



SYNTHESIS AND CHARACTERIZATION OF SnO_2 USING JAPANESE PAPAYA LEAF EXTRACT (*Cnidoscolus aconitifolius*) AS CAPPING AGENT BY HYDROTHERMAL METHOD

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ABSTRACT

*Synthesis of SnO_2 has been carried out by using extract of *Cnidoscolus aconitifolius* leaf as a natural capping agent. The synthesis aims to determine the effect of the use of a capping agent on the crystallinity and the size of crystals. The mass variations of the capping agent used were 5, 10, and 15 gram using the hydrothermal method at relatively low temperature (95-100°C). The synthesized of SnO_2 were characterized using FTIR (Fourier Transform Infra Red Spectroscopy) and XRD (X-Ray Difraction). The results of FTIR characterization show the stretching vibration absorption band of Sn-O-Sn in the spectrum with and without capping agent at wave number 599 cm^{-1} and 599.333 cm^{-1} . XRD diffractogram shows that SnO_2 without a capping agent and using a capping agent has relatively the same crystallinity. Mass variations of 5, 10, and 15 gram resulted in crystals measuring 14.37 nm, 13.75 nm, and 11.78 nm and SnO_2 without capping agent measuring 15.93 nm. The results showed that the *Cnidoscolus aconitifolius* leaf solution could be used as a natural capping agent to produce SnO_2 . The results of SEM characterization show that aggregate reduction can be seen in the addition of Japanese papaya (*Cnidoscolus aconitifolius*) capping agent.*

ABSTRAK

Sintesis SnO_2 telah dilakukan dengan memanfaatkan ekstrak daun tanaman pepaya jepang (*Cnidoscolus aconitifolius*) sebagai *capping agent* alami. Sintesis ini bertujuan untuk mengetahui pengaruh penggunaan *capping agent* terhadap kristalinitas dan ukuran kristal yang dihasilkan. Variasi massa *capping agent* yang digunakan yaitu 5, 10, dan 15 gram dengan menggunakan metode hidrotermal pada suhu yang relatif rendah (95-100°C). Hasil sintesis SnO_2 dikarakterisasi menggunakan Spektrofotometer FTIR (Fourier Transform Infra Red Spectroscopy) dan XRD (X-Ray Difraction). Hasil karakterisasi FTIR menunjukkan pita serapan getaran peregangan Sn-O-Sn pada spektrum tanpa *capping agent* terjadi di bilangan gelombang 599 cm^{-1} dan pada bilangan gelombang 599,333 cm^{-1} untuk SnO_2 dengan *capping agent*. Difraktogram XRD menunjukkan SnO_2 yang dihasilkan tanpa *capping agent* dan menggunakan *capping agent* memiliki kristalinitas yang relatif sama. Variasi massa *capping agent* 5, 10, dan 15 gram menghasilkan kristal berukuran 14,37 nm, 13,75 nm, dan 11,78 nm sedangkan SnO_2 tanpa *capping agent* berukuran 15,93 nm. Hasil ekstrak daun tanaman pepaya jepang (*Cnidoscolus aconitifolius*) dapat digunakan sebagai *capping agent* alami untuk menghasilkan material SnO_2 . Hasil dari karakterisasi SEM menunjukkan adanya reduksi agregat dapat dilihat pada penambahan *capping agent* pepaya jepang (*Cnidoscolus aconitifolius*).

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INTRODUCTION

Nanotechnology is a science that studies and creates materials on the nanometer scale (Poole & Owens, 2003). Nanotechnology continues to be developed by researchers from the academic and industrial worlds. One area that is of interest to researchers is the development of nanoparticle synthesis methods. Nanometer-sized materials or commonly called nanoparticles have a number of chemical and physical properties that are superior to large-sized materials. One example of nanoparticles is SnO_2 which is an oxide semiconductor material. Making nanoparticles really depends on the preparation method in the form of nanospheres or nanocapsules.

Several methods that can be used for SnO_2 synthesis are solvothermal, sol-gel and hydrothermal (Viet *et al.*, 2016). Hydrothermal synthesis can be defined as a method of synthesis from single crystals that depends on dissolved substances from minerals in hot water under high pressure. In this method, a hydrolysis reaction occurs, namely when the temperature increases, hydrolysis of the metal salt precursor will produce metal hydroxide. The morphology of the SnO_2 produced can be influenced by several parameters such as time, surfactant, temperature, solvent, reactant concentration and capping agent. Capping agent functions as an inhibitor and control agent in aggregate growth in SnO_2 synthesis.

