

Microencapsulation of Green Cardamom (*Elettaria cardamomum*) Essential Oil with Maltodextrin and Its Applications in Coffee Drink

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Article History :

Received : 30 July 2022

Received in revised form : 23 September 2022

Accepted : 29 September 2022

Keywords :

Cardamom,
Essential oil,
Herbal coffee drink,
Microencapsulation.

ABSTRACT

Microencapsulation of green cardamom (*Elettaria cardamomum*) essential oils and its application is considered an innovative technique to improve the functional properties of coffee drinks. This study aims to determine the best formulations (M) of combination of maltodextrin and cardamom essential oil, parameters such as microcapsule yields, water content, and solubility were measured. To find out whether the microcapsule was applicable for coffee drink, antioxidant activity and taste and aroma of coffee drink mixed with the best formulation were analyzed. Completely Randomized Block Design (CRBD) with four replications was used; the data were analyzed by ANOVA and further tested by the LSD test with a level of 5%. The results showed that formulations significantly affected the yields and solubility of cardamom essential oil microcapsules. The best formulation treatment was formulation M3 with a yield score, water content and solubility of 89.03%, 11.85%, and 93.50%, respectively. Antioxidant activity, aroma and taste scores of ground coffee mixed with M3 were $77.61 \pm 0.23\%$, 4.31 (typical of cardamom, little coffee), and 4.88 (typical of cardamom, little coffee), respectively. While ground coffee only has antioxidant activity of $76.32 \pm 0.31\%$. In conclusion, ground coffee mixed with microencapsulated cardamom essential oil has the potential as a herbal coffee drink.

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1. INTRODUCTION

Cardamom (*Elettaria cardamomum*) is plant of Zingiberaceae family and as one of the spices produced in Indonesia. The cardamom essential oil is generally used in industry, such as the food, beverage, pharmaceutical, as aroma and flavoring component (Singh *et al.*, 2018). The volatile oil contains about 1.5% α -pinene, 0.2% β -pinene, 2.8% sabinene, 1.6% myrcene, 0.2% α -phellandrene, 11.6% limonene, 36.3% 1,8-cineole, 0.7% γ -terpinene, 0.5% terpinolene, 3% linalool, 2.5% linalyl acetate, 0.9% terpinen, 2.6% α -terpineol and 31.3% α -terpinyl-nerolidol (Korikontimath *et al.*, 1999; Al-Zereini *et al.*,

2022). The main compound of cardamom are 1, 8-cineole (representing 50% or more), with smaller amounts of limonene, α -terpenyl acetate, α -terpineol, borneol, camphor and α -pinene. Although widely used in various fields, essential oils are susceptible to oxidative deterioration and loss of volatile compound, especially when exposed to heat, oxygen, and moisture (Petrovic *et al.*, 2010; Calvo *et al.*, 2012). The quality of a product fortified with the oil may deteriorate due to oxidative degradation, formation of off-flavor, and the generation of free radicals. These changes have negative effect on the shelf life stability, sensory properties and overall acceptability (Bakry *et al.*, 2016). Microencapsulation technology could be a viable option to maintain their biological and functional characteristics of cardamom essential oil.

Microencapsulation is a technique in which tiny particles or droplets are surrounded by a coating wall, or are embedded in a homogeneous or heterogeneous matrix, to form small capsules (Silva *et al.*, 2014). It can envelope a core material which was originally a liquid or gaseous substances within another substance i.e. a solid form in a very small sealed capsule. The core material is gradually diffused through the capsule wall, offering controlled release under desired condition (Fang & Bhandari, 2010). Therefore, microencapsulation technique can be used to deliver bioactive components, improving their handling properties. Wall composition and microencapsulation technique may determine functional properties and potential applications of encapsulated components. Wall materials have to be able to provide a thin layer that is cohesive with the core material, is chemically miscible, inert, and has suitable properties for coating purposes. Generally, carbohydrates include gelatin, lactose, sucrose, maltodextrin, pullulan, whey protein and gum arabic are used as wall material applied to foodstuffs in the microencapsulation process (Koç *et al.*, 2010). Maltodextrin has advantages in the form of low viscosity and high solubility, as well as having high binding power in forming a product matrix (Balasubramani *et al.*, 2015). Microencapsulation of cardamom essential oil with maltodextrin may protect distinctive taste and aroma, be easily dissolved in brewing, and provide more hygienic when it is applied to coffee drink.

