Food Research 7 (5) : 53 - 60 (October 2023)
Journal homepage: https://www.myfoodresearch.com

FOOD RESEARCH

FULL PAPER

Optimization of coating materials on microencapsulation of red galangal (*Alpinia purpurata*, K. Schum) rhizome essential oil with freeze dry method

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Article history:
Received: 2 February 2022
Received in revised form: 10 March 2022
Accepted: 29 July 2022
Available Online: 13 September 2023

Keywords:
Essential oil,
Red galangal rhizome,
Microencapsulation,
Optimization

DOI:
https://doi.org/10.26656/fr.2017.7(5).033

Abstract

The potential use of large red galangal (*Alpinia purpurata,* K. Schum) essential oil has a weakness that is prone to compound damage. Thus, it is necessary to make efforts to maintain the active compounds contained in the red galangal rhizome essential oil, one of which is by microencapsulation. The purpose of this study was to determine the appropriate formulation of the material coating used in the microencapsulation process using the freeze-drying method. The optimization of the microencapsulation process was carried out using the response surface method (RSM) with the central composite design (CCD). The optimization results of essential oil microcapsules showed that the yield response of 44.01%, water content of 5.75%, TPC of 34.227 mg GAE/g, antioxidant activity (IC_{50}) of 136.01 ppm, and solubility of 87.70%. The GC-MS analysis has shown that there were five main compounds detected from the microcapsules, Diacetone alcohol of 31.36%, Chavicol of 15.26%, Methyl oleate of 6.87%, Alloaromadendrene of 5.82% and Methylhexadecanoate of 3. 44%. Overall, the choice of coating material can affect the characteristics and morphology of the microcapsules.

1. Introduction

Red galangal rhizome essential oil is an essential oil that can be obtained from the distillation of red galangal flowers, stems and leaves (Gülçin *et al*., 2012). The amount of demand for essential oils in the market is very high because they can be used in the pharmaceutical, food industry, agriculture, cosmetics, and other industries. In Indonesia, red galangal essential oil production is quite high because it is considered to have good quality with a eugenol content of around 70-90% (Atanasova *et al*., 2017; Nejad *et al*., 2017; Uddin *et al*., 2017).

The compounds contained in the essential oil of red galangal (*Alpinia purpurata,* K. Schum) consist of 1.8-cineole, -pinene, methylcinamat, and eugenol as the cause of the spicy taste of galangal (Sujono *et al*., 2019). Cineol (1.8-cineole) is a naturally occurring monoterpene, also known as eucalyptol. This compound is one of the main compounds in many plant essential oils. Cineol has been shown to have therapeutic benefits in inflammatory airway diseases, such as asthma and chronic obstructive pulmonary disease (Juergens, 2014). The potential use of large red galangal essential oil has the disadvantage that it is susceptible to compound damage due to oxidation reactions, storage temperature, UV light, and humidity (Petrović *et al*., 2013; Calvo *et al*., 2012).

Microencapsulation is one way to protect against the loss of active compounds that may occur in the storage process with a thin coating by certain coating materials (Nugraheni *et al*., 2016). The application of microencapsulation can prolong the oxidative stability of the capsule core and ensure a more controlled release of substances during the process (Özbek and Ergönül, 2017). According to Gharsallaoui *et al*. (2007), microencapsulation changes the liquid material into a solid to protect the core material from loss of flavour. One method that can be used in the microencapsulation process is freeze-drying because it can be carried out at very low temperatures. The processes that take place during freeze-drying are sublimation and desorption (Ogrodowska *et al*., 2017).

The microencapsulation process of red galangal rhizome essential oil will be carried out using the freeze-drying method. This method is relatively suitable for heat-sensitive materials because it can keep oxidative reactions to a minimum (Cano-Higuita *et al*., 2015). The coating materials used in this study were maltodextrin and gum-arabic in a ratio of 1:3, 1:1, and 3:1. In

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eISSN: 2550-2166

addition, to determine the characteristics of the results of microencapsulation of red galangal essential oil in optimal treatment. The purpose of this study was to determine the appropriate formulation of the coating material used in the microencapsulation process using the freeze-drying method. In addition, to determine the characteristics of the results of microencapsulation of red galangal essential oil in optimal treatment.

