MUKHTABAR
JOURNAL OF MEDICAL LABORATORY TECHNOLOGY

VOLUME 3 (1), 2025, 1-8 | RESEARCH ARTICLE
e-ISSN: 2987-0224

Formulation and Evaluation of Clove Flower Extract (*Syzygium aromaticum* L.) Toothpaste Aidie With Variations of NA CMC Concentration as a Binder

Sinta Herawayanti[1], Marlina Indriastuti[1*]

[1]Pharmacy Departement, STIKes Muhammadiyah Ciamis, West Java, Indonesia

*Corresponding author: marlina.tirtahadidjaya@gmail.com

ABSTRACT

Background & Objective: Toothpaste is a semi-solid preparation that contains abrasives, cleansers, and additives to clean teeth without damaging oral tissues. Clove flower (*Syzygium aromaticum* L.) is known to contain eugenol, saponins, and flavonoids, which are analgesic. The purpose of this study was to make and evaluate toothpaste preparations from clove flower extract.
Method: The research was conducted experimentally with toothpaste formulation using variations in Na CMC concentration as a binder: 2%, 1%, and 0.5%. Evaluation included organoleptic test, homogeneity, pH, viscosity, spreadability, foam formation, and physical stability. Data were analyzed using SPSS.
Result: All formulas met the evaluation standards. Formula I was closest to the positive control in organoleptic, having an average viscosity of 9600 mPa.s and spreadability of 5.2 cm. Formula II showed the best pH with an average of 8.74. All three formulas produced foam well. Physical stability for 4 weeks showed that the texture and pH remained stable.
Conclusion: Na CMC affects the viscosity and spreadability of toothpaste preparations. Formulas with the right concentration of Na CMC can produce herbal toothpaste that is stable and meets quality requirements.

Keywords: Toothpaste; Clove Flower Extract; Toothpaste Evaluation.

INTRODUCTION

Clove (*Syzygium aromaticum* L.) is a spice plant that has long been used in various industries, such as food, pharmaceuticals, and cosmetics. The flower part of this plant contains major active compounds such as eugenol, saponins, flavonoids, and tannins. Eugenol acts as an analgesic and anti-inflammatory through the mechanism of cyclooxygenase and lipooxygenase inhibition, while saponins and flavonoids show antioxidant, antibacterial, and immunostimulatory activities.

Tannins themselves have potential as antiseptics and antitumors (Ilmiah et al., 2017); (Andries et al., 2014); (Susilowati & Wahyuningsih, 2014).

Extraction of active compounds from plant materials can be done through cold methods such as maceration and percolation, as well as hot methods such as reflux, Soxhlet, digestion, infusion, and decoction. Important factors in the extraction process include particle size, solvent selection, temperature, and process duration. Stirring can accelerate the diffusion of active compounds and increase extraction efficiency (Moh RI, 2000; (Winarno, 2004)

Toothpaste, as a semi-solid preparation, consists of various components such as abrasives, humectants, surfactants, binders, sweeteners, preservatives, and water. The binder used in this study is sodium carboxymethyl cellulose (Na CMC), which is known to have high viscosity, is stable at various pH and electrolytes, and is able to increase the stability of the preparation. Other components have specific functions such as plaque removal, maintaining moisture, producing foam, and providing flavor and aroma (SNI 12-3524-1995; (Makanan, 2021).

Making toothpaste preparations can be done through mixing or melting methods, where all components are mixed homogeneously. Evaluation of preparation quality is carried out through various tests such as organoleptic tests, homogeneity, pH, viscosity, spreadability, foam formation, and physical stability. These evaluation standards are important to ensure the physical stability and convenience of product use. (Winarno, 2004); (Indonesia, 2000)

Previous studies have shown that the ethanol extract of clove flowers can inhibit the growth of Streptococcus mutans, the main bacteria that cause caries. In addition, the concentration of Na CMC was shown to affect the physical characteristics of the preparation, especially viscosity and spreadability, which are indicators of the stability and ease of application of toothpaste. (Andries et al., 2014); (Ilmiah et al., 2017).

