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Formulation and Antibacterial Activity of Coffee Ground Waste Extract Face Serum

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ABSTRACT

Coffee is one of the most popular beverages around the World, which generates over million tons of coffee ground waste each year. Using technologies, the waste can be converted into value-added products such as cosmetic. The objective from this study is to formulate the coffee ground waste extract to face serum for anti-acne treatment. Samples are collected from several coffee shops, then macerated using ethanol. Formulation consisting: HPMC, tween 80, glycerin, PEG 400, sodium EDTA, sodium benzoate and triethanolamine. Evaluation is conducted to the extract antibacterial activity, face serum bacterial activity and face serum stability. The extract exhibits 0.860 gram/mL MIC50 Cutibacterium acnes, meanwhile, the face serum provides 15.30 ± 4.03 mm inhibition zone. Ultimately, the face serum is stable after 4 weeks testing using accelerated stability test in terms of organoleptic, pH and viscosity.
Keywords : Cutibacterium acnes; accelerated stability; face serum; cosmetic

INTRODUCTION

Coffee is one of the most popular beverages around the World. The coffee business generates over million tons of coffee ground waste (CGW) each year, which will become environmental problems in the future (Lee et al., 2023). The waste from coffee business viewed as environmental problems, but using technologies, it can be converted into value-added products (Lee et al., 2023).

There is a hidden potency in the coffee ground waste because it still contains phenolic, caffeine, chlorogenic acids and caffeic acids which exhibit various health benefit like antioxidant (Choi et al., 2018; Ludwig et al., 2014; Usman et al., 2023), anticancer (Mojica et al., 2018), anti-inflammatory agent (Choi et al., 2018) and antibacterial (Ludwig et al., 2014; Singh Arora et al., 2009).

Based on literature study, there are still few research which utilise the CGW for acne treatment. One of the mechanism for anti-acne treatment is preventing overwhelm Cutibacterium acnes activity (Banki & Castiglione, 2015). Since crude extract cannot be apply directly onto human skin, suitable formulation will be needed, not only to optimize the CGW extract activity but also increasing user convenience. Serum formulation exhibit stable and easy-use application for face application. Hence, serum formulation can provide high concentration application through the skin, which crucial for inhibiting bacterial activity on skin (Aggnihotri, 2021; Burlando et al., 2010).

In this research, we formulate CGW into serum to enhance user convenience and maximize its potency. Also, we evaluate its antibacterial activity and product quality including accelerated stability test, so it can be further developed as choice for antiacne treatment in the future.

METHODOLOGY

Sample preparation

The coffee ground waste (CGW) sample were collected from several coffee shop in Yogyakarta, Indonesia. All the coffee ground waste composed by 30% arabica and 70% robusta. The samples were sieved then dried at oven at 70 °C for 24 hours to remove excessive water content from previous brewing process. The moisture content was determined using moisture balance.

Extraction of coffee ground waste

Dry coffee ground waste sample was subjected into maceration tube, then ethanol was added with 1:3 ratio. The mixture was stirred each 3 hours for 48 hours maceration time. Next, the extract

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was filtered then concentrated using water bath. The concentrated extract stored in refrigerator at 4 °C for further use.

Face serum formulation

The face serum was formulated based on formula in table II. The HPMC was developed using warm water and used as gelling agent. The extract was dissolved in water with tween 80, PEG400, and glycerin as a co-solvent. While, the sodium EDTA and sodium benzoate were dissolved in water and act as preservative solution. The extract mixture was added into HPMC solution followed by gentle stirring. After the extract completely dissolved, the preservative solution and triethanolamine added.

Accelerated stability test

The accelerated stability test was conducted in 40 °C temperature and 75 % RH for 4 weeks in climatic chamber. Each week, the organoleptic test, pH, and viscosity were measure, to assess the stability.

