



SYNTHESIS AND CHARACTERIZATION OF SnO_2/ZnO COMPOSITE USING JAPANESE PAPAYA LEAF EXTRACT (*Cnidioscolus aconitifolius*) WITH HYDROTHERMAL METHOD

Heni Puspita¹, Rika Agustin¹, Asdim[✉]¹, Eka Angasa¹, Evi Maryanti¹, Agus Martono Hadi Putranto¹

¹Program Studi Magister (S2) Kimia, FMIPA, Universitas Bengkulu, Kandang Limun, Muara Mangkahulu, Bengkulu, 38119, Indonesia.

DOI: 10.20414/spin.v6i2.10997

History Article

Accepted:

July 23, 2024

reviewed:

December 02, 2024

Published:

December 20, 2024

Keywords:

Composite

SnO_2/ZnO ; Extract;

Hydrothermal;

Japanese papaya leaf

(*Cnidioscolus*

aconitifolius).

ABSTRACT

*SnO_2/ZnO composites were synthesized using the hydrothermal method using Japanese papaya (*Cnidioscolus aconitifolius*) leaf extract. This study aims to determine the effect of using Japanese papaya leaf extract (*Cnidioscolus aconitifolius*) on the formation of crystallinity and morphology in synthesizing SnO_2/ZnO composites. Synthesis was carried out with variations in mass of 5, 10, and 15 grams using the hydrothermal method for 12 hours at 160°C . The results of X-Ray Diffraction (XRD) characterization show that wide diffractogram peaks are identified as the peaks of the SnO_2 compound with a tetragonal structure and sharp peaks are identified as the peaks of the ZnO compound. The Fourier Transform Infrared (FTIR) characterization shows the peak wave number of 665 cm^{-1} which is the Sn-O-Sn strain and the peaks at wave numbers 598 cm^{-1} and 501 cm^{-1} which are the Zn-O strain. Characterization of Scanning Electron Microscopy (SEM) in the synthesis of SnO_2/ZnO composites after adding Japanese papaya (*Cnidioscolus aconitifolius*) leaf extract had relatively reduced particle size and aggregate formation compared to no extract. The best effective mass of Japanese papaya leaf extract (*Cnidioscolus aconitifolius*) is the mass variation of 15 grams with 28.49 nm crystals.*

ABSTRAK

Sintesis komposit SnO_2/ZnO dilakukan dengan metode hidrotermal menggunakan ekstrak daun pepaya jepang (*Cnidioscolus aconitifolius*). Penelitian ini bertujuan untuk mengetahui pengaruh penggunaan ekstrak daun pepaya jepang (*Cnidioscolus aconitifolius*) terhadap pembentukan kristalinitas dan morfologi dalam sintesis komposit SnO_2/ZnO . Sintesis dilakukan dengan variasi massa 5, 10, dan 15 gram menggunakan metode hidrotermal selama 12 jam pada suhu 160°C . Hasil karakterisasi X-Ray Diffraction (XRD) menunjukkan adanya puncak difraktogram lebar yang teridentifikasi sebagai puncak senyawa SnO_2 dengan struktur tetragonal dan puncak tajam teridentifikasi sebagai puncak senyawa ZnO . Karakterisasi Fourier Transform Infrared (FTIR) menunjukkan puncak bilangan gelombang 665 cm^{-1} yang merupakan regangan Sn-O-Sn dan puncak pada bilangan gelombang 598 cm^{-1} dan 501 cm^{-1} yang merupakan regangan Zn-O. Karakterisasi Scanning Electron Microscopy (SEM) pada sintesis komposit SnO_2/ZnO setelah penambahan ekstrak daun pepaya jepang (*Cnidioscolus aconitifolius*) memiliki ukuran partikel dan pembentukan agregat yang relatif lebih kecil dibandingkan dengan tanpa ekstrak. Massa efektif terbaik ekstrak daun pepaya jepang (*Cnidioscolus aconitifolius*) adalah variasi massa 15 gram dengan kristal 28,49 nm.

How to Cite

Puspita, H., Agustin, R., Asdim.,, & Putranto, A. M. H. (2024). Synthesis and Characterization of SnO_2/ZnO Composite Using Japanese Papaya Leaf Extract (*Cnidioscolus Aconitifolius*) With Hydrothermal Method. *SPIN-Jurnal Kimia & Pendidikan Kimia*. 6(2). 202-213.