Thus, the use of clove flower extract in Na CMC-based toothpaste formulations has great potential as a natural alternative in maintaining oral health and is able to meet the quality parameters of topical preparations according to standards.

OBJECTIVE

The purpose of this study was to formulate toothpaste preparations with active ingredients in the form of clove flower extract (*Syzygium aromaticum* L.) and use sodium carboxymethyl cellulose (Na CMC) as a binder, and evaluate the physical quality and stability of the resulting preparations.

METHOD

This research is an experimental study that aims to design and evaluate toothpaste preparations based on clove flower extract (*Syzygium aromaticum* L.) with varying concentrations of sodium carboxymethyl cellulose (Na CMC) as a binding agent. This study was conducted at the pharmacy technology laboratory of STIKes Muhammadiyah Ciamis. Three formulas were developed with Na CMC concentrations of 2%, 1%, and 0.5%, respectively, while the concentration of clove flower extract used remained at 5%. The extraction process was carried out using the maceration method with 70% ethanol solvent. Each formula was made in three replicates and evaluated through a series of physical and stability tests.

Tools and Materials

The materials used are NaCMC, glycerin, CaCO3, clove flower extract, methyl paraben, xylitol, SLS, and distilled water. The tools used are Blender, glassware, analytical scales, rotary evaporator, drying oven BOV-T140C, hotplate, mortar and pestle, and blender.

Clove Flower (*Syzygium aromaticum* L.) Extraction

Clove flowers (*Syzygium aromaticum* L.) were obtained from the Umbungagung plantation, Panumbangan, Ciamis. Clove flowers were dried and pollinated. The powdered simplisia was extracted using the maceration method.

Formulation

The formula in this study is listed in Table 1.

TABLE 1. Formulation of Clove Flower Extract Toothpaste (*Syzygium aromaticum* L.)

Ingredient	F1 (gr)	F2 (gr)	F3 (gr)
Na CMC	1	0.5	0,25
Glycerin	12	12	12
$CaCO_3$	24.5	24.5	24.5
Clove flower extract	2.5	2.5	2.5
Metyl paraben	0.125	0.125	0.125
Xylitol	0.5	0.5	0.5
SLS	1	1	1
Aquadest	Add 50	Add 50	Add 50

Research Procedure

Na CMC was dispersed with hot water (mixture 1). Calcium carbonate and clove flower extract were crushed, plus glycerin was stirred homogeneously (mixture 2). Mixture 1 was added to mixture 2 and stirred until homogeneous (mixture 3). Methyl paraben and xylitol that had been dissolved in the remaining water were added to mixture 3, stirred homogeneously, then SLS was added and stirred until a paste mass was formed.

Evaluation of Physical Preparations

1. Organoleptical Test, Homogeneity, and pH Test

   For the organoleptical test, the color, aroma, and texture of the preparation were observed. Homogeneity is indicated by the absence of coarse grains in the preparation. The pH test is carried out using a pH meter. (Triananda & Wijaya, 2021).

2. Viscosity and Spreadability Test

   The good viscosity of toothpaste is 2000-50000 mPa.s (Syurgana et al., 2017) And the spreadability of a good preparation is between 5-7 cm. (Triananda & Wijaya, 2021).

3. Foam Formation Test

   The toothpaste foam formation test is performed by dissolving 1 gram of toothpaste with 15 ml of distilled water, then shaking 5 times and observing the height of the foam formed. There is no foam height requirement for a toothpaste product. (Syurgana et al., 2017); (Daud et al., 2016).

4. Physical Stability Test

   This test was conducted by storing samples in conditions designed to accelerate changes, namely in a room, a humid room, and a room exposed to direct sunlight. Organoleptical observations were made for 4 weeks, and pH measurements were taken after storing the preparation for 4 weeks. (Sayuti, 2015).

