Formulation and Characterization of Cosmetic Serum Containing Argan Oil as Moisturizing Agent

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Abstract: Nowadays, cosmetics are becoming more high demand in daily life and it was used frequently by many of people per year. Moisturizing serum is a bland of oleaginous substances that are applied to the skin by rubbing which used to replace natural skin oil, to cover tiny fissures in the skin and to provide a soothing protective film. Argan oil is a plant oil that produced from Argan tree (*Argania spinose)* which become one of the main roles in the dermocosmetic field due to higher in moisturizer contents. The main objective of the research is to evaluate the characteristics of cosmetic serum and to identify the best formulation of cosmetic serum containing Argan oil as moisturizing agent. Five formulations of cosmetic serum (F1, F2, F3, F4 and F5) with different concentration of Argan oil have been developed. They were evaluated for its physical appearance, pH, rheology, spreadability, skin moisturizing, stability test and analysed by using ANOVA single factor. The product was milky white, rose cheek smell, non-greasy, non-oily and homogen. The result showed that pH of five formulations have shown significant different ($p<0.05$) and was fall into acceptable range (pH 5-6). Stability study at low and room temperature shown all formulation were stable except for high temperature (at 40°C) only F2 after week 1. Statistical result indicated that there was significant different between all formulations in moisturizing effect ($p<0.05$) and F3 gave higher percentage of moisture rising. Rheological analysis shows all five formulations were pass over 30% of torque point. Formulation 2, 3 and 4 gave more than 50% of spreadability percentage compared with formulation 1 and 5. In this study, it can be concluded that the F2 of formulated cosmetic serum are stable and could delivered high moisturizer effect on the skin.

1 INTRODUCTION

The cost increase in worldwide living standard has created a rise in demand for cosmetic products. The cosmetic industry established in Malaysia is one of the important economic sources. The importance of cosmetics has increased as many people want to stay young and attractive.

Cosmeceutical refers to the combination of the cosmetics industry with the pharmaceutical industry. Cosmetics companies produce cosmetics and pharmaceutical ones produce medicines. Cosmeceuticals are skincare products that combine cosmetics and medicines. What this means to the consumer is that they actually work (DeHaven, 2007). “Cosmetic Product” by definition from Guideline on The Control of Cosmetic Products, Health Science Authority, Revised in 2014, is any product that is intended to be placed in contact with the various external parts of the body, such epidermis, hair system, nails, lips, eyes, teeth and the mucous membranes of the oral cavity and external genital organs with purposes mainly to cleaning, perfuming, changing their appearance, correcting body odours, protecting or keeping them in good condition (Authority, 2011).

The available cosmetic products are classified mainly in five classes which are skin care products, hair care products, colour cosmetic products, personal care products, and fragrances (T. Mitsui, 1993). The skin care and maintenance are included moisturizers, massage oils, creams, fairness creams, petroleum gels, sunscreens, anti-itching creams, and antiseptic ointments.

Serum is one of the cosmetic products with very high concentration of active ingredient in their formula for providing intensive nutrition to the deeper skin layer and non-greasy finish product which suitable for skin.

Cosmetic serum was classified based on its rate of absorption and the ability to penetrate into the deeper layers of the skin. This research was planned to

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develop an ideal cosmetic serum containing Argan oil as a moisturizing agent to the skin. Serum are differ from others moisturizers in that serum do not "make room" for emollients or thickener. Likewise, serum will not contain sunscreen active like daytime moisturizer would but serum preserve the extra space for other beneficial ingredients or even more antioxidants than any others product.

2 METHODOLOGY

2.1 Materials

Argan oil, Squalane light (Chem Soln), Triglyceride (Sigma), Glycerin USP (Chem Soln), Hyaluronic acid HMW, Sodium EDTA, PEG-100, PEG-12 Dimethicon, Sodium acrylate, Phenoxyethanol-SA, distilled water.

2.2 Apparatus and Equipments

Rheometer (Brookfield® R/S CP), pH meter (Hanna®), Moisture Checker (Scalar®), homogenizer (Ultra Turrax), beaker, glass rod, filter paper (Whatman), syringe with needle, aluminium foil, dropper.

