



Optimization of Supercritical Extraction of Oil from Onion by Supercritical CO₂

JIAN-ZHONG JIN* and JIAN-YING TONG

College of Biological and Environmental Engineering, Zhejiang Shuren University, Hangzhou 310015, P.R. China

*Corresponding author: Fax: +86 571 88297098; Tel: +86 571 88297171; E-mail: hzjjz@sohu.com

Received: 18 February 2013;

Accepted: 18 June 2013;

Published online: 26 December 2013;

AJC-14490

In this study, the supercritical CO₂ extracting technology was applied to extract volatile oil from freeze-dried onion powders, single factor experiment and orthogonal array design were used to study the influence of extracting pressure, extracting temperature, extracting time and flow rate of CO₂ on the extracting ratio of volatile oil. The optimum extracting parameters were established with extracting pressure of 20 MPa, extracting temperature of 35 °C, flow rate of CO₂ of 14 kg·h⁻¹ and the extracting time of 2.5 h. The optimum technology for supercritical CO₂ extraction of volatile oil from freeze-dried onion powders was very efficient and extraction yield is 0.53 %.

Keywords: Onion, Supercritical CO₂ extraction, Volatile oil.

INTRODUCTION

Nowadays, the users prefer natural products mainly in the food and cosmetic sectors. For this reason there has been an increasing interest in essential oils extracted from various plants.

Onions (*Allium cepa* L.), which are used as both a vegetable and a spice, are grown commercially in several areas of China. Onions possess strong, characteristic aromas and flavors, which have made them important ingredients of food. Onions and onion oil are important seasonings widely used in food processing¹. Many researches have demonstrated that onions and onion oil possess several biological properties, such as antimicrobial^{2,3} and antioxidant activities⁴. The medicinally most significant components of onion oil are the organosulfur-containing compounds^{5,6}. These compounds are reactive, volatile, odor producing and lachrymatory⁷.

The most widely used methods of essential oils from various plants are based on hydrodistillation and solvent extraction^{8,9}. The first is often associated with low yields and in damage to some of the more thermolabile compounds, the latter uses solvents such as hexane in most of the cases. Such solvents are dangerous to handle and are harmful to human health.

In all cases the researchers aim to shorten the extraction time, use less solvent, increase the yield and improve the quality of the extracted product. One such technology that has emerged over the last three decades as an alternative to the traditional extraction of natural products is the supercritical fluid extraction (SFE) technique¹⁰⁻¹³. Supercritical carbon dioxide (SC-CO₂) extraction offers a number of clear advantages, among which

are (a) the ease with which the solvent can be separated after the extraction, leaving virtually no trace in the processed matrix, (b) its being neither toxic nor harmful and (c) its featuring a low extraction temperature, which helps minimizing the product thermal degradation¹⁴. Besides, the energy costs associated with this novel extraction technique are lower than that for traditional solvent extraction methods. Thus, supercritical fluid extraction technology is increasingly gaining importance over the conventional techniques for extraction of natural products.

In the present work, the supercritical CO₂ extracting technology have been applied to extract volatile oil from freeze-dried onion powders, single factor experiment and orthogonal array design were used to study the influence of extracting pressure, extracting temperature, extracting time and flow rate of CO₂ on the yield of volatile oil.

EXPERIMENTAL

Freeze-dried onion powder was purchased from Alixiang Food Industry Co. Ltd. (Wuxi, China). Carbon dioxide (99 %) was supplied by Hangzhou Minxing industrial and trading Co. Ltd. (Hangzhou, China).

Supercritical-CO₂ extraction: The supercritical fluid extraction system (0.5 L sample capacity) used in this study was purchased from Hangzhou Hulia Pump Co. Ltd. (model HL-0.5/50MPaIII; Hangzhou, China). 100 g freeze-dried onion powder was placed in the extraction vessel. After an initial air purge, liquefied carbon dioxide was pumped into the extraction vessel by a high-pressure pump to a given pressure,

the temperature inside the vessel was raised to and maintained at, the desired level by a heating jacket encasing the vessel and the CO₂ flow rate was maintained at a given amount. Fractional separation was obtained setting the separator at 5 MPa and 40 °C.