Anti-bacterial activity

To assess antibacterial activity of the coffee ground waste extract, Cutibacterium acnes ATCC 6919 was used, the bacteria was obtained from Faculty of Pharmacy, Gadjah Mada University. The bacteria was dissolved in 0.9 % NaCl and incubated at 37 °C for 24 hours in anaerobic jar. Next, 40 µL extract was pipetted into microplate, BHI media (150 µL) was added. Then, 10 µL bacterial suspension was added into microplate. Incubation was performed at 37 °C for 4 hours in anaerobic condition (Juniatik et al., 2017). DMSO was used as negative control and clindamycin as positive control. After incubation in chamber incubator, the microplate optical density was measured using microplate reader in 600 nm. Anti-bacterial activity of sample was indicated based on the decreasing of optical density compared with bacterial control. Hence, the 50% minimum inhibitory concentration (MIC50) value was determined using probit analysis (Pum, 2019).

On the other hand, to assess the face serum antibacterial activity, Brain-Heart Infusion (BHI) agar was used. Fifty microliter of bacterial suspension was pipetted and be diffused using spreader glass. The paper disk was prepared and 30 µL bacterial suspension was added. Incubation was carried out at 37 °C using anaerobic jar. Inhibition zone was measured and analyzed (Yosefa et al., 2023).

RESULT AND DISCUSSION

Coffee ground waste extraction result

The coffee ground waste (CGW) was collected from several coffeeshop in Yogyakarta Indonesia which had problem with coffee waste disposal. The samples still had strong coffee aroma, indicating that there were still several substances like caffeine, chlorogenic acids and caffeic acids although the coffee had been brewed previously. After cleaning and drying process, CGW was macerated using ethanol 70 % to withdraw non-polar substances from CGW.

The sample was cleaned to remove debris, then dried using oven at 70 °C for 24 hours. Then, the processed sample weighed and the moisture content measured, which the results can be observed at table I. After drying process, the moisture content was 3.57 ± 0.15 %, in this situation microbes cannot grow and there will no further hydrolysis reaction. After solvent evaporation to concentrated extract, 7.57 ± 0.89 % of CGW extract obtained.

Antibacterial activity of coffee ground waste extract

The antibacterial activity of the coffee ground waste (CGW) extract was determined using microdilution assay to calculated 50% minimum inhibitory concentration (MIC50), while antibacterial of the CGW face serum measure using disk diffusion method using inhibition zone (Veiga et al., 2019). For microdilution assay, the bacteria was incubated for 48 hours to obtained 0.08 – 0.12 optical density at 600 nm wavelength. This value was equivalent with 1.5 x 108 CFU/mL of bacteria in exponential phase (Mytilinaios et al., 2012; Zhang et al., 2015). The percentage Cutibacterium acnes inhibition can be seen in figure 1. To calculate MIC50 can be determined using probit analysis, the MIC50 of the coffee ground waste extract was 0.860 gram/mL. The MIC50 value

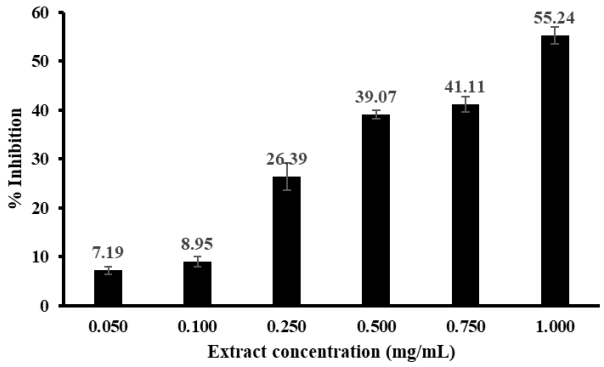

Figure 1. The antibacterial activity of coffee ground waste extract against Cutibacterium acnes

