Jurnal Ilmiah Berkala: Sains dan Terapan Kimia
Vol. 18 No. 2, Juli 2024

Formulation and Assesment of Sheet Mask Essence Derived from Cassava Leaf Extract (*Manihot utilissima* Crantz.)

Formulasi dan Evaluasi Esens Masker *Sheet* dari Ekstrak Daun Singkong (*Manihot utilissima* Crantz.)

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ABSTRACT

Cassava leaves are rich in flavonoids and vitamin C, serving as antioxidants to inhibitoxidation caused by free radicals. This enables the utilization of cassava leaves in cosmetic applications, such as incorporating them into formulation, such as sheet mask essence. This study aims to determine the characteristics of essence sheet mask cassava leaf extract. The preparation is made with as many 3 formulas, with cassava leaf extract concentrations of 1.0%(F1), 3.0%(F2), and 5.0%(F3). The research method used for the manufacture of essence preparation is to mix all the ingredients and add cassava leaf extract and perfume. The essence preparation is evaluated including organoleptic, pH, viscosity, and stability tests. The result of this study obtained a yellow-orange yellow essence, with a characteristic aroma of lemongrass oil and liquid consistency. An increase in the concentration of the extract results in a decrease in pH and an increase in viscosity. The findings of this research indicated the succesfull of cassava leaves extract formulation into sheet mask essence, furthermore the evaluation were covered on physical and chemical aspect, including organoleptic test, pH, viscosity, and stability.

Keywords: Sheet Mask Essence, Manihot Esculenta

ABSTRAK

Daun Singkong merupakan tanaman yang memiliki kandungan flavonoid dan vitamin C yang berperan sebagai antioksidan untuk menangkal reaksi oksidasi akibat radikal bebas sehingga manfaat dari daun singkong ini dapat diformulasikan ke dalam bentuk sediaan topikal seperti kosmetik salah satunya sebagai esens masker *sheet*. Penelitian ini bertujuan untuk menentukan karakteristik esesns masker *sheet* ekstrak daun singkong. Sediaan dibuat 3 formula dengan konsentrasi ekstrak daun singkong 1,0% (F1), 3,0% (F2), dan 5,0% (F3). Metode penelitian yang digunakan untuk pembuatan sediaan esens yaitu dengan mencampurkan semua bahan, tambahkan ekstrak daun singkong dan parfum. Sediaan esens kemudian dievaluasi fisik uji organoleptik, pH, viskositas dan uji stabilitas. Hasil dari penelitian ini diperoleh esens berwarna kuning-kuning kejinggaan, dengan aroma khas *lemongrass oil* dan konsistensi cair. Peningkatan konsentrasi ekstrak mengakibatkan penurunan pH dan peningkatan viskositas. Kesimpulan dari penelitian ini yaitu variasi konsentrasi ekstrak daun singkong berpengaruh terhadap karakteristik fisik sediaan esens masker *sheet* pada uji organoleptis, pH, viskositas, dan pada uji stabilitas *mechanical test* ketiga formula tidak stabil terhadap uji pH dan Organoleptis, dan pada uji *cycling test* semua formula juga tidak stabil secara fisik terhadap uji viskositas dan pH.

Kata kunci: Esens Masker *Sheet*, *Manihot esculenta*.

Received: 3 January 2023; **Accepted**: 7 October 2023: **Available online**: 31 July 2024

1. INTRODUCTION

Cosmetics are topical preparations that are used to treat the external parts of the human body. A sheet mask is one of the cosmetics in the form of a sheet made of cellulose fiber soaked in a liquid or essence containing active ingredients that are beneficial for the skin. The essence of this sheet mask contains ingredients partially water and humectants. Its consistency or viscosity in essence is lower than that of serum and ampoules (West *et al*., 2019; Chao, 2021). The application of the essence on this sheet mask will help its absorption into the skin more optimally by using the working principle of the sheet mask, namely ODT (Occlusive Dressing Treatment) which will help penetrate and absorb the essence better on the skin (Surjanto *et al*., 2016).

