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Research article

Nanostructured copper oxide films by a perfume atomizer technique well suited for gas sensor

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Abstract

In a pursuit of searching potential materials suited for alternative sources of energy, the copper oxide is found to be a versatile and useful candidate material capable of addressing many technological needs and hence the results of Copper Oxide thin films are presented in this work. Apart from its' role in solar energy converting devices, gas sensors, superconducting sensors, etc., the specific usage of low cost solar cells for terrestrial applications has become vital in recent investigation. Though there are much studies on the fabrication of Copper Oxide thin films, keeping in view of the situation in demand, we wish to employ a simple and low cost spray pyrolysis or perfume atomizer method here as it has not yet been reported. Here, the results of Copper Oxide thin films of different concentrations (with constant temperature of 673K) along with structural, morphological, and optical and gas sensing properties have been reported .**Copyright © IJRETR, all rights reserved.**

Keywords: Copper oxide thin films, Optical properties, Substrate temperature

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1. Introduction

Ever since the invention of the principle of photovoltaic[1], the future of energy resources has been increased rapidly with plenty of novel materials. In the process of searching of suitable materials for terrestrial low cost solar cells, the investigators found that the choice of Copper Oxide such as CuO and Cu_2O has been justified and interesting due to many reasons. Certain salient features of Copper oxide semiconductors are high optical absorption, non-toxic and low cost fabrication. Moreover, the CuO and Cu_2O materials are known to be *p*-type semiconductors and hence potentially useful in constructing *pn* junction devices, transistors, light emitting diodes, Schottky barrier solar cells, etc.,[2-5]. Further, these materials have been employed as heterogeneous catalyst for several environmental processes [5-6], solid state gas sensors hetero-contact[7-8], and microwave dielectric materials[9]. The copper oxide thin films have been prepared by almost all the techniques like dip coating, spin coating, chemical vapour deposition, sputtering, and vacuum evaporation technique and so o [10-13]. As there is no report in literature about the fabrication of CuO thin films using extremely low- cost perfume atomizer technique, it has been aimed at preparing Copper oxide thin films of different molarities but keeping the temperature of **673K** as constant, using a low-cost simplified spray pyrolysis technique[14-15] and predicting the structural, optical, morphological, properties, which are of greater importance in low-cost solar devices of terrestrial applications. It is for the obvious reason that the substrate temperature of 673K has been considered during thin film fabrication as the *in-situ* temperature of terrestrial solar cells falls in the range.

Among various metal oxides Cuo thin films have attracted researchers due to their potential applications in the field of solar cells and gas sensors [16-18]. Ethanol is the most important alcohol owing to its divers applications. It is widely used in food industry brewing process control, medical and clinical applications and bio-medical technological processes. Those working on ethanol synthesis have great chances of being victims of respiratory and digestive track cancer. Hence, there is a great demand for monitoring ethanol gas at trace level semiconductor metal-oxide based gas sensors are commonly used for environmental monitoring and industrial applications, due to this advantages such as small dimension, low cost and convenient operation. To date n-type metal oxides are widely investigated, Such as ZnO [19], InO_3 [20], and SnO_2 [21], due to their extensive sensing performance. Recently increasing interest has been taken in gas sensors based on p-type semiconductors. Among a variety of p-type semi-conductors, copper oxide (CuO) has proved itself to be one of the most attractive materials for gas sensor applications from the point of view of gas sensitivity as well as chemical stability. CuO has been found to be sensitive to HCHO, NO_2 H_2S and CO [22]. However, research on P-type CuO based ethanol sensor and the sensing properties of CuO to ethanol is satisfactory.

2. Experimental investigation

2.1. Preparations of thin films by spray pyrolysis using a perfume atomizer

In the present work, a simple and elegant method, namely, a simplified spray pyrolysis has been employed. In comparison to the conventional spray technique, this simplified spray technique using perfume atomizer has several advantages and fulfils the requirements demanded by terrestrial low cost solar cells[23]. Atomization is based on hydraulic pressure and hence there is no need to dry the sprayed micro particles of the starting precursor solution before they arrived at the substrate surface. This leads to an insufficient wettability between the micro articles and the previously deposited layers, which resulted films with low packing density and many pinholes. But perfume atomizer increases the wettability between sprayed micro particles and the previously deposited layers and thereby enhances the quality of the films. The intermittent spraying process enables the substrate to attain the required temperature before the start of the next spray. Perfume atomizer facilitates fine atomizer of the precursor solution and avoids deposition of large droplets. The block diagram of a simplified spray pyrolysis technique using perfume atomizer is depicted in Fig. 1.

