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Fabrication of Fluorine-Doped Tin Oxide (FTO): From experiment to its application in physics learning

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Abstract: The paper reports the experiment in the laboratory in making FTO by employing spray pyrolysis methods, its application in DSSC, and its implementation in physics learning. The methodology in this research was mixed methods (experiment and qualitative methods). The experimental section was conducted by dissolving $SnCl_2.2H_2O$ and NH_4F in 96% alcohol and then depositioned on a glass substrate on a hotplate with a temperature of 450◦C using an Omron NE-C28 nebulizer. Spraying was carried out for 20 minutes then characterized morphology (SEM), content (EDS), crystal structure (XRD), transmittance (UV-VIS), and voltage-current (IV). Based on investigations using Scanning Electron Microscopy and Electron Dispersive Spectroscopy, it is known that transparent conductive SnO_2:F particles have been formed. By using XRD, Nanometer-sized crystalline particles have also been identified. Furthermore, the FTO electrode is utilized as an electrode on DSSC and its performance is measured. In the qualitative section, students and researchers were interviewed about the physical aspect of the FTO fabrication process. Then, interview results were used to design laboratory physics learning.
Keywords: FTO; spray pyrolysis; resistance; transmittance

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Introduction

Laboratory work carries out several procedures and creates products with certain specifications (Wilujeng et al., 2019) especially in Physics. The laboratory works in physics increase high chances for students to exercise scientific knowledge in the laboratory (Nggadas et al., 2019). Through experiments, students are systematically exposed to different ways of scientific method which are understand the method, get to know some equipment and perform analysis studies. Other great opportunities also involves activities that requirere modelling and remaking some products.

One of the products that can be produced in the laboratory is a fluorine-doped tin oxide (FTO). Fabricating FTO is an interesting experiment because its often used to replace full name of ITO* (ITO). On the other hand, ITO require a high cost to make (Way et al., 2019). The exercise is targeting at harnessing student's science process skills which can be improved through many related activities. This is due to FTO fabrication that produce products in the form of conductive transparent glass. The process of producing the product are equally important during experiment for them to learn technological developments. Such as, FTO is used as one of the components of energy conversion tools, namely dye-sensitized solar cells (DSSC). Therefore planning an experiment with clear context for technology application is expected to increases student enthusiasm for learning (Prabavathy et al., 2021).

In previous studies (Saehana et al., 2019; Darsikin et al., 2018), FTO was generated using the spray pyrolysis method. The FTO is then applied as an electrode in a DSSC solar cell. The photovoltaic effect can be observed under sunlight, then the current and voltage are measured and the efficiency is calculated. The process of making FTO up to its application to DSSC solar cells involves physical and chemical processes related to several physics materials taught in high schools to universities (Tan et al., 2021). At the university level, several courses are relevant to the processes that occur in the laboratory in FTO fabrication, such as the Modern Physics course.

In modern physics courses taught in universities, some physical phenomena such as the photovoltaic effect (Ochieng, 2020) and x-ray spectroscopy (Cruz, 2022). So far, the learning of these two materials is limited for their theoretical aspects, hence students faced difficulties to bulit understanding. The likelihood for students to understand material development can be slimmer if experiment is poorly design during teaching and learning. Therefore, experimentation design in teaching and learning for material development is needed, this involves examples of material fabrication applications and their applications. The aim of the experiment thereby not only targeting at executing fabrication but expand students' insights which lead to greater learning outcomes.

In the FTO fabrication experiment, several physical aspects can be learned by students through a series of existing experiments. The manufacturing process with the spray pyrolysis method involves physical and chemical processes. Mixing the $SnCl_2$ solution with NH_4Cl causes a reaction to form SnO_2:F which is then deposited on a glass substrate with the help of a nebulizer. In addition, the existence of electronic equipment such as solar cells is also relevant to learning in modern physics courses, especially the concept of photovoltaics. The results of the solar cell characterization are also related to the characteristics of diodes related to basic electronics courses (Montalvo-Galicia et al., 2022).

In this paper, this study reported on the resulting fabrication of FTO by the spray pyrolysis method for its application in the context of Physics learning. In order to get a characterization of FTO, Scanning Electron Microscopy (SEM), Energy Dispersive X-Ray (EDX), X-Ray Diffraction (XRD), Uv-Vis Spectroscopy (Uv-Vis) and Four Point Probe are reported as well. Apart from that, the application of FTO in DSSC devices is embedded in the learning process for students to obtain learning experiencefrom physics theory, followed by experimentation and application.

