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Exploration of Spectroscopic Study of Screen-Printed Tungsten Oxide: Tin Oxide Nano-Composite Powder Fired at High Temperature

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Abstract: ***Thick films of pure Tin oxide (SnO_2) and composite 1%, 3%, 5%, 7% and 9 % tungsten oxide (WO_3) incorporated into pure Tin oxide (SnO_2) were prepared with standard screen printing technique. All samples were prepared on glass substrate and subjected to drying and firing at 500^0C at 5 hours in muffle furnace. Thick films of tin oxide (SnO_2) and composite 1%, 3%, 5%, 7% and 9 % Tungsten Oxide (WO_3) incorporated into pure tin oxide (SnO_2) were examined for Scanning Electron Microscopy (S.E.M), Energy Dispersive X-ray Analysis (E.D.A.X), X-ray diffraction (X.R.D), Fourier Transform infra-Red (F.T.I.R) and Ultra-Violet-visible spectroscopy (U.V) to investigate surface morphology, elemental analysis, crystalline Phases of films, vibrational and spectroscopic study respectively. The spectrophotometric parameters such as absorbance and absorption coefficient were also a part of investigation. The UV-visible spectrophotometric study was encountered in terms of absorbance and absorption coefficient along with varying percentage composition.***
Keywords: morphology, vibrational and spectroscopic study, crystallite phases of films absorbance, absorption coefficient, etc.

I. INTRODUCTION

Recently nanostructures of transition metal oxide have paid much attraction and interest because of their size, shape and high degree of crystalinity. Among all tungsten oxide has good photochromic, electrochromic, electric and photocatalytic properties. Tin oxide (SnO_2) with oxidation number four (+4) plays an important role in oxidizing agents for organic synthesis. Micro-sensors constructed with SnO_2 material is found to be more reliable material because of its use in transistors, electrodes, gas sensors, liquid crystal displays, catalysts, photovoltaic devices, photo sensors, antistatic coatings, polishing and ceramic glazes. Screen printing technique was introduced in the decade of 1950. Screen printing is most popular technique and versatile chemical method to deposit thick films on glass or alumina substrate [1]. Screen printing technique is method to construct robust, compact and relatively cheap hybrid circuit for different technological applications such as gas sensor, electric appliances, transistors and optical devices [2]. Different methods has been developed to grow pure tin oxide (SnO_2) and different composites material of tungsten oxide (WO_3) incorporated into pure tin oxide (SnO_2) such as spray pyrolysis, vaccum evaporation, chemical vapour deposition, magentron sputtering, pulsed laser deposition, sol-gel technique and screen printing technique [3-4]. Among all screen printing method has found to gain popularity for the formation of superconducting oxide films. An advantage of thick film techniques includes fast processing, low cost and economical use of paste [5]. Metal oxides have non-stoichiometric structure, defects in crystal lattice, different morphology and crystallite pattern which make them to employ into electrical and optical devices. Absorption spectra of tin oxide (SnO_2) nanoparticles obtained in UV-Visible region shows blue shift in the absorption edge at 268 nm as compared to bulk [6]. The observed relative peak shift was useful in different applications like thick film resistor and gas sensing. The band gap and absorption edge modification of tin oxide takes place when different dopant material like tungsten oxide (WO_3), Zinc oxide (ZnO), Titanium oxide (TiO_2), Cuprous oxide (CuO), Zirconium Oxide (ZrO_2) and many other binary and ternary phase were added into different varying concentration [7-8]. Tungsten Oxide (WO_3) has absorption edge at 300nm to 360 nm when Tungsten Oxide (WO_3) films were prepared from spray pyrolysis deposition proving the possibility to use Tungsten Oxide (WO_3) to build a photochemical cell as a device for pollutant photo-degradation and/or hydrogen production working in aqueous media at pH=6 [9]. In current research paper a different composition of Tungsten oxide (WO_3): Tin oxide (SnO_2) composite nanomaterial prepared by screen printing method followed by firing in muffle furnace at 500^0C for 5 hours has investigated for structural, morphological and spectroscopic analysis in terms of absorbance and absorbance coefficient with minimal concentration to maximum concentration in solvent phase [10].

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II. MATERIALS AND METHODS

A. *Preparation of Thick Film*

Tin-oxide (SnO_2) and composite thick films of tungsten oxide (WO_3) blended in tin oxide (SnO_2) with percentage composition were prepared on glass substrate with particular dimension by using standard screen-printing technique [11-12]. The analytical grade tin oxide (SnO_2) and Tungsten Oxide (WO_3) chemical powder (99.99 %) was weighed. The calcined Tin oxide (SnO_2) and tungsten oxide (WO_3) powder of different composition was mixed and crushed thoroughly with glass frit which acts as permanent binder and ethyl cellulose acts as a temporary binder. The mixture was then mixed with butyl carbittol acetate as organic vehicle to form the paste. The paste was then screen printed onto the surface of glass substrate [13-16]. The paste was controlled to obtain rheological properties.