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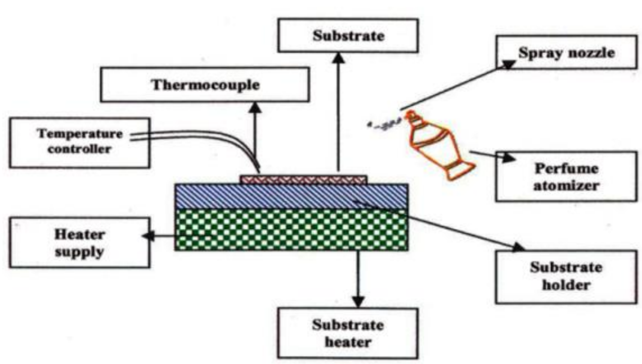

Figure1: Block diagram showing a simplified spray pyrolysis technique

The starting material of CuO thin films was prepared by dissolving Copper acetate in 30*ml* of water with different concentration 0.1,0.2 and 0.3M respectively; samples are labeled as **C1, C2** and **C3**.The deposition temperature of Copper oxide thin films has been kept constant at **673K**. As-deposited CuO thin films were found to be well adherent to the glass substrate. The spray rate was maintained at 2ml/min ,the substrate was kept 30*cm* angle between nozzle and substrate at 45^o.The overall reaction process can be expressed as heat decomposition of Copper oxide in the presence of water and air and its pyrolytic reaction is of the following form:

$$Cu(CH_3COO)_2.H_2O+H_2O \rightarrow CuO+ 2CH_3COOH + H_2O \quad (1)$$

The structural studies of the CuO thin films were carried out by using X-ray diffractometer. The Morphological characterization was done using scanning electron microscope. The optical studies of the CuO thin films were analyzed using UV-Vis-NIR photo-spectrometer.

The CuO films optimized with deposition conditions were used to fabricate the gas sensor with two thick silver pads on two sides of the film to take out electrical contacts. A schematic of the gas sensing setup used for ethanol sensing is discussed in detail by the authors elsewhere [24]. Ethanol was injected into the container and gasified by heating, Air in the atmosphere was used as the diluted gas, which afforded 100 ppm tested vapor.

3. Results and discussion

3.1. Structural Properties

It was seen in the XRD patterns shown in Fig.2that the patterns appear there include peaks which have different intensities and widths. The presence of different peaks such as CuO on the XRD patterns indicates that all copper oxide films have polycrystalline structures, and shows the characteristics peaks corresponding to CuO phase. Further the XRD diffractogram of CuO films deposited at different concentration with constant temperature **(673K)** are shown in Fig. 2, which is matching to the observed and the standard *(h k l)* planes confirm that the deposited films are polycrystalline having monoclinic CuO. The diffractogram has shown the prominent (-1 1 1) peak copper oxide located at 2θ value of (35.74)°with *d* value 2.510Å. Another high intensity peak corresponding to (1 1 1) atomic plane of copper oxide located at 2θ value of (38.95)°with *d* value 2.310Å has also been observed. The position and the *d* values of the diffraction peaks for CuO films are in good agreement with those reported earlier for the spray deposited CuO thin films [25-26]. The intensity of prominent peak is maximum when films have been deposited at different concentration with constant temperature (673K). Also, Diffraction angle (2θ), the inter-planar spacing (*d*), miller indices (h k l), crystal systems the observed and standard intensities (I and Io) of copper oxide thin films are shown in Table.1 which indicates the substrate temperature, sample code, position of the observed XRD peak 2θ(°), observed and standard relative intensity of peak and texture coefficient T *(h k l).*

