Nanocrystalline Titanium Dioxide Dipped with AlCl2 as a Humidity Sensors[1]

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ABSTRACT

Titanium dioxide and AlCl2 was mixed in different stoichiometry in mol wt. % for the study. By using the screen printing technique, the thick films of humidity sensors are prepared. All of the humidity sensor devices are tested and finally concluded that the sample T-1 at constant temperature 400C to 700C exhibited high sensitivity and fast response time to humidity sensing at room temperature. In the case of conductivity, curves are frequently crowded and mixed together. The conductivity of Sample films linearly responds to Relative Humidity. The resistance of thick films decreases by increasing the humidity of sensors at room temperatures due to surface oxygen vacancies of TiO_2 acting as electron donors.

Keywords: *Thick films; TiO2-AlCl2; Sensitivity; Humidity sensors*

INTRODUCTION

These humidity chambers are designed to maintain temperature and relative humidity at set points controllable by the operator at the front panel.[1-4] Air is constantly being circulated through the chamber, scheduled for comparison to set points. Heat is produced by electric resistance heaters that turn off and on for temperature control. On units with cooling there is a refrigeration unit continuously on. Chamber humidification is reached by means of a low-pressure vapor generator injecting water vapor into the chamber through a small orifice. The water vapor is reached into the chamber at the blower discharge. Chambers were programmable, and networked or Web- enabled test chambers are also available. In the present study is to synthesize and structural characterization of TiO_2 nanoparticles by using liquid phase method with effectively more surface area in short reaction time at room temperature and this method is the simplest, cost effective, eco-friendly method. It is also probed for its effect on nanocrystalline size structure via XRD studies of TiO_2 nanoparticles.[5-9]

EXPERIMENTAL METHOS:

All the chemicals used in this present work were of GR grade purchased from Sd-fine chemicals, India having purity 99.99%.TiO_2 nanoparticles are synthesised by sol-gel method. The synthesis of TiO_2 nanoparticles is divided into various steps, such as mixing, stirring, filtering, drying and calcination. Finally by the calcinating the powder at 400 °C for 3 hours, the TiO_2 is obtained in the nanoparticles form.

Screen printing is a technique in which a mesh is used to transfer ink onto a substrate, except in areas made impermeable to the ink by a blocking stencil. A material or gel which is formed is moved across the screen to fill the open mesh apertures with ink, and vice versa then causes the screen to touch the substrate momentarily along a line of contact. This causes the material to wet the substrate and be pulled out of the mesh apertures as the screen springs back after the blade has passed. In the similar way, instead of ink we use paste of nanomaterial and glass slides as substrate.

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So, we have use here mesh which has permeable stencil area smaller in area than glass slide which we are used. Firstly we take nanomaterial (90%) and in order to make its paste mix with solid binder (10%) known as Ethyl cellulose. When we make a well grind mixture of nanomaterial and solid binder then we add drop by drop liquid binder. We should careful about that liquid binder is added in appropriate quantity. So the perfect thick paste of nanomaterial is prepared. Then we follow the procedure that with the help of squeegee spread paste of permeable mesh for well layered nanomaterial on substrate (Glass slides). As these Titanium dioxide Nanoparticle thick films are prepared, firstly we allow them to dry in the atmosphere. Then they are subjected to the vacuum oven at 80°C for 1 hour. Then we put these thick films at Muffle furnace at 250°C for 3 hours. Now these Thick films are dip in Aluminium Chloride with different dipping time [10-12].

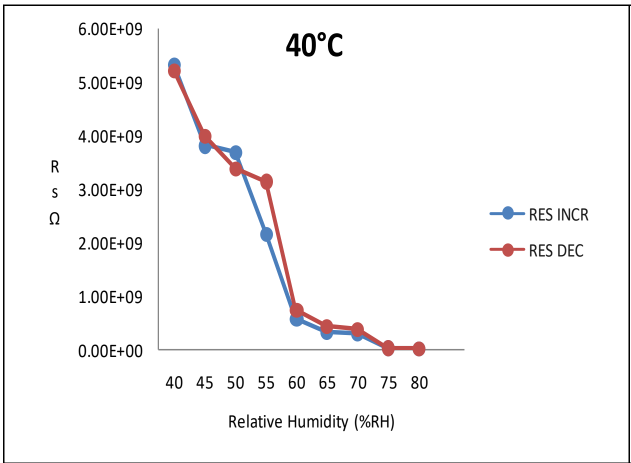
The main compound of aluminium chloride ($AlCl_2$) is aluminium and chlorine. The compound is regularly cited as a Lewis acid. In the dipping method, we have used Aluminium chloride for the dipping method. As we prepared solution of $AlCl_2$ then we dipped the thick film of Titanium dioxide for different time parameters. We take 1, 2, 3 minutes dipping and after firing these slides for 1 hour at 250°C finally the three thick films slides for different dipping time is prepared and one thick film taken for pure.

