

EFFECT OF CMC-NA CONCENTRATION VARIATION AS A THICKENING AGENT IN SHAMPOO FORMULATION CONTAINING GRAPESEED OIL (*VITIS VINIFERA L.*)

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Abstract

Grapeseed oil (*Vitis vinifera L.*) is a natural ingredient rich in antioxidants and essential fatty acids that has potential applications in hair care products. This study aimed to determine the effect of sodium carboxymethyl cellulose (CMC-Na) concentration on the physical characteristics of grapeseed oil shampoo and to identify the optimum formulation. Three shampoo formulations containing 10% grapeseed oil were prepared with different CMC-Na concentrations, namely 1% (F1), 3% (F2), and 5% (F3). The formulations were evaluated for organoleptic properties, homogeneity, pH, foam height, and viscosity. All formulations produced homogeneous shampoos with acceptable appearance and foam stability, while the pH values ranged from 6.30 to 6.41 and complied with the Indonesian National Standard (SNI) requirements for shampoo preparations. Increasing the CMC-Na concentration significantly increased the viscosity of the shampoo. Among the tested formulations, F1 containing 1% CMC-Na exhibited viscosity within the recommended range while maintaining satisfactory physical characteristics, whereas F2 and F3 produced excessively high viscosity values. Therefore, the formulation containing 1% CMC-Na was considered the optimum formulation for grapeseed oil shampoo.

Keywords: Grape Seed Oil , shampoo, viscosity

1. INTRODUCTION

Shampoo is a detergent-based preparation designed and packaged for cleansing hair. It functions to remove dirt, excess oil, and impurities from the scalp and hair without adversely affecting the natural condition and health of the hair. As a result, the hair becomes clean, fragrant, shiny, smooth, and easy to manage [15].

Grapes (*Vitis vinifera L.*), commonly known as grapes, are not native to Indonesia. They originated in the Middle East, particularly in Mesopotamia (present-day Iraq). The initial spread of grapes worldwide occurred primarily through processed products such as wine rather than fresh fruit) [14]. Grapeseed oil is obtained from grape seeds and is rich in unsaturated fatty acids, particularly linoleic acid, as well as vitamin E [11].

Based on phytochemical screening, grapeseed oil contains alkaloids, flavonoids, tannins, terpenoids, and saponins. It also contains approximately 75% linoleic acid, 15% oleic acid, 6% palmitic acid, 3% stearic acid, and 1% linolenic acid. In addition, grapeseed oil is a rich source of vitamin E and possesses high antioxidant activity, which contributes to its stability[11].

Research involving variations in the concentration of gelling agents is important because these substances can influence the physical characteristics and stability of shampoo formulations. Excessive concentrations of gelling agents may produce shampoos with overly high viscosity, making them difficult to dispense from containers. CMC-Na (Sodium Carboxymethyl Cellulose) is a cellulose derivative widely used in industry as a thickener, emulsion stabilizer, and suspension stabilizer. As a thickening agent, CMC-Na forms a colloidal dispersion system and increases viscosity. The presence of CMC-Na helps

suspended particles remain uniformly distributed within the system, thereby preventing sedimentation due to gravitational forces. Thickening agents are substances used to increase the viscosity of a formulation. The viscosity and flow characteristics of shampoo can be determined through viscosity measurements [9].

Viscosity is an important parameter in shampoo formulation. CMC-Na is commonly used in pharmaceutical formulations, both oral and topical, due to its viscosity-enhancing properties [12]. Furthermore, CMC-Na is highly effective in aqueous systems with high water content and can be easily combined with both water-soluble ingredients and oil components [9]. Therefore, CMC-Na has the potential to serve as an effective thickening agent for increasing the viscosity of shampoo formulations.

Based on the background described above, this study was conducted to determine the effect of varying concentrations of CMC-Na as a thickening agent on the viscosity of grapeseed oil shampoo. The evaluation included pH testing, foam height testing, homogeneity testing, and organoleptic assessment to identify the most effective concentration of CMC-Na capable of producing a shampoo formulation that meets the required quality standards. The concentrations of CMC-Na investigated in this study were 1%, 3%, and 5%.

2. METHODOLOGY

The stages of this research included the preparation of grapeseed oil extract, formulation of grapeseed oil shampoo, and evaluation of the physical and chemical properties, hedonic characteristics, and qualitative antioxidant activity of the shampoo preparation.

2.1 Plant Determination

The grape seeds (*Vitis vinifera* L.) used in this study were identified at the Indonesian Research Institute for Medicinal and Aromatic Plants (BALITRO), Bogor, West Java. Identification was carried out using morphological characteristics and taxonomic references to ensure accurate classification of the plant material.