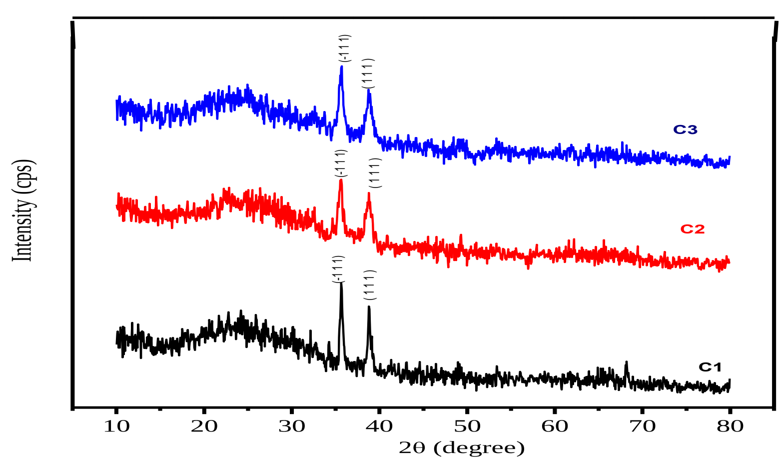

Figure 2: XRD patterns of CuO films of three different concentrations.

Table 1: Diffraction angle**(2*θ*)**, the inter planar spacing(***d***), miller indices**(h k l),** the observed and standard intensities (**I** and $\mathbf{I_0}$) of copper oxide thin films

Substrate tem-perature (K)	**Sample code**	**2*θ*(°)**	***d* (Å)**	**(h k l)**	**I**	$\mathbf{I_0}$
673	**C1**	35.6 38.79	2.51857 2.31989	(-1 1 1) (1 1 1)	100.00 58.29	100.00 100.00
673	**C2**	35.53 38.7	2.52471 2.32240	(-1 1 1) (1 1 1)	100.00 76.53	100.00 100.00
673	**C3**	35.58 38.8	2.52127 2.31973	(-1 1 1) (1 1 1)	100.00 80.39	100.00 100.00

In order to understand the structural properties in detail, the grain size(D), dislocation density (δ) ,number crystallites per unit area (N) , for preferential orientations were calculated and shown in Table.2 using Scherer formulae , as given below.

$$D = 0.94\lambda/\beta\cos\theta \quad \textbf{[nm]} \tag{2}$$

$$\delta=1/D^2 \quad \textbf{[lines/m}^2\textbf{]} \tag{3}$$

$$N=t/D^3 \quad \textbf{[m}^3\textbf{]} \tag{4}$$

whereβ- the half width of peak with maximum intensity (in radian), D-grain size, θ - Bragg angle,λ -wavelength of the light used and*d* -inter-planar spacing.

Table 2: Calculated values of grain size, dislocation density and number crystallites per unit area of CuOfilms of 0.1, 0.2 and 0.3M.

Sample code	**Grain size [nm]**	**Dislocation density [lines/ m²]**	**Number of grain per unit area [m³]**
C1	8.7986	3.98	1.08
C2	9.1230	1.31	3.15
C3	12.354	1.29	5.83

3.2. Thickness measurements

The thickness of the copper oxide thin films were measured by weight gain method using the following equation, that is,

$$T = (W2-W1)/(\rho A) \quad [nm] \tag{5}$$

where T- the thickness of the thin films,W1-the weight of the glass substrate before coating, W2 - the weight of glass substrate after coating, ρ - the density of substrate and A - the area of the substrate.

The thickness of 0.1M, 0.2M and 0.3M concentrated thinfilms found to be 136, 239 and 397 *nm* respectively. It is very evident that the thickness of the thin film is found increase as the concentration of the source material increases as depicted in Fig. 3.

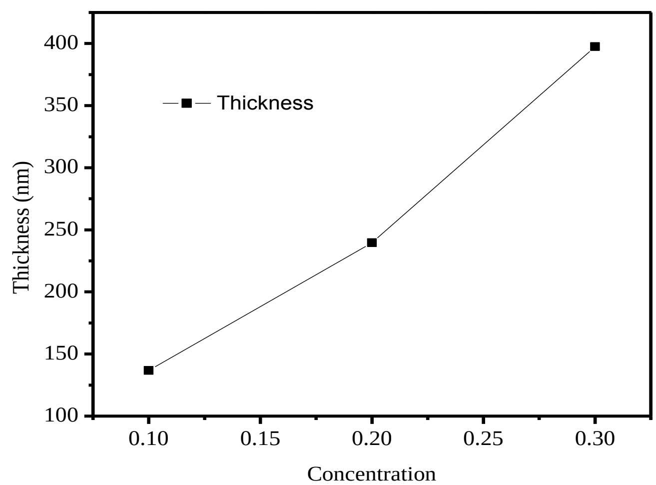

Figure3: Thickness Vs Concentration of CuO thin films.

