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SYNTHESIS, CHARACTERIZATION AND GAS SENSING PROPERTIES CO_3O_4 AND NiO NANOPARTICLES BY THICK FILMS FABRICATION

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ABSTRACT:

Nanoparticles have wide application in the field of material science. These tiny particles have great surface area and inherent properties found to be applicable in various sections of science and technology. The present research focuses on the synthesis of nanoparticles such as Co_3O_4 and NiO. The nanomaterials of Co_3O_4 and NiO were prepared by Co-precipitation method and sol-gel method respectively, subsequently the materials were characterized by X-ray diffraction, from which the calculated size of Co_3O_4 and NiO by Scherrer formula found to be 30.17 nm and 10.88 nm respectively. These materials were also subjected to SEM analysis for studying the morphological properties. The EDS shows the basic elemental composition of prepared nanoparticles of Co_3O_4 and NiO. The average nanoparticles size found to be in the range of 30.0 nm to 64.4 nm. The material was also applied for gas sensitivity performance of ethyl alcohol and observed the sensitivity at 100^0C.

KEYWORDS:

Co_3O_4 and NiO, XRD, SEM and EDS

INTRODUCTION:

In the recent years nanomaterials are having vast applications in the field of research. Nanomaterials can be prepared by various methods like sol-gel method, co-precipitation method, hydrothermal method etc. These nanoparticles are used as a catalyst because of their large surface area. The metal oxides nanoparticles can be useful as gas sensors for most of gases. Metal oxides give huge surface zone those aides in catalytical utilization of materials. By and large, metal oxide thin and thick films [1, 2, 3, and 4] gave a decent record for gas sensors. Extensive quantities of gasses are tried for assortment of nanomaterials. Favorable position of gas detecting study gives affectability for different gasses, which manages their fixation at different spots, incorporates businesses, research facility, mining, air etc. Upkeep of contamination gasses and unsafe gasses can be sense at the specific level and temperature for various metal oxides.

Metal oxide dopants and their variety likewise gives great reactivity for chose gasses, dopants builds a few properties of metal oxides such as conductivity, electrical properties, affectability towards gasses surface range and reactivity, consequently the greater part of the analyst doped nanomaterial by a few move and inward move components to build the few properties as specified. In Both cases that is doped and undoped nonmaterial gives great catalytical applications as a gas sensors Ethanol (C_2H_5OH), an inflammable substance compound, is a standout amongst the most usually and generally utilized alcohols and has numerous applications in sustenance, biomedical, transportation and concoction enterprises and additionally wellbeing and security. Most extreme prescribed presentation level of ethanol as per the Occupational Safety Health and Administration (OSHA) set up to be 1000 ppm, Exposure to ethanol vapour brings about medical issues, for example, trouble in breathing, cerebral pain, languor, aggravation of eyes, liver harm etc. In like manner interest for ethanol recognition is high. [5, 6, 7, 8].

A large portion of alkali (NH_3) displays in the climate is radiated from concoction plants, developed farmland (manure), and engine vehicles. No wellbeing impacts have been found in people presented to run of the mill ecological convergences of NH_3. In any case, introduction to large amounts of smelling salts can bring about aggravation and genuine smoulders on the skin and in the mouth, throat, lungs, and eyes. The United States Occupational Safety and Health Administration has set an adequate 8h presentation restrict at 25 ppm by volume. While wellbeing and environment issues are characterized in ppm, NH_3 was found to influence semiconductor creation at ppb level. The ppb-level concentrated NH_3 can change the photochemical properties of photograph oppose and prompt to T-topping deformities. It is additionally photograph responsive and can store on optical surfaces of lithography frameworks bringing about fog. NH_3 is radiated into wafer prepare from different semiconductor forms including wafer cleaning, and additionally from human body [9, 10, 11].

MATERIALS AND METHODS:

All the in chemicals used in synthesis are of AR grade purchased from Merk chemicals Mumbai and used without further purification. Chemicals as Cobalt nitrate, cobalt sulphate, Nickel chloride, sodium hydroxide.