INTRODUCTION

Materials consisting of two or more materials that are still separate and macroscopically incompatible when forming one component are called composite materials (Kurniawan et al., 2013; Minah et al., 2017). Where the properties of each material that makes up the composite have different chemical and physical properties and produce composites with new properties (Oroh et al., 2013). The synthesis of SnO_2/ZnO composites is carried out using several synthesis methods. Some methods that have been reported are the solid state reaction method (Dony et al., 2013), the sol-gel method (Ghaderi et al., 2015), the solvothermal method (Viet et al., 2016; Prabakaran et al., 2019; Suthakaran et al., 2019), and the hydrothermal method (Zhu et al., 2015). The hydrothermal method has advantages over other methods, namely producing particles with high crystallinity, high purity and homogeneous particle distribution (Arrafiqie et al., 2016). The synthesis of SnO_2/ZnO composites using the hydrothermal method begins with the hydrolysis of metal salt precursors into metal hydroxides as the temperature increases in a closed system. Then when the system reaches a higher temperature, the hydroxide dehydrates to produce metal oxides (Ortiz-Landeros et al., 2012). Synthesis of SnO_2/ZnO composite materials using the hydrothermal method in addition to using synthetic chemicals, can also use natural materials. Sudhparimala and Vaishnavi (2016) have successfully synthesized SnO_2/ZnO composites using ethanol extract of aloe vera leaves producing spherical particles with a size of 66 nm. Honarmand et al. (2020) have successfully synthesized SnO_2/ZnO composites using Teucrium

polium extract as a structure directing agent (SDA) that can control the shape and size of particles influenced by phenolic compounds contained in Teucrium polium extract. Jiao et al. (2016) have also successfully synthesized SnO_2/ZnO aggregates using cellulose aerogel with an aggregate shape measuring 45.4 nm. It has also been reported that the synthesis of SnO_2/ZnO composites using Acroptilon repens flower extract as a reducing and stabilizing agent successfully formed non-spherical SnO_2/ZnO nanocomposites with particle sizes of 5-40 nm (Golmohammadi et al., 2021). This study shows that the use of different materials produces SnO_2/ZnO composites with varying shapes and sizes. Therefore, intensive exploration of natural materials in the synthesis of SnO_2/ZnO continues to be carried out. In this study, it is proposed to use Japanese papaya leaf extract (*Cnidocolus aconitifolius*) in the synthesis of SnO_2/ZnO composites. To the best of our knowledge, the use of Japanese papaya leaf extract (*Cnidocolus aconitifolius*) in the synthesis of SnO_2/ZnO composites has never been reported. Japanese papaya leaf extract (*Cnidocolus aconitifolius*) contains secondary metabolite compounds such as alkaloids, phenols, glycosides, saponins, steroids, phlobatannin, flavonoids, and tannins (Somade et al., 2021). The saponin content in Japanese papaya leaf extract (*Cnidocolus aconitifolius*) is around 7.84% (Okpara and Akwukwaegbu, 2020). The use of Japanese papaya leaf extract (*Cnidocolus aconitifolius*) in the synthesis of nanoparticles has been reported, one of which is in the synthesis of silver nanoparticles (AgNPS) using AgNO_3 precursor (Fabiyyi, 2021). The results of SEM analysis showed that the morphology

of the obtained AgNPS was spherical with nanoparticle sizes ranging from 2-20 nm. Based on the background above, this study focuses on the synthesis of SnO₂/ZnO composites by the hydrothermal method using Japanese papaya leaf extract (*Cnidoscopus aconitifolius*) as a capping agent or structure-directing agent to control the shape and size of the particles. The characteristics of the obtained composites will be studied in the form of crystallinity and morphology using X-ray Diffractometer (XRD), Fourier Transform Infrared (FTIR), and Scanning Electron Microscope (SEM) instruments.

METHOD

Tools

The tools used in this study were Fourier Transform Infrared (FTIR) (Alpha Platinum-ATR), X-ray Diffractometer (XRD) (PAN Analytical Philip), Scanning Electron Microscopy (SEM) (Hitachi 3800), oven (Philip Harris Ltd), analytical balance (Sartorius), hotplate magnetic stirrer, hydrothermal reactor, spatula, stirring rod, scissors, spray bottle, suction ball and glassware such as Erlenmeyer, beaker, measuring pipette, dropper pipette, measuring flask, watch glass, funnel and petri dish.