3.3. The Optical constants: The refractive index (n) and absorption coefficient (α)

Fig.4 shows the plot of absorption versus wavelength of CuO films deposited with different concentration. Determination of the optical constants n and α perhaps one of the most challenging tasks when studying the optical properties of materials since this involves complex equations and a great deal of computing. A number of methods and different approaches exist to determine the optical constants [27-35].

The easiest of them are those, which depend on single transmittance measurement. The refractive index n and the absorption coefficient (α) of the copper oxide thin films studied here were determined from the transmittance data only using PUMA approach. Point-wise unconstrained minimization approach (PUMA for the estimation of the thickness d and optical constant n and α of thin films) software is a procedure described by Birgin et al [36]. This method implements the complex optical equation, shown below, derived and formulated by Heavens [37] and Swanepoel [38].

The linear absorption coefficient ($\alpha = \mathbf{A} / \mathbf{d}$; where **A** is the absorbance and **d** is the thickness of the films) spectra of the copper oxide thin films are shown in Fig. 4. It is inferred from the figure that C1 and C3 films have low absorption coefficient at high wavelengths as compared to the other films. We think that this may be a result of their high transmission values. Besides, it was seen from Fig. 4 that the absorbance decreases with in increases in wavelength range of 400-1100 nm for C3 and C1 sample. But, C2 sample have high absorption co-efficient value attain at 300 nm. So, it was concluded that the fundamental absorption region of C2 films shifts to the higher wavelength. Besides, not all of the films have very sharp absorption edges. This indicates that these films have low crystallinity level. So, it was concluded that all films contain high defect density near the band edge [39].

The refractive indices of copper oxide thin films shown in Fig. 5. It was determined from these figures that the refractive index decreases depending upon the concentration. Decrease of the refractive index indicates that the films density decreases. That is, when the incident light interacts with a material which has low amount of particles, the reflection will be low, and thus the refractivity of the films decreases. It was clearly seen that concentration has the strong effect on the optical properties of copper oxide thin films. The produced copper oxide films are p-type materials; hence their absorption property is a very important parameter. So, as can be Fig. 4, C2 films are suitable films due to their optical absorption properties.