Table I. Formula of the coffee ground waste face serum

Ingredients	Weight (gram)
Cofffe ground waste extract	2.00
HPMC	0.75
Tween 80	1.00
Glycerin	7.00
PEG 400	4.00
Sodium EDTA	0.10
Sodium Benzoate	0.10
Triethanolamine	0.10
Water	Ad 100

Table II. Coffee ground waste extract results

Description	Value
Moisture content	3.57 ± 0.15 %
Yield	7.57 ± 0.89 %

can be used to justify the effectivity of antibacterial activity of substances (Butterfield et al., 2012). The lower the MIC50 value, the better the effectivity of the compound. Compared with other natural products, CGW extract was effective inhibit Cutibacterium acnes (Hou et al., 2019; Jantarat et al., 2018; Poomanee et al., 2018; Weber et al., 2019). Since the CGW extract came from waste product, application of CGW extract for antibacterial agent will beneficial not only for health but also for environment.

Face serum formulation

Extract solubility was one of the most crucial aspects in face serum formulation, so co-solvent like tween 80 was added to enhance extract solubility. Tween 80 was used because it can enhance coffee ground waste (CGW) extract solubility (Niazi, 2016). Humectant like glycerin is added to promote hydration in skin surface, also to assisted extract solubility. Skin hydration also enhanced active ingredients penetration through stratum corneum (Draelos, 2018). To adjust pH in desired range 4.5-5.5, TEA was added (Niazi, 2016).

Face serum evaluation

The accelerated stability test was conducted based on International Conference on Harmonisation (ICH) Guideline with slight modification at 40 °C temperature and 75 % RH for 4

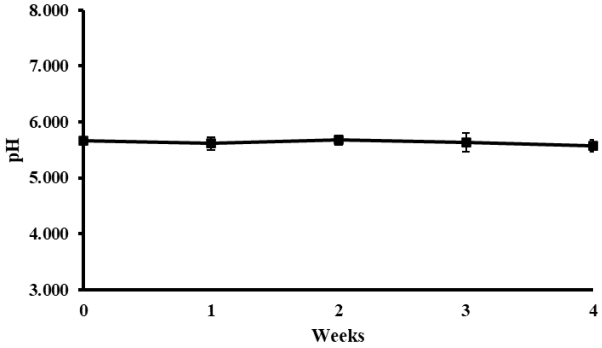

Figure 2. The pH stability profile in accelerated study test

Table III. The organoleptic test results of accelerated stability test

Weeks	Conditions	Parameters		
		Color	Homogenity	Odor
0	40 °C / RH 75%	Dark brown	Homogenous	Coffee-like
1	40 °C / RH 75%	Dark brown	Homogenous	Coffee-like
2	40 °C / RH 75%	Dark brown	Homogenous	Coffee-like
3	40 °C / RH 75%	Dark brown	Homogenous	Coffee-like
4	40 °C / RH 75%	Dark brown	Homogenous	Coffee-like

weeks in climatic chamber (González-González et al., 2022). Overall, after 4 weeks test, there was no significant changes on formulation in terms of organoleptic tests, pH, and viscosity.

Organoleptic tests were important to assess the physical acceptability of the coffee ground waste (CGW) face serum. The CGW face serum had consistent homogenous dark brown color, the color came from the CGW extract that had strong black-brown color. The texture was thick but not greasy in skin and washable by water. There was no lump observed in CGW face serum and no significant color difference in the product.

In terms of odor, the coffee odor was quite strong followed with tween 80 odor, since the tween 80 also exhibited strong odor. Moreover, after 4 weeks testing in extreme condition, there was no significant change observed in terms of the color, homogeneity, and odor. Based on the data obtained, all the organoleptic tests were acceptable.

The addition of tween 80, glycerin, PEG400 decreased pH value of the product, since tween 80 had low pH (Taurina et al., 2021). Conversely, triethanolamine (TEA) in low amount enhanced product pH significantly because it had 10.5 in pH value. The balance between these components majorly contributed to the CGW face serum pH value.