Artefacts produced during physics learning and documented and published through online media (youtube) so that it can be used as a learning media (Rahmatika et al., 2021). Adding to that, the experimental results become as actual examples during Modern Physics lectures to be discussed in activities. The activities and narratives revolves around the manufacturing process, examination of formed particles, and other physical properties. In the end, students are introduced to the FTO application in DSSC (Ikpesu et al., 2020). We hope that the manufacture of FTO glass and its application in DSSC solar cells can be used as one of the laboratory-based physics learning models both in high schools and universities.

Method

The methodology in this research was mixed methods (experiment and qualitative methods). In this study, an experiment was carried out to produce FTO glass and then the product was used as a physics learning medium. A combination of laboratory experimental approaches and qualitative research is carried out in research to achieve the research goal of creating laboratory-based learning (Montalvo-Galicia et al., 2022).

Experiment In Fabrication of FTO

Spray pyrolysis was the method of fabricating FTO glass. FTO itself comes from materials in the form of the chemicals Tin(II) Chloride Dihydrate ($SnCl_2.2H_2O$, Sigma Aldrich Germany) and Ammonium Fluoride (NH_4F, Sigma Aldrich Germany) were dissolved in Ethanol 96%. These materials were then depositioned on a glass substrate using the OMRON NE-C28 nebulizer as shown in Figure 1(a) (Darsikin et al., 2018). Particle formation on the substrate was depicted in Figure 1(b). Moreover, the process of characterizing the physical properties of FTO glass was carried out using X-Ray Diffraction spectroscopy (XRD, PANalytical X'Pert PRO), Energy-dispersive X-ray spectroscopy and Scan Electron Microscopy (SEM and EDS, FEI Inspect-S50), Uv-Vis Spectroscopy (Analytic Jena Specord 200 plus), and IV Characteristic (Keithley).

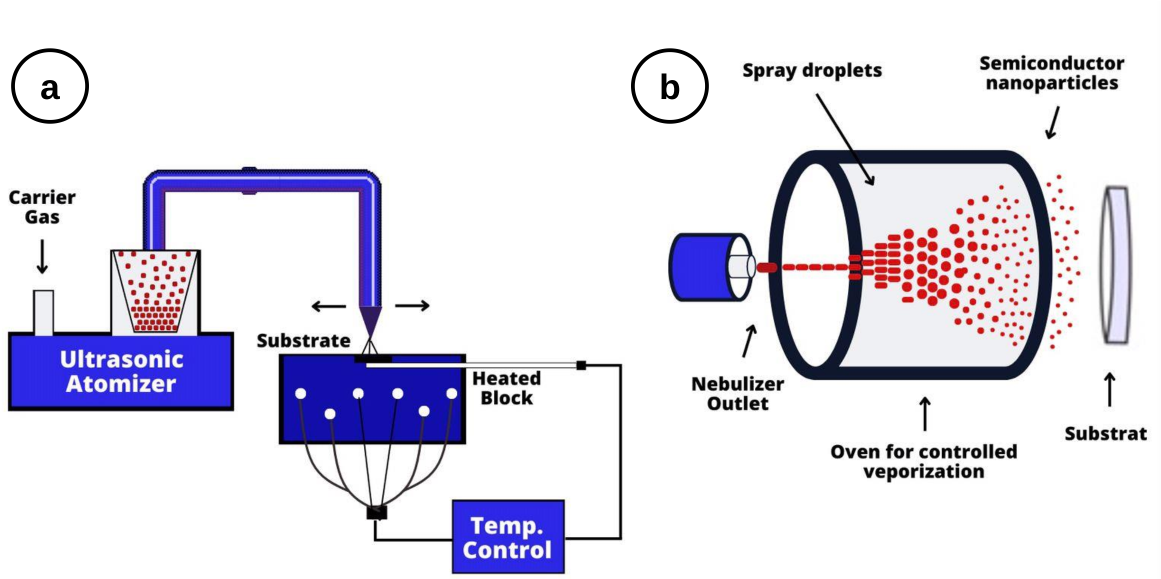

Figure 1. Scheme of Fabricating FTO layers: (a) Spray Pyrolysis Methods; (b) Inset deposition process on the substrate (Darsikin et al., 2018)

After the FTO was generated, it was applied as an electrode to dye-sensitized solar cells (DSSC). DSSC was made with a sandwich structure and measured the amount of current and voltage under the sun with a certain intensity using KEITHLEY equipment (Saehana et al., 2019). Then, the efficiency and fill factor are calculated so that the performance of the solar cells produced can be known. All of the experimental processes in the laboratory are documented in the form of a video and then uploaded to free service providers (*youtube*) so that they can be used for the offline and online learning process (Izzah et al., 2021).