Synthesis of SnO_2 using SnCl_4 precursor and *triethylenediamine* (TEDA: $\text{C}_6\text{H}_{12}\text{N}_2$) as a capping agent using the hydrothermal method produces tetragonal SnO_2 (Kim *et al.*, 2016). In the synthesis of SnO_2 using the hydrothermal method using the precursor $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$, Urea ($\text{CH}_4\text{NH}_2\text{O}$) and Sodium Hydroxide

(NaOH) produces a tetragonal morphology.

Apart from using synthetic chemicals as capping agents, researchers also use natural materials as capping agents in the synthesis of SnO_2 . Bhosale *et al.*, (2018) have succeeded in synthesizing SnO_2 using *Calotropis gigantea* leaf extract as a capping agent with XRD pattern analysis of the resulting material showing a tetragonal structure with a size of 35 nm. It has also been reported that the synthesis of SnO_2 using *Parkia speciosa Hassk* extract as a capping agent, succeeded in synthesizing SnO_2 with the resulting nanoparticle size below 2.7 nm (Begum & Ahmaruzzaman, 2018). Green synthesis SnO_2 uses the capping agent *citrus sinensis* leaf extract and $\text{SnCl}_2 \cdot \text{H}_2\text{O}$ as a precursor to produce SnO_2 measuring 1632 cm^{-1} . The synthesis of SnO_2 using the capping agent *Cassia alata* leaf extract with the precursor $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ has been reported and it was found that the size of the resulting nanoparticles ranged from 25-50 nm. The use of *Carica papaya* leaf extract as a capping agent for SnO_2 synthesis has also been reported by Jadhav & Kokate.,, (2020) with the size of the resulting SnO_2 ranging between 7.10 nm.

In contrast to the leaf extract of the papaya plant (*Carica papaya*), the leaf extract of the Japanese papaya plant (*Cnidoscolus aconitifolius*) has never been reported as a capping agent in the synthesis of SnO_2 . The Japanese papaya plant (*Cnidoscolus aconitifolius*) or commonly known as Chaya in its country of origin is a plant that originates from Central America. The term "papaya" is based on the shape and texture of the leaves which are similar to papaya leaves, although the morphology of the plant as a whole is more like a cassava

plant. The Japanese papaya plant (*Cnidoscolus aconitifolius*) has been proven to be a widespread plant with increasing popularity. Japanese papaya (*Cnidoscolus aconitifolius*) leaf extract contains many secondary metabolite compounds such as alkaloids, saponins, tannins, phenols and flavonoids. The saponin content in Japanese papaya (*Cnidoscolus aconitifolius*) leaf extract is around 7.84%. The use of Japanese papaya (*Cnidoscolus aconitifolius*) leaf extract as a capping agent in nanoparticle synthesis has been successfully carried out by Fabiyi (2021) in the synthesis of silver nanoparticles (AgNPs) which were synthesized using AgNO_3 precursor with surface plasmon resonance absorption at 410, 423 and 428 nm. SEM analysis confirmed the formation of spherical morphological AgNPs with nanoparticle sizes ranging from 2-20 nm.

This research focuses on the synthesis of SnO_2 using the hydrothermal method in an alkaline environment with Japanese papaya (*Cnidoscolus aconitifolius*) leaf extract as a natural capping agent and its characterization using FTIR and XRD instruments.

METHODS

Tools and materials

The tools used in this research were Fourier Transform Infrared (FTIR) Spectrophotometer (*Alpha Platinum-ATR*), X-ray Diffractometer (XRD) (*PAN Analytical Philip*), Scanning Electron Microscopy (SEM) (*Hitachi S-3400*), oven (*Philip Harris Ltd*), analytical scales (*Sartorius*), hotplate magnetic stirrers, Teflon-lined autoclaves, spatulas, stir sticks, scissors, spray bottles, suction balls and glassware such as Erlenmeyer, beakers, measuring pipettes, dropper pipettes, volumetric flasks, glass watch, funnel and petri dish.