After the extraction was completed, the extraction vessel was depressurized and the extract was collected. The yield is defined in Eq. (1). It was measured for each run and averaged for the three values.

$$y = \frac{\text{g of oil extracted}}{\text{g of onion power}} \quad (1)$$

RESULTS AND DISCUSSION

Influence of extracting pressure: The influence of extracting pressure on the yield of oil was given in Fig. 1 under other identical extraction conditions. The extracting pressure varied from 15 to 30 MPa while keeping the extracting temperature at 40 °C, extracting time at 2 h and flow rate of CO₂ at 10 kg·h⁻¹. The yield of oil increases with extracting pressure, reaching a maximum at 20 MPa and almost no changing thereafter.

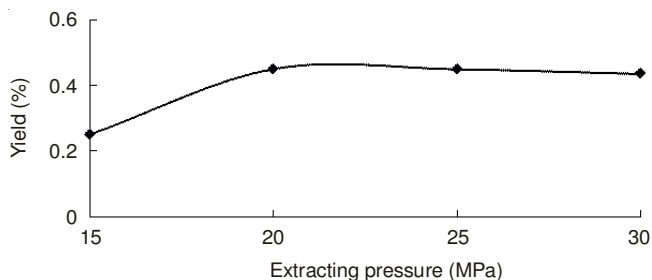


Fig. 1. Effects of extracting pressure on yield of volatile oil

Influence of extracting temperature: The extracting temperature was varied from 30 to 45 °C keeping the extracting pressure at 20 MPa, extracting time at 2 h and flow rate of CO₂ at 10 kg·h⁻¹. The results are represented in Fig. 2. With the increase in temperature from 30 to 40 °C, the yield of oil increases from 0.26 to 0.45 %. With further increase in temperature, little decreased yield was observed.

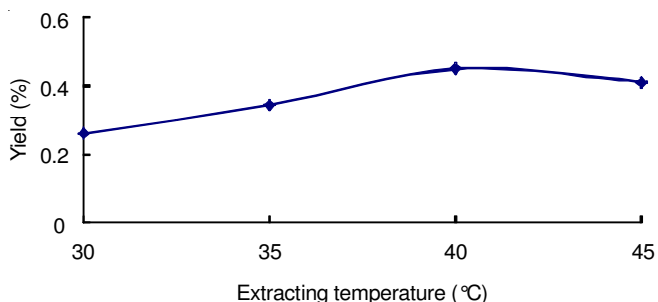


Fig. 2. Effects of extracting temperature on yield of volatile oil

Influence of flow rate of CO₂: Flow rate of CO₂ was varied from 8 to 14 kg·h⁻¹ keeping the extracting pressure at 20 MPa, extracting time at 2 h and extracting temperature at 40 °C. The result was shown in Fig. 3. The yield increased from 0.27 to 0.51 % with the change in flow rate of CO₂ from 8 to 12 kg·h⁻¹. With further increase in flow rate of CO₂, a slightly decrease in yield was observed.

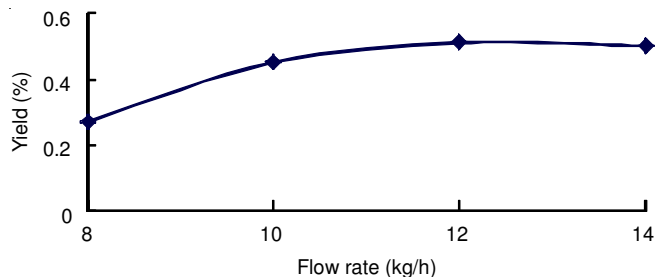


Fig. 3. Effects of flow rate on yield of volatile oil

Influence of extracting time: The influence of extracting time on yield of volatile oil was given in Fig. 4 under other identical extracting conditions. A gradual rise in yield was seen with increase in duration of extracting time. As seen from Fig. 4, in 2 h of extracting time, 0.51 % of yield is obtained, where as at the end of 2.5 h only 0.52 % of the extraction is complete. This suggests that 2 h is sufficient to optimize the extracting parameters.