Qualitative Approach

The FTO glass fabrication process was then conveyed to physics education study program students who are currently programming an MK in Modern Physics in the even semester of the 2022/2023 academic year. After participating in the lesson, students are then given a perception questionnaire about the fabrication, characterization, and application of the FTO. This questionnaire has been validated by experts and uses a Likert Scale to determine the level of student perception (Rizal et al., 2022).

To find out the physics aspects of the FTO fabrication process, interviews were conducted with three students who had passed the modern physics course at the Physics Education Study Program, FKIP Tadulako University. The interviews were conducted by asking five questions about students' perceptions of (1) the Physical aspects of the experiment of making Fluorine doped Tin Oxide glass; (2) The application of FTO glass in technology and (3) The relationship between the fabrication process and the content of modern physics courses. The results of the interviews are then used as a basis for compiling recommendations on the learning process so that learning is more interesting and displays physics content (Moll et al., 2017).

Results and Discussion

FTO Glass Fabrication with Spray Pyrolysis Method

Fluorine-doped Tin Oxide glass is a type n semiconductor that is transparent and conductive. The spray pyrolysis method carried out by researchers to produce FTO glass in this study can be seen at the following link: https://www.youtube.com/watch?v=e1gEl-aC478 (Palamba et al., 2021). The FTO glass produced through the sintering and ionization processes in this experiment can be seen in Figure 2. The resulting glass appears transparent so that objects, logos, and writing behind it can be seen.

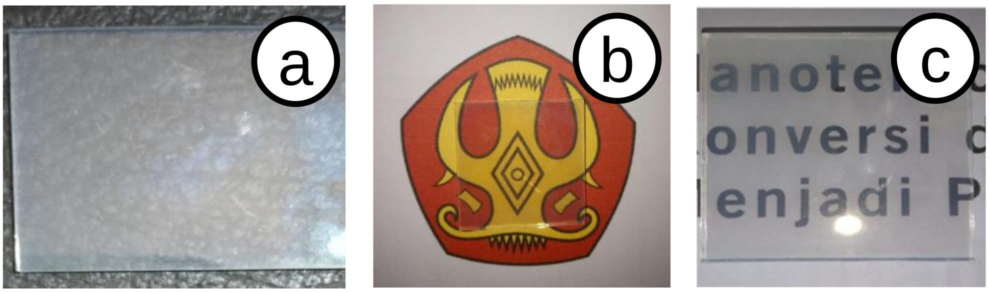

Figure 2. FTO glass was produced through this experiment. (a) Glass placed in front of an object; (b) Glass is placed in front of an image/logo; and (c) Glass is placed over a letter

In FTO glass fabrication a chemical reaction occurs, which is described as follows (Guermat et al., 2022):

a. Formation of the SnO_2 layer from the separation of $SnCl_2.2H_2O$ compounds:
   $SnCl_2 + 2H_2O \rightarrow SnO_2 + 2HCl$

b. The decomposition of NH_4F is as follows:
   $NH_4F \rightarrow NH_3\uparrow + NF\uparrow$

c. Evaporation of H_2O and HN_4F and oxidation
   $SnCl_2.2H_2O + NH_4F \rightarrow SnO_2{:}F$ (*Flourine Tin Oxide*)

The morphology and content of the FTO film are known through examination using SEM and EDX as shown in Figure 3 and Figure 4.