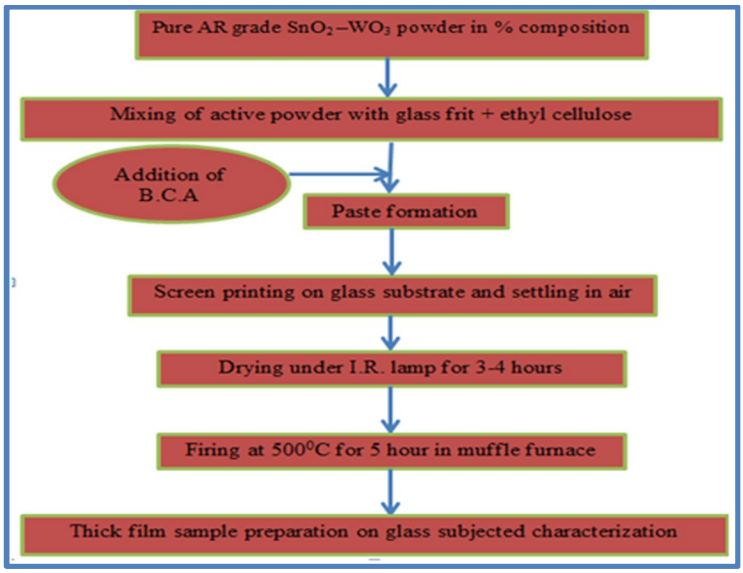

Fig. 1 Flowsheet diagram of preparation of thick films for Nanomaterial

The detail of the technique was described in reference book of Harper [13]. After screen printing the thick films were dried under ordinary visible lamp of 200 watt for 2-3 hr. followed with firing into muffle furnace, dried under air atmosphere at 500^0C for 5 hr. After preparation of pure tin oxide (SnO_2) and tungsten oxide (WO_3)-tin oxide (SnO_2) composite thick films were subjected to structural, morphological and spectroscopic study.

B. *Structural, Morphological and spectroscopic characterization*

1) Using X-ray diffraction (Miniflex Model, Rigaku, Japan) analysis from 20-80, 2θ was carried out to investigate the crystalline phases of the tin oxide (SnO_2) and tungsten oxide additives thick solid films samples. The instrumental broadening was nullified using silicon standard sample. The single line approximation method has selected for the crystallite size determination using high intense peak. The average crystallite size of pure and composite tin oxide thick film samples were measured by using the Scherer formula [17].

$$D = \frac{0.9\lambda}{\beta COS\theta} \qquad (1)$$

Where, 0.9 is shape factor
D-average crystallite size,
λ-1.542 Å (X-ray wavelength),
β - Peak FWHM
θ- Peak position.

2) The microstructure and chemical composition of the thick films of tin oxide and additive material of tin oxide were analyzed using a scanning electron microscope [Nova nano SEM NPEP303] coupled with an energy dispersive spectrometer (EDS JEOL, JED-2300, Germany).

3) FTIR is useful technique to interpret qualitative and quantitative measurement for organic and inorganic samples. It is useful for solids, liquids and gaseous samples and measures the wide range of wavelengths in Infra-red spectra that are absorbed by the material. The FTIR is done by a standard method of solid phase potassium bromide (KBr) pellet technique. The FTIR spectra is recorded at room temperature for pure tin oxide (SnO_2) and composite 1%, 3%, 5%, 7% and 9 % tungsten oxide (WO_3) mixed in pure Tin oxide (SnO_2) in IRAffinity-1 Shimadzu FTIR instrument. A small amount 2 to 5 mg sample powder is mixed in approximate 250mg-300 mg Potassium Bromide (KBr) of spectroscopic grade purity made up of UVASOL Company to form clear transparent proper 13mm circular pallet or disc with 1mm thickness when mixture was pressed in KBr Press Model M-15 at a pressure of about 5×106 Pa in an evacuated die. The following FTIR spectra were recorded between 400 cm^{-1} to 4000 cm^{-1} consist of transmittance at different spectral resolution of 2 cm^{-1} and taking 32 scans for each sample.

4) UV spectroscopy is intense non-destructive testing technique for exploring the properties of semiconductor nanomaterial in terms of absorbance, transmittance and reflectance. Absorbance depends on nature of surface, oxygen deficiency, band gap and impurity centers. The UV absorbance of pure tin oxide (SnO_2) and composite 1%, 3%, 5%, 7% and 9 % tungsten oxide (WO_3) incorporated into pure tin oxide (SnO_2) was recorded on UV spectrophotometer 2012 made analytical technology Ltd. 1mg of all sample powder is dissolved into 10ml 1N H_2SO_4 as a solvant to form clear solution followed by 10 minutes sonication for all samples. A cuvette of 1 cm^3 was employed for measurment with scan step 5 nm with wide range of 200 nm to 800 nm. Duterium amd Tungston lamp was used as a sorce of radiation for broad range with photo-dioade array detector. A solvant was emplyed for dissolution purpose. The solvent effect was nullified as blank reading. The absorption coefficient α is related to absorbance by following equation [18-19]

$$\alpha = \frac{2.303 * A}{d} \qquad (2)$$

Where, α- absorption coefficient
A-Absorbance of material,
d- Path length of cuvette or cell