2. Materials and methods

2.1 Tools and ingredients

The tools used in the microencapsulation process were a hot plate stirrer, glassware such as a 250 mL glass beaker, 500 mL glass beaker, 100 mL measuring cup, funnel, and 500 mL Erlenmeyer. The main tool used for the microencapsulation process was a freeze dryer (Labconco). The tools used for analysis included a moisture analyzer (OHAUS MB25), UV-Vis spectrophotometer (Analytik Jena Specord 200 Plus), Gas Chromatography-Mass Spectrometry (Shimadzu QP2010 Plus), Scanning Electron Microscopy (HITACHI), Particle Size Analyzer (CILAS 1090 DRY), and a digital scale (Sartorius 5201-1S). The main ingredient needed was red galangal rhizome, which was then refined to obtain the oil.

2.2 The emulsion and microencapsulation sample preparation

Red galangal rhizome essential oil was obtained from distillation with Pulsed Electrical Field (PEF) pretreatment. Giving voltages to the rhizome serves to increase the oil yield. The voltage used in the pre-treatment was 4000 volts with a long exposure of 30 mins. The microcapsules production is made of an emulsion of red galangal rhizome essential oil. The coating material consists of maltodextrin and gum-arabic (1:3, 1:1, 3:1). Then, distilled water was added as a solvent to each coating material ratio in a ratio of 1:1. The emulsion solution was homogenized at a speed of 6000 rpm, a temperature of 50°C for 15 mins. After that, 5 mL of red galangal rhizome essential oil was added to the encapsulation material and homogenized for 15 mins at a speed of 10,000 rpm. The emulsion was placed in a special tube to be frozen before freeze-drying was carried out.

2.3 Experimental design

The optimization of the microencapsulation process was carried out using the Response Surface Method (RSM) research method with the Central Composite Design (CCD). The experimental design is using Design-Expert software version 8 (trial). The experimental design using Central Composite Design is exposed in Table 1. The factors used were a comparison of the ratio of maltodextrin (X_1) and the ratio of gum-arabic (X_2). The responses measured in this study were yield (Y_1), water content (Y_2), total phenolic content (Y_3), antioxidant activity (Y_4), and total solubility (Y_5). The response data in this study can be seen in Table 2. The TPC response was selected with the maximum target to obtain the highest total phenol yield and the DPPH response (IC_{50}) was selected with the minimum target to obtain the highest antioxidant activity. Meanwhile, the yield response, water content, and solubility level were selected based on the range of the lower and upper limits of each response.

Table 1. Central composite design proses microencapsulation.

Factors	- α	-1	0	+1	+α
Ratio MD (g)	2.20	3.75	7.50	11.25	12.80
Ratio GA (g)	2.20	3.75	7.50	11.25	12.80

Table 2. Central composite design data response

No.	Factors		Response				
	X_1: MD (g)	X_2: GA (g)	Yield (%)	Water content (%)	TPC (mg GAE/g)	DPPH (ppm)	Solubility (%)
1	3.75	3.75	42.960	3.96	17.2165	167.03	84.5
2	11.25	3.75	43.575	2.27	19.7938	166.12	83
3	3.75	11.25	42.675	3.26	25.9794	136.70	86.2
4	11.25	11.25	42.764	2.18	11.5464	172.84	90.4
5	2.20	7.50	42.551	4.6	30.6186	122.89	82.1
6	12.80	7.50	43.281	1.83	2.26804	186.03	89.2
7	7.50	2.20	43.673	1.33	2.78351	184.63	96.7
8	7.50	12.80	41.363	1.66	5.87629	175.44	91.9
9	7.50	7.50	48.275	8.49	20.3093	152.59	93.5
10	7.50	7.50	47.925	7.76	21.8557	149.34	93.4
11	7.50	7.50	48.375	6.05	22.3711	145.19	94.2
12	7.50	7.50	48.075	7.52	21.3402	150.90	93.9
13	7.50	7.50	48.300	6.96	21.8557	148.99	94

https://doi.org/10.26656/fr.2017.7(5).033

2.4 Microcapsule characterization of red galangal essential oil

2.4.1 Yield determination

Determination of the yield of red galangal rhizome essential oil microcapsules was carried out by measuring the emulsion using a digital scale (Sartorius 5201-1S) and recorded as the weight of the starting material. The results of the microcapsules were weighed using a digital scale and recorded as the final sample weight. The percentage of material yield can be calculated by dividing the final sample weight and initial sample weight multiplied by 100%.

