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OBTAINING EXTRACTS FROM PLANT RAW MATERIALS USING CARBON DIOXIDE

K. Gafurov[1], cand. of technical sciences, associate professor
B. Muhammadiev[2], cand. of biological sciences, associate professor
Sh. Mirzaeva[1], doctoral candidate,
F. Kuldosheva[1], assistant,
[1] Department of machinery and equipment for the food and chemical industries.
[2] Department of Chemistry
Bukhara Engineering and Technology Institute, Bukhara, Uzbekistan

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Correspondence:

Sh. Mirzaeva
E-mail: shohista.m@rambler.ru

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Abstract. The unique properties of supercritical carbon dioxide as a solvent are widely used for extraction. In supercritical media, the dissolution of molecules of various chemical nature is possible. The purpose of this research was to study the extraction process and obtain extracts from valuable raw materials from local plants by CO_2 extraction under subcritical and supercritical conditions. The objects of research were: ground seeds of muskmelons and pumpkins, liquorice roots, mint and mulberry leaves, and oleaster flowers. For extraction, a laboratory installation was used. It allowed extraction by means of a heat pump, with sub- and supercritical CO_2 being supplied with a high-pressure plunger pump. The pressure range was 3–15 MPa, the range of temperatures was 295–330 K, and the volumetric flow rate of supercritical CO_2 was 800–900 g. The experiments with ground seeds of muskmelon and pumpkin have shown that 4 successive one-feed extraction cycles, with supercritical CO_2 parameters (315–330 K; 3–7.5 MPa), resulted in a decrease in the mass of the muskmelon seeds by 90 g (and the pumpkin seeds lost 80 g of their total weight). During the total extraction time (2.5 hours), 20 kg of CO_2 were pumped through the reactor (25 litres at 290 K and 6.8 MPa), with the average oil content in the extract being 4 g per 1 kg of CO_2 (3.0 g per 1 litre of $sCCO_2$). In the experiments with oleaster flowers, the maximum amount of extractable solids (2% by weight of the raw material) was obtained at the temperature in the extractor 308 K and the pressure 7.5 MPa. During extraction under subcritical conditions, there was no liquid phase in collector 2, only yellow-green paste was released in it. According to the results of the experiments with mint leaves, the maximum yield of greenish liquid was observed at T=315 K and P=4 MPa, and for mulberry, it was at T=306 K and P=6.0 MPa. The results of extracting oils and obtaining extracts from ground seeds of muskmelons and pumpkins, liquorice roots, mint and mulberry leaves, and oleaster flowers confirm that the yield of the extractive substance is maximum with the supercritical CO_2 parameters in the extractor: 310 K, 7.5 MPa. When liquid CO_2 is used for extraction (300 K and 6–8 MPa), up to 2% of a yellow substance is extracted, which does not differ in appearance from supercritical extract.

Keywords: supercritical extraction, muskmelon seeds, pumpkin seeds, mint leaves, liquorice roots, mulberry leaves, rose flowers.

Introduction. Formulation of the problem

The development of energy-efficient and resource-saving technologies, which allow receiving new high-quality products in the pharmaceutical, perfumery, and food industries, is due to the acute public demand for high-quality medicines and food, as well as for environmentally friendly industries.

In recent years, studies from different countries have established that the most practical way to obtain high-quality extracts from plant materials is using liquefied and supercritical gases as solvents. One of the approaches to this problem is using liquefied and supercritical carbon dioxide as an extractant. CO_2 extraction is gaining popularity in the world. This is primarily because it is cost-effective, technologically advanced, and allows processing not only high-quality raw materials, but production wastes as well, so as to extract their main components and thus improve the quality of low-grade products. In CO_2 extraction, two different technologies are traditionally distinguished: subcritical and supercritical (SC). The differences are as follows: during subcritical CO_2 extraction, the gas extractant contacts with the raw material in the form of a liquid, and in the case of supercritical CO_2 extraction, it is in the fluid state [1-4].