III. RESULT AND DISCUSSION

A. X-Ray Diffraction Analysis

X-ray diffraction is a plot of peak position with intensity along the spectra. The screen printed thick film of additives of tungsten oxide (WO_3) with tin oxide and tin oxide (SnO_2) base material was highly crystalline and all are well oriented along planes (110), (101) and (211). It confirms the presence of polycrystalline nature. No characteristic diffraction peaks of impurities and other compounds were found as percentage composition was increased from 0% to 9% which were quit consistent with standard peaks of tin oxide [20]. The percentage composition also brought a change in intensities along spectrum. It has been depicted from the figure the polycrystalline nature of spectrum well orient along (110) plane. All observed peaks are well agreement with JCPDS card no. 41-4145 [8]. The higher peak intensities in an XRD pattern along the spectrum are due to better crystalinity and bigger grain size attributed to agglomeration of nanoparticles as depicted in S.E.M images also. The molar density along the spectra was less or more hence observed peaks have low or denser in their intensities. The crystallite sizes were calculated using Scherer formula (eq.1). The average crystallite size lies in the range of 19 nm to 24 nm.

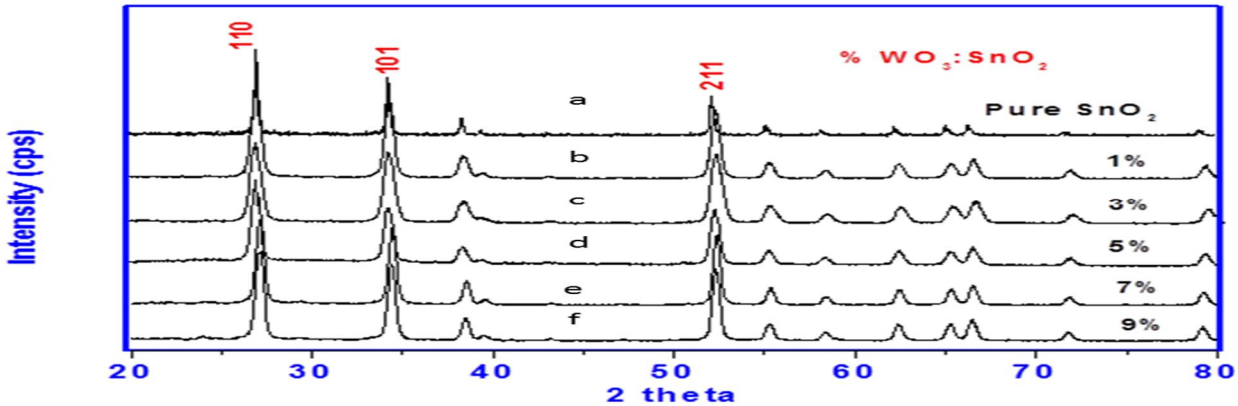

Fig 1: XRD spectra of (a) Pure Tin oxide (SnO_2) (b) 1% Tungsten Oxide (WO_3):Tin oxide (SnO_2) (c) 3% Tungsten Oxide (WO_3):Tin oxide (SnO_2) (d) 5% Tungsten Oxide (WO_3):Tin oxide (SnO_2) (e) 7% Tungsten Oxide (WO_3):Tin oxide (SnO_2) (f) 9% Tungsten Oxide (WO_3):Tin oxide (SnO_2)

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B. Scanning Electron Microscopy and EDAX analysis

Scanning electron microscopy delievers high-resolution imaging which in turn utilized for evaluating various materials to surface fractures, flaws, contaminants and corrosion. Focussed beam of secondary electrons interact with atoms present in sample produces various signals that consists of information about the surface topography and composition of sample [21]. All images were scanned at 30000x with 3 µm dimension scale. The following S.E.M images reveals that all nanocomposite micrograph were well agglomerated meso-porous in nature with interconnected pores. Some of the images has miniature crack into their surface nature. The avarage grain size of scanned images in between 45 nm to 63 nm. The average grain size of nanomaterial is inversly proportional to its avarage surface area. The exposed grain size for any physical or chemical property like absorbance measurement is a function of its surface area.

