



## Effect of Extraction Temperature, Time and Volume of Diluent on Oil Yield from Ginger (*Zingiber officinale*) in a Batch-Mode Process

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**ABSTRACT:** This study focuses on modelling, optimizing and examining the effect of extraction temperature, volume of diluent and extraction time on the yield of oil from *Zingiber officinale* (ginger) using a batch-mode solvent extraction process with ethanol as solvent. Fresh ginger seeds were obtained from a local vendor, washed with distilled water and sun-dried. The dried sample was then milled to further reduce the particle size. A four-level-three-factor D-optimal approach was used for designing the experiments and for developing a statistical model for the optimization of the process variables. A quadratic model was developed for modelling and optimization of the extraction parameters. The model was significant with p-value less than 0.0001 and the "Lack of Fit" p-value was 0.1019, which means that the lack of fit was insignificant. A high  $R^2$  value of 0.983 by the model shows that the model was able to adequately represent the actual relationship between the variables studied. Extraction temperatures, volume of diluent and extraction time were found to have significant effect on the yield of oil from ginger. The optimum conditions predicted by the model were 82.22°C extraction temperature, 300 ml volume of diluent, and 30 minutes extraction time. The predicted yield was 21.451% and the results obtained from three replications gave an average yield of 20.533% which was close to the predicted value.

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*Zingiber officinale* commonly known as ginger is a monocotyledon plant that belongs to the family called *Zingiberaceae*. The plant is a commonly consumed dietary condiment that is generally considered to be safe and used to cure various diseases (Arshad *et al.*, 2014). Gingerols, the pungent component in the rhizome of ginger, were reported to have antiemetic, analgesic, antipyretic, anti-inflammatory, chemopreventive, and antioxidant properties (Dugasani *et al.*, 2010). Thus, ginger has been universally used both in modern and traditional medicine (Mekuriya and Mekibib, 2018). Its therapeutic roles in diseases management via modulation of biological activities include anti-inflammatory and anti-oxidative activities together with regulation of genes mechanism of action (Arshad *et al.*, 2014). The extraction of oil is dependent on various factors which include extraction time, particle size, the volume of diluents and extraction temperature. The interaction among factors has been scarcely considered. In this respect, a statistical experimental design can be employed to determine the effects of process parameters and their interactions and to optimise the adsorption process, avoiding the traditional one-factor-at-a-time experiments (Carmona *et al.*, 2005; Huang *et al.*, 2008). The design determines which factors have

important effects on a response as well as how the effect of one factor varies with the level of the other factors (Carmona *et al.*, 2005; Montgomery, 2005; Box *et al.*, 1978; Brasil *et al.*, 2005; Pavan *et al.*, 2007; Annadurai *et al.*, 2002). The determination of factor interactions could only be attained using statistical designs of experiments (Montgomery, 2005; Box *et al.*, 1978) since it cannot be observed when the system optimization is carried out by varying just one factor at the time and fixing the others. Response surface methodology (RSM) is suitable as it is robust and a widely used method in optimization process of process parameters (Tan *et al.*, 2012; Rezzoug *et al.*, 2005).

Previous studies have reported various extraction methods to obtain phytochemicals from ginger, such as supercritical fluid extraction (Hawthorne, 1990), sonication (Abdurahman *et al.*, 2013) and accelerated water extraction (Herrero *et al.*, 2005). However, there are few studies on the process parameter optimization of the extraction of this essential oil using D-Optimal approach. This study, therefore, focuses on the application of D-Optimal approach for the optimization of extraction of essential oil from ginger. The D-Optimal approach is a systematic application of analysis of experiments for the sole purpose of

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designing and improving the quality of the product. It is principally used for evaluating several process parameters at a time with the smallest number of experimental runs based on a table.