RESULTS

The results of the pH test evaluation of clove flower (*Syzygium aromaticum* L.) extract toothpaste aide are listed in Table 2.

TABLE 2. The results of the pH test evaluation of clove flower (*Syzygium aromaticum L.*) extract toothpaste

Formula	Test Result	x̄ ± SD	P-Value
K (+)	8.72 8.71 8.74	8.72±0.01	0.785
F1	8.78 8.68 8.81	8.75±0.06	
F2	8.76 8.78 8.70	8.74±0.04	
F3	8.69 8.85 8.77	8.77±0.08	

The results of the viscosity test evaluation of clove flower extract toothpaste (*Syzygium aromaticum L.)* can be seen in Table 3.

TABLE 3. The results of the viscosity test evaluation of clove flower (*Syzygium aromaticum L.*) extract toothpaste

Formula	Test Result	x̄ ± SD	P-Value
K (+)	9850 9570 9180	9533.33±336.50	0.023
F1	9850 9850 9100	9800.00±433,01	
F2	6590 6760 6360	6570.00±200.74	
F3	5910 5530 5950	5796.66±231.80	

The results of the spreadability test evaluation of clove flower *(Syzygium aromaticum* L.) extract toothpaste aide are listed in Table 4.

TABLE 4. The results of the spreadability test evaluation of clove flower (*Syzygium aromaticum* L.) extract toothpaste

Formula	Test Result	x̄ ± SD	P-Value
K (+)	5.1	4.13±0.05	0.021
	5.2		
	5.1		
F1	5.3	4.20±0.10	
	5.3		
	5.2		
F2	5.8	5.30±0.30	
	5.6		
	5.7		
F3	6.2	6.16±0.25	
	6.9		
	6.5		

The evaluation results of the clove flower extract (*Syzygium aromaticum* L.) toothpaste foam formation test can be seen in Table 5.

TABLE 5. The evaluation results of the clove flower extract (*Syzygium aromaticum* L.) toothpaste foam formation test

Formula	Test Result
K (+)	8
	8.5
	8
F1	7
	8
	8
F2	9.5
	9.5
	9
F3	11
	12
	12.5

DISCUSSION

Organoleptical Test

Based on the test results, it shows that formulations I, II, III of clove flower extract toothpaste are brown (beige) and have a distinctive smell of cloves, this is influenced by the active substances used because macroscopically the ethanol extract of clove flowers is concentrated brown with a distinctive smell of cloves derived from essential oil components which contain eugenol compounds, so that it can affect the color and smell of toothpaste preparations. The texture obtained shows different results in each formulation. This is due to the concentration of Na CMC, which is different in each formula, so that the higher the concentration of Na CMC used as a binder, the thicker the toothpaste preparation is produced. The positive control has a thick texture, orange brown color, and a distinctive smell of cloves.

Homogeneity Test

Formulations I, II, III, and positive control have a good and homogeneous composition with no coarse grains on the object glass because, in the process of mixing the preparation, good stirring is carried out. The stirring process is one of the factors to improve the homogeneity of a preparation. In addition, substances that are difficult to mix (methyl paraben and xylitol) are dissolved first using distilled water to minimize the risk of preparations that are difficult to mix. So this is in line with the research of Auna et al. (2017) that the indicator of a homogeneous toothpaste is when there are no coarse grains on the glass object.

pH Test

The average results obtained from pH testing on all toothpaste formulations fall within the standard pH range of toothpaste, namely, pH between 4.5-10.5 (Warnida et al., 2016). So, the pH of toothpaste from the three formulations between formulations I, II, and III, is eligible. The pH results obtained have alkaline properties because the pH of Na CMC is stable against Calcium Carbonate, which is the largest component in the toothpaste preparation. This is in line with Eko's research (2012) that Na CMC is stable in the pH range of 5.5 to 9.5, is stable to electrolytes and calcium ions, and is suitable for most toothpaste formulations. (Oliver, 2013).

