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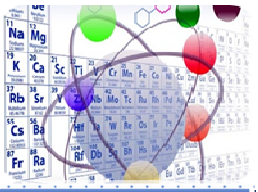

Formulation dan Phyical Stability Test Of Turi Leaf Extract *(Sesbania grandiflora L.)* Silver Nanoparticle Soap

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ABSTRACT

One metal that is widely researched is silver (Ag) which can be used as an antibacterial and antifungal. The method that is widely developed is the biological method, also called biosynthesis. Biosynthesis is a synthesis method using media from plant extracts. This research was conducted to determine the best formulation of Ag nanoparticle soap from turi (sesbania grandiflora) leaf extract based on its stability test. To obtain turi leaf extraction using the infusion method, a flavonoid compound test was carried out to determine the flavonoid compound content. After knowing the existence of flavonoid compounds, the wavelength was determined using UV-Vis Spectrophotometry. In the nanoparticle soap formula, a nanoparticle soap preparation was made which was then tested for the physical quality of Ag nanoparticle soap from turi leaf extract (Sesbania Grandiflora L). The gel-shaped preparation has a characteristic oleum rosae odor and a yellow to blackish soap color. Test the homogeneity to see if there are coarse grains on the surface of the soap. The specific gravity test results of all soap formulations meet the criteria for good soap. The high foam value in soap preparations is between 6.1 – 12.5 mM. The results of measuring the absorption values for concentrations of 1 mM, 2 mM, and 3 mM show that the optimum absorption results in liquid soap preparations are a concentration of 3 mM with a wavelength of 300 nm with an absorbance value of 1.105. The research results showed that turi leaf extract (sesbania grandiflora L) had an effect on the physical stability of Ag nanoparticle soap and met the standards set by SNI. Among formulation 1 with a concentration of 0.03, formulation II with a concentration of 0.06 and formulation III with a concentration of 0.09, the turi leaf extract soap preparation showed the best physical properties in formulation 1 with a concentration of 0.03 in the pH stability test.

Keywords*: Physical Stability, Turi Leaf Extract (sesbania grandiflora L.), Ag nano particle soap*

1. INTRODUCTION

Nanotechnology has become an important and interesting field of physics, chemistry, biology and engineering in recent years. One of the developments in nanotechnology that is being developed is nanoparticles. Nanoparticles are

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particles with a nanometer size, namely around 1-100 nm. Based on research by Wahyudi et al., (2018) nanoparticles are developing rapidly because they can be widely applied such as in the environmental, electronic, optical and biomedical fields.[1] The nanoparticles that attract a lot of attention are metal nanoparticles because of their wide application, including in the fields of optics, electronics, biology, catalysts and medicine. One of the most researched metals is silver (Ag). The most widespread application of silver nanoparticles is as an antibacterial and antifungal agent.

Many nanoparticle syntheses have been developed using biological methods, better known as the biosynthesis method. Biosynthesis is a method of synthesizing nanoparticles using media from biological materials, either microorganisms or extracts from plants. The use of environmentally friendly materials provides environmental safety benefits and is suitable for biomedical and pharmaceutical applications.

According to research by Rahmi, A. (2023), the use of natural ingredients has the potential to be developed into pharmaceutical dosage forms, along with the development of technology in the chemical and pharmaceutical fields, many types of preparations have been developed towards natural products.[2] Turi leaf extract as a natural base ingredient is used as a preparation. silver nanoparticle soap because it contains flavonoids which can kill microorganisms. And based on the results of research conducted by Wilda, *et al.,* (2017) it is also stated that turi leaves contain a higher flavonoid content.[3] Therefore, with the flavonoid content in them, turi leaves can be used to make liquid soap. The silver nanoparticle soap that is made needs to be tested for its physical stability, in order to guarantee the quality and quality so that it is safe when used.

Liquid soap has several advantages over solid soap, which is based on consumer opinion that liquid soap is more hygienic, liquid soap products are more profitable, practical and economical for consumers and soap production is easier and profitable for producers. The quality requirements for liquid soap based on the Indonesian National Standard (SNI) No. 06-4085-1996 are a pH ranging between 8-11. Physical stability test of preparations to ensure that the preparations made still meet the parameter criteria during storage. In research, Sativareza *et al.,* (2021) stated that the physical instability of liquid soap preparations is characterized by separation (coalescence formation) and other physical changes. To obtain the stability value of a pharmaceutical preparation in a short time, the stability can be accelerated, one of which is the cycling test.[4]

So the aim of the research carried out was to find out whether there was an effect of AgNO3 on the physical stability of turi leaf extract (sesbania grandiflora L.) silver nanoparticle soap and in what formulation the silver nanoparticle soap showed the best physical properties.

