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## Analysis and Characterization of Interactions Between Components in Claymask Formulations

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### ABSTRACT

*The basic properties that need to be considered and maintained in the use of an ingredient in cosmetic and pharmaceutical formulations are: colloidal size, plasticity of crystal structure, mineralographic composition, high cation exchange capacity, development capacity, high specific surface area, and consequently strong adsorption and absorption capacity. Clay masks can contain active ingredients in the form of antibacterial with alternative natural ingredients such as babadotan leaf extract. The analysis of the functional cluster in the study was obtained using the Fourier Transform Infrared (FT-IR) method. Evaluation of clay mask preparations includes testing of physical and chemical properties. Testing of physicochemical properties consists of organoleptics, pH, homogeneity, dispersibility, adhesion and antibacterial test against bacteria p. Acnes. The stability of the preparation will be damaged if the mixing system is disturbed, mainly due to temperature changes and changes in composition due to excessive addition of one of the phases or if the emulsifying substances are not mixed with each other. The extraction results obtained from babadotan the yield produced is 17.63%. The FTIR spectrum obtained for the clay mask formulation shows the presence of new peaks at wave numbers 3310, 2120, 1430 and 2126  $\text{cm}^{-1}$  where these peaks appear due to the additional ingredients used in the cosmetic formulation. The interaction between the components of the clay mask depends on the type of clay being applied. Interactions can be seen in FTIR with additional spectral peaks caused by other additives such as glycerin, in which the interaction shows the OH group involved in water absorption. This is because the results of all evaluations, Formula I tends to be stable close to the requirements of the clay mask preparation*

Keywords: kaolin, bentonite, clay mask, characterization FTIR of clay and product clay cosmetic

### 1. INTRODUCTION

The use of clay minerals for cosmetic and pharmaceutical purposes has increased in recent years, due to the increasing success of natural remedies. In addition, clay is basically considered a non-toxic and non-irritating material at the level used in pharmaceutical and cosmetic products. Clay is mainly composed of

mostly hydrated alumino-silicates arranged in tetrahedral or octahedral sheets connected by oxygen atoms occupying the nodes, while aluminum and silicon occupy the center of the polyhedra<sup>2</sup>

The basic properties that need to be considered and maintained in the use of an ingredient in cosmetic and pharmaceutical formulations are: colloidal size, plasticity of crystal structure, mineralographic composition, high cation exchange capacity, development capacity, high specific surface area, and consequently strong adsorption and absorption capacity<sup>1</sup>. Clay mask is a type of paste-shaped cosmetic made from clay minerals. One type of clay used as a clay mask is bentonite and kaolin. Clay is a commonly used raw material in health and cosmetic applications after many researchers studied and reported on its physical and chemical properties<sup>5</sup>.

In formulating clay masks, there are several problems that need to be considered, especially in terms of the combination of kaolin and bentonite base variations. According to research conducted by Hidayati, Amananti and Santoso (2019), the results of the evaluation of clay mask preparations show that less stable formulas are found in formulas that only add kaolin or bentonite singly, in terms of pH, adhesion and dispersion that do not meet the requirements<sup>1</sup>. The choice of clay mask as the form of preparation is because it has advantages in the structure and texture of the preparation where the advantages of clay mask are high absorption capacity, high surface area, solubility in water, reactivity with acids, high refractive index, large heat retention capacity, opacity, low hardness, high reflectance and good rheological properties.<sup>4</sup>

Clay masks can contain active ingredients in the form of antibacterial with alternative natural ingredients such as babadotan leaf extract. Tanaman bandotan (*Ageratum conyzoides* L.) is one of the plants that is efficacious as a medicine<sup>6</sup>. Babadotan leaves have pharmacological effects that have benefits as wound healing, including acne wounds. Bandotan leaves contain secondary metabolites for anti-oxidants, prevention and treatment of acne. Natural extracts can be a valuable addition to clay mask preparations to provide additional benefits to the skin. The addition of plant extracts is considered safer with minimal toxic effects on its use<sup>7</sup>.