Viscosity Test

The viscosity test results show numbers that are in the range of the viscosity standards specified. So it can be concluded that formulations I, II, and III meet the requirements of a good viscosity test on toothpaste, namely 2000-50000 mPa.s [24]. Based on Table 3, an increase in the concentration of Na CMC as a binder in the preparation can increase the viscosity of the paste preparation. This means that the more the concentration of Na CMC, the thicker the toothpaste preparation produced. This is in line with the research of Dyera et al. (2020) that increasing the concentration of Na CMC in the preparation can increase the viscosity of the preparation using a chain extension mechanism. The chain extension is by forming a gel matrix in toothpaste preparations. The Na CMC matrix is formed from the extension of the polymer chain; the more Na-CMC used, the tighter the matrix formed. (Forestryana et al., 2020)

Spreadability Test

The results of the spreadability test on formulations I, II, and III have met the requirements of the spreadability test. Increasing the concentration of Na CMC as a binder in the preparation can reduce the spreadability of toothpaste preparations because the resulting preparation becomes thicker. This is in line with the research of Dyera et al. (2020) that the higher the concentration of Na CMC, the more the spreadability will decrease. Spreadability is inversely proportional to viscosity, where the higher the viscosity, the spreadability will decrease. (Forestryana et al., 2020)

Foam Formation Evaluation

Based on the results of the foam formation evaluation, the three toothpaste formulas can form foam well. There is no foam height requirement for a toothpaste product. This is attributed to the aesthetic value that consumers like (Syurgana et al., 2017). The more foam produced, the more consumers will like it because it shows that the toothpaste has high cleaning power. In addition, it can also be associated with the ability of a surfactant to produce foam (Syurgana et al., 2017). In this study, SLS (Sodium Lauryl Sulfate) was used as a surfactant that produces good foam in each formulation. In line with the research of Marwah et al. (2017) that the foam produced from

a toothpaste preparation is generally influenced by the surfactant used. SLS is an anionic surfactant that has characteristics as a good foam-former and has high cleaning power (Syurgana et al., 2017).

Physical Stability Test

Based on the results of organoleptic observations of toothpaste preparations carried out for 4 weeks of storage in three different rooms, it shows that the toothpaste preparation has not changed in terms of texture, color, or odor of the preparation. This shows that the toothpaste is stable in storage on organoleptic parameters, because there is no physical change during the 4 weeks of storage, either in the room, a humid room, or a room exposed to direct sunlight. This contradicts the statement that the physical instability of toothpaste preparations is characterized by blanching or the appearance of color, odor, change, or phase separation, syneresis, change in consistency, gas formation, and other physical changes. (Sayuti, 2015).

CONCLUSION

In this study, it can be concluded that first, clove flower extract (*Syzgium aromaticum L.*) can be formulated into toothpaste preparations using variations in Na CMC concentration in formulation I (Na CMC 2%), formulation II (Na CMC 1%), and formulation III (Na CMC 0.5%). Second, the evaluation tests carried out include organoleptic, homogeneity, pH, viscosity, spreadability, foam formation, and physical stability tests of the preparation. Evaluation results that differ significantly due to variations in Na CMC concentration are in the viscosity and spreadability tests. It can be concluded that the greater the concentration of Na CMC, it will effect on the physical properties of toothpaste, which will cause an increase in the viscosity of toothpaste and a decrease in the spreadability of toothpaste. And finally, the results of the various formulation tests stated that all formulations met the requirements of the toothpaste evaluation test, both in formulation I (Na CMC 2%), formulation II (Na CMC 1%), and formulation III (Na CMC 0.5%).

ACKNOWLEDGEMENT

The author would like to thank all parties so that this research can be completed.

