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RESEARCH ARTICLE

Lactic acid clay mask formulation with plasticiser variations

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Abstract

Background: Lactic acid can inhibit the growth of acne-causing bacteria, moisturise, and remove dead skin cells. It can be formulated in clay mask preparations. One of the components of a clay mask is a plasticiser, which increases stability and controls the drying of preparations. **Objective:** To compare the effect of sorbitol and glycerol as plasticisers on the physical stability of lactic acid clay masks. **Method:** Clay masks were made with plasticiser concentrations of 0%, 2%, 3%, 4%, and 5%, then evaluated (organoleptic, homogeneity, pH, spreadability, viscosity, and flow properties) for three weeks. Furthermore, statistical analysis was done using two-way ANOVA and Friedman on the evaluation results with a p -value < 0.05 . **Result:** Lactic acid clay mask with sorbitol had a range of pH values of 4.68–5.46, spreadability of 2.97–3.78 cm, drying speed of 10.92–15.08 minutes, and viscosity of 110.73–292.71 cps. In comparison, masks with glycerol had a pH value range of 4.64–5.54, spreadability of 2.67–3.73 cm, drying speed of 10.57–14.67 minutes, and viscosity of 107.417–282.55 cps. **Conclusion:** Sorbitol and glycerol had the same effectiveness as a plasticiser in increasing the physical stability of lactic acid clay mask preparations.

Introduction

A clay mask is a popular facial treatment that rejuvenates and refreshes the skin. Clay masks can also absorb excess oil, remove blackheads, and clean facial skin so that the skin looks brighter and cleaner (Ahmad *et al.*, 2017). The active ingredient that can be combined in clay masks is Alpha-Hydroxy Acid (AHA) (Yulianti & Binaharjo, 2010). One of the compounds that belongs to the AHA group is lactic acid. A lactic acid concentration of 2% can maintain an acidic pH on the skin and prevent the growth of bacteria to maintain a healthy facial skin balance (Bruning *et al.*, 2020). In cosmetic products, lactic acid is used as a moisturiser and exfoliating agent for the skin (El Enshasy *et al.*, 2015).

In addition, lactic acid can also inhibit the activity of the tyrosinase enzyme responsible for skin tanning (Usuki *et al.*, 2003) and inhibit the growth of *Propionibacterium acne* (Chen *et al.*, 2006). Given its many benefits, this compound needs to be formulated in clay mask preparations to optimise its use.

One of the components of a clay mask is a plasticiser. A plasticiser is a non-volatile organic material with a low molecular weight that can reduce stiffness, reduce brittleness, and increase mask flexibility (Wypych, 2017; Setiarto, 2020). One example of a plasticiser is sorbitol, which has hygroscopic characteristics, is stable, non-toxic, does not cause skin irritation, and is not easily oxidised at room temperature (Rowe *et al.*, 2009; Esmaeli *et al.*, 2017). Sorbitol also has the function of reducing internal hydrogen bonds so that it will increase intermolecular distances and can increase flexibility (Hidayati *et al.*, 2015; Esmaeli *et al.*, 2017). Another example of a plasticiser is glycerol. This compound can dissolve completely in water to produce a stable mixture. It is hygroscopic, so it can maintain moisture in preparation, is not easily oxidised when stored at room temperature, is non-toxic, and does not cause irritation to the skin (Rowe *et al.*, 2009). In addition, glycerol can provide flexibility to preparations, make them easy to shape, increase polymer elasticity, and break down hydrogen bonds to

enlarge the internal space in the molecular structure (Setiarto, 2020).

In clay masks, plasticisers affect the spreadability, viscosity, and stability of preparations in the long term and can control the drying of preparations (Ahmad *et al.*, 2017). There is no information about the quality of lactic acid clay masks using sorbitol or glycerol as plasticisers. Based on this, it is necessary to research and compare the effect of increasing the concentration of plasticisers (sorbitol and glycerol) on the physical stability of lactic acid clay masks.

Methods

Materials

The materials used were an analytical balance (Ohaus), pH meter (HANNA), a mixer (Miyako), a viscometer (Brookfield RV DV-E), lactic acid (Buana Chemical), bentonite (Brickstore), kaolin (Brickstore), xanthan

gum (PT. Sumber Berlian Kimia), sorbitol (PT. Sumber Berlian Kimia), glycerol (PT. Wilmar Nabati Indonesia), lauryl glucoside (Banaransoap), nipasol (Pharmastore), and distilled water.

