FORMULATION AND PHYSICAL CHARACTERISTICS OF VITAMIN C EMULGEL

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Abstract.

Vitamin C (ascorbic acid) acts as a potent antioxidant, capable of neutralizing the harmful effects of free radicals. This compound is used in both oral and topical preparations; however, topical formulations are generally preferred for dermatological applications. This study aims to formulate a vitamin C emulgel using various concentrations of carrageenan as a gelling agent. Emulgels are designed to address the limitations of gels, as gel formulations often allow for faster drug release compared to conventional ointments or creams.

The formulation process began with the stepwise incorporation of carrageenan as a gelling agent, with concentrations ranging from 0.5% (F1), 0.75% (F2), 0.85% (F3), 0.95% (F4), 1% (F5), 1.125% (F6), 1.25% (F7), 1.5% (F8), up to 2% (F9). Each formula maintained the same composition of other ingredients: Tween 20 and Span 20 as emulsifiers (1% and 1.5%, respectively), liquid paraffin as the oil phase (7.5%), propylene glycol as a humectant (10%), and propyl and methyl parabens as antimicrobial agents (0.01% and 0.03%, respectively).

The formulations were evaluated for physical properties, including pH, spreadability, viscosity, freeze-thaw stability, and general stability, with the goal of identifying the optimal carrageenan concentration that provides the best stability. Among the variations, the emulgel with 1% carrageenan

demonstrated the most favorable and stable physical properties, making it the optimal formulation for a vitamin C emulgel.

Keywords: Physical Caracteristic, Emulgels,Vitamin C

Introduction

Vitamin C is essential for maintaining collagen structure, a protein that connects various fibrous tissues, including skin, tendons, cartilage, and other vital tissues in the human body. A strong collagen structure contributes to the healing of bone fractures, bruises, bleeding, and minor wounds (Naidu, 2003). Additionally, Vitamin C plays a crucial role in iron absorption and mental alertness (Davies, 1991). As a powerful antioxidant, it neutralizes free radicals throughout the body (Kim Do, 2002) and has a mild laxative effect, aiding in the excretion of waste (Naidu, 2003). It also combats carcinogenic nitrites, with research from the Massachusetts Institute of Technology showing an 81% reduction in nitrosamine formation among students given Vitamin C (Naidu, 2003).

Deficiency in Vitamin C, or hypoascorbemia, can lead to issues such as cracked tongue, rough skin, unhealthy gums, fatigue, muscle weakness, and even mental health impacts like depression. This deficiency has also been linked to health concerns, including high cholesterol, heart disease, arthritis, and respiratory infections.

Topical formulations of Vitamin C are often preferred for local effects in skincare and cosmeceuticals. However, skin permeability remains a primary challenge in ensuring active ingredients penetrate effectively. The outermost skin layer, the stratum corneum, presents a barrier to many medicinal substances, especially those that are not easily absorbed (Shashi, 2012). Drug delivery innovations, such as emulgels—which combine emulsions with gels—have improved the bioavailability of certain drugs by enhancing skin absorption, particularly for fat-soluble molecules (Shashi, 2012). Emulgels also allow for the inclusion of penetration enhancers, further boosting drug permeability through the skin (Raut SV, 2014). Factors such as the active ingredient properties, formulation, and patient-specific variables—such as skin thickness, pH, injury state, and age—also significantly influence drug penetration and absorption.

Vitamin C emulgels are increasingly popular in cosmeceutical preparations for their ease of production, stability, and aesthetic appeal. Emulgels offer high stability and effectiveness by integrating a gelling agent that improves emulsion formulation stability, resulting in a product with both elegance and efficacy. Physical and chemical properties—such as organoleptic characteristics, pH, viscosity, spreadability, adhesion, and stability—are used to evaluate and optimize these emulgel formulations (Garg et al., 2017; Kumar & Utreja, 2019).

This study aims to develop an optimal Vitamin C emulgel formulation that meets the specified physicochemical properties, providing both stability and high efficacy.

Methodology

A. Tools and Materials

All functions are equipped as follows: Analyzers, Micropipette, Magnetic Stirrer, Homogenizer, Water Bath, pH Meter, Bookfield Viscometer, Daily Food Processor, Food Cleaner, Oven, Food. Ingredients in this formula include: Vitamin C (Brataco Chemical), Span 20 (Brataco Chemical), Tween 20 (Brataco Chemical), Liquid Paraffin (Brataco Chemical), Propylene Glycol (Brataco Chemical), Methyl and Propyl Paraben (Brataco Chemical), Triethanolamine (Brataco Chemical), Carrageenan, Aquamineralisata (Brataco Chemical).

B. Method

Emulgel base orientation

5% (F1), 0.75% (F2), 0.85% (F3), 0.95% (F4), 1% (F5), 1.125% (F6), 1.25% (F7), 1.5% (F8) and 2% (F9). Composite formulas included in Table 1.