2.4.2 Water content analysis

The microcapsules produced from each treatment were measured for moisture content using a moisture analyzer (OHAUS MB25). Samples that were inserted into the tool were as much as 1.2 g. The water content measurement result would appear on the screen.

2.4.3 Total phenolic content analysis

Determination of the total phenolic content of red galangal rhizome essential oil microcapsules was carried out using the Folin-Ciocalteau method. Microcapsules were dissolved in distilled water and then mixed with 10% Folin-Ciocalteau reagent and 7.5% Na_2CO_3. The solution was homogenized and incubated for 30 mins in the dark and at room temperature. Then the absorbance was measured using a UV-Visible spectrophotometer with a wavelength of 760 nm. The number of phenolic compounds in the sample was measured based on the standard curve of Gallic Acid Equivalent (GAE)/g microcapsules.

2.4.4 Antioxidant activity analysis

Determination of the antioxidant activity of red galangal rhizome essential oil microcapsules was carried out using the DPPH method. The absorbance of the sample was measured using a UV-Visible spectrophotometer with a wavelength of 517 nm. The level of antioxidant activity was expressed by the percentage of free radical inhibition (% inhibition).

2.4.5 Solubility analysis

The solubility of microcapsules in water was calculated using Whatman filter paper no. 42 through the percentage by weight of the residue that could not pass through the filter. The filter paper was weighed before use. The solubility level was measured by weighing 0.1 g of microcapsule sample (a) into 10 mL of distilled water and stirring using a magnetic stirrer. The solution was filtered through filter paper and the unfiltered sample was heated in an oven for 1 hr at 105°C, then cooled in a desiccator for 15 mins, and then weighed. The weight of the unfiltered sample (b) was obtained from the difference between the weight of the final filter paper and the weight of the initial filter paper. The percentage of solubility level can be measured by calculating the difference in the weight of the sample divided by the weight of the microcapsule sample and multiplied by 100%.

2.5 Chemical component analysis of microcapsule

This test was conducted to determine the phytochemical content of essential oils and microcapsules produced using Gas Chromatography-Mass Spectrometry. The analysis was carried out by injecting the sample into the instrument with the injection port conditions up to 250°C, oven temperature up to 60°C and increasing periodically to 280°C. The detector used was a mass spectrometer (MS) with an ionization energy of 70 eV, scanned mass range (m/z) 28 –600, with a detector temperature of 250°C.

3. Results and discussion

3.1 Microcapsule characteristic of red galangal essential oil

3.1.1 Yield response

The highest yield response of 48.375% was obtained from the treatment with a maltodextrin ratio of 7.5 g and a ratio of 7.5 g of gum arabic. The lowest yield of 41.364% was obtained from the treatment with a maltodextrin ratio of 7.5 g and a ratio of 12.8 g of gum arabic. The research data showed that the amount of yield depends on the amount of emulsion produced, so it depends on the amount of coating material used. Research by Wilkowska *et al*. (2016) explained that the amount of yield in the microencapsulation process depends on the method used. The results of the analysis of variance showed that there was a significant effect of the factor on the yield response, which was indicated by a p-value <0.05.