## MATERIALS AND METHODS

**Feedstock Collection, Preparation and Pretreatment:** Rhizomes of fresh ginger were obtained from Uselu market in Benin City, Edo state, Nigeria. They were washed with water and sliced longitudinally to facilitate drying by the sun. The sliced rhizomes of ginger were dried consistently under the sun for three weeks to reduce the moisture content. The dried rhizomes of ginger were finely ground so as to facilitate the extraction process. The sample was stored under dry conditions prior to use.

**Extraction Process:** The fine rhizomes of ginger were subjected to solvent extraction process using ethanol as the solvent. The extraction process was carried out using the Soxhlet apparatus. During the extraction process, 30 g of the ground dried ginger rhizomes was used during each experimental run.

**Experimental Design:** A four-level-three-factor D-Optimal factorial design, which resulted in twenty experimental runs, was generated with the Design Expert 7.0.0 (Stat-ease, Inc. Minneapolis, USA). Response surface methodology (RSM) based on D-Optimal design was applied to evaluate the effect of the following controlled independent variables: extraction time, extraction temperature and volume of diluents.

**Process modelling and optimization:** The following generalized second order polynomial equation was used to estimate the response of the dependent variables.

$$Y = b_0 + \sum b_i X_i + \sum b_{ii} X_i^2 + \sum \sum b_{ij} X_i X_j + E \quad (1)$$

Where  $Y_i$  is the predicted response,  $X_i$  and  $X_j$  are the independent variables,  $b_0$  is offset term,  $b_i$  and  $b_{ij}$  are the single and interaction effect coefficients and  $E$  is the error term.

Based on RSM, this equation was used to evaluate the linear, quadratic and interactive effects of independent variables on the chosen response. For the model, the calculations from the linear and cross regression were performed.

The  $R^2$  value, the residual error, the pure error calculated from the repeated measurements and the lack of fit were calculated. ANOVA was utilized to estimate the measurable attributes of the model fitting.

## RESULTS AND DISCUSSIONS

**Statistical modelling:** By applying a multiple regression analysis on the experimental data and the following second degree polynomial was obtained:

$$Y = 24.95 + 1.36A + 1.08B + 2.37C - 1.47AB + 0.19AC + 1.97BC - 12.16A^2 + 1.30B^2 - 1.45C^2 \quad (2)$$

Where  $Y$  represents ginger extract yield as a function of extraction temperature ( $A$ ), volume of diluent ( $B$ ) and extraction time ( $C$ ).

The predicted levels of yield of ginger oil based on Eq. (2) together with the experimental data are given in Table 1. Tables 2 and 3 show the results of analysis of variance (ANOVA) carried out to determine the fit of the statistical model. Table 1 gives the experimental design matrix using D-Optimal, the experimental yields of oil from ginger and the predicted yields. A comparison of the experimental and predicted yields showed very little deviations. This small deviation between experimental yields and predicted yields is indicative of the goodness of the statistical model. The root mean square error (RMSE) was chosen as an indicator of the performance of the statistical model.

The predicted data, when compared to the experimental data, gave RMSE value of 0.2766. The value of RMSE calculated shows that the model derived from the experimental data were able to predict the extraction yield of oil from ginger quite well. An  $R^2$  value of 0.983 indicates that the model explained 98.3% of the variability in the response for the region studied. The predicted  $R^2$  of 0.913 is also in reasonable agreement with an adjusted  $R^2$  of 0.969.

The Coefficient of Variance (CV) is the ratio of standard error of the estimate to the mean value. A low value of CV indicates high reliability and precision of the experiment. It is considered reproducible once it is not greater than 10% (Montgomery, 2005; Mason *et al.*, 1989). In this study, CV obtained was 7.794 %, hence it is considered reproducible.

This parameter shows the degree of precision with which the runs were carried out. An Adequate precision value of 24.031 was obtained. This parameter measures the signal to noise ratio and a value greater than 4 is generally desirable (Cao *et al.*, 2009). A ratio of 24.031 indicates an adequate signal meaning that the model can be used to navigate the design space.