2.2 Preparation of Grapeseed Oil Extract

A total of 200 g of grape seed powder was placed into a clean glass bottle. Ninety-six percent ethanol was added until the powder was completely immersed, with approximately 1–2 cm of solvent remaining above the powder surface (approximately 1 L of ethanol). The bottle was tightly sealed and stored in a dark place for 24–48 hours. The mixture was stirred periodically to maximize extraction efficiency.

After the maceration process, the extract was filtered until a clear yellowish-green filtrate was obtained. The filtrate was then transferred to a wide evaporating dish and concentrated using a water bath maintained at a temperature of 40–50°C. The solution was heated gently without boiling to prevent degradation of the oil components. Ethanol was allowed to evaporate completely until a concentrated, clear oil-like extract was obtained. The final extract was transferred using a pipette into a pre-weighed container and adjusted to a final weight of 20 g.

2.3 Preparation of Grapeseed Oil Shampoo

The shampoo formulation was prepared by first preparing all equipment and accurately weighing the required ingredients according to the formulation. CMC-Na was dispersed in hot water and mixed until a homogeneous gel was formed. Grapeseed oil extract and menthol solution were then added and mixed thoroughly.

Phenoxyethanol and Cocamide DEA were subsequently incorporated while stirring continuously until homogeneous. In a separate container, Cocamidopropyl Betaine was mixed with Sodium Lauryl Sulfate solution and then added gradually to the main mixture with continuous stirring. Fragrance was added in a sufficient quantity, and the mixture was stirred until uniform. The finished shampoo preparation was then transferred into suitable containers and appropriately packaged.

Table 1. Formulation of Grapeseed Oil Shampoo Preparations

Ingredient	Concentration (%)			Function
	F1	F2	F3	
Grapeseed Oil	10	10	10	Active ingredient
Sodium Lauryl Sulfate	10	10	10	Surfactant
Cocamide DEA	3	3	3	Foaming agent
Sodium Carboxymethyl Cellulose (CMC-Na)	1	3	5	Thickening agent
Cocamidopropyl Betaine	3	3	3	Emollient
Menthol	0,2	0,2	0,2	Cooling agent
Phenoxyethanol	0,5	0,5	0,5	Preservative
Fragrance	Qs	Qs	qs	Fragrance agent
Purified Water (Aquadest)	ad100	ad100	ad100	Solvent

2.4 Evaluation of Shampo Formulation

2.4.1 Organoleptic Evaluation

The organoleptic evaluation was carried out by observing the physical characteristics of the shampoo preparations, including texture, appearance, odor, and color.

2.4.2 Homogeneity Test

The homogeneity test was conducted to determine the presence or absence of coarse particles and to assess the physical uniformity of the shampoo preparations. A small amount of shampoo from each formulation was spread onto a watch glass and visually examined. The preparation was considered homogeneous if it exhibited a uniform appearance without any visible coarse particles [9].

2.4.3 Viscosity Test

The viscosity of the shampoo formulations was measured using a Brookfield Viscometer (DV-E model). Approximately 200 mL of shampoo sample was transferred into a beaker glass and placed beneath the viscometer. An appropriate spindle was selected according to the viscosity of the formulation and immersed into the sample until fully submerged. The viscosity measurement was then performed at predetermined rotational speeds, and the results were recorded in centipoise (cPs) [12].

2.4.4 Foam Height Test

The foam height was measured using a simple method. One gram of shampoo preparation was placed into a graduated test tube containing 10 mL of distilled water and then tightly capped. The mixture was shaken for 20 seconds, and the height of the foam formed was measured and recorded [10].

2.4.5 Acidity (pH) Test

The pH test was performed using universal pH indicator paper. One gram of shampoo sample was dissolved in 100 mL of distilled water and allowed to stand for several minutes. The pH paper was then immersed in the solution, removed, and the resulting color was compared with the standard color scale provided by the manufacturer. According to the Indonesian National Standard [6], the acceptable pH range for shampoo preparations is 4.0–8.0.

3. RESULTS

3.1. Results of Grape Plant Identification

The determination of the grape (*Vitis vinifera*) specimens was conducted at the Indonesian Research Institute for Medicinal and Aromatic Plants (BALITRO), Bogor, West Java. Based on morphological and taxonomic examination, the plant material used in this study was confirmed as *Vitis vinifera* L.

