Optimization of Subcritical Fluid Extraction in Zingiberene

by Vita Paramita

Submission date: 03-Jul-2019 07:53AM (UTC+0700)
Submission ID: 1148821552
File name: C16,4292-15239-1-PB.pdf (750.42K)
Word count: 2291
Character count: 12374

Journal of Vocational Studies on Applied Research

Optimization of Subcritical Fluid Extraction in Zingiberene

Vita Paramita[1*], Mohamad Endy Yulianto[1], Indah Hartati[2], Udin Bahrudin[3], RTD WisnuBroto[1]

[1]Vocational School, Diponegoro University, Prof. Sudarto, SH street Tembalang-Semarang
[2]Wahid Hasyim University, MenorehstreetSampangan-Semarang
[3]Medical Faculty, Diponegoro University, Prof. Sudarto, SH street Tembalang-Semarang

e-mail: vparamita@live.undip.ac.id

Abstract- *The establishment of subcritical fluidapplicationfor zingiberene extraction was studied through the use of water as a green solvent. The zingiberene content was observed by using Liquid Chromatography-Mass Spectrophotometry while the independent variables include temperature, extraction time, ginger-solvent ratio and aging time. Through the application of the subcritical water, the critical value of zingiberene content obtained was 0.036%. It was achieved by using surface modeling of alpha for rotatability design at 125℃, 20 minextraction time, 0.08 ginger to solvent ratio and 9 min aging time. The surface modeling was validated by plotting the observed and predicted value of zingiberene content which later showed R-square at 0.76.*

Keywords **-** *ginger, zingiberene, subcritical water, extraction, optimization*

Submission: January 07, 2019 Correction: January 15, 2019 Accepted: January 28, 2019

Doi: http://dx.doi.org/10.14710/jvsar.1.1.2019.18-21

[How to cite this article: Paramita, V., Yulianto, M.E., Hartati, I., Bahrudin, U., and Broto, RTD. W.(2019). Optimization On Subcritical Fluid Extraction Of Zingiberene. *Journal of Vocational Studies on Applied Research, 1*(1), 18-21. doi: http://dx.doi.org/10.14710/jvsar.1.1.2019.18-21]

1. Introduction

Zingiberene is well known as one of the main components of ginger (*Zingiberofficinale*) essential oil [1,2]. This oil has a wide application such as in antimicrobial activity [3,4], as well as herbal medicine [5,6]. Many extractions methods have been applied to obtained ginger active compounds [7,8]. Sharma and Gupta reported that the conventional extraction method is by juicing and adding acetone as the solvent which makes it more effective than for ethanol [9]. Yang *et al.* compared three different methods for ginger oil extraction and concluded that solid-phase microextraction provided a higher yield than petrol-ether or steam distillation extractions [10].

In the last decade, the application of subcritical water as a solvent medium gained its popularity. This method was adopted because its implementation is environmentally friendly. The subcritical water serves as a natural form of acid catalyst, at the range of 150-370°C and 4-220 bar of temperature and pressure respectively [11]. Recently, Saripreported its use for the extraction of ginger oil at a wider temperature range (100-374.5°C) and saturated pressure [12]. It is mostly employed because of the selective polarity of water which can make it be either polar or non-polar when applied as a solvent in specific conditions [13,14]. Therefore, this study was designed to discussthe optimization of subcritical water extraction of ginger fresh rhizome to obtain a valuable component of zingiberene with regards to the halal issue.

2. Experiment

2.1 Apparatus

The quantity of distilled water needed was set to the operation condition temperature and loaded into the pressurized hot water (PHW) extractor, and weighed ground fresh ginger was added based on various specific ginger-solvent ratio (0.06, 0.07, 0.08, 0.09 and 0.10). The stainless-steel lid of the PHW extraction cell was securely covered after the raw materials had been well arranged. Nitrogen gas was passed over the extractor for 2 min to ensure air removal from the cell and to sustain a fixed pressure of 2 bar. The extraction was conducted at a temperature of 115, 120, 125, 130, and 135°C for 10, 15, 20, 25, and 30 minutes respectively. Table 1 shows the parameter.The extract was subsequently cooled at 25°C and 1 MPa in the cooling cell, concisely after the extraction process was finished [14]. The extracted compound was aging for 0, 5, 10, 15 and 20 minutes before sampling to ensure the completion of theadvanced extraction process.