The materials used in this research were SnCl_4 98% (*Sigma Aldrich*), NaOH (*Merck KgaA*), ethanol (*Merck*), aqua DM (*brataco*), ordinary filter paper, tissue, AgNO_3 (*Merck KgaA*), aluminum foil and Japanese papaya (*Cnidoscolus aconitifolius*) leaf extract with varying sample masses, sequencely 5, 10 and 15 grams.

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

Japanese papaya (*Cnidoscolus aconitifolius*) leaf extract is prepared by washing Japanese papaya (*Cnidoscolus aconitifolius*) leaves thoroughly with aqua DM to remove bound dust particles and chopping until smooth, then drying at room temperature ($\pm 28^\circ\text{C}$) for three weeks. Then weighed with different mass variations, sequencely 5, 10, and 15 grams with a watch glass. The prepared Japanese papaya (*Cnidoscolus aconitifolius*) leaf sample was placed in a 250 mL beaker and 100 mL of aqua DM was added and then heated with a hot plate while stirring with a magnetic stirrer at a speed of 650 rpm for 60 minutes at a temperature of 60°C then cooled. After reaching room temperature, the boiled water is poured and filtered using ordinary filter paper. Obtained Japanese papaya leaf extract (*Cnidoscolus aconitifolius*) (Fabiyi, 2021).

Synthesis of SnO_2 with Japanese Papaya Leaf Extract (*Cnidoscolus aconitifolius*) Using the Hydrothermal Method

Synthesis of SnO_2 with natural capping agents is based on previous research conducted by Guan *et al.* (2013). A total of 25 mL of 0.4 M SnCl_4 was put into a 250 mL beaker, added with 25 mL of Japanese papaya (*Cnidoscolus aconitifolius*) leaf extract with varying sample masses, namely 10, 15 and 20 grams, then stirred

with a magnetic stirrer at a speed of 500 rpm for 30 minutes with the last 10 minutes 50 mL of 0.4 M NaOH being poured slowly after the mixture is finished, put into a Teflon lined autoclave. Then heated at 95-100°C in the oven for 3 hours (Utami, 2020). The mixture was cooled to room temperature, then filtered using filter paper and the residue was washed 5-8 times using aqua DM, the filtrate was tested with a 0.01 M AgNO_3 solution. The residue was washed again once using ethanol. The solid obtained was dried using a hot plate at a temperature of 70°C. The powder obtained was then characterized using FTIR, XRD and SEM.

Characterization of SnO_2 with Japanese Papaya (*Cnidoscolus aconitifolius*) Leaf Extract

Characterization was carried out using an X-ray Diffractometer (XRD) to analyze the crystal phase and average crystal size of the SnO_2 formed, identification of the SnO_2 produced using a Fourier Transform Infrared Spectrophotometer (FTIR) and identification of the morphology of the SnO_2 produced using Scanning Electron Microscopy (SEM).

RESULT AND DISCUSSION

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

The Japanese papaya (*Cnidoscolus aconitifolius*) leaves used to make the extract were obtained in the Bengkulu City area. The Japanese papaya leaves obtained are washed using running water until clean and the final wash uses Aqua DM, which aims to remove dirt or dust stuck to the leaves. After that, the leaves are dried by not separating the leaves from the petioles so that the leaves do not rot. Drying is done not under direct sunlight but is air-dried at

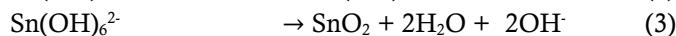
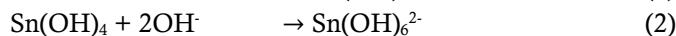
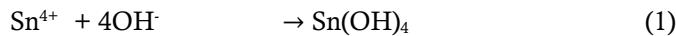
room temperature. The solvent used in the extraction process is aqua DM, because aqua DM is a solvent that is free from minerals and is environmentally friendly so it does not interfere during the extraction process. The sample was heated using a hot plate at 60°C for 60 minutes. Cooling is done at room temperature and filtered to obtain Japanese papaya leaf extract. The resulting Japanese papaya leaf water extract filtrate is bright brownish red to faded brownish red, which indicates that the Japanese Papaya leaf extract has been extracted. There are color differences for each mass variation. This is because the greater the sample mass used, the more concentrated the extract obtained will be. The number of secondary metabolites obtained increases due to the higher variation in sample mass. The resulting water extract from Japanese papaya leaves (*Cnidoscolus aconitifolius*) is then used as a capping agent in the synthesis of SnO_2 .