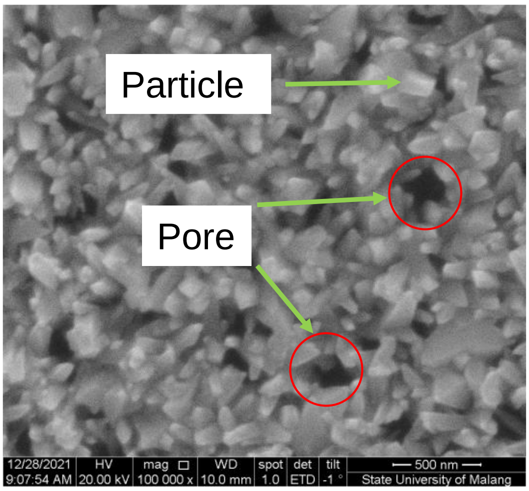

Figure 3. Characterization results by Scanning Electron Microscopy

Figure 3 shows the results of morphological characterization using SEM. Using a magnification of 100,000x can be seen the layers that have formed. A considerable number of pyramid-shaped $SnO_2{:}F$ particles can be observed. Likewise, the space between particles (pores) also indicates that the particle fabrication process has not been fully homogeneous (Zhao et al., 2016). This causes particle contact to be not maximal so the layer resistance is large (El-Yazeed et al., 2021).

By using Electron Dispersive Spectroscopy (EDS) equipment, the compiling elements of the FTO layer can be known and formed in Figure 4(a). According to several pieces of literature, their composition affects the conductivity of FTO glass (Lisnic et al., 2023) (Guermat et al., 2022).

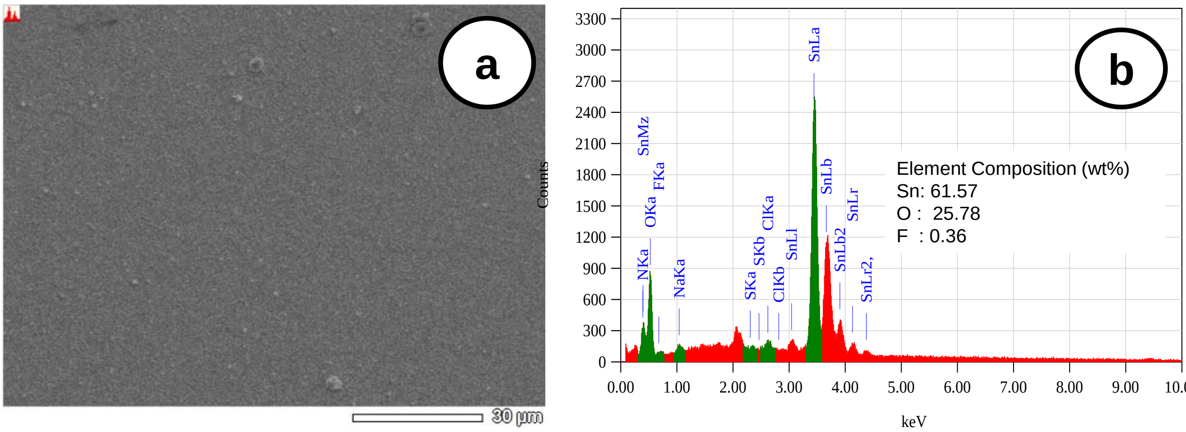

Figure 4. The results of characterization of the compiling elements of the film with Electron Dispersive Spectroscopy (EDS)

Figure 4(b) states that the resulting film contains a certain number of atoms with a certain presentation. The three dominant atoms are Sn, O, and F (Ikhmayies, 2017). The ratio between the composition of the compiling elements, especially F and Sn, is still relatively small (0.005) when compared to the ideal condition of 0.05. It means the composition must be increased tenfold to fit ideal conditions (Zheng et al., 2017).