CONFLICT OF INTEREST

No conflict of interest

REFERENCES

1. Andries, J. R., Gunawan, P. N., & Supit, A. (2014). Uji Efek Anti Bakteri Ekstrak Bunga Cengkeh Terhadap Bakteri Streptococcus mutans Secara In Vitro. *E-GIGI*, *2*(2). https://doi.org/10.35790/eg.2.2.2014.5763
2. Daud, N. S., Desi, S. A., & Ifaya, M. (2016). Formulasi Pasta Gigi Infusa Daun Jambu Biji (Psidium guajavaLinn.) Dengan Variasi Konsentrasi Na. CMC Sebagai Bahan Pengikat. *Jurnal Ilmiah Ibnu Sina*, *1*(1), 42–49.
3. Forestryana, D., Surur Fahmi, M., & Novyra Putri, A. (2020). Pengaruh Jenis dan Konsentrasi Gelling Agent pada Karakteristik Formula Gel Antiseptik Ekstrak Etanol 70% Kulit Buah Pisang Ambon. *Lumbung Farmasi: Jurnal Ilmu Kefarmasian*, *1*(2), 45. https://doi.org/10.31764/lf.v1i2.2303
4. Ilmiah, P., Qomariah, R. I. A. L., Farmasi, P. S., Farmasi, F., & Surakarta, U. M. (2017). *Formulasi*

pasta gigi ekstrak etanol bunga turi (.
5. Indonesia, D. K. R. (2000). No Title. *Farmakope Indonesia Edisi IV*.
6. Makanan, B. P. O. dan. (2021). No Title. *Pedoman Teknis Evaluasi Pasta Gigi*.
7. Oliver, J. (2013). No Title. *Perbedaan Penggunaan Bahan Pengikat Na CMC Dan HPMC Terhadap Sifat Fisik, Kimia Dan Uji Hedonik Sediaan Pasta Gigi Enzim Papain Pepaya (Carica Papaya L.)*, *53*(9), 1689–1699.
8. Sayuti, N. A. (2015). Formulasi dan Uji Stabilitas Fisik Sediaan Gel Ekstrak Daun Ketepeng Cina (Cassia alata L.). *Jurnal Kefarmasian Indonesia*, *5*(2), 74–82. https://doi.org/10.22435/jki.v5i2.4401.74-82
9. Susilowati, E. P., & Wahyuningsih, S. S. (2014). Optimasi Sediaan Salep yang Mengandung Eugenol dari Isolasi Minyak Cengkeh (Eugenia Caryophylatta Thunb.). *IJMS-Indonesian Journal On Medical Science*, *1*(2), 29–34.
10. Syurgana, M. U., Febriana, L., & Ramadhan, A. M. (2017). Proceedings of the 6th Mulawarman Pharmaceuticals Conferences FORMULASI PASTA GIGI DARI LIMBAH CANGKANG TELUR BEBEK. *Mulawarman Pharmaceuticals Conferences*, *November* 7–8. https://doi.org/10.25026/mpc.v6i1.275
11. Triananda, A. L., & Wijaya, A. (2021). *Formulasi Dan Uji Fisik Sediaan Gel Ekstrak Daun Petai Cina (Leucaena leucocephala (Lam .) De . Wit) Dengan Basis Hydroxy Propyl Methyl Cellulose (HPMC) Formulation And Physical Properties Test Of Petai Cina (Leucaena leucocephala (Lam .) De. Wit* . 29–36.
12. Warnida, H., Juliannor, A., & Sukawaty, Y. (2016). Formulation of Bawang Dayak (Eleutherine bulbosa (Mill.) Urb.) Extract into a Gel Toothpaste. *Jurnal Sains Farmasi & Klinis*, *3*(1), 42–49. http://jsfkonline.org/index.php/jsfk/article/view/98
13. Winarno, F. G. (2004). No Title. *Kimia Pangan Dan Gizi*.