2. EXPERIMENTAL

2.1. Chemicals, Equipment and Instrumentation

The tools used in this research are digital scales, measuring cups, glass beakers, evaporating dishes, stirring rods, funnels, dropper pipettes, microscopes, spirit stoves, asbestos gauze, pycnometers, viscometers, mortars and stampers, horn spoons, pH paper, flannel cloth, stopwatch, filler, test tube and UV-Vis spectrophotometer.

The materials used in this research were turi leaves (*sesbania grandiflora L*) which were extracted using infusion, AgNo3 (silver nitrate), SLS (Sodium lauryl sulfate), Na2S04 (Sodium sulfate), STTP, Citric acid, Essential oil (Oleum rosae), Foam booster, distilled water, 95% ethanol, 2N HCl and concentrated HCl.

2.2. Research Procedure

2.2.1. Making infusion

The extraction process is carried out using the infusion method. Start by weighing 10 grams of simplicia powder of turi leaves (sesbania grandiflora L). Put it in a beaker glass and add 100 ml of distilled water. Place it on a Bunsen fire using

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a pan filled with water for 1 hour after the solution reaches a temperature of 80 degrees Celsius, while stirring. Wait until the temperature drops then filter the boiled water using a sterile flannel cloth into an Erlenmeyer flannel

2.2.2. Test the content of flavonoid compounds

A total of 2 ml of turi leaf infusion extract (sesbania grandiflora L) was put into a test tube. Add 1 ml of 95% ethanol and 2 ml of 2N HCL, then add 10 drops of concentrated HCL. If there is a change in color to red, yellow, orange, purple or blue, it can indicate that the extract contains flavonoid compounds.

2.2.3. *Synthesis and characteristics of Silver Nanoparticles*

Referring to research (Syahruddin Kasim *et al.,* 2020), 1 ml of extract was mixed into 15 ml of AgNo3 solution with varying concentrations of 1 Mm, 2 mM, 3 mM. then synthesized using a magnetic stirrer for 1 hour until the color change occurs in the solution. If the color of the solution changes to brownish yellow, it can be said that silver nanoparticles are formed

2.2.4. *UV-Vis Spectrophotometric Measurements*

Turn on the UV-vis spectrophotometry instrument, let it sit for 30-60 minutes. Prepare a blank distilled water solution and sample solution with varying concentrations of 1 mM, 2 mM, 3 mM. Enter into each cuvette according to the boundary marks. Perform a set scan wavelength of 200-600 nm, with an interval value of 5. Record the absorbance value at each wavelength setting.

2.2.5. *Nanoparticle Soap Formulation*

Table 1. Modified Formula for Silver Nanoparticle Soap Preparations

Material Name	**Formulation (%)**			**Utility**
	I	II	III	
Nanopartikel Ag & Ekstrak daun turi	1nm	2nm	3nm	Active substance
SLS (Sodium lauryl sulfate)	9	9	9	Foaming surfactant
Na_2SO_4 (Natrium sulfate)	6	6	6	Speed up mixing
STTP (Sodium tripolyphosphate)	3	3	3	preservative
Asam sitrat	0,15	0,15	0,15	pH neutralizer
Essensial oil (oleum rosae)	q.s	q.s	q.s	fragrance
Foam booster	1,4	1,4	1,4	Foam enhancer
Aquades	Ad 100 ml	Ad 100 ml	Ad 100 ml	Solvent

Information:
F1= soap formula with AgNO3 content 0.03 grams
FII= soap formula with AgNO3 content 0.06 grams
FIII= soap formula with AgN03 content 0.09 grams

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2.2.6. *Making Liquid Soap*

Prepare the tools and weigh all the materials needed. The first step is to add the SLS material little by little into the mortar while stirring, then add the foam baster and stir again slowly. Add Na_2S0_4 to the mortar little by little. STTP dissolved in 5 ml of distilled water, stir slowly, mix the STTP solution slowly into the mortar. When it is mixed, add citric acid that has been diluted in 5 ml of distilled water, stir gently. Then add the mixture ($AgN0_3$, 10 ml distilled water, and 1 ml turi leaf extract) which has been in the magnetic stirrer for 15 minutes. Then add oleum rosae or green tea to create a fragrant aroma in the liquid soap. Then add the remaining distilled water into the mortar and stir until homogeneous. Next, the soap preparation is evaluated by physical tests consisting of organoleptic tests, pH tests, homogeneity tests, viscosity tests, specific gravity tests and foam height tests.