A] Synthesis of Co_3O_4 nanoparticles by Co-precipitation method:

For the synthesis of Co_3O_4 nanoparticles take 1:2 mole ratio of salts of cobalt i.e. dissolve 0.01 moles of cobalt nitrate and 0.02 moles of cobalt sulphate in minimum amount of double distilled water. Place this cumulative mixture of metal solution over the magnetic stirrer and heat this solution for 80^0 C with constant stirring up to half hour, add ammonium hydroxide solution to the same solution of metal and balance the PH up to 12 of the solution. The precipitate obtained is firstly washed with distilled water, then with ethanol, dried under IR lamp for half hour, and then calcined for the dry precipitate for 7-8 hours, dark brown coloured nanoparticles of Co_3O_4 are obtained.

B] Synthesis of nanoparticles of nickel oxide by Sol-gel method:

The NiO nanoparticles were synthesized by Sol-gel method utilizing absolute alcohol and sodium hydroxide. Nickel chloride Hexahydrate were firstly added into 70ml absolute alcohol with continues stirring at 80^0 C. Then 0.5 gm. NaOH was dissolved in 100ml absolute alcohol in another beaker. Then this solution was added drop wise into nickel chloride solution with continuous stirring for 2hours, light green coloured sol was formed. With the continue heating the sol was converted into gel. The gel was dried under IR lamp for 15-20 minutes then crushed in **mortal and piston** to get fine particles of NiO gel. Finally these particles then placed in crucible and heated 6-7 hours for 800 to 900 ^{0} C. The blacked colour nickel oxide nanoparticles are obtained.

C] Preparation of thick films of CO_3O_4 and NiO nanoparticles:

The powder nanoparticle of CO_3O_4 and NiO converted into paste form was used to prepare thick films by screen printing method maintaining the inorganic to organic materials ratio at 70:30. The inorganic part consists of nanoparticles of CO_3O_4 and NiO. The organic part consisted of 8% ethyl cellulose (EC) and 92% butyl carbitol acetate (BCA). The nanoparticles of CO_3O_4 and NiO mixed with ethyl cellulose (EC) were mixed thoroughly in an acetone medium with mortar and pestle. A solution of BCA which was added drop wise until proper thyrotrophic properties of the paste achieved. Now thick film was prepared on glass substrate by using standard screen -printing technique. The film was dried under IR lamp for 45 minutes to remove the organic volatile impurities and then fired at temperature 400 ^{o} C for 1.5 to 2 hours in muffle furnace. The prepared thick films are now ready for characterization.[12,13,14]

RESULT AND DISCUSSION:

A] SEM Analysis:

The Scanning electron microscopy (SEM) images of prepared Nanomaterial Co_3O_4 are as shown in **fig.1.** The images show surface texture, colour and porosity Co_3O_4 nanoparticles. It has heterogeneous surface, microspores and mesopores as seen from its surface micrographs. It is greyish black in colour, various sized nanoparticles imaged can be seen from SEM images as shown in **fig.1** [15]