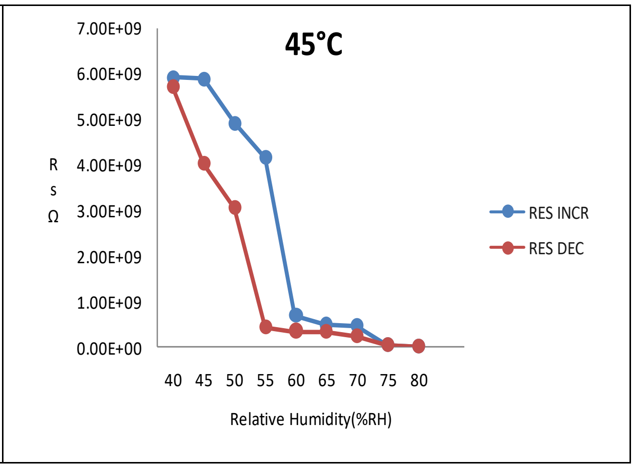
Humidity is a quantity representing the amount of water vapour in the atmosphere or in a gas. The level of comfort is determined by a combination of two factors namely relative humidity and ambient temperature. Humidity measurement is an important factor for operating certain equipment. A rule of thumb is to assure a relative humidity near 50% RH at normal room temperature (20–25°C). This may vary from a low as 40% RH for the clean rooms to 60% RH in hospital operating rooms. By the use of hygrometers humidity can be measured. The first hygrometer was invented by Sir John Leslie. Humidity measurement is an important quantity for predicting the climate outdoors as well as controlling the climate indoors. [13-18]

RESULTS AND DISCUSSION:

Hysteresis:

The phenomenon in which the value of a physical property lags behind changes in effect causing is known as Hysteresis. In our present work, humidity sensing with TiO_2 nonmaterial thick films shows the hysteresis plot of sample T-1, T-2,T-3,T-0 at constant temperature 40°C. Hysteresis plot shows variation between resistance of sample with respect to relative humidity in increasing and decreasing order of 40 to 80 % RH and 80 to 40 %RH in steps of 5 % RH. The resistance measurement was done with Keithley 2400 source meter at different constant temperature 40°C to 70^0C in steps of 10^0C.





