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Screening the Effect of Process Parameters on the Yields of Bioactive Compounds Extraction from *Zingiber officinale* Using Full Factorial Design

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Abstract

In this study the use of solvent extraction technique for the extraction of bioactive compounds from Zingiber officinale (Ginger) *was investigated. The effect of three parameters namely extraction temperature, volume of solvents and the extraction time on the extract yield was screened using full factorial experimental design. The experimental runs which consist of the treatment combinations of the three parameters were performed in Soxhlet extraction apparatus to obtain various yields of the ginger oil extract. The results revealed that the extraction temperature, volume of solvents and the extraction time have significant individual and interaction effect on the extract yield resulting in highest extract yield of 29.48%. Based on the pareto analysis, the individual effects of the three parameters on the extract yield can be ranked as follows: extraction temperature > volume of solvent > extraction time. While the interaction effects can be ranked as extraction temperature-volume of solvent > extraction temperature-extraction time > volume of solvent-extraction time. The GC-MS analysis of the ginger oil extract revealed the presence of 24 different components with various medicinal potentials.*

1. Introduction

Zingiber officinale is a medicinal plant which belong to the family Zingiberaceae with origin from Island Southeast Asia [1]. It is commonly used as condiment and spices for cooking [2]. *Zingiber officinale* has been reported to be rich in bioactive compounds with high medicinal values which can be employed as antioxidants and in therapeutic treatment [3]. Ghasemzadeh et al. [4] reported the antioxidant activities, phenolic and flavonoids contents of different parts of ginger plant. The study revealed that ginger displayed high antioxidant activities which validates its medicinal potential. Also, Nicoll and Henein [3] in their extensive review reported the various application of ginger extracts as a hot remedy for cardiovascular diseases. The review showed that various laboratory tests on animals indicated suitability of ginger extract as anti-inflammatory, antioxidant, anti-platelet, hypotensive and hypolipidemic effect. The presence of various bioactive compounds in ginger has been reported to be responsible for its medicinal potentials. Nishidono et al. [5] identified various chemical constituents of ginger that can be employed for thermogenesis. Some of the bioactive compound identified include [10]-gingerol [10]-shogaol, [10]-gingerdiols and [10]-gingerdiols 3,5-diacetate.

Exploring the full medicinal potential of ginger is often constrained with challenges of extracting the bioactive compounds. To this effect, several techniques have been employed for the extraction of these bioactive compound from ginger. Amiri et al.[6] and Ko et al.[7] employed subcritical water extraction technique for extraction of bioactive compounds from ginger. The authors investigated the effect of parameters such addition of co-solvents, temperature, retention time and particle size on the yield of the extraction. The use of supercritical fluid extraction has also been used for extraction of bioactive compounds from ginger as reported by Salea et al.[8]. The authors investigated the influence of pressure, temperature, and carbon dioxide flow rate on the extraction yield from ginger. Also, enzyme-assisted three phase partitioning techniques has been reportedly used for the extraction of oleoresin from ginger. The study revealed that the yield of the oleoresin obtained from ginger was improved by the enzymatic pre-treatment. ionic liquid-based ultrasonic-assisted extraction has been used for simultaneous extract of hydrophobic and hydrophilic bioactive compounds from ginger. Effects of parameters such as ionic liquid type, ionic liquid concentration, solid/liquid ratio, ultrasonic power, extraction temperature and extraction time on the recovery of gingerols were investigated. The study revealed that the ionic liquid-based ultrasonic-assisted extraction substantially enhanced the yield of total gingerols and shortened the extraction time. A recent study by Uwadiae et al. [9] investigated the optimization of oil yield from ginger using D-optimal approach. An optimum condition of 82.22 ^{o}C, 300 ml and 30 min were obtained for the extraction temperature, volume of solvent and extraction time, respectively. However, initial screening of the factors to determine their significant effect for optimization studies was not reported. The present study therefore focuses on screening the effect of extraction temperature, volume of solvent and extraction time on bioactive compounds from ginger using full factorial design of experiment. Full factor design has been employed for screening of process parameters prior to optimization study as reported by Hossain et al.[10].