2.2.7. *Physical Quality Testing of Nanoparticle Soap*

1. Organoleptic test

Organoleptic tests are carried out to determine the quality of the preparation using the five senses which include the shape, color and smell of the Turi leaf extract Ag nanoparticle soap preparation which will be observed every week for 4 weeks (Leny *et al.,* 2020).

2. Homogeneity test

The homogeneity test is carried out by placing 1 gram of the material to be tested on glass or other suitable material. It should have even measurements without grits (Yuni Daisa, Nurul and Sri, 2019).

3. Specific gravity test

Specific gravity checks were carried out every week for 4 weeks using a pycnometer. The pycnometer used is clean and dry, weigh the pycnometer empty weight (W_0). Weigh the pycnometer filled with distilled water (W_1) and weigh the pycnometer filled with Ag nanoparticle soap preparation (W_2).

Specific gravity formula: $BJ = \frac{W_2 - W_0}{W_1 - W_0}$

4. pH test

Use up to 1 gram of soap for pH testing, dilute to 10 ml with distilled water. Dip the pH meter after calibrating in the prepared soap and wait until the pH meter reading is stable and the pH reading becomes constant (SNI by Korompis *et al.,* 2020). pH was measured at weeks 1, 2, 3, and 4 (Muna *et al.,* 2021).

5. Viscosity test

Viscosity measurements were carried out using an Ostwald viscometer. Enter the preparation through tube A then suck it into tube B, right up to the A limit and prepare a stopwatch to measure time. Then the liquid is allowed to fall to line B. Note the time it takes to flow from line A to line B. Wash the viscometer thoroughly

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and dry it again. The work as above is repeated using a comparison fluid (aquadest). Calculate the specific gravity of the liquid using a pycnometer (Irawati *et al.,*2017). Calculate the viscosity of a liquid with the formula:

$$\frac{\eta 1}{\eta 2} = \frac{\rho 1\ t1}{\rho 2\ t2}$$

information :

η1 = Viscosity of the test substance

η2 = Viscosity of the reference fluid

ρ1 = Specific gravity of the test substance

ρ2 = Specific gravity of the reference liquid

t1 = Test substance time

t2 = Comparison fluid time

6. Test foam height

Testing the height of the foam, measuring is an easy way to put 1 gram of soap into a 100 ml measuring cup and add 50 ml of distilled water then cover and shake for 5 seconds and calculate the height of the foam formed, measurements are carried out every week for 4 weeks.

$$\text{Foam height test formula} = \frac{\text{final foam height}}{\text{Initial foam height}} \times 100\ \%$$

3. RESULTS AND DISCUSSION

3.1. Analysis of Characterization Results

3.1.1. Silver nanoparticle

In the process of synthesizing Ag nanoparticles using turi leaf extract, the color change was observed from clear to yellowish to brown. The color change that occurs indicates the presence of Ag nanoparticles. The solution mixture containing AgNo3 and turi leaf extract changed color from clear to yellow after 30 minutes then turned brown after around 1 hour. Furthermore, with increasing reaction time, the brown color of the combined solution increases.

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3.1.2. Test for flavonoid compounds

Table 2. Flavonoid compound test results

Perlakuan uji senyawa flavonoid	Hasil identifikasi	Keterangan	gambar
1 ml ekstrak daun turi + 2 ml etanol 95% + 2 ml HCl2N + 10 tetes HCl pekat	Kuning	+	

The flavonoid test was carried out so that in making Ag nanoparticle soap, the active substances used are expected to work optimally in cleaning hands from invisible microorganisms such as attached germs, fungi, bacteria or viruses. To determine the content of flavonoid compounds in turi leaves, you can do this by reacting turi leaf extract with 95% ethanol, HCl2N and concentrated HCl. Flavonoid compounds are polar compounds so they must be dissolved in a polar solvent, namely ethanol which has a high enough polarity so that more flavonoid compound extract can be obtained. The addition of concentrated HCl in the flavonoid test is used to hydrolyze flavonoids into their aglycones. And the addition of HCL2N functions as an attractant for flavonoid compounds in the extract. From the research results, a color change occurs, namely a yellow color is formed, this is because the falvon reacts with the reagents used.