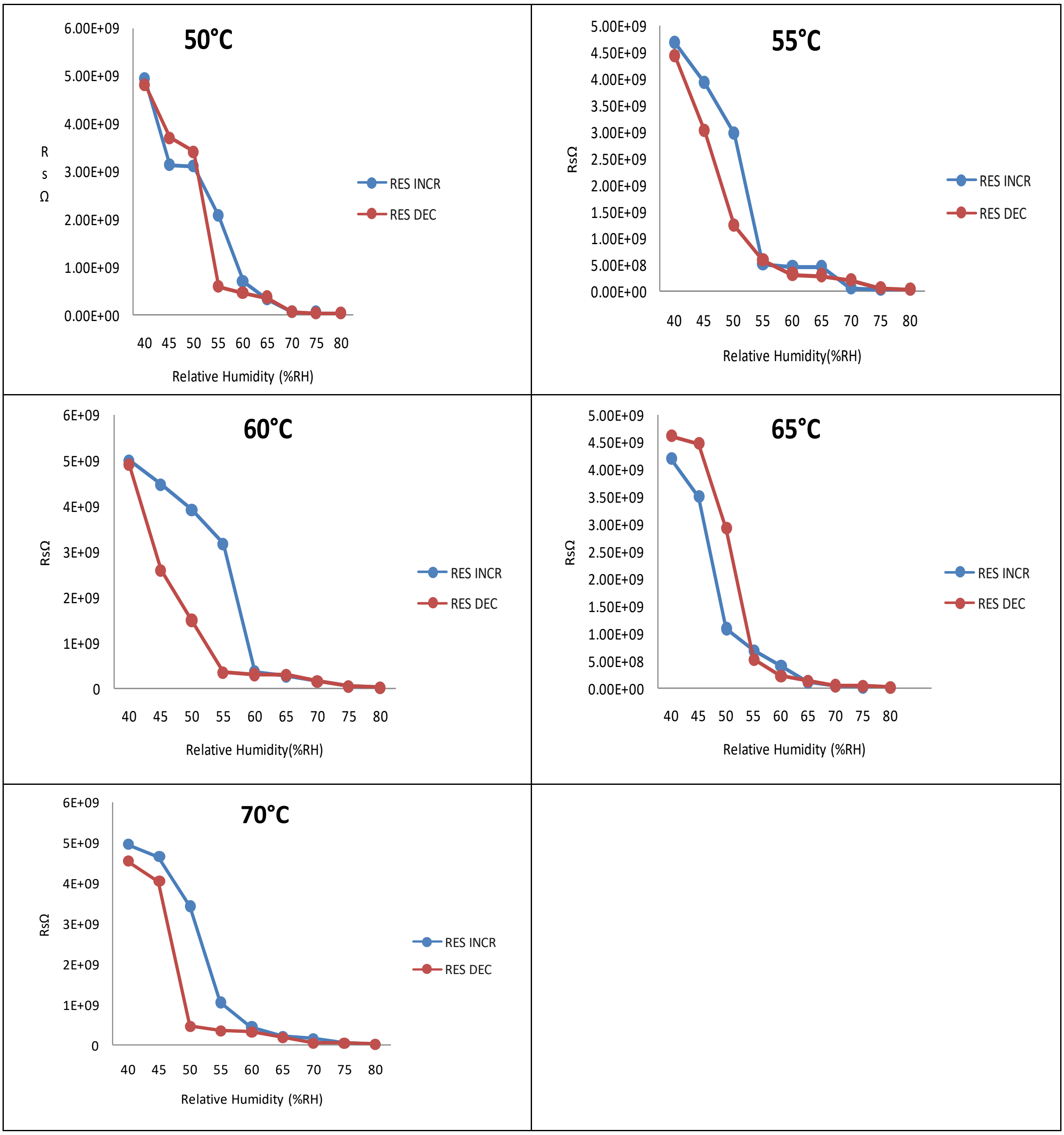


Figure 1.1 T-1 Metal oxide Thick Film (1-minute) hysteresis plot

Similarly, we can plot the graphs for remaining samples. In present work it is observed that from hysteresis plot series of a sample which is TiO_2 nanomaterial film T-1,T-2,T-3 dipped in Aluminium Chloride for different dipping time i.e. for 1,2 and 3 minutes respectively and hysteresis plot of pure sample of TiO_2 nanoparticle film at respective constant temperature. From hysteresis plot, it is clearly seen that there is very small hysteresis is present during forward (increasing) and reverse (decreasing) cycle of RH. It is observed that there is very significant average change was observed in value of resistance of sample in the range of 10^{10} to 10^{8}Ω.m . From 40 to 80 % RH except in the sample T-1 (1 minutes) change in the value of resistance from 10^{9} to 10^{7}Ω.m.There is a noticeable change in the value of resistance of sample T-1 at constant temperature 40°C to 70°C.

Table 1.1 Sample Codes

Sr. No.	Sample	Thickness x10^{-6} m	Material
1.	T-0	24	Pure TiO_2
2.	T-1	18	TiO_2 +Dipped with $AlCl_2$ for 1 minutes dipping and after firing these slides for 1 hour at 250°C.
3.	T-2	19	TiO_2 +Dipped with $AlCl_2$ for 2 minutes dipping and after firing these slides for 1 hour at 250°C.
4.	T-3	20	TiO_2 +Dipped with $AlCl_2$ for 3 minutes dipping and after firing these slides for 1 hour at 250°C.

The hysteresis was observed because of the process of adsorption and de-adsorption are not so faster at particular humidity. As the adsorption would not be efficient which causes small change in the value of resistance, the physiobsorbed water molecules is converted into chemisorbed by donating the surface electron at the constant temperature and for de-adsorption it requires large activation energy. As we observed that on the other hand sample shows the comparable decrease in resistance with increase in % RH which indicates that conduction occurs grain surface by release of electron from water molecule. Hence, the sample as shows the noticeable change in resistance between the values in between humidity range 40 -80 % RH. [19-20]

