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Application of Taguchi Method to Optimize Extracted Ginger Oil in Different Drying Conditions

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Abstract. Ginger (*Zingiber officinalis,* Roscoe), contains potent anti-inflammatory agents and oleoresin; consequently ginger oil has great anti-inflammatory effects. Supercritical Fluid Extraction (SFE) has received a great deal of attention because it is inert, inexpensive, easily available, odorless, tasteless and environment friendly. This study has successfully applied the SFE technique to extract a variety of functional compounds from drying ginger. This study applied the Taguchi method to determine optimum extraction conditions for drying ginger to produce a high yield of ginger oil. The control factors included reaction time, drying temperature, extraction pressure and particle size of the ginger power. The following optimal extraction conditions were obtained: 60 °C, 4000 psi and a ginger power size of less than 595 μm at a 90 min extraction time. Using optimal extraction conditions for drying ginger, the maximum yield of ginger oil is 2.74% and S/N ratios of 8.76. In addition to these results, the study found that high temperature would cause starch gelatinization, which might affect the extraction process and produce a lower yield of ginger oil.

Keywords: Ginger (*Zingiber officinalis,* Roscoe), supercritical fluid extraction (SFE), Taguchi method.

1. Introduction

Ginger, *Zingiber officinalis,* Roscoe, is a monocotyledonous herbaceous perennial belonging to the Zingiberaceae family, which is characterized by a pale-yellow pungent aromatic rhizome that is the important part of this spice. It contains oleoresin and essential oils [1]. Ginger is used worldwide as a food ingredient and medicine. It has long been used to treat many gastrointestinal disorders and is often promoted as an effective antiemetic [2,3]. Gingerols, the pungent principles in the rhizome of ginger, were reported to have antiemetic, analgesic, antipyretic, anti-inflammatory, chemopreventive, and antioxidant properties [4-6]. They were also suggested for treating various illnesses, including arterial sclerosis, migraine headaches, rheumatoid arthritis, high cholesterol, ulcers, and depression [7-9]. In ginger oil, 6-Gingerol, 6-shogaol, and other structurally related substances in ginger inhibit prostaglandin biosynthesis through suppressing 5-lipoxygenase or prostaglandin synthetase [10]. In general, low temperature drying usually can preserve active compounds in the material, which may decompose under the effects of high temperature [11-13]. However, heat treatment of ginger has been suggested in order to enhance its curative effect [14,15]. Due to these properties, it has gained considerable attention as a botanical dietary supplement in the US and Europe in recent years, and especially for its use in treating chronic inflammatory conditions [4].

A supercritical fluid extraction (SFE) technique is a separation process based on the contact of a substance containing an extractable compound with a solvent, such as CO_2 under supercritical conditions. SFE has become a promising separation technique [16]. Supercritical CO_2 fluid shows numerous characteristics as well, such as being inert, inexpensive, easily available, odorless, tasteless, and environmentally friendly [17]. The selectivity of the compound to be extracted is dependent on the density of the supercritical fluid, which can be altered by varying process conditions. Using this technique, pure organic extracts with no trace of the solvent can be obtained [18,19]. Hence, this study used SFE technology to extract ginger oils from dried ginger powder, which produces purer ginger oil.

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In order to obtain a high yield of product, optimization of the existing SFE process is necessary. The Taguchi method is a systematic application of analysis of experiments for the purpose of designing and improving product quality. It is used especially for evaluating several process factors at a time with the smallest number of experimental runs based on a table, known as the orthogonal array. The conclusions drawn from small scale experiments are valid over the entire experimental region spanned by the control factors and their settings [20]. In recent years, the Taguchi method has become a powerful tool employed in industry for improving productivity during research and development, so that high quality products can be produced quickly and at very low cost. These reports used the Taguchi method to optimize operation conditions in food science and engineering, such as fermentation processes [21-23], and agriculture product drying [23]. The objective of the present study was to apply the Taguchi method to determine the optimum extraction conditions for producing a high yield of ginger oil from drying ginger in different drying conditions.

2. Materials and Methods

2.1. Sample preparation

Fresh Ginger (*Zingiber officinalis,* Roscoe) was purchased from a local market in Pingtung City, in southern Taiwan, and was used in our experiments. The schematic representation of extraction and isolation of ginger oil from dried ginger slices by supercritical fluid extraction was shown in Fig. 1. After peeling and coring, samples were cut into slices about 2~3 mm thick, and 4.80~5.00 g of samples was prepared per batch at at -80 °C freeze drying (79340-01A, Labconco corporation, Missouri, USA) and 40-80 °C by hot-air drying (DV20, Sdom Apparatus Mfg. company, Tainan, Taiwan). The drying process was terminated when the water activity (A_w) reached 0.4 and made powder (D3V-10, Yu Chi Machinery CO., LTD., Chang Hua, Taiwan). The average moisture content of the material taken from inside the drying chamber was evaluated using an AOAC method [24,25].