Materials

The materials used in this study were SnCl₄ 98% (Sigma Aldrich), Zn(CH₃COO)₂·2H₂O (Sigma Aldrich), ethanol (tennis), aqua DM, AgNO₃, NaOH, plain filter paper, tissue, and Japanese papaya leaf extract (*Cnidoscopus aconitifolius*) with varying sample masses of 5, 10, and 15 grams.

Preparation of Japanese Papaya Leaf Extract (*Cnidoscopus aconitifolius*) with Mass Variations of 5, 10, and 15 grams

The preparation of Japanese papaya leaf extract (*Cnidoscopus aconitifolius*) with mass variations of 5, 10, and 15 grams was prepared following the following procedure, namely Japanese papaya leaves (*Cnidoscopus aconitifolius*) were washed until clean and finally rinsed with DM aqua to remove impurities that were attached. After being clean, the leaves were cut into small pieces and dried (± 5 days) at room temperature. After drying, they were weighed with different mass variations, namely 5, 10, and 15 grams using a watch glass. The weighed Japanese papaya leaf sample (*Cnidoscopus aconitifolius*) was put into a 500 mL beaker and 100 mL of DM aqua was added and then heated using a hotplate while stirring with a magnetic stirrer at a speed of 650 rpm for 1 hour at a temperature of 60°C. After that, it was cooled to room temperature. After cooling, it was filtered using ordinary filter paper and obtained Japanese papaya leaf extract (*Cnidoscopus aconitifolius*) (Fabiyyi, 2021).

Synthesis of SnO₂/ZnO Composite Material Without Extract Using Hydrothermal Method

Synthesis of SnO₂/ZnO composite material without natural material extract was modified from research (Angasa et al., 2020). A total of 20 mL of 0.1 M SnCl₄ and 20 mL of 0.2 M Zn(CH₃COO)₂·2H₂O were mixed for 20 minutes at room temperature while stirring using a magnetic stirrer. Next, 40 mL of NaOH was slowly added to the solution by stirring using a magnetic stirrer for 10 minutes. After that, the mixture was transferred into a Teflon lined autoclave and then heated at 160°C for 12 hours (Jiao et al., 2016). After the reaction was complete, the reactor was cooled to room temperature. After cooling, the mixture was filtered with Whatman filter paper no. 1 and the residue was washed three times with DM aqua, then

the filtrate was tested with 0.01 M AgNO_3 , then the residue was washed again with ethanol. The precipitate obtained was dried using a hotplate at a temperature of 70°C for 60 minutes after which it was dried again at a temperature of 60°C for 12 hours using an oven (Yu et al., 2019). The samples obtained were then characterized using an X-ray diffractometer (XRD), Fourier Transform Infrared (FTIR), and Scanning Electron Microscopy (SEM). The same procedure was carried out for all variations of the extract and without using the extract.

Synthesis of SnO_2/ZnO Composite Material with Japanese Papaya Leaf Extract (*Cnidoscopus aconitifolius*) Using the Hydrothermal Method

The synthesis of SnO_2/ZnO composite material with natural material extracts was modified from the research conducted (Angasa et al., 2020). A total of 20 mL of 0.05M SnCl_4 and 20 mL of 0.1 M $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ were mixed for 10 minutes at room temperature while stirring using a magnetic stirrer. Next, 10 mL of Japanese papaya leaf extract (*Cnidoscopus aconitifolius*) was added slowly into the solution by stirring using a magnetic stirrer for 10 minutes. Next, 40 mL of NaOH was added slowly into the solution by stirring using a magnetic stirrer. After that, the mixture was transferred into a 100 ml Teflon-lined autoclave and then heated at 160°C for 12 hours (Jiao et al., 2016). After the reaction was complete, the reactor was cooled to room temperature. After cooling, the mixture was filtered with Whatman filter paper no. 1 and the residue was washed three times with DM aqua, then the filtrate was tested with 0.01 M AgNO_3 , then the residue was re-washed with ethanol. The obtained precipitate was dried using a hotplate at 70°C for 60 minutes and then dried again at 60°C for 12 hours using an

oven (Yu et al., 2019). The samples obtained were then characterized using an X-ray diffractometer (XRD), Fourier Transform Infrared (FTIR), and Scanning Electron Microscopy (SEM). The same procedure was carried out for all extract variations and without using the extract as a control.

Characterization of SnO_2/ZnO Composite Material with Japanese Papaya Leaf Extract (*Cnidoscopus aconitifolius*)

Characterization was carried out using an X-ray diffractometer (XRD) to analyze the crystal phase and average crystal size of the formed SnO_2/ZnO composite. The morphology of the samples was analyzed using Scanning Electron Microscopy (SEM) and identification of functional groups contained in the SnO_2/ZnO composite using a Fourier Transform Infrared Spectrophotometer (FTIR).