3.2 Results of Grapeseed Oil Extraction

The grapeseed oil used in this study was obtained from the Indonesian Research Institute for Medicinal and Aromatic Plants (BALITRO), Bogor, West Java. Phytochemical screening results indicated that the grapeseed extract contained several bioactive compounds, including alkaloids, flavonoids, tannins, terpenoids, and saponins. These secondary metabolites are known to possess various biological activities, including antioxidant properties and potential benefits for hair and scalp health.

3.3. Organoleptic Evaluation Results of Grapeseed Oil Shampoo

3.3.1 .Organoleptic Evaluation

The organoleptic evaluation of grapeseed oil shampoo formulations was conducted by observing the color, odor, appearance, and texture of each formulation

Table 2 Organoleptic Evaluation Results of Grapeseed Oil Shampoo Formulations

Physical Parameter	Color	Odor	Appearance	Texture
F1	Yellowish White	Grape Aroma	Slightly Gel-like	Moderately Viscous
F2	Yellowish White	Grape Aroma	Gel	Viscous
F3	Yellowish White	Grape Aroma	Gel	Viscous



Organoleptic Evaluation Results of Grapeseed Oil Shampoo Formulations

3.3.2. Homogeneity Test

The homogeneity test was conducted to evaluate the uniformity of the shampoo preparations and to determine the presence or absence of coarse particles. The results of the homogeneity test for grapeseed oil shampoo formulation.

Table 3 Homogeneity Test Results of Grapeseed Oil Shampoo Formulations

Formula	Homogeneity
F1	Homogeneous, no coarse particles observed
F2	Homogeneous, no coarse particles observed
F3	Homogeneous, no coarse particles observed



Homogeneity Evaluation Results of Grapeseed Oil Shampoo Formulations

3.3.3 Viscosity Test

The viscosity test was performed using a Brookfield viscometer to evaluate the effect of different concentrations of CMC-Na on the viscosity of the shampoo formulation.

Table 4. Viscosity Test Results of Grapeseed Oil Shampoo Formulations

Speed (rpm)	Formulation			Standard Requirement
	F 1 (cPs)	F 2 (cPs)	F 3 (cPs)	
2,5	4.000	16.000	36.000	400 – 4.000 Schmitt dan William (1996)
4	3.500	13.750	35.750	
5	3.400	15.000	34 .000	
10	2.600	8.500	24 .000	
20	2.100	6.000	13 .000	

The viscosity test demonstrated that increasing the concentration of CMC-Na resulted in a significant increase in viscosity. F1, containing 1% CMC-Na, exhibited viscosity values ranging from 2,100 to 4,000 cPs, which complied with the standard viscosity range for shampoo preparations [12]. In contrast, F2 and F3 showed substantially higher viscosity values, exceeding the recommended range. This increase can be attributed to the greater thickening effect of CMC-Na at higher concentrations, which enhanced the internal resistance of the formulation and produced a thicker gel-like consistency.

These results indicate that F1 provided the most appropriate viscosity characteristics for shampoo preparations, while higher concentrations of CMC-Na produced excessively viscous formulations that may reduce ease of use and product dispensing.

3.3.4 Foam Height Test

The foam height test was conducted to evaluate the foaming ability of the shampoo formulations. Foam formation is an important characteristic of shampoo products because it influences consumer acceptance and reflects the cleansing performance of the formulation.

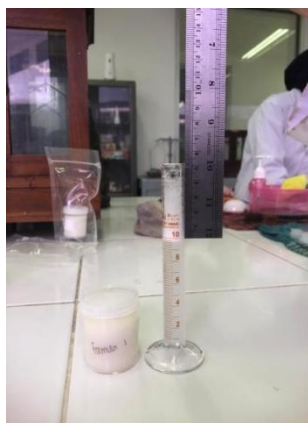
Table 5. Foam Height Test Results of Grapeseed Oil Shampoo Formulations

Parameter	Formulations			Standard Requirement
	1	2	3	
Foam Height	5 cm	4 cm	5 cm	1,3 – 22 cm (Wilkinson 1982)

F1

F2

F3



Photographs of the Foam Height Test of Grapeseed Oil Shampoo Formulations

3.3.4 Acidity (pH) Test

The pH test was conducted to determine whether the shampoo formulations met the acceptable pH range for shampoo preparations according to the Indonesian National Standard [6].