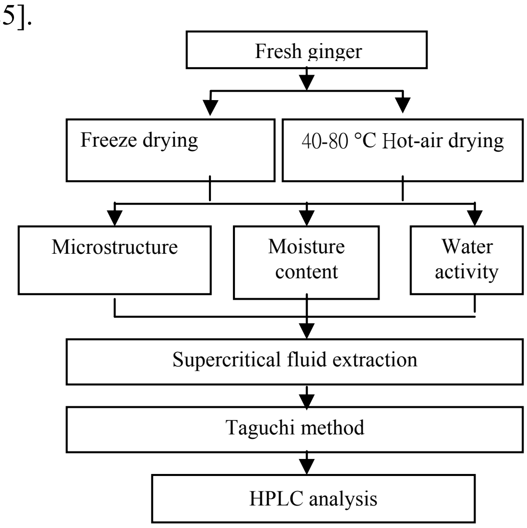


Fig. 1. Schematic representation of extraction and isolation of 6-shogaol compound from dried ginger slices by Supercritical fluid extraction.

2.2. Supercritical carbon dioxide extraction equipment

A batch-operated supercritical carbon dioxide extraction system (shown in Fig. 2), was used to carry out the extraction of lipophilic compounds from previously dried samples. Fifteen grams of powder ginger were loaded into a 100 mL extractor vessel. Supercritical fluid extractions were conducted at 3500 psi and 40-60 °C, during 90 minutes in a static mode, followed by a 30-minute dynamic extraction. The extract was collected in an empty volumetric flask, and then dissolved in acetone for subsequent analysis. The yield of ginger oils can be expressed as:

$$Y_{ginger\,oil}\,(\%) = \frac{C_{extraction}\,(g)}{W_{samples}\,(g)} \times 100\% \qquad (1)$$

where $Y_{ginger\,oil}$ (%) is the yield ratio of ginger oil by SFE system from drying sample, $C_{extraction}$ (g) is the capacity of ginger oil by SFE system and W_{sample} is the weight of drying ginger.

2.3. Water activity

Water activity (a_w) was measured with a water activity meter (AQUA LAB CX-2, Decagon Devices, Pullman, WA, USA). After beginning the experiment, a sample was removed from the drying cabinet, and the a_w was measured every hour using a water activity meter [26].

2.4. Taguchi method

The Taguchi method is mainly used to achieve high quality SFE and effectively reduces the number of experimental trials. The control factors shown in Table 1 include different kinds and levels of dynamic extraction time, extraction temperature, extraction pressure, and powder particle size during the SFE process. L_9 (3^4) orthogonal arrays were selected for the experiments, and there were 9 experimental runs with 4 factors (columns) and 3 levels (rows). The signal-to-noise ratio (S/N ratio, η) was calculated from experimental data by a loss function, which created a transformation function of the repetitive data to another value and was used as a measure of the variation present in the experiment [20,21]. The loss function depends on the criteria for the quality characteristic being optimized, and a high S/N ratio value is used as an indicator of optimal conditions [20]. The objective in this study was to find the optimum SFE conditions to enhance the yield of ginger oil on drying ginger. Here, the yield of ginger oil was treated as having larger-the-better performance characteristics. The loss function of S/N ratio (η_{ij}) larger-the-better can be expressed as [20]:

$$\eta_{ij} = -10 \log\left(\frac{1}{n}\sum_{k=1}^{n}\frac{1}{y_{ijk}^{2}}\right) \quad \text{(db)} \tag{2}$$

The optimal level of the process parameters were identified and could be computed based on the selected levels of the strong effect by significant factors for forecasting the optimal performance of the S/N ratio (η_f). The estimated S/N ratio (η_f) equation can be expressed as [20]:

$$\eta_f = \overline{\eta} + \sum_{i=1}^{\alpha}(\eta_i - \overline{\eta}) \quad (db) \tag{3}$$

Where, $\overline{\eta}$ is the total mean of the S/N ratio, and η_i is the S/N ratio for choosing the optimal procedure parameters and its level, and α is the number of the process parameters that significantly affected the optimal conditions on SFE process.

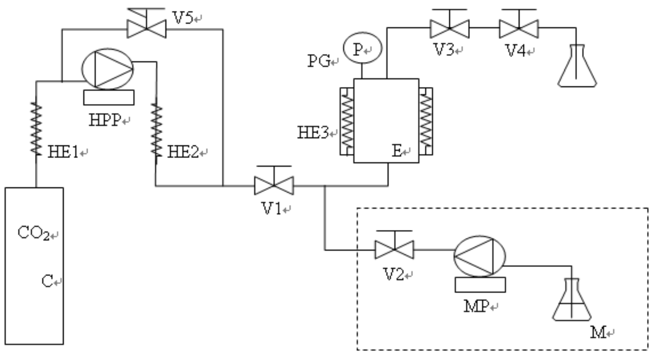


Fig. 2. Scheme of supercritical carbon dioxide extraction equipment at batch-stirred apparatus for synthesis under high pressure: (C) CO2 tank, (E) Extract vessel, (HE1-3) Heat exchangers, (HPP) High pressure pump, (M) Modifier, (MP) Modifier pump, (PG) Pressure gauges, (V1-3) exhaust valve, (V4) Needle valve, (V5) Back pressure valve.