Tabel 1. Composition of Emulgels Base Orientation

Ingredients	Concentrations (% w/w)								
	F1	F2	F3	F4	F5	F6	F7	F8	F9
Carrageenan	0.5	0.75	0.85	0.95	1	1.125	1.25	1.5	2
Liquid Paraffin	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.5
Tween 20	1	1	1	1	1	1	1	1	1
Span 20	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Propylene Glycol	10	10	10	10	10	10	10	10	10
Nipagin	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03
Nipasol	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
TEA	2	2	2	2	2	2	2	2	2
Aqua ad.	100	100	100	100	100	100	100	100	100

Making Vitamin C Emulgel

Prepare emulgel by dispersing carrageenan in distilled water (75 °C) with continuous stirring at medium speed with a mechanical stirrer and adjust the pH to 5.5-6.5 with triethanolamine (TEA). The oil phase is prepared by dissolving Span 20 in liquid paraffin. The water phase was prepared by dissolving Tween 20 in distilled water. 0.03 g of methylparaben and 0.01 g of propylparaben dissolved in 10 g of propylene glycol and vitamin C mixed with the water phase. The oil and water phases are heated separately to 70-80 °C. Then the oil phase was added to the water phase with continuous stirring until it cooled to room temperature. The emulsion is poured into the gel with gentle stirring until a homogeneous emulsion is formed (V. Naga Sravan et al, 2014)

Methylparaben and other preservatives are greatly reduced in the presence of non-ionic surfactants due to micellization. However, propylene glycol (10%) has been shown to enhance the antimicrobial activity in the presence of non-ionic surfactants (Rowe, 2006).

Emulgel Evaluation

Physical parameters of prepared formulations: All prepared formulations were visually checked for color, appearance, homogeneity, phase separation and freeze-thaw test.

Determination of pH

pH measurements were performed using a digital pH meter (Mettler Toledo). The gel (1 g) was dissolved in 25 ml of distilled water and the electrode was then immersed in the gel formulation until a constant reading was observed. Determination of pH values for each formulation was done in three replicates (V. Naga Sravan et al., 2014).

Determination of Viscosity

The viscosity of each formulation was determined at ambient temperature using a Brookfield digital viscometer with spindle no. 5 at 50 rpm (V. Naga Sravan et al, 2014).

Spreadability test

Weigh (350 mg) of Emulgel on a glass plate (10 x 5 cm). Another glass plate (10 x 5 cm and 5.8 ± 1 g) was dropped from a distance of 5 cm. The diameter of the spreading circle was measured after 1 minute (V. Naga Sravan et al., 2014). The gel types based on spreading are given in Table 2.

Tabel 2. Jenis Gel Berdasarkan Penyebaran (Dignesh, 2012)

Jenis Gel	Pengukuran (cm)
Gel cair	Lebih dari 2,4
Gel semi-cair	1.9-2.4
Gel semi kaku	1.9-1.6
Gel kaku	1.6-1.4
Gel sangat kaku	Kurang dari 1,4

Results and Discussion

A. Results

Emulgel-based orientation results

Based on the results in Table 3 and Table 4. Formulas F5, F6 and F7 show better results in consistency, phase separation and freeze-thaw tests compared to formulas F1, F2, F3, F4, F8 and F9, only formulas F5, F6 and F7 are characterized by easy spreading conditions and no phase separation after freeze-thaw test, while F8 and F9 show no phase separation but have harder consistency and are very stiff.

Table 3 Emulgel-based orientation results

Formulas	Color	Odor	Consistency	Phase Separation
F1	White	Odorless	Thinner	Separated
F2	White	Odorless	Thinner	Separated
F3	White	Odorless	Thinner	Separated
F4	White	Odorless	Thinner	Separated
F5	White	Odorless	Viscous, easy to spread	None
F6	White	Odorless	Viscous, easy to spread	None
F7	White	Odorless	Viscous, easy to spread	None
F8	White	Odorless	Harder	None
F9	White	Odorless	Harder	None

Table 4 Freeze Thaw result

Formulas	Phase separation at the cycling-					
	1	2	3	4	5	6
F1	(-)	(+)	(+)	(+)	(+)	(+)
F2	(-)	(+)	(+)	(+)	(+)	(+)
F3	(-)	(-)	(+)	(+)	(+)	(+)
F4	(-)	(-)	(+)	(+)	(+)	(+)
F5	(-)	(-)	(-)	(-)	(-)	(-)
F6	(-)	(-)	(-)	(-)	(-)	(-)
F7	(-)	(-)	(-)	(-)	(-)	(-)
F8	(-)	(-)	(-)	(-)	(-)	(-)
F9	(-)	(-)	(-)	(-)	(-)	(-)

The freeze-thaw test for the vitamin E emulgel formulation shows that F5, F6 and F7 have good stability at a concentration of 5%. All formulations are shown in Table 5.