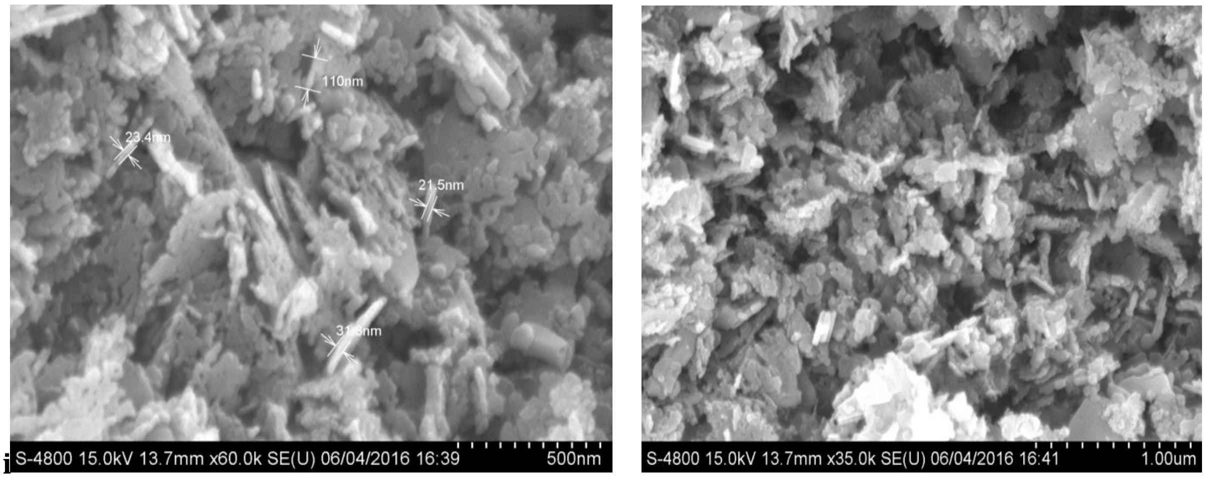


Fig.1. Fe-SEM Images of prepared Co_3O_4 nanomaterial

B] XRD analysis:

The XRD spectrum for prepared Co_3O_4 and NiO is as shown in fig.2and 3 respectively.The spectrum shows the main 2θ peaks at 31.2^0 ,For Co_3O_4 and $42.4^0$for NiO from which the average particle size calculated by Scherer formula Eq. (**1**) is 30.17 and 10.88 for Co_3O_4 and NiO respectively. $D = K\lambda/\beta \cos\theta$ ……………. (1)

Where K=constant (0.89 to1.39), λ=Radiation of wavelength (1.54 A^0), β=FWHM (Full Width Half wave Maxima), θ=Bragg angle in degree, D=Particle Size.[16].

