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Synthesis By Sol Gel Method And Characterization Of Co3O4 Nanoparticles.

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Abstract- A Co_3O_4 nanoparticles was synthesized by sol gel technique employed with appropriate reactants.in this case, we used metal salt and Tetrahydrofuran (THF) as starting material and the obtained product were calcined at 500^{0}C & 550 ^{0}C.the prepared nanoparticles were characterized by XRD, SEM, EDAX . Particle structure was confirmed by x-ray diffraction analysis; size was determined by Scherrer formula. SEM analysis showed that prepared nanoparticles were in Nano regime, somewhat round shaped and interlinked with each other. EDAX analysis showed that composition obtained was near stoichiometry.

Keywords: Cobalt oxide , EDAX ,Nanoparticles, SEM ,Sol-Gel Method, XRD**.**

I. Introduction

Nanostructured materials have been widely investigated for the fundamental scientific and technological interests in accessing new classes of fundamental materials with unprecedented properties and applications. Due to their small size, nanoparticles exhibit novel material properties that are significantly different from those of their bulk counterparts.[1] Transition metal oxide such as Cobalt oxide Nano powder which is p-type of semiconductor is widely used in many field such as Magnetic, Gas sensor, Lithium batteries ,Catalysis and Electrochemical based on size, structure, shape, phase & surface morphology[2,3,4]. Co_3O_4 nanoparticles were successfully synthesized by various methods like Sol-gel[4],Thermal method[5],Precipitation methods[6],Pyrolysis process[7],and Sono chemical method[8].

In this study, we have reported the synthesis of Co_3O_4 nanoparticles using Sol-gel method, characterized its structural, morphological properties using UV-visible,XRD,SEM with EDAX.

II. Experimental

2.1 Chemicals And appratus

All the chemicals used in the experiment were of analytical grade. The chemical used in the synthesis were cobalt salt, THF and Ammonia. All the solutions were prepared in double distilled water.

2.2 Synthesis of Co_3O_4 Nanoparticles

Metal Salt was mixed in double distilled water with THF continuous stirring for half an hour. The 25% of ammonia was then mixed with homogeneous metal salt solution drop wise with continuous stirring ,till gray coloured homogeneous gel were obtained. The gel was kept overnight into the muffle furnace at 40^0C was turned into the black mass and followed by calcination at 500 & 550 ^{o}C . Black colour Co_3O_4 nanoparticles were thus obtained.

2.3 Characterization techniques

The size, structure and morphology of as prepared metal nanoparticles were characterized by X-ray diffraction (XRD) and Scanning Electron microscopy (SEM) with Electron diffraction X-ray analysis (EDAX).

III. Result & Discussion

3.1 XRD Analysis

The XRD pattern of the find product obtained from the method as shown in Fig.1 (a,b).the XRD pattern indicate the monophasic fcc Co_3O_4 phase obtained by the Sol-Gel method at different calcination temperature. The XRD pattern revealed peaks at 2θ values of (111),(202),(311),(222),(400),(313),(422),(333),(404) can be perfectly indented to pure cubic phase with Fd-3m(227) space group and lattice constant a= 8.09 A^o reported in the literature (JCPDS073-1701)[9,10].the particle size of the investigated Co_3O_4 solid was calculated from line broadening analysis of some diffraction line of metal oxide phase using Sherrer equation. The inspection of these figure revealed that the Co_3O_4 solid prepared by this method and calcined at 500^{o}C &550^{o}C consist of crystalline Co3O4 phase which quality of crystallinity increases by increases of calcination temperature. The Co_3O_4 nanoparticles prepared from this method and calcined at 500^{o}C & 550^{o}C have crystalline average size67nm and 8nm respectively which calculated from XRD pattern and Scherrer equation. The size of the sample produced by the Sol-Gel method were calculated by XRD pattern and Scherrer equation implies that the Co_3O_4 nanoparticles produced at 550^{o}C is the best result obtained by this work.