2. Methodology

2.1 Materials

The ginger used for the study was purchased from a local market in Benin City, Edo State, Nigeria. The ginger was cut into smaller piece in order to facilitate the rate of drying. Subsequently, the gingers were sun dried for about 3 weeks to reduce its moisture and prevent bio-contamination by fungal growth. The dried ginger was thereafter ground to powdery form for ease of solvent penetration.

2.2 Method

Full factorial design was employed to generate the number of experimental runs based on the extraction temperature (78 ^{o}C-90 ^{o}C), extraction time (30 -90 min) and volume of the solvent (300-400). The detail of the experimental design is shown in Table 1. The extraction process was performed batchwise using Soxhlet apparatus as reported by Azian et al.[11]. For a typical run, a fixed mass (30g) of dried ginger in powdery form was placed in the extractor section of the Soxhlet apparatus. For the first run shown in Table 2, 300 ml of ethanol was measured into the distillation flask. The heating mantle was set to a temperature of 90 ^{o}C for 90 min. The vapour containing the bioactive component were trapped at the Soxhlet extractor. After 90 min, the ethanol in the extractor was poured back into the distillation flask and the one contained in the ginger sample was pressed into the distillation flask. The extract was collected from the distillation flask and subsequently analyzed using GC-MS to determine the various components. The procedure was repeated for all the runs at constant mass of 30g. The yield of the extracted ginger oil was calculated as shown in Equation (1)

$$\text{Yield (ginger oil)}(\%) = \frac{\text{C (extraction) (g)}}{\text{W (sample) (g)}}\ x\ 100 \qquad (1)$$

where
Y is the yield of the ginger oil (%), C is capacity of the ginger oil by the Soxhlet apparatus, W is the weight of the ginger.

Table 1: Detail of the parameters used for the Full factorial experimental design

Factors	Name	Units	Low Actual	High Actual	Low Coded	High Coded	Mean	Std. Dev.
A	Extraction Temperature	ºC	78	90	-1	1	84	6
B	Volume of solvent	ml	300	400	-1	1	350	50
C	Extraction time	min	30	90	-1	1	60	30

3. Results and Discussion

3.1 Full factorial experimental design

The extraction yields obtained from various combinations of the parameters in the experimental runs are summarized in Table 2. It can be seen that the extraction yield varies depending on combination of the factors. The lowest extraction yield of 13.25% was obtained using 300 ml of the ethanol, at 78ºC for 30 min. While the highest extraction yield of 29.48% was obtained using 400 ml of ethanol, at 90 ºC for 90 min. The normal plots of the residual of the actual values of the extraction yield is depicted in Figure 1. The data points are normally distributed.

Table 2: Detail of full factorial experimental design showing the factors and the response

Experimental runs	Extraction Temperature (ºC)	Volume of solvent (ml)	Extraction Time (min)	Extraction yield (%)
1	78	300	90	24.59
2	78	400	30	17.00
3	78	400	30	17.20
4	78	400	90	28.00
5	90	300	90	26.21
6	90	400	90	29.48
7	90	400	90	29.45
8	78	300	30	13.23
9	90	300	30	17.95
10	78	400	90	23.95
11	90	300	90	26.24
12	78	300	30	13.25
13	78	300	90	24.61
14	90	300	30	17.99
15	90	400	30	18.00
16	90	400	30	18.21

