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J.HASANOV (ASRUzIGICh), A.BERDIEV (TChTI),
ZHANG MIANSONG, ZHANG YU,
LIU CHANGHENG (Shandong Academy of Sciences Institute of Biology),

EXTRACTION OF DOG ROSE SEED WITH SUPERCRITICAL CARBON DIOXIDE

Тадқиқотнинг асосий мақсади наъматак уруғини юқори критик суюқликли экстракциялаш жараёнини ўрганиш ҳисобланади. Жараён 30 МПа, 50 ^{0}С ва оқим тезлиги 0.228 л/мин, юқори критик углерод қўшоксидини қўллаб ҳамда сепарациялаш ҳарорати 40 ^{0}С бўлган шароитларда амалга оширилган. Тажрибалар 120 минут давомида олиб борилган. Сепарациялаш жараёнидан кейин мойдаги қолдиқ миқдори хромотографик таҳлил асосида ўрганилган ва наъматак уруғидаги мойда тўйинмаган ёғ кислоталари миқдори аниқланган.

Таянч сўзлар: *юқори критик экстракция, наъматак уруғи, қолдиқ миқдори, ажратиш, тўйинмаган ёғ кислоталари, тўйинган ёғ кислоталари.*

Целью исследования является изучение процесса сверхкритической жидкостной экстракции (SFE) семени шиповника. Процесс осуществлен при 30 МПа, 50 ° С и скорости потока 0,228 л/мин с применением сверхкритического диоксида углерода и при температуре сепарации 40°С. Эксперименты проводились в течение 120 минут. Изучено количество остатков в масле после процесса сепарации на основе хроматографического анализа определено содержание ненасыщенных жирных кислот в маслосодержащем семение шиповника.

Ключевые слова: *сверхкритическая жидкостная экстракция, семена рожицы, содержание остатков, разделение, ненасыщенная жирная кислота, насыщенная жирная кислота.*

The aim of this research is to study Supercritical Fluid Extraction (SFE) process of dog rose seed. The process implemented at 30 MPa, 50 ^{0}C, and flow rate of 0.228 L/min by applying supercritical carbon dioxide and at 40 ^{0}C separation temperatures. The experiments were carried out over the period of 120 minute. Content of residue in SFE oil determined. The GC analyses revealed the oil from dog rose seeds was rich of unsaturated fatty acids.

Keywords: *Supercritical fluid extraction, dog rose seed, residue content, separation, unsaturated fatty acid, saturated fatty acid.*

1.1. Introduction

Supercritical fluid extraction has emerged as an attractive separation technique for the food and pharmaceutical industries due toa growing demand for “natural” processes that do not introduce any residual organic chemicals. Supercritical $CO_2$is by far the most commonly used supercritical fluid. The unique solvent properties of supercritical CO_2 have made it a desirable compound for separating antioxidants, pigments, flavors, fragrances, fatty acids, and essential oils from plant and animal materials. In the supercritical state, CO_2behaves as a lipophillic solvent and so, is able to extract most non polar solutes. Separation of the $CO_2$from the extract is simple and nearly instantaneous; leaving no solvent residue in the extract, as would be typical with organic solvent extraction. The replacement for the conventional method appeared was SFE, which it revealed huge amount of merits, as well as the most marketable green technology. CO_2 as a solvent above critical condition demonstrates itself as gas like and liquid like futures. As supercritical fluid CO_2 has especially properties such as odorless, environmentally safe, nonflammable, pure, none toxic recyclable and extraction process can be implemented low temperature compared to conventional [1-5]. As we know numerous impacts of the technological aspects of supercritical fluid extraction on quality and quantity properties of the oil. It can be seen from experiment indexes that more influence of pressure, temperature, fluid flow rate, particle size and filling quantity, period of the extraction on oil recovery rate and also quality as well.

Particle sizes are one of the factors, which have more effect on Supercritical fluid extraction, such as a pressure, temperature and flow rate. Theoretically, reduction of the particle size increases extraction effectiveness. This is because diminishing the size of the particles lead to reduce of the diffusion path and greater contact surface area as a result extraction process accelerates. However, the appearance of the minuscule particles can make the bed and occurs some channels affect consequently, diminish the extraction rate. Above-mentioned concepts were proved by researches, which have been done experiments on the plant of cocoa liquor, cocoa nib, canola seeds as well as the effects of particle size on the oil content studied. Results revealed that decreasing of the particle size has special impact on volume of the intended substances also, an effect of particle size on triglycerides and fatty acids composition at 35 MPa, 60 ^{0}C, the flow rate of carbon dioxide was 2 ml/min [6, 7].