Table 5 Formulasi Emulgel Vitamin C

Component	Concentration (% w/w)		
	F5	F6	F7
Carrageenan	1	1.125	1.25
Vitamin C	2	2	2
Liquid Paraffin	7.5	7.5	7.5
Tween 20	1	1	1
Span 20	1.5	1.5	1.5
Propylene glycol	10	10	10
Metyl Paraben	0.03	0.03	0.03
Propyl Paraben	0.01	0.01	0.01
TEA	2	2	2
Aqua ad.	100	100	100

Physical stability of Vitamin C Emulgel

Table 6 and Figure 1 show the physical properties of Emulgel in F5, F6 and F7. The research results show that F5 has a better formula based on parameters such as pH measurement and dispersion test. F5 continues the stability test and Figure 2 shows the stability study data of F5 formula.

Table 6. Physical Characteristics of Vitamin C Emulgel

Formulation Code	Organoleptic characteristics	Time Storage (days)				
		0	7	14	21	28
F5	Phase separation	No	No	No	No	No
	Color	White	White	White	White	White
	Odor	Odorless	Odorless	Odorless	Odorless	Odorless
	Texture	Smooth	Smooth	Smooth	Smooth	Smooth
	Consistency	Viscous	Viscous	Viscous	Viscous	Viscous
	Homogeneity	Homogenous	Homogenous	Homogenous	Homogenous	Homogenous
F6	Phase separation	No	No	No	No	No
	Color	White	White	White	White	White
	Odor	Odorless	Odorless	Odorless	Odorless	Odorless
	Texture	Smooth	Smooth	Smooth	Smooth	Smooth
	Consistency	Viscous	Viscous	Viscous	Viscous	Viscous
	Homogeneity	Homogenous	Homogenous	Homogenous	Homogenous	Homogenous
F7	Phase separation	No	No	No	No	No
	Color	White	White	White	White	White
	Odor	Odorless	Odorless	Odorless	Odorless	Odorless
	Texture	Smooth	Smooth	Smooth	Smooth	Smooth
	Consistency	Viscous	Viscous	Viscous	Viscous	Viscous
	Homogeneity	Homogenous	Homogenous	Homogenous	Homogenou	Homogenous