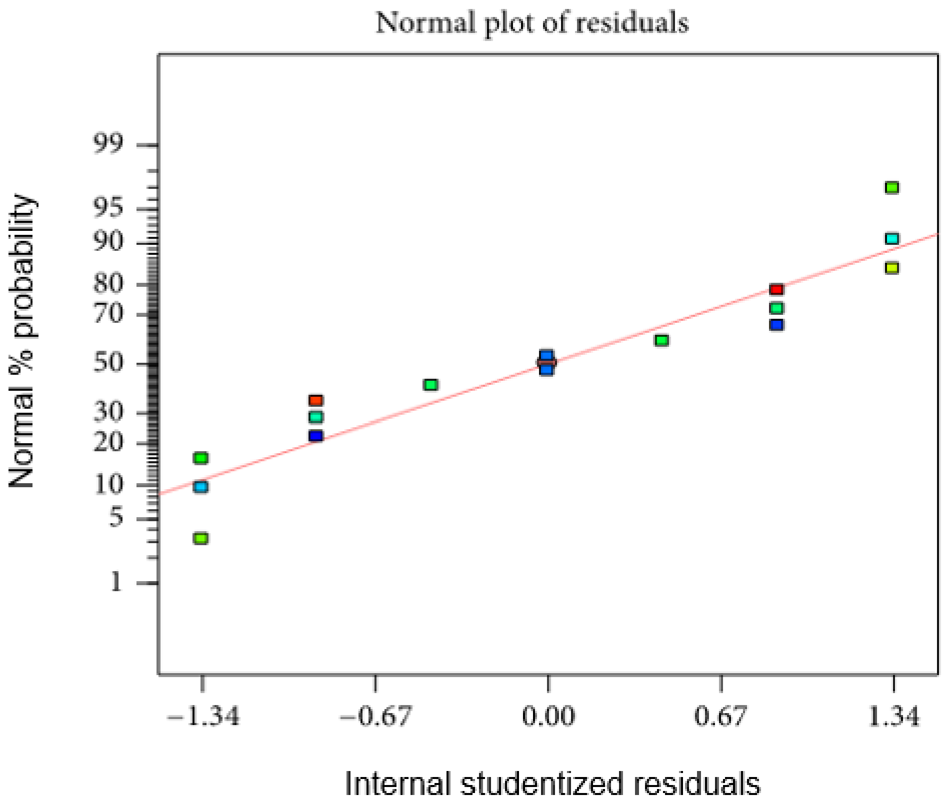


Figure 1: Normal plots of the residuals

The three-dimensional plots showing the effects of the three factors on the extract yields are depicted in Figure 2-4. Figure 2 shows the effect of volume of solvent and extraction temperature on the extract yield. The volume of the solvent and the extraction yield have a direct linear effect on the extract yield. This implies that the extract yield increases with increase in the volume of ethanol and temperature. This can further be confirmed from the analysis of variance (ANOVA) summarized in Table 3. The p-values of the individual effect of volume of solvent and the extraction temperature are < 0.05 which implies that they both have significant effect on the extract yield. Moreover, the interaction effect of the volume of solvent and the extraction temperature which is < 0.05 is significant on the extract yield. The highest individual effect of volume of solvent was achieved using 400 ml of ethanol to obtain 21.55% of ginger oil extract. While the highest individual effect of extraction temperature was achieved at 90 ^{o}C resulting in 22.48% ginger oil extracted from ginger. Chen et al.[12] has reported that the extraction temperature significant influenced ginger oil yield. Also, Alfaro et al.[13] reported that the use of ethanol as solvent of extraction significantly enhanced ginger oil yield compared to solvent such as hexane, petroleum ether and dichloromethane.