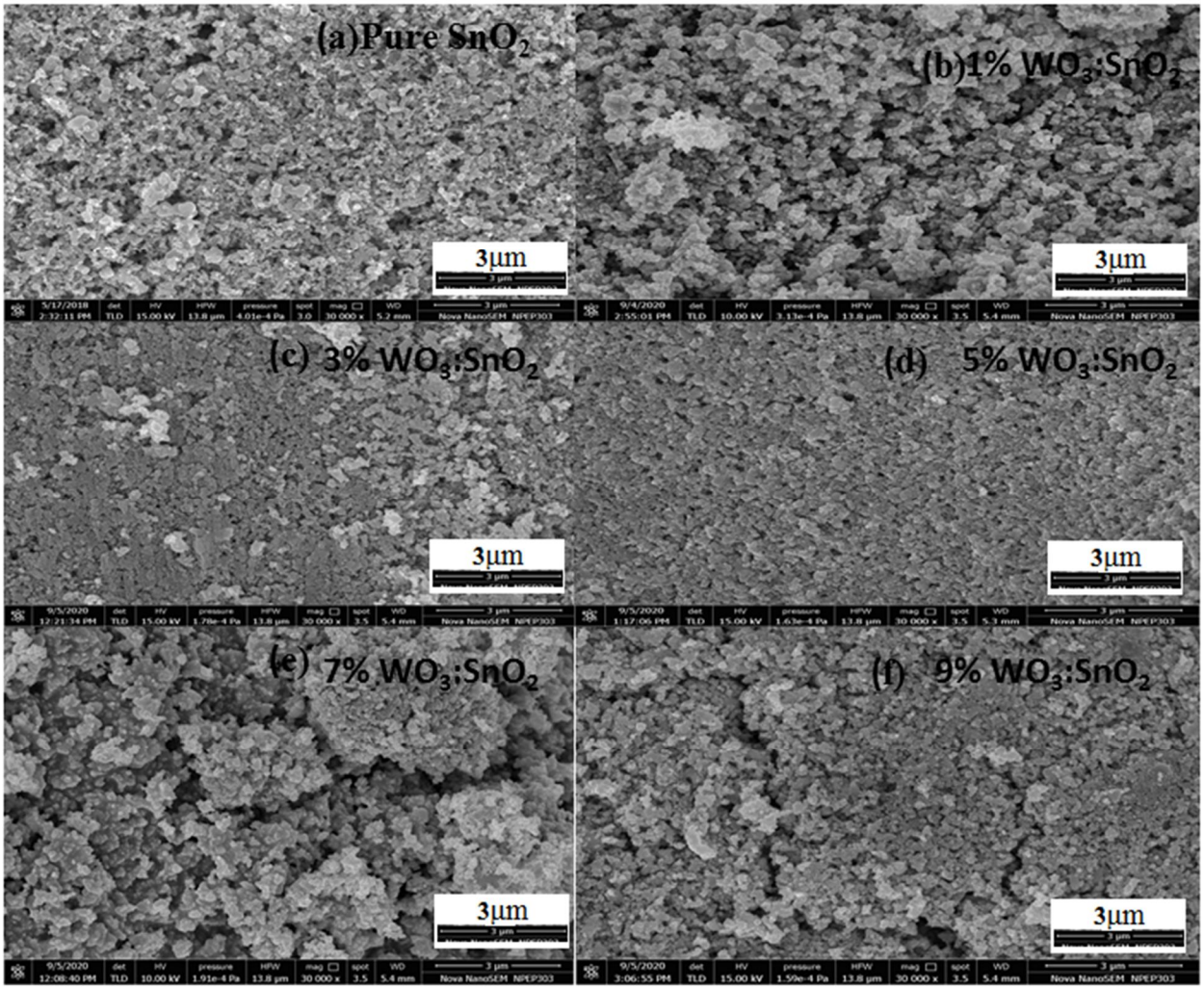

Fig 2: SEM micrographs corresponding to a) Pure Tin oxide (SnO_2) (b) 1% Tungsten Oxide (WO_3):Tin oxide (SnO_2) (c) 3% Tungsten Oxide (WO_3):Tin oxide (SnO_2) (d) 5% Tungsten Oxide (WO_3):Tin oxide (SnO_2) (e) 7% Tungsten Oxide (WO_3):Tin oxide (SnO_2) (f) 9% Tungsten Oxide (WO_3):Tin oxide (SnO_2)

Elemental analysis by E.D.A.X is useful to explain quantitative measurment of elemental analysis and present of qualitative parameter to investigate the presence of nanomaterial. From fig. 3 it has been concluded to presence of pure tin oxide (SnO_2) and composite tungsten oxide (WO_3)-tin oxide (SnO_2) nanomaterial in appropriate mixture, indicating percentage composition were succesfully prepared.