**Table 1 :** D-Optimal design matrix for optimization of variables and the response values

Run No	Factors			Response Yield of oil (%)	
	A	B	C	Experimental	Predicted
1	90.00	400.00	51.66	13.45	13.69
2	85.18	359.82	30.00	20.02	20.71
3	90.00	334.96	90.00	12.99	13.69
4	82.23	400.00	90.00	28.78	29.13
5	82.23	400.00	90.00	30.02	29.13
6	78.00	300.00	30.00	9.23	8.51
7	78.00	359.54	65.77	11.98	12.40
8	78.00	400.00	30.00	9.29	9.68
9	85.18	359.82	30.00	20.29	20.71
10	78.00	300.00	90.00	9.01	8.94
11	78.00	300.00	90.00	9.09	8.94
12	90.00	300.00	30.00	12.95	13.80
13	79.10	303.11	60.00	13.76	14.71
14	85.14	300.00	65.71	25.21	25.30
15	78.00	400.00	30.00	9.79	9.68
16	90.00	337.21	52.50	14.23	13.73
17	90.00	300.00	30.00	14.11	13.80
18	90.00	400.00	90.00	17.89	18.14
19	78.00	350.00	31.09	8.67	7.98
20	85.37	361.63	67.50	25.61	25.50
				<b>RMSE</b>	<b>0.2766</b>

**Table 2:** ANOVA for Quadratic Model

Source	Sum of Squares	dF	Mean Square	F Value	p-value	
Model	906.468	9	100.719	65.006	< 0.0001	Significant
Temperature (A)	18.674	1	18.674	12.052	0.0060	
Volume (B)	10.730	1	10.730	6.925	0.0251	
Time (C)	58.736	1	58.736	37.909	0.0001	
AB	15.862	1	15.862	10.237	0.0095	
AC	0.249	1	0.249	0.161	0.6968	
BC	29.544	1	29.544	19.068	0.0014	
A <sup>2</sup>	425.217	1	425.217	274.446	< 0.0001	
B <sup>2</sup>	5.412	1	5.412	3.493	0.0912	
C <sup>2</sup>	6.871	1	6.871	4.435	0.0615	
Residual	15.494	10	1.549			
Lack of Fit	11.984	5	2.397	3.415	0.1019	not significant
Pure Error	3.509	5	0.702			
Cor Total	921.961	19				

**Table 3:** Statistical information for ANOVA

Parameter	Value
R <sup>2</sup>	0.983
Adjusted R <sup>2</sup>	0.968
Predicted R <sup>2</sup>	0.913
Mean	15.970
Standard deviation	1.245
CV %	7.794
Adequate Precision	24.031

*Effect of independent variables on the yield of oil from ginger:* The effect of the variables on the yield of oil from ginger is shown in the response surface plots in Figures 1-3. The plots which were generated from the statistical model aid in understanding the interactions between the factors and also to locate the optimum levels by varying the values of two variables with the response while keeping the other two variables constant.

Figure 1 shows the interaction effect of extraction temperature and volume of diluent on the yield of ginger oil. It is seen from Figure 2 that there is an

increase in the yield of ginger oil with an increase in extraction temperature, with a maximum yield of ginger oil in the extraction temperature range of 84 °C to 86 °C. Beyond this value range, the ginger oil yield starts to decrease with an increase in the extraction temperature. This negative effect may be due to the evaporation of the ethanol solvent after reaching its boiling point of 87 °C. It was however generally noticed that an increase in solvent volume resulted in an increase in ginger oil yield. Figure 2 shows the interaction effect of extraction temperature and extraction time on the yield of ginger oil

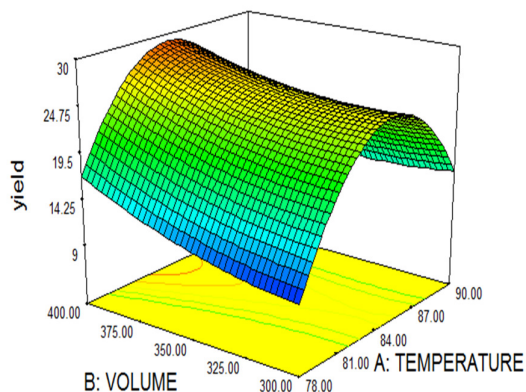


Fig 1: Response surface plot showing the effect of volume of diluent and extraction temperature on the yield of oil from ginger