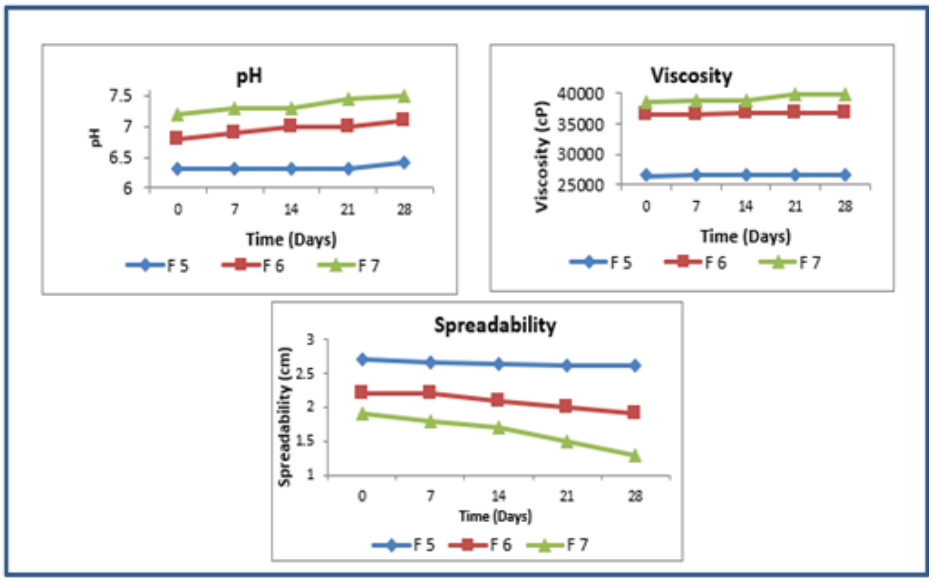

Figure 1. Test Results for 4 weeks of Emulgel Vitamin C Formula F5, F6 and F7

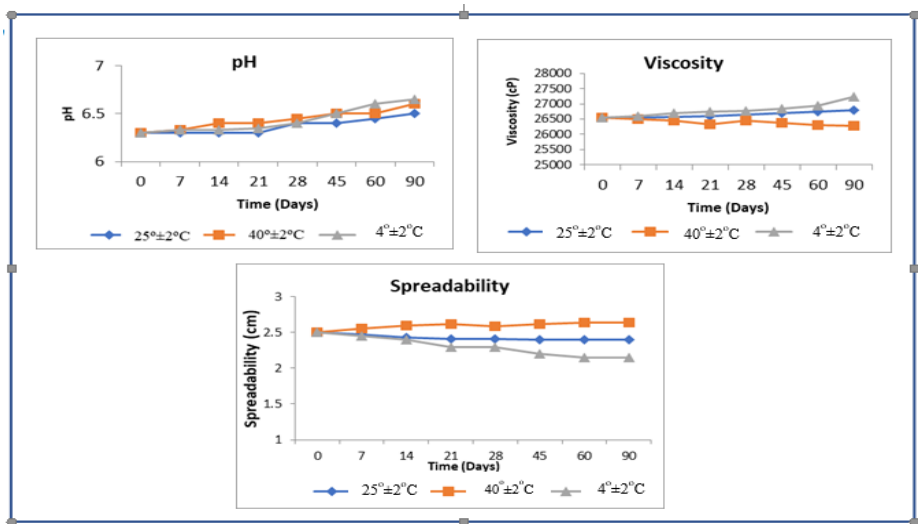

Figure 5. Stability study of promising F5 formula during storage time at temperature (25oC±2oC), at hot temperature (40oC±2oC), and at cold temperature (4oC±2oC)

B. Emulgel Base Alignment Results

Nine emulgel formulations using carrageenan as gelling agent were prepared as shown in Table 1. During preliminary tests, it was found that when more than 1.25% carrageenan was used, the gel base obtained was so thick that its use was not viable. Similarly, at concentrations below 1%, viscosity is very poor. Therefore, carrageenan is used in F5-F7 formulations in the range of 1-1.25%. The results of the

various physical parameters evaluated are shown in Tables 3 and 4. F1-F4 formulation is liquid due to the low carrageenan concentration. F8-F9 formulation is thick due to the higher carrageenan concentration. F5-F7 formulations had a creamy appearance and no phase separation was observed in the centrifugation test with freeze-thaw test. Based on the results of measuring viscosity, spreading and pH of F5-F7 formula, it was found that F5 formula is the best for vitamin C emulsifying gel formulations based on the pH measurement parameters of F5 which has a pH between 6 and 6.5, while F6 and F7 have a pH above 6.8 which is slightly above the safe level for topical skin needs which is in the range of 4.5–6.8. For the spreading test, F5 was included in the liquid gel category and F6 and F7 formulations with higher carrageenan concentrations were included in the semi-liquid gel and semi-rigid gel category for rigid gels respectively. As the concentration of gelling agent in the formulation increases, the spreadability of the formulation decreases. The results are shown in Figure 2. Accelerated stability studies were conducted for F5 formulation for three months. The samples were analyzed for their appearance, viscosity, pH and spreading ability for 0, 7, 14, 28, 45, 60 and 90 days.

Results of physical appearance determination, the results of organoleptic observations show in Table 6 that the emulgel did not show any changes in smell and color during the storage period.

Results of viscosity determination, the results of viscosity measurements during the storage period are shown in Figure 5. The viscosity values when stored at room temperature are more stable than when stored at high temperatures in the climatic chamber and at cold temperatures. This is in line with the Arrhenius kinetic equation, in which the viscosity is inversely proportional to the temperature. The higher the temperature, the lower the viscosity value. In addition to the temperature, the storage time also affects the stability of the dosage form. The longer the preparation is stored in the climatic chamber, the lower the viscosity value becomes due to the influence of high humidity, so that the preparation absorbs water vapor and causes an increase in the volume of the preparation.

Results of pH measurement, the pH value of the manufactured Emulgel preparation is still at a safe level for topical skin needs and is in the range of 4.5–6.8. The measured values are shown in Figure 5, which shows the suitability of the Emulgel for topical application.

Results of distribution measurement, the results of measuring the spreadability during storage time are shown in Figure 5. During spreading, it was observed that the spreading force decreased with increasing viscosity. The spreading value is more stable when stored at room temperature than when stored at high temperatures in the climate chamber and at cold temperatures.

Conclusion

Based on the formulation results of vitamin C emulgel with different concentrations of carrageenan as a gelling agent using concentrations ranging from 5% (F1), 0.75% (F2), 0.85% (F3), 0.95% (F4), 1% (F5), 1.125% (F6), 1.25% (F7), 1.5% (F8) and 2% (F9). From the preliminary investigation, three formulas were obtained which met the requirements of physical properties, namely, formula F5 with a carrageenan concentration of 1%, formula F6 with a carrageenan concentration of 1.125% and formula F6, namely a formula with a carrageenan concentration of 1.125%. Subsequently, a new physical evaluation of these three formulas was carried out, namely pH, viscosity and spreadability, resulting in the F5 formula providing results that are in the range that still meets the requirements for emulgel preparations. Formula F5 is a formula that contains carrageenan at a concentration of 1%.

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