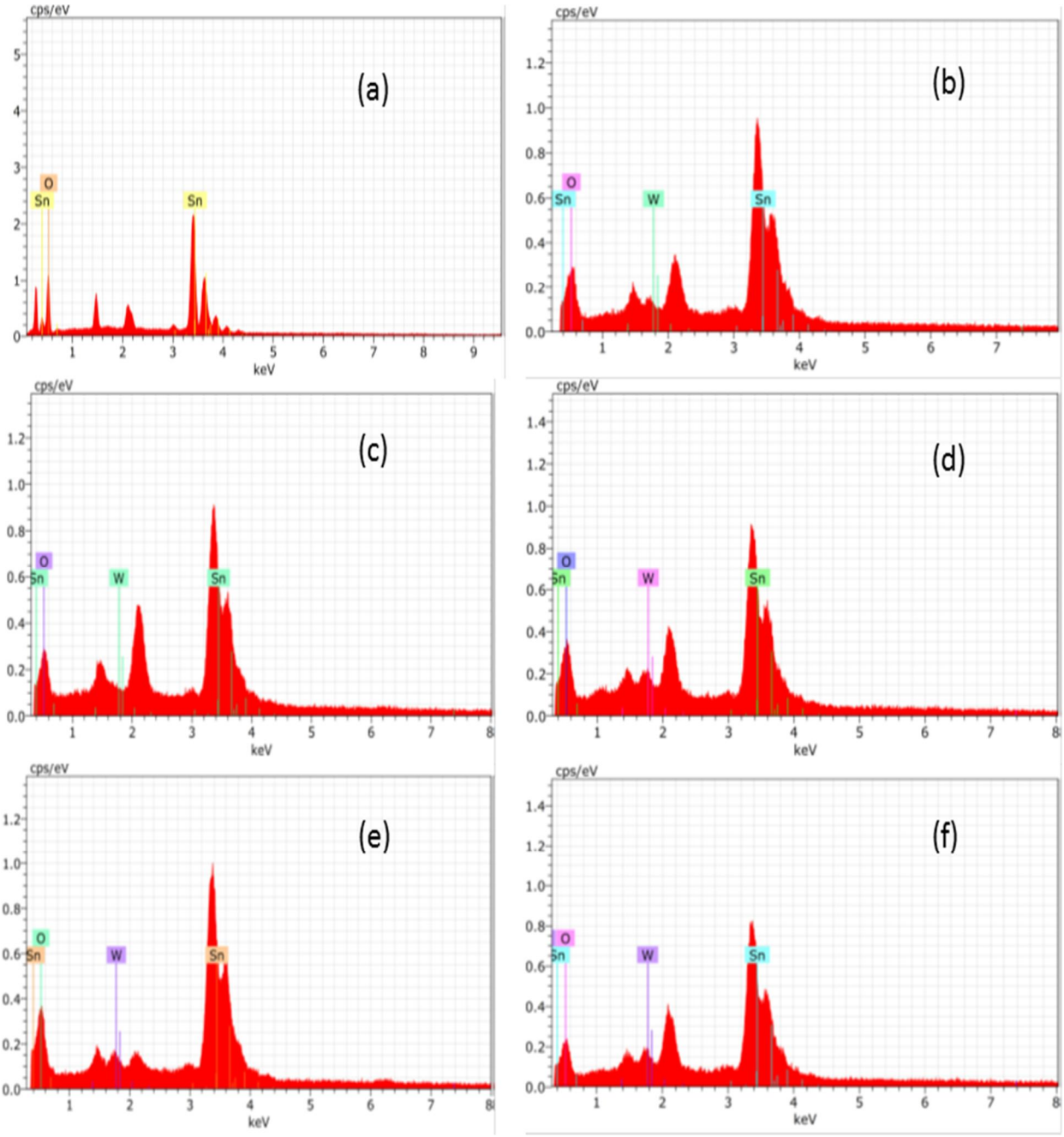

Fig 3: EDAX spectra of corresponding to a) Pure Tin oxide (SnO_2) (b) 1% Tungsten Oxide (WO_3):Tin oxide (SnO_2) (c) 3% Tungsten Oxide (WO_3):Tin oxide (SnO_2) (d) 5% Tungsten Oxide (WO_3):Tin oxide (SnO_2) (e) 7% Tungsten Oxide (WO_3):Tin oxide (SnO_2) (f) 9% Tungsten Oxide (WO_3):Tin oxide (SnO_2)

C. Ftir Analysis

Functional property of composite tungsten oxide (WO_3)-tin oxide (SnO_2) is investigated in fig. 4. The presence of characteristic frequencies is as shown in following figure. It has been evident that the absorbance band at the extreme left at 619 cm^{-1} to 690 cm^{-1} and 329 cm^{-1} to 400 cm^{-1} corresponds to Sn-O and W-O bonds confirming tin oxide (SnO_2) and tungsten oxide (WO_3) nanoparticles $\upsilon(W\text{-}O_{INTER}\text{-}W)$ and a weak shoulder peak is observed for oxygen-oxygen bond at 860 cm^{-1} [22]. The IR band at 1629-1637 cm^{-1} attributes to deformation mode of OH groups and stretching vibrations[23]. The IR bands of broad band region at the 3448 cm^{-1} to 3490 cm^{-1} corresponds to O-H confirming the presence of hydroxyl groups on the surface of thick films of pure Tin oxide (SnO_2) and composite tungsten oxide (WO_3) incorporated into pure tin oxide (SnO_2) [22-23].