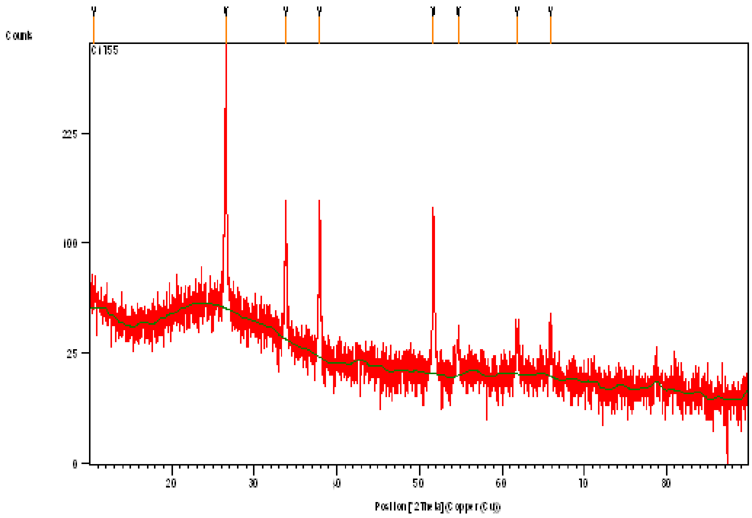

Figure 5. Crystal characterization results with XRD

Based on JPCDS data, it is known that the diffraction peak of fluorine-doped tin oxide is identified through a peak of 26.5◦; 38.0◦; 52.0◦; 55.0◦; 62.0◦ and 66.0◦ (Sharaf et al., 2021). Thus, through this experiment as shown in Figure 5, it is known that crystalline SnO_2:F particles have been produced (Van et al., 2021).

The formation of SnO_2: F can be seen from the peak formed from the results of XRD testing. The results of XRD testing can be used to determine the crystal size (crystallite size) with a certain phase by referring to the main peaks of the diffractogram pattern through the formulated Debye Scherrer equation (Kurapati &; Srivastava, 2018):

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (1)$$

Where
:
D = Crystal size
K = Form factor of the crystal (0.9-1)
λ = Wavelength of X-rays (1.54 Å = 0.154 nm)
β = Value of Full Width at Half Maximum (FWHM) (rad)
θ = Diffraction angle (degrees)
According to the Scherer equation, it is known that the crystal size of SnO_2:F is 0.85 nm.

One of the uniqueness of FTO glass is invisibility. The graph in Figure 6 shows that photons can be transmitted more than 70% through the FTO glass medium in the range of 370 to 1000 nm. This means that the experiments carried out have succeeded in making translucent glass although it is still lower commercialized. (Bandara et al., 2021). This is supported by visible observations as shown in Figure 6. The object behind the FTO glass can be observed.

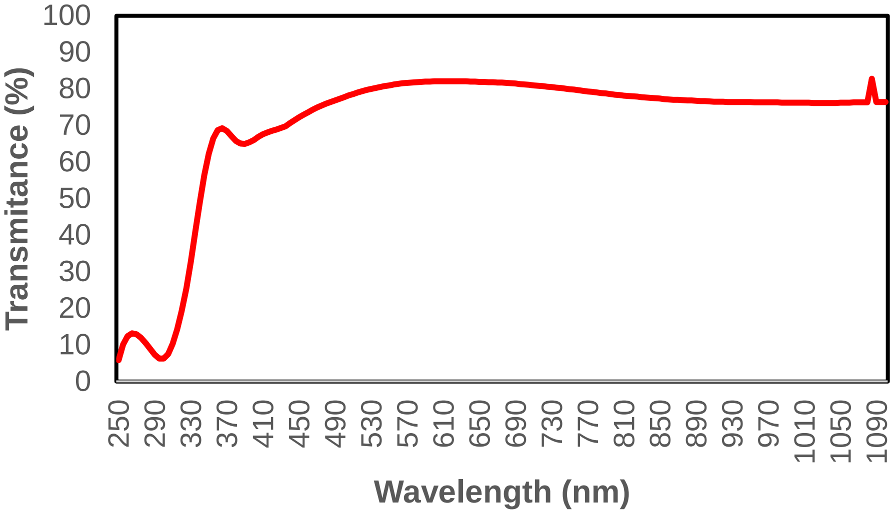

Figure 6. Results of layer transmittance measurement using Uv-Vis Spectroscopy

FTO resistance measurements were performed using four-point probe equipment (Hameed et al., 2022; Elsherif et al., 2016). Those were measured on nine samples, with concentration variations. Table 1 shows different resistance values obtained on four different sides and then averaged.

