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THE EFFECT OF VARIOUS PRECURSORS AND SOLVENTS ON THE CHARACTERISTICS OF FLUORINE-DOPED TIN OXIDE CONDUCTING GLASS FABRICATED BY ULTRASONIC SPRAY PYROLYSIS

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ABSTRACT

Transparent conductive oxide (TCO) glass is one of most important components in dye-sensitized solar cell (DSSC) device. In addition to its high electrical conductivity, transparency is another important requirement that must be achieved in fabricating TCO. One TCO film is fluorine-doped tin oxide (FTO), which can be considered as the most promising substitution for indium-doped tin oxide (ITO), since the latter is very expensive. However, the fabrication techniques for TCO film need to be carefully selected; the synthesis parameters must be properly optimized to provide the desired properties. In this work, FTO glass has been fabricated by the ultrasonic spray pyrolisis technique with different precursors, i.e. tin (II) chloride dihydrate ($SnCl_2.2H_2O$) and anhydrous tin (IV) chloride ($SnCl_4$), as well as different solvents, i.e. ethanol and methanol. For both conditions, ammonium fluoride (NH_4F) was used as the doping compound. The resulting thin films were characterized by use of a scanning electron microscope (SEM), x-ray diffraction (XRD), ultraviolet-visible (UV-Vis) spectroscopy and a four-point probe test. The results of the investigation show that the highest transmittance of 88.3% and the lowest electrical resistivity of 8.44×10^{-5} Ω.cm were obtained with the FTO glass processed with 20 minutes of spray pyrolysis deposition and 300^{o}C substrate heating, using $SnCl_4$ as the precursor and methanol as the solvent. It can be concluded that TCO fabrication with tin chloride precursors and ammonium fluoride doping using ultrasonic spray pyrolisis can be considered as a simple and low cost method, as well as a breakthrough in manufacturing conductive and transparent glass.

Keywords: Ammonium fluoride doping; Anhydrous tin (IV) chloride precursor; Conductive glass; Electrical resistivity; Optical transmittance; Tin (II) chloride dihydrate precursor

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1. INTRODUCTION

Transparent conductive oxide (TCO) glass is one of the most important components in gas sensors, substrates for electro-deposition, optoelectronic devices, hetero-junction photovoltaic cells and dye sensitized solar cells (DSSC). In addition to its high conductivity, transparency is another important criterion for the TCO. Nowadays, the most commonly used and commercialized TCO is indium tin oxide (ITO). However, due to the scarcity of its raw material, i.e. the indium (I) element, the production cost is very high, which consequently results in a high price for ITO glass. Therefore, a new alternative material is needed to substitute ITO. Tin oxide (SnO_2) has been considered as one of the oxides with characteristics sufficiently close to those of ITO, including high chemical stability, mechanical strength, thermal resistance and low cost (Hassanien et al., 2016).

For FTO fabrication, various deposition techniques have been employed, such as evaporation (Muranaka et al., 1986), electron beam evaporation (Shamala et al., 2004), sputtering (Brousseau et al., 1997), spray pyrolysis (Paraskevi & Munkegade, 2008), the sol-gel method (Ganz et al., 1997), and chemical vapour deposition (Ray et al., 1997). In this context, the spray pyrolysis technique has been considered as a simple, low cost, but versatile method, in which the film formation can be practically realized by using an ultrasonic nebulizer. One of the governing factors determining the final characteristics of TCO glass by this method is the thickness of the layer formed during the deposition process as a result of the particles forming a bond on the substrate. It is known that when a liquid is atomized with an ultrasonic nebulizer, the liquid-gas interface is broken and the aerosol is formed. This happens because the rapid expansion and contraction of the bubbles in liquids causes exploitation, or damage, to the surface of the liquid under ultrasonic excitation. Therefore, the use of an ultrasonic nebulizer as an atomizing equipment allows the formation of nanoparticles in the film of desirable characteristics (Raabe, 1976).

FTO fabrication with a simple ultrasonic nebulizer has been carried out by Trisdianto et al. (2016), who demonstrated that the addition of fluorine ions into the tin (Sn) precursor improved the properties of the thin film performance by comparing the undoped and doped samples in the same condition. Another research work using the same technique was conducted by Arini et al. (2016), which confirmed that longer deposition times and increased substrate temperatures increased the thickness of FTO films, resulting in expectedly decreased electrical resistivity. Despite its promising potential, however, there are still challenges in various synthesis conditions affecting the quality of the final TCO glass, including the type of precursors and solvents. The present work is therefore aimed at investigating the effect of using tin (II) chloride dihydrate ($SnCl_2.2H_2O$) and anhydrous tin (IV) chloride ($SnCl_4$) precursors, as well as ethanol and methanol solvents, on the final characteristics of transparent fluorine-doped tin oxide glass.