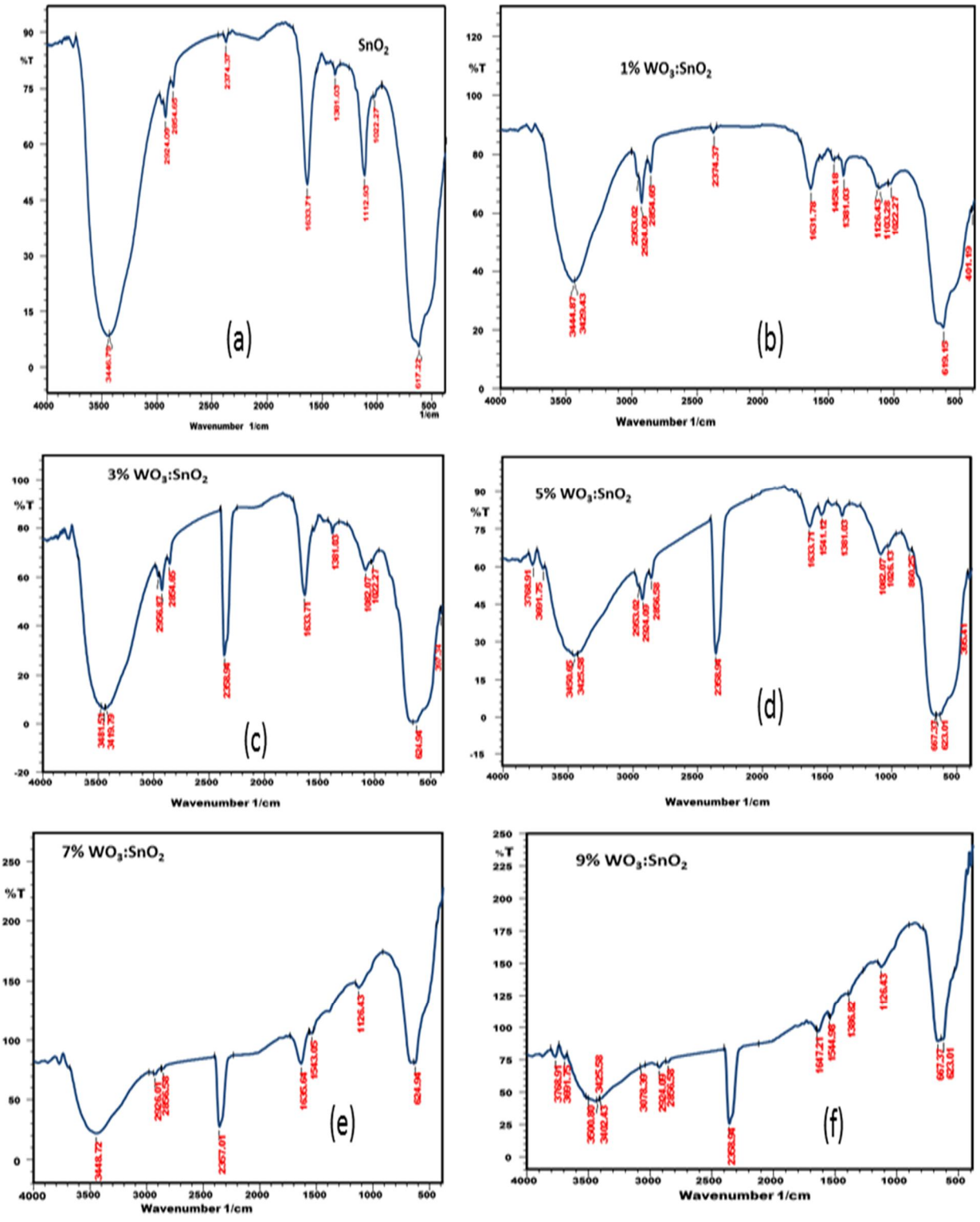

Fig 4: FTIR spectra of corresponding to a) Pure Tin oxide (SnO_2) (b) 1% Tungsten Oxide (WO_3):Tin oxide (SnO_2) (c) 3% Tungsten oxide (WO_3):Tin oxide (SnO_2) (d) 5% Tungsten Oxide (WO_3):Tin oxide (SnO_2) (e) 7% Tungsten Oxide (WO_3):Tin oxide (SnO_2) (f) 9% Tungsten oxide (WO_3):Tin oxide (SnO_2)

D. Spectroscopic Analysis

Spectrophotometer can be divided into according to wavelength and application context. The absorbance of tungsten oxide (WO_3)-Tin oxide (SnO_2) nano powder with different composition has depicted in following figure.