Based on the description above, in this study will be carried out "Preparation and Characterization of Clay Mask Formulation with Babadotan Leaf Extract (*Ageratum conyzoides* L.)". In the study, variations of kaolin and bentonite will be carried out to obtain the best formulation conditions, therefore the structural properties, chemical composition, and physical/mechanical properties of clay mask preparations will also be analyzed.

## **2. EXPERIMENTAL**

### *2.1. Chemicals, Equipment and Instrumentation*

The materials used in this research are Pharmaceutical grade kaolin and bentonite purchased from Chemical stores, glycerin (C<sub>3</sub>H<sub>8</sub>O<sub>3</sub>), Sodium lauryl sulfate (SLS), Nipagin (C<sub>8</sub>H<sub>8</sub>O<sub>3</sub>), xanthan gum purchased from Chem-Lab, Bandotan leaf extract (*Ageratum conyzoides* L.) and aquadest. -400 spectrometer). The equipment used in this research are 100 ml measuring cups (pyrex), 250 and 100 ml glass beaker (pyrex), stirring rods, analytic scales, petri (iwaky) cups, object glass, watch glass, spatula, glass jars, droppers and pH meter and Fourier Transform Infrared Spectrophotometer (FTIR)

## 2.2. Research Procedure

### 2.2.1 Extraction of babadotan leaves (*Ageratum conyzoides* L.)

A sample of bandotan leaves (*Ageratum conyzoides* L.) in the form of simplicia powder is macerated as much as 200 grams and put into a glass jar with 96% ethanol solvent until submerged for 3x24 hours. In the maceration process, 3 repetitions are carried out and then filtered so that filtrates and residues are obtained. The filtrate obtained is concentrated with a vacuum rotary evaporator so that a concentrated extract is obtained.

### 2.2.2 Preparation of Clay Mask Formulation

The clay mask formulation for the 100 g formula is to put 56.4 ml of aquadest into the mortuary, put 1 g of bentonite into the mortar until the bentonite is wet, add 25 g of kaolin (Formula 1) little by little and grind until homogeneous. Add 0.5 g of xanthan gum and grind again. Add 8 g of glycerin to the grease until homogeneous. In another container dissolve 0.1 g of nipagin with hot water (solution 1) and dissolve 2 g of sodium lauryl sulfite sulfate with aquadest (solution 2). Pour solution 1 into the lampshade gently. Pour solution 2 into the lampung little by little, grind until homogeneous and form a clay mask base. Add the bandotan leaf extract to the grease until homogeneous.

**Table 1.** Formulation of Clay Mask

Material	Weight per Formula (% concentration)			Function
	F1	F2	F3	
Babadotan leafextract	5 %	10 %	15 %	Active Substances
Bentonite	1 %	2%	3%	Basis <i>clay mask</i>
Kaolin	25 %	30 %	35 %	Basis <i>clay mask</i>
Xanthan Gum	0,5 %	0,5 %	0,5 %	Thickener
glycerine	8%	8%	8%	Humectants
Nipagin	0,1 %	0,1 %	0,1 %	Preservatives
Sodium LaurylSulfate	2%	2%	2%	Surfactants
Aquadest	Ad 100	Ad 100	Ad 100	Solvent

### 3. RESULTS AND DISCUSSION

#### 3.1. Extraction Results

Extraction is the process of withdrawing active components/substances from a mixture of solids and/or liquids using certain solvents. The selection of the extraction method depends on the properties of the material and the compound being isolated. Maceration extraction is an extraction method by soaking materials with solvents that are suitable for the active compounds to be taken with low sorption or without a heating process.

The extraction results obtained from babadotan leaf extract which has a dark greenish color, thick shape and smell typical of babadotan leaves and the yield produced is 17.63%. The magnitude of the yield indicates the number of components extracted during the maceration process (Rosidi, et al., 2014). The high yield of the extract from this extraction process is in accordance with the standard of good yield value of >10% according to the herbal pharmacopoeia.