The stability of the preparation of sheet mask essence must be able to maintain its quality according to the specifications that have been set during the period of storage and use to ensure the identity of the strength, quality, and purity of a product (Rismana *et al*., 2015; Ariani & Wulandari, 2021). Stability tests are carried out to ensure the durability of the preparations has the same properties after manufacture and during storage and whether the preparations have met the predetermined criteria parameters (Sayuti, 2015).

One of the plants that can be used as the main ingredient in sheet mask essences is the cassava plant. Cassava plants are usually used by the community as food ingredients, and the leaves can be used as traditional medicines such as diarrhea and headaches (Nafilah *et al*., 2017). Cassava leaves contain flavonoid, phenolic, and vitamin C compounds that can act as antioxidants. According to research by Siyumbwa *et al*. (2014), ethanol extract of cassava leaves has an IC50 value of 95.12 mg/L, which belongs to the group of strong antioxidant activity. The benefits of antioxidants on facial skin are that they can help prevent premature aging, can protect against ROS (Reactive Oxygen species), and can protect against exposure to UV rays that can damage skin tissue (Rompis *et al*., 2019). The utilization of cassava leaves in cosmetic products is still limited, and looking at the potential of cassava leaves, researchers can use cassava leaves as ingredients for cosmetic preparations.

2. MATERIALS AND METHODS

2.1. Materials

The tools used in this study were glassware (Pyrex, Iwaki), oven (Memmert), blender (Cosmos CB-802), stir bar, pH meter (Hanna Instrument), mortar and stemper, water bath, vial, spatel, analytical balance (Ohaus pioneer PA123) and viscometer (Brookfield type LV), sieve no 20 (Retsch AS 200) Centrifugation (Clements GS 150 Centrifuge), Homogenizer (RW 20. n IKA Labortechnik) and SPSS 21 software (IBM Statistic).

The ingredients used are cassava leaves, 96% ethanol, butylene glycol, glycerol (Glycerin PH, Pharmaceutical grade, P&G Chemical), methylparaben (Methyl paraben®, Cosmetic grade, Sumber Berlian Kimia), PEG-40 hydrogenated castor oil, Gum Arabic (Gum arabic powder KB-120®, Food grade, CHEMIPAN), distilled water, oleum lemongrass oil (Lemongrass Oil®, Pharmaceutical grade, Happy Grade), phosphate buffer pH 4.0 and pH 7.0, filter paper, aluminium foil, foil bag, sheet mask paper (facial mask paper).

2.2 Method

2.2.1 Extraction

Sampling of cassava leaves was taken from, Cindai Alus area, Banjar Regency, South Kalimantan. The cassava leaves were first separated from the stems and other impurities, then washed with running water and dried in

an oven at a temperature of 50°C±2°C. The dried cassava leaves were then ground into powder, then sieved with a 20 mesh size sieve, then the simplicia powder was put into a maceration vessel and added 96% ethanol solvent with a ratio of powder and ethanol solvent 1:10 the solvent used was 5.5 L macerated for 3x24 hours (Kemenkes RI, 2020; Malik *et al*., 2020). Every 1x24 hours filtering is carried out until the maceration process is perfect. After obtaining the extraction results, the solvent was evaporated using a water bath at a temperature of 50°C±2°C to obtain a thick extract (Rikomah *et al*., 2017; Solikhah *et al*., 2019). Then the weight of the thick extract obtained was then calculated and the % yield was calculated.

2.2.2 Essence Sheet Mask Fabrication

For the first, each ingredient is weighed as needed (Table 1). Then gum arabic is dissolved with a little distilled water in a mortar of 10 mL of distilled water (mass I). After that, the methylparaben was dissolved with 10 mL of distilled water at a temperature of ±70ºC (mass II). Mix mass II into mass I to get mass III. After that, butylene glycol, glycerol, and PEG-40 hydrogenated castor oil were mixed (mass IV). Then mass IV is mixed into mass III (mass V). Then mass V was added with ethanol extract of cassava leaves which had been dissolved in 96% ethanol, then added water up to 100 mL and sufficient fragrance and then stirred until the preparation was homogeneous. After the essence of the sheet mask is used up, the empty sheet mask sheet is folded according to the package size and put into a foil bag. Then, 20 mL of essence is poured into a foil bag and tightly closed. The following formula Essence Mask Sheet Cassava Leaf Extract can be seen in table 1.