Table 1. Results of resistance measurement with the Four Point Probe method

	Sheet Resistance (ohm/cm^2)				
Samples	**Side 1**	**Side 2**	**Side 3**	**Side 4**	**Average**
A	10.000	10,000.0	10000.0	10,000.0	10,000.0
B	1371.0	1470.0	1470.0	1,215.0	1,381.5
C	860.0	320.0	270.0	860.0	577.5
D	142.0	98.0	107.0	170.0	129.3
E	62.0	48.0	45.0	67.0	55.5
F	34.0	23.0	21.0	26.0	26.0
G	27.0	24.0	24.0	40.0	28.8
H	39.0	30.0	30.0	25.0	31.0
I	15.0	13.0	14.0	16.0	14.5

Based on Table 1, sample A has the greatest resistance of 10,000.0 Ohms/cm^2. This is thought to be because the SnO_2:F layer has not formed properly. In terms of morphology, it is caused by the presence of many pores formed (the film is not yet homogeneous) so the contact between particles is not optimal (Mokaripoor et al., 2015). The smallest resistance was obtained in sample I with an average of 14.5 ohms/cm^2.

Application of FTO in Dye-Sensitized Solar Cells (DSSC)

The FTO produced through this study (resistance 55.5 Ω/cm^2), was then applied as an electrode to DSSC solar cells (Pinheiro et al., 2023). After TiO2 was depositioned by the spray method, it was then soaked in a dye solution. DSSC was made using a sandwich structure with platinum-coated counter electrodes. The process of making solar cells can be watched in the video at the following link: https://www.youtube.com/watch?v=DXiYNy6zZYo. Then, the characterization of solar cells was carried out at an intensity of 100 mW/cm2. The nature of sensitivity to the presence of light is shown by the solar cell device made. An efficiency of 2.01% is obtained from measurements using the Keithley IV meter as shown in Figure 7.

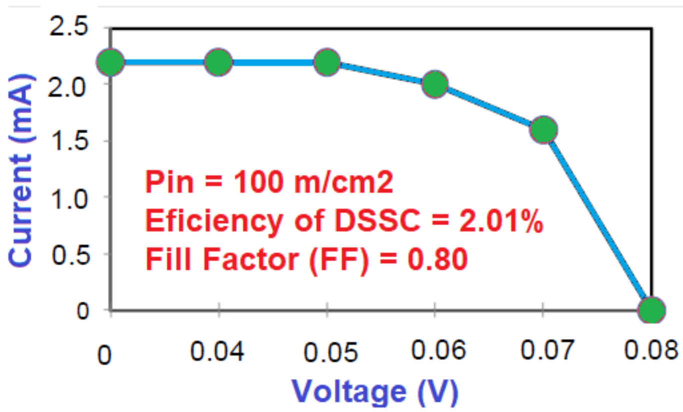

Figure 7. Characterization of DSSC solar cells, with the resulting FTO, in dark (a) and light (b) conditions

Based on the calculated fill factor value (0.80), the researchers suspect that in Figure 7, the resulting solar cells still have a large internal resistance so they have not produced efficiency below today's commercial DSSC solar cells (Arbab et al., 2020).

A few of the experimental activities carried out in making FTO glass and making DSSC solar cells in this study have been documented in video and have been uploaded online so that students can watch them (Palamba et al., 2021). This is expected to help support laboratory-based learning on some relevant physics materials such as Ohm's Law, X-ray Diffraction, Efficiency, and Electric Power.

Perception of Student to Fluorine doped Tin Oxide and Its Application

After following the learning related to the process of making fluorine-doped tin oxide glass, its characterization, and application, students are then given a perception questionnaire about FTO. This questionnaire contains a total of 10 questions with four possible answers (Likert scale-Strongly agree, agree, disagree, stringly diagree) as shown in Table 2.

Table 2. Student Perceptions of FTO

No	Question	Score
1	FTO is made by the spray pyrolysis method which involves physical and chemical processes	4.0
2	FTO is made up of microscopic particles that cannot be seen with the naked eye	4.0
3	FTO constituent particles can be seen using SEM	4.0
4	The composition of the FTO constituent particles can be determined using EDS	3.0
5	The size of the crystals that make up the FTO can be determined using XRD	4.0
6	The FTO resistance can be measured with an ohmmeter/four-point probe	4.0
7	FTO transparency can be determined by inspection using UV-Vis equipment	4.0
8	FTO can be used as a DSSC-type solar cell electrode	3.0
9	FTO can be applied to other technology products	4.0

No	Question	Score
10	Learning modern physics courses with this material makes learning interesting	4.0
	Average Score	3.8

Based on the analysis, it shows that the average means score is $\bar{x} = 3.8$ which gravitate to strongly agree. Thus, based on the questionnaire in Table 2 student perceptions about the manufacture, characterization, and application of FTO imply that learning with FTO material enable students to relate the activities with the technology application.