Table 1. The control factors and levels in the SFE process

Factors	A	B	C	D
	Extraction time (min)	Temperature (°C)	Pressure (psi)	Powder particle size (µm)
Level 1	30	40	1500	> 2000
Level 2	60	50	2500	595 – 2000
Level 3	90	60	3500	< 595

2.5. Statistical analysis

Analysis of variance (ANOVA) was used to investigate the significance of the influence and confidence of the processing parameters on the performance [27,28]. A regression analysis of the experimental data was obtained using SAS® 8.2 (Statistical analysis system, Cary, NC, USA). Duncan's multiple-range test was used to compare the difference between means at a probability level < 0.05. The sum of squares of factors (SS_{Factor}) and error (SS_{Error}) were calculated as:

$$SS_{Factor} = \sum_{k=1}^{L} \frac{(\sum_{i=1}^{n} y_{ki})^2}{n} - \frac{T^2}{N} \quad (4)$$

$$SS_{Error} = \sum_{j=1}^{L} (SD)_j^2 (r-1) \quad (5)$$

Here, $L, k, n, i, y, T, N, M, j$ and r are the level number, factor level, and number of experimental results at each factor level, each experimental trial considered at factor level, experimental result, sum of all experimental results, total number of experimental results, number of experimental trials, each experimental trial and number of tests at each experimental trial, respectively. The factors variance (V_{Factor}) and F-ratio (F_{ratio}) were obtained as:

$$V_{Factor} = \frac{SS_{Factor}}{DOF_{Factor}} \quad (6)$$

$$F_{ratio} = \frac{V_{Factor}}{V_{Error}} \quad (7)$$

3. Results and Discussion

3.1. Drying Performance

The water content of fresh ginger is 9.05 kg H_2O/kg dry basis at a water activity of 0.98. A water content curve versus time of ginger slices under different drying processes is shown in Fig. 3. Results show that 80 °C hot-air drying had the fastest drying rate of all the drying processes. It took 2 hrs to reach a moisture content of 0.35 kg H_2O/kg dry basis, at a water activity of 0.32. The second one was 70 °C hot-air drying at 22 hrs. Among them, the freeze drying took the longest, requiring at least 14 hrs to reach a moisture content under 0.40 kg H_2O/kg dry basis. For 40 °C hot-air drying, it took 21.5 hrs to reach moisture content from 9.05 to 0.35 kg H_2O/kg dry basis, whereas traditional solar drying took 46 hrs to reach the same value.

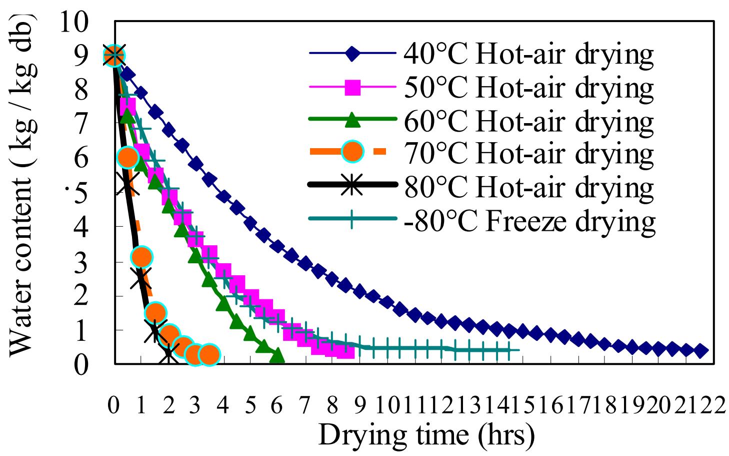


Fig. 3. The drying characteristic curve of ginger slices under different drying temperature.

3.2. Production of ginger oil

Results of the L_9 (3^4) orthogonal array for producing ginger oil on the SFE process are shown in Table 2. The S/N ratio was calculated from the average experimental data (the yield of ginger oil) by a loss function for the higher-the-better performance as Eq. 2, and as shown in Table 2. The results showed in Fig. 2 on the optimal SFE conditions for $A_3B_3C_3D_3$, meaning those with less than 595 μm ginger powers at 3500 psi and 60 °C to extract for 90 min. Summary results of the ANOVA are shown in Table 3, and indicated that all of the selected factors were significant parameters at the 95% confidence level ($p < 0.05$) on the SFE process.