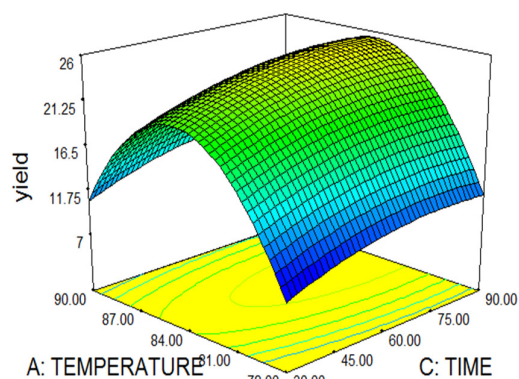


Fig 2: Response surface plot showing the effect of Extraction temperature and Extraction time on the yield of ginger extract.

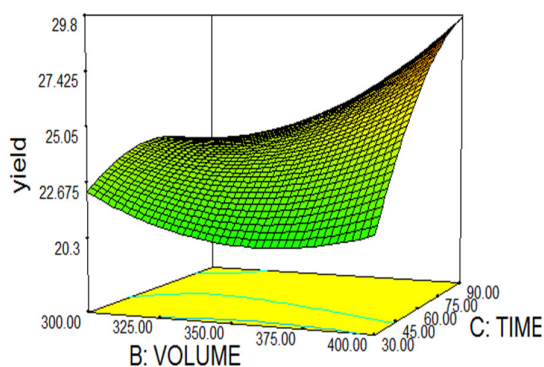


Fig 3: Response surface plot showing the effect of Volume of diluent and extraction time on the yield of ginger extract.

. The extraction time is seen to have a positive effect on the yield of ginger oil. This trend may be due to the fact that a longer extraction time will result in the dissolution of more ginger oil resulting in an increased yield. Figure 3 shows the interaction effect of the volume of diluent and extraction time on the yield of ginger oil. It is seen that an increase in the volume of diluent only resulted in a slight increase in yield of

ginger oil; whereas an increase in extraction time generally resulted in a significant increase in yield of ginger oil. The extraction temperatures, volume of diluent and extraction time were optimized based on the model developed. The optimum yield of ginger oil predicted by the model was 21.451%. This optimum yield was obtained at an extraction temperature of 82.22 °C, a volume of diluents of 300 ml and extraction time 30 minutes.

**Validation of statistical model:** The validity of the results predicted by the regression model was ascertained by carrying out repeated experiments under optimal extraction conditions. The results obtained from three replications demonstrated that the average yield of ginger oil of 20.533 % was close to the predicted value of 21.451 %, giving an error of 4.47 %. The low percentage error of the predicted yield of oil shows that there is good agreement between the predicted and experimental values indicating the validity of the response model.

**Conclusion:** D-optimal was used to evaluate and to optimize the effect of extraction temperature, diluent volume and extraction time on the yield of ginger oil. Second order polynomial has been shown to adequately predict the yield of ginger oil and also to optimize extraction parameters for the extraction of oil from ginger. It was found that the yield of ginger generally increased with an increase in extraction time, slightly increased with an increase in diluent volume, but decreased when the temperature was above 86 °C.

**REFERENCES**

Abdurahman, NH; Azhari, NH (2013). Performance of Ultrasonic Membrane Anaerobic System (UMAS) in Membrane Fouling Control. *Int. J. Eng. Sci. Inno Technol.* 2(6): 480-491.