2.1. Materials and methods

Prior to operation, seeds moisture content and oil content of doge rose seeds were determined. The doge rose seed oil content and moisture content were 10.3 % and 5.8 % in order. The dog rose seeds were milled as well as sieved into groups. The dog rose seed particles weighted and sieved m_1=78.2 to d_1<0.27 *mm*, m_2= 48.2 g to 0.27*mm*<d_2<0.7*mm*, m_3=68.2 g, 0.7 *mm*< d_3<1.4 *mm* of groups respectively.

2.1.2. Supercritical fluid extraction

Supercritical CO_2 extraction process was carried out above-mentioned apparatus revealed in fig.1. Carbon dioxide with density 500 kg/m^3 supplied whole the system from tube. Carbon dioxide leads to filter then cooled by conditioner until 7 ^{0}C, liquid phase delivered by pump till extraction vessel through filters and heater. Carbon dioxide preheated to the temperature above the intended critical condition. Supercritical fluid interacts with particles of the doge rose seed in the extraction vessel. Temperature and pressure fixed by controllers inside of the extractor. Local temperature of the extraction process was measured inside the vessel by thermometer with accuracy of ±0.01^{0}C. The pressure of the vessel, which was extraction process occurred, measured by manometer with accuracy ±0.01MPa. The pressure controlled by the valves of 3, 6, 8. There are two non-return valves on the line of the pipe from the CO_2 tube between extraction vessels. Then mixture of the oil and CO_2 from extraction through the heater transported to the separator with the volume of 2 L. At this point of the system, oil released and CO_2 recycled to the system. Extraction process continued 120 minutes and measuring intervals were 30 minutes.

2.2.3. Chemical analysis

The fatty acids composition was determined by applying Thermo fisher 1310 gas chromatograph (with FID detector) and chromatographic column TR-WAX (0.25 µm; 30mm* 0.32 mm).

Fatty acid methyl esters (FAMEs) were prepared by using 0.2 g samples into a 50mL round bottom flask with nitrogen protection and 6 mL methanol solution with 0.5mol/L sodium hydroxide was added. Column temperature was 100℃ for 5 min and then increased to 240℃ at the rate of 4℃/min. Detector and vaporization temperature were 250℃ and flow rate of the nitrogen was 3.5 ml/min. The injection volume of the sample was 0.5 µL into GC applying with diversion ratio 50:1.

Table 1. Presents the content of the fatty acids of the oil from doge rose seed as percentages from the total area. The oil content was rich with unsaturated fatty acids, especially linoleic (C 18:2) and linolenic (C18:3), oleic (c18:1) accounted for 53.08% and 22.68%, 15.18 % in order. Moreover the content of the SFA and UFA was 8.31%, 91.68 % respectively.

3.1. Results and discussions

Supercritical fluid extraction (SFE) of the dog rose (Rosa canina) seeds studied, so that optimal extraction time and oil content were determined. Experiments were carried out on different sieved particle sizes of the seeds (d_1<0.27mm, 0.27<d_2<0.7 mm, 0.7< d_3<1.4 *mm*) other extraction characteristics were the same remained (temperature of the extraction process 50 ^0C, separation temperature 40 ^0C, extraction vessel pressure 30 MPa, CO_2 flow rates 0.216 L/min).

Table 1.

Fatty acid content of the oil from dog rose

№	Formula	Retention time (min)	Relative peak area (%)
1	C13:0	17.188	0.03
2	C15:0	19.672	0.01
3	C16:0	22.048	4.08
4	C16:1	22.6	0.12
5	C17:0	24.313	0.06
6	C17:1	24.772	0.04
7	C18:0	26.505	2.67
8	C18:1	26.887	15.18
9	C18:2	27.898	53.08
10	C18:3	28.555	0.09
11	C18:3	29.21	22.68
12	C20:0	30.602	1.10
13	C20:1	30.942	0.30
14	C20:2	31.892	0.10
15	C21:0	32.547	0.03
16	C22:0	34.415	0.16
17	C22:1	34.75	0.07
18	C22:1	36.21	0.05
19	C24:0	37.958	0.12
20	C24:1	38.365	0.02
Total:			99.99