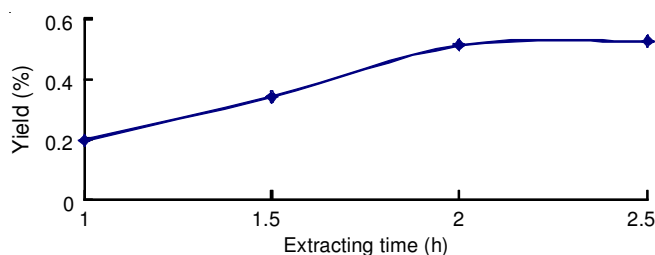


Fig. 4. Effects of extracting time on yield of volatile oil

Orthogonal array design: In order to fully examining the effect of supercritical CO₂ extraction of factors, an orthogonal array design (OAD) test, L₉(3)⁴, was employed as a chemometric method for investigating supercritical fluid extraction of oil from freeze-dried onion powder after single-factor experiments. As listed in Table-1, four processing parameters, namely, extracting pressure (P), extracting temperature (T), flow rate of CO₂ (F) and extraction time (t), were studied and optimized.

The experimental results are listed in Table-2. The extracting pressure was found to be the most important determinant of yield. The results indicated that the optimal conditions for extraction of oil from freeze-dried onion powder by supercritical fluid extraction was A₂B₁C₃D₃, namely 20 MPa of extracting pressure, 35 °C of extracting temperature, 14 kg·h⁻¹ of flow rate, and 2.5 h of extracting time. Under the optimal conditions, 100 g sample was extracted by supercritical fluid extraction for three times, the average yield of oil was 0.53 %.

Level	A P(MPa)	B T(°C)	C F(kg·h ⁻¹)	D T(h)
1	15	35	10	1.5
2	20	40	12	2
3	25	45	14	2.5

Conclusion

The optimum extracting parameters were established with extracting pressure of 20 MPa, extracting temperature of 35 °C, flow rate of CO₂ of 14 kg·h⁻¹ and extracting time of 2.5 h.

TABLE-2
RESULTS OF ORTHOGONAL TEST AND ANALYSIS

No.	A	B	C	D	Yield (%)
1	1	1	1	1	0.22
2	1	2	2	2	0.28
3	1	3	3	3	0.37
4	2	1	2	3	0.52
5	2	2	3	1	0.49
6	2	3	1	2	0.41
7	3	1	3	2	0.42
8	3	2	1	3	0.31
9	3	3	2	1	0.21
k1	0.29	0.39	0.31	0.31	
k2	0.47	0.36	0.34	0.37	
k3	0.31	0.33	0.43	0.40	
R	0.18	0.06	0.12	0.09	

The optimum technology for supercritical CO₂ extraction of volatile oil from freeze-dried onion powders was high efficient, extraction yield 0.53 %.

REFERENCES

1. C.L. Ye and Y.F. Lai, *Chem. Eng. Technol.*, **35**, 646 (2012).
2. G. Griffiths, L. Trueman, T. Crowther, B. Thomas and B. Smith, *Phytother. Res.*, **16**, 603 (2002).
3. N. Benkeblia, *LWT-Food Sci. Technol.*, **37**, 263 (2004).
4. C.L. Ye, D.H. Dai and W.L. Hu, *Food Control*, **30**, 48 (2013).
5. A. Dron, D.E. Guyeru, D.A. Gage and C.T. Lira, *J. Food Process Eng.*, **20**, 107 (1997).
6. N.K. Sinha, D.E. Guyer, D.A. Gage and C.T. Lira, *J. Agric. Food Chem.*, **40**, 842 (1992).
7. E. Block, S. Naganathan, D. Putman and S.H. Zhao, *J. Agric. Food Chem.*, **40**, 2418 (1992).
8. Q.H. Zhang, Z.P. Wang, *Food Drug*, **11**, 62 (2009).
9. Y. Wen, *China Pharmaceuticals*, **19**, 54 (2010).
10. G.M. Svetlana, D.L. Zika, P.Z. Zoran, S.V. Senka, *Hem. Ind.*, **65**, 147 (2011).
11. J.A.R. Uribe, J.I.N. Perez, H.C. Kauil, G.R. Rubio and C.G. Alcocer, *J. Supercrit. Fluids*, **56**, 174 (2011).
12. A. Piras, A. Rosa, D. Falconieri, S. Porcedda, M.A. Dessì and B. Marongiu, *Molecules*, **14**, 2573 (2009).
13. K.L. Nyam, C.P. Tan, O.M. Lai, K. Long and Y.B. Che Man, *Food Bioprocess Technol.*, **4**, 1432 (2011).
14. M. Bravi, F. Spinoglio, N. Verdone, M. Adami, A. Aliboni, A. D'Andrea, A. De Santis and D. Ferri, *J. Food Eng.*, **78**, 488 (2007).