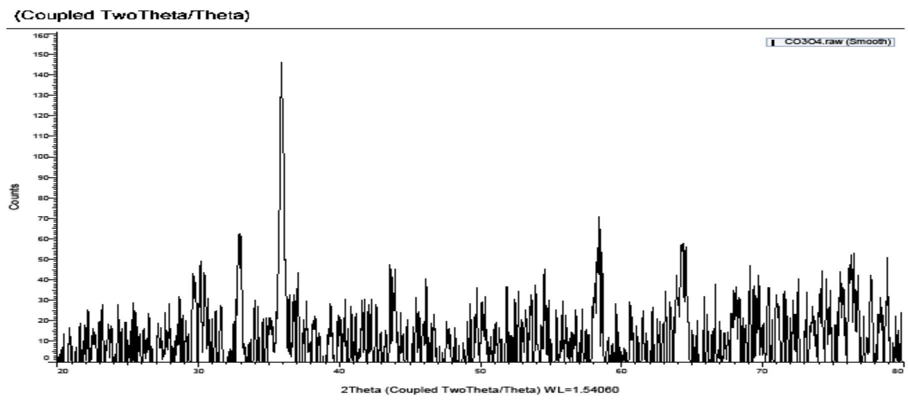


Fig.2.XRD spectra of $Co_3O_4$of nanoparticles

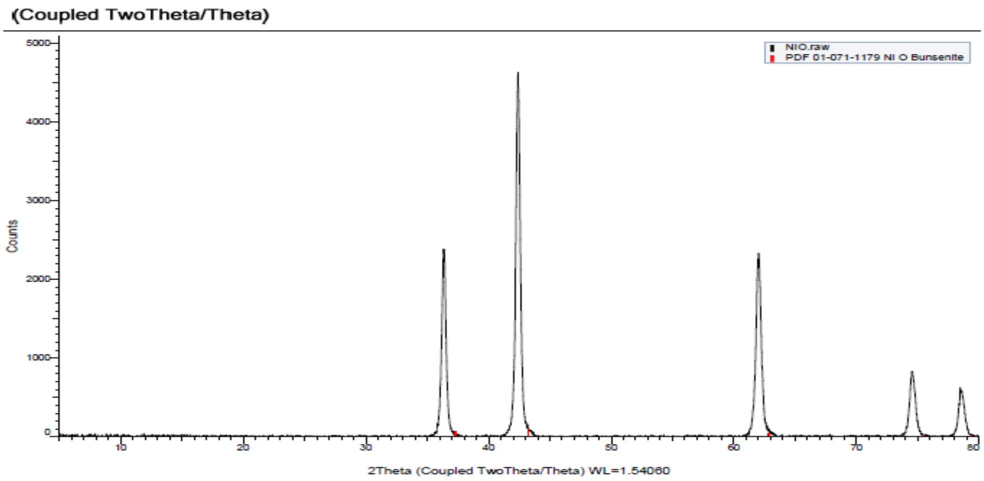


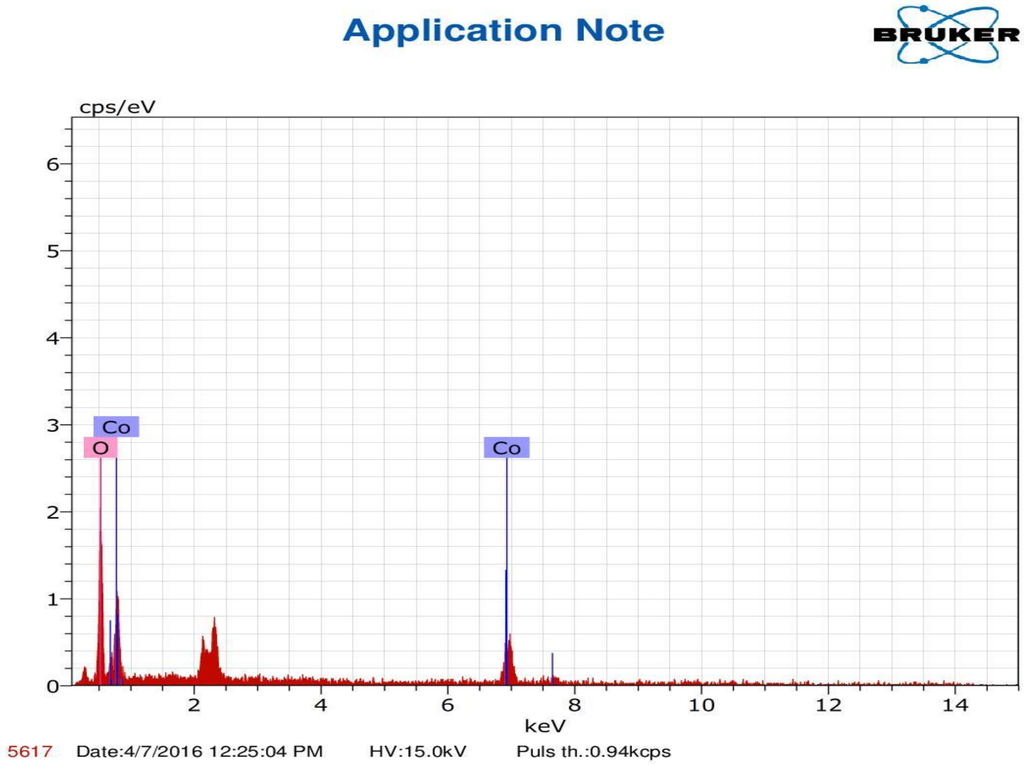
Fig.3 XRD spectra of NiO of nanoparticles

C] Elemental Analysis:

Electron dispersive X-Ray spectroscopy (EDS) is widely used technique to detect the elemental composition of metals. The spectrum is as shown in fig.4 from which one can get the elemental composition of synthesized material contains 69.47% of oxygen and 30.17 % of cobalt

S.N	Element	Elementary Weight % calculated from EDS
1	Oxygen	69.47%
2	Cobalt	30.17%

Table1:1 Percentage of elements and its elementary weight in synthesized Co_3O_4 Nanoparticles



El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
O	8	K-series	16.18	38.19	69.47	3.38
Co	27	K-series	26.18	61.81	30.53	1.30
		Total:	42.36	100.00	100.00	

Spectrum: 5617

El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
O	8	K-series	16.18	38.19	69.47	3.38
Co	27	K-series	26.18	61.81	30.53	1.30
		Total:	42.36	100.00	100.00	

2

Fig.4. EDS spectrum of synthesized Co_3O_4 Nanomaterial.

