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Optical And Electrical Properties Of Synthesized Cobalt Oxide (Co_3O_4) Nanoparticles Using Thermal Decomposition Method

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ABSTRACT

In this research the Nanoparticles (NPs) of cobalt oxide (Co_3O_4) in the desired size range with a narrow size distribution has been prepared by chemical thermal decomposition of cobalt hydroxide. The cobalt oxide (Co_3O_4) was characterized by X-Ray Diffraction (XRD) and Energy-Dispersive X-ray spectroscopy (EDX). The resulting metal phase shows the diffraction pattern of cobalt oxide NPs in the desired size range with a narrow size distribution of average size (13-30nm), and further investigated by Transmission Electron Microscope (TEM). The surface morphology was characterized by Scanning Electron Microscope (SEM) images showing the topography of sample. The functional group of the sample has been characterized by Fourier Transform Infrared spectroscopy (FTIR). The optical properties were studied by UV-vis spectroscopy and electrical properties were obtained by Impedance meter (LCR).

Keywords: *: Co_3O_4; NPs; XRD; UV; SEM; TEM*

1. INTRODUCTION

Materials with features on the scale of nanometer often have properties dramatically different from their bulk scale counterparts [1-3]. Nanotechnology in recent years requires the miniaturization of devices into nanometer size while their ultimate performance is dramatically enhanced. This raises many issues regarding the new materials for achieving specific functionality and selectively [2, 4-6]. Nano structure, nano magnetism, and nano phase materials, a new branch of materials research, are attracting a great deal of attention because of their potential applications in areas such as magnetic data storage, optics, catalyses, ceramics, electronics and nanocomposites. The improved performance and the unique properties of nanomaterials are determined by their sizes, surface structures and interparticle interactions [4, 7-9]. Nanocrystalline materials are single phase or multiphase polycrystals, the crystal size of which is of the order of few nanometers so that about 40 to 80 % of the atoms are in the grain boundaries[1, 6]. Also in the recent years the interest of the scientific community in the catalytic behavior of metal nano-crystallites has increased tremendously. With decreasing crystallite size a higher number of surface atoms per gram of the active component/metal are exposed and available for reaction. In case of surface insensitive reactions the activity per gram should therefore increase with decreasing crystallite size [10, 11]. In magnetism for

a typical size of a magnetic domain of the order of 1mm and particles much smaller than that will be mono domain [7, 11]. A magnetic semiconductors are numerous, the spinel cobalt oxide Co_3O_4 is one of them and widely used catalyst for a variety of reactions[12]. And many efforts have been devoted to the synthesis of Co_3O_4 nanostructures with different morphologies such as hollow spheres, nano porous, nanospheres, cubic single crystals, fibers, nano particles, nano rods, nano plates, nano wires, nano tubes and nano cubes structures have been prepared [12, 13]. 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 [2, 12]. Co_3O_4 is a very important material extensively used in catalysis, gas sensors, electrochromic films devices, battery cathodes, ceramic pigments, solid-state sensors, solar energy absorbers, p-type semiconductor, energy storage, heterogeneous catalytic materials and magnetic materials [2, 12, 14].

2. MATERIALS and METHOD

To prepare Co_3O_4 NPs the following chemicals were used: n-hexane and acetone, Berol050 (Penta ethylene glycol dodecylether) cobalt nitrate (Co (NO3)2·6H2O) and 25wt.% ammonia solution.

2.1 Synthesis of Cobalt Oxide Nanoparticles:

The Co_3O_4 NPs has been prepared by some chemical method one of them is called thermal decomposition of cobalt hydroxide, and cobalt hydroxide prepared by mixing the Berol 050 and n-hexane and stirring for1h. Cobalt nitrate solution $Co(No_3)_2.6H_2O$ was added drop wise. Here in order to affect the size of the reverse micelle the water to surfactant ratio varied over a wider range while the ratio of surfactant to n-hexane kept constant, by addition of 25wt. % ammonia solution in a molar ratio of 4:1(NH_3 to cobalt) drop wise under stirring, precipitation of cobalt was achieved resulting in a quick color change from pink/orange to light green. The mixture was left under stirring for 30 min before break up micelle structure by added 500ml acetone drop wise. The residual surfactant was removed by wash extensively with acetone; the green precipitation was dried over night at 120℃ in air. A calcinations at 200℃ in air for 5h yields a Co_3O_4 powder[10].