The process of making FTO using the spray pyrolysis method involves physical and chemical processes. The chemical processes are known through direct and video observations that chemical reactions occur through mixing existing solutions (Jo et al., 2020). On the other hand, the physical reaction occurs through the evaporation of the solution flowing through the nebulizer and the formation of a layer on the substrate which is placed on the hotplate at 450◦C (Kumar et al., 2018).

FTO has constituent particles that cannot be seen by the naked eye. This is believed by all students because based on the results of direct observation that FTO glass is physically indistinguishable from ordinary glass. Explanations by lecturers as well as information obtained through learning resources increase students' confidence that FTO has constituent particles in the microscopic order (Rajendran et al., 2023).

The constituent particles of the FTO can be seen using SEM and are highly believed by all students ($\bar{x} = 4.0$). The composition of the FTO constituents can be known through examination with EDX which is believed by students with an average score of 3.0. The size of the crystals that make up the FTO can be known by XRD and is also believed by students with a maximum average score ($\bar{x} = 4.0$). FTO resistance as one of the important FTO parameters can be measured using a multimeter in ohmeter mode. This is known by all students with a maximum score of 4.0. Likewise, FTO transparency can be known by UV-Vis examination by students. Thus, all students who were respondents to this study knew very well that FTO could be characterized in terms of morphology, structure, optics, and electricity using certain equipment (Ramírez-Amador et al., 2019).

The use of FTO in DSSC as a main or auxiliary electrode is known by all students with an average score of $\bar{x} = 3.0$. This can be known by students through the lecturer's explanation by showing the structure or through video shows. On the other hand, the application of FTO in various electronic components is believed by all students with a maximum score of $\bar{x} = 4.0$. All students who took part in learning activities related to FTO were very happy. This is because a variety of interesting information is presented not only through explanations by the teacher but also through the multimedia used. In this section, a maximum average score of 4.0 is obtained.

To obtain more detailed information regarding FTO, interviews were then conducted with three students regarding several related aspects, namely:

(1) Physics aspects in the experiment of making Fluorine doped Tin Oxide glass;

Respondent R-1 said that the FTO made using the spray pyrolysis method uses a physical-chemical approach. Where the $SnCl_2$ and NH_4Cl solutions were dissolved in alcohol and then sprayed with a nebulizer on the substrate glass to form SnO_2:F.

(2) Product FTO Glass Characterization

Respondent R-2 said that the particles that make FTO up can be determined using equipment such as SEM, EDS, XRD, Uv-Vis, and ohmmeter. The teacher's explanation by showing the results of these characterizations strengthens students' beliefs about this.

(3) Application of FTO glass in technology

Respondent R-3 said that FTO glass has some potential applications in the world of technology. One of its applications is as an electrode component in DSSC.

(4) The link between the fabrication process and the content of modern physics courses.

All respondents stated that the FTO fabrication process was related to the content of modern physics courses. Materials that are relevant to FTO fabrication are diffraction (Abuelwafa et al., 2021), particle size (Wang et al., 2017), transmittance (Abdel-Galil et al., 2021), and photovoltaic effect (Qiao et al., 2020).

Conclusion

In this study, experiments have demonstrated the manufacture of FTO using the spray pyrolysis method. SnO_2:F particles have been formed by which conductive and transparent layers are formed. The results of surface morphology characterization with SEM show that there are differences in FTO morphology with nozzle-to-collector distance, heating time, and temperature. FTO was also confirmed by characterization of the structure using XRD at diffraction peaks of 26.5◦; 38.0◦; 52.0◦; 55.0◦; 62.0◦ and 66.0◦. The best transmittance and resistance of FTO films are above 80% with a resistance of 55 ohms/cm^2. The application of FTO as an electrode in DSSC solar cells got an efficiency of about 0.87% and a fill factor of 0.30. Experimental activities in making FTO in this study are expected to help support laboratory-based learning on several relevant physics materials such as Ohm's Law, X-ray Diffraction, Efficiency, and Electrical Power. Furthermore, the process of making FTO and its application is taught in the Modern Physics course so that information is obtained about student perceptions of the manufacturing process, characterization, and application of this technology in DSSC solar cells. Students have a positive perception of learning the material for making FTO with the spray pyrolysis method. Some experimental proofs and applications can also be known by students.

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