2. EXPERIMENTAL METHOD

The materials used in this work were tin chloride dihydrate ($SnCl_2.2H_2O$), anhydrous tin (IV) chloride ($SnCl_4$), ammonium fluoride (NH_4F), distilled water, ethanol and methanol. All the chemical reagents had analytical grade purity and were purchased from Merck. The FTO fabrication process was started by cleaning the commercial soda lime glass substrates using a normal detergent, followed by ethanol immersion in a glass beaker. To complete the cleaning process, both the glass substrates and the beaker were ultra-sonicated for 15 minutes. The substrates were further dried in an oven at 60^oC overnight before being used.

The tin precursor solution was prepared by dissolving 12 grams of $SnCl_2.2H_2O$ in 100 ml of ethanol and stirring this mixture for 30 minutes. In turn, the fluorine doping solution was made

by dissolving NH_4F in distilled water, followed by rigorous stirring until a homogeneous solution was obtained. This doping solution was added with a dropper to the tin precursor solution, and the mixture was stirred for 30 minutes. The ratio of NH_4F to $SnCl_2.2H_2O$ was fixed at 2 wt%. The same process was also conducted with the methanol solvent. In this work, another variation was made by using a different tin precursor, i.e. anhydrous tin (IV) chloride ($SnCl_4$) dissolved in each ethanol and methanol solvents. For this purpose, 8.18 ml of the $SnCl_4$ solution was dissolved in 91.82 ml of ethanol. Furthermore, as much as 2 wt% of the NH_4F solution was added dropwise into the former tin precursor solution over a period of one hour. Consistent with the aim of the study, the same experiment was also performed for the $SnCl_4$ precursor with methanol as the solvent.

Moreover, the deposition process of the mixture solutions on the soda lime substrates for FTO fabrication was carried out by using spray pyrolysis with an ultrasonic nebulizer (GEA Medical 402A1). The work was performed at a distance of 10 cm and a speed of ± 30 ml/10 min, at a fixed heating temperature of 300°C and with deposition times of 20 minutes. The characterizations of the fabricated FTO glass were performed with scanning electron microscope-energy dispersive spectroscopy (SEM-EDS; JEOL-JSM 6390A), x-ray diffraction (XRD; Shimadzu XRD-7000), ultra violet- visible spectroscopy (Thermo UV-Vis Genesys 10s), and a four-point probe (FPP5000).

3. RESULTS AND DISCUSSION

The current study is mainly concerned with the effect of various tin precursors and solvents during the spray pyrolysis process of the glass substrates on their final characteristics. For the study purposes, SEM-EDS observation was carried out first on the fabricated glass with the following variations: (a) $SnCl_2.2H_2O$ with ethanol; (b) $SnCl_2.2H_2O$ precursor with methanol; (c) $SnCl_4$ precursor with ethanol; and (d) $SnCl_4$ precursor with methanol. All the setting combinations in the experiment were accomplished at a fixed heating temperature of 300°C and 20 minute deposition periods. It can be seen that by using the $SnCl_2.2H_2O$ precursor with the ethanol solvent, as depicted on the right hand side of Figure 1a, solid-interconnected and large particles with an average size of 0.91 μm were found to cover the substrate surface. By using the methanol solvent with the same precursor, as demonstrated in Figure 1b, the particles grew smaller, with an average size of 0.50 μm, while quite a large number of holes were still found among the particles, causing several porous voids.

On the other hand, fabricating the FTO with the $SnCl_4$ precursor which was dissolved in the ethanol solvent has produced a layer consisting of individual and separated small particles with an average size of 0.51μm (Figure 1c). Here, several pores were also observed among the particles. Figure 1d shows that by using the methanol solvent, the resulting FTO film looks more solid, with an average size of 0.76 μm. It is clearly seen that the particles become more compact and interconnected, accompanied by a significant reduction in pores. A previous study showed that FTO with many grain boundaries causes high electrical resistance values (Muruganantham et al., 2011).

