Effectiveness of sunscreen on *galangal* (*Alpinia galangal*) crude extract using the microwave-assisted extraction method

C W Rizkita and Sukardi

Department of Agroindustrial Technology, Faculty of Agricultural Technology Universitas Brawijaya

Email: citrawahyur@student.ub.ac.id

Abstract. Sunscreen is widely used in cosmetics as skin protection to reduce or prevent damage caused by ultraviolet (UV) radiation. One natural ingredient that has sunscreen potential is *Alpinia galangal* which has anti-UV properties. *Galangal* contains flavonoid compound which has a significant role in warding off UV rays. At the same time, it also includes other compositions that have an anti-UV such as phenolic, saponin, alkaloid and terpenoids. The purpose of this research was to determine the best extraction condition and effectiveness of *galangal* extracted sunscreen. The sample of *galangal* was extracted using microwave-assisted extraction (MAE) method with ethanol as a solvent. The resulting extract was then used for sunscreen cream and physically tested to identify the effectiveness, indicated by SPF and irritation tests. The yield extract obtained was around 5.5% for 2 hours, with 500 W for power and ethanol ratio 1:3. The *galangal* extract Sunscreen has an SPF value of approximately 15, categorised in the high protection category, and the physical properties are stable. In this study, it was concluded that *galangal* extract has anti-UV properties, and the irritation test proved that sunscreen with *galangal* extract cause no irritation symptoms and was considered safe for use on normal skin.

1. Introduction

*Alpinia galangal* is found in Indonesia with a slightly spicy aroma and taste. *Galangal* extract is 0.8–2% [3], consisting of flavonoids, terpenoids, saponins, and phenolics [12]. *Galangal* extract has benefits such as for ingredient in the food industry, medical purposes, cosmetics, antioxidants, anti-acne, anti-UV, and so on. *Galangal* extract can be produced through microwave-assisted extraction (MAE). MAE process is influenced by temperature, boiling point, solubility, and microwave power as well as the extraction time, affecting the amount of yield. The advantage of the MAE method is less time consumed with more yield production. Hence, it is more energy-saving [9].

Currently, people are starting to realise the importance of skin protection due to the presence of UV rays which can cause skin problems such as darkening, premature aging, and red spots to skin cancer [11]. Sunscreen creams can absorb at least about 85% of sunlight containing UV-A and UV-B [6]. Thus, sunscreens are sought after by the people, especially those with natural ingredients that are in higher
demand. *Galangal* extract can be used as an anti-UV on sunscreen. *Galangal* extract contains complex aromatic compounds that can be used as an organic sunscreen product. There have been many studies on extraction using the MAE method, resulting in high yields. This study aimed to determine the yield components and optimal conditions of *galangal* extract and to determine the effectiveness and concentration of *galangal* extract as a sunscreen.

2. Materials and methods

2.1. Raw material preparation
The materials used were *galangal* and 96% ethanol as a solvent. *Galangal* rhizome was dried to reduce the moisture content. The rhizome was dried for two days, then cleaned and dried, and cut into 0.5 cm in size. The sample size is one of the factors that need to be considered since the smaller particle size increases the efficiency of the extraction process.

2.2. *Galangal* extraction with MAE
The extraction of *galangal* rhizome was carried out using the microwave-assisted extraction (MAE) method. The chopped *galangal* rhizome was weighed according to the predetermined ratio of ingredients and solvents at ratio 1: 2, 1: 3, and 1: 4. Then the chopped *Galangal* was put into MAE which already contained 1 L of 96% ethanol solvent. MAE was carried out on predetermined microwave power at the variation of 400, 500, and 600 watts during the time according to the variation, 30 minutes, 1 and 2 hours respectively. The heating time is needed to reach the appropriate temperature to start the degradation process [10]. In this MAE method used ethanol as a solvent. Ethanol solvents are used to increase solvent extraction, which has a tan of more than 0.1 [13]. In MAE extraction, ethanol solvent was used because it has low polarity for extraction of the compounds like flavonoids, terpenoids, phenolics and saponin, and other compounds in *galangal* [8]. The MAE process is carried out by immersing the sample in a solvent in a vessel and irradiating it with microwaves to generate heat directly in the material. The ability of a solvent to absorb microwave energy and convert it into heat depends on its dissipation factor (tan) which can be found by Equation 1 where the capacity is polarised by the electric field divided by the dielectric loss. So that the higher the dissipation factor, the greater the amount of heat energy produced. Based on equation 1, $\varepsilon'$ is the dielectric constant of solvent which reflects the capacity to be polarised, and $\varepsilon''$ is the dielectric loss factor related to the efficiency of the transformation of electromagnetic energy into heat. So, the higher the dissipation factor, the greater the heat energy will be produced [14].

$$\tan \delta = \frac{\varepsilon'}{\varepsilon''} \tag{1}$$

After obtaining the extract, then the solvent and the extract is separated using a rotary evaporator. After receiving the *galangal* extract, GC-MS and UV-Vis tests were carried out to determine the composition of the *galangal* extract.