Table 4. pH Test Results of Grapeseed Oil Shampoo Formulations

Parameter	Formulation			Standard Requirement (SNI 8860:2020)
	1	2	3	
Ph	6,30	6,36	6,41	4,0 – 8,0



F1

F2

F3

Photographs of the pH Evaluation of Grapeseed Oil Shampoo Formulations

The pH values ranged from 6.30 to 6.41 and complied with the Indonesian National Standard (SNI 8860:2020), which specifies an acceptable pH range of 4.0–8.0 for shampoo formulations [6]. These results indicate that the formulations are safe for scalp application and are unlikely to cause irritation.

4. CONCLUSIONS

The concentration of CMC-Na significantly affected the viscosity of grapeseed oil shampoo formulations, with higher concentrations producing higher viscosity values. All formulations met the required physical quality parameters, including organoleptic properties, homogeneity, pH, and foam height. Among the tested formulations, Formula 1 containing 1% CMC-Na exhibited the most favorable characteristics and was identified as the optimum formulation

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REFERENCES

- [1] G. Agoes, *Sediaan Kosmetik (SFI-9)*. Bandung: Institut Teknologi Bandung, 2015, pp. 273–284.
- [2] B. Anam and M. A. Putri, *Inovasi Pembuatan Shampo dari Ekstrak Seledri dengan Metode Ultrasonic Extraction–Microwave Distillation (USE-MD)*. Surabaya: Fakultas Vokasi, Institut Teknologi Sepuluh Nopember, 2017.
- [3] Badan Pengawasan Obat dan Makanan Republik Indonesia, *Pengertian Kosmetik, Bab 1*. Jakarta: BPOM, 2004.
- [4] P. Coniwanti, M. Dani, and Z. S. Daulay, “Pembuatan Natrium Karboksimetil Selulosa (Na-CMC) dari Selulosa Limbah Kulit Kacang Tanah (*Arachis hypogaea* L.),” *Jurnal Teknik Kimia*, vol. 21, no. 4, 2015.
- [5] T. L. Da Silva, E. C. Bernardo, B. Nobre, R. L. Mendes, and A. Reis, “Extraction of Victoria and Red Globe Grapeseed Oil Using Supercritical Carbon Dioxide with and without Ethanol,” *Journal of Food Lipids*, vol. 15, pp. 356–357, 2008.
- [6] Departemen Kesehatan Republik Indonesia, *Farmakope Indonesia Edisi VI*. Jakarta: Direktorat Jenderal Pengawasan Obat dan Makanan, 2020.
- [7] S. Fajriati, *Formulasi Sediaan Sampo dari Ekstrak Wortel (Daucus carota Linn.)*. Jakarta: Akademi Farmasi Hang Tuah, 2013.

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- [8] H. A. Gea, *Formulasi Sediaan Shampo dari Ekstrak Etanol Daun Bandotan (Ageratum conyzoides L.)*. Medan: Institut Kesehatan Helvetia, 2018.
- [9] M. Karsheva, S. Georgieva, and S. Handjieva, "The Choice of the Thickener – A Way to Improve the Cosmetic Sensory Properties," University of Chemical Technology and Metallurgy, Bulgaria, 2006.
- [10] F. C. C. Korompis, P. V. Y. Yamlean, and W. A. Lolo, "Formulasi dan Uji Efektivitas Antibakteri Sediaan Sabun Cair Ekstrak Etanol Daun Kersen (*Muntingia calabura L.*) terhadap *Staphylococcus epidermidis*," *Jurnal Ilmiah Farmasi*, vol. 9, no. 1, p. 33, 2020.
- [11] M. U. Maheswari and P. G. M. Rao, "Antihepatotoxic Effect of Grape Seed Oil in Rat," *Indian Journal of Pharmacology*, vol. 37, no. 3, p. 179, 2005.
- [12] D. Rashati and M. C. Eryani, "Evaluasi Sifat Fisik Sediaan Sampo Ekstrak Daun Katuk (*Sauropus androgynus (L.) Merr.*) dengan Berbagai Variasi Viscosity Agent," *Jurnal Riset Kefarmasian Indonesia*, vol. 1, no. 1, 2019.
- [13] R. I. Tranggono and F. Latifah, *Buku Pegangan Ilmu Pengetahuan Kosmetik*, 1st ed. Jakarta: Gramedia Pustaka Utama, 2007.
- [14] B. T. W. Wiryanta, *Membuahkan Anggur di Dalam Pot dan Pekarangan*. Jakarta: Agro Media Pustaka, 2004.
- [15] S. Yuniarti, "Formulasi Shampo Anti Ketombe dari Daun Teh (*Camellia sinensis L.*)," Undergraduate Thesis, Universitas Muhammadiyah Purwokerto, 2013.