Cross sectional SEM images on the right hand side of Figures 1a–1d also provide information about the thickness of the fabricated FTO films, which were measured as 2.38 μm, 600 nm, 340 nm and 1.06 μm with the variations of $SnCl_2.2H_2O$+ethanol, $SnCl_2.2H_2O$+methanol, $SnCl_4$+ethanol, $SnCl_4$+methanol, respectively. Based on the energy dispersive spectroscopy (EDS) results as presented in Table 1, it can be seen that O and Sn elements were found on the surface of the fabricated FTO thin films, which can be traced back to the origin of the tin hydrate precursor used in the current spray pyrolysis technique. The same case was also found

with the Fluor (F) element, which was originally from ammonium fluoride. It is highly possible that the other elements found were from the soda lime glass itself (Karlsson et al., 2012).

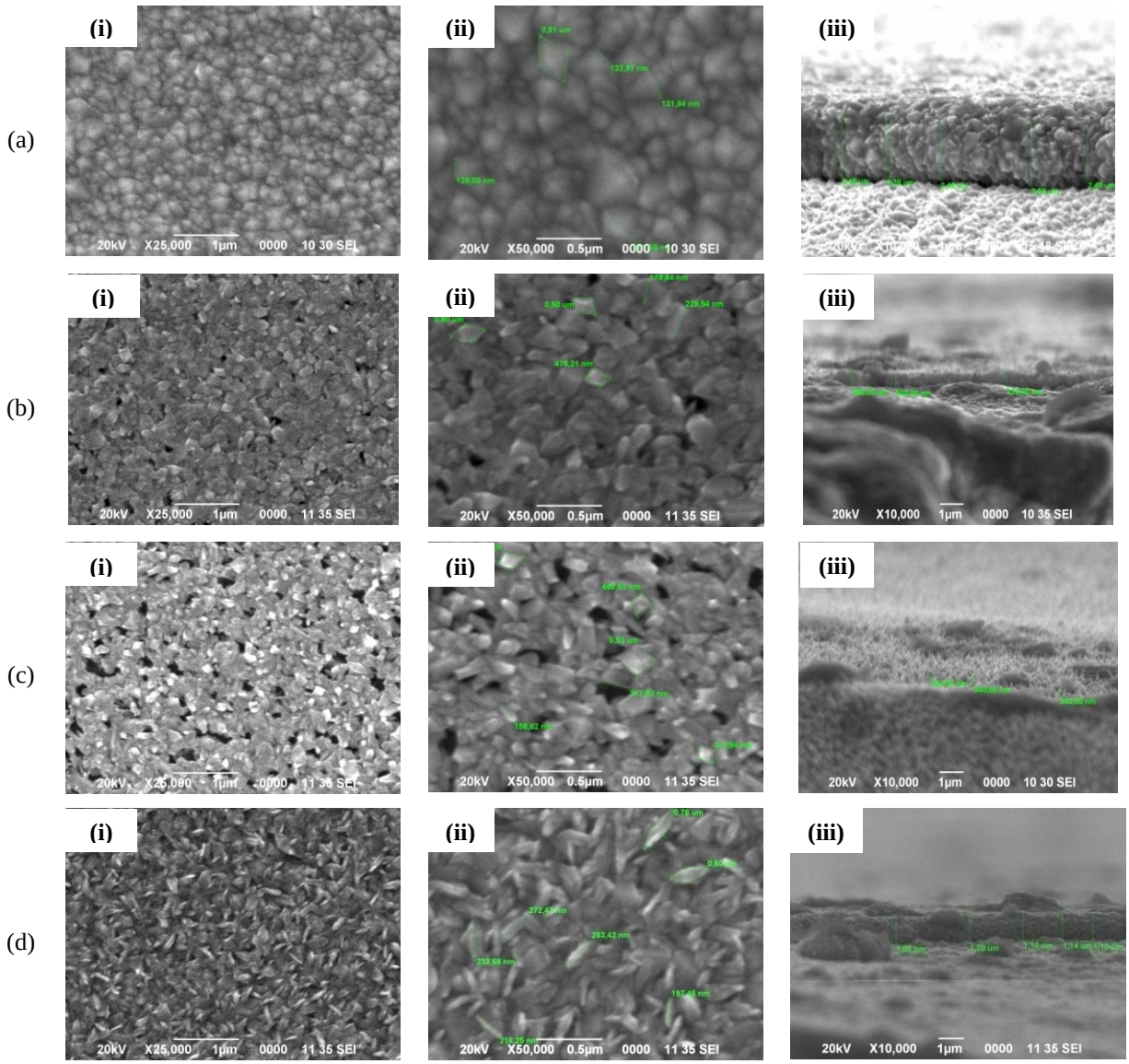

Figure 1 Top view (left) and cross section (right) SEM images of 2 wt% fluorine doped thin films fabricated at a substrate heating temperature of 300°C and 20 minute deposition time, with precursor and solvent variations of: (a) $SnCl_2.2H_2O$+ethanol; (b) $SnCl_2.2H_2O$+methanol; (c) $SnCl_4$+ethanol; and (d) $SnCl_4$+methanol