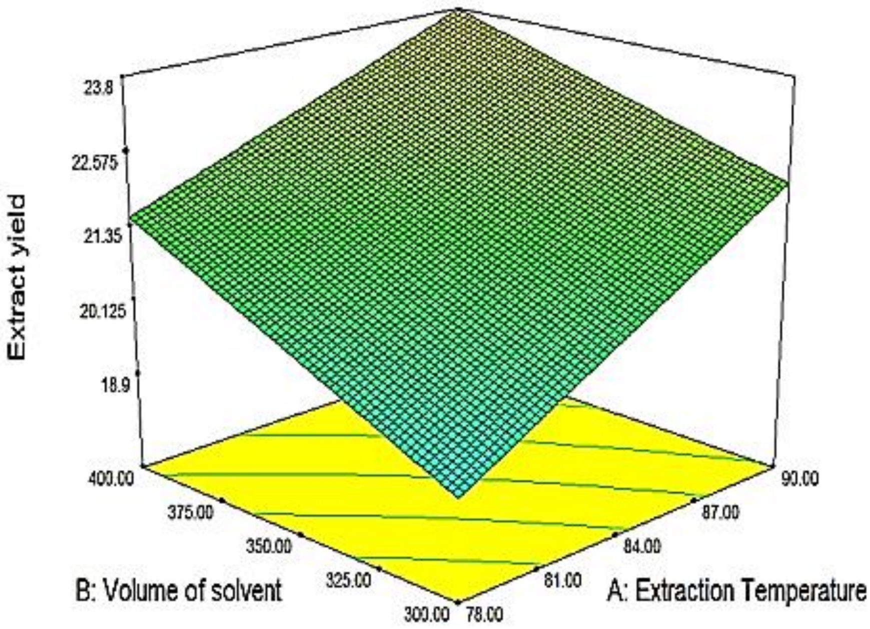


Figure 2: Three-dimensional plots showing the effect of volume of solvent and extraction temperature on the extract yield

Figure 3 shows the three-dimensional plot of the effect of extraction time and extraction temperature on the extract yield. Both the extraction time and extraction temperature have direct linear effect on the extract yield which implies that an increase in either the extraction time or the extraction temperature will result in a corresponding increase in the extract yield [12]. As it can be seen from Table 3, both the individual and the interaction effects of the extract time and extraction temperature on the extract yield are significant since their p-values are < 0.05. At extraction time of 90 min, there was a significant effect on the extract yield which was obtained as 25.45% while extraction temperature has significant individual effect on the extract yield at 90 ^{o}C [8].

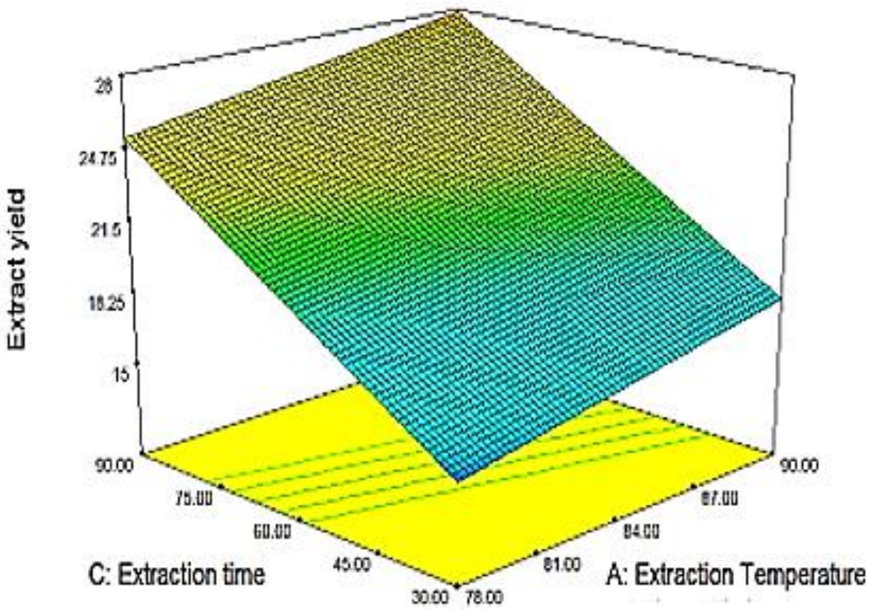


Figure 3: Three-dimensional plots showing the effect of extraction time and extraction temperature on the extract yield

Figure 4 depicts the three-dimensional plot showing the effect of extraction time and the volume of solvent on the extract yield. A direct linear effect of the extraction time and the volume of solvent on the extract can be inferred from Figure 4. This implies that an increase in either the extraction time or volume of the solvent result in a corresponding increase in the extract yield. This can further be confirmed from the individual and the interaction effects of the two parameters which has p-values < 0.05, an indication that these parameters are significant at both individual and interaction level. The ranking of the individual and interaction effects of the extraction temperature, volume of solvent and extraction time on the extract yield is depicted in Figure 5. The individual effects of the three parameters on the extract yield can be ranked as follows: extraction temperature > volume of solvent > extraction time. While the interaction effects can be ranked as extraction temperature-volume of solvent > extraction temperature-extraction time > volume of solvent-extraction time.

