Cosmetic Serum Loaded Arabica Coffee (Coffea arabica) Extract: Formulation and Antioxidant Study

Harum Nadyanti Usman¹, Liza Pratiwi²*, Bambang Wijianto³
¹Pharmacy Department, Faculty of Medicine, Tanjungpura University, Pontianak, Kalimantan Barat, Indonesia
²Pharmaceutical Technology Department, Pharmacy Department, Medical Faculty, Tanjungpura University, Pontianak, Kalimantan Barat, Indonesia
³Pharmaceutical Chemistry Department, Pharmacy Department, Medical Faculty, Tanjungpura University, Pontianak, Kalimantan Barat, Indonesia

ABSTRACT

Arabica coffee (Coffea arabica) is a natural antioxidant that can be used in the prevention of skin aging. This study was conducted to determine the comparison of variations in the composition of HPMC, CMC-Na, and Carbomer in the optimum formula of arabica coffee serum, then to determine the physical properties of the preparation, as well as the IC₅₀ value of arabica coffee extract and arabica coffee serum in the DPPH test. Arabica coffee extract was obtained by maceration method using 70% ethanol. Determination of the optimum formula of arabica coffee serum using the Simplex Lattice Design method. The optimum formula of arabica coffee serum was evaluated for physical properties including organoleptic test, homogeneity test, pH test, spreadability test, and adhesion test. The antioxidant activity of arabica coffee extract and the optimum formula of arabica coffee serum was tested using the DPPH method. Data analysis used SPSS One sample T-test statistics. The results showed that the composition variation of HPMC, CMC-Na, and Carbomer in the optimum formula of arabica coffee serum using the Simplex Lattice Design method was 0.1%:0.8%:0.1%. The resulting predicted values are a pH value of 4.88 and a spreadability value of 7.95 cm. The optimum formula of arabica coffee serum has the physical properties of the preparation, namely brown color, coffee aroma, light texture, a cool sensation when applied to the skin, homogeneous, has a spreadability of 7.93 cm, stickiness of 1 second, and pH value of 4.9. The IC₅₀ value of arabica coffee extract and the optimum formula of arabica coffee serum are 8.13 ppm and 250 ppm. The results of the SPSS One sample T-test statistical analysis showed that there was no significant difference between the prediction response and the results of the optimum formula evaluation.

Keywords: Arabica Coffee; Antioxidant; Serum; Simplex Lattice Design

INTRODUCTION

Skin aging is one of the most commonly discussed dermatological problems (Ahmad, 2018). Skin aging is a degenerative process of the skin that occurs faster than its time. Symptoms that arise are fine lines, wrinkles, and dark spots on the facial skin (Ginting, 2020). Factors of skin aging consist of intrinsic factors and extrinsic factors. Intrinsic factors can be caused by an increase in free radicals and DNA damage, while extrinsic factors are caused by UV exposure and smoking behavior (Nisa, 2016; Dewias, 2017). Skin aging treatments can be done by using photoprotector ingredients, topical medications containing retinoid acid or hydroquinone, chemical peels, microdermabrasion, botox injections, filler injections, laser therapy, and antioxidants (Taylor, 2005; Knaggs, 2008).

Antioxidants are compounds that have a molecular structure that can provide electrons to free radical molecules and can break the chain reaction of free radicals. Antioxidants are widely used for health and cosmetic products. Antioxidants are found in nature, for example in plants and animals. Many plants contain secondary metabolites that have antioxidant activity, one of which is coffee (Darmawan, 2013; Ajhar, 2020).

Coffee is one of the plantation products that are widely found in Indonesia and has a fairly high economic value. Based on previous research, it is stated that the 70% ethanol extract of coffee beans has very strong antioxidant activity with an IC₅₀ value of 12.42 ppm because it contains flavonoids, tannins, saponins, alkaloids, and steroidal compounds. Coffee beans have been widely used in cosmetic preparations. The extract cannot be used directly in topical use, so it requires a dosage formulation such as serum (Mardhiani, 2018; Ajhar, 2020).