Figure 2 shows the diffractogram of FTO thin films under various tin precursors and solvents. Several peaks of 26.51, 33.76, 37.84, 51.61, 61.69, and 65.75^o appear at 2θ degrees, which can be associated with (110), (101), (200), (211), (310), and (301) crystal planes, respectively. This confirms the tetragonal rutile structures of the cassiterite SnO_2 phase, as has been previously reported by Banyamin et al. (2014).

A previous research carried out by Noh et al. (2012) indicates that the SnO_2 has (110), (101), (211) and (200) crystal planes depending on the composition of the precursor and the layer thickness. Figures 2c and 2d show the crystal planes (110), with the ethanol solvent having a higher peak than that of methanol.

Table 1 Semi-quantitative EDS-SEM analysis of FTO thin films with a variety of tin (Sn) precursors and solvents at 2% wt fluorine doping, substrate heating temperature of 300°C, and 20 minute deposition time

Element	% mass			
	$SnCl_2.2H_2O$		$SnCl_4$	
	Ethanol	Methanol	Ethanol	Methanol
O	13.14	20.26	21.62	15.35
F	0.58	0.74	0.41	0.30
Na	-	-	3.15	-
Mg	-	-	1.44	-
Si	-	14.47	25.45	1.29
Cl	-	0.42	0.50	-
Ca	-	3.64	6.86	-
Sn	86.28	60.46	40.58	83.05

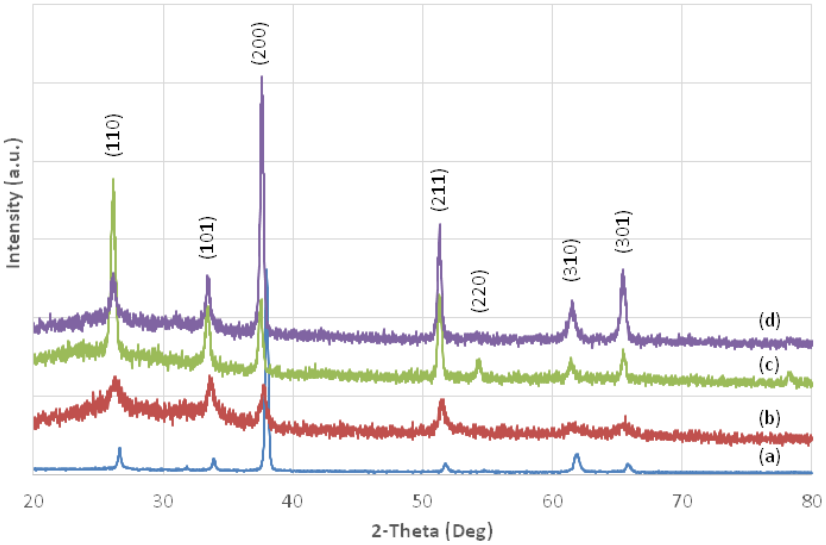

Figure 2 X-ray diffraction pattern of 2 wt% fluorine doped SnO_2 thin films fabricated with a substrate heating temperature of 300°C and 20 minute deposition time with precursor and solvent variations of: (a) $SnCl_2.2H_2O$+ethanol; (b) $SnCl_2.2H_2O$+methanol; (c) $SnCl_4$+ethanol; and (d) $SnCl_4$+methanol