2.3 Testing of antioxidant activities and sunscreen effectiveness
The *galangal* extract was diluted and then added with DPPH solution, which had previously been diluted with 0.025g / L methanol. The mixture was left to rest for 20 minutes at room temperature. After obtaining a homogeneous mixture, continued with measuring the absorbance at 515nm with UV-Vis to determine the DPPH inhibition (%). Compared with the blank solution.

The effectiveness of the sunscreen of *galangal* extract was tested by dissolving it in 96% ethanol and made in a concentration of 0.02% and then measured in wavelength at 290-320nm with UV-Vis. Compared with the blank solution. After that, the sunscreen cream can be made. In anti-UV testing, SPF (Sun Protection Factor) is an indicator that shows the effectiveness of a product. The higher the SPF value, the more effective it is to protect the skin from the effects of UV rays. SPF can be interpreted as the amount of UV energy needed to cause MED (Minimal Erythema Dose) in the active ingredients of sunscreen that is protected by the skin compared to the amount of energy required to cause MED without sunscreen protection as shown in equation 2 [15].
\[
SPF_i = \frac{MED_{IP}}{MED_{IU}}
\]

2.4 Irritation test and photoprotector activity

After the sunscreen cream was made, the skin irritation test was carried out for three consecutive days. Photo protector activity test was also used to measure the SPF value using an SPF Spectrometer.

3. Results and discussion

3.1 Galangal extraction with MAE

The dried galangal rhizome was used to reduce the moisture to increase the oil produced. Water escaped to the surface and evaporated during the drying process so that yields are higher [3]. Besides, chopped galangal can increase the yield compared to intact galangal because galangal that has been cut into small pieces will have a larger surface diameter so that the contact with microwaves and solvents will be more significant, so the extract will be easier to take [2].

Furthermore, MAE is heated faster than conventional heating processes. Microwave radiation is used for the extraction of organic compounds to speed up sample preparation, allow for the use of multiple samples simultaneously and use less volume of solvent and reduce residue. Microwave heating can distribute even heat more to all parts, so that heat utilisation is more efficient [5]. The solvent used was ethanol which has polar properties and can extract the complex compounds in galangal. Ethanol has strong hydrogen bonds, so it is hygroscopic and can dissolve in ionic compounds. Extraction with solvents will produce more oil because the oil will be bound together with the solvent, and there is less possibility of oil evaporation [3].

In microwave extraction of galangal, the yield increase with the increase in time. The yield increase is also influenced by the ratio of ingredients and solvents, microwave power and extraction time. For microwave power, the 500W was the most suitable power obtained, as shown in Figure 2. The high microwave power can cause thermal degradation and reduce yield [1]. The higher power level can break galangal cells so that the targeted compounds can be obtained. The moisture in the cell will absorb microwaves which cause internal heating in the cell, which result in significant pressure on the cell wall [2].

![Figure 1. MAE process.](image-url)
Microwave heating can produce high yields because the heat will be more evenly distributed so that it is more effective in utilising energy for galangal extraction. It is known that the yield tends to increase during the irradiation time, but higher exposure to microwaves can cause degradation of the components [1]. Within ±2 hours, the yield was the highest as Figure 3.

However, when the extraction time was longer, there is a risk of degradation of the extract components. For the ratio of ingredients and solvent, the most effective was at 1:3 because at a greater ratio, the solvent is saturated. The more the ratio of materials and solvents used does not necessarily result in a higher yield. The GCMS results showed that the galangal contained more than 15 volatile components, including flavonoids, terpenoids, saponins, phenolics, galangin, 1,8-cineol and other essential oil compounds. The main components of the galangal extract are flavonoids and phenolic.

3.2 Antioxidant testing, sunscreen effectiveness and irritation test
DPPH antioxidant testing is used to determine free radicals contained in antioxidants. Phenolic components have antioxidant activity. Also, flavonoids have a role in antioxidant activity that can reduce free radicals, including O²⁻, H₂O², OH⁻. Alkaloids also can break the chain of reactions from free radicals [7]. The presence of antioxidant compounds can prevent diseases caused by UV radiation. The DPPH value of galangal extract was 10.50 mg/ml.
The sunscreen with the highest concentration of galangal extract had the greatest effectiveness. In testing the effectiveness of sunscreens, the SPF value is still in the range 15-20, so it is classified as moderate protection for sunscreens. This effectiveness is obtained because galangal extract contains relatively high flavonoids which function as an antidote to free radicals in the skin. So the skin can be protected UV radiation. The SPF value is around 15 and is included in moderate protection. The higher the SPF value, the more effective it is to protect the skin from the effects of UV rays. The flavonoid compound has the potential as sunscreen due to the presence of a chromophore group which is a conjugated aromatic system that causes the ability to absorb the light around UV light wavelengths. The smaller the IC<sub>50</sub>, the antioxidant activity and the SPF value are higher [7].

Irritation test experimented on mice, sunscreen is applied on mice skin and then observed for any sensitivity, edema and erythema. It is known that there are no signs that indicate sensitivity, erythema and edema, conforming that the sunscreen with natural ingredients galangal extract is safe to use.