Table 1. Essence Formulation of Cassava Leaf Extract Sheet Mask.

Ingredients	Function	Formula Essence Mask sheet		
		F1	F2	F3
Cassava Leaf Extract	Active ingredients	1.0%	3.0%	5.0%
Butylene Glycol	preservative	5.0%	5.0%	5.0%
Glycerol	Humectants	5.0%	5.0%	5.0%
Methyl Paraben	preservative	0.2%	0.2%	0.2%
Ethanol 96%	Solvent	3.0%	3.0%	3.0%
PEG-40 *Hydrogenated Castor Oil*	Surfactant	0.1%	0.1%	0.1%
Arabic Gum	Gelling agent	0.5%	0.5%	0.5%
Oleum Lemongrass oil	Parfumes	3 drops	3 drops	3 drops
Aquades	Solvent	until 100 mL	until 100 mL	until 100 mL

2.2.3 Evaluation

1. Organoleptic test

An organoleptic test is an examination carried out by observing changes in color, odor, and consistency of the preparation (Kusumawati *et al*., 2020).

2. pH test

The pH meter was calibrated with a buffer solution of pH 4.0 and pH 7.0. Furthermore, the electrode was rinsed using distilled water then the pH was dipped into the preparation

and waited until the pH value on the pH meter screen was constant. The pH limit of the preparation that can be tolerated by the skin is 4.5-6.5 (Wikantyasning *et al*., 2019).

3. Viscosity test

Viscosity was measured with a Brookfield-type LV viscometer at room temperature. Nanoemulsion was put into a 100 ml container. The spindle number is set to 3 and the speed is set to 6 rpm. Then calculated the results of the scale reading (dial reading) and multiplied by a certain correction factor. The standard thickness of the essence sheet mask is between 137-275 cps (Kusumawati *et al*., 2020).

4. Stability Test

a. The cycle test method was carried out for 6 cycles with one test cycle consisting of storing essence samples in a refrigerator at 4ºC for 24 hours, after which they were transferred to an oven at 40ºC for 24 hours (Sinaga *et al*., 2014). Parameters observed were organoleptic preparations and pH.
b. In the mechanical test method, the essence preparation was put into a 10 mL tube and then centrifuged at 3000 rpm for 1 hour (Juttulapa *et al*., 2016). Parameters observed were organoleptic preparations, pH, and viscosity.

3. RESULTS AND DISCUSSION

3.1 Cassava Leaf Extract Results

The extract obtained was 106.55 grams with a percent yield of 19.51%. This percentage was higher than other researcher works - 10.48% (Rikomah *et al.*, 2017) or 0.8875% (Sari and Meltisa, 2017). The different result may arise due to different area of cultivation land resulting the variability of cassava leaves compositions. The extraction method and the extraction solvent may also contributed to the difference (Senduk *et al*., 2020). The color of the thick extract of cassava leaves obtained is blackish brown and has a characteristic smell, of thick extract, and can be seen on Figure 1.

3.2 The Result of Sheet Mask Essence

The sheet mask essence formulations were evaluated on physical and chemical properties, such as organoleptic, pH, viscosity, and stability.

3.3 Physical Evaluation of Preparations

1. Organoleptic Test Results

The results of the organoleptic test of the three formulas are that formula 1 has a transparent yellow color, formula 2 has a brownish yellow color, and formula 3 it has an orange-yellow color. The color difference in the three formulas is caused by the large concentration of extract contained in the preparation, so the more concentration of extract added, the more concentrated the color obtained (Lubis, 2020). The three formulas are scented with lemongrass to cover the distinctive aroma of cassava leaves and have a liquid consistency. The results obtained in the organoleptic test can be seenin Figure 2.