Indonesia is listed as the third largest coffee producer in the world after Brazil and Vietnam. The consumption of coffee increased along with the increase in coffee production. Generally, coffee drinks are sold in the market in the form of instant coffee with added sugar, milk, or vegetable fat (creamer). Development of new flavor of coffee such as herbal coffee can enhance the coffee. Herbal coffee is a type of coffee drink that has herbs added to it. The mixture of coffee and herbs or spices may produce coffee drink with such a taste, aroma of herbs while providing health benefits which may be attractive to consumers. The purpose of this study was to determine the best formulation of maltodextrin as coating material in the microcapsule production of cardamom essential oil, to find out the most panelist acceptability of coffee when it was mixed with cardamom microcapsule, and to measure the antioxidant activity of the coffee mixed with cardamom microcapsules.

2. MATERIALS AND METHODS

2.1. Materials

Materials used were acetylated maltodextrin MOR-REX™ 1910 with 10-12 DE (MD) and cardamom essential oil (EO) of food grade, and super quality of Robusta coffee of Ulu Belu, Lampung production. This research was conducted using Completely Randomized Design (CRD) with 4 replications with one factor of variation of maltodextrin levels used in the emulsion. The combinations of essential oil and maltodextrin levels (v/w) were

M1 (1:2.5), M2 (1:5), M3 (1:7.5), M4 (1:10), M5 (1:12.5). Differences in mean values were analyzed by analysis of variance (ANOVA) and then tested with Least Significant Difference (LSD) using IBM SPSS Statistics 22 software ($\alpha = 0.05$).

2.2. Microencapsulation Process

Microencapsulation of cardamom essential oil (EO) was carried out based on modified procedure [Yuliasari *et al.* \(2016\)](#), focusing on the amount of coating material used. The first stage is making an emulsion between cardamom essential oil and maltodextrin coating material, (essential oil: maltodextrin) 1:2.5, 1:5, 1:7.5, 1:10, and 1:12.5. From the formulation, into each of 10, 20, 30, 40 and 50 g of maltodextrin were added with 100 mL of distilled water while stirring using a magnetic stirrer at a speed of 500 rpm for 15 minutes at 55 ± 2 °C. Following, 4 mL of cardamom EO was added into the solution of maltodextrin and aquades, and stirred using a magnetic stirrer (78HW-1, Biomixer, Sao Paulo, Brazil) for 4 hours at a speed of 500 rpm at room temperature. To prepare of microcapsule of cardamom EO, the mixtures were placed in 100 mL sealed glass bottles then prefrozen in refrigerator at -20 °C for 4 hours. The microcapsule of cardamom EO was then frozen in a vacuum freeze-dryer at -52.2 °C (Liotop L101, Liobras, São Carlos, Brazil) for 56 h. The microcapsule of cardamom EO was analyzed for the yields, solubility, and moisture content.

2.3. Observation and Measurements

2.3.1. Moisture Content and Water Solubility

Moisture content was determined gravimetrically and measured through constant weight method with a Petri dish at 105 °C for 1 h ([AOAC, 2005](#)). The Petri dish was dried with a constant weight (m_1). Microcapsule samples weighing (1 g) a mass of m_2 were added and then it was dried in an oven for 3 h until constant weight. The final dried total weight of Petri dish was weighed as m_3 . Moisture content (MC) was calculated as:

$$MC = \frac{m_3 - m_1}{m_2} \times 100 \quad (1)$$

Water solubility of encapsulated oil was determined according to [Xu *et al.* \(2022\)](#). One gram of microcapsules (as a) was dispersed in 20 mL of distilled water and then stirred using a magnetic stirrer for 5 min. Subsequently, the mixture was filtered using a vacuum filter. The filter paper was dried at 105 °C for 30 minutes before use and weighed (as b). Filter paper along with the residue was dried again in the oven (Conthern, NZ) at 105 °C to a constant weight for approximately 3 hours (as c). Water solubility (WS) was calculated using equation (2).