RESULT AND DISCUSSION

Preparation of Japanese Papaya Leaf Extract (*Cnidoscopus aconitifolius*) with Mass Variations of 5, 10, and 15 grams

Extraction was carried out one by one for each variation of the different Japanese papaya leaf mass (*Cnidoscopus aconitifolius*) after being air-dried, namely 5 grams, 10 grams, and 15 grams. In the extraction process, the solvent used was aqua DM, where aqua DM is an environmentally friendly solvent and is also free from minerals so that it does not interfere with the extraction process. The extracted sample was heated with the help of a hotplate at a temperature of 60°C for 60 minutes. After that, it was cooled at room temperature and filtered using filter paper so that Japanese papaya leaf extract (*Cnidoscopus aconitifolius*) would be obtained.

Synthesis of SnO₂/ZnO Composite by Hydrothermal Method

SnCl₄ precursor was used as the reactant for the formation of SnO₂ and Zn(CH₃COO)₂.2H₂O precursor as the reactant for the formation of ZnO. The addition of extract to the SnCl₄ and Zn(CH₃COO)₂.2H₂O solution caused the mixture which was initially white to become blackish brown. With the addition of NaOH, the blackish-brown mixture faded to a brownish-yellow color.

The synthesis of the composite was carried out in a Teflon-lined autoclave which functions as a place for the dissolution and growth of SnO₂/ZnO

crystals. Teflon-lined autoclave is a thick-walled cylindrical container that is resistant to high pressure and temperature to change the crystal structure (Byrappa and Yoshimura, 1992). The heating temperature used was 160°C for 12 hours so that the synthesis process of SnO₂ and ZnO composites was maximized (Jiao et al., 2016).

Adapted from Hemmati et al. (2011) illustration of the mechanism in the formation of SnO₂/ZnO in general through the reaction of SnCl₄, Zn(CH₃COO)₂.2H₂O and NaOH after the hydrothermal process can be written as follows:

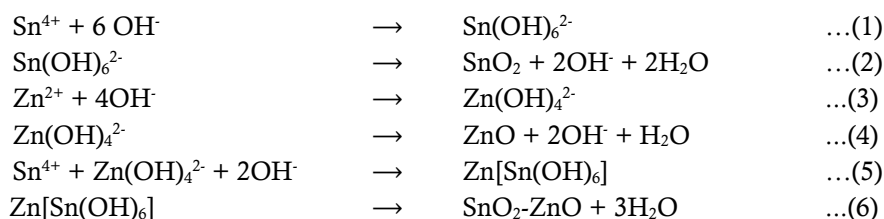
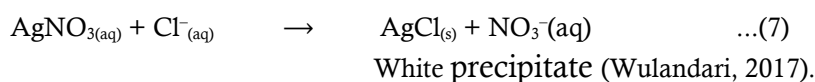


Illustration of the sample formation mechanism is shown in the early stages of precipitation, when SnCl₄ which is the precursor reacted with solvents to form tin ions Sn⁴⁺ and Zn(CH₃COO)₂.2H₂O which are precursors reacted with solvents to form Zn²⁺. When NaOH is slowly added to the solution, a white precipitate will form (Wang et al., 2007).

When Sn⁴⁺ and Zn²⁺ react with OH⁻, they will produce a complex compound Sn(OH)₆²⁻ in reaction (1) and a precipitate Zn(OH)₄²⁻ in reaction (3) which is marked by the formation of a white precipitate in the solution. Sn(OH)₆²⁻ will decompose into SnO₂ in reaction (2) and Zn(OH)₄²⁻ decomposes into ZnO in reaction (4) as the temperature and pressure increase during the hydrothermal reaction. Mixing SnCl₄,

Zn(CH₃COO)₂.2H₂O and NaOH at room temperature also produces Zn[Sn(OH)₆] with a cubic structure in reaction (5). then Zn[Sn(OH)₆] decomposes and recrystallization occurs to form SnO₂/ZnO during the hydrothermal process in reaction (6).