The response surface factor curve to the yield response can be seen in Figure 1 (a). Based on the curve, it can be seen that the two factors have a significant influence. The graph shows a quadratic model which shows there is a peak as the optimal condition and there is a decrease based on the two factors used. Based on the research of Cid-Ortega *et al*. (2020), the combination of maltodextrin and gum arabic coating materials affects the amount of yield produced in the microencapsulation process (Table 2). The greater the viscosity, the lower the yield, this is due to the increasing content of dissolved solids. Gum arabic coating material can cause an increase in the viscosity of the emulsion which can

https://doi.org/10.26656/fr.2017.7(5).033

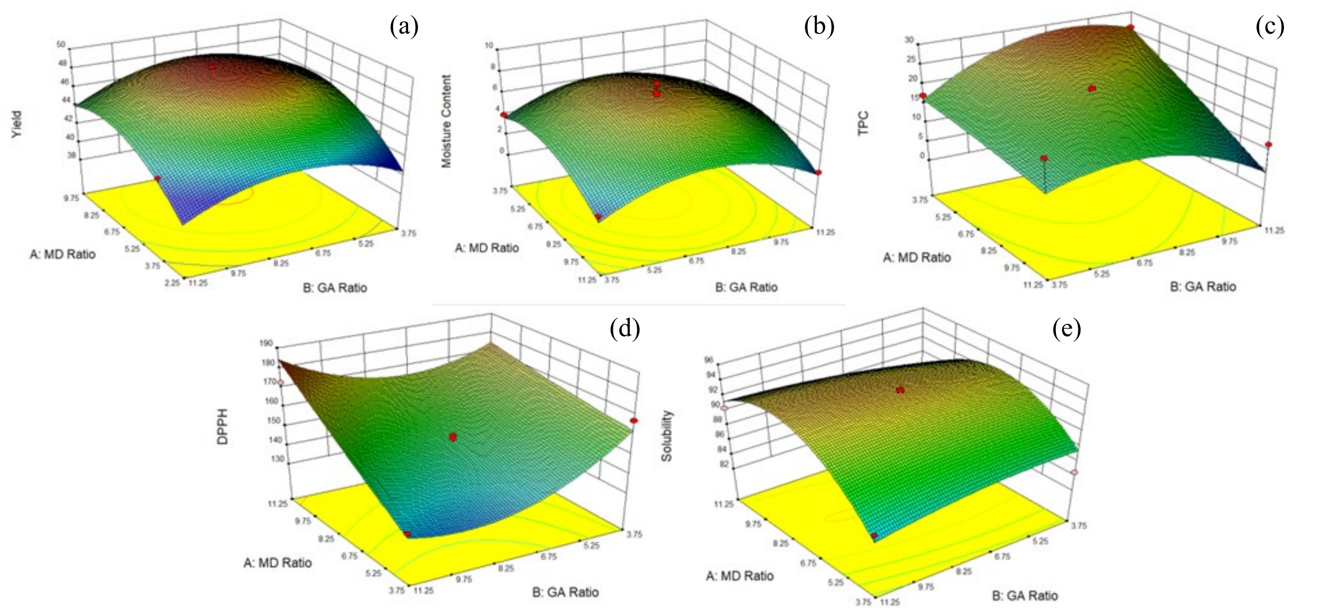

Figure 1. Response surface curve of ratio coating materials on (a) yield, (b) water content, (c) TPC, (d) DPPH, and (e) solubility of red galangal essential oil microencapsulation.

cause drying in the microencapsulation process not to run perfectly (Khasanah *et al*., 2015). Thus, in this study, the smallest yield was the treatment with the highest ratio of gum arabic.

3.1.2 Water content response

The highest yield response of 8.49% was obtained from the treatment with a maltodextrin ratio of 7.5 g and gum arabic ratio of 7.5 g. The lowest water content of 1.33% was obtained from the treatment with a maltodextrin ratio of 7.5 g and a ratio of 2.2 g of gum Arabic. From the research data, it was shown that the water content resulting from the microencapsulation process was not significantly affected by the ratio of the coating material. According to the research of Du *et al*. (2014), the different chemical structures of the coating materials resulted in varying the water content of each combination. Gum arabic coating material is a complex heteropolysaccharide with a highly branched structure and has short chains and more hydrophilic groups (Silva *et al*., 2013). The higher concentration of maltodextrin causes lower water content in microcapsules (Tuyen *et al*., 2010). Thus, the greater the ratio of gum arabic used as a coating material, the higher the water content. The results of the analysis of variance (ANOVA) showed that there was a significant effect of the factors on the water content response, which was indicated by a p-value <0.05.

