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# GROWTH AND HUMIDITY SENSING PROPERTY OF METAL OXIDE THIN FILM BY SPRAY PYROLYSIS

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**ABSTRACT:** Wide variety of metal oxide nanostructures such as iron oxide, tin-oxide, copper oxide, tungsten oxide, molybdenum oxide, zirconium-titanium mixed oxides, indium oxide and their applications such as gas-, bio- and chemical sensors, optical sensors, dye-sensitized solar cells etc. To control the humidity of air means the increasing or decreasing of moisture content of air during summer and winter respectively in order to produce comfortable & healthy efficiency of the worker. In general, for summer the summer relative humidity should not be less than 60% whereas for winter it should not be more than 40%. It may be noted that human being feels comfortable when air is at $21^0$C and 56% relative humidity. The sensor device which is used to measure the relative humidity is known as humidity sensor. A humidity sensitivity meter is use for measuring the change in relative humidity of the chamber. A steel box is taken to use as a chamber & rubber is stripper at its mouth and then a glass cover is put over it to make it air resistive. Then the wires comes from resistivity meter is switched at the terminals of one glass slide. It may be noted that the film on the glass slide should be equal and of same thickness for all the films. The graph between humidity and resistivity of $SnO_2$ which is made-up by spray, which gives many variations. When the humidity is increases then the resistivity is continuously increases and it gives slopes which show the sensitivity.it is linearly decreases. So from the graph this indicated the linear decrease in resistance with increasing humidity in 25-70% RH and thus proves the suitability of composite for humidity sensing purposes. The sensitivity was calculated with slope of curve and is found approximate at 30%RH. Further change in resistance at 90%RH with time is measure and curve which reveals the response time 10 sec. for 85%RH of material is very low and recovery time after blowing dry air is very high i.e. 13sec.

**Keywords: Humidity Sensor, Metal Oxide, Thin film, Spray Pyrolysis.**

## INTRODUCTION:

Nature has endowed human and animals with five primary sense organs namely eyes, ears, tongue, skin and nose." A device that detects or senses heat, light, sound, motion, etc., and then reacts to it in a particular way is called Sensor" **[1-7].** Here are sure structures which have to be well thought-out when we choose a sensor. For example-Accuracy, Environmental condition, Range, Calibration, Resolution, Cost, Repeatability etc. And the sensors are classified into Primary Input quantity (Measured), Transduction values (Using physical and chemical effects), Material and Skill Property. Transduction value is the important standards which are trailed for an well-organized approach. Regularly, material and technology standards are selected by the growth engineering group. The classification is based on the temperature, Pressure, flow, Level Sensors, Proximity and displacement, Biosensors, Image, Gas and chemical, Acceleration.

In1938, Wagner & half discovered that atoms and molecules interact with the semiconductor surfaces and influences such surface properties as conductivity & surface potential. The effect of the ambient atmosphere upon the electrical conductance of semiconductors was described by Brattain and Bardeen **[8]**. Wide variety of metal oxide nanostructures such as iron oxide, tin-oxide, copper oxide, tungsten oxide, molybdenum oxide, zirconium-titanium mixed oxides, indium oxide and so on and their versatile applications such as gas-, bio- and chemical sensors, optical sensors, pressure sensors, lithium ion batteries, electrochemical performance for energy storage, dye-sensitized solar cells etc.

In recent years, numerous metal oxides have been investigated due to their interesting properties and applications in sensing, catalysis and magnetism**. [9,10].** $SnO_2$, is the inorganic compound with the formula $SnO_2$. The inorganic form of $SnO_2$ is called cassiterite, and this is the chief ore of tin. This colorless, diamagnetic solid is amphoteric.it is insoluble in water and alcohol and it is soluble in the concentrated alkine and acid. The properties of $sno_2$ depends on the nature of interaction between oxide and dopant. In order to form a good material with good humidity sensing performance, adsorption and condensation of water molecules are important process. So the ideal sensor may possess the properties such as porous texture, high surface area and high thermal stability.**[11-13]**

In this paper tin oxide/zinc oxide composites with various molar ratios were synthesized and explored as humidity- sensing materials.