DC Conductivity

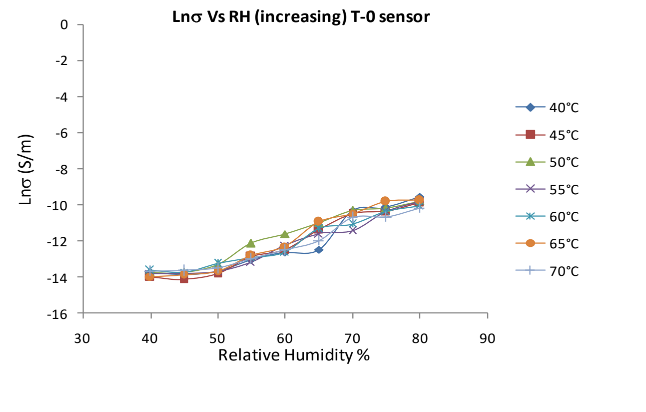


Fig. 1.2.a.

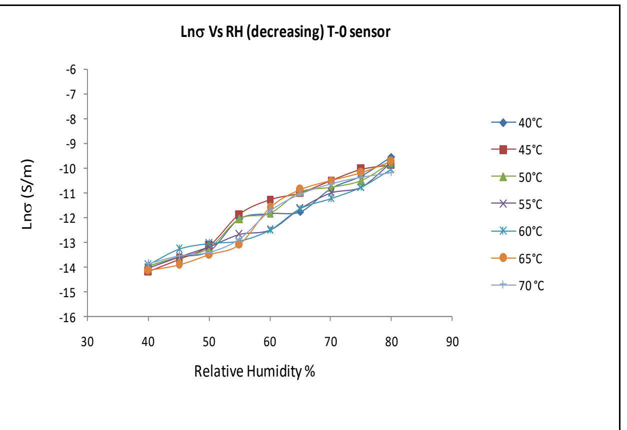
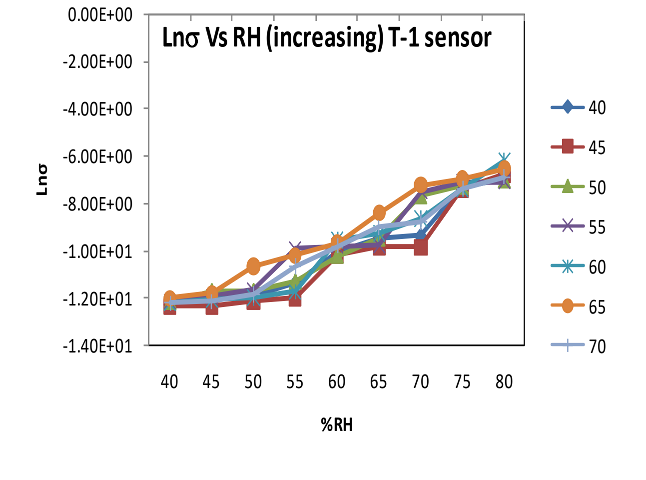


Fig. 1.2.b.



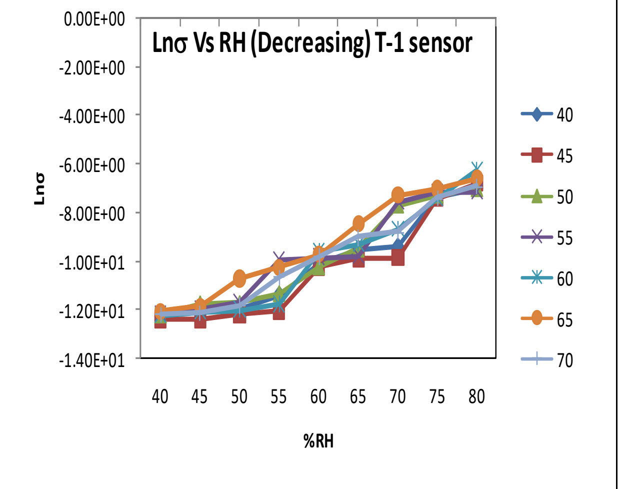


Fig. 1.2.c.