$$WS = 100 - \left(\frac{c - b}{a} \right) \times 100\% \quad (2)$$

2.3.2. Yield of product ([Salimi *et al.*, 2012](#))

The yield of the product is the overall product of the microcapsules which is calculated from the mass of the microcapsule product from the total solids (encapsulation material and core material) then calculated in percent.

$$Y (\%) = \frac{\text{microcapsules weight (g)}}{\text{total weight of solid (g)}} \times 100 \quad (3)$$

2.3.3. Antioxidant Activity

Antioxidant activity analysis was carried out following the procedure performed by [Ismail et al. \(2012\)](#) and [Shimamura et al. \(2014\)](#), which has been modified with using the DPPH (1,1-diphenyl-2-picrihydrazyl) method. The sample was prepared by weighing 0.1 g and adding 2 mL of 96% ethanol, vortexes until dissolved. Then 0.1 mL of the solution was put into a test tube which was covered with aluminum foil. Then 1 mL of 0.2 mM DPPH solution was added rapidly (made by weighing 0.0078 g of DPPH powder and adds ethanol to a volume of 100 ml). Then the solution is homogenized vortex for 60 seconds. The blank solution is 1 mL 0.2 mM DPPH was pipetted into a test tube and 0.1 ml of ethanol was added. The solution was incubated in the dark at room temperature for 30 minutes, then pipetted in a cuvette to be read absorbance at a wavelength of 517 nm using a spectrophotometer.

The decrease in the absorption value of the DPPH solution after the addition of the sample is a measure of the antioxidant capacity of the sample. The absorbance of the DPPH solution is the control absorbance (A_k). Solution absorption sample is as sample absorbance (A_s). DPPH solution absorbance value and the sample solution was calculated as the percent antioxidant activity with the formula as follows:

$$RSA = \left(\frac{A_k - A_s}{A_k} \right) \times 100\% \quad (4)$$

where *RSA* is radical scavenging activity in %.

2.4. Sensory analyzes

Firstly was preparation of mixed of ground coffee and microcapsules. The coffee beans were ground using hand mill, and as much as 20 g ground coffees was mixed with 5 g of cardamom essential oil microcapsules, and then well mixed ([Febrianto et al., 2015](#)). A 5 g of the mixture were brewed with 75 mL of boiling water in a coffee jar and swirling it around for 5 sec and kept in for 5 min. Secondly, coffee drink sample was served in coffee cup. Approximately 50 mL of coffee drink sample was presented per cup. The coffee drink was analyzed for taste and aroma, as well as antioxidant activity.

Assessment of aroma and taste was done by using a scoring test, while Multiple comparison test was to determine the comparison between cardamom coffee and control coffee (original coffee, without addition of cardamom EO microcapsules) ([Soekarto, 1985](#)). Each panelist was given a sample containing one control sample (Mo) of original coffee and 5 cardamom EO coffees of various formulations. A total of 10 untrained panelists from Dr. Coffee Lampung were asked to evaluate cardamom coffees and compare each of them with control of original coffee. The evaluation included cardamom coffee assessment was better, equal to, or worse than control of original coffee. The level of differences in multiple comparison sensory tests was shown in Table 1. The score for aroma and taste in the scoring test was 1-5 (score 5 = typical of cardamom, 4 = typical of cardamom and a little coffee, 3 = balanced of cardamom and coffee, 2 = typical of coffee, a little cardamom, 1 = typical of coffee) ([Lim, 2011](#)).

Table 1. Degree of difference in multiple comparison sensory tests

Differences	Comparison scale	Numerical scale
Better than R	Much better than R	1
	Better than R	2
Same as R	The same as R	3
Worse than R	Worse than R	4
	Much worse than R	5

3. RESULTS AND DISCUSSION

3.1. Yield, Water Content, and Solubility

The analysis of variance showed that the formulation of cardamom EO with maltodextrin had a significant effect on the yield of cardamom essential oil microcapsules produced (Table 2). It showed that the formulation of M4 was not significantly different from M3, M2, and M1, but significantly different with M5. The highest yield was found in the formulation M4 of 90.27%, while the lowest yield was found in the formulation M5 of 79.36%. The yield of the microcapsule product was determined by comparing the mass of the dried microcapsules with the total active ingredients and coating materials used. It considers both the mass and the pore spaces. Therefore, larger and non-uniform particles require more volumes and have lower yields. The use of maltodextrin as wall component of microencapsulation could serve as a mass enhancer, so the more maltodextrin added, the higher the yield was. Another parameter that can affect the yields is moisture content. High moisture content causes more adhesion of particles, and resulting in decrease of the yield measurement. This was occurred on formulation M5 of which with the addition of high maltodextrin. [Salami *et al.* \(2018\)](#) reported same result. [Yuliawaty & Susanto \(2015\)](#) reported that high used of maltodextrin increased the yields.