Table 1: Formula of cosmetic serum

Materials	Quantity (%)				
	F1	F2	F3	F4	F5
PHASE A					
Water	73.8				
EDTA	0.2				
Hyluronic acid HMW	2				
PHASE B					
PEG-12	4				
Squalene light	6	5	4	3	2
Argan oil	1	2	3	4	5
Triglyceride	3				
PHASE C					
Glycerin USP	5				
PEG-12 Dimethicon	3				
Sodium acrylate	1				
Phenoxyethanol-SA	1				

2.3 Preparation of Cosmetic Serum

All the ingredients were weighed according to the different percentage listed (Table 1). The net weight of all formulated serum was 100g. Add water and EDTA into a disinfected glass beaker and stir, until EDTA has dissolved. Add hyaluronic acid and mix thoroughly with a stick blender or homogenizer until phase A is free of lumps. Add phase B to phase A, stir well after each ingredient has been added. Mix with stick blender. Add phase C to phase A/B, again, stirring well after each ingredient. Especially after sodium acrylate uses the stick blender. Serum should be free of any lumps. Viscosity can be adjusted by adding, 2.5% of the sodium acrylate, if needed.

2.4 Organoleptic, Physicochemical and Stability Testing of Cosmetic Serum

The properties and stability testing of cosmetic serum were carried out for the five different formulations of cosmetic serum. The observation were recorded on day one of preparation, week one, week two, week three, week four and two month after preparation for determination of organoleptic properties, pH, homogeneity, rheological, skin moisture and stability study.

2.4.1 Physical Appearance

Observe the color of the serum formulation sample which should be in white milky and glossy appearance. Next, feel some serum formulation sample on the skin to access the texture which should smooth homogeneous texture and non-greasy finish.

2.4.2 pH Test

The pH test will be determined by using Digital pH meter. Dipper of digital pH will be deep into the sample of serum formulation and the pH value will be recorded. The pH of the formulation should having acidic pH as the skin is having an acidic pH of around 4–6.

2.4.3 Homogeneity

This will be confirmed by spread some of the serum formulation on the transparent glass and observe it. The formulation should produce uniform distribution of serum.

2.4.4 Rheological Study

Viscosity of the formulation is determined by Brookfield® Viscometer at 100rpm, using spindle type model S64.5 ml of the serum. The serum will placed in a big mouth container with the spindle dipped in it for about 5 minutes before the measurement.

2.4.5 Spreadability Test

The product spreads on the skin or affected area and denotes the extent of area to which the serum was applied. Some sizes of filter paper are chosen and each filter paper is measure the total area of filter paper (A1) and weighing of each filter paper (W1). Choose the formulation to be tested and draw several milliliters into the B-D 5mL syringe and drawn onto the center of filter paper for 20 drops. When latest drop hits the filter paper, start a timer or stopwatch to count down for exactly 10 minutes. During the 10 minute test, the liquid will spread in a relatively uniform circular pattern over the filter paper. After 10 minutes, cut exactly on the line between saturated spread and dry filter paper by using scissor. Weigh the remaining dry (unsaturated) filter paper. Record this weight as W2. Measure the diameter of the saturated portion of filter paper. If the spread was not a perfect circle, then take several diameter readings around the spread area and determine an average diameter. Record this measurement as A2.

% Spread by Area = (A2/A1)100

2.4.6 Skin Moisture Test

Skin moisture will be measure by using Scalar moisture checker after applying cosmetic serum on the skin. The scale will show the moisture of the skin after using the cosmetic serum.

2.4.7 Stability Test

It is to determine physical and chemical stability of the product with accelerated stability analysis which subjects the material to elevated temperatures. Short term accelerated stability study was carried out for the period of 3 months for the formulation. The samples were stored at different storage conditions of temperatures and samples are withdrawn on monthly interval and analyzed.

3 RESULTS AND DISCUSSION

3.1 Physical Appearance

Physical appearance was evaluated by observation on the texture, color and smell of the formulated cosmetic serum. All formulations gave milky white finish products with non-greasy and non-oily properties after 4 weeks observation. The rose smell of formulation 1 and 4 started to disappear after week 2 while formulation 2, 3 and 5 after week 3. Homogeneity of all formulations were uniformly distributed of contents while observing serum on the transparent glass slide.

The use of fragrance in cosmetic serum is to cover the unpleasant smell of Argan oil. The 1 part of concentrated rose cheek fragrance was diluted into 10ml distilled water to get 0.1% fragrance before used in all five formulations. The diluted fragrance used in formulation made them less smell and unstable to stand in longer time. Thus, all smelling of five formulations started to disappear at week 4.

Fragrance is not always readily detected in cosmetics either often masking agents are used to disguise the natural smell of the active ingredients (which often have really unpleasant smells). These masking agents so “clean” smell that often consumers think the product is fragrance-free (Dan Thompson, June 2014).