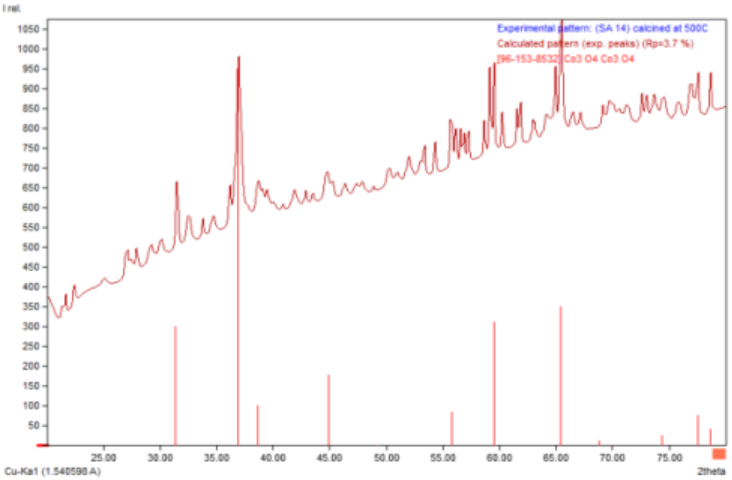

Figure 1(a): XRD pattern of Cobalt Nano particles were taken after calcination at 500 ^{0}C.

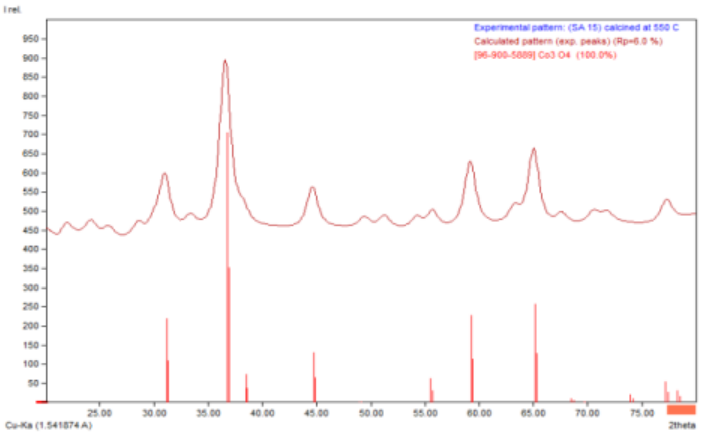

Figure 1(b): XRD pattern of Cobalt Nano particles were taken after calcination at 550^{0}C

3.2 SEM Analysis

The morphology and the structure of the product were characterized by scanning electron microscopy(SEM) Fig.2(a,b) shows the typical SEM image of Co_3O_4 nanoparticles with various size of 65.5nm, 66.1nm,67.6nm,70.2nm,72.8nm,89.8nm and 94.1 nm with different calcination temperature of 500 ^{0}C and 550 ^{0}C respectively ,which clearly conformed by XRD result. Fig 2 shows SEM images of nanosphere obtained from the method.the structure of nanosphere is shown obviously in fig. which its diameter suggest to be about 65.5 nm[2].

The morphology and structure of sample calcined at 550 ^{0}C [Fig2(b)] indicate that smallest nanoparticle size related to the sample calcined at 500 ^{0}C [Fig2(a)].

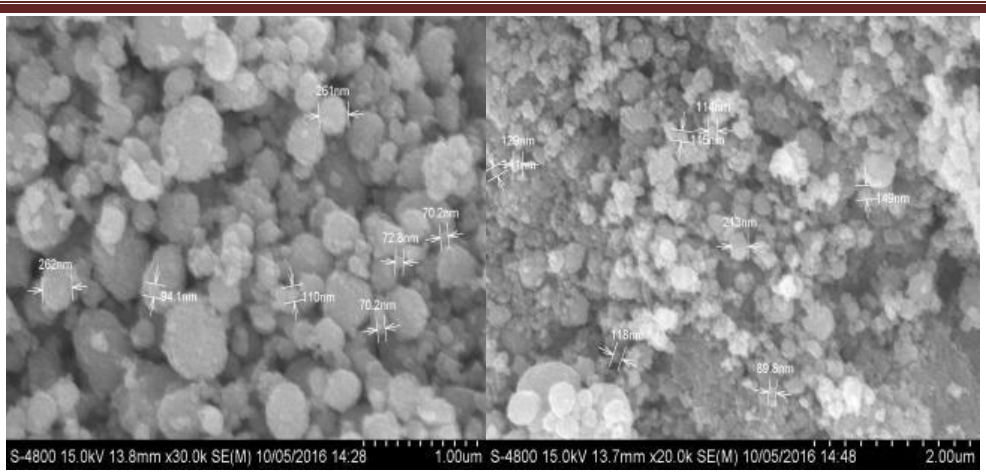

Figure 2(a): SEM image of Cobalt Nano particles were taken after calcination at 500 ^{0}C