The serum is one of the skincare products used to treat skin health and beauty. Serum has a certain content or special function that suits the needs of facial skin. This preparation has the

*Corresponding author: Liza Pratiwi
Email: lyza_pratiwi@yahoo.com
advantage of a light texture so that it is easily absorbed by the skin. In addition, serum In also has a higher concentration of active substances compared to preparations, so other cosmetic it is expected to provide greater effectiveness of active substances. The factor that needs to be considered in formulating serum is the selection of gelling agent (Mardhiani, 2018; Tilarso, 2022).

The gelling agent is a material that can affect the structure of serum gel because it can affect the viscosity and stability of the preparation (Tilarso, 2022). Gelling agents that are often used are HPMC, CMC-Na, and Carbomer. The advantages of HPMC are that the resulting gel has a neutral pH, is clear, and has a stable viscosity. The advantage of CMC-Na is that the resulting gel is clear, but has a large cohesion power (Widyaningrum, 2019; Hati, 2021). The advantage of carbomer is that the resulting gel is transparent and bioadhesive (Dewi, 2016). Serum with the variation of gelling agent carbomer and CMC-Na has large viscosity and small spreadability. The ideal criteria for serum are light and spreadable when used on the skin. HPMC can provide good viscosity stability so it is expected that the addition of HPMC can improve serum viscosity (Hati, 2021). The dosage formula can be determined by trial and error or using a method, namely the Simplex Lattice Design.

Simplex Lattice Design is an optimization method used to determine the optimum formula for a preparation. This method can avoid determining the formula by trial and error so that it can determine the formula quickly and practically (Hidayat, 2020). Based on this description, a researcher is interested in research to determine the optimum formula for arabica coffee serum preparations with variation gelling agents (HPMC, CMC-Na, and Carbomer) using the Simplex Lattice Design method. This research has the novelty that there has been no previous research formulating Arabica coffee as a serum preparation with variations in the composition of HPMC, CMC-Na, and Carbomer.

**METHODOLOGY**

**Preparation of Arabica Coffee Bean Extract**

A total of 500 g of roasted arabica coffee bean powder was macerated using 1 L of 70% ethanol solvent. Soaked for 3 days, with stirring once a day. Repetition was carried out until the maceration process ended. The collected macerate was then put into a glass beaker and heated at 60°C.
in a water bath until the solvent evaporated. Arabica coffee thick extract was obtained (Hilda, 2021). In the previous study, standardization of arabica coffee extract was carried out. Standardization consists of specific parameters and non-specific parameters. Specific parameters consisted of the level of water-soluble content and the level of ethanol-soluble content. Non-specific parameters consisted of loss of dry, water content, total ash content, and acid-insoluble ash content (Muchtaridi, 2017).

Arabica Coffee Serum Formulation

Arabica coffee serum was made with variation gelling agents (HPMC, CMC-Na, and carbomer). The arabica coffee serum formula in Table I.

The serum preparation starts with developing the gel base. The gelling agent is dispersed with warm aquadest in a mortar and stirred to form a gel base. Arabica coffee bean extract was dissolved in aquadest then added to the base and stirred until uniform. Methylparaben, glycerin, and DMSO were added and mixed well. Then add aquadest ad up to 100% (Aulifa, 2020).

Arabica Coffee Serum Optimum Formula

The optimum formula of arabica coffee serum was determined using software design expert version 13 with the Simplex Lattice Design method. Response parameters used were pH value and spreadability.

Evaluation of Physical Properties of Arabica Coffee Serum Optimum Formula

The evaluations to be carried out are an organoleptic test, homogeneity test, pH test, spreadability test, and adhesion test.

Organoleptic Test
Organoleptic test on serum including color, aroma, and skin sensation by observing the visual appearance and skin sensation (Tilarso, 2022).

Homogeneity Test
A homogeneity test on serum was done by applying serum on a glass plate and then visually observing to see the color and texture of the serum. Homogeneity was indicated by the absence of coarse grains and the color of the serum was the same overall (Tilarso, 2022).

pH Test
The pH test was carried out using a pH meter. The pH meter was dipped into the serum preparation. Calibration was carried out first by dipping the pH meter into pH 4 and 7 buffer solutions (Tilarso, 2022).