Lactic acid clay mask

The lactic acid clay mask with sorbitol (MCLAS) and lactic acid clay mask with glycerol (MCLAG) formula can be seen in Table I. The clay mask was made by hydrating bentonite (M1) and xanthan gum (M2) in aqua fervida for 24 hours. Nipasol was dissolved in aqua fervida (M3). Next, the kaolin was mixed with sorbitol/glycerol and stirred at 500 rpm until homogeneous. M1, M2, M3, and the remaining distilled water were mixed and stirred until homogeneous (M4). Lauryl glucoside was gradually added to M4 while stirring until homogeneous. Then, lactic acid was added to the mixture while stirring at the same speed until homogeneous. Finally, TEA was added until the acidity of the preparation reached 4.5–6.

Table I: Lactic acid clay mask formula

Components	Base of clay mask (% b/b)	MCLAG (% b/b)				MCLAS (% b/b)			
		I	II	III	IV	I	II	III	IV
Lactic acid	2	2	2	2	2	2	2	2	2
Bentonite	1	1	1	1	1	1	1	1	1
Kaolin	34	34	34	34	34	34	34	34	34
Xanthan gum	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8
Glycerol	0	2	3	4	5	0	0	0	0
Sorbitol	0	0	0	0	0	2	3	4	5
Lauryl glukoside	2	2	2	2	2	2	2	2	2
Nipasol	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Triethanolamine	2	2	2	2	2	2	2	2	2
Distilled water until	100	100	100	100	100	100	100	100	100

Evaluation of lactic acid clay mask

Evaluations were conducted at weeks zero, one, two and three as follows;

Organoleptic and homogeneity observation of clay masks

It was done by observing the colour, smell, and texture. Colour observations were carried out with the naked eye using a black background, odour observations by aerating, and texture observations using fingertips (Elmitra, 2017). As much as 0.5 grams of the mask was smeared on the glass object, and the surface was observed visually to determine the homogeneity of the preparation (Mardikasari *et al.*, 2017).

Measurement of pH

The clay mask pH was measured with a pH meter (Utari *et al.*, 2019).

Measurement of spreadability

A total of 0.5 grams of the mask was placed between two round glass scales. It was left for one minute, and the diameter of the preparation spread from four sides was measured. A load weighing 50 grams was added and left for one minute, after which the measurements were taken. The process was repeated, bringing the total load to 100 grams (Yulianti & Binaharjo, 2010).

Determination of rheology

Viscosity measurements were carried out using a Brookfield viscometer with spindle number five and a speed of 1 rpm. Meanwhile, the flow properties used spindle number five with speeds of 0.3, 0.5, 0.6, 1, and 1.5 rpm. Next, a flow property curve was made between the shear stress (x-axis) and the shear rate (y-axis) of the preparation (Sinko, 2022).

Measurement of drying speed

As much as 0.5 grams of the mask was rubbed on the back of the hand, and using a stopwatch, the time it took for the preparation to dry (a dry mask layer forms) was determined (Luthfiyana et al., 2019).

Data analysis

Test data that were normally distributed were analysed using two-way ANOVA with a 95% confidence level and then continued with the Tukey HSD test. The test data that were not normally distributed were analysed using non-parametric Friedman with a 95% confidence level.

Results

The resulting MCLAS and MCLAG had the same organoleptic and homogeneity: a white colour, a distinctive smell, a thick texture, and homogeneity. The homogeneity of the preparation was indicated by the absence of particles or lumps when the preparation was applied to the object glass. The results of organoleptic test observations for three weeks of storage showed no change in odour, colour, texture, or homogeneity in each formula.

Flow properties are related to the characteristics of the preparation, such as consistency and dispersion ability. The ten clay mask formulas had thixotropic plastic flow properties (Figure 1); The plastic flow property had a yield value characteristic. The measurement showed an increase in the pH of the clay mask preparation, as well as an increasing concentration of sorbitol or glycerol (Tables II and III).