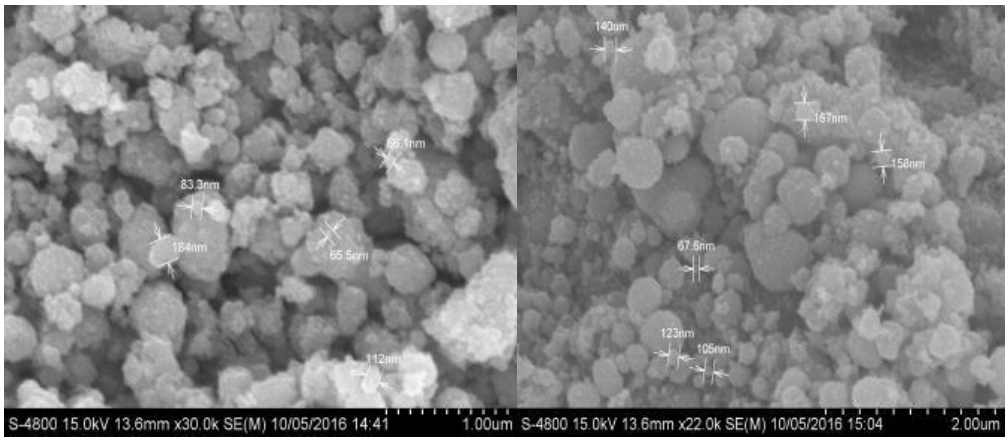

Figure 2(b): SEM image of Cobalt Nano particles were taken after calcination at 550 ^{0}C

3.3 EDAX Analysis

To check the chemical composition of synthesizes Co3O4 nanoparticles was measured by Electron Diffraction X-ray Analysis .The spectrum shows the strong X-ray peaks associated with Co and O were found in the EDAX spectrum as shown in fig.3.[10,11].

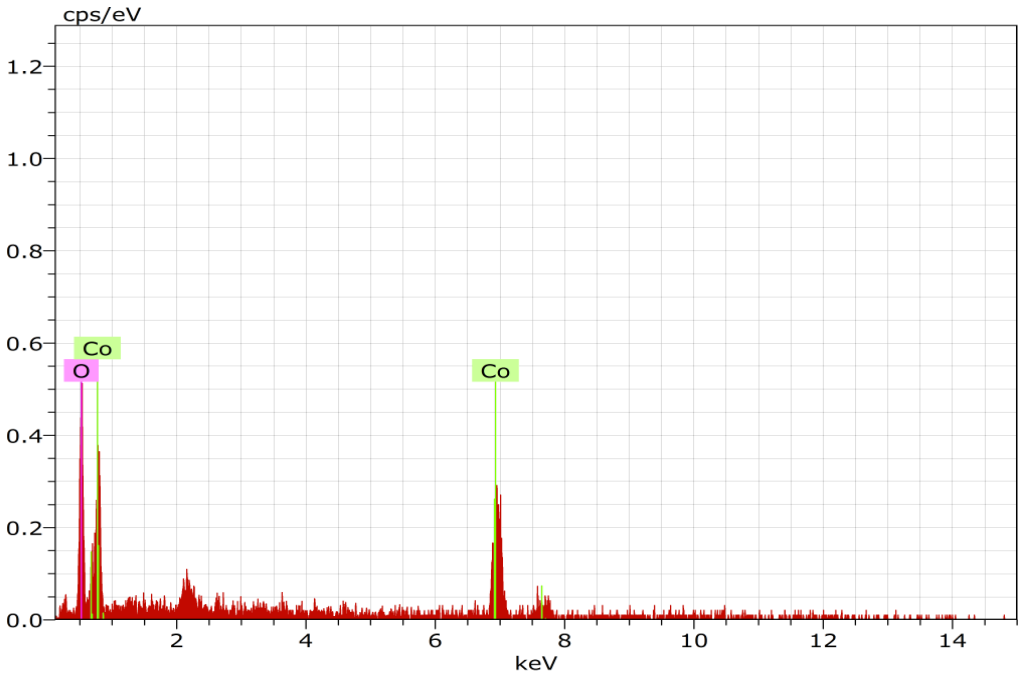

Figure 3: EDAX Spectrum of Cobalt Nanoparticles

IV. Conclusion

Nanocrystalline Co_3O_4 was prepared using Sol gel method at low temperature .X-ray diffraction pattern conformed the formation of (structure) of Co_3O_4. SEM & EDAX indicate that the obtained samples are sphere like morphology and the grain size is in the range of 65nm - 95nm.also the calcination temperature affect the morphology and reduce the surface area of nanoparticles with best calcination temperature 550 ^{0}C.

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