3. RESULTS and DISCUSSION:-

Figure (1) showed FT-IR spectrum (Shimadzu, Thermo FT-IR 8400s instrument by using potassium bromide pellets) to determine a functional groups of Co_3O_4 NPs and the absorption peaks appeared at 3453, (1330-1600) cm^{-1} and 553, 661cm^{-1}. The Co-O expanding was found in absorption band at 553cm^{-1}. O-Co-O bond was found in absorption band at 661cm and dedicated to the bridging vibration of it. The weak band in the range (1330-1600) $cm^{-1}$is assigned to H-O-H bending vibration mode were submitted due to humidity. The band at 3453cm^{-1} is due to crystallization water. Energy-Dispersive X-ray (EDX) spectroscopy (JSM- 600 at 15kv) of Co_3O_4 NPs in figure (2) showed significant absorption peaks at 6.9, 7.7kev assigned to finding the cobalt element and absorption of weak peaks at 0.5, 0.78kev assigned to finding the Oxygen. The chemical composition of CoO NPs formation supported by EDX, the analysis fitting coefficients of cobalt and oxygen in Co_3O_4 NPs have been showed at ZAF Method Standard less Quantitative in table (1).

Phase structure of Co_3O_4 NPs was analyzed by Lab X Shimadzu model: 6000 diffractometer with CuK_{α_1} radiation ($\lambda = 1.5406\,Å$) at room temperature operated at voltage $50\,kv$ and current of $40mA$, the XRD patterns (particularly the pattern in 2θ = 37.39º) which assigned in Figure (3) shows the cobalt oxide has cubic phase structure. The relation between 2θ and relative intensities gives peaks at (2θ = 17.7º, 30.1º, 35.6º, 37.39º, 43.5º, 54.46º, 57.9 and 64.06º) imply to Co_3O_4 NPs has a cubic structure and no peaks of impurity. To calculate the average grain size of Co_3O_4 NPs using Scherer relation [15-17], and it was found to be about 30 nm.

$$\boldsymbol{d = \frac{0.9\lambda}{\beta \cos\theta}} \quad \ldots\ldots\ldots (1)$$

Whereλ is the wavelength of the X-ray used, θ is the Bragg angle, β is the full width at half maximum (FWHM) of the diffraction peak.

Figure (4): shows that the morphology and spherical agglomerated Co_3O_4 NPs was analyzed by SEM Scanning Electron Microscopy (JEOL, JSM- 600

operating at 15kv), also in Figure (4a and 4b) the particles take irregular morphology with different size particle, if the micrograph of the Co_3O_4 NPs calcinated at 200 °C. Therefore, the irregular morphology and agglomerate due to the magnetic induction between the particles

Table (1): ZAF Method Standard less Quantitative

Element	Kev	Mass	Atom	sigma	k
O k	0.525	13.88	37.25	0.06	10.4321
Co k	6.924	86.12	62.75	0.21	89.5679
Total		100	100		100