3.1.3. Organoleptic Test

Table 3. Organoleptic test results for liquid soap

Organoleptis	Week	FI	FII	FIII
smell	1	Oleum rosae	Oleum rosae	Oleum rosae
	2	Oleum rosae	Oleum rosae	Oleum rosae
	3	Oleum rosae	Oleum rosae	Oleum rosae
	4	Oleum rosae	Oleum rosae	Oleum rosae
color	1	yellow	chocolate	Purplish brown
	2	Brownish yellow	chocolate	Blackish brown
	3	Light brown	Chocolate	black
	4	Light brown	chocolate	black
Shape and race	1	Transparent gel (gentle)	Transparent gel (gentle)	Transparent gel (gentle)
	2	Transparent gel (gentle)	Transparent gel (gentle)	Transparent gel (gentle)
	3	Transparent gel (gentle)	Transparent gel (gentle)	Transparent gel (gentle)
	4	Transparent gel (gentle)	Transparent gel (gentle)	Transparent gel (gentle)

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Organoleptic tests were carried out with the aim of looking at the physical form of the Ag nanoparticle soap preparations made including shape, odor, color and taste. The dosage form obtained is in gel form. The smell of the resulting preparation is that of oleum rosae. This distinctive aroma comes from a combination of the aroma of oleum rosae fragrance and the natural aroma of mixed turi leaf extract. The color of Ag nanoparticle soap is from yellow to blackish due to the addition of Ag to the turi leaf extract. And finally, the taste of the Ag nanoparticle soap preparation obtained is that the preparation is very gentle on the skin.

3.1.4. Homogeneity Test

Table 4. Homogeneity Test Results

formulation	Week	homogeneity
F1	1	Homogeneous
	2	homogeneous
	3	Homogeneous
	4	Homogeneous
F2	1	Homogeneous
	2	Homogeneous
	3	Homogeneous
	4	Homogeneous
F3	1	Homogeneous
	2	Homogeneous
	3	Homogeneous
	4	Homogeneous

The homogeneity test, carried out from week 1 to week 4 for each formula, showed that there were no coarse grains or particles on the surface of the Ag nanoparticle soap that were visible to the naked eye. This shows that each formula is homogeneous in the Ag nanoparticle soap preparation.

3.1.5. pH test

Table 6. pH test results

Formulation	Week			
	1	2	3	4
F1	10	10	10	10
F2	9	9	9	8
F3	9	9	9	9

The pH test was carried out to determine the alkalinity level of Ag nanoparticle soap. This is because Ag nanoparticle soap comes into direct contact with the skin and can cause problems if the pH of the Ag nanoparticle soap preparation does not match the skin's pH. The skin has a resistance capacity and can quickly adapt to products that have a pH of 8.0-10.8 (Frost et al., 1982). From the results of tests that have been carried out, Ag nanoparticle soap from all formulas is stable only in Formula 1 (F1) and Formula 3 (F3) from week 1 to week 4. Changes and differences in pH values can be caused by the ingredients. Another constituent of soap is citric acid which is acidic. And also changes in pH can be influenced by the decomposition medium, such as storage temperature, which can increase acid or alkaline

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levels. According to SNI, the pH of soap is allowed to be between 8-11. The results show that all soap formulations produced meet the criteria for good soap. A high pH can cause skin irritation because it has a high level of free alkali. The level of free alkali in soap is caused by the presence of alkali which does not react with fatty acids and the saponification process (Zulkifli and Estiasih, 2014). The amount of alkali in each formula is the same, so the pH between formulas does not have a significant difference.

3.1.6. Viscosity Test

Table 7. Viscosity Test Results

Week	Formulation	Viscosity (centipoise)
1	F1	2,138
	F2	2,672
	F3	3,746
2	F1	2,134
	F2	2,929
	F3	2,956
3	F1	2,404
	F2	3,810
	F3	3,748
4	F1	2,221
	F2	2,751
	F2	3,638

The viscosity test aims to see the viscosity resulting from the preparation being made. The viscosity test was carried out using an Ostwald viscometer. The viscosity value based on SNI soap standards is 400-4000 cPs. From the viscosity test above, it can be seen that the viscosity test results for Ag nanoparticle soap preparations are between 2.138-3.810, meaning that the soap preparations made meet the standards set by SNI. The viscosity value obtained was unstable due to an increase or decrease from week 1 to week 4.