On the other hand, the formulation did not significantly affect the moisture content of the cardamom EO microcapsules (Tabel 2). Moisture content is an important characteristic of microcapsules as it relates to their viscosity, storage stability and the ease flow characteristic ([Premi & Sharma, 2017](#)). The moisture content the microcapsules in our study ranged from 10.63-12.15%. The high moisture content in this study could be caused by characteristic of maltodextrin used as coating materials and the water-retention capacity of cardamom EO microcapsules. The ability of maltodextrin to bind water was influenced by the value of DE and the hydrophilic groups ([Balasubramani *et al.*, 2015](#)). Maltodextrin with low DE is non-hygroscopic, while maltodextrin with high DE tends to absorb water (Phisut, 2012). In this study, the maltodextrin used was high DE maltodextrin (DE 10-12). In addition, the hydrophilic groups of maltodextrin on the surface of the product enhance the binding water from the air. This study was not in agreement with [Purnomo *et al.* \(2014\)](#) reported that natural dye microencapsules coated with maltodextrin, whey and carrageenan have water content ranging from 4-6%. The high moisture content of cardamom EO microcapsules in our study may not meet the recommended moisture content for dry powder, which should be <8%. High moisture content may shorten the storage time and also accelerate lipid oxidation rate and produces a peculiar smell ([Ghasemi *et al.*, 2017](#)).

The results of the analysis of variance showed that the formulation of cardamom EO and maltodextrin had a significant effect on the solubility of the cardamom EO microcapsules (Tabel 2). The M4 formulation was not significantly different from the formulation of M3 and M5, but significantly different from the formulation of M1 and M2. The highest solubility was found in the formulation of M4, 96.00%, while the lowest was in the formulation of M1, 90.50%. Overall, the solubility value of microcapsules was very high, above 90%. Increasing maltodextrin concentration had significance effect on the solubility of the cardamom EO microcapsules. This result attributed to the presence of the carbohydrate-based wall material maltodextrin, which increased the hydrophilic sites in the capsule samples. These hydrophilic groups (-OH) increased the solubility of the particles by promoting the interaction between

water and the microcapsule molecules. This study was in line with [Xu et al. \(2022\)](#) reported that solubility of tea oil powder with ranged from 77.16-87.13% which as a result of using maltodextrin as microcapsule wall. Solubility is one of the important parameters in the manufacture of microcapsules, the higher the solubility, the easier it is to release the active ingredient from the microcapsules so that it is easier to apply.

Tabel 2. Least significant difference (LSD) at 5% level on the yield, moisture content (MC), and solubility (WS) of cardamom essential EO

Formulation	Yields (%)	MC (%)	WS (%)
Cardamom EO: maltodextrin			
M5 (1:12.5)	79.36 ± 4.67 ^b	10.93 ± 1.2	93.25 ± 1.00 ^{ab}
M4 (1:10)	90.27 ± 2.76 ^a	10.63 ± 2.3	96.00 ± 1.91 ^a
M3 (1: 7.5)	89.03 ± 5.31 ^a	11.84 ± 1.13	93.50 ± 2.5 ^{ab}
M2 (1: 5)	86.69 ± 5.72 ^{ab}	12.14 ± 3.1	90.75 ± 1.82 ^b
M1 (1: 2.5)	80.21 ± 7.75 ^{ab}	11.54 ± 3.2	90.50 ± 2.21 ^b

Note: The numbers and the standard deviation in the same column followed by the same letter indicated no significantly different in the 5% LSD.