Synthesis of SnO_2 using the Hydrothermal Method

SnO_2 synthesis was carried out by making a mixture of solutions from SnCl_4 , NaOH and Japanese papaya leaf extract. A total of 25 mL of 0.4 M SnCl_4 was reacted with 25 mL of Japanese papaya (*Cnidoscolus aconitifolius*) leaf extract with sample mass variations of 5, 10 and 15 grams into a 250 mL beaker. The mixture turned light brown and was then stirred using a magnetic stirrer for 30 minutes. Stirring is done so that the solution is completely mixed and forms a homogeneous solution. In the last 10 minutes of stirring time, NaOH solution was added drop by drop to obtain an even dispersion of material size. The more NaOH solution added, the thicker a saturated solution (colloid) will be formed. Where alkaline conditions can increase the ability of various functional groups present

in natural capping agents, which play a role in the synthesis of nanoparticles so as to increase their stability. Meanwhile, acidic conditions will reduce the ability of functional groups in material synthesis.

When Japanese papaya leaf extract is added to SnCl_4 , the extract will reduce SnCl_4 to Sn^{4+} metal ions. The function of adding NaOH is as a precipitating agent in the solution. Then, the mixture is put into a



The formation of SnO_2 occurs in 2 stages, namely hydrolysis and nucleation. In the initial stage, when the precursor is dissolved in the solvent, Sn^{4+} ions are formed. When NaOH is added slowly to the solution, a white precipitate of $\text{Sn}(\text{OH})_4$ will form. The white precipitate of $\text{Sn}(\text{OH})_4$ will eventually dissolve when it reacts with abundant hydroxide ions (OH^-) to form a transparent solution of $\text{Sn}(\text{OH})_6^{2-}$. In the next stage, the concentration of the $\text{Sn}(\text{OH})_6^{2-}$ complex increases with increasing heating time, until it reaches a critical concentration where SnO_2 nuclei form spontaneously through a



White precipitate

After that, the precipitate obtained was dried using a hot plate at a temperature of 70°C to remove the remaining water and ethanol content. Once dry, the solids is crushed to obtain a powder. The SnO_2 powder is then crushed into smaller particles.

Teflon lined autoclave which functions as a place for the reaction and growth of SnO_2 crystals. Heating was carried out at a temperature of $95\text{-}100^\circ\text{C}$ for 3 hours.

The reaction mechanism that occurs during the SnO_2 formation process generally uses the hydrothermal method with SnCl_4 and NaOH precursors (Inderan *et al.*, 2015):

condensation reaction (Inderan *et al.*, 2015).

The resulting precipitate was washed using aqua DM 5-8 times depending on the volume of aqua DM added and the washing was stopped when the filtrate was tested with AgNO_3 solution to see whether the filtrate was free from Cl^- ions. To speed up washing, the more aqua DM you add, the faster the filtrate will be free of Cl^- ions. When the filtrate is reacted with the AgNO_3 solution, a positive result is indicated by the presence of a white precipitate. The reaction that occurs between the AgNO_3 solution and Cl^- ions is reaction (4) as follows (Wulandari, 2017):

Characterization of SnO_2

Characterization Using X-ray Diffractometer (XRD)

Characterization using XRD is a qualitative analysis instrument which aims to see the size and crystallinity of a material that has been synthesized based on the X-ray diffraction pattern in the sample. Figure 1 displays the diffractogram formed based on test variations.