3.2 pH Evaluation of Cosmetic Serum

The result shows on the day 1, pH of five formulations are fall within the range of pH 5 to pH 6. This is because in the outer layers of the stratum corneum, the moisture barrier has a slightly acidic pH (4.5 to 6.5). These slightly acidic layers of the moisture barrier are called the acid mantle. The acidity is due to a combination of secretions from the sebaceous and sweat glands. The acceptance range for the effectiveness of a dermocosmetic product is lies between pH 4 to pH 6 (Shan Sasidharan, 2014). After two months, the pH of these formulations rises gradually, which closed to the neutral pH (pH 7). However, all formulation was considerably fulfilling the pH requirement as a dermocosmetic product. The distribution of pH test between formulation 1, 2, 3, 4 and 5 showed no significantly different ($p < 0.05$).

3.3 Rheological Study of Cosmetic Serum

From the test, the torque value of all five formulations were more than 30% which mean the

resulted value of viscosity is in the acceptable viscometer range.

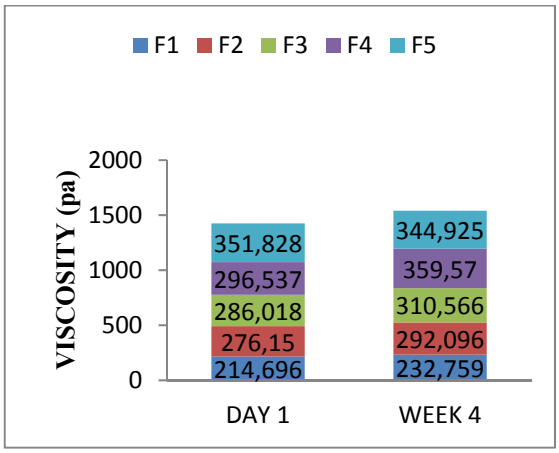

Figure 1: Viscosity study of the formulation

Viscosity of all formulation 1, 2, 3, 4 and 5 are gradually increased after 4 weeks placed in the room temperature (25±2) from 214.696, 276.15, 286.018, 296.537 and 351.828 to 232.759, 292.096, 310.566, 359.57 and 344.924 respectively.

Since the F1, F2, F3, F4 and F5 having an increasing in emollient which are 1%, 2%, 3%, 4% and 5% of Argan oil, the viscosity of these formulation are increasing from day 1 to week 4. A product viscosity is determined by its structure and the greatest impact on it has the continuous phase; eg water. The effect on the viscosity is depending on the size, shape and concentration of the suspended particles and how they are interact with the continuous phase (Tharwat F. Tadros, 2013).

Viscosity is a measurement of internal fluid friction which is resistance to flow when one layer of fluid is forced to move in over another layer and typically measured with Brookfield Viscometer. A fluid may be made up of molecules that vary in size, shape, and cohesiveness or a single type of molecule. As these molecules are forced to move or flow past each other, the molecular properties will determine just how much force is required to move them past each other. The force required to cause movement is referred to as shear. Shear force are importance in cosmetic product in order to determine the packaging of pumping product during manufacturing and also the spreading of serum on the skin.

According to an article, most of cosmetic products mostly categorize under shear thinning for suspensions and emulsions, where their viscosity decreases with increasing shear rate. This behavior is also referred to as pseudoplastic and is the result of structural breakdown within the fluid (Kelly Dobos, 2017).

Table 2: pH result of the formulation

	Mean ± Standard deviation (N=3)						Mean ± Standard deviation
	Day 1	Wk 1	Wk 2	Wk 3	Wk 4	Wk 8	
F1	5.87±0	5.89±0.01	6.03±0.02	6.03 ±0.05	6.14±0.02	6.89±0.01	6.13±0.02
F2	6.01±0.01	5.98±0.01	6.04±0.01	6.08±0.01	6.06±0.01	6.21±0.01	6.06±0.01
F3	6.01±0.01	6.01±0.02	5.99±0.01	6.05±0.02	6.14±0.02	6.52±0.01	6.12±0.004
F4	5.86±0.01	5.88±0.01	5.87±0.01	5.92±0.01	5.95±0.01	6.22±0.01	5.95±0.002
F5	5.90±0.01	5.90±0.02	5.93±0.02	5.91±0.01	5.95±0.02	6.18±0.02	5.97±0.005

3.4 Spreadability Study of Cosmetic Serum

Spreadability indicates the area on which a semi-solid topical formulation spreads on application to the skin. This parameter plays a key role in determining both the efficacy and the consumer acceptance of the product. A poor spreadability may result in an uneven distribution of the formulation on the skin, thus affecting the amount of the dose applied and the efficiency of active ingredient(s) skin permeation. On the other hand, consumers perceive a poor spreadability as a weakness of the product, which could lead to the choice of other products with a better performance, independently on their actual efficacy.