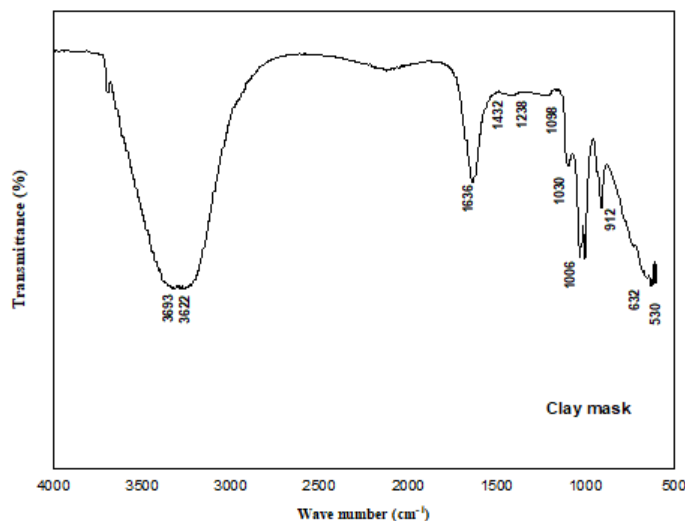
**Figure 1.** Extraction of babadotan leaf

#### 3.2 Preparation of Clay Mask

The preparation process in the clay mask formula is carried out using tools such as mortar and pestle that are heated for sterilization to avoid contaminants to microorganisms that contaminate the material and final product. In the preparation of preparations, Babadotan leaf extract is added after the clay base has formed and the base temperature has begun to decrease, with the aim that the extract's antibacterial active compounds are not lost or damaged. The result of the preparation is a homogeneous preparation with different textures from each formulation

#### 3.3 Characterization of Clay Materials and Clay Mask Products

The analysis of the functional cluster in the study was obtained using the Fourier Transform Infrared (FT-IR) method, which is an infrared spectroscopic method equipped with Fourier transform for the analysis of the spectral results. The results of the FTIR of the clay base and the clay mask formula are presented in figure 1 where the X-axis is the wave number and the Y-axis is the transmittance percentage.



**Figure 2.** Result from FTIR clay mask

Based on the FTIR spectrum in Figure 4.1, it can be seen that the clay base and clay mask formula are in the wavelength range of 4000-500  $\text{cm}^{-1}$ . Kaolinite peaks are in the wavelength range of 3692, 3620, 1003 and 911  $\text{cm}^{-1}$ . FTIR on a clay basis showed that there was absorption at the OH group wave number which was observed at wave numbers 3692 and 3620  $\text{cm}^{-1}$ , while there was a bending of the Al-O group at a peak of 911  $\text{cm}^{-1}$  which could be associated with the presence of aluminosilicate minerals. According to previous research (Madikizela et al., 2017) it was reported that pure kaolinite shows two peaks at 797 and 750  $\text{cm}^{-1}$  and well-crystallized kaolinite shows two sharp peaks at 3690 and 3620  $\text{cm}^{-1}$ . The FTIR spectrum obtained for the clay mask formulation shows the presence of new peaks at wave numbers 3310, 2120, 1430 and 2126  $\text{cm}^{-1}$  where these peaks appear due to the additional ingredients used in the cosmetic formulation.

### 3.4 Evaluation of clay mask preparations

The decision analysis was carried out to determine the best formulation of the clay mask of babadotan leaf extract (*Ageratum conyzoides* L.) with the difference in the kaolin and bentonite bases that have been made. Decision making is carried out by considering the parameters which are the physical and chemical properties as well as the characteristics of the preparation. In this study, the preparation and characterization of the clay mask formulation of babadotan leaf extract (*Ageratum conyzoides* L.) with variations of kaolin and bentonite as a base has met the requirements and is stable based on the results of the tests carried out. Organoleptic test result can be seen from the following table 2.

The pH in a formulation can affect the stability of the resulting preparation. Formula III has the highest pH caused by the concentration of kaolin, where formula III has the highest concentration of kaolin at 35%. The best pH value according to SNI requirements is the smallest pH value but still falls within the pH value range of topical preparation requirements.

Based on the results of the spreadability test, all clay mask formulas are stable despite the increase.