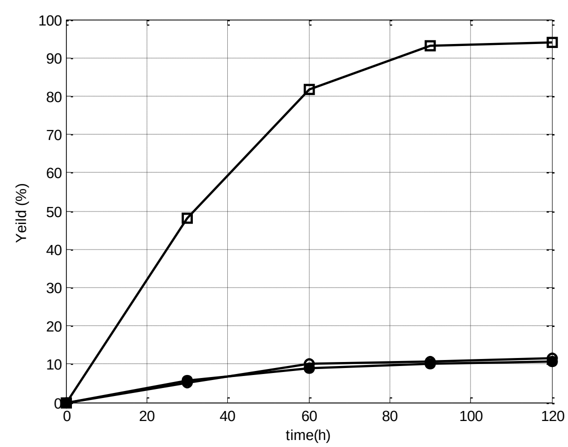

Fig.1. ***Effect of the particle sizes on the oil recovery.***
□-d_1<0.27*mm*; ○- d_2<0.7*mm*; ●- d_3<1.4 *mm*

From the overall picture of the Supercritical fluid extraction (**Fig.1.**) of the dog rose seeds oil quantity curves give information about the similarity of the process theory, whereas the quantity of the oil was different with varies of the sieve fraction. The results show that, the slop of the extraction curves sharply increased until the 90 minutes with the particle size of the seeds in d_1 compared to d_2 and d_3. This is because the main cause of the large difference of the yield of the oil between d_1 and d_2, d_3 was much more percentage of the tiny particles in d_1 than others. At the rest of the time recovering oil volume were almost remains constant among sieved particles. Theoretically proven that, reducing of the particle sizes caused enhance of surface area, diminish diffusion path as well as disruptions of the cell walls.

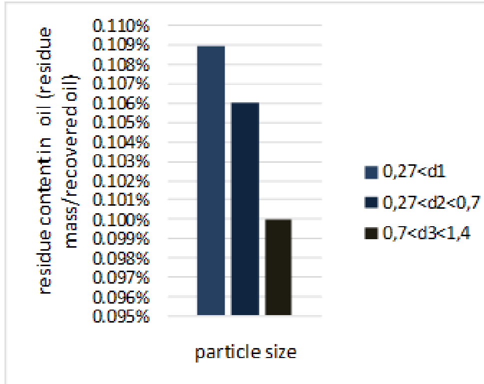

Fig.2. ***Influence of the sizes of the sieved particles on residue content in oil.***

Results fig. 2. show that reducing the size of the sieved particles brings to be more residue content in oil of dog rose seed. Therefore, the residue content in d_1,d_2, and d_3 was 0.109%, 0.106% and 0.1% in order.

4.1. Conclusion

Supercritical fluid extraction of the dog rose seeds at the condition of pressure 30 mPa, temperature 50 ^{0}C, separation temperature 40 ^{0}C and the flow rate of the CO_2 13 L/h has been studied. Dog rose seed oil was recovered 94.1%, 11.46% and 10.66 % in order. The residue content in oil demonstrated contrary wise effect. The main content of the oil from doge rose seeds were unsaturated fatty acids such as linoleic (C 18:2) and linolenic (C18:3), oleic (c18:1).

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Hasanov Jahongir Hikmatiloevich – Senior Researcher of "Processes and Apparatuses of Chemical Technology" Academy of Sciences of the Republic of Uzbekistan Institute of General and Inorganic Chemistry, mob. Tel.: 998909317253(), E-mail: jh1980@rambler.ru;
Zhang Miangsong – Student of master's degree, researcher of "Fishery Biotechnology" Academy of Sciences of Shandong Institute of Biology, home address: st. Zhongshi, Ginan, China, Index 250103;
Berdiev Amon – Senior teacher of [a]Department of food process engineering and basics of mechanics, Toshkent chemical technological institute, mob. Tel.: +998934307425 E-mail Amon84_1@mail.ru;
Zang Yu – Student of master's degree, researcher of "Fishery Biotechnology" Academy of Sciences of Shandong Institute of Biology, home address: st. Zhongshi, Ginan, China, Index 250103;
Liu Changheng – Phd, head of the Laboratory of "Biotechnology in Food" Shandong Academy of Sciences Institute of Biology, home address: st. Zhongshi, Ginan, China, Index 250103.