The response surface factor curve to the water content response can be seen in Figure 1 (b). Based on the curve, it can be seen that the two factors have a significant influence. The graph shows a quadratic model which shows there is a peak as the optimal condition and there is a decrease based on the two factors used. In previous studies, the results of microencapsulation with low water content will increase hygroscopicity because it can absorb moisture related to the water concentration between the microcapsules and the atmosphere (Tonon *et al*., 2011).

3.1.3 Total phenolic content response

The highest total phenol response was 30.6187 mg GAE/gr which was obtained from the treatment with a maltodextrin ratio of 2.2 g and a ratio of 7.5 g of gum arabic. The lowest total phenol was 2.268 mg GAE/gr which was obtained from the treatment with a maltodextrin ratio of 12.8 g and a ratio of 7.5 g of gum arabic. The research data showed that the total phenol tends to increase with a smaller ratio of maltodextrin. Based on the research of Busch *et al*. (2017) who explained that the gum arabic coating material can protect more bioactive compounds caused by the branched structure of the gum, making it easier for substances to protect the phenolic content by bonding. The results of the analysis of variance showed that there was a significant effect of the factors on the TPC response, which was indicated by a p-value <0.05.

The response surface factor curve to the total phenolic content response can be seen in Figure 1 (c). Based on the curve, it can be seen that the two factors have a significant influence. The graph shows a quadratic model which shows that there is a peak as the optimal condition and a decrease based on the two factors used. Based on the research of Blainski *et al*. (2013), the higher the ratio of the coating material used, the less the intensity of the blue colour in the Folin-Ciocalteau reagent, causing the total phenol content to decrease.

3.1.4 Antioxidant activity response

The highest antioxidant activity response was 186.03 ppm which was obtained from the treatment with a maltodextrin ratio of 12.8 g and a ratio of 7.5 g to gum-arabic. The lowest antioxidant activity of 122.89 ppm was obtained from the treatment with a maltodextrin ratio of 2.2 g and a ratio of 7.5 g of gum-arabic. The research data showed that the antioxidant activity tends to increase with the increasing ratio of maltodextrin. Based on the research of Ananingsih *et al*. (2014), a maltodextrin concentration that is too high can cause the value of the antioxidant activity to be lower because maltodextrin only plays a role in increasing the yield of the product. The results of the analysis of variance showed that there was a significant effect of factors on the response of antioxidant activity as indicated by p-value <0.05.

The response surface factor curve to the antioxidant activity response (IC_{50}) can be seen in Figure 1 (d). Based on the curve, it can be seen that the two factors have a significant influence. The graph shows a quadratic model which shows there is a peak as the optimal condition and there is a decrease based on the two factors used. Gum arabic which was used as a coating material can protect the encapsulated bioactive compounds during the drying and storage process (Noghabi and Mohammad, 2019). According to Böger *et al*. (2018), the combination of maltodextrin and certain gum arabic can increase antioxidant activity.

3.2 Solubility analysis response

Solubility is an important property of microcapsules because they can rehydrate when used as food or pharmaceutical ingredients (Syamaladevi *et al*., 2012). Based on the tests carried out, it can be seen that the response of the highest solubility level of 96.7% was obtained from the treatment with a maltodextrin ratio of 7.5 g and a ratio of 2.2 g of gum-arabic. The lowest solubility level of 82.1% was obtained from the treatment with a maltodextrin ratio of 2.2 g and a ratio of 7.5 g of gum-arabic. The research of Krisnitya *et al*. (2020), explained that the higher the concentration of maltodextrin, the better the percentage of microcapsule powder solubility. The results of the analysis of variance showed that there was a significant effect of the factors on the solubility level response, which was indicated by a p-value <0.05.

The response surface curve factor to the solubility level response can be seen in Figure 1 (e). Based on the curve, it can be seen that the two factors have a significant influence. The graph shows a quadratic model which shows there is a peak as the optimal condition and there is a decrease based on the two factors used. Based on the research of Yuliawaty and Wahono (2015), the high solubility is due to the presence of hydroxyl groups found in maltodextrin which will interact with water. The group with a high amino acid charge can cause the group to become soluble in polar solvents such as water (Ningsih *et al*., 2019).