3.1.7. *Test foam height*

Table 8. Foam height test results

Week	F1 (cm)	F2 (cm)	F3 (cm)
1	8,6	12,5	10,6
2	9,6	7,6	7,3
3	9,6	8,3	6,1
4	8,6	7,1	6,3

The foam height test aims to see the foaming power of liquid soap. The characteristics of soap foam are influenced by several factors, namely foam stabilizers, surfactants and other soap constituent ingredients. Based on SNI, the foam height of liquid soap preparations must be between 13-220 mm. From the test above, it shows that the foam height value in the Ag nanoparticle soap preparation is between 6.1 – 12.5 mm. which means it still meets the standards set by SNI. If too much foam is produced it can cause skin irritation. The function of the foam in soap is to remove oil

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or fat from the skin. If the foam in the soap is too high, it can make the skin dry. When the fat in the skin is lost, it will make the skin more susceptible to irritation, because the fat in the skin is useful as a defense. The top layer of the skin is called the skin barrier, one of the components of the skin barrier is fat. Fat will make the skin barrier tighter, so that bacteria and microorganisms cannot easily enter the body.

3.2. Uv-Vis spectrophotometric

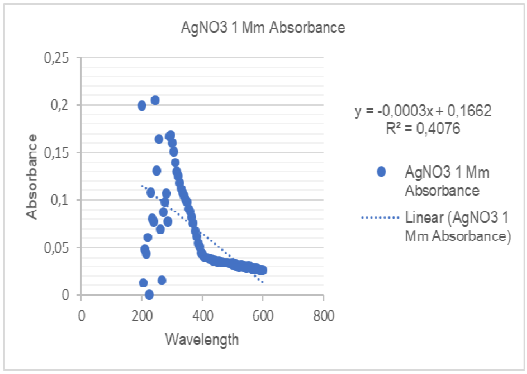

Figure 1. Absorption Spectrum of 1 mM Turi Leaf Extract Nanoparticle

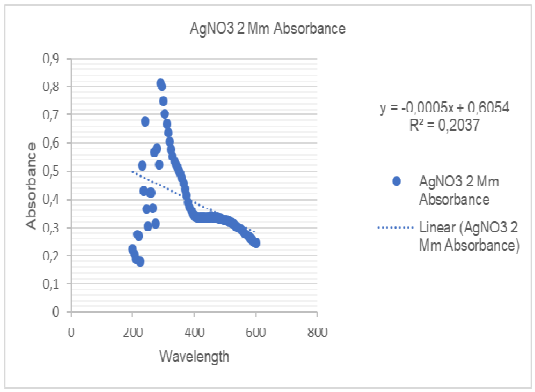

Figure 2. Absorption Spectrum of 2 mM Turi Leaf Extract Nanoparticles

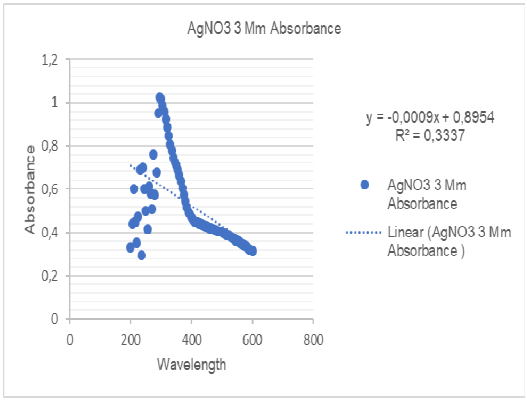

Figure 3. Absorption spectrum of 3 mM turi leaf extract nanoparticles

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Based on the results of the spectrum uptake of Ag nanoparticles from turi leaf extract (sesbania grandiflora L). at each concentration, namely 1 mM, 2 mM, 3 mM, shows the highest peak produced at a value of 280-290 nm. From image 1. A concentration of 1 mM produces a wavelength of 295 with an absorption value of 0.169, from image 2 a concentration of 2 mM produces a wavelength with an absorption value of 0.814, and from the results of image 3 a concentration of 3 mM produces a wavelength of 300 with an absorbance value of 1.105. So the results show that the optimum concentration variation in soap preparation is a concentration of 3 mM. from research (Ndikau et al., 2017) states that the greater the concentration of silver nitrate, the greater the indication of its formation. And the higher the absorption value, the higher the concentration of nanoparticles in the solution.

4. CONCLUSION

The research results show that turi leaf extract (sesbania grandiflora L) affects the physical stability of Ag nanoparticle soap and has met the standards set by SNI in organoleptic testing, homogeneity testing, specific gravity testing, pH testing, viscosity testing and foam height testing. Among formulation 1 with a concentration of 1 mM, formulation II with a concentration of 2 mM and formulation III with a concentration of 3 mM turi leaf extract soap preparations showed the best physical properties in formulation 1 with a concentration of 0.03 seen from the pH stability test. The compound content test showed positive results for the presence of flavonoid compounds. Based on UV-vis spectrophotometric measurements of the three concentrations, the most optimal in liquid soap preparations is a concentration of 3 mM with a wavelength of 300 nm with an absorbance value of 1.105.

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