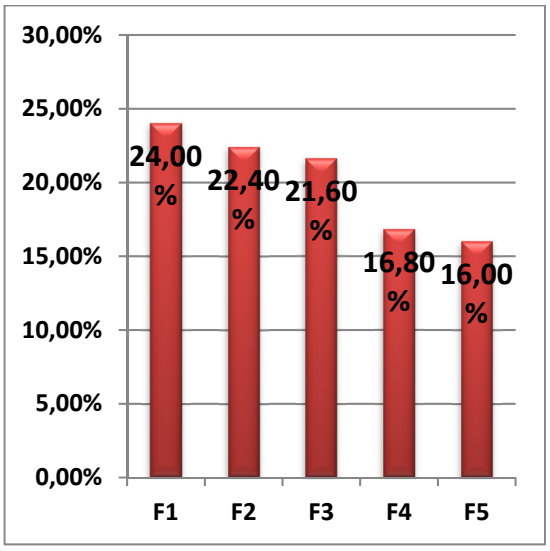

Figure 2: Percentage spreadability of the formulation

From the result of evaluation, it is showed that formulation 1 give the higher percentage of spreadability with 24% compared to other formulations. Secondly, the formulation 2 and 3 also showed more than 20% percent of spreadability during the test carried out while formulation 4 and 5 only give 16.8% and 16% of spreadability respectively. Thus, it was assumed that formulation 1 had a good spreadability and consumer satisfaction when compared to others formulation. Other authors reported a linear relationship between viscosity and spreadability for topical formulations, as the lower the viscosity of a lotion, the lower the surface tension and the higher the spreadability on the skin (Lardy F, 2000). When compare with the viscosity test, formulation 1 having lowest viscosity, thus it affect its spreadability which having the highest percentage of spreadability.

For the rheological study and spreadability, there are having linear relationship between viscosity and spreadability, as the lower the viscosity, the lower the surface tension and the higher the spreadability on the skin.

3.5 Skin Moisture Study of Cosmetic Serum

A total of 15 volunteers, ages ranging from 18 to 25 years old were included in the study. The volunteers are healthy with no preceding skin diseases. Non-invasive skin moisture measurements were carried out using Scalar moisture checker at 1 minute, 5 minutes and 10 minutes to determine the short term improvement in skin moisture properties after a single application of cosmetic serum. Five different cosmetic serums containing different concentration of emollient (Argan oil) which are 1%, 2%, 3%, 4% and 5% were used in this evaluation.

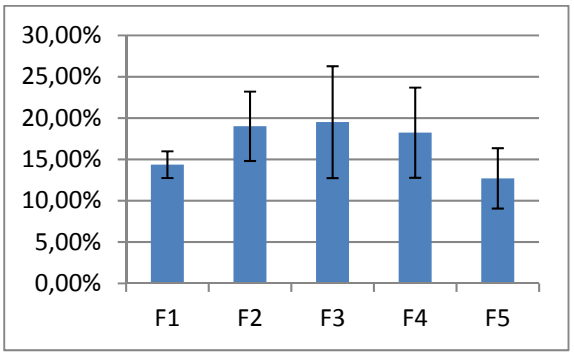

Figure 3: Percentage of moisture increase of the formulation

From the moisture test of formulation 1, moisture increment in volunteer 1, 2 and 3 are 14.81%, 12.57% and 15.70%. The total percentage increment is 14.36 % (±1.61). In the formulation 2, moisture increment in volunteer 1, 2 and 3 are 21.28%, 14.16% and 21.57%. The total increment of formulation 2 is higher than formulation 1, 4 and 5 which is 19.00 % (±4.20) and slightly lower by 0.67% from the formulation 3. The formulation 3 shows the highest percentage of moisture increment in all three volunteers which is 19.67 % (±6.49). While in the formulation 4, total increment of moisture from the individual's volunteer 1, 2 and 3 (23.99%, 13.13%, 17.57%) is 18.23% (±5.46). lastly, formulation 5 show the least moisture increment in the volunteer 1, 2 and 3 (16.83%, 11.40% and 9.88%) with the total moisture increment is 12.70% (±3.65). Thus from the result, formulation 3 show the highest moisture levels raised compared to others formulation.

According to the one way ANOVA using data percentage of moisture increment for 15 volunteer at 1 minute, 5 minutes and 10 minutes for all formulation, all data show high significant different ($p<0.05$) among each volunteer. The moisture level of each volunteer varied due to the different thickness of skin between male and female, environment condition and lifestyle of volunteers.