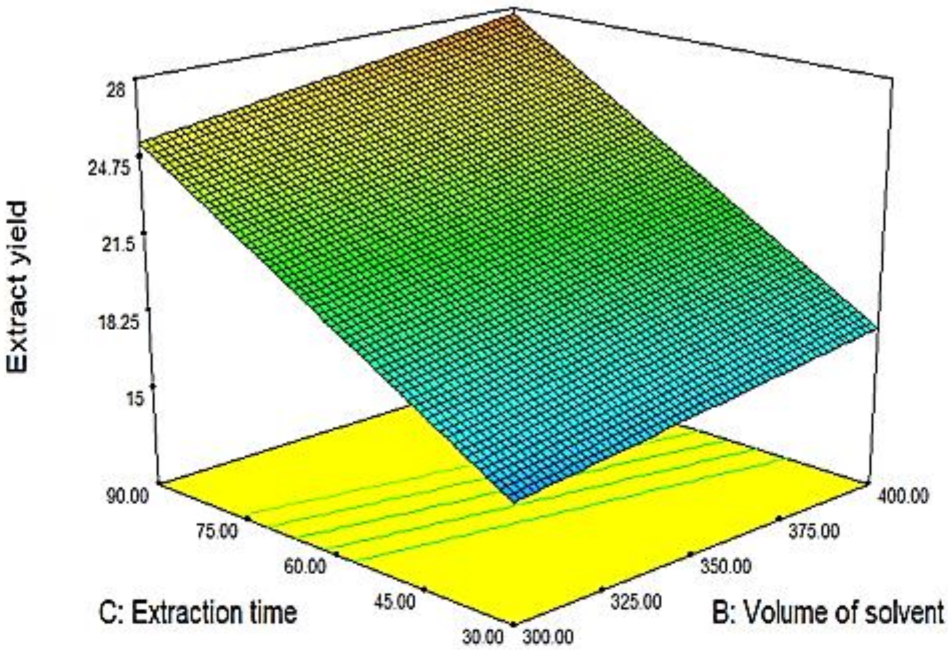


Figure 3: Three-dimensional plots showing the effect of extraction time and extraction temperature on the extract yield

Table 3: Analysis of variance of the three factors investigated

Source	Sum of Squares	Df	Mean Square	F value	P-value Prob>F	
Model	456.79	7	65.26	199.87	<0.0001	Significant
A-temperature	19.56	1	19.56	487.61	<0.0001	
B-volume	0.035	1	0.035	1725.61	<0.0001	
C-time	377.82	1	377.88	30.06	<0.0001	
AB	8.75	1	8.75	7.68	0.0150	
AC	23.64	1	23.64	32.17	<0.0001	
BC	0.96	1	0.96	43.08	<0.0001	
ABC	26.04	1	26.04		0.0863	
Curvature	19.78	1	19.78	4.39	0.5728*	