Spreadability Test
The spreadability test was carried out by placing a 0.5-gram sample on a petri dish and waiting for 1 minute, then measuring the diameter of the sample spread. After that, a weight of 150 grams was added and allowed to stand for 1 minute and then a constant diameter was measured (Tilarso, 2022).

Adhesion Test
The adhesion test was carried out using a sample of 0.25 grams placed between 2 glass preparations, then pressed by a load of 1 kg for 5 minutes. After that, the serum release time was recorded (Tilarso, 2022).

Antioxidant Activity Test of Arabica Coffee Extract
An antioxidant activity test was conducted using the DPPH method. An a-concentration series of 150, 175, 200, 225, and 250 ppm were made using methanol PA. After that, a 0.5 mM DPPH stock solution was added with a volume ratio (1:1), then incubated for 30 minutes at room temperature using a dark container, and absorbance measurements were taken using a UV-Vis Spectrophotometer with a wavelength of 516 nm (Mardhiani, 2018).

Antioxidant Activity Test of Arabica Coffee Serum Optimum Formula
An antioxidant activity test was conducted using the DPPH method. An a-concentration series of 50, 70, 80, 200, and 250 ppm were made using methanol PA. After that, a 0.5 mM DPPH stock solution was added with a volume ratio (1:1), then incubated for 30 minutes at room temperature using a dark container, and absorbance measurements were taken using a UV-Vis Spectrophotometer with a wavelength of 516 nm (Mardhiani, 2018).

Data Analysis
Data analysis was conducted using SPSS statistics. Data analysis used one sample T-test analysis. Differences between groups of serum
formulas were observed with the effect of the type and concentration of the gelling agent on pH and spreadability (Tilarso, 2022).

**RESULTS AND DISCUSSION**

**Extract Ethanol 70% Arabica Coffee Beans**

The thick extract obtained was 108 g of 500 g of arabica coffee powder, resulting in a yield of 21.6%.

**Formulation and Physical Properties Test of Arabica Coffee Serum**

The physical properties tests carried out were pH tests and spreadability tests. The pH and spreadability values were used as response parameters for determining the optimum formula. The pH test was conducted to see the acidity of the serum preparation. The pH of topical preparations that are safe for the skin is in the pH range of 4.5-6.5 (Aziza, 2020). The equation of the effect of the gelling agent on pH value can be seen in Table IV. The results show that the gelling agent that has a higher effect on increasing the pH value is HPMC, followed by CMC-Na then carbomer. Carbomer is acidic so if the formula levels are increased it can cause a decrease in pH value. Increasing the levels of HPMC and CMC-Na in the formula can increase the pH value of the preparation (Tambunan, 2018; Marchianti, 2021).

The spreadability test was conducted to see an overview of the serum’s spreading ability when applied to the skin. Good topical preparation has a spreadability of 5-7 cm. The wider the spreadability, the greater the effect is given by the active substance because it expands the contact area with the skin (Aziza, 2022). The equation of the effect of gelling agent on the spreadability can be seen in Table IV. The results show that the gelling agent that has a higher effect on increasing the spreadability value is CMC-Na, followed by carbomer and then HPMC (Tambunan, 2018; Marchianti, 2021).

**Arabica Coffee Serum Optimum Formula**

The optimum formula for arabica coffee serum was determined using the Simplex Lattice Design method. Determination of the optimum formula is measured using the degree of
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Figure 1. Contour Plot of pH and Spreadability

Figure 2. Arabica Coffee Serum

Table VI. Equation of Effect of Gelling Agent on pH Value and Spreadability

<table>
<thead>
<tr>
<th>Response</th>
<th>Equation</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td>+4.87(A) + 4.70(B) + 4.45(C) + 1.83(AB) + 0.69(AC) + 1.33(BC) - 8.62(ABC)</td>
</tr>
<tr>
<td>Spreadability</td>
<td>+ 6.26(A) + 8.18(B) + 7.86(C)</td>
</tr>
</tbody>
</table>

Description: A = HPMC; B = CMC-Na; C = Carbomer

Table VII. Goal Setting, Lower Limit, and Upper Limit of Response Parameters

<table>
<thead>
<tr>
<th>Response</th>
<th>Goal</th>
<th>Lower Limit</th>
<th>Upper Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td>In range</td>
<td>4.8</td>
<td>6</td>
</tr>
<tr>
<td>Spreadability (cm)</td>
<td>Maximize</td>
<td>5.9</td>
<td>8.5</td>
</tr>
</tbody>
</table>

The desirability, a formula that has a degree of desirability close to 1 is chosen as the optimum formula (Hidayat, 2020).