The statistical optimization method used provides a model for predicting the optimal performance (η_f) as shown in Eq. 3. The expected values of the optimal S/N ratio and productivity were 8.14 *db* and 2.55%, respectively. A confirmation run was conducted based on these optimal conditions and the response was 8.76 *db*, in which the corresponding production of ginger oil was 2.74%. In the validation experiment, the study obtained a maximum yield of ginger oil under optimal conditions which was enhanced 4-fold higher than original conditions (0.70%).

Table 2. The results of L9 (34) orthogonal array experiments for different operation conditions on the SFE process and its signal-to-noise ratio (S/N ratio) on the yield of ginger oil

Trial No.	Extraction time (min)	Temperature (°C)	Pressure (psi)	Extraction time (min)	Ginger oil yield (%)	Standard deviation	S/N ratio (*db*)
1	Level 1	Level 1	Level 1	Level 1	0.70	0.06	-3.12
2	Level 1	Level 2	Level 2	Level 2	1.58	0.10	3.95
3	Level 1	Level 3	Level 3	Level 3	2.10	0.21	6.39
4	Level 2	Level 1	Level 2	Level 3	1.30	0.13	2.19
5	Level 2	Level 2	Level 3	Level 1	1.48	0.17	3.30
6	Level 2	Level 3	Level 1	Level 2	1.44	0.38	2.70
7	Level 3	Level 1	Level 3	Level 2	1.82	0.27	5.05
8	Level 3	Level 2	Level 1	Level 3	1.58	0.13	3.95
9	Level 3	Level 3	Level 2	Level 1	1.57	0.42	3.47
Average					1.51	0.21	3.10

Table 3. Analysis of variance (ANOVA) of factors affecting ginger oil SFE process

Factor	Sum of squares	DOF	Variance	*F*-ratio	Confidence (%)	Significant
A	0.2142	2	0.1071	3.7716	95.71	*
B	0.5757	2	0.2878	10.1350	99.89	*
C	0.9386	2	0.4693	16.5247	99.99	***
D	0.6025	2	0.3013	10.6079	99.91	**
Error	0.5112	18	0.0284			

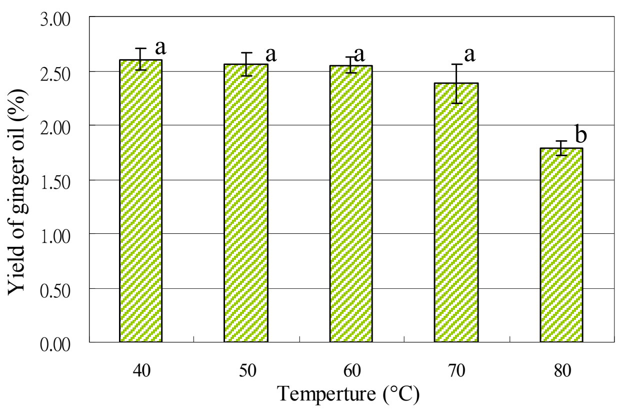
***: Significant at least at the 0.1% level, $p < 0.001$

** : Significant at least at the 1% level, $p < 0.01$

* : Significant at least at the 5% level, $p < 0.05$

3.3. Ginger oil of SFE

The study has focused on extracting ginger oils by optimal operation conditions on the SFE process from different drying temperatures, and the results are shown in Fig. 5. It was found that the yield of 40 °C to 70 °C hot-air drying ginger has no significant influence ($p < 0.05$), and the yield of ginger oil was 2.4 % to 2.6%. But, the results also show unambiguously that the yield of ginger oil is reduced when the drying temperature is over 70 °C. The study assumes that the ginger, at high drying-temperatures, may exhibit starch gelatinization. Hence, the yield of ginger oil in 80 °C hot-air drying decreased to 1.8%. Furthermore, the starch gelatinization of ginger would indeed affect the ginger oil extraction yield in the SFE process, and it also generated a bad smell during drying process.



[ab] Means with the same letters in a rectangle do not significantly differ from each other (by Duncan's multiple-range test, $p < 0.05$).

Individual bars represent data as the mean of 3 replicates ± the standard deviation.

Fig. 5 Extraction yield of ginger oil under different temperature drying methods in SFE process.

4. Conclusions

The study successfully applied the Taguchi method to determine optimal SFE conditions for high yields of ginger oil, and was validated by running a four-factor, three-level experimental design. The dynamic extraction time, extraction temperature, extraction pressure, and powder particle size during the SFE process were all observed. As a result, high quality operation can be achieved without increasing production costs on the basis of these optimal SFE conditions. This research can contribute to the future scale-up of ginger oil production in the food, biochemistry and medical industries.

5. Acknowledgements

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