Table 1. Parameters for the subcritical fluid extraction of zingiberene

Parameter Level	Temperature (°C)	Ratio ginger to solvent (-)	Time (min)	Aging Time (min)
Low Star Point	115	0.06	10	0
Low Level	120	0.07	15	5
Central Level	125	0.08	20	10
High Level	130	0.09	25	15
High Star Point	135	0.10	30	20

3. Analysis

3.1 Bioactive Components Determination

The subcritical water ginger extract samples were analyzed using LC-MS (LC-MS Waters Xevo-TQD, Waters Corp., MA, USA). The ionization mode of chromatograph was electrospray positive (ESI+) and the measurement method was Single Ion Reaction (SIR). The parent ion of zingiberene was 205m/z.The mobile phase was methanol and aquabidest at 90:10 ratio and 0,5 ml/min of flow rate. Nitrogen was used as the gas carrier at a linear velocity of 50L/h. The desolvation temperature was 400 °C. The capillary and conevoltages were 3500 and 30V, respectively. 10ml of subcritical water of fresh ginger rhizome extract was evaporated until it reached one third and dissolved with pro-ethanol LC-MS upto 10ml. The solution was filtered using microphore 0.45µm and the filtrate placed on the sample vial. The datawas interpreted based on the optimum condition of the apparatus for the ginger metabolite.

3.2 Response Surface Methodology (RSM)

The central composite of alpha for rotatability design was applied to the Response Surface Methodology (RSM) to optimize the parameter process during the zingiberene extraction process. A set of random experimental work was subjected to the design of experiment (DOE) in order to execute the optimized value. The independent variables were temperature, extraction time, aging time and ginger-solvent ratio, while the dependent was zingiberene content. There were 26 actual experiments with 4 factors (k=4), and 3 levels to construct a central composite design of rotatability (e.g., star points ± 2). The ranges of the experimental design were based on previous results [14]. Variables, ranges, and levels of the experimental work are presented in Table 1.

4. Results and Discussion

The LC-MS analysis of zingiberene extracts was obtained with operation condition as mentioned in Table 1

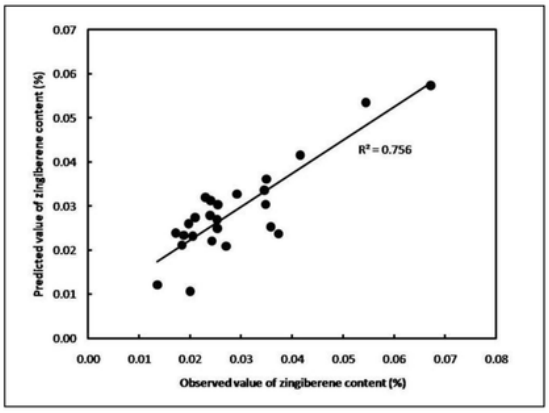

Figure 1. Observed vs predicted plot of zingiberene content

and presented as an observed value with respect to the predicted value in Table 2. The plot of the observed and predicted value resulted in the linear correlation of least square at the R-square value of 0.756. This indicates that the values were well correlated.

The analysis of variance showed that the temperature (Q) significantly ($p<0.05$) affected the zingiberene content (Table 2). Varying the extraction, ginger-solvent ratio and aging time resulted in p-value higher than 0.05 and this shows that there is no significant effect on the extracted content. This is also supported by the standardized effect estimate of Pareto chart (Fig. 2).