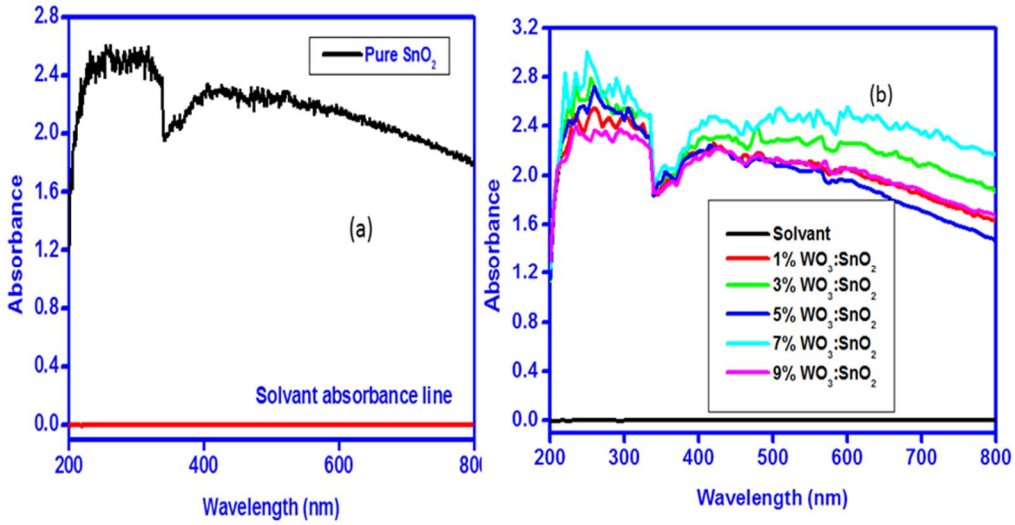

Fig 5: UV-Visible spectra of (a) Pure Tin oxide (SnO_2) (b) Composite % tungsten oxide (WO_3): Tin oxide (SnO_2) and zero absorbance line of solvent

It has been clear to view the zigzag nature of graph. The solvant effect was first nullified as blank reading. path length of cylindrical cuvette/cell was 1 cm^3. Zero slovant absorbnace line clearly indicates that there is no effect of solvant on the absorbance spectra of pure Tin oxide (SnO_2) and composite 1%, 3%, 5%, 7% and 9% tungsten oxide (WO_3) incorporated into pure tin oxide (SnO_2). The variation of absorption coefficient with percentage compososition follows the linear graph which was inclined in downward direction as depicted in fig . 6. Particle size and morphology plays an important role on the spectoscopic investigation of different constituent of tin oxide composite material[21,24]

% Composition of SnO_2-WO_3	Absorbance	Alpha
0	2.6005	5.988952
1	2.5482	5.868505
3	2.7891	6.423297
5	2.7263	6.278669
7	3.0061	6.923048
9	2.404	5.536412

Table.1. indicates Percentage composition of Absorbance (A) and absorption coefficient (α).

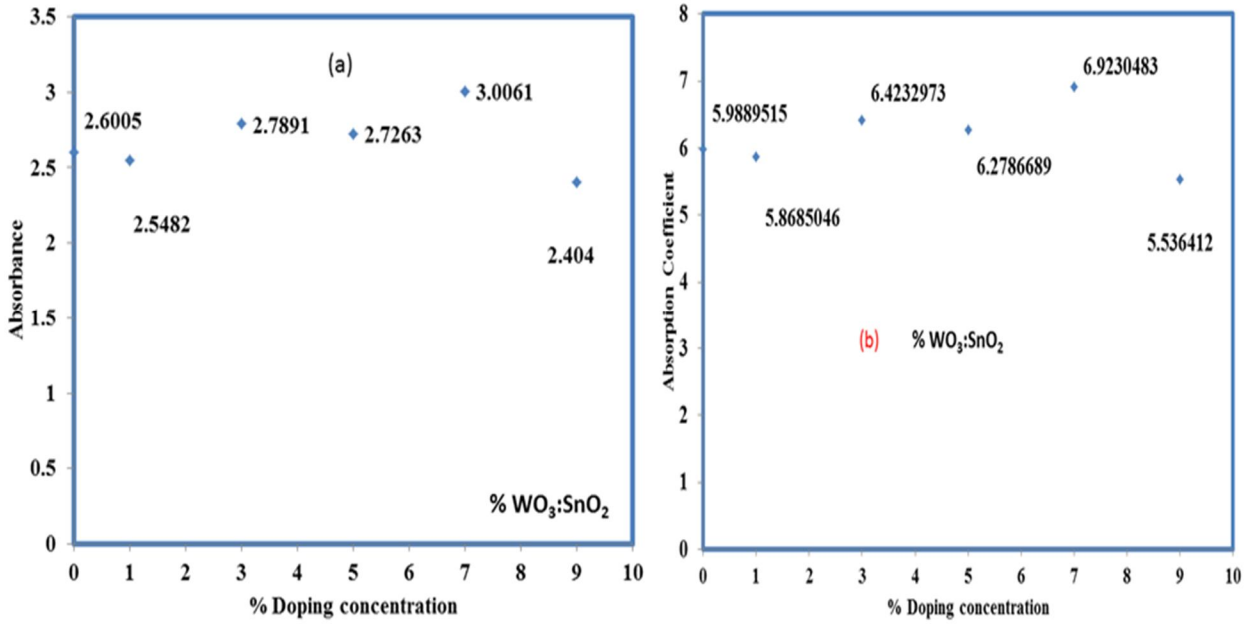

Fig 6: Spectroscopic graph of (a) absorbance Vs. % doping concentration of composite % tungsten oxide (WO_3): tin oxide (SnO_2) (b) Absorption coefficient Vs. % doping concentration of composite % tungsten oxide (WO_3): tin oxide (SnO_2)