However, clay masks are still considered to meet the requirements because they are still entering the range of 2-5 cm spread. According to Numberi et al, in 2020 the spread of masks is said to be good if the spread of masks increases along with the increasing burden given. The adhesion test of each formula has also met the requirements. The larger the base level, the longer the adhesion to the formula. The adhesion of the preparation is related to the concentration of the bases i.e. kaolin and bentonite. The adhesion of a good formulation is more than 4 seconds.

**Table 2.** Organoleptic test result

Formulation		Observation		
	Colour	Smell	Homogeneity	Texture
F1	Dark green	Typical	Homogen	Semi-solid paste like cream
F2	Green	Typical	Homogen	Semi-solid paste like a denser cream
F3	Light green	Typical	Homogen	Semi-solid paste

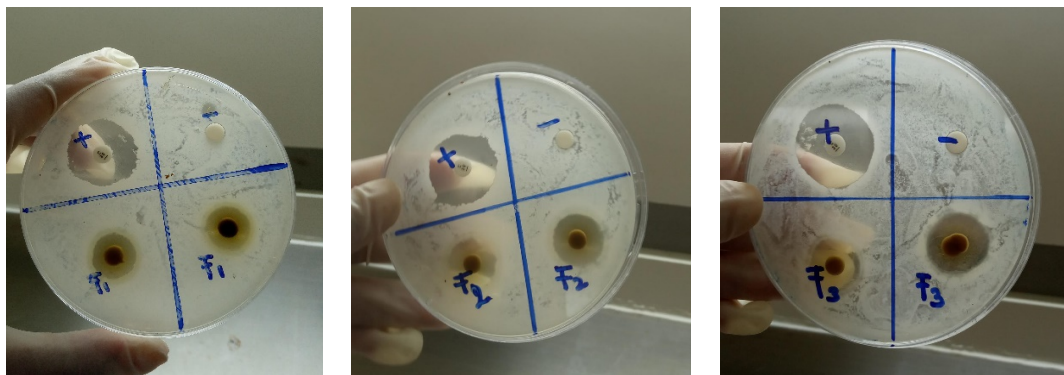
In terms of homogeneity, using a 100x magnification light microscope, a clay mask preparation of babadotan leaf extract (*Ageratum conyzoides* L.) met the requirements and was stable. Based on the test results, the base of kaolin and bentonite does not affect the homogeneous form of a preparation. For organoleptic testers, stable preparations were obtained that met the requirements, this was based on a questionnaire obtained from 10 respondents. In addition, the preparation does not cause allergic reactions and irritation when applied to the respondents' skin. Based on the results of the antibacterial activity test, the clay mask formulation made has antibacterial activity against *p.acnes* bacteria

**Table 3.** Antibacterial Activity Test Results of Babadotan Leaf Extract Clay Mask Formula

Treatment	Flat diameter of the resistance zone (mm)
	<i>Propionibacterium acnes</i>
Kloromfenikol (control +)	21,7
DMSO (control -)	0
Formulation I	17,25
Formulation II	17,45
Formulation III	18,3

The test of the potential antibacterial activity of clay masks aims to determine the antibacterial ability

of the extract to be made into preparations in inhibiting bacterial growth. The prepared preparation tested is a clay mask formula with a difference in the concentration of kaolin and bentonite base. In this study, the bacteria used is *p.acne*. The results of the antibacterial test can be seen in figure 3 below:



**Figure 3.** Images of Antibacterial Activity Test Results of each Clay Mask Formula

From the results of the inhibition zone, it can be said that the formulation of clay mask preparations can increase the antibacterial activity of babadotan leaf extract compared to the extract alone. This is because the clay mask formulation has several ingredients that can support the antibacterial activity such as the two bases used.

#### 4. CONCLUSION

The addition of babadotan leaf ethanol extract to clay mask preparations shows the peak of absorption which indicates the -OH group, secondary C-O alcohol, aromatic C-H, C=O and aromatic C=C derived from flavonoid compounds contained in babadotan leaf ethanol extract (*Ageratum conyzoides* L.) where flavonoid compounds. The optimum formulation of clay mask preparations was obtained in formulation III which was reviewed from chemical tests in terms of pH, adhesion, spreadability, homogeneity and antibacterial activity tests.

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