**Experimental set up:**

**Preparation of precursor solution:**

**Stannic Oxide:**

Similarly a solution of $SnCl_4$ in water was prepared by dissolving approximate amounts of $SnCl_4$ in water under magnetic stirring until colorless and transparent solution was obtained. The solution was then heated and kept at the temperature to evaporate water and hydrochloride.

1. $SnCl_4.5H_2O + H_2O \rightarrow SnO_2 + HCl$

**Zinc oxide:** The precursor for spray deposition is prepared by dissolving Zinc acetate; ethanol & diethanolamine (DEA) is respectively. ZnO Solution was obtain by dissolving Zinc acetat under magnetic stirring in a solution of ethanol and DEA (some drops) for 1 hour.

$Zn(CH3COO)\ 2+ C_2H_5OH+ DEA \rightarrow ZnO$ + other product

**Ratio of precursor solution:**

Firstly we have to make pure precursor solution of $SnO_2$ and ZnO solution. Then we take the ratio of the mixture of above solution in 2:1, 3:1, 4:1 and 5:1. It may be noted that when the quantity of ZnO solution is more than a precipitate is formed.

**Substrate cleaning:**

The substrates used to deposits Stannic Oxide thin films are glass slides (BK7) slide of dimension Prior to this deposition of the film, a thorough cleaning of the glass slides has been done.

Initially they have been cleaned with a soap solution and then ultrasonic cleaner in acetone & methanol for removal of dust grease. Finally the substrates are washed ultrasonically in distilled water & the wet slides were dried by a hot air gun.

**Spray pyrolysis:**

In this method, following apparatus are used to make a thin film on glass slide. By spray pyrolysis precursor solution to be sprayed is introduced in the solution container which is connected to the liquid inlet of the atomizer by a tube having a solution flow controller. The experimental set-up of the spray pyrolysis is shown in the figure.

After the films are the formed they are put in electric oven for 2 hour then they are taken in butter paper or in cover in a box so that no moisture or dust particle is absorbed on the surface of film.

**APPLICATIONS:**

To control the humidity of air means the increasing or decreasing of moisture content of air during summer and winter respectively in order to produce comfortable & healthy efficiency of the worker. In general, for summer the summer relative humidity should not be less than 60% whereas for winter it should not be more than 40%. It may be noted that human being feels comfortable when air is at $21^0$C and 56% relative humidity. "The sensor device which is used to measure the relative humidity is known as humidity sensor." Modern civilization is showing an increasing fascination toward the air conditioning system. This demands increasing day by day to fulfill the human comfort. According to American Society of Heating Refrigeration and Air-conditioning Engineers - human comforts is defined as that condition of mind which express satisfaction with the thermal environment. Hence the subject has a broad sense which indicates the conditioning of air for industrial purposes, life style, hafiz society, food processing, storage of food and other materials like chemicals & computerize and sophisticated instrumentation, auditorium, cinema halls etc.