Figure 1. Cassava Leaf Condensed Extract

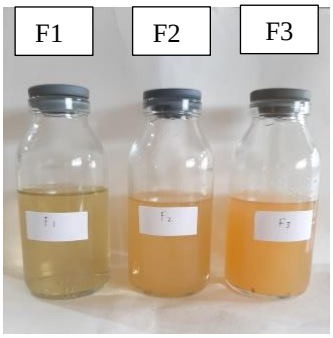


Figure 2. Results Essence Leaf Extract Sheet Mask

2. pH Test Results

The results obtained from the pH test on the three formulas entered the skin's physiological pH range of 4.5-6.5 (Wikantyasning *et al*., 2019). The results of the pH test of the three formulas for variations in the concentration of cassava leaf extract showed the addition of the extract concentration and a decrease in the pH value of the preparation. This happens because of the H+ group released from the cassava leaf extract which makes the pH of the preparation more acidic (Lubis, 2020). The results of statistical tests showed that most of the p-value <0.05, which means that the variation of extract concentration from the three formulas significantly affected the pH value. The results of the pH test can be seen in table 2.

Table 2. pH Test Results of Essence Mask Sheet Cassava Leaf Extract

Formula	Replication 1	Replication 2	Replication 3	Results (Mean ± SD)
1	5.82	5.79	5.85	5.82± 0.030
2	5.73	5.62	5.63	5.66± 0.061
3	5.29	5.31	5.35	5.32± 0.031

3. Viscosity Test Results

The results obtained from the viscosity for the three formulas have met the range of the essence viscosity which ranges from 137-275 cps (Kusumawati *et al*., 2020). The factor of variation in concentration of cassava leaf extract affects the viscosity of the preparation, the more concentration of extract added, the greater the viscosity of the preparation. The results of statistical tests showed that most of them had a p-value <0.05, which means that the variation of the extraction concentration of the three formulas significantly affected the viscosity value. The results obtained from the viscosity test can be seen in table 3.

Table 3. Viscosity Test Results of Essence Mask Sheet Cassava Leaf Extract

Formula	Replication1 (cps)	Replication 2 (cps)	Replication 3 (cps)	Results (Mean ± SD) (cps)
1	140	180	160	160.00± 20.000
2	200	220	240	220.00± 20.000
3	280	240	260	260.00± 20.000

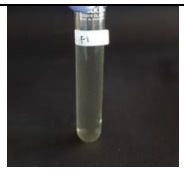
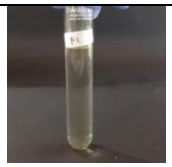
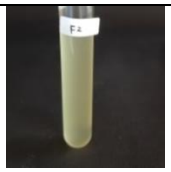
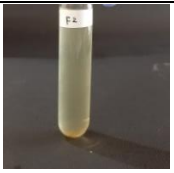
4. Stability Test Results

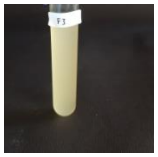
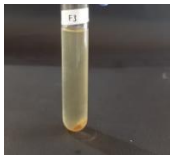
a. *Mechanical Test*

The results of the organoleptic test on the mechanical test are that in formula 2 and formula 3 there is a precipitate after centrifugation, while in Formula 1 there is no precipitate. This occurs as a result of the centrifugation process which causes the particles of dissolved substances in the preparation to become condensed because they are influenced by the force of gravity, particles that have a lower density will rise to the surface and form a higher density which will form a layer at the bottom of the preparation (Dimonie *et al*., 2013). The resulting precipitate probably occurred due to an increase in the concentration variation of cassava leaf extract in each formula.

The results of the pH test showed that the three formulas after being centrifuged experienced a decrease in pH, in formulas 1 and 2 the tolerable pH range was 4.5-6.5, while formula 3 had a value of less than 4.5. The reason the pH decreased was due to the centrifugation process resulting in an increase in temperature which caused the decomposition of compounds in the preparation, this might occur because the H+ from the cassava leaf extract was released so that the pH of the preparation decreased or became more acidic (Putra *et al*., 2014). The results of statistical tests showed a p-value <0.05 which of the three formulas had a significant effect after centrifugation on the pH value. The results of the organoleptic and pH tests from the mechanical test can be seen in table 4.