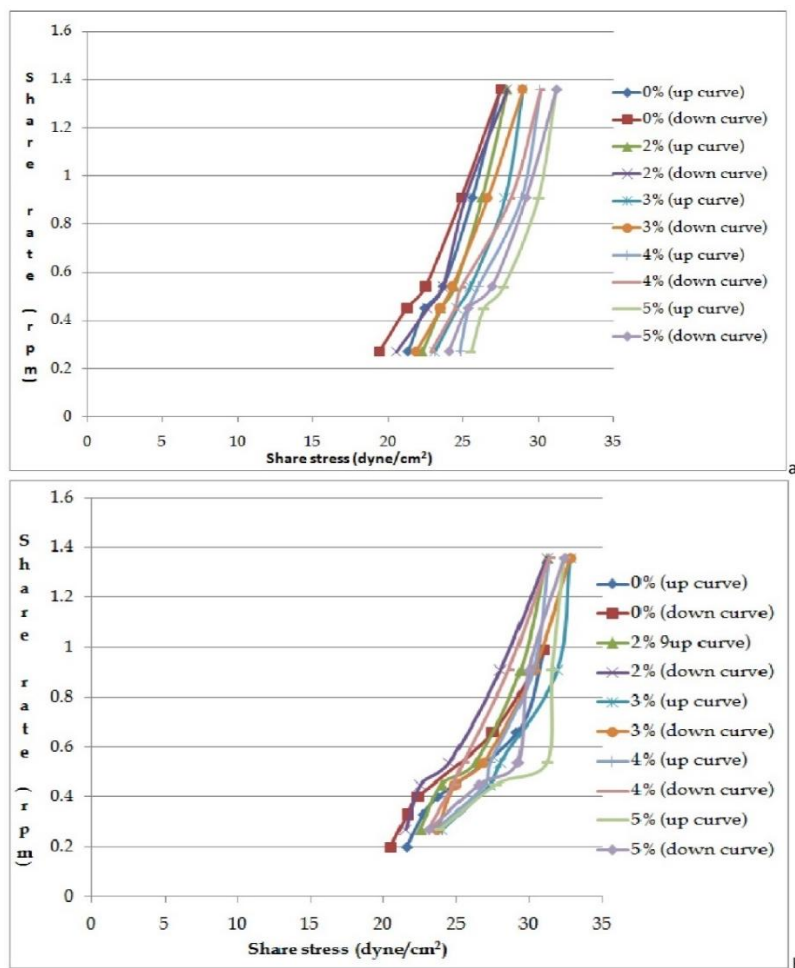


Figure 1: Rheology of MCLAG (a) and MCLAS (b)

Table II: Physical stability of MCLAG

Parameters	Time (week)	Glycerol (%)				
		0	2	3	4	5
pH value*	0	4.64 ±0.04	4.82±0.02	4.91±0.02	4.97±0.02	5.12±0.06
	1	4.68±0.03	4.89±0.02	5.06±0.08	5.24±0.03	5.36±0.04
	2	4.82±0.04	5.17±0.02	5.27±0.02	5.38±0.03	5.47±0.04
	3	4.93±0.03	5.22±0.03	5.34±0.04	5.44±0.03	5.54±0.05
Spreadability (cm)*	0	3.73±0.03	3.49±0.05	3.38±0.03	3.24±0.04	3.16±0.03
	1	3.6±0.03	3.34±0.03	3.23±0.03	3.14±0.05	2.99±0.02
	2	3.44±0.04	3.22±0.04	3.07±0.02	2.998±0.02	2.78±0.01
	3	3.32±0.02	3.11±0.04	2.92±0.04	2.78±0.03	2.67±0.03
Viscosity (cp)*	0	107.41±0.21	108.11±0.16	109.51±0.34	110.50±0.19	114.62±0.31
	1	209.31±0.36	220.73±0.20	221.72±0.21	231.60±0.74	234.71±0.13
	2	212.79±0.77	233.03±0.15	241.25±0.27	246.20±0.21	250.34±0.23
	3	241.44±0.15	251.59±0.18	262.19±1.12	274.39±1.10	282.55±0.62
Drying speed (minute)*	0	12.05±0.40	12.68±0.28	13.23±0.03	13.71±0.27	14.67±0.33
	1	11.34±0.05	11.68±0.30	12.23±0.03	12.63±0.32	13.15±0.04
	2	11.15±0.02	11.37±0.03	12.04±0.04	12.24±0.03	13.04±0.03
	3	10.57±0.02	11.25±0.04	11.53±0.03	12.10±0.03	12.53±0.04