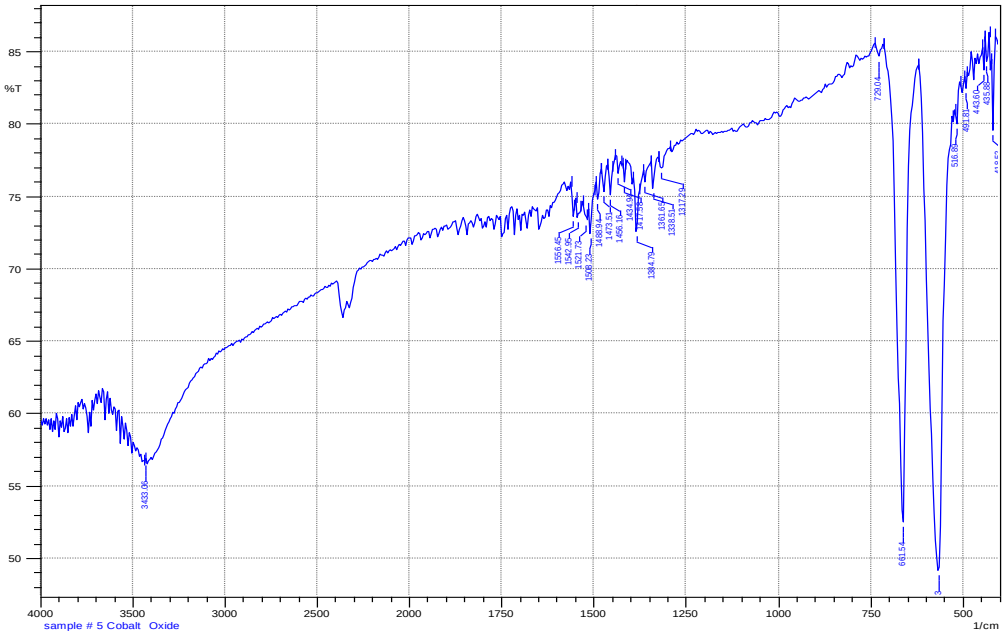

Fig (1): FT-IR spectrum of Co_3O_4

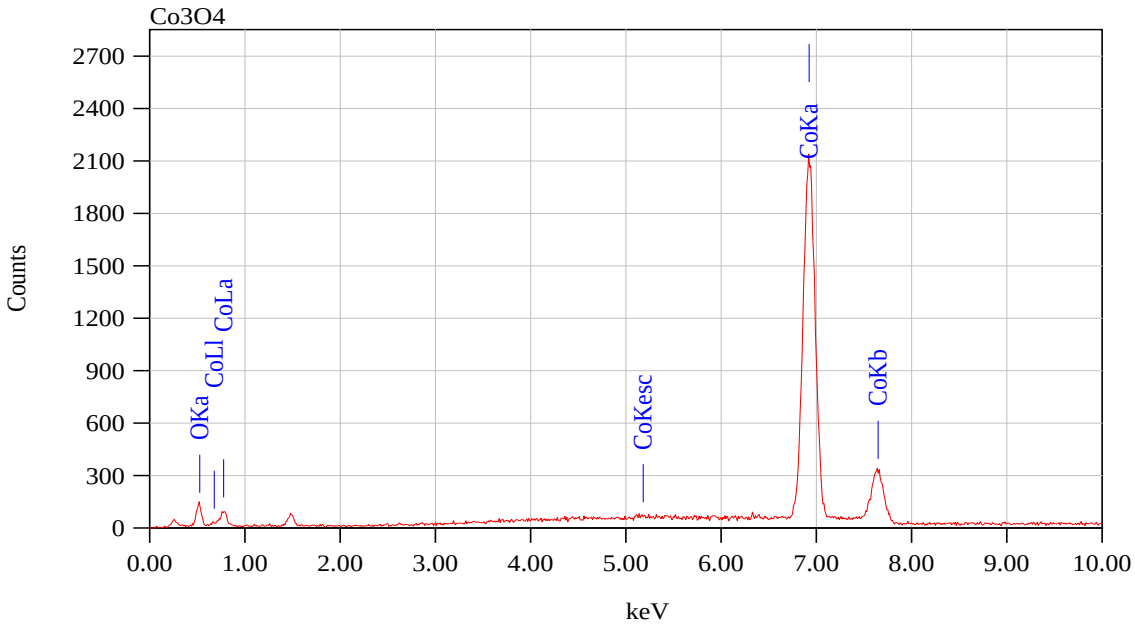

Fig (2): EDX spectrum of Co_3O_4

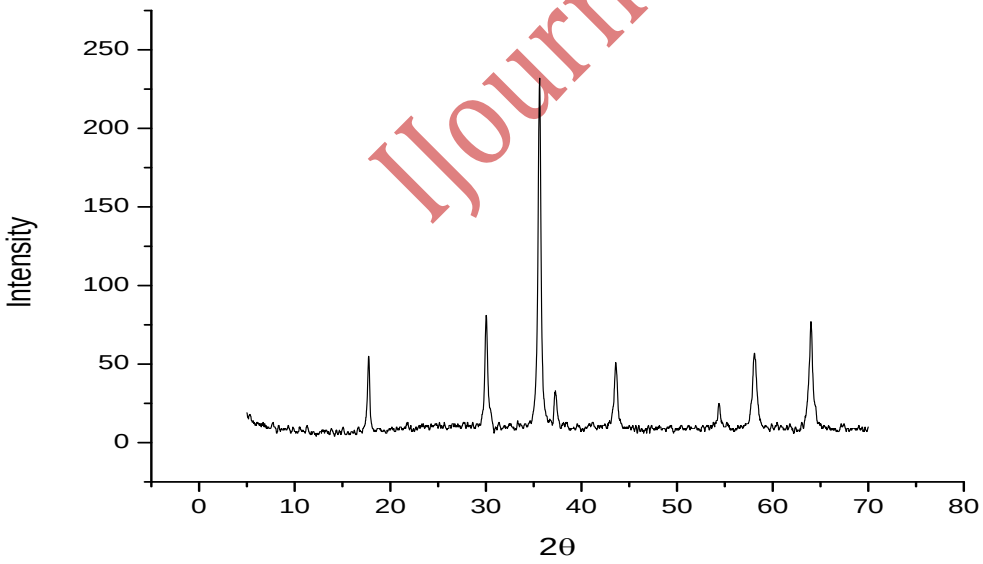

Fig (3) XRD of Co_3O_4 NPs

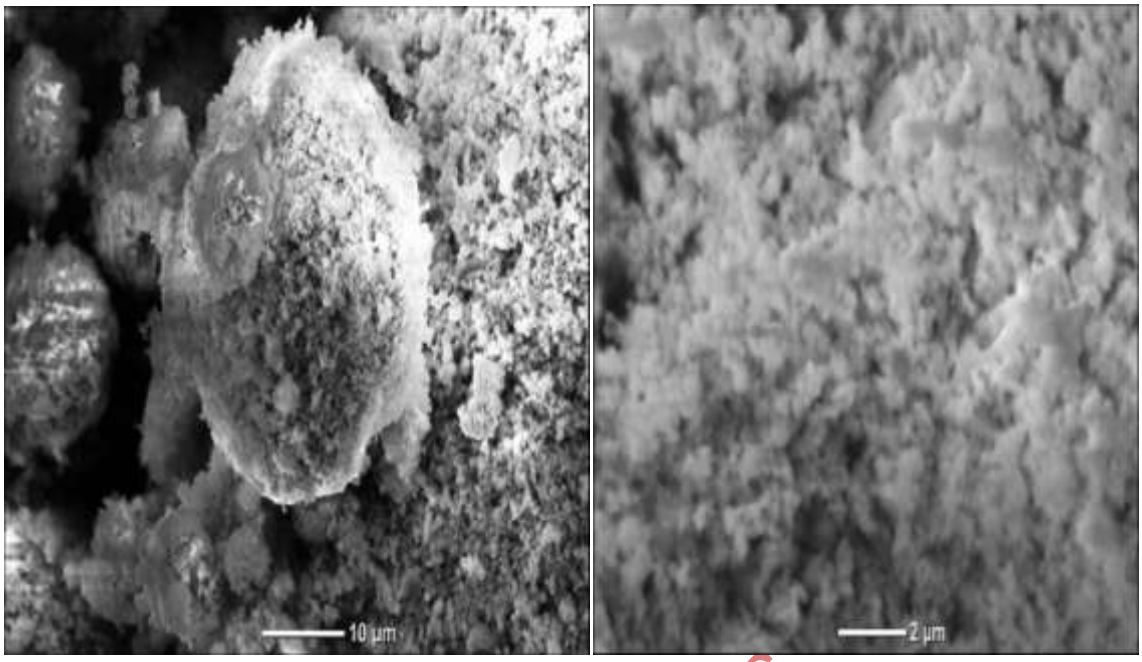

Fig (4): Show the SEM images of Co_3O_4 NPs (a) in 10 μm and (b) in 2 μm and

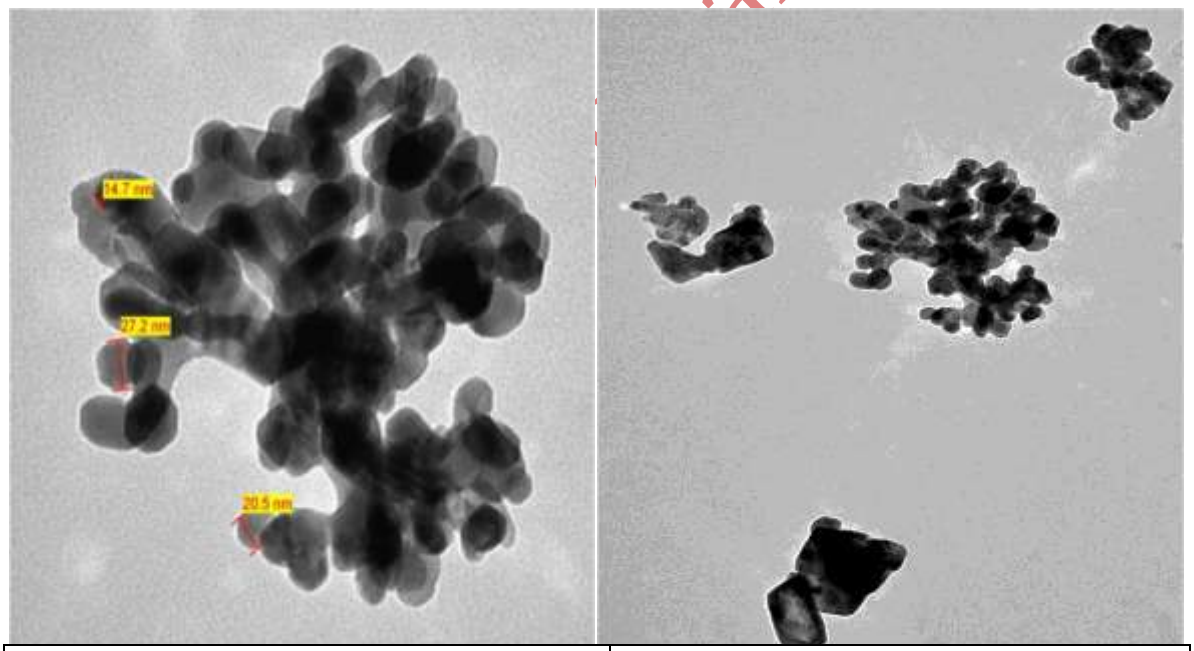

Fig (5): The particle size and nanographs of Co_3O_4 NPs in room temperature measured by using TEM (magnification=300000, model JEM 1400, operating at 90 kV, for 1h at room temperature)

The resin method used to prepare samples. The sizes of Co_3O_4 NPs appear from TEM image in both cases is of a very narrow distribution. The average sizes of Co_3O_4 NPs obtained from TEM about (14-30 nm) corresponding well with the results obtained from the XRD characterization.

To study the electrical properties of nanoscale experimental design impedance/dielectric spectroscopy has been used (LCR meter 8105G in a wide range of frequency (10Hz - 1MBHz) and voltage.). This technique enables us to characterize the real and imaginary components of the complex impedance and related electrical parameters of sample. Figure (6) showed that the complex impedance decrease with the increase of frequency as in the literature (17). Figure (7) Showed the electrical conductivity as a function of frequency of Co_3O_4 NPs where the main parameter of the electrical properties can be studied by the equation bellow[18]:

$$\sigma^* = {}^{1}/_{\rho^*} = \sigma^{/} + i\sigma^{//} \quad \ldots\ldots\ldots (2)$$

$$\rho^* = \rho^{/} + i\,\rho^{//} \quad \& \quad \rho^{/} = {}^{Z^{/}A}/_{d} \quad \& \quad \rho^{//} = {}^{Z^{//}A}/_{d} \ldots\ldots\ldots\ldots\ldots (3)$$