3.3 Galangal extract characteristics
The characteristic of galangal extract is it has a light-yellow colour resulting from methyl cinnamate and terpenoid compounds shown in Figure 3. The resulting aroma is a distinctive aroma of galangal, which is slightly pungent. Other parameters that can be determined are the average density of galangal extract of about 0.8 g / ml and the refractive index of around 1.48.

![Figure 4. Colour of galangal extract.](image)

4. Conclusions
Galangal extraction using 500W microwave-assisted extraction method with 96% ethanol solvent can produce high yields. The longer the extraction time, the more yield is produced. The high yield is also influenced by the high ratio of ingredients and solvents and microwave power. Galangal extract also has a composition that can be used as anti-UV and also has antioxidant properties. Because of these two properties, galangal extract has the potential to be used as a sunscreen mixture, and it is shown that the sunscreen with galangal extract has a moderate protective effect and is safe to use.

References
[1] Akbari S, Nour H A, Rosli M Y 2019 Optimisation of saponins, phenolics and antioxidants extracted from fenugreek seeds using microwave assisted extraction and response surface methodology as an optimising tool J Comptes Rendus Chimie 22 714-727
[2] Akhtar I, Sumera J, Madeeha A, Nadia G, Amina T 2020 Process optimisation for microwave assisted extraction of Foeniculum vulgare Mill using response surface methodology J Science 32 1451-1458
[3] Ermiat, Basuni H, Gatot P 2004 Kajian teknologi ekstraksi ekstrak lengkuas merah (Alpinia galangal L.Swart) (The study of red galangal (Alpinia Galangal L. Swart) extract extraction technology) J Agribisnis dan Industri Pertanian 3 3 34-41 [In Indonesian].
[4] Fauruschou, A, Wulf, H. C 2007 The Relation Between Sun Protection Factor and Amount of Sunscreen Applied in Vivo Dermatology. 156 716-719.
[5] Feriyanto Y E, Patar J S, Mahfud, Pantjawani P 2013 Pengambilan Minyak Atsiri dari Daun dan Batang Serai Wangi (Cymbopogon winterianus) Menggunakan Metode Distilasi Uap dan Air dengan Pemanasan Microwave (Extracting essential oils from the leaves and stems of...
citronella (Cymbopogon winterianus) using the steam and water distillation method using microwave heating. \textit{J Teknik Pomits} \textbf{2} \textit{1} 2337 – 3539 [In Indonesian]

[6] Geraldine E T, Ema D H 2018 Formulasi krim tabir surya ekstrak buah parijoto (\textit{Medinilla speciosa Blume}) dan uji nilai SPF secara in vitro (The sunscreen cream formulation of parijoto fruit extract (Medinilla speciosa Blume) and the in vitro SPF value test). \textit{J Farmasi Sains dan Komunitas}. \textbf{15} \textit{2} 92-98 [In Indonesian].

[7] Khairi N, Suryani A, Khairuddin D, Gemini A 2018 The determination of antioxidants activity and sunblock \textit{Sterculia Populifolia} extract-based cream. \textit{J Pharmaceutical and Biomedical Research}. \textbf{4} \textit{1} 20-26.

[8] Mandal S C, Vivekananda M, Anup K D 2015 Essential of Botanical Extraction (Amsterdam: Elsevier).

[9] Morais S 2013 Ultrasonic and microwave-assisted extraction and modification of algae components \textit{Functional Ingredients from Algae for Foods and Nutraceuticals} ed Herminia D (Cambridge: Woodhead Publishing) pp 585-605

[10] Seoane R P, Florez-Fernandez, N, Conde P E., Dominguez G H 2017 Microwave-Assisted Water Extraction \textit{Water Extraction of Bioactive Compounds} ed Herminia D G, Maria J G M (Amsterdam: Elsevier Inc) pp 163-198.

[11] Sopyan I, Dolih G, Sylvia T 2017 Formulation of tomato extract (\textit{Solanum lycopersicum L.}) as a sunscreen lotion \textit{J Physiology, Pharmacy and Pharmacology} \textbf{8} \textit{3} 453-458.

[12] Tang X, Changmou X, Yavuz Y, Amarat S, Maurice R M 2018 Phytochemical profiles, and antimicrobial and antioxidant activities of Greater \textit{Alpinia galangal} (\textit{Alpinia galangal} (Linn)) Swartz flowers \textit{J Food Chemistry} \textbf{225} 300-308

[13] Orsat V, Winny R 2017 Microwave-Assisted Extraction of Flavonoids \textit{Water Extraction of Bioactive Compounds} ed Herminia D G, Maria J G M (Amsterdam: Elsevier) pp 221-244

[14] Vernes L, Maryline V, Farid C 2020 Ultrasound and microwave as green tools for solid-liquid extraction \textit{Liquid-Phase Extraction} ed Colin F P (Amsterdam: Elsevier) pp 355-374

[15] Yanti E, Aprilita R, Ratih D P, Irvani R, Tyas P U 2018 In-Vitro and In-Vivo Sunscreen Activity of Active Compounds Isolated from Fruits of \textit{Phaleria marcocarpha} (Scheff.) Boerl \textit{J Young Pharm}. \textbf{10} \textit{2} 106-110