

Fig. 2. The ED image of Co_3O_4 synthesized via sol-gel route.

Table 1. Joint Committee on Powder Diffraction Standard (JCPDS) file number 9-418 for Co_3O_4.

System: Cubic.

d	I/I_1	hkl
4.669	20	111
2.869	40	220
2.430	100	311
2.333	12	222
2.021	25	400
1.6505	12	422
1.555	35	511
1.4293	45	440
1.0863	8	642
1.6524	16	731

the Co_3O_4 is a promising material in science and engineering as a result of the large surface area it contains. The large surface area will promote the adsorption characteristics of the oxide.

CONCLUSION

The Co_3O_4 has been synthesized from cobalt (II) acetylacetonate, methanol and ethyleneglycol via a sol-gel route. The synthesized

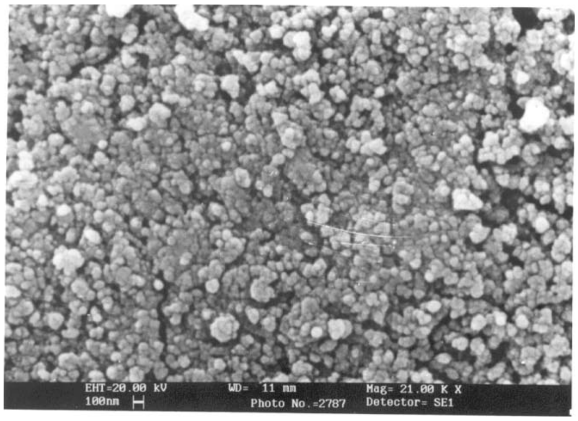

Fig. 3. The SEM image of Co_3O_4 synthesized via sol-gel route.

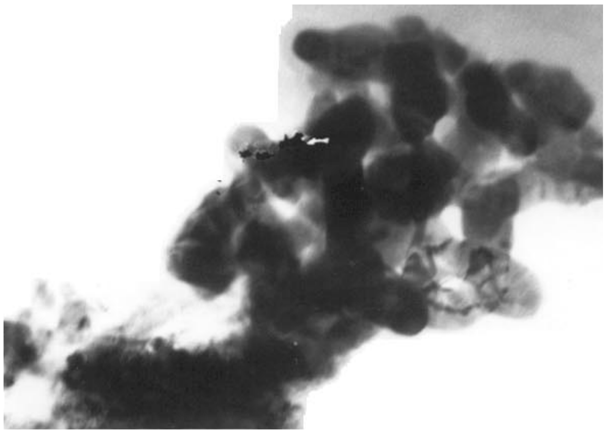

Fig. 4. The TEM image of Co_3O_4 synthesized via sol-gel route.

Co_3O_4 is a nanopolycrystal with cubic structure. The surface area of this oxide suggests that it may be used extensively in science and technology as a catalyst. A typical example of its use may be in the catalytic decomposition of hydrogen peroxide for the commercial production of oxygen gas.

ACKNOWLEDGEMENT

The thanks are due to TWAS for sponsoring the research and to Prof. C. N. R. Rao of JNCASR, Bangalore, India, for providing the facilities and discussions.

REFERENCES

Antolini, E. (1997). Formation of lithium-cobalt spinel oxide ($Li_xCo_{3-x}O4$) by solid state reaction of a Li_2CO_3-Co_3O_4 powder mixture. *Mat. Res. Bull.*, 32: 9-14.

Cansell, F., Cheralier, B., Demourgues, A., Etourneau, J., Even, C., Garrabos, Y., Pessey, V., Petit, S., Tresaud, A. and Weill, F. (1999). Superficial fluid processing: a new route for materials synthesis. *J. Mater. Chem.* 9(1): 67-75.

Ita, B. I. (2000). Combustion synthesis of nanocrystalline $LaFeO_3$. *^AC_H-Models in Chemistry*. 137: 671-676.

Mahesh, R., Kannan, K. R. and Rao, C. N. R. (1992). Giantmagnetoresi –stance effect in inorganic oxides. *J. Solid State Chem.*, 114: 294-297.

Rao, C. N. R. (1999). Novel materials, materials design and synthetic strategies: recent advances and new directions. *J. Mater. Chem.*, 9 (1): 1-14.