This proven that Argan oil contains higher contents of triglyceride which mainly act as moisturizing agent through the percentage of moisture rising after first application. Emollients provide some occlusivity and improve the appearance of the skin by smoothing flaky skin cells. There are many

different types of emollient esters and oils available. Emollients are generally grouped by their ability to spread on the skin. By combining emollients with the different spread rates, it can tailor the skin feel of a moisturizer. In this cosmetic serum formulation, three types of emollient were used such as Argan oil, squalene and triglyceride. Additionally, emollient lipids similar to those naturally found in the skin may also increase the rate of barrier repair (Dobos, 2016). Thus, by increasing the percentage of emollient would be increasing the skin's moisture level.

3.6 Stability Study of Cosmetic Serum

The sample of formulations were stored at three different storage conditions and withdrawn weekly to analyze the physical stability.

Table 3: Stability studies of the formulation

	Room temperature (25°C±1)				Cold room (2-3°C)				Oven (40°C)			
	W1	W2	W3	W4	W1	W2	W3	W4	W1	W2	W3	W4
F1	X	X	X	X	X	X	X	X	/	/	/	/
F2	X	X	X	X	X	X	X	X	/	/	/	/
F3	X	X	X	X	X	X	X	X	X	/	/	/
F4	X	X	X	X	X	X	X	X	/	/	/	/
F5	X	X	X	X	X	X	X	X	/	/	/	/

X : No phase separation
/ : Phase separation

All 5 formulations were stable at room temperature (25°C±2) and cold room (2-3°C) for a month without having any phase separation. At the high temperature storage (40°C), all 5 formulations show stable during first three days. After that, formulation 1, 3, 4 and 5 started to have phase separation while formulation 2 stable for a week and become unstable at the week 2. This process results from the external forces usually gravitational or centrifugation. When such forces exceed the thermal motion of droplets, a concentration gradient builds up in the system with the larger droplets moving faster to the top or bottom of container depends on their density (Tharwat F. Tadros, 2013).
Oil in water emulsion is considered to be special liquid-liquid colloidal dispersions. The kinetic stability is a consequence of small droplet size and the presence of n interfacial film around the oil droplets and is caused by stabilizing agent or emulsifiers. In the serum formulation, polyethylene glycol-100 (PEG-100) had been used as oil in water emulsifier to form a homogenous mixture by keeping water and oil together. These stabilizers suppress the mechanisms that would involve in the breakdown of emulsion such as sedimentation, aggregation, coalescence and phase inversion.
These serums are stabilized by films that form around the oil droplets at the water-oil interface.
Temperature can affect emulsion stability significantly. Temperature affects the physical properties of oil, water, interfacial films, and surfactant solubilities in the oil and water phases. These, in turn, affect the stability of the emulsion. Perhaps the most important effect of temperature is on the viscosity of emulsions because viscosity decreases with increasing temperatures. This decrease is mainly because of a decrease in the oil viscosity. In this evaluation, temperature of the oven (40°C) increases the thermal energy of the oils droplets and, therefore, increases the frequency of drop collisions. It also reduces the interfacial viscosity, which results in a faster film-drainage rate and faster drop coalescence.
The effect of temperature on crude oil/water interfacial films was studied in some detail by Jones *et al.*, who showed that an increase in temperature led to a gradual destabilization of the crude oil/water interfacial films. However, even at higher temperatures, a kinetic barrier to drop coalescence still exists. Temperature influences the rate of buildup of interfacial films by changing the adsorption rate and characteristics of the interface. It also influences the film compressibility by changing the solubility of the crude oil surfactants in the bulk phase.

4 CONCLUSIONS

The formulated cosmetic serum was successfully developed and evaluated using different standard parameters including skin moisture rising properties. Based on all cosmetic serum formulations studied, all F1, F2, F3, F4 and F5 formulation showed significantly different in rising of skin moisture level ($p<0.05$). On the evaluation of the finished serum's texture, all formulations give a milky white in color, non-greasy, non-oily and homogeneous contents. Without any pH adjustment, all the formulations gave the pH value within the limits of normal skin pH range. Besides that, in the stability study of

cosmetic serum, all formulations stable until 4th week in the room temperature (25°C±1) and cold room (2-3°C) but unstable in the oven (40°C) after the first 3 days except for F2 which stables for a week before undergo phase separation.

Thus, from the studied, the F2 is the best formulation of cosmetic serum as it is the most stable formulation and could give highest percentage of moisture increased.

Besides that, further studies are warranted to prove safety and efficacy of the formulated cosmetic serum and extend formulation to gel and cream formulation.

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CONFLICT OF INTEREST

Declare none

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