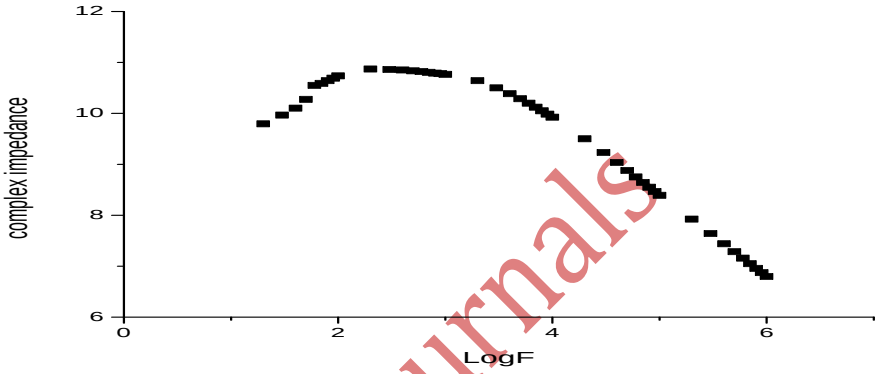

Fig (6): The complex impedance of Co_3O_4 NPs.

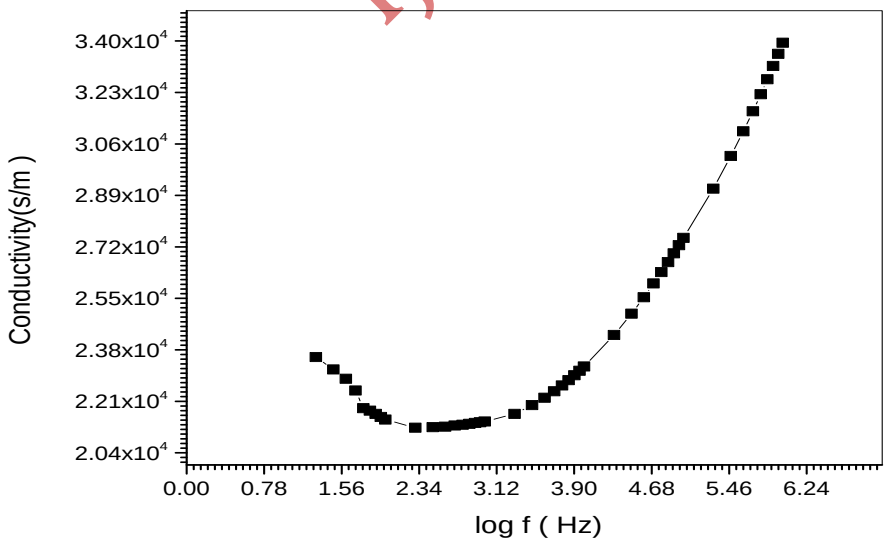

Fig (7): The conductivity of Co_3O_4 NPs.

Figure (8): shows the relation between optical absorption and wavelength of UV-*vis* spectroscopy of the Co_3O_4 NPs at room temperature and a sample has two absorbance bands in 192 nm and 197 nm wavelength ranges. Figure (9) shows energy band gap of the sample that relates to absorption coefficient and photon energy and can be determined by the Tauc relation [13, 17, 19]:

$$\boldsymbol{\alpha h\upsilon = B(h\upsilon - E_g)^r} \quad \ldots\ldots\ldots (4)$$

Where α , E_g , B and $(h\upsilon)$ is absorption coefficient, energy gap, constant for different transitions, energy of a photon respectively and r the index which assumes the values 1/2, 3/2, 2 and 3 depending on the nature of the electronic transition responsible for the reflection. Therefore, the band gap can be estimated by plotting a graph between h□ □ and$(\alpha h\upsilon)^2$and the straight line which intersect the h□ axes gives the value of band gap (2.85e.v) as in the standard measurement [13].