3.3 Optimal response solution

The optimal result of the response that was processed by Design Expert 8.0 (trial) was obtained in the treatment of the maltodextrin ratio of 3.75 and the ratio of gum-arabic 9.14, with the predicted value of each response in Table 3. The optimal solution was obtained at a desirability value of 0.894%. According to Akçay and Anagün (2013), the desirability value comes from each value of the converted response and is made on a scale from 0 to 1. A value of 0 indicates the solution is not optimal and the closer to 1 the results are more optimal. This shows that the prog can achieve the desired optimization goals.

Table 3. Optimal solution.

Parameters	Prediction Standard
Ratio MD (g)	3.75
Ratio GA (g)	9.14
Yield (%)	44.6941
Water content (%)	5.64335
TPC (mg GAE/g)	28.4251
DPPH (ppm)	131.282
Solubility (%)	86.282
Desirability	0.894
Noted	*Selected*

The optimal results of the response were known, and the verification stage was carried out by conducting research again using the factors that have been generated in the optimal solution. Then, each response was measured and recorded. The results of the verification were then compared with the predicted values by the prog. The comparison of the actual and predicted verification results in the prog can be seen in Table 4. According to Amran *et al*. (2014), the accuracy value obtained above 50% indicates that the model is accurate enough to predict optimal results. All actual verification results were still within the predicted interval value. This is following the statement of Kim and Kyung (2008), that the prediction interval is used as the upper and lower limits to determine the accuracy of the results on the optimal solution.

3.4 Gas chromatography-mass spectroscopy

The GC-MS analysis on the results of the optimal treatment of the microencapsulation process aimed to determine the chemical content of microcapsules. The

https://doi.org/10.26656/fr.2017.7(5).033

Table 4. Comparison between actual verification and prediction.

Parameters	Prediction	Interval Prediction	Data Actual Verification	Accuracy (%)
Ratio MD (g)	3.75	3.75	3.75	
Ratio GA (g)	9.14	9.14	9.14	
Yield (%)	44.6941	43.72 – 45.66	44.01	98.45
Water content (%)	5.64335	3.51 – 7.77	5.75	98.15
TPC (mg GAE/g)	28.4251	12.54 – 44.30	34.227	83.05
DPPH (ppm)	131.282	108.45 – 154.11	136.01	96.52
Solubility (%)	86.282	77.93 – 95.07	87.70	98.38

analysis was carried out by diluting the microcapsule powder from each treatment using methanol as a solvent in a ratio of 1: 10 (Maulidna *et al*., 2020). Based on Table 5, it can be seen that there were five main compounds detected from the results of the microcapsule dilution, namely, Diacetone alcohol of 31.36%, Chavicol of 15.26%, Methyl oleate of 6.87%, Alloaromadendrene of 5.82% and Methylhexadecanoate of 3.44%.

Table 5. Chemical component of microcapsule of red galangal essential oil.

No.	Chemical components	Area (%)
1.	Diacetone alcohol	31.36
2.	Chavicol	15.26
3.	Methyl oleate	6.87
4.	Alloaromadendrene	5.82
5.	Methyl hexadecanoic	3.44

Microcapsule particle size analysis using CILAS 1090 DRY Particle Size Analyzer. The results of measuring the diameter particle size distribution of microcapsule powder obtained an average value of 243.17 µm (Table 6).

Table 6. Particle size distribution of microcapsule of red galangal rhizome essential oil.

Diameter 10% (µm)	Diameter 50% (µm)	Diameter 90% (µm)	Average Diameter (µm)
150.34	249.49	356.67	243.17

4. Conclusion

The results showed that the optimal coating material ratio was found in the MD ratio of 3.75 g and the GA ratio of 9.14 g. From the results of the comparison between the actual verification value and the predicted value, the model is found to be suitable. The optimal coating material ratio can produce a good yield response, moisture content, TPC, DPPH, and solubility. Based on the observation of morphological characteristics, the selected coating material can affect the shape and size of the red galangal rhizome essential oil microcapsules.

Conflict of interest

The authors declare no conflict of interest.

Acknowledgments

The research work was funded by the Faculty of Agriculture Technology Universitas Brawijaya-Malang-Indonesia.

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