The resulting precipitate is washed using DM aqua 10-15 times depending on the volume of DM aqua added and the filtrate is tested with AgNO₃ solution to see if the filtrate is free from Cl⁻ ions. In washing, the more DM aqua added, the faster the filtrate is free from Cl⁻ ions. When the filtrate is reacted with AgNO₃ solution, a positive result is indicated by the presence of a white precipitate. The reactions that occur between AgNO₃ solution and Cl⁻ ions are as follows:



After the precipitate is free from Cl^- ions, a final rinse is carried out using ethanol. The rinsing process using ethanol aims to remove unwanted organic impurities in order to obtain a SnO_2/ZnO composite with good purity. After that, the precipitate obtained was dried on a hotplate at a temperature of 70°C for 60 minutes to remove the water and ethanol content in it. After that, it was dried again at a temperature of 60°C for 12 hours using an oven (Yu et al., 2019).

Characterization of SnO_2/ZnO Composites

Characterization Using an X-ray Diffractometer (XRD)

Qualitative analysis techniques based on X-ray diffraction patterns on samples, and characterization using an X-ray diffractometer (XRD) seek to determine the phase, crystallinity, and crystal size of the synthesized SnO_2/ZnO composite. The diffractogram of the SnO_2/ZnO synthesis results is shown in Figure 1. From Figure 1, it can be seen that there are two broad diffractogram peaks at the 2θ (theta) peak of 26.19° and 51.59° for all samples

synthesized without and using extracts. These two broad peaks are identified as the peaks of the tetragonal structured SnO_2 compound with miller indices (110) and (211). This is in accordance with the standard diffractogram of the Inorganic Crystal Structure Database (ICSD) No. 00-001-0625. The miller indices (110) and (211) are the orientation of the crystal plane of SnO_2 which will have different shapes and directions according to the index of each SnO_2 crystal plane. From Figure 1 it can also be seen that there are sharp peaks for samples synthesized without and using extracts at peak 2θ (theta) 31.83° , 34.49° , 36.35° , 47.64° , 56.71° , 63.01° , 68.12° , 69.27° . These sharp peaks are identified as the peaks of ZnO compounds with a hexagonal structure with miller indices of the crystal planes (100), (002), (101), (102), (110), (103), (112), and (201). This is in accordance with the standard diffractogram of ICSD No.01-075-0576. These results indicate that the SnO_2/ZnO composite has been successfully synthesized without and using Japanese papaya leaf extract (*Cnidocolus aconitifolius*).

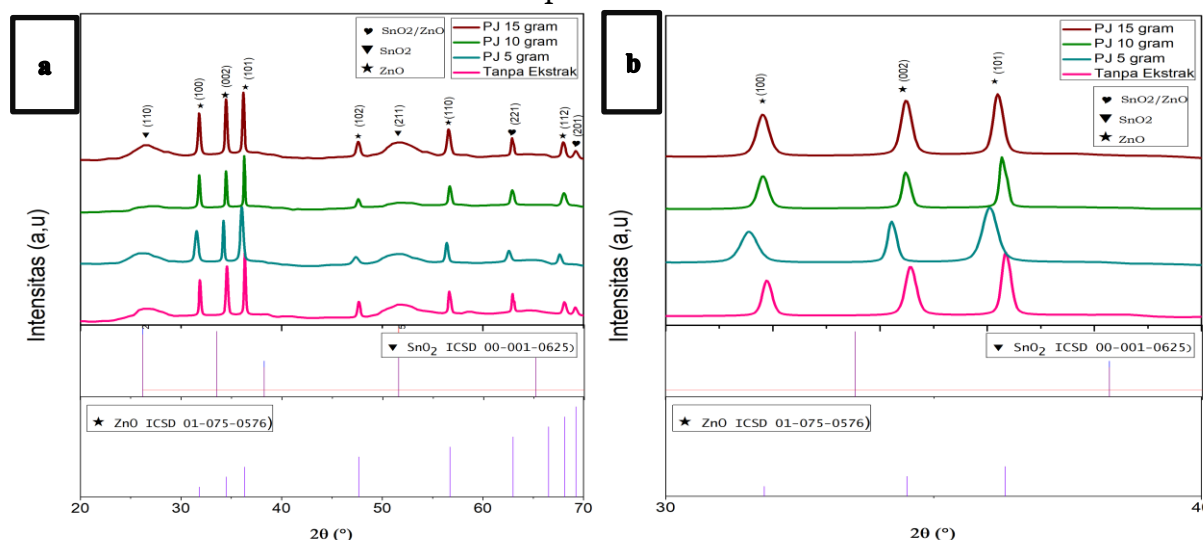


Figure 1. (a) Diffractogram of XRD analysis results on samples synthesized without and with the use of extract (b) Enlarged diffractogram of analysis results.