The desirability value in this study was 0.79 obtained from the composition of HPMC:CMC-Na: Carbomer (0.1%:0.8%:0.1%). The resulting predicted values are the pH value of 4.88 and the spreadability value of 7.95 cm.

Evaluation of Arabica Coffee Serum Optimum Formula

The serum was remade using the optimum formula obtained from the Simplex Lattice Design method and then the preparation was evaluated. An organoleptic test aims to determine the appearance of the preparation in the form of form, color, and smell (Afianti, 2015). The results
obtained are that the serum is brown according to the color of the coffee extract, the aroma of the serum smells of coffee, the serum has a light texture, and a cold sensation when applied to the skin.

The homogeneity test aims to determine whether the ingredients in the formulation are evenly mixed or not (Asky, 2022). The results showed that there were no coarse grains or even colors in the preparation, so it can be
concluded that the serum made has good homogeneity.

The pH value in the arabica coffee serum optimum formula is safe for the skin because it is in the pH range of 4.5-6.5. The results of the One Sample T-test analysis of pH value obtained a significance value (sig. (2-tailed)) of 0.41 > α (0.05), so there is no significant difference between the predicted value and the evaluation results of the optimum formula.

The spreadability value in the arabica coffee serum optimum formula is quite large so that the serum can spread widely on the skin surface. The results of the One Sample T-test analysis of the spreadability value obtained a significance value (sig. (2-tailed)) in the spreadability test was 0.97 > α (0.05), so there was no significant difference between the predicted response and the evaluation results of the optimum formula.

The adhesion test aims to determine how long the attachment time of the preparation on the surface of the skin is (Rohmani, 2019). Serum preparations have a small adhesion ability because the texture is not too thick with the aim that it can be spread evenly on the skin. The gelling agent that gives the effect of increasing the higher adhesion value is HPMC, followed by carbomer and then CMC-Na (Tambunan, 2018; Santoso, 2022).

**Antioxidant Activity Test of Arabica Coffee Extract and Arabica Coffee Serum Optimum Formula**

An antioxidant activity test was conducted using the DPPH method. The DPPH method procedure is to measure the decrease in DPPH absorption at its maximum wavelength by adding a DPPH reagent solution so that it is proportional to the concentration of free radical inhibitors. Antioxidant activity is seen from the IC$_{50}$ value (Martiani, 2017).

The IC$_{50}$ value in the arabica coffee serum optimum formula is 250 ppm, which is in the range of 100-250. So the arabica coffee serum optimum formula has antioxidant activity with moderate category. The antioxidant activity of arabica coffee serum is not as strong as arabica coffee extract, this is due to the lack of maximum release of arabica coffee extract from the gel base when reacting with DPPH when testing antioxidant activity, resulting in lower IC$_{50}$ values.
in less strong antioxidant activity. So it is necessary to increase the concentration of arabica coffee extract in the serum to obtain stronger antioxidant activity.

CONCLUSION
The conclusion of this study is the comparison of variations in the composition of gelling agents in the optimum formula of arabica coffee serum, namely 0.1%-0.8%-0.1% (HPMC:CMC-Na:Carbomer). The optimum formula of arabica coffee serum has the physical properties of the preparation, namely brown color, coffee aroma, light texture, a cool sensation when applied to the skin, homogeneous, has a spreadability of 7.93 cm, stickiness of 1 second, and pH value of 4.9. The IC50 value of arabica coffee extract and the optimum formula for arabica coffee serum are 8.13 ppm and 250 ppm.

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