3.2. Sensory Analyses

Sensory analyses of cardamom coffee were conducted by using the scoring method and multiple comparison tests. Parameters observed by the scoring method included aroma and taste. The multiple comparison method is used to determine the comparison of the treatment sample with the control on the sensory attributes of taste and aroma ([Soekarto, 1985](#)). Each panelist was given a sample containing one standard sample (R) of ground coffee without the addition of cardamom, and 5 samples of ground coffee mixed with cardamom microencapsulation M1, M2, M3, M4, and M5. A total of 20 semi-trained panelists (coffee drinkers) were asked to evaluate the aroma and taste of the sample coffee and compare it to the control ground coffee. The aroma and taste assessment included whether it was better, equal to, or worse than the aroma and taste of the control ground coffee. The level of difference in the multiple comparison sensory test is presented in Table 1.

3.2.1. Score of Aroma

The results of the analysis of variance showed that the formulation of cardamom EO with maltodextrin had a significant effect on the aroma score of the cardamom coffee drink (Table 3). The formulation of M2 was not significantly different from the M3 and M1, but was significantly different from M4 and M5. The highest score of aroma was found in M2 of 4.6 (Typical cardamom, a little coffee), while the lowest aroma score was found in M5 of 3.75 (cardamom and coffee balanced). The distinctive aroma score of cardamom produced in cardamom coffee could derive from addition of microcapsules of cardamom EO. This may be caused by cineol, terpene, terpineol and borneol compounds of cardamom EO. Generally they are used as a flavoring agent ([Singh et al., 2018](#)). Aroma is the smell caused by chemical stimuli that are smelled by the olfactory nerves in the nasal cavity ([Lim, 2011](#)).

3.2.2. Score of Taste

Taste is the main factor for determining consumer acceptance and plays an important role in making decisions about the level of preference for a product. The results of the analysis of variance showed that the formulation of cardamom EO with maltodextrin

had a significant effect on the score of taste of the cardamom coffee drink (Table 3). The formulation of M3 was not significantly different from the formulation of M2, but significantly different from the M4, M5 and M1. The highest taste score was in the formulation of M3, 4.88 (typical cardamom, a little coffee), while the lowest score was in the M1, 4.00 (Typical cardamom, a little coffee). The distinctive taste of cardamom produced in cardamom coffee could come from the essential oil. Essential oils contain volatile mixtures and non-volatile mixtures that cause characteristic aroma and taste (deNascimento *et al.*, 2020).

Table 3. The LSD at 5% level on aroma and taste of cardamom EO microcapsules

Formulation	Aroma	Taste
Cardamom EO: maltodextrin		
M5 (1:12.5)	3.75 ± 0.28 ^c	4.31 ± 0.14 ^{bc}
M4 (1:10)	4.0 ± 0.20 ^{bc}	4.37 ± 0.14 ^{bc}
M3 (1: 7.5)	4.31 ± 0.12 ^{ab}	4.87 ± 0.2 ^a
M2 (1: 5)	4.62 ± 0.14 ^a	4.68 ± 0.23 ^b
M1 (1: 2.5)	4.25 ± 0.2 ^{ab}	4.0 ± 0.23 ^c

Note: The numbers and standard deviation in the same column followed by the same letter indicated no significant difference in the 5% LSD

3.2.3. Multiple Comparison Test

The comparison of cardamom coffee with control coffee was analyzed using the multiple comparison tests. The results of the LSD test at the 5% level on the difference between cardamom coffee and control coffee was shown in Table 4. It showed that the formulation of M4 was not significantly different from the M3, M5, and M1, but significantly different from M2. The highest value of multiple comparisons was found in M4, 4.75 (worse than R) (Table 1), while the lowest score was found in M2, 3.94 (same as R). The addition of cardamom EO microcapsules to ground coffee affected the panelists' preference for the acceptance of cardamom coffee. The panelists did not like cardamom coffee. This study was not in agreement with Febrianto *et al.* (2015) reported that panelists' preferences to cardamom herbal coffee was moderately like to like. Dalimunthe *et al.* (2021) reported that panelists' preferences were strongly influenced by the ripeness of cherries. Furthermore, Larasati *et al.* (2021) found that fermenting modified cherries using *S. cerevisiae* cocultures reduced the caffeine content that the panelists preferred.