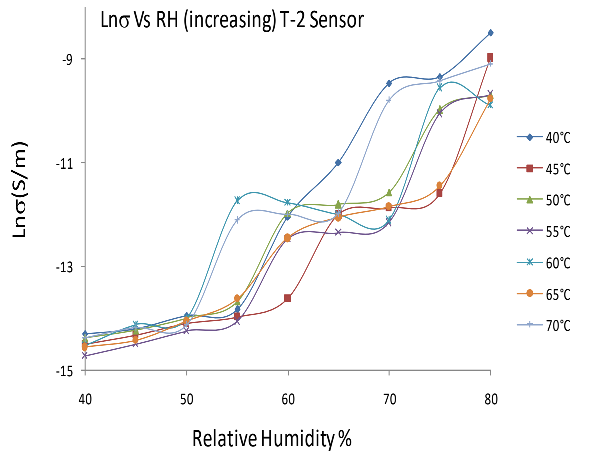


Fig. 1.2.e.

Fig. 1.2.d.

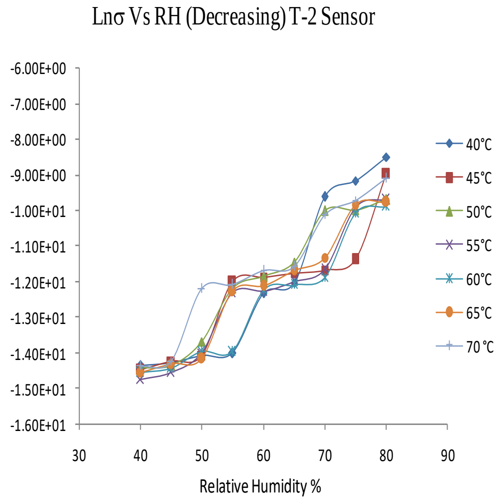


Fig. 1.2.f.

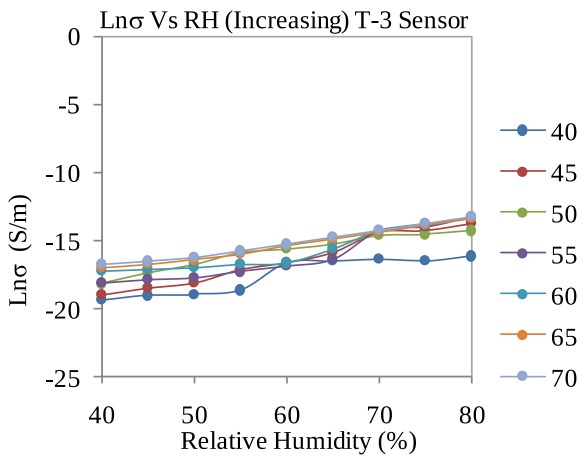


Fig. 1.2.g.

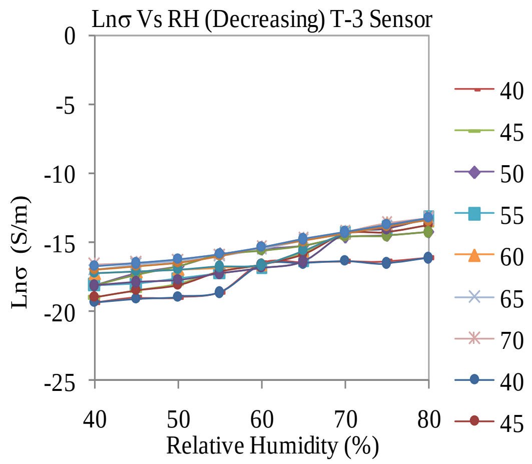


Fig. 1.2.h.

Fig. 1.2 Variation (a-d) Ln*σ* Vs RH at different constant temperature (40^{0}C to 80^{0}C)

The variation of Lnσ with increasing and decreasing (40 to 80 % RH and 80 to 40 % RH) of the series sample. Respectively at constant temperature 40ºC to 70ºC. It is observed that the conductivity increases partially linearly with relative humidity from 40 to 80 % RH and vice-versa. When the temperature of sample T-1 increases, the conductivity also increases. In all the series of sample, the conductivity found to be lowest at temperature 40ºC, while it is highest at high temperatures.[21-22]