Based on the diffraction pattern of Figure 1 for all samples there are no other

peaks, this indicates that the SnO_2/ZnO composite obtained has good purity. From

Figure 1 it can also be seen that the ZnO peak has a narrow and sharp gap with high intensity indicating good crystallinity. However, for SnO₂, the peak is wide with low intensity indicating that SnO₂ has low crystallinity. Materials with high crystallinity have sharp and clear diffraction peaks (Mihaiu et al., 2015). To determine the crystal size of the synthesized product, data processing was carried out obtained

from the results of the XRD analysis, namely by using the Debye-Scherrer equation (Sanjaya et al., 2017). The calculation of the crystal size of the synthesized product without extract and with the addition of Japanese papaya leaf extract (*Cnidoscopus aconitifolius*) can be seen with the Debye-Scherrer equation, the average crystal size of the SnO₂/ZnO composite is obtained as shown in Table 1.

Table 1. Average crystal size of SnO₂/ZnO composite with the addition of Japanese papaya leaf extract (*Cnidoscopus aconitifolius*) and without using the extract.

No	Japanese papaya leaf extract (<i>Cnidoscopus aconitifolius</i>) with mass variation.	Average crystal size (D)
1	Without Extract	39,82 nm
2	5 grams	30,81 nm
3	10 grams	44,96 nm
4	15 grams	28,49 nm

Based on the data in Table 1, the addition of Japanese papaya leaf extract (*Cnidoscopus aconitifolius*) as a natural capping agent can affect crystal growth. This can be seen from the varying crystal sizes with the addition of Japanese papaya leaf extract (*Cnidoscopus aconitifolius*), namely 30.81; 44.96; and 28.49 nm for mass variations of 5, 10, 15 grams, respectively. The crystal size for the 5 gram and 15 gram extract variations is smaller than the crystal size without the addition of extract, which is 39.82 nm. This is an indication of the influence of secondary metabolite compounds contained in Japanese papaya leaf extract (*Cnidoscopus aconitifolius*). Where the -OH group contained in the secondary metabolite compound can protect the surface of the SnO₂/ZnO composite and suppress its crystal growth (Amanta, 2022). The greater the capping agent added, the more functional groups are available to synthesize the SnO₂/ZnO composite, thus reducing the number of particles (Singh et al., 2018). However, the use of capping agent for Japanese papaya leaf extract

(*Cnidoscopus aconitifolius*) is not in accordance with the literature. This is most likely due to the effect of additional metabolites found in Japanese papaya leaf concentrate (*Cnidoscopus aconitifolius*) so that it can work with particle development. The most effective extract mass to produce the smallest crystal size is 15 grams.

Characterization Using Fourier Transform Infrared Spectroscopy (FTIR)

Characterization using the Fourier Transform Infrared (FTIR) instrument aims to identify functional groups in the synthesized material. The FTIR spectrum will read the presence or absence of the SnO₂/ZnO composite material produced and ensure the presence or absence of other organic compounds from Japanese papaya leaf extract (*Cnidoscopus aconitifolius*) left in the sample. The SnO₂/ZnO material powder was characterized in the wave number range of 500 cm⁻¹ to 4000 cm⁻¹. The FTIR spectrum of the SnO₂/ZnO composite synthesis results can be seen in Figure 2.