Table 4. The LSD at 5% level on the difference between cardamom coffee and original coffee

Formulation (cardamom EO : maltodekstrin)	Multiple Comparison
M5 (1:12.5)	4.75 ^a
M4 (1:10)	4.59 ^{ab}
M3 (1: 7.5)	4.52 ^{ab}
M2 (1: 5)	4.19 ^{ab}
M1 (1: 2.5)	3.94 ^b
LSD (0,05) = 0,816	

Note: The numbers followed by the same letter indicated no significant difference in the 5% LSD.

3.3. Determination the Best Treatment

Determination of the best treatment for cardamom coffee was based on the results of the Least Significant Difference (LSD) test at 5% level on yield, solubility, sensory analysis. The recapitulation of the 5% LSD test was presented in Table 5. The M3 formulation treatment was determined as the best treatment because it had high yield and high solubility and a distinctive cardamom coffee aroma, of which was not significantly different from the M2 and M4. From the multiple comparison test, the lowest score was recommended because it was acceptable to consumers. In addition, yield and solubility are considered as important parameters. Aroma was also important because it produced a distinctive aroma of cardamom that attracts consumers. The best formulation treatment was applied to produce coffee cardamom.

Table 5. Determination of the best treatment based on LSD test on the yield, solubility and organoleptic parameters of cardamom coffee

Parameters	Formulations				
	M1	M2	M3	M4	M5
Yields	80.21ab	86.69ab	89.03ab	90.27a*	79.36b
Water solubility	90.50b	90.75b	93.50ab	96.00a*	93.25ab
Aroma	4.25ab	4.63a*	4.31ab	4.00bc	3.75c
Taste	4.00c	4.69ab	4.88a*	4.38bc	4.31bc
Multiple comparation	4.19ab	3.94b*	4.59ab	4.75a	4.52ab
Note	-	**	*	**	-

Antioxidants are inhibitors that work to inhibit oxidation by reacting with reactive free radicals to form unstable, unreactive free radicals. The antioxidant test method used in this study was the free radical DPPH method. DPPH is a free radical that can react with compounds that can donate hydrogen atoms. Antioxidant compounds can change the color of the DPPH solution from purple to yellow (Dehpour *et al.*, 2009). The antioxidant activity measured was cardamom coffee mixed with microcapsules with the best treatment being M3. The results of % inhibition activity in cardamom coffee of M3 and control coffee (without addition cardamom EO capsules were $77.61 \pm 0.23\%$ and $76.32 \pm 0.31\%$, respectively. This may because cardamom essential oil contains phenolic compounds such as cineol, terpineol, and borneol as antioxidants which function as scavengers of free radicals (Al-Zeremi *et al.*, 2022). It is difficult to explain which compounds have antioxidant activity of cardamom essential oil because of the chemical complexity of essential oils. However, Jena *et al.* (2021) reported that the presence of 1,8-cineole, camphene, -pinene, caryophyllene oxide as the main compound with free potential radical scavenger. 1,8-Cineole and -pinene present in the essential oil of *E. cardamomum* known to protect against hydrogen peroxide-induced oxidative stress in mouse pheochromocytoma cells (Wang *et al.*, 2019). Hamzaa & Osman (2012) reported that coffee mixed with cardamom could ameliorate the hazardous effects of oxidative stress due the phenolic compound in cardamom EO.

4. CONCLUSIONS

Formulation of cardamom EO and maltodextrin significantly affected the yields, solubility, but it did not affect moisture content of the cardamom EO microcapsules. In

addition, the formulations also significantly affected the sensory parameter. The best formulation was M3, cardamom EO mixed with maltodextrin at ratio 1:7.5. Ground coffee mixed with microencapsulated cardamom EO (cardamom coffee) M3 had antioxidant activity of $77.61 \pm 0.23\%$, while original coffee was $76.32 \pm 0.31\%$. Even though panelist preference to the cardamom coffee was low or did not like, microencapsulated cardamom EO can be mixed with ground coffee to produce herbal cardamom coffee drink which may have beneficial health effect.

ACKNOWLEDGMENT

This paper has been presented in the National Seminar of *Himpunan Ilmu Tanah Indonesia (HITI)* on July 7, 2022 in University of Lampung, Bandar Lampung, Indonesia.

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