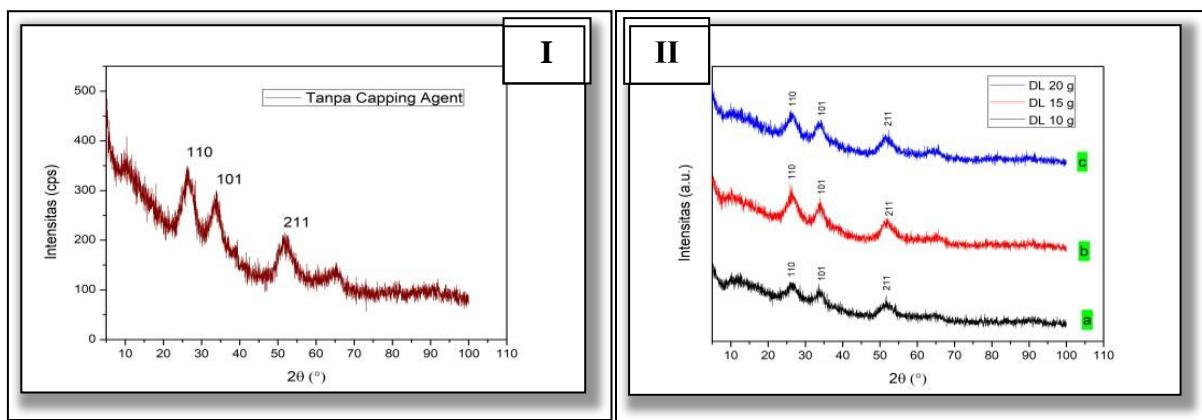


Figure 1. Diffractogram (I) diffractogram without capping agent of Japanese papaya (*Cnidoscolus aconitifolius*) leaf extract which was synthesized at a temperature of 95-100 °C for 3 hours and dried at a temperature of 70 °C (II) Diffractogram of the synthesis result with variations in the mass of added Japanese papaya (*Cnidoscolus aconitifolius*) leaf extract capping agent (a) 5 grams (b) 10 grams (c) 15 grams.

The synthesis of the SnO_2 material uses the same molar ratio of SnCl_4 and NaOH as the method of Guan *et al.* (2013), namely using concentration in a ratio of 1:2. Where the peaks of the diffractogram on the crystallinity of SnO_2 are influenced by the concentration of NaOH . The higher concentration makes the crystallinity in SnO_2 have a high intensity and narrow diffraction angle peaks. However, as the NaOH concentration increases, the solution becomes saturated, small crystal nuclei form and begin to grow (Guan *et al.*, 2013).

Based on the diffractogram in Figure 2 (a) showing XRD without capping agent of lamtoro leaf extract, several sharp peaks can be seen and identified as a tetragonal structure with 3 peaks appearing with the diffraction angle values of SnO_2 being 26.3029, 33.7044 and 51.2956 with a crystal lattice (110) , (101) and (211). This shows that the diffraction angle value is close to the diffraction angle value in the research of Viet *et al.* (2016) that the results of XRD analysis gave rise to 4 SnO_2 peaks at 26.6, 33.8, 51.8 and 65.9 with crystal lattices (110), (101), (211) and (301). This can be stated in accordance with the *Joint*

Committee on Powder Diffraction Standard (JCPDS No. 41-1445).

Based on all the XRD data, it shows that SnO_2 without the addition of a capping agent and with the addition of a capping agent from Japanese papaya leaf extract has relatively almost the same crystallinity, that is, it has a crystallinity that is not too high. The XRD SnO_2 synthesis results obtained look the same in the diffractogram of research by Junbo *et al.* (2005), namely the XRD diffractogram of $\text{Sn}(\text{OH})_4$. It is estimated that the influence of the temperature used in this study is relatively low, namely 95-100°C, on the synthesis process, so that the diffractogram resulting from the synthesis has less sharp and wider peaks. The high crystallinity and sharp peaks in the XRD diffractogram results occur, this is thought to be because the temperature in the drying process has not reached a temperature high enough to fulfill the optimal crystal growth process (Mustari *et al.*, 2019). Based on the literature from all XRD data, there is a similarity in peaks between the SnO_2 synthesis results and the diffractogram of $\text{Sn}(\text{OH})_4$ synthesis results, indicating the possibility that the SnO_2 synthesis results are mixed with $\text{Sn}(\text{OH})_4$.

material. Due to the similarities between the peaks in the XRD diffractograms of SnO_2 and $\text{Sn}(\text{OH})_4$, it is possible that there is material overlap between the two. This shows that the results of the SnO_2 synthesis in this study have low purity because other materials, namely $\text{Sn}(\text{OH})_4$, can be read.

XRD analysis can determine the size of SnO_2 crystals by processing data obtained from XRD results, namely using the Debye-Scherrer equation.

Table 1. Average size of SnO_2 crystals with the addition of capping agent Japanese papaya leaf extract (*Cnidoscolus aconitifolius*).