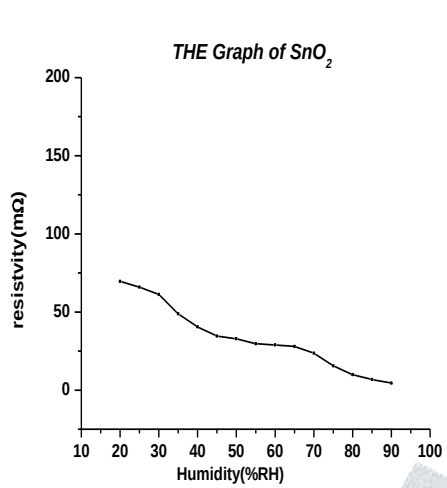
**Humidity measuring set up:**

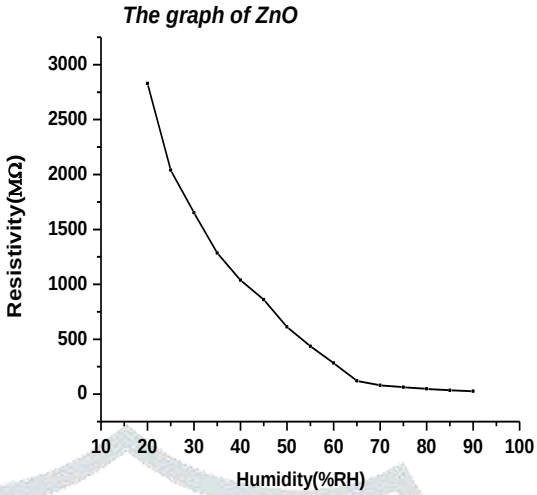
A humidity sensitivity meter is use for measuring the change in relative humidity of the chamber. A steel box is taken to use as a chamber & rubber is stripper at its mouth and then a glass cover is put over it to make it air resistive. Then the wires comes from resistivity meter is switched at the terminals of one glass slide. It may be noted that the film on the glass slide should be equal and of same thickness for all the films. Also the humidity sensor is put in this box & the machine is taken in as manually. The potassium hydroxide (KOH) and potassium sulphate ($K_2SO_4$) chemicals are taken in to decreases or increases the humidity of the chamber is respectively. When the humidity is increases the resistivity is decreases.

The graph between humidity and resistivity of $SnO_2$ which is made-up by spray is shown in the fig. which gives many variations. When the humidity is increases then the resistivity is continuously increases and it gives slopes which show the sensitivity and at certain point (30%RH) it is linearly decreases. The solution of 1:4 i.e. 1 ml of zinc oxide: 4ml of stannic oxide is used as a best sensor because is giving very high resistivity at low humidity and it gives a maximum slope at 30% RH. Again pure ZnO is used as a sensor but it is poor then 1:4 because its slope and its response & recovery time are less than 1:4. Pure $SnO_2$ is not used as a sensor because it gives minimum resistance at minimum humidity and it does not give a slope. Both have similar results. i.e. 1:4 is used as a good sensor and $SnO_2$ is not used as a sensor.

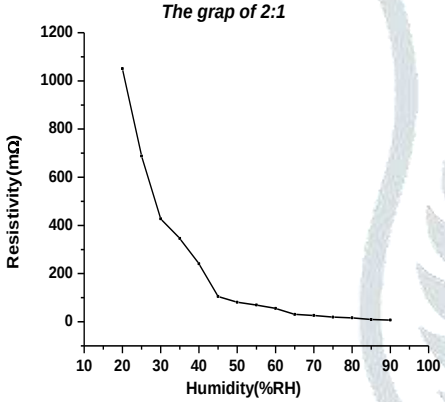
To trial the constancy, the sensor was showing in air for 2-3 days, surveyed by measurement resistance at various %RH. There are almost no changes in the resistance, which also confirms the good stability of the sensor. From the criteria as discussed above, the sensor has prominent stability and is quite promising for a practical humidity sensor.