*n = 3

Table III: Physical stability of MCLAS

Parameters	Time (week)	Sorbitol (%)				
		0	2	3	4	5
pH value*	0	4.68±0.02	4.87±0.02	4.91±0.03	4.98±0.01	5.06±0.02
	1	4.7±0.01	4.89±0.01	4.97±0.02	5.05±0.06	5.10±0.01
	2	4.76±0.02	5.08±0.09	5.21±0.05	5.33±0.03	5.43±0.01
	3	4.85±0.04	5.09±0.09	5.23±0.06	5.36±0.01	5.46±0.01
Spreadability (cm)*	0	3.78±0.09	3.62±0.07	3.61±0.02	3.55±0.02	3.52±0.01
	1	3.43±0.06	3.42±0.04	3.41±0.05	3.37±0.01	3.19±0.12
	2	3.40±0.01	3.26±0.04	3.19±0.01	3.17±0.01	3.10±0.02
	3	3.34±0.02	3.23±0.01	3.18±0.01	3.17±0.00	2.97±0.05
Viscosity (cp)*	0	110.73±0.31	112.87±1.86	114.77±0.93	115.06±0.58	118.53±1.10
	1	240.20±0.20	250.67±0.11	250.77±0.68	252.33±1.10	265.36±1.06
	2	241.03±0.68	261.13±1.79	268.00±7.03	272.03±1.91	273.53±0.01
	3	278.23±6.03	278.48±0.16	279.7±0.46	280.83±0.63	292.71±0.27
Drying speed (minute)*	0	11.52±0.07	12.55±0.04	13.39±0.15	14.18±0.15	15.08±0.11
	1	11.30±0.17	12.47±0.16	13.17±0.17	13.79±0.45	14.49±0.1
	2	11.25±0.14	12.36±0.14	12.95±0.32	13.40±0.22	14.18±0.06
	3	10.92±0.36	12.08±0.06	12.49±0.08	13.10±0.06	13.52±0.07

*n = 3

Discussion

The measurement results showed an increase in the pH of the clay mask preparation, along with an increasing

concentration of sorbitol or glycerol (Tables II and III). This is because the structure of sorbitol and glycerol has an OH-group, so the higher the concentration of sorbitol or glycerol, the higher the OH-concentration in

the preparation, which increases the pH value of the preparation (Hidayati *et al.*, 2019). Good topical preparations have a pH value within the pH range of the skin, namely 4.5–6. If the pH of the preparation is too acidic (< 4), it can irritate, and if the pH is too alkaline (> 7), it causes dry and scaly skin (Yulianti & Binaharjo, 2010; Nisa *et al.*, 2017). During the three weeks of storage, all preparations met the skin pH requirements, which is good, so it does not irritate the skin. The ten formulas for clay mask preparations experience an increase in pH every week because sorbitol and glycerol can be decomposed by the presence of air, which can increase acid or alkaline levels in clay mask preparations (Putra *et al.*, 2014).

The test results showed a decrease in the spreading power of the clay mask preparation along with the increasing amount of sorbitol or glycerol, but it still met the good spreadability range (Tables II and III). The decrease in spreading power occurs because sorbitol or glycerol can increase the size of the molecular units, causing the preparation to become viscous and reducing the ability to spread (Hidayati *et al.*, 2015). Clay mask preparations have good spreading power, ranging from 2 cm to 5 cm. Clay masks experienced a decrease in spreadability during storage, but still met a good range of spreadability. This happens because sorbitol and glycerol can also bind water, so that the preparation will become thicker and cause a decrease in the ability to spread the mask during storage (Hidayati *et al.*, 2015). Besides plasticisers, using polymers can also significantly reduce the spreadability of semisolid preparations (Wiyono & Mustofani, 2019). Using glycerol at 10% on the gel mask will increase the spreadability and slow the drying time due to a decrease in the viscosity of the preparation, so it is necessary to add a dry accelerator to the formula (Silvia & Dewi, 2022).