No.	Mass variations of Japanese papaya leaf extract (<i>Cnidoscolus aconitifolius</i>)	Average crystal size (D)
1.	0 grams	15.93 nm
2.	10 grams	14.37 nm
3.	15 grams	13.75 nm
4.	20 grams	11.78 nm

Based on Table 1, there are differences in the average crystal size of each SnO_2 produced. The calculation data obtained shows that the SnO_2 produced is on a nanometer (nm) scale. The average size of the crystals synthesized without the addition of capping agent and with the addition of various masses of Japanese papaya (*Cnidoscolus aconitifolius*) leaf extract. Based on the data in the table, the addition of Japanese papaya leaf extract (*Cnidoscolus aconitifolius*) as a natural capping agent can influence crystal growth. As is the function of the capping agent itself. This is indicated by the decreasing crystal size when adding Japanese papaya (*Cnidoscolus aconitifolius*) leaf extract, namely 14.37; 13.75, and 11.78 nm with mass variations of 5, 10, and 15 grams when compared with the crystal size without the addition of capping agent, namely 15.93 nm. This is an indication of the influence of secondary metabolite compounds contained in Japanese papaya (*Cnidoscolus aconitifolius*) leaf extract. Where the -OH group contained in secondary

$$D = \frac{K \lambda}{\beta \cos \theta} \quad (5)$$

Where, D is the average crystal size (nm); λ is the X-ray wavelength (nm); β is the FWHM (Full Width Half Maximum) value and K is the Scherr Constant (0.9) (Davar *et al.*, 2010).

In this equation, the average crystal size (D) of SnO_2 is obtained which can be seen in Table 1.

metabolite compounds can protect the surface of SnO_2 nanoparticles and suppress crystal growth (Khairunnisa, 2015).

The average crystal size that works most effectively with the addition of a capping agent is obtained by adding a capping agent from Japanese papaya leaf extract (*Cnidoscolus aconitifolius*) with a mass of 15g, namely 11.78 nm because the capping agent functions very well in controlling particle size. With variations in the mass of the capping agent, Japanese papaya leaf extract (*Cnidoscolus aconitifolius*) showed different average crystal size results, where the resulting data showed that the higher the concentration of the capping agent used, the smaller the crystal size.

Characterization Using Fourier Transform Infrared (FTIR)

Characterization using the FTIR instrument was carried out in the wave number range of 500 cm^{-1} to 4000 cm^{-1} . The FTIR spectrum of the SnO_2 synthesis can be seen in Figure 2.

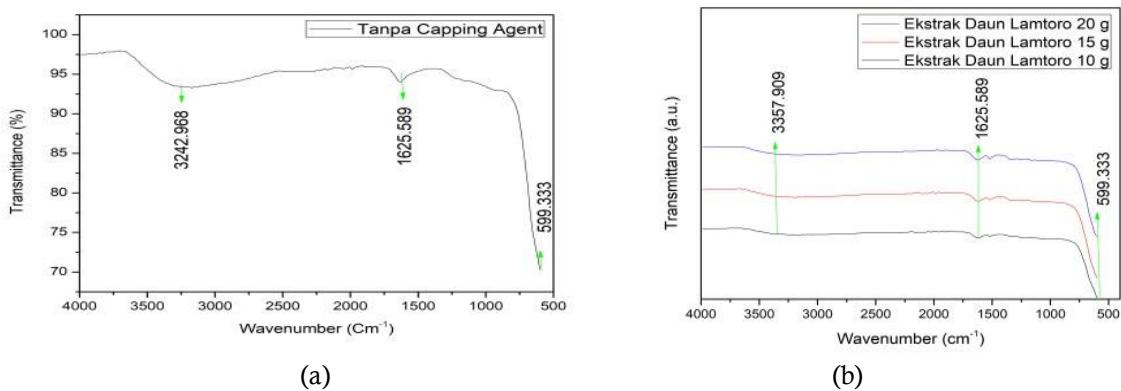


Figure 2. FTIR spectra of SnO_2 synthesis results: (a) without capping agent (b) with variations in the mass of added capping agent, Japanese papaya (*Cnidoscolus aconitifolius*) leaf extract 5, 10, and 15 grams

Based on the FTIR spectra in Figure 2, variations without and with the addition of Japanese papaya leaf capping agent show that the spectrum is almost the same, there is no significant difference. The spectrum without using a capping agent and that using a capping agent does not show a significant difference. Where a strong absorption band is seen at a wave number of 599 cm^{-1} for SnO_2 without capping agent and at a wave number of $599,333 \text{ cm}^{-1}$ for SnO_2 with Japanese papaya leaf (*Cnidoscolus aconitifolius*) capping agent. This spectrum and spectra show the stretching vibration spectrum of Sn-O-Sn. This is in accordance with the literature, where the SnO_2 absorption spectrum is at wave numbers $400-800 \text{ cm}^{-1}$ (Honarmand *et al.*, 2019).