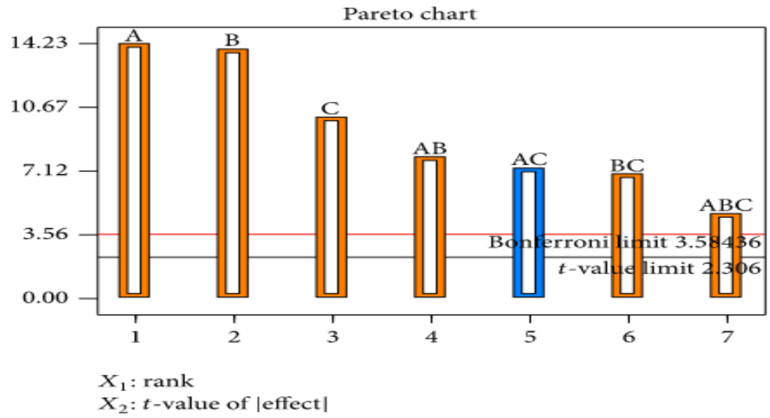
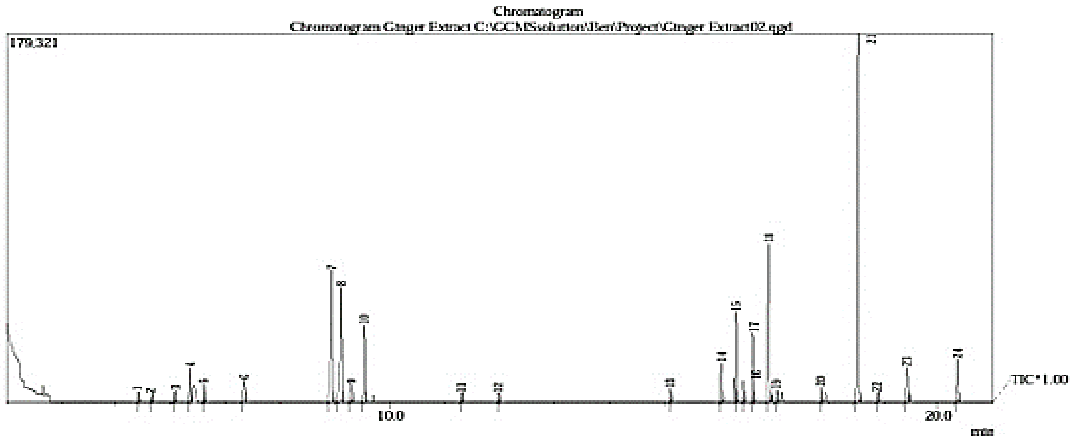


Figure 5: Pareto chart showing the order of significance of the individual and the interaction effects of the parameters on the extract yield

3.2 GC analysis of the ginger oil extract

The chromatogram of the GC-MS analysis of the ginger oil extract is depicted in Figure 6. The chromatogram shows the presence of 24 different phenolic compounds in the ginger oil extract which is consistent with that reported by Salea et al.[8]. Some of the bioactive compounds identified from the ginger oil extract include gingerol, paradol, Trifluoromethyl peroxynitrate, Formamide, N-Formyl-N-meth Propionanilide, 3-bicycloy that exhibit antioxidants, anti-tumor and anti-inflammatory properties [14]. Gingerol also known as [6]- gingerol has been identified as one of the most bioactive components in ginger. It possesses some interesting pharmacological activities like analgesic effect [15].