IV. CONCLUSION

The screen printed thick films of tungsten oxide-tin oxide (WO_3)-tin oxide (SnO_2) nanomaterial fired at 500^0C for 5 hours are highly crystalline and porous as described in X.R.D. and S.E.M study. Elemental analysis indicates the presence of tungsten oxide (WO_3) - tin oxide (SnO_2) powder. FTIR study reveals bonding and frequencies in transmittance region and gives qualitative aspect. The absorbance of 7 % tungsten oxide (WO_3)-Tin oxide (SnO_2) maximum with absorbance value 3.0061 and corresponding absorption coefficient value is 6.92 cm^{-1}. The absorbance of 9 % tungsten oxide (WO_3)-Tin oxide (SnO_2) minimum to absorbance value 2.404 and corresponding absorption coefficient value is 5.53 $cm^{-1.}$

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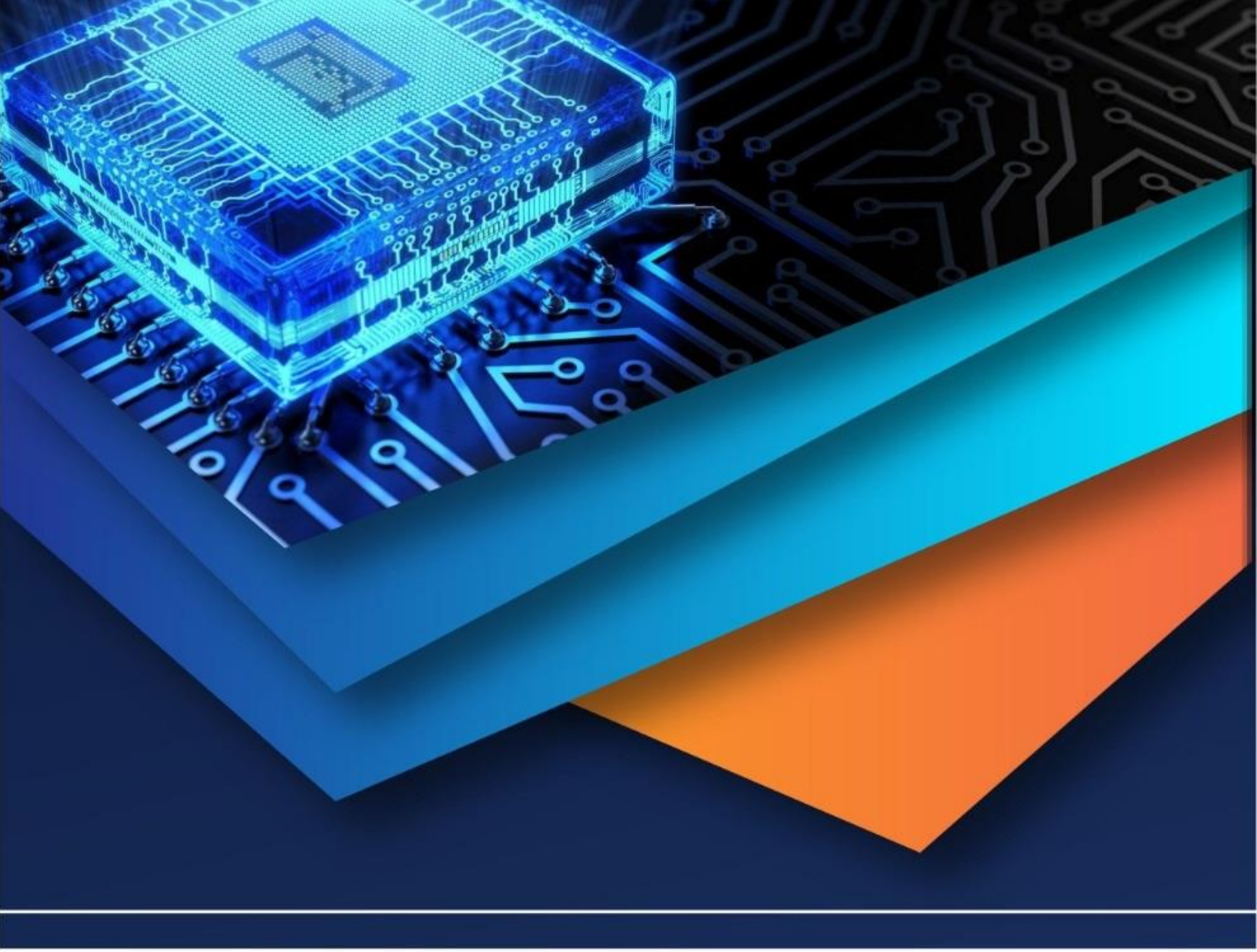

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