Table 4. Results of Organoleptic and pH Tests on Mechanical Tests of Sheet Mask Essences from Cassava Leaf Extract

Formula	Before Centrifugation	After Centrifugation	Results
F1	 pH : 5.82±0.030	 pH : 4.74± 0.010	There are no sediment
F2	 pH : 5.66± 0.061	 pH : 4.57± 0.053	There is sediment

F3	 pH : 5.32± 0.031	 pH : 4.43± 0.021	There is sediment

b. *Cycling Test*,

The results of the organoleptic test on the cycling test were that the three formulas did not change color, or scent during the test, but in formula 2 and formula 3 in cycles 3 and 6, there was a precipitate. The color of the precipitate obtained is brown, it is possible that the precipitate came from the less soluble extract particles due to the addition of variations in the concentration of cassava leaf extract. The results obtained from the organoleptic test to the cycling test can be seen in table 6.

The results of the pH test on the cycling test showed that the three formulas experienced a decrease in pH in each cycle. Changes in pH can occur due to environmental influences, namely temperature, and poor storage (Putra *et al*., 2014). The results of statistical tests showed that most had a p-value <0.05, which means that the pH of the preparation in cycle 0, cycle 3, and cycle 6 was unstable. The results obtained from the pH test against the cycling test can be seen in table 7.

The results of the viscosity test on the cycling test showed that from the three formulas the viscosity value of the preparation in cycle 0 to cycle 6 experienced a decrease in viscosity. This can be caused by environmental factors that can affect the viscosity of an increase in temperature at conditions with a temperature of 40°C heating occurs which causes the molecules of a liquid to move so the interaction force between molecules weakens so that the viscosity of the preparation decreases due to an increase in temperature, then The way the preparation is stored can also affect the viscosity. The addition of ingredients with a liquid consistency such as glycerol, butylene glycol, and PEG 40 hydrogenated castor oil (Sayuti, 2015). The results of statistical tests showed that most of them had a p-value <0.05, which means that the viscosity of the preparation in cycle 0, cycle 3, and cycle 6 was unstable. The results obtained from the pH test against the cycling test can be seen in table 8.

Table 6. Organoleptic Test Results of Essence Mask Sheet Cassava Leaf Extract

Cycle	Formula 1	Formula 2	Formula 3
0	Transparent yellow, distinctive smell of lemongrass, liquid	Brownish yellow, distinctive smell of lemongrass, liquid	Orange yellow, lemongrass smell, liquid
3	Transparent yellow, distinctive smell of lemongrass, liquid	Brownish yellow, characteristic smell of lemongrass, liquid and there is a sediment	Yellow-orange, distinctive smell of lemongrass, liquid and there is sediment
6	Transparent yellow, distinctive smell of lemongrass, liquid	Brownish yellow, characteristic smell of lemongrass, liquid and there is a sediment	Yellow-orange, distinctive smell of lemongrass, liquid and there is sediment

Table 7. pH Test Results against Cycling Test Essence Mask Sheet Cassava Leaf Extract

Formula	pH Value of Essence on Cycle		
	0	3	6
1	5.82 ± 0.030	5.23 ± 0.015	4.54 ± 0.010
2	5.66 ± 0.061	5.13 ± 0.021	4.43 ± 0.015
3	5.32 ± 0.031	5.06 ± 0.055	4.23± 0.021

Table 8. Viscosity Test Result of Mask Sheet Essence Cassava Leaf Extract

Formula	Viscosity Value (cps) of Essence on Cycle		
	0	3	6
1	160.00 ± 20,000	120,00± 20,000	60,00± 20,000
2	220.00± 20,000	123,33± 41,633	80,00± 20,000
3	260.00± 20,000	200,00± 20,000	126,66± 30,550

4. CONCLUSIONS

This study concludes that the successful of fabrication of cassava leaf extract into mask sheet essence. The variation of extract concentration was employed to be evaluated on physical and chemical characteristics. The organoleptic results obtained were transparent yellow to orange yellow, liquid consistency with desired scent. The formula were shown stability over mechanical stress, except for their acidity (pH).

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