e) Electrical Characterization:

The D.C. resistance of the film samples was measured by using half bridge method as a function of temperature in home built measurement system .The home built characterisation system consists of glass chamber (25 litres) and heater (1000W) of nichrome wire (Resistance-120ohm at room temperature). The heater was used to change the film sample temperature from room temperature(RT) to 400^{0}C By changing its voltage using dimmer stat (maximum current limit upto-8A).The electrical terminals were brought out from the thick film resistor by using insulated feed-through mounted on the stainless steel base plate. The aluminium foil with pressure contacts system use for external contacts. The electrical resistances of thick film in air (*R*a) and in the presence of gas (*Rg*) were measured to evaluate the gas response (*S*) given by the relation. S = Ra-Rg/Ra

Electrical resistant of Co_3O_4 Thick films:

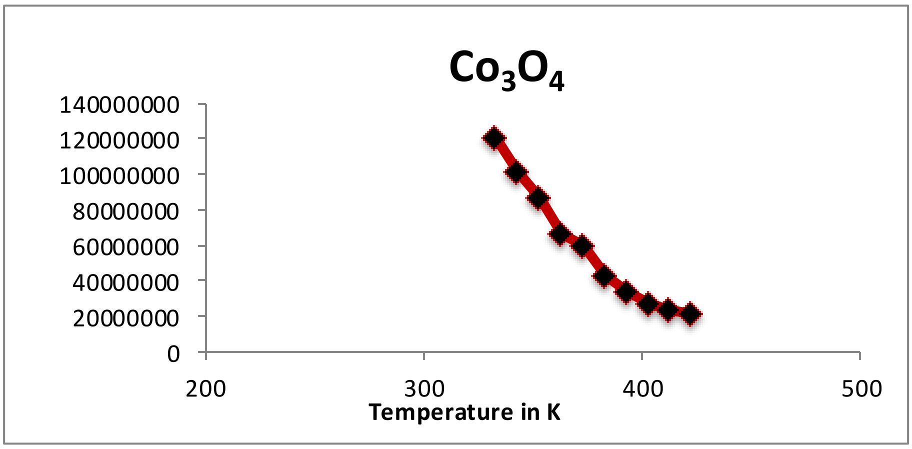


Fig. 5 showing graph of Electrical resistance V/s Temperature for Co_3O_4 thick films

Fig. 5 shows the graph of electrical resistance against temperature for prepared thick films of Co_3O_4.The optimum temperature ranging between 2 00 to 500 Kelvin. The graph shows semiconducting behaviour for prepared material of Co_3O_4 as the electrical resistance is decreasing with increase in temperature, showing a typical NTC semiconducting behaviour.

Electrical resistant of NiO Thick films: The graph shows semiconducting behaviour for prepared material of NiO as the electrical resistance is decreasing with increase in temperature, showing a typical NTC semiconducting behaviour shown in fig.6

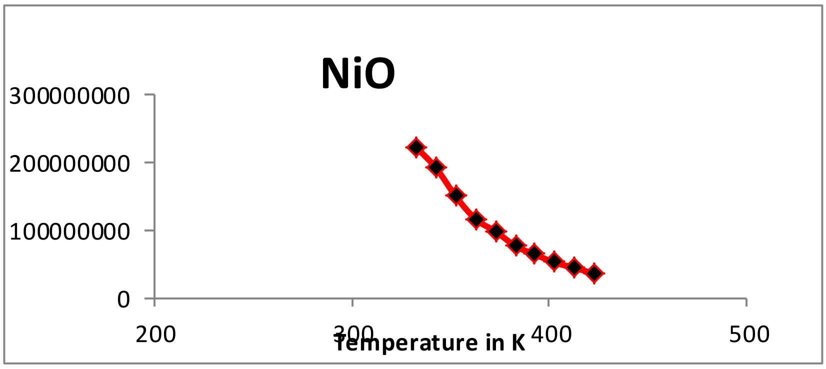


Fig. 6. Showing graph of Electrical resistance V/s Temperature for NiO thick films