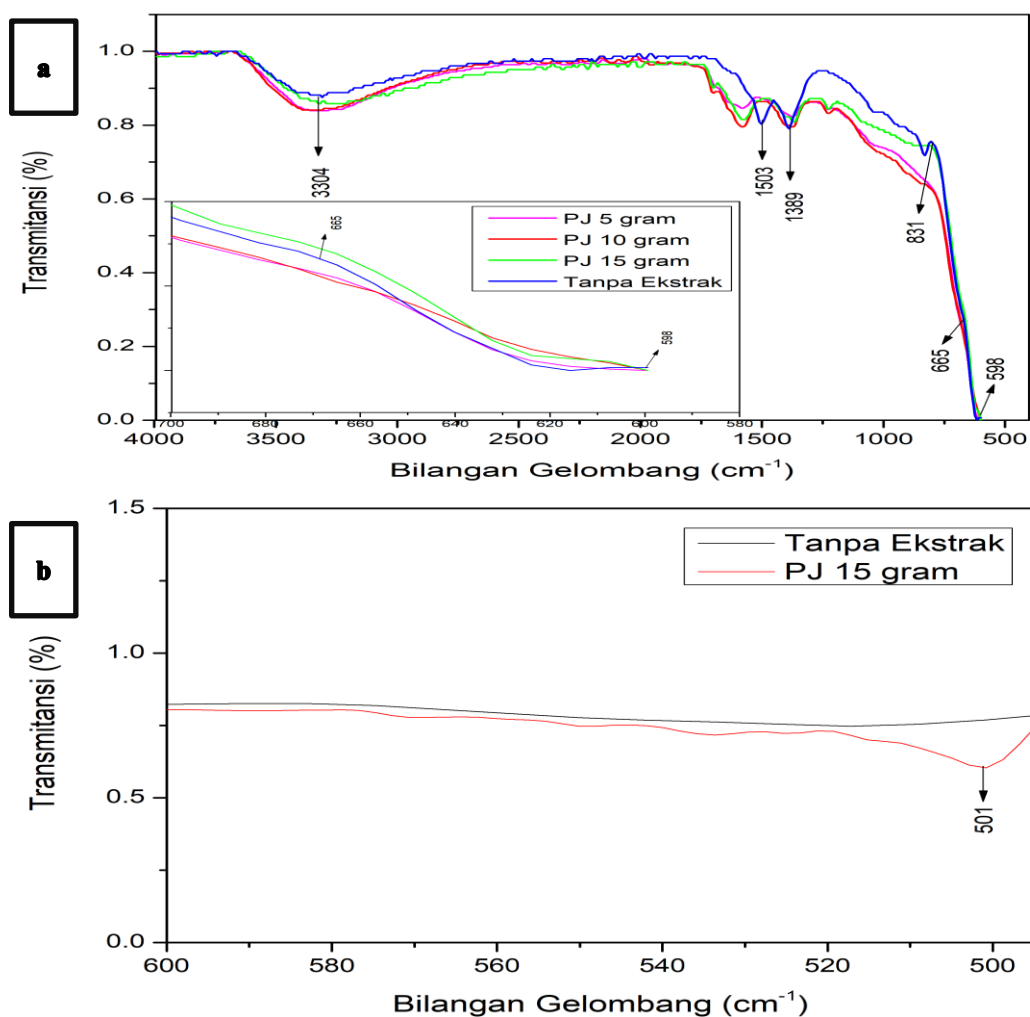


Figure 2. FTIR spectrum of the synthesis of SnO₂/ZnO Composite synthesized at a temperature of 160°C for 12 hours (a) comparison of the wave spectrum of 4000-500 cm⁻¹ (b) comparison of the FTIR spectrum of the wave number 600-490 cm⁻¹

Based on the FTIR spectrum in Figure 2, the spectrum of the sample without extract and using variations of Japanese papaya leaf extract (*Cnidioscolus aconitifolius*) shows almost the same spectrum, with no significant difference between the two. The resulting FTIR spectrum shows that there is a spectrum indicating the SnO₂/ZnO compound which is marked by the presence of a peak at wave number 665 cm⁻¹ which is the Sn-O-Sn stretch and peaks at wave numbers 598 cm⁻¹ and 501 cm⁻¹ which are the Zn-O stretch. The peak spectrum that appears at other waves such as the 3304 cm⁻¹ wave spectrum is the O-H group of water. The water content that is still present in the powder is likely water due to the presence of water

vapor during sample storage. The peak at wave number 1503 cm⁻¹ comes from the aromatic C=C group, the peak at the 1389 cm⁻¹ spectrum wave comes from the C-H group and the peak at wave 831 cm⁻¹ comes from the alkane C-H group. Other organic spectra that appear such as O-H, C=C, and C-H are caused by the sample washing process that has not been maximized.

Characterization Using Scanning Electron Microscope (SEM)

Characterization using the Scanning Electron Microscope (SEM) instrument is one of the analyses that aims to see the morphology of the sample such as the shape and size of the particles that have been synthesized. The SEM results for the

SnO₂/ZnO composite synthesized with Japanese papaya leaf extract (*Cnidoscolus aconitifolius*) and without using the extract to

see the effect of the extract on the growth of the SnO₂/ZnO composite aggregate are shown in Figure 3.