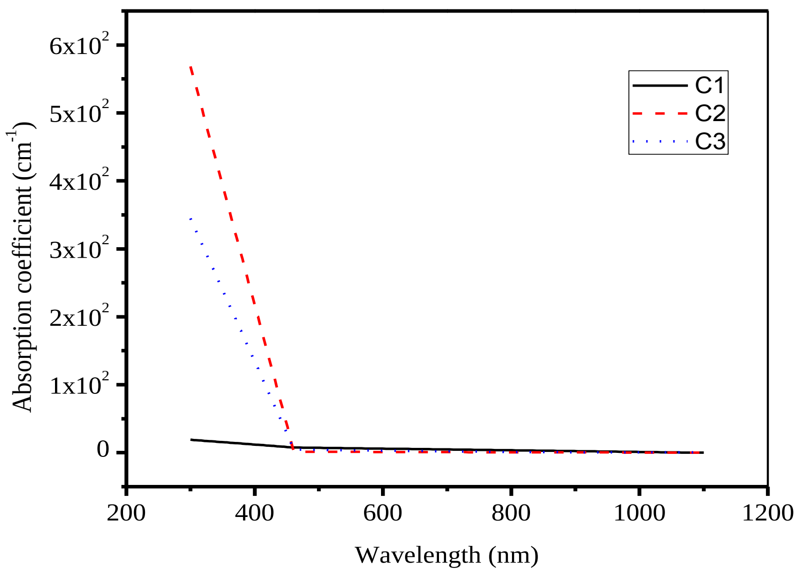

Figure 4: Linear absorption coefficient spectra of CuO thin films

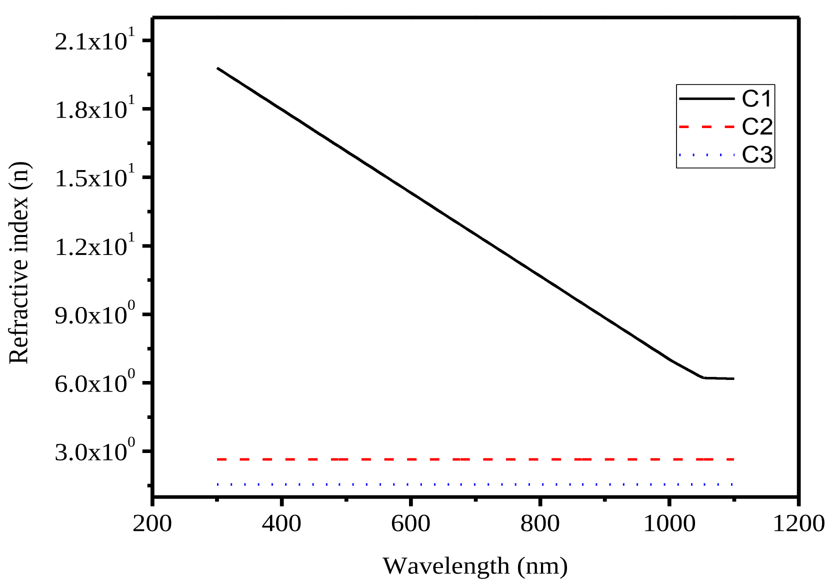

Figure 5: Refractive index of CuO thin films.

3.4. Band Gap measurements

Optical absorption and transmission measurements were found for CuO thin films deposited in different concentration with constant temperature. When plot was made between $(\alpha h\upsilon)^2$ and $h\upsilon$ a linear graph resulted shown in the Fig.6, which clearly suggested the direct nature of band gap in copper oxide thin films. Furthermore, the intercept on hυ axis corresponding to energy determined earlier from the absorption data [40]. The band gap values of the deposited copper oxide thin films being 1.2, 1.3 and 1.4 eV.

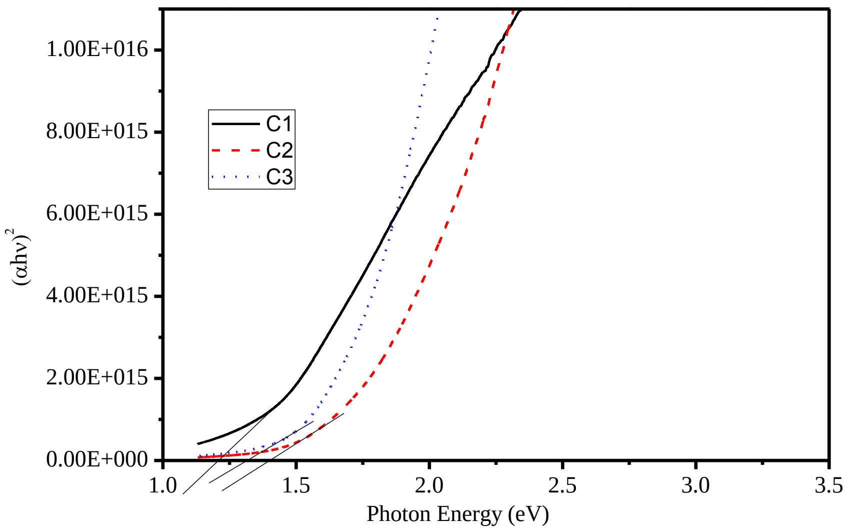

Figure 6: Band gap calculations of CuO thin films.

3.5. Surface properties

As surface property of the film used in photovoltaic applications isanother important physical property, the Scanning electron micrographs were taken to investigate the surface morphologies of the copper oxide thin films and shown in Fig. 7. It was determined that the surface homogeneity of the films improves with increasing the of the starting material concentration at constant temperature **[673K]**. The film is dense, uniform and crack-free indicating that film deposition using simplified spray pyrolysis technique is more effective in achieving crack-free copper oxide films. The particles are of uniform crystallites of spherical type and about 2*μm* in size. These spherical grains are distributed uniformly throughout the film with the above size which is larger than indicated by the XRD pattern. The SEM pictures show that the films were uniform, well adherent and completely devoid of pin holes and cracks. Film with 0.1M of solution concentration has smooth surface grains were perceptible. With 0.2M of solution, the surface starts to modify and smaller granular particles found uniformly distributed all over the surface. When the solution concentration is increased to 0.3M, the surface becomes probably filled with the clusters of the larger grains, as shown in Fig. 7.

