Green synthesis and characterization of SnO$_2$ and ZnO nanoparticles:
Study their electrical conductivity and gas sensing properties

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ABSTRACT

In the recent years nanomaterial’s directed attention of many researchers because their vast application in the field of research. Nanomaterials that can used as catalyst have potent application in the field of electrical conductivity, Gassensing, optical materials, ceramic materials, signal transducers, optoelectronic devices. The present study involves preparation of transition metal oxides such as SnO$_2$ and ZnO(Green method). In addition to these materials thousands of materials have been prepared successively and reported their application in same fields. SnO$_2$ and ZnO, prepared by Co-precipitation method, characterization was accomplished by XRD for Nanomaterial size confirmation. Their thick films prepared by screen printing method, electrical conductivity was measured by a conventional apparatus. Gas sensing properties of Ammonia gas performed by preparing thick films of SnO$_2$ & ZnO nanomaterials. Good results were recorded for conductivity properties and gas sensing properties of prepared nanomaterial of SnO$_2$ and ZnO.

Keywords: Coriander sativum extract, zincoxide Nanoparticles, zinc nitrate, Stanous chloride, electrical conductivity, Co-precipitation, Thick films, X- Ray Diffraction(XRD).

INTRODUCTION

Nanomaterial’s can be prepared by various methods; some of the reported methods are sol-gel method, combustionmethod, chemical vapour deposition, chemical bath deposition, co-precipitation method etc. Successfully these nanoparticles are prepared by the methods as mention above. Due to large surface area provided by metal oxide nanoparticles effectively can be used as a catalyst [1]. Many of the research papers flaunting that metal oxides nanoparticles can be useful as a gas sensors for the most of the gases. In several cases researchers showed that the metal oxide nanoparticles can be effectively used as a photo catalyst for the degradation of dyes and environmental pollutants.

In the recent years activity towards preparation of gas sensors by using nanomaterial’s has been increased. Most of the researchers performed activity of research for development of gas sensors. An efficient gas sensor shows excellent catalysis for selected gases. Conventional apparatus designs can be made for the sensitivity of gases. The majority of nanomaterial’s are belongs to the transition metal oxides. These metal oxides can also be prepared by doping the several transition metals or inner transition elements to the metal oxides. In most of the nanomaterial’s doped by metals improves several properties such as catalytical activity, conducting properties, electrical properties, optoelectrical properties, sensing activity. [2, 3]

In the present study ZnO & SnO$_2$ prepared by co-precipitation method. Typically ZnO shows n-type semi conduction with a band gap of 3.37 ev Zinc oxide particles are mostly used in preparation of solar cells, photo detectors, LED’s photo-detectors etc.
The most used gas sensor devices can be divided into three big groups depending on the technology applied in their development: Solid state, Spectroscopic and Optical. While spectroscopic and optical sensors are very expensive for domestic use and sometimes difficult to implement in reducing spaces. So-called Solid State sensors present great advantages due to their fast sensing response, simple implementation and low prices. These Solid State gas sensors are semiconductor metal oxides sensors based on the change of the physical and/or chemical properties of sensing materials when exposed to various gas atmospheres. These are resistive type sensors. The detection principle of resistive sensors is based on changes of the resistance of a metal oxide thick film (semi-conducting in nature) upon adsorption of the gas molecules to be detected. The gas-solid interactions influence the density of electronic species in the film and thereby the resistance of the film. Metal oxide sensors are also sometimes called chemiresistors. These are the simplest type of the gas sensors among the various technologies and hence perhaps are the most attractive gas sensor type for portable applications. They possess advantages of compact size, simple fabrication, low cost and simple measurement electronics.

Metal oxides provide large surface area that helps in catalytical application of materials. Generally, metal oxide thin [4, 5, 6] and thick films gives a good account for gas sensors. Large numbers of gases are tested for various nanomaterials. Advantage of gas sensing study gives sensitivity for various gases which helps to regulate their concentration at various places includes industries, laboratory, mining, atmosphere etc. Maintenance of pollutant gases and harmful gases can be sense at the particular level and temperature for different catalyst metal oxides.

Metal oxide dopants and their variation also gives good reactivity for selected gases dopants increases several properties of metal oxides such conductivity, electrical properties, sensitivity towards gases,[7,8,9,10,11] surface area ,reactivity etc. Hence most of the researcher doped nanomaterial by several transition and inner transition elements to increase the several properties as mentioned earlier. In Both cases that is doped and undoped nanomaterial provides good catalytical applications as a gas sensors. [12,13,14,15,16,17,18].

MATERIALS AND METHODS

The Chemicals required for synthesis Of ZnO and SnO$_2$ are Zinc nitrate hexahydrate, sodium hydroxide ,Stanous chloride dihydrate, oxalic acid, acetone ,and ammonia double distilled water, Corundum extract .All the materials used were analytical grade and used without further purification.