Peak Report TIC

Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H	Mark	Name
1	5.403	5.383	5.425	8683	0.76	5098	0.79	1.70		1H-Pyrrole, 2-ethyl- $$ 2-Ethylpyrrole $$
2	5.646	5.633	5.658	3582	0.31	3642	0.56	0.98	MI	Formamide, N-formyl-N-methyl- $$ Form
3	6.076	6.050	6.100	10971	0.96	5542	0.85	1.98		Formamide, N-formyl-N-methyl- $$ Form
4	6.357	6.333	6.383	23474	2.05	16623	2.56	1.41		Trifluoromethyl peroxynitrate $$ Trifluor
5	6.601	6.583	6.625	12742	1.12	8854	1.36	1.44		3-Buten-2-one, 4-(1H-pyrrol-2-yl)-, (E)- $
6	7.331	7.283	7.358	27508	2.41	10607	1.63	2.60		3-Buten-2-one, 4-(1H-pyrrol-2-yl)-, (E)- $
7	8.929	8.875	8.958	126086	11.04	64122	9.88	1.97		Trifluoromethyl peroxynitrate $$ Trifluor
8	9.100	9.042	9.125	123594	10.82	56064	8.63	2.20		Benzene, 1-(1,5-dimethyl-4-hexenyl)-4-me
9	9.297	9.275	9.317	14752	1.29	8539	1.32	1.73		1,3-Cyclohexadiene, 5-(1,5-dimethyl-4-he
10	9.540	9.500	9.567	60321	5.28	37286	5.74	1.62		Ethanone, 1-cyclopropyl-2-(4-pyridinyl)- $
11	11.313	11.300	11.333	5785	0.51	4183	0.64	1.38	MI	N-(Dimethylthiophosphinyl)ethylamine $
12	11.974	11.958	11.992	5646	0.49	4204	0.65	1.34		N-(Dimethylthiophosphinyl)ethylamine $
13	15.112	15.100	15.125	6180	0.54	6292	0.97	0.98	MI	Trifluoromethyl peroxynitrate $$ Trifluor
14	16.034	16.017	16.058	25782	2.26	19043	2.93	1.35		Borane, ethylisopropylmethyl-
15	16.325	16.300	16.350	62182	5.44	43701	6.73	1.42		Propionanilide, 3-(bicyclo[2.2.1]hept-2-yl
16	16.445	16.425	16.467	17234	1.51	10350	1.59	1.67		Propionanilide, 3-(bicyclo[2.2.1]hept-2-yl)
17	16.627	16.608	16.650	43412	3.80	33620	5.18	1.29		Trifluoromethanesulfonic anhydride $$ M
18	16.901	16.867	16.942	130598	11.43	70997	11.86	1.70		4-(4-Methylpent-3-enyl)-3,6-dihydro-1,2-d
19	17.062	17.042	17.083	9523	0.83	5610	0.86	1.70		2R-Acetoxymethyl-1,3,3-trimethyl-4t-(3-m
20	17.865	17.842	17.892	13575	1.19	7303	1.12	1.86		Trifluoromethyl peroxynitrate $$ Trifluor
21	18.532	18.483	18.575	330068	28.89	179321	27.62	1.84		N-(Dimethylthiophosphinyl)ethylamine $
22	18.899	18.875	18.925	9893	0.87	4596	0.71	2.15	MI	N-(Dimethylthiophosphinyl)ethylamine $
23	19.419	19.383	19.450	34523	3.02	16779	2.58	2.06		Gingerol $$ 3-Decanone, 5-hydroxy-1-(4-
24	20.346	20.317	20.375	36210	3.17	20927	3.22	1.73		5-Methoxycarbonylpyridine-2-carboxylic
				1142384	100.00	649303	100.00			

Figure 6: The chromatogram of the GC-MS analysis of the ginger oil extract

4. Conclusion

This study examined the effect of extraction temperature, volume of solvent and extraction time on the extract yield on ginger oil using full factorial experimental design. The various treatment combinations of the parameters resulted in different values of the extract yields of the ginger oil. The three factors were found to have significant individual and interaction effects on the extract yields of the ginger oil. The individual effects can be ranked as extraction temperature > volume of solvent > extraction time. While the interaction effects can be ranked as extraction temperature-volume of solvent > extraction temperature-extraction time > volume of solvent-extraction time. Based on the GC-MS analysis, 24 different bioactive compounds with various medicinal potentials were identified in the ginger oil extract. The optimum conditions of the bioactive compounds extraction from the ginger can further be investigated using the three parameters.

References

[1] Kou X, Ke Y, Wang X, Rahman MRT, Xie Y, Chen S, et al. Simultaneous extraction of hydrophobic and hydrophilic bioactive compounds from ginger (Zingiber officinale Roscoe). Food Chem 2018;257:223–9. doi:10.1016/j.foodchem.2018.02.125.

[2] Varakumar S, Umesh KV, Singhal RS. Enhanced extraction of oleoresin from ginger (Zingiber officinale) rhizome powder using enzyme-assisted three phase partitioning. Food Chem 2017;216:27–36. doi:10.1016/j.foodchem.2016.07.180.