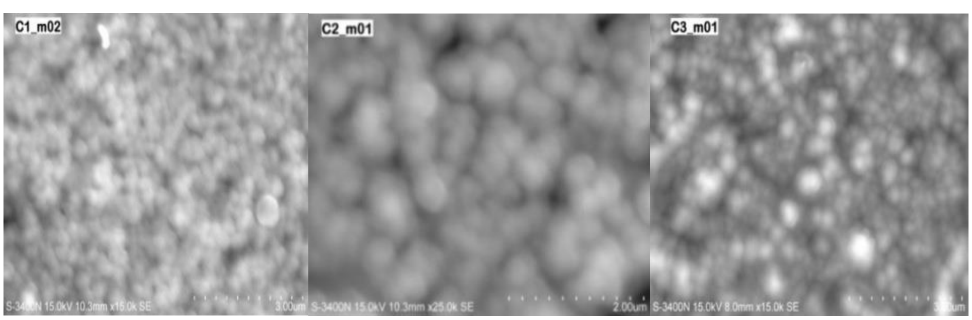

Figure 7:SEM Images ofCuO films of different concentrations

3.6. Elemental Analysis

Energy Dispersive Spectra (EDS) of the copper oxide thin films are shown in Fig.8. As in picture **Cu** and **O** elements are present in the film but **Si, S, Cl, Ca,** and **Mg** are not expected to be in the films and have resulted from the glass substrate. Also, it was clearly seen that the amounts of Cu and O elements increases with different concentration kept at constant temperature **[673K]**. Here, for instance, EDS of Copper oxide thin film of 0.1M concentration only is shown below as the others also depict the same.

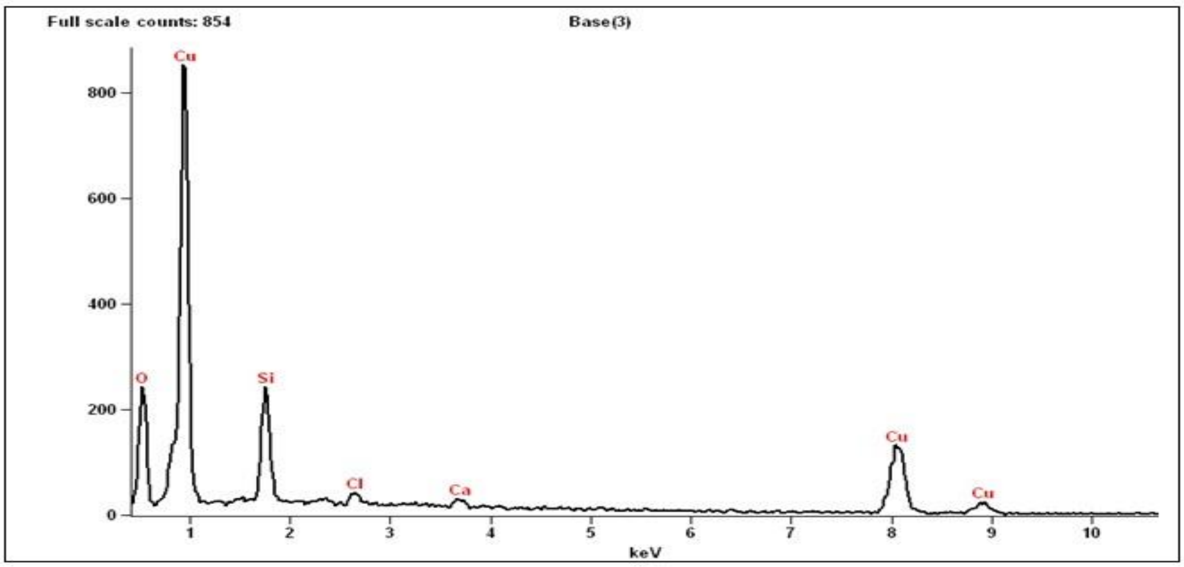

Figure 8: Energy Dispersive Spectra (EDS) of CuO Thin films

3.7. Variation in response time and sensitivity of CuO film sensor

The dynamic response curve is reported for a copper oxide thin film at various concentrations 0.1, 0.2 and 0.3 mol respectively. Upon exposure to ethanol vapour, the sensor decreased upto 140s, as seen from Fig .9 (C1). The reason for decrease in the resistance may be due to the oxidation of the ethanol vapour up on coming in contact with the oxide Semiconducting surface, which liberates free electrons and H_2O. The atmospheric oxygen chemisorbs on the surface of the oxide semiconductor as O^{2-} or O^- removing an electron from the conduction band of the semiconductor ethanol vapors react with the chemisorbed oxygen and re-inject the carrier thereby reducing the resistance of the material. This interaction of oxygen ions on the surface of CuO with ethanol molecules can be described by the following reaction [41]