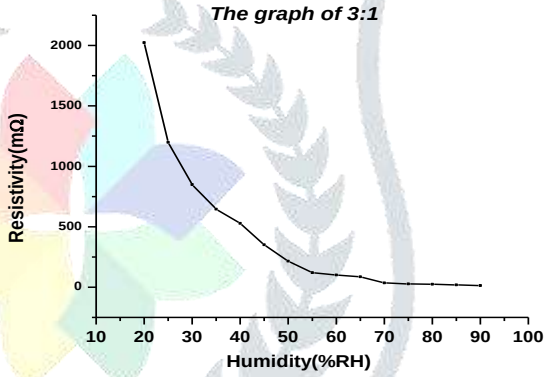
**Figure:-4**



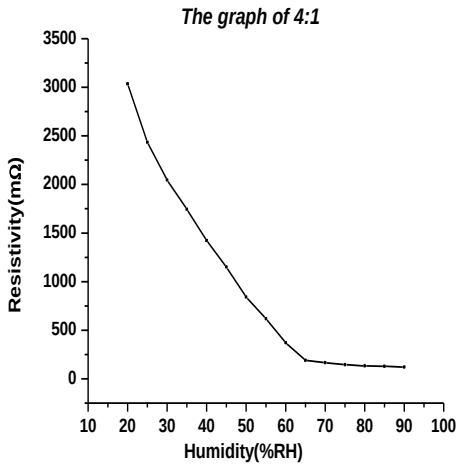




**(a)(b)**

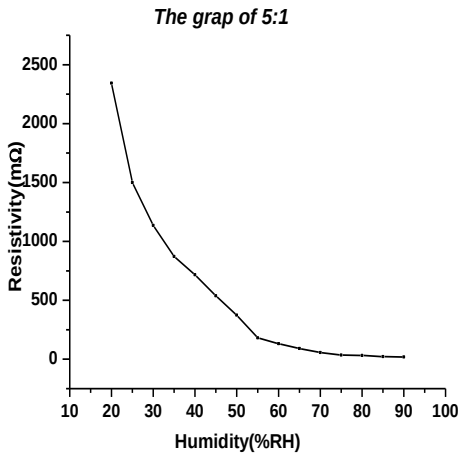


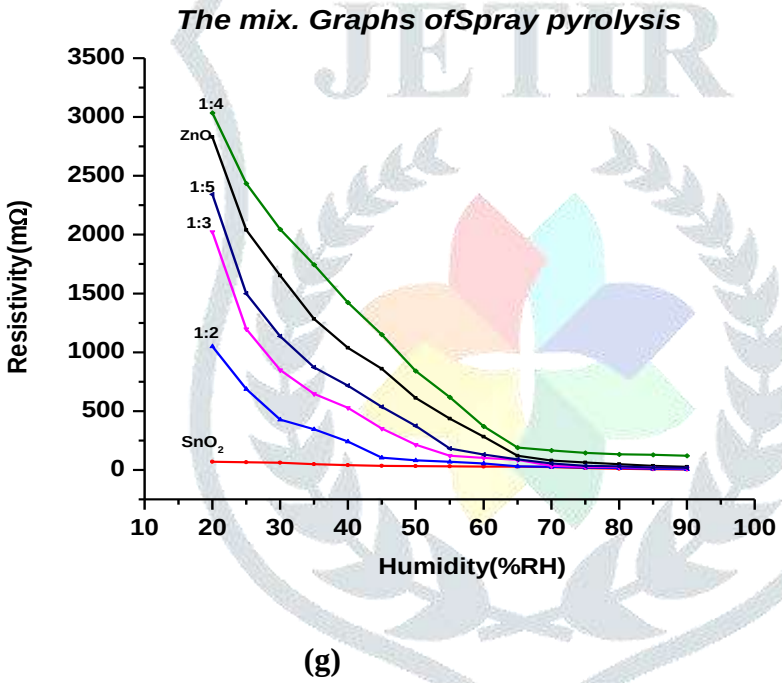

**(c)** **(d)**




(e)




(f)




(g)

**Fig 4.** a, b, c, d, e, f Shows the humidity sensing graph separate ZnO, $SnO_2$ , ratios 2:1, 3:1, 4:1,5:1 and g shows the combined graph of all the samples

Every sample has a lowest humidity sensitivity and highest sensitivity point after which no changes is observed. Every sample have a different slope and different response & recovery time Those sample which have a maximum slope & minimum response &recovery time which used as a good sensor i.e. 1:4. When one temperature is increased by air blower outside one chamber the humidity suddenly falls and as a result the resistivity increases. Here all the observations are taken at room temperature effect of ratio of precursor solution. It is clear from the above graph that following effect can be noticed.

The graphs show that there is linear decline in resistance with growing humidity in 25-70% RH and thus proves the suitability of composite for humidity sensing purposes. The sensitivity was calculated with slope of curve

and is found approximate at 30%RH. Further change in resistance at 87%RH with time is measure and curve which reveals the response time 10 sec. for 83%RH of material is very low and recovery time after blowing dry air is very high i.e. 13sec.

**Conclusions:**

From above which indicates the linear decrease in resistance with increasing humidity in 20-90% RH and this proves the suitability of composite for humidity sensing purposes. The sensitivity was calculated with slope of curve and is found approximate at 30%RH. The composite was electrically responsive to humidity under a closed chamber in the range of 20-90%RH. The synthesized organic/ inorganic composite of can be used to prepare electrochemical humidity sensors/ the sensors based on composite shows better sensitivity, linearity and quicker response resistivity. Since 1:4 has a approximate 65%RH slope and 10sec. response and 13sec. recovery time so it used as a good sensor. The UV graphs gives the film is transparent and a transmission spectrum fluctuates between 68 - 90% in visible and infrared region with sharp cut off at 300nm.

Since these film gives PL in the range 380-650 nm. So it peak at the centered at 390 nm which gives violet colour and different film have different peak when the ratios are increases than the peak is increases and ZnO have lowest peak.

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