[3] Nicoll R, Henein MY. Ginger (Zingiber officinale Roscoe): A hot remedy for cardiovascular disease? Int J Cardiol 2009;131:408–9. doi:10.1016/j.ijcard.2007.07.107.

[4] Ghasemzadeh A, Jaafar HZE, Rahmat A. Antioxidant activities, total phenolics and flavonoids content in two varieties of malaysia young ginger (Zingiber officinale Roscoe). Molecules 2010;15:4324–33. doi:10.3390/molecules15064324.

[5] Nishidono Y, Saifudin A, Nishizawa M, Fujita T, Nakamoto M, Tanaka K. Identification of the chemical constituents in Ginger (Zingiber officinale) responsible for thermogenesis. Nat Prod Commun 2018;13:869–73. doi:10.1177/1934578x1801300722.

[6] Amiri ZN, Najafpour GD, Mohammadi M, Moghadamnia AA. Subcritical Water Extraction of Bioactive Compounds from Ginger (Zingiber officinale Roscoe). Int J Eng 2018;31:1991–2000. doi:10.5829/ije.2018.31.12c.01.

[7] Ko MJ, Nam HH, Chung MS. Conversion of 6-gingerol to 6-shogaol in ginger (Zingiber officinale) pulp and peel during subcritical water extraction. Food Chem 2019;270:149–55. doi:10.1016/j.foodchem.2018.07.078.

[8] Salea R, Veriansyah B, Tjandrawinata RR. Optimization and scale-up process for supercritical fluids extraction of ginger oil from Zingiber officinale var. Amarum. J Supercrit Fluids 2017;120:285–94. doi:10.1016/j.supflu.2016.05.035.

[9] Uwadiae SE, Aifesume H, Ayodele BV. Effect of extraction temperature, time and volume of diluent on oil yield from ginger (*Zingiber officinale*) in a batch-mode process. J Appl Sci Environ Manag 2019;23:611. doi:10.4314/jasem.v23i4.5.

[10] Hossain MA, Ayodele B V., Cheng CK, Khan MR. Syngas production from catalytic CO_2 reforming of CH_4 over $CaFe_2O_4$ supported Ni and Co catalysts: Full factorial design screening. Bull Chem React Eng Catal 2018;13:57–73. doi:10.9767/bcrec.13.1.1197.57-73.

[11] Azian MN, Anisa ANI, Iwai Y. Mechanisms of Ginger Bioactive Compounds Extract Using Soxhlet and Accelerated Water Extraction. Int J Chem Mol Nucl Mater Metall Eng 2014;8:444–8.

[12] Chen H, Chung C, Wang H, Huang T. Application of Taguchi Method to Optimize Extracted Ginger Oil in Different Drying Conditions. Int Conf Food Eng Biotechnol 2011;9:310–6.

[13] Alfaro MJ, Bélanger JMR, Padilla FC, Paré JRJ. Influence of solvent, matrix dielectric properties, and applied power on the liquid-phase microwave-assisted processes (MAP™)11MAP is a Trademark of Her Majesty the Queen in Right of Canada as represented by the Minister of the Environment. extraction of . Food Res Int 2003;36:499–504. doi:https://doi.org/10.1016/S0963-9969(02)00198-9.

[14] Vipin AV, Raksha Rao K, Kurrey NK, Anu Appaiah KA, Venkateswaran G. Protective effects of phenolics rich extract of ginger against Aflatoxin B1-induced oxidative stress and hepatotoxicity. Biomed Pharmacother 2017;91:415–24. doi:https://doi.org/10.1016/j.biopha.2017.04.107.

[15] Tohma H, Gülçin İ, Bursal E, Gören AC, Alwasel SH, Köksal E. Antioxidant activity and phenolic compounds of ginger (Zingiber officinale Rosc.) determined by HPLC-MS/MS. J Food Meas Charact 2017;11:556–66. doi:10.1007/s11694-016-9423-z.