Annadurai, G; Juang, RS; Lee, DJ (2002). Factorial design analysis for adsorption of dye on activated carbon beads incorporated with calcium alginate. *Adv. Environ. Res.* 6 (2):191-198,

Brasil, JL; Martins, LC; Ev, RR; Dupont, J; Dias, SLP; Sales, JAA; Airoidi, C; Lima, EC (2005). Factorial design for optimization of flow-injection preconcentration procedure for copper(II) determination in natural waters, using 2-aminomethylpyridine grafted silica gel as

- adsorbent and spectrophotometric detection. *Int. J. Environ. Anal. Chem.* 85: 475–491.
- Box, GEP; Hunter, WG; Hunter, JS (1978). *Statistics for Experimenters—An Introduction to Design, Data Analysis and Model Building*, John Wiley and Sons Inc, N.Y.
- Cao, G; Ren, N; Wang, A; Lee, DJ; Guo, W; Liu, B; Feng, Y; Zhao, Q (2009). Acid hydrolysis of corn stover for biohydrogen production using *Thermoanaerobacterium thermosaccharolyticum* W16. *Int. J. Hydrogen Energ.* 34: 7182–7188.
- Carmona, MER; da Silva, MAP; Leite, SGF (2005). Biosorption of chromium using factorial experimental design. *Process Biochem.* 40: 779–788.
- Dugasani, S; Pichika, MR; Nadarajah, VD; Balijepalli, MK; Tandra, S; Korlakunta, JN (2010). Comparative antioxidant and anti-inflammatory effects of [6]-gingerol, [8]-gingerol, [10]-gingerol and [6]-shogaol. *J. Ethnopharmacol.* 127(2): 515–20
- Famurewa, AV; Emuekele, PO; Jaiyeoba, KF (2011). Effect of drying and size reduction on the chemical and volatile oil contents of ginger (*Zingiber officinale*). *J. Med. Plants Res.* 14: 2941–2944.
- Guan, X; Yao, H (2008). Optimization of Viscozyme L-assisted extraction of oat bran protein using response surface methodology. *Food Chem.* 106(1): 345–351
- Hawthorne, SB (1990). Analytical-scale supercritical fluid extraction. *Anal. Chem.*, 62 (11): 633A–642A
- Herrero, M; Martín-Álvarez, PJ; Senorans, FJ; Cifuentes, A; Ibanez, E (2005). Optimization of accelerated solvent extraction of antioxidants from *Spirulina platensis* microalga. *Food Chem.* 93 (3): 417–423
- Huang W; Zhenshan L; Niu H; Li D; Zhang, J(2008). Optimization of operating parameters for supercritical carbon dioxide extraction of lycopene by response surface methodology. *J. Food Eng.* 89: 298–302.
- Pavan, FA; Gushikem, Y; Mazzocato, AC; Dias, SLP; Lima, EC (2007). Statistical design of experiments as a tool for optimizing the batch conditions to methylene blue biosorption on yellow passion fruit and mandarin peels. *Dyes Pigm.* 72: 256–266.
- Kunamneni, A; Singh, S (2005). Response surface optimization of enzymatic hydrolysis of maize starch for higher glucose production. *Biochem. Eng. J.* 27(2): 179–190.
- Mason, RL; Gunst, RF; Hess, JL (1989). *Statistical Design and Analysis of Experiments*. John Wiley and Sons, Inc., N.Y.
- Mekuriya, W; Mekibib, B (2018). Review on the Medicinal Values of Ginger for Human and Animal Ailments. *J. Vet. Sci. Technol.*, 9(2): 519–523
- Montgomery, DC (2005). *Design and Analysis of experiments*. Wiley and Sons Inc, N.Y.
- Rezzoug, SA; Boutekedjiret, C; Allaf, K (2005). Optimization of operating conditions of rosemary essential oil extraction by a fast controlled pressure drop process using response surface methodology. *J. Food Eng.* 71: 9–17.
- Tan, QLP; Kieu, XNT; Kim, NHT; Hong, XNT (2012). Application of Response Surface Methodology (RSM) in condition optimization for essential oil production from *Citrus latifolia*. *Emir J. Food Agric.* 24(1): 25–30.