$$O_{2\,(ads)} + e^- \leftrightarrow 2O^-_{(ads)} \quad (6)$$

$$C_2H_5OH_{(ads)} + 3O_2 \rightarrow 2CO_{2(g)} + 3H_2O_{(g)} + 3e^- \quad (7)$$

The free electrons liberated move into the conduction band of CuO resulting in a decrease in the resistance of CuO film. The response time of the CuO sensor was found to be 140 s Fig. 9(C1). After 140 s the resistance started increasing as the gaseous species on the surface desorbed. However, it took longer time for the sensor response to reach its original value, as the ethanol vapors do not easily absorb. Because of the water liberation, the surface got saturated after sometime, which required cyclic heating and flushing the chamber with hot air.

The resistance of the CuO film was 5 MΩ, 4.1MΩ and2.1 MΩ, while for the 0.3 mol it is only 2.1 MΩ, after exposure to ethanol. It was also observed that the sensor shows much lower resistance for 0.3 mol films Fig. 9 (C3), and the response time of the ethanol was 130 s, indicating that as the grain size increased with increasing concentration, the CuO sensors shows higher response. The behavior of CuO based sensor to ethanol is based on changes in electrical resistance induced by adsorption or desorption of the gas on its surface [42]

Thus, the C3 sample CuO sensor which possesses well-defined microstructure with uniform grain size, revealed large gas response compared to C1, C2 sample. It is suggested that this tendency mainly reflects an increasing order of the mesopore size or Knudsen diffusion co-efficient (D_k) involved among the films [43].