The pH of the CGW face serum must be on the range between 4.1 – 5.8 to prevent stratum corneum disruption and causing irritation like atopic dermatitis, ichthyosis and dry skin (Draelos, 2018; Patil et al., 2019; Proksch, 2018). Since the pH of the CGW face serum was 5.578 ± 0.108 after 4 weeks testing (figure 2), the product can be justified as safe product for face application in terms of pH acceptability. Moreover, the significant change of pH also can be used as measure product stability. Significant pH change caused by several factors, microbial contamination and substance incompatibility. The growing microbe might produce acid metabolic that decreased pH significantly, so some preservative was needed to prevent microbial contamination. On the other hand, chemical reaction between each substance may altered pH change caused by release of H+ ion in the product.

The viscosity, HPMC formed hydrogen bridge that absorbs water from face serum continuously and tend to increased viscosity of the product (Niazi, 2016; Taurina et al., 2021). If the amount of HPMC added was too high, the thick and gel-like formulation will be obtained, but in this study, there was no significant viscosity increase caused by excess amount of HPMC. The accelerated

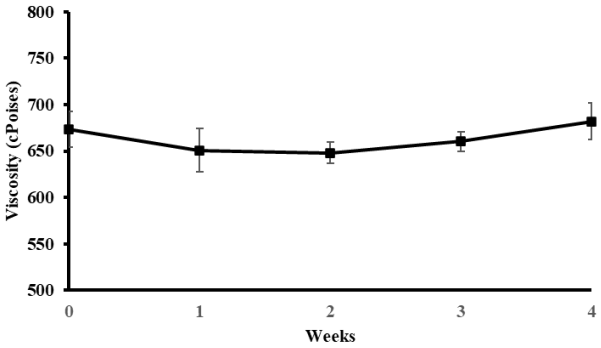

Figure 3. The viscosity stability profile in accelerated study test

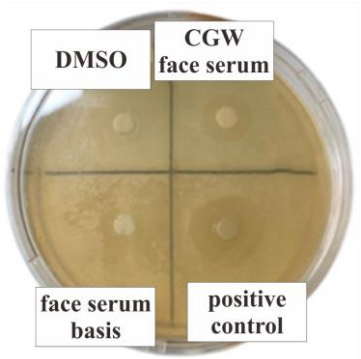

Figure 4. Antibacterial of the CGW face serum using disk diffusion method

stability test can be observed in figure 3. Based on the figure 3, after 4 weeks there were no significant change in viscosity. After 4 weeks of test, the CGW face serum had 681.95 ± 20.01 cP in viscosity.

Antibacterial activity of CGW face serum

The result of the antibacterial activity of the CGW face serum can be observed in figure 4. There were clear spots around the disk from the CGW face serum, positive control and face serum basis, indicated potent antibacterial activity. Meanwhile, in DMSO disk, there was no clear zone observed. The positive control was used in this research was marketed face serum that had claim for anti-acne properties and exhibited 18.91 ± 1.77 mm of inhibition zone. Meanwhile, the CGW face serum showed antibacterial activity with 15.30 ± 4.03 mm inhibition zone. The larger of the inhibition zone, the stronger its antibacterial activity. The addition of viscosity enhancer, co-solvent, and other components were not eliminate antibacterial activity of CGW extract. Even though the extract was formulated in viscous formulation, the active ingredients like phenolic, caffeine, chlorogenic acids and caffeic acids of CGW extract can be released from the face serum basis and promoted antibacterial activity (Irianto et al., 2023; Ludwig et al., 2014; Singh Arora et al., 2009).

CONCLUSION

To sum up, both of coffee ground waste extract and its face serum formulation exhibited antibacterial activity with 0.860 gram/mL MIC50 and 15.3 ± 4.03 mm inhibition zone against Cutibacterium acnes. The coffee ground waste face serum was stable after 4 weeks testing using accelerated stability test in terms of organoleptic, pH and viscosity.

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