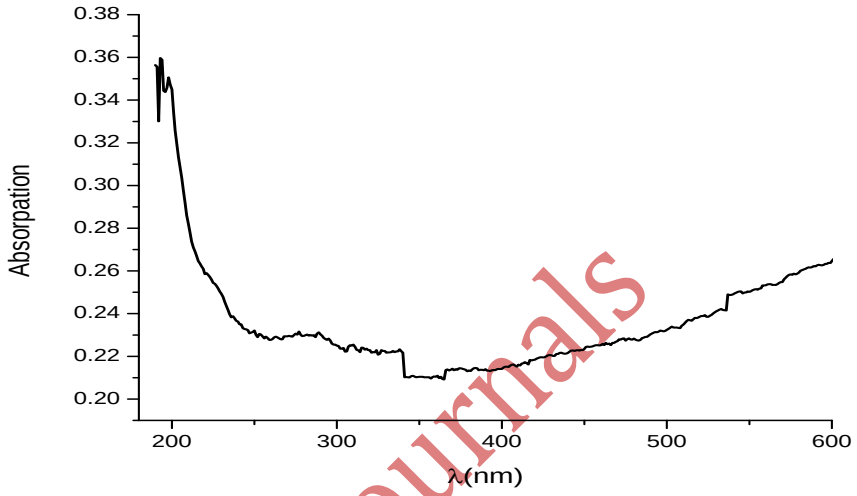

Fig (8): the absorbance spectrum of the Co_3O_4 sample

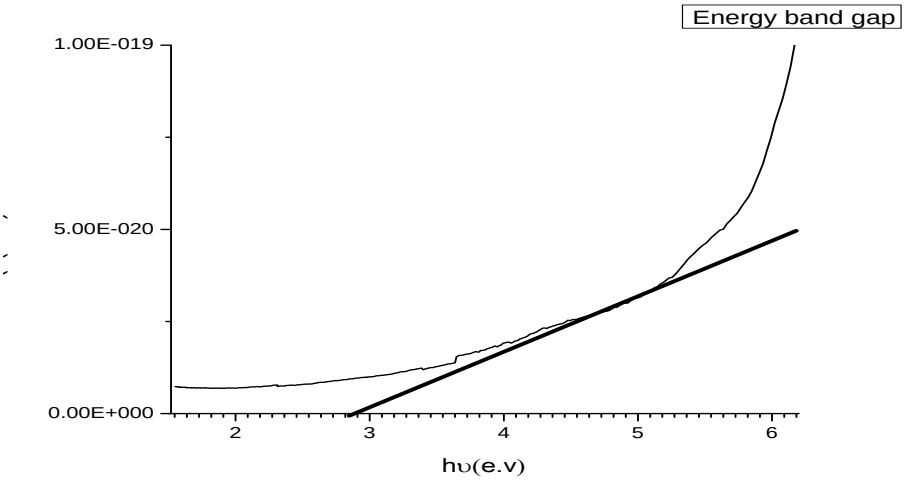

Fig (9): UV–*vis* spectrum and $(\alpha h\upsilon)^2 - h\upsilon$ curve of theCo_3O_4 Nanoparticles

4. Conclusion:

In summery the nanosized Co_3O_4 were synthesized by thermal decomposition method with narrow sized distribution in the range (13-30 nm).The formation of the Co_3O_4 NPs analysis by EDX and FT-IR spectroscopy is in agreement. XRD used to characterize the crystal structure of cobalt oxide nanoparticles at 200 °C. The spherical agglomerated particles of Co_3O_4 nanoparticles are shown by SEM and the average size was supported by TEM pictures. The optical absorption band gaps of the Co_3O_4 NPs were studied by UV–*vis* spectroscopy and were estimated to be approximately 2.85ev. The optical conductivity was studied by the impedance spectroscopy. This method is suitable to prepare a high-purity Co_3O_4NPs, low cost, safe, and simple for several applications.

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