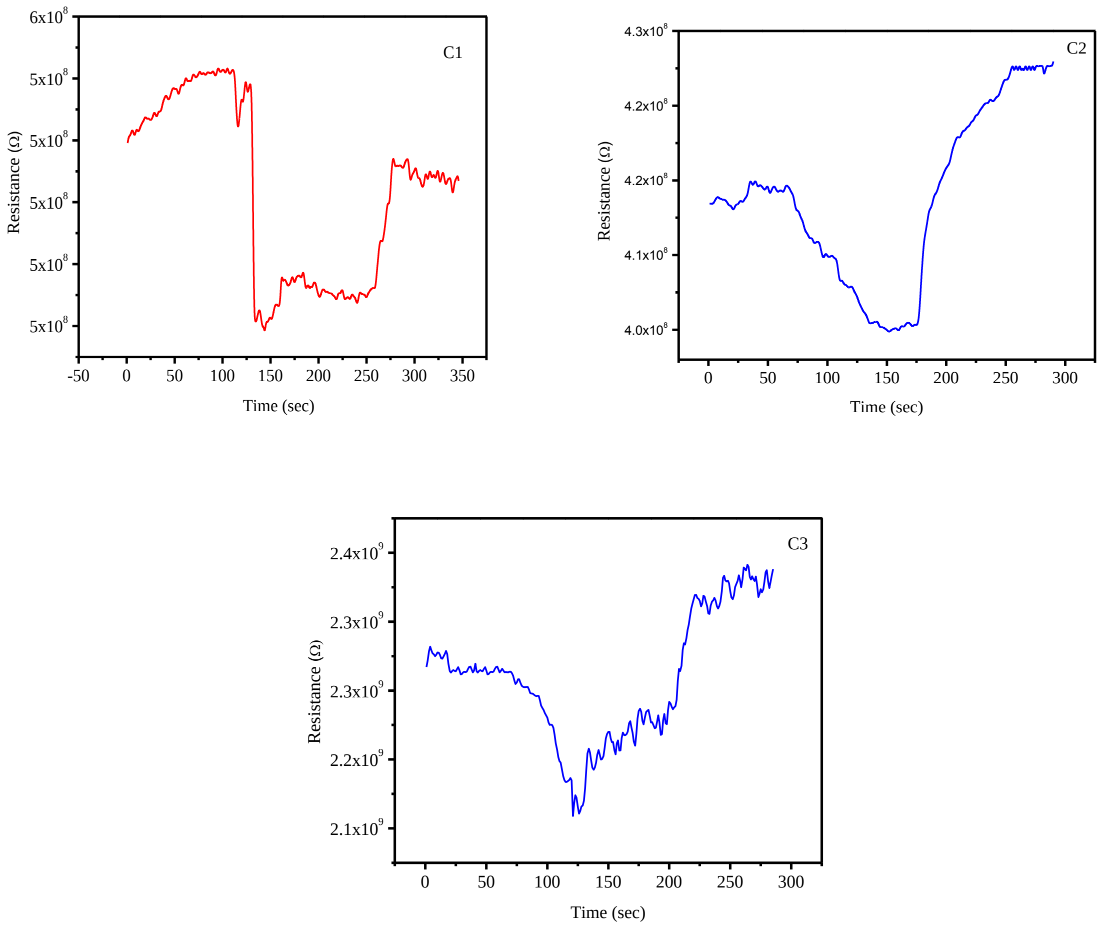

Figure 9: The response of CuO sensor prepared at various concentration, upon exposure to 100ppm of ethanol gas at room temperature.

The sensitivity of CuO gas sensor has been calculated using the expression [44]

$$S = (R_{gas} - R_{air}) / R_{air} \quad (8)$$

Where R_{air} is the sample resistance in dry air and R_{gas} is the resistance in the presence of test gas.

The gas sensing mechanism belongs to the surface controlled type [45] which is based on the change of the electrical conductance of the semiconducting material upon exposure to ethanol vapours. The gas sensitivity is a function of grain size, surface state and oxygen adsorption [46]. The surface area generally provides more adsorption-deposition sites and thus high sensitivity. The ethanol sensing mechanism is based on the change in conduction of CuO thin films, which is controlled by ethanol species and the amount of chemisorbed oxygen on the surface. It is known that atmospheric oxygen molecules are absorbed on the surface. The sensitivity is found to be maximum in C1 sample, It showed maximum sensitivity of 1.07 to 100 ppm of ethanol gas, which may be due to air renders more oxygen vacancy generation, which enhances the gas sensitivity.

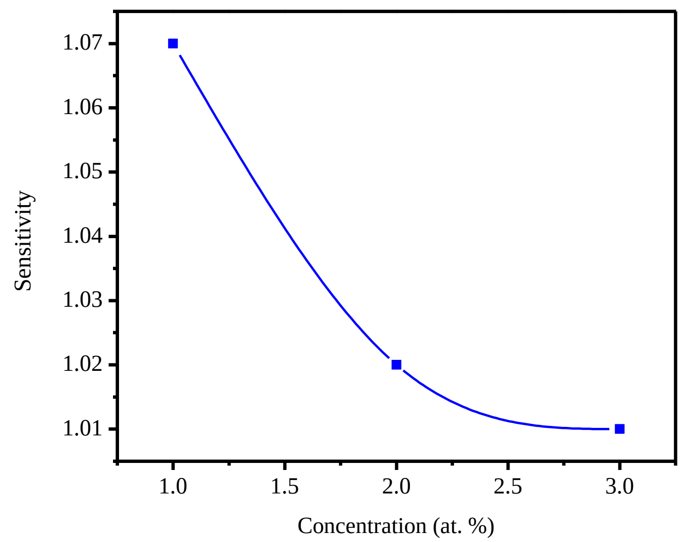

Figure 10: Variation in sensitivity with various concentration of CuO sensor, toward 100 ppm of ethanol.

4. Conclusion

In this paper, the feasibilities of fabricating the Copper oxide thin films (at a constant temperature of **673K)** using a low-cost and much simplified spray pyrolysis technique, were investigated as they are of useful in many applications and in particular, in terrestrial applications. The structural, surface and optical properties of Copper oxide thin films of three different concentrations were studied and reported with adequate results. The response of the 0.1, 0.2 and 0.3M CuO thin films to the ethanol (100 ppm) gas was studied and compared. The 0.3 mol of CuO films showed higher sensor response than the 0.1, 0.2 mol CuO films. The result suggested that 0.3 mol causes significant effect on the sensing performance of CuO to ethanol.

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