There is a wide spectrum absorption band without a capping agent, which is an O-H stretching vibration at numbers $3,300 \text{ cm}^{-1}$ and 1625 cm^{-1} and at wave numbers $3,308,649 \text{ cm}^{-1}$ and $1,642,009 \text{ cm}^{-1}$ for SnO_2 with capping agent. These peaks are caused

by stretching vibrations ($3000-3600 \text{ cm}^{-1}$) and vibrations (1600 cm^{-1}) of hydroxyl groups (OH) which can come from water molecules on the SnO_2 surface. This spectrum appears due to the presence of absorbed water which is possible from water vapor when storing SnO_2 powder (Tammina *et al.*, 2018; Honarmand *et al.*, 2019).

Characterization Using Scanning Electron Microscopy (SEM)

Based on Figure 3, it can be seen that the results of aggregate reduction can be seen from the addition of Japanese papaya (*Cnidoscolus aconitifolius*) capping agent. This can be seen from Figure 3 (a). SnO_2 without a capping agent particles formed tend to be larger in size than in Figure (b). Aggregate formation can be reduced due to hydrogen bonds between the hydroxyl groups on the SnO_2 surface and the hydroxyl and carbonyl groups on the capping agent (Masjedi & Salavati, 2016).

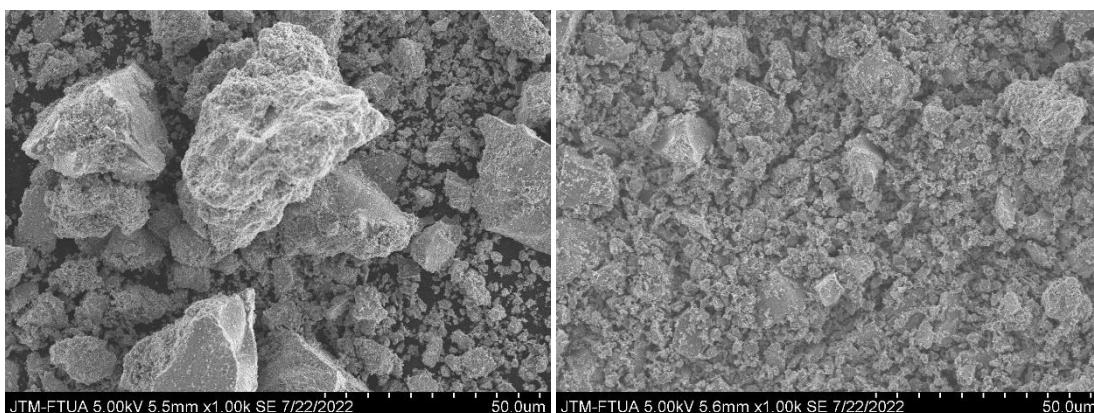


Figure 3. SEM images (a) SnO_2 synthesized without using a capping agent (b) SnO_2 synthesized using a capping agent from Japanese papaya leaf extract (*Cnidoscolus aconitifolius*) with 1000x magnification.

CONCLUSION

Synthesis of SnO_2 material has been carried out to produce low purity using the hydrothermal method with the addition of capping agent from Japanese papaya leaf extract (*Cnidoscolus aconitifolius*) at varying masses of 5, 10 and 15 grams and without capping agent from Japanese papaya leaf extract (*Cnidoscolus aconitifolius*). The best mass of Japanese papaya leaf extract in synthesizing SnO_2 is the use of a capping agent with a mass variation of 15 grams which works effectively by producing the smallest crystal size, namely 11.78 nm. Characterization using FTIR and the results of SEM characterization show that aggregate reduction can be seen in the addition of Japanese papaya (*Cnidoscolus aconitifolius*) capping agent.

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