XRD OF TITANIUM DIOXIDE

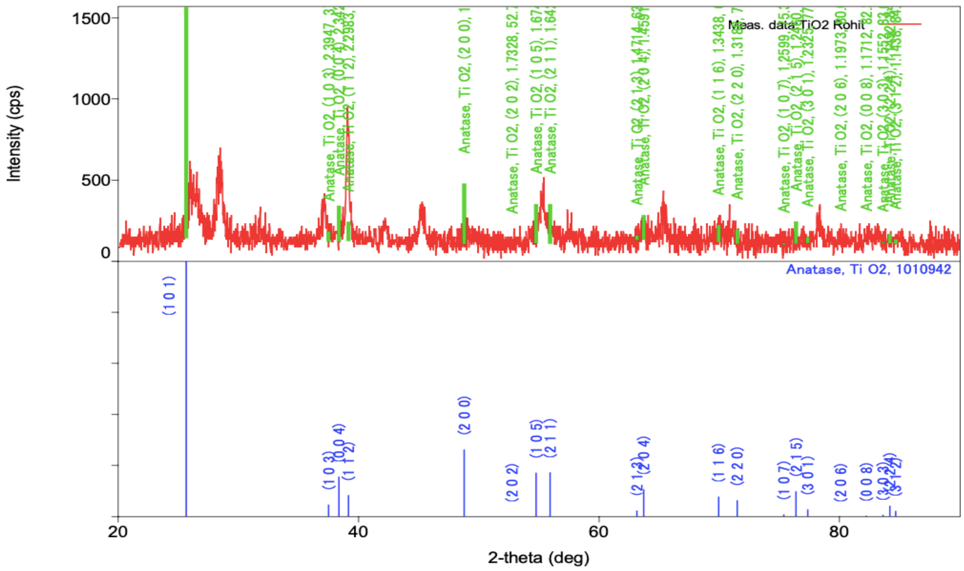


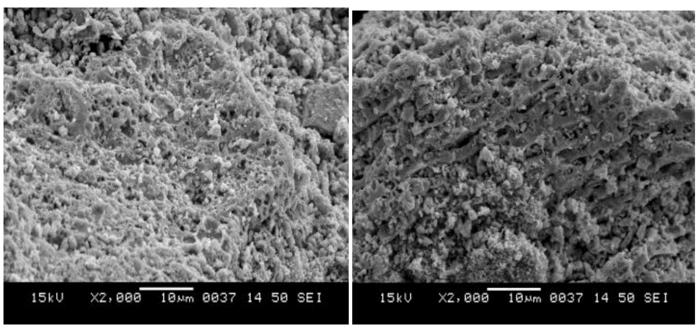
Fig. 1.3 XRD pattern of Periclase Titanium dioxide (TiO_2)

The XRD pattern of Periclase Titanium dioxide (TiO_2) nanostructure synthesized by liquid phase via co-precipitation method calcinated at 300°C. It is clearly observed that the highest intensity peak is obtained at (200) crystal planes and other peaks lying at (101), (004), (200), (105) and (211) of TiO_2.The (200) is observed as intense and sharp peak. Periclase phase in diffraction pattern exactly matches with JCPDS card no. 21-1272. All the peaks are perfectly match with Pericles TiO_2 nanostructure, which indicates that Periclase TiO_2 nanoparticles obtained. The average crystalline size was found to be 39.69 nm calculated by using Debye-Scherer formula. Debye Scherer formula as follows,

$$D = K\lambda / \beta . Cos\theta$$

Where, D is crystalline size, λ is wavelength of X-ray, βis Full Width Half Maxima, θ is Diffraction angle, K is constant closed to unity.[23]

SEM PICTURES:



SEM pictures of TiO_2 at 2000 magnification

The FE-SEM morphology of nanocomposites shows the particles are nanoporous, small size, like spherical shape. One can see that nanocrystalline and porous TiO_2, is formed on the surface. The Prepared sample shows agglomerated in nature. Grains exhibit a variety of sizes and forms, as well as a nanometer-scale order and a nanoporous structure. Because of the nano capillary pore and the structure's huge surface area, the adsorption and condensation of water molecules are anticipated to be made easier. Due to this porosity, humidity can be responded to and recovered from effectively.

CONCLUSIONS

The study of humidity sensors has led to novel sensing mechanism that can exploited in a different condition. It is observed that there is very significant average change was observed in value of resistance of the sample in the range of 10^{10} to 10^{8} ohm. There is noticeable change in the value of resistance of sample T-1 at constant temperature 40ºC to 70ºC. In the case of conductivity, curves are frequently crowded and mixed together. Conductivity of Sample films linearly responds to Relative Humidity. The Sample films shows the appreciable results regarded with Humidity Sensing. All the peaks are perfectly match with Periclase TiO_2 nanostructure, which indicates that Periclase TiO_2 nanoparticles obtained. No other peaks were detected in spectrum within detection limit of XRD instrument, indicating the pure periclase TiO_2 Nanomaterial is synthesized [19-25].

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Conflict of Interest: None

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