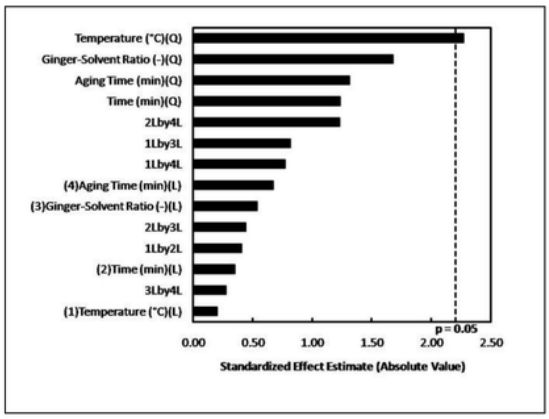

Fig. 2. Standardized effect estimate of Pareto chart

The higher value of independent parameters than p-value (0.05) indicates that they significantly affected the observed parameter value (i.e. zingiberene content), and vice versa. Chernoff emphasized that influential variables firmly deal with proper analysis rather than measuring their effects [15].

Table 2. ANOVA of process variables

	SS	Df	MS	F	p
(1)Temperature (°C)(L)	0.000003	1	0.000003	0.039789	0.845533
Temperatur (°C)(Q)	**0.000419**	**1**	**0.000419**	**5.148575**	**0.044382**
(2)Time (min)(L)	0.000010	1	0.000010	0.121035	0.734481
Time (min)(Q)	0.000124	1	0.000124	1.519879	0.243336
(3)Ginger-solvent ratio (-)(L)	0.000023	1	0.000023	0.287184	0.602698
Ginger-solvent ratio (-)(Q)	0.000229	1	0.000229	2.812954	0.121660
(4)Aging time (min)(L)	0.000037	1	0.000037	0.450312	0.516030
Aging time (min)(Q)	0.000140	1	0.000140	1.718981	0.216534
1Lby2L	0.000013	1	0.000013	0.164476	0.692845
1Lby3L	0.000054	1	0.000054	0.660878	0.433493
1Lby4L	0.000048	1	0.000048	0.592369	0.457714
2Lby3L	0.000016	1	0.000016	0.192065	0.669686
2Lby4L	0.000123	1	0.000123	1.510672	0.244681
3Lby4L	0.000006	1	0.000006	0.075067	0.789174
Error	0.000895	11	0.000081		
Total SS	0.003677	25			