The test results (Tables II and III) showed that the viscosity of the preparation was in the viscosity range for clay masks, namely 100,000 – 296,000 cps (Santoso *et al.*, 2018). During the three weeks of storage, the ten formulas experienced an increase in viscosity value due to the large amount of water content bound by sorbitol or glycerol, causing an increase in the viscosity value that causes the clay mask preparations to become thick and difficult to flow (Hidayati *et al.*, 2015).

The MCLAS viscosity was greater than that of MCLAG. This is due to differences in the molecular weights. Sorbitol has a molecular weight of 182.17 g/mol and has six carbon atoms and six hydroxy groups (Rowe *et al.*, 2009). While glycerol has a molecular weight of 92.09 g/mol, its structure only has three carbon atoms and three hydroxyl groups (Rowe *et al.*, 2009). The large molecular weight of plasticiser compounds

increases the free volume of polymer chains that can bind water molecules (Cheng *et al.*, 2006). The more water molecules bound to the polymer chain (xanthan gum), the higher the density and viscosity of the preparation.

In the formation of potato starch films, the use of sorbitol (1.45–1.43 g/cm³) produces a greater density than glycerol (1.39–1.30 g/cm³) (Hazrol *et al.*, 2021). Likewise, in the formation of palm sugar starch films, the film density with sorbitol (1.523 – 1.496 g/cm³) is greater than that with glycerol (1.493 – 1.407g/cm³) (Sanyang *et al.*, 2016). The two studies showed that glycerol and sorbitol produced insignificant differences in density values, so the viscosity values were not significantly different.

The results of the drying speed test showed that increasing the sorbitol or glycerol concentration would extend the mask's drying time (Tables II and III). The increase in mask drying time occurred because sorbitol has a hydroxyl group that can bind water, so the more sorbitol concentration, the more water will be bound and cause the preparation to become viscous and have a longer drying time (Riyanto *et al.*, 2017). Meanwhile, glycerol has hygroscopic properties with a high affinity for water, can absorb moisture, and can hold water molecules, thereby reducing the evaporation of water content from the preparation and maintaining stability (Sukmawati *et al.*, 2014). The same thing is also seen in the formation of thermoplastic starch; sorbitol molecules replace the position of starch molecules and form hydrogen bonds with water, resulting in increased mobility, reduced stiffness, and lengthened drying time of starch (Esmali *et al.*, 2017). In clay mask preparations, the drying time is good, i.e. 10-20 minutes (Septiani *et al.*, 2011), and all preparations meet the requirements for a good clay mask drying time.

Statistical analysis of evaluation data of MCLAS used Friedman (pH, spreading power, and viscosity) and two-way ANOVA (drying speed). In contrast, MCLAG data were analysed using two-way ANOVA (pH, spreading power, and viscosity) and Friedman (drying speed). From both analyses, a significant value of < 0.05 was obtained; this indicated that the use of sorbitol or glycerol at 1% intervals and storage time significantly affected the evaluation of the mask. At a plasticiser concentration of 2%, the pH and spreading power data were analysed statistically using an independent parametric sample t-test and obtained a significant value > 0.05, which indicated that the use of sorbitol or glycerol and storage time did not change the pH and spreading power value significantly.

To compare the ability of sorbitol and glycerol, the data on viscosity and drying speed were used from the

formula, using a plasticiser concentration of 2% at week zero. The results of the independent parametric sample t-test analysis obtained a significant value < 0.05 , which indicates that the use of sorbitol or glycerol affects the viscosity and drying speed value significantly. In the third week, the independent parametric sample t-test analysis obtained a significant value > 0.05 , which shows that the use of sorbitol or glycerol resulted in an insignificant difference in the stability of the viscosity and drying speed value.

Conclusion

Applying sorbitol or glycerol as a plasticiser at 1% intervals at a 2-5% concentration can increase clay mask preparations' physical characteristics (pH, spreading power, viscosity, and drying speed). The differences in physical and chemical properties between sorbitol and glycerol only affect the properties of the clay mask preparation, but do not affect the ability of the two plasticisers to maintain physical stability for three weeks of the clay mask preparation.

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