Gas sensing properties of Co_3O_4 in presence of C_2H_5OH atmosphere:

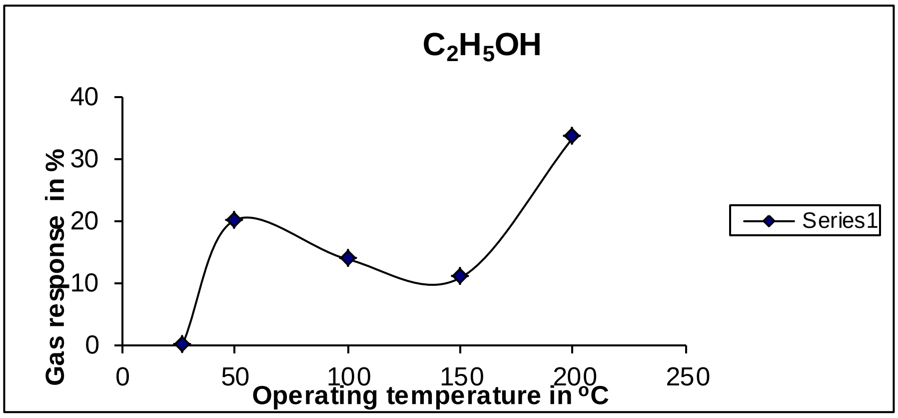


Fig. 7. Showing graph variation of gas response against operating temperature for C_2H_5OH Gas over Co_3O_4 thick films

The variation of gas response of the Co_3O_4 thick films sample with C_2H_5OH (Ethanol) gas observed in the range of 50-300 ^{0} C temperature is represented in Figure-7.The film was exposed to different gas concentrations of vaporised ethanol. The sensitivity values were observed maximum at 200 ^{0}C. Similarly optimum temperature for NiO thick films was 50-200 ^{0}C, shows the maximum response at 150 ^{0}C, as depicted in **Fig. 8** [17, 18].

Gas sensing properties of NiO thick films in presence of ammonia atmosphere:

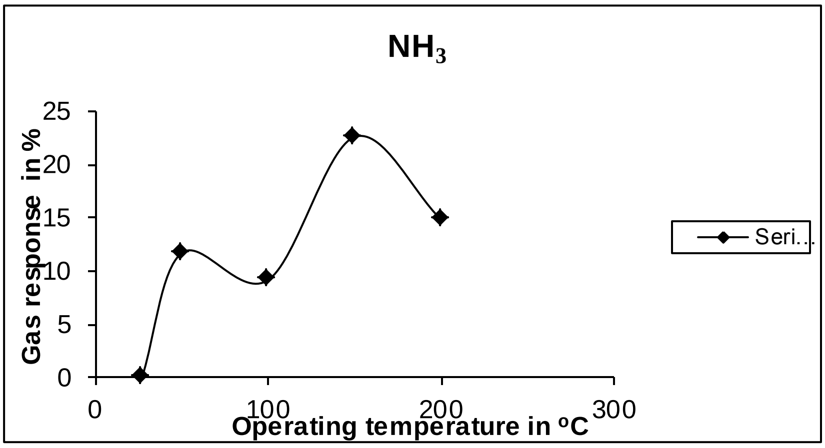


Fig. 8.Showing graph variation of gas response against operating temperature for ammonia Gas over NiO thick films

CONCLUSIONS:

The nanoparticles and of Co_3O_4 and NiO material successfully prepared by co precipitation and sol-gel method respectively and their thick films also prepared by conventional screen printing method. Characterization carried by XRD from which calculated size of Co_3O_4 and NiO from Scherer formula found to be 30.17nm and 10.18nm respectively. From SEM studies it is observed that the heterogeneous surface of Co_3O_4 microspores and mesopores as seen from its surface micrographs. It is greyish black in colour, various sized nanoparticles images ranging from 30.0 nm to 64.4 nm can be seen from SEM images. The prepared nanoparticles Co_3O_4 have fixed elemental composition confirmed from EDS. The electrical characterization and gas sensing properties examined for Co_3O_4 and NiO thick films for vaporized ethanol and ammonia gas respectively.

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