**Synthesis of ZnO by Co-precipitation method:** [Green synthesis method]

Take 0.02M of Zinc nitrate hexahydrate and dissolved it into 50ml distilled water with constant string on magnetic stirrer followed by addition of aqueous leaf extract of corriandum is introduces into same solution of zinc nitrate in the following particular sets 0.25,0.5,1,1.5,2,3,4,5ml with constant stirring. After this 2M solution of NaOH was added to make the pH -12 of the same solution resulted in pale white aqueous solution stirred for 120 minute. Pale white precipitate was then taken out and washed over and over again with distilled water and followed by washing with ethanol to get free from impurities. Precipitate is obtained was dried at room temperature for 12-15hr then powder of pale white coloured is dried on hot plate for 60-70$^\circ$C for 4 hours , pale white coloured nanoparticles of ZnO were obtained.

**Synthesis of SnO$_2$ by Co-precipitation method:**

In preparation of SnO$_2$,0.1m of stannous chloride dihydrate was dissolved in 50 ml of water after complete dissolution, about 4 ml ammonia solution was added to above aqueous solution with constant stirring continued for 20 minutes, white precipitate is formed. It is allowed to settle for 12 hr. then it is filtered and washed 2-3 times with hot water.

The obtained precursor dried for 24 h at 60$^\circ$C. Dried powder is crushed and heated at 600 $^\circ$C for 4 hours in presence of air.

**Thick films preparation of ZnO and SnO$_2$ by screen printing method:**

The Nano-powder of ZnO paste used to prepare thick films by using screen printing technique. Thick films prepared by maintaining the inorganic to organic materials ratio at 70:30. The inorganic part consists of a functional material (ZnO). The organic part consisted of 8% ethyl cellulose (EC) and 92% butyl carbitol acetate (BCA). The ZnO with ethyl cellulose were mixed thoroughly in an acetone medium with mortar and pestle. A solution of BCA was added drop wise until proper thixotropic properties of the paste were achieved. ZnO thick films were prepared on glass substrate by using standard screen-printing technique. The films were dried under IR radiation for 45 minutes to remove the organic part and then fired at temperatures 400$^\circ$C for 1.5 to 2 hrs. in muffle furnace.

Same method was followed for preparation of thick film of SnO$_2$. 

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RESULTS AND DISCUSSION

Electrical characterisation:

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 as shown in fig.1. The home built characterisation system consists of glass chamber (25 litres and -12’ diameter) 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°C By changing its voltage using dimmer stat (maximum current limit up to -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 spring press contacts were used during the measurement. The temperature of the sample was measured by using the temperature indicator with the help of Cr-Al thermocouple. The high resistance of the sample was determined by using the half bridge method.

**Electrical resistance (Heating): ZnO**

The thick film of ZnO was introduced in chamber followed by increasing temperature. The series of readings were performed on the ZnO sample. Fig.2 shows resistance in ohm for ZnO Thick films measured by Half-bridged method.
Electrical resistance (Heating): SnO$_2$:

From figure 2 & 3 ZnO & SnO$_2$ shows resistance in ohm. The resistance of ZnO decreases with increasing temperature. Confirming semiconducting behaviour of ZnO.

Gas sensing properties of ZnO (Green synthesis) using NH$_3$ gas:

From Fig. 4 ZnO shows gas response for NH$_3$ gas at 50 °C.
From Fig. 5 SnO$_2$ thick films show gas response for NH$_3$ gas at 50 °C

Characterization of ZnO and SnO$_2$ nanoparticles by XRD:

ZnO XRD pattern:

![ZnO XRD Spectra](image)

The XRD analysis is carried out by using D8 ADVANCE (Burker) model x-ray diffractometer using cu-k-α wavelength 1.54 Å$^3$. By using Scherrer’s formula the size of ZnO nanoparticles is calculated by $D = K\lambda/\beta\cos \theta$

Where
K=constant(0.89to1.39)
λ=Radiation of wavelength
β=FWHM(Full Width Half wave Maxima)
θ=Bragg angle in degree
D=Particle Size

The XRD pattern of ZnO shows the spectrum having main peak at 2θ. From which calculated size of ZnO nanoparticle is $51\text{nm}$. The XRD spectrum of ZnO as shown in the Fig. 6.
SnO$_2$ XRD pattern:

The XRD pattern of SnO$_2$ shows the spectrum having main peak at 2$\theta$. From which calculated size of SnO$_2$ nanoparticle is 35nm. The XRD spectrum of SnO$_2$ as shown in the Fig-7

CONCLUSION

1) The nanomaterial’s ZnO and SnO$_2$ successively prepared by Co-precipitation method.
2) The electrical resistance of ZnO and SnO$_2$ was calculated. The figures 2 & 3 shows that for both nanomaterial’s i.e. ZnO and SnO$_2$, resistance decreases with increasing temperature. Confirming semiconducting behaviour of ZnO and SnO$_2$
3) Thick films of ZnO (Green synthesis) and SnO$_2$ were prepared successfully by screen printing method.
4) The nanomaterial’s ZnO and SnO$_2$ were characterized by XRD from which nanoparticle size for ZnO and SnO$_2$ was confirmed as 51nm and 35 nm respectively.
5) The thick films prepared of ZnO and SnO$_2$ shows sensitivity for NH$_3$ gas at 50$^\circ$C for both materials.

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