According to their investigation, it was proposed that a tendency of (110) and (200) crystal planes to grow occurred when the coating thickness increased. This is also in line with another study by Korotcenkov et al. (2000), confirming that the presence of (110) and (200) crystal planes depends on the thickness of the coating. Our current study has confirmed this phenomenon; as can be seen in the SEM cross-sectional images on the right hand side of Figure 1., a thick layer of SnO_2 for $SnCl_2.2H_2O$+methanol and $SnCl_2.2H_2O$+ethanol increased from 600 nm to 2.38 µm, while the $SnCl_4$+ethanol and $SnCl_4$+methanol layer thickness increased from 340 nm to 1.06 µm. Figures 2c and 2d also show that the (211) crystal plane fabricated with ethanol solvent has a lower peak than that of methanol. This result is in line with a previous work by Hassanien et al. (2016), which proposed that the change in the (200) and (211) crystal planes depends on the solvent used for solution preparation. Agashe and Mahamuni (2010) observed that the intensity of the (211) peak increased with the growth rate of SnO_2 thin layers, which can be controlled by the type and concentration of precursors. Furthermore, a research by Muruganantham et al. (2011) proposed that the growth rate increases as the concentration of solution increases. Another study by Tatar et al. (2013) found

that the orientation of cassiterite SnO_2 crystal planes on the glass substrate is influenced by the major components: solvent, concentration, speed, deposition temperature and spray pressure.

Table 2 shows the results of the resistivity measurement on the FTO films fabricated with different precursors and solvents. It can be seen that the FTO samples prepared by using the $SnCl_2.2H_2O$ precursor provided lower resistivity values compared with those of the $SnCl_4$ precursor. This result shows that chemical processes occurred during the pyrolytic reaction, leading to the formation of the SnO_2 compound. It has been suggested by Gordillo et al. (1994) that in the $SnCl_2$ precursor, Sn and Cl atoms are bound by ionic bonds, whereas in the $SnCl_4$ precursor, the Sn and Cl atoms are bonded with covalent bonds. In the case of the $SnCl_2.2H_2O$ precursor, the FTO films fabricated by using the ethanol solvent have a resistivity value as low as 3.32×10^{-5} Ω.cm, in comparison to that of methanol, which has a value of 2.62×10^{-4} Ω.cm. This result can be correlated to the chemical nature of $SnCl_2.2H_2O$, which is highly soluble in ethanol (Patnaik, 2003).

Table 2 Resistivity measurement results of the FTO films fabricated by using precursors and solvents at a ratio of 2% wt fluorine doping, substrate temperature of 300°C, and 20 minute deposition time

Precursor	Solvent	Resistivity (Ω.cm)
$SnCl_2.2H_2O$	Ethanol	3.32×10^{-5}
	Methanol	2.62×10^{-4}
$SnCl_4$	Ethanol	2.04×10^{-2}
	Methanol	8.44×10^{-5}

In the case of the $SnCl_4$ precursor, the FTO film fabricated with the methanol solvent has a comparable resistivity to that of the $SnCl_2.2H_2O$ precursor in ethanol, i.e. the same order of 10^{-5} Ω.cm. This value is much lower than that of FTO film with the $SnCl_4$ precursor but dissolved in ethanol, which still has an order of 10^{-2} Ω.cm. The result can be correlated again by the chemical characteristic of solubility of $SnCl_4$ in methanol solvent (Patnaik, 2003). When an $SnCl_4$ compound is dissolved in methanol, the hydrated SnO_2 molecules are formed. An exothermic reaction takes place when part of the $SnCl_4$ in methanol ionizes to Sn^{4+} and Cl^-, and the remainder reacts with alcohol to form complex molecules such as $SnCl_4$. $2CH_3OH$ and $SnCl_4$. $5CH_3OH$. The second complex is neutral, and thus can react to form SnO_2 on the substrate. The solution is sprayed onto a hot substrate and decomposes to form thin films F: SnO_2 (Moholkar et al., 2008).

The results of electrical resistivity given in Table 2 above can also be correlated to the resulting nature of the particles, as well as the crystal growth of the SnO_2 phase, as has been shown with the SEM and XRD analyses in Figures 1 and 2. For the $SnCl_2.2H_2O$ precursors, the values decreased significantly down to one order lower, i.e., from 2.62×10^{-4} to 3.32×10^{-5} Ω.cm when the solvent was changed from methanol to ethanol. With the use of the methanol solvent (Figure 1b), the spray pyrolysis process resulted in small particles accompanied by pores or voids as a consequence of incomplete connections between the particles. These pores acted as a sink for electronic charges, resulting in the resistivity of the film being higher. When the solvent was changed to ethanol, the SnO_2 crystallites grew larger, and interconnection was well established (Figure 1a). Additionally, the thickness of the film increased to 2.38 μm. As demonstrated visually by the compacted particles and reduced porosity in Figure 1a, the electronic transfer between particles became easier, and resistivity was reduced significantly (Zhao et al., 2008).