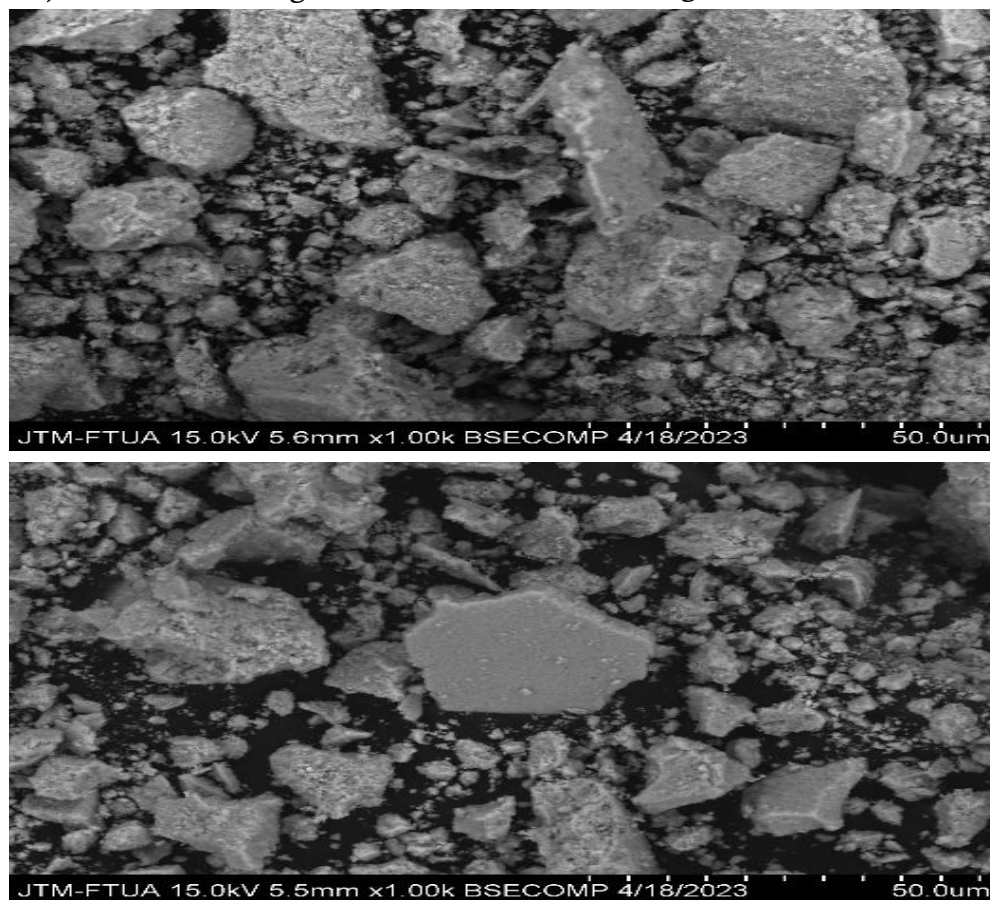


Figure 3. SEM characterization results of SnO₂/ZnO composites (a) without Japanese papaya leaf extract (*Cnidoscolus aconitifolius*) (b) using Japanese papaya leaf extract (*Cnidoscolus aconitifolius*) with a mass variation of 15 grams with a magnification of 1,000 times.

From Figure 3, it can be seen that the SnO₂/ZnO composite synthesized without using Japanese papaya leaf extract (*Cnidoscolus aconitifolius*) shows the formation of agglomeration, resulting in particles with relatively large sizes. While the addition of Japanese papaya leaf extract (*Cnidoscolus aconitifolius*) still causes agglomeration, but the lumps are less than without the extract. This shows that Japanese papaya leaf extract has the ability of a capping agent in the synthesis of SnO₂/ZnO composites, but it is not optimal.

CONCLUSION

Japanese papaya leaf extract (*Cnidoscolus aconitifolius*) can be used in the synthesis of SnO₂/ZnO composites with

different characterizations. Characterization using XRD shows that the synthesized SnO₂/ZnO composites have good crystallization characterized by sharp peaks and high intensity. Characterization using FTIR of the SnO₂/ZnO composite shows that SnO₂ has Sn-O-Sn stretching in the spectrum of wave numbers 665 cm⁻¹ and Zn-O group stretching is found at waves 598 cm⁻¹ and 501 cm⁻¹. The results of SEM analysis show that the morphology of the resulting SnO₂/ZnO is irregular. The mass of Japanese papaya leaf extract (*Cnidoscolus aconitifolius*) in producing the smallest crystal size of the SnO₂/ZnO composite (28.49 nm) is the use of extract with a mass of 15 grams.

ACKNOWLEDGEMENTS

The author would like to thank the Chemistry Study Program, FMIPA, University of Bengkulu for all the facilities provided and to all parties who helped during this research.

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