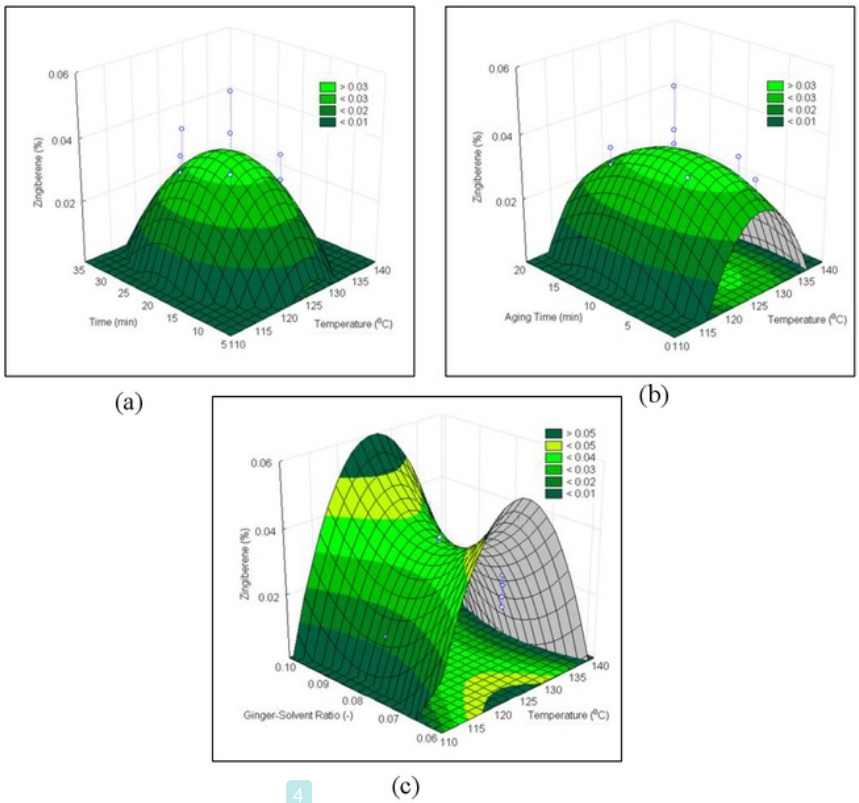

Figure 3. Response surface contour; (a) effect of time and temperature on the zingiberene content at 0.08 of ginger to solvent ratio for 10 min of aging time; (b) effect of aging time and temperature on the zingiberene content at 0.08 of ginger to solvent ratio for 20 min of extraction time; (c) effect of ginger-solvent ratioand temperature on the zingiberene content at 20 min of extraction time and 10 min of aging time.

The 3D-contour responses of the surface plots are as presented in Fig. 3. The plot of temperature against time (Fig. 3a) and aging time (Fig. 3b) showed a maximum value of zingiberene content at the value of 0.036%. Nevertheless, this value was obtained as minimum value by plotting the temperature against ginger-solvent ratio. Expanding this ratio to higher and lower variables resulted in a higher value of zingiberene extracted content. This shows that optimum value was obtained regardless of the ratio.

The observed values of zingiberene extract were presented in percentage from all of the components obtained (i.e. zingiberene, gingerol, paradol, and shogaol). The zingiberene content was identified by deprotonating the fragmented pattern of its molecular weight [16]. Therefore, the critical value of zingiberene (0.036 %) was obtained at 125°C temperature, 20 min extraction time, 0.08 ginger to solvent ratio and 9 min aging time.

5. Conclusion

Through the application of subcritical water, a critical value of 0.036% was obtained for zingiberene content. This value was approached by the surface modeling of alpha for rotatability design performed at 125°C temperature, 20 min extraction time, 0.08 ginger to solvent ratio and 9 min aging time of extraction parameters. The surface modeling was validated by plotting the observed and predicted value of zingiberene content and evidenced by the value of R-square at 0.76.

6. Acknowledgment

This research was supported by the 2017 research grant from the Ministry of Research, Technology and Higher Education, Indonesia.

References

[1] El-Ghorab AH, Nauman M, Anjum FM, Hussain S, Nadeem M. 2010. A comparative study on chemical composition and antioxidant activity of ginger (*Zingiber officinale*) and cumin (*Cuminum cyminum*). Journal of Agricultural and Food Chemistry 58(14): 8231-8237.

[2] Nampoothiri SV, Venugopalan VV, Joy B, Sreekumar MM, Menon AN. 2012. Comparison of Essential oil Composition of Three Ginger Cultivars from Sub Himalayan Region. Asian Pacific Journal of Tropical Biomedicine 2(3), Supplement: S1347-S1350.

[3] Yamamoto-Ribeiro MMG, Grespan R, Kohiyama CY, Ferreira FD, Mossini SAG, Silva EL, FilhoBAdA, Mikcha JMG, Junior MM. 2012. Effect of Zingiber officinale essential oil on Fusarium verticillioides and fumonisin production. Food Chemistry 141: 3147-3152.

[4] Singh G, Kapoor IP, Singh P, de Heluani CS, de Lampasona MP, Catalan CA. 2008. Chemistry, antioxidant and antimicrobial investigations on essential oil and oleoresins of *Zingiber officinale*. Food and Chemical Toxicology 46: 3295-3302.

[5] Cafino EJV, Lirazan MB, Marfori EC. 2016. A simple HPLC method for the analysis of [6]-gingerol produced by multiple shoot culture of ginger (*Zingiber officinale*). International Journal of Pharmacognosy and Phytochemical Research8(1): 38-42.

[6] Butt MS, Sultan MT. 2011. Ginger and its health claims: Molecular aspects. Critical Reviews in Food Science and Nutrition 51(5): 383-393

[7] Guo JB, Fan Y, Zhang WJ, Wu H, Du LM, Chang YX. Extraction of gingerols and shogaols from ginger (*Zingiber officinale* Roscoe) through microwave technique using ionic liquids, Journal of Food Composition and Analysis 62: 35-42.