The opposite result occurred when the $SnCl_4$ precursor was dissolved in ethanol and methanol. Here, the resistivity values decreased more significantly down to three order lower i.e. from 2.04×10^{-2} to 8.44×10^{-5} Ω.cm, when the solvent was changed from ethanol to methanol. With the use of the ethanol solvent (Figure 1c), the resulting particles were quite small but still accompanied by the presence of pores or voids, which caused incomplete connections between the particles, and thus higher electrical resistivity. By contrast, when methanol was used as the solvent (Figure 1d), the SnO_2 crystallites grew larger, and the particle interconnection was well established, accompanied by an increase in film thickness, up to 1.06 μm. As a result, electronic transfer between the particles became easier, and thus electrical resistivity was significantly reduced.

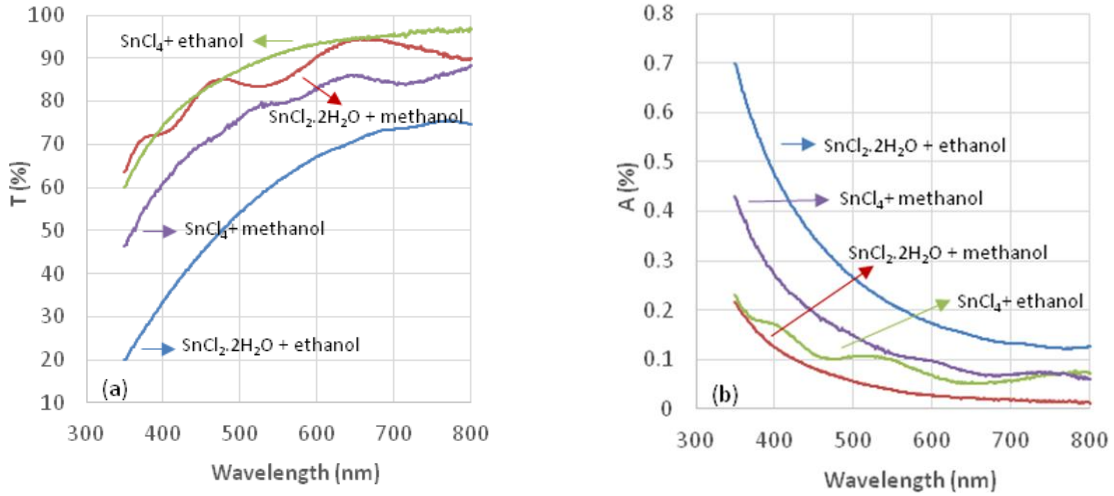

Figure 3 UV-Vis spectra with: (a) transmittance; and (b) absorbance modes for FTO thin films fabricated with various precursors and solvents, a doping ratio of 2% wt, 300°C substrate heating temperature, and 20 minute deposition time

UV-Vis spectroscopy was performed to evaluate the effects of various precursors and solvents on the transparency of the fabricated FTO films, and the results are presented in Figure 3. The highest optical transmittance of ~97% was obtained by the FTO glass fabricated with $SnCl_4$+ethanol, and the use of methanol for the same precursor lowered the optical transmittance to 88.3%. Such a decrease could be related to the increases in thickness of the FTO film, as revealed by the cross-sectional SEM results of the samples on the right hand side of Figures 1c and 1d. The same thing happened for the $SnCl_2.2H_2O$ precursors, where the optical transmittance value obtained with the FTO film processed with $SnCl_2.2H_2O$+methanol was ~95%. This value decreased to ~76% for the sample derived from $SnCl_2.2H_2O$+ethanol. Again, such decreases were due to the increases in thickness of the FTO film, as shown on the right hand side of Figures 1a and 1b.

4. CONCLUSION

On the basis of the investigation, it has been found that the use of different tin precursors and types of solvents during the spray pyrolysis technique using an ultrasonic nebulizer affected the characteristics of FTO films. In the case of the $SnCl_2.2H_2O$ precursor, the use of an ethanol solvent produced better thin layer characteristics compared to the use of methanol. However, an opposite trend was observed for the $SnCl_4$ precursor, where a better quality FTO film was obtained by using methanol instead of ethanol. The optimum value achieved in the study was shown by FTO film fabricated with $SnCl_4$ precursor in a methanol solvent with an electrical resistivity value of 8.44×10^{-5} Ω.cm and an optical transmittance value of 88.3%.

5. ACKNOWLEDGEMENT

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