[8] Sadineni V, Umesh KV, Singhal RS. (2016). Enhanced extraction of oleoresin from ginger (Zingiber officinale) rhizome powder using enzyme-assisted three phase partitioning. Food Chemistry. 216: 27-36.

[9] Sharma SS, Gupta YK. 1998. Reversal of cisplatin-induced delay in gasric emptying in rats by ginger (Zingiber officinale), Journal of Ethnopharmacology 62(1): 49-55.

[10] Yang Z, Yang W, Peng Q, He Q, Feng Y, Luo S, Yu Z. 2009. Volatile phytochemical composition of rhizome of ginger after extraction by headspace solid-phase microextraction, petroleum ether extraction and steam distillation extraction. Bangladesh Journal of Pharmacology 4: 136-143.

[11] Moreschi SRM, Leal JC, Braga MEM, Meireles MAA. 2006. Ginger and turmeric starches hydrolysis using subcritical water + CO2: the effect of the sfe pre-treatment. Brazilian Journal of Chemical Engineering 23(2): 235-242.

[12] Sarip MSM, Morad NA, Ali MNA, Yusof YAM, Yunus MAC. 2014. The kinetics of extraction of the medicinal ginger bioactive compounds using hot compressed water. Separation and Purification Technology 124: 141-147.

[13] Bogdanov MG, Svinyarov I. 2013. Ionic liquid-supported solid–liquid extraction of bioactive alkaloids. II. Kinetics, modeling and mechanism of glaucine extraction from Glaucium flavum Cr. (Papa veraceae). Separation and Purification Technology 10: 279-288.

[14] Yulianto ME, Kusumo P, Hartati I, Wahyuningsih. 2017. Subcritical water extraction of gingerol from *Zingiber Officinale*. Rasayan Journal of Chemistry 10(3): 738-743.

[15] Chernoff H, Lo S-H, Zhing T. 2009. Discovering influential variables: A method of partitions. The Annals of Applied Statistics 3(4): 1335-1369.

[16] Tao Y, Li W, Liang W, Breemen RBv. 2009. Identification and quantification of gingerols and related compounds in ginger dietary supplements using High Performance Liquid Chromatography-Tandem Mass Spectrometry. Journal of Agricultural and Food Chemistry 57(21): 10014-10021.

Optimization of Subcritical Fluid Extraction in Zingiberene

ORIGINALITY REPORT

7%	5%	6%	6%
SIMILARITY INDEX	INTERNET SOURCES	PUBLICATIONS	STUDENT PAPERS

PRIMARY SOURCES

1	baadalsg.inflibnet.ac.in Internet Source	2%
2	journals.sagepub.com Internet Source	2%
3	www.ijcrls.com Internet Source	1%
4	Leonardo Sepúlveda, Aloia Romaní, Cristóbal Noé Aguilar, José Teixeira. "Valorization of pineapple waste for the extraction of bioactive compounds and glycosides using autohydrolysis", Innovative Food Science & Emerging Technologies, 2018 Publication	1%
5	Submitted to Universiti Sains Malaysia Student Paper	1%
6	Submitted to University of Stellenbosch, South Africa Student Paper	<1%
7	Submitted to Tezpur University	

Student Paper

<1%

8 Duy Nguyen, Vittoria Balsamo, Jenny Phan. "Effect of Diluents and Asphaltenes on Interfacial Properties and Steam-Assisted Gravity Drainage Emulsion Stability: Interfacial Rheology and Wettability", Energy & Fuels, 2013

Publication

<1%

Exclude quotes	Off	Exclude matches	Off
Exclude bibliography	Off		

Optimization of Subcritical Fluid Extraction in Zingiberene

GRADEMARK REPORT

FINAL GRADE

/0

GENERAL COMMENTS

Instructor

PAGE 1

PAGE 2

PAGE 3

PAGE 4