The main advantages of SCF (supercritical fluids) as solvents are: fast mass transfer due to low viscosity and high diffusion coefficient; dissolving power highly sensitive to changes in pressure and temperature; very low interfacial tension which, combined with low viscosity and high diffusion coefficient, allows SCF to penetrate porous media more easily than liquids do; the simplicity of separation of SCF and substances dissolved in them when the pressure is released [1-4].

Of food solvents, liquid carbon dioxide is the most suitable for this purpose. This extractant shows absolute affinity for the most important biologically active substances, allows extracting them from raw materials at room temperatures (from 18 to 22°C), and completely disappears from the miscella (oil-solvent mixture) when the pressure in the apparatus decreases to atmospheric [1-4].

In this regard, improving the technology of obtaining CO_2 extracts from domestic (local) raw materials, for the further use of these extracts in functional products, is very important.

Analysis of recent research and publications

The advantages of CO_2 extraction, in comparison with traditional extraction methods, have been shown in a number of works [1,2]. Industrial methods and equipment to obtain CO_2 extracts from plant materials are diverse [3,4].

A significant contribution to the development of theoretical and practical foundations for the preparation and use of CO_2 extracts has been made by well-known scientists: Mark A. McHugh, Val J. Krukonis, G. Kasyanov, Larry T. Taylor, E. Kosheva, R. Blyagoz, Tony Clifford, Jerry W. King, V. Karamzin, B. Leonchik, A. Pekhov, T. Roslyakova, T. Timofeenko, R. Shazzo and others.

Abroad, certain experience has been gained in the production and use of CO_2 extracts from plant materials, but the existing technology and equipment for their production are still far from perfect. So, in particular, extracting valuable components from raw materials takes quite a long time (from 3 to 5 hours). The imperfection of the system of stripping the solvent from the evaporator and subsequent liquefaction of the vapour in the condenser often results in violation of the process parameters.

Carbon dioxide (CO_2) can be an alternative to organic solvents (acetone, petrol, alcohol, etc.) widely used in the food and pharmaceutical industries to extract biologically active substances (BAS), dietary supplements (DS), and ingredients for medicines from plant materials [4,5]. That is why the use of CO_2 in this capacity is an important and promising area of technology. The selectivity and dissolving power of supercritical (SC) CO_2 is determined by its phase state, which, in turn, depends on the parameters of extraction – temperature and pressure [6,7]

SC solvents are a new technical tool. In recent years, they have proved helpful in two modern and promising areas of technological research: obtaining nano- and microparticles as carriers of dosage forms, and creating systems for slow drug release in the body. Today, technologies have been developed to produce nano- and microforms of drugs using SCF as solvents and precipitants. Depending on the properties of pharmaceutical substances and their solubility in SCF, various technological options are possible.

The use of SC solvents in biotechnological processes has some advantages, which was shown for the biosynthesis of citric acid as an example [8]. In comparison with existing technologies, the use of SCF media allows low-temperature biosynthesis, sterilisation of biomass, and automation of basic process operations [4]. There are supercritical apparatuses that allow obtaining up to 12 different products from the same plant material. The SC technology gave birth to such concepts as CO_2 chemistry, CO_2 metallurgy, CO_2 nanotechnology, CO_2 purification, and this is a very telling fact. Indeed, not without reason has it been called in the West the true "21st century technology."

It has been proved that the addition of small quantities of surfactants (0.01–0.1%) to the extractant improves the extraction process. In most cases, either there is an increase in extracted substances (alkaloids, essential oils, etc.), or the completeness of extraction is achieved with a smaller extractant volume [8]. There are cases, though, when mechanism of action of surfactants on the rate and completeness of extraction remains unclear. Undoubtedly, surfactants lower the surface tension of the solution and improve wettability. The solubilising ability of surfactants is important, too.

Thus, the completeness and rate of extraction depend on many factors. The effect of these factors should be skilfully regulated. Various supplements that can increase the efficiency of CO_2 extraction need further study and are the object of researchers' close attention.

Today, in the world, the ways of obtaining valuable ingredients from plant materials are being actively researched, and a number of extraction technologies have been developed. Examples of these ingredients are antioxidant compounds from oregano [9], carotenoids, tocopherols, and sitosterols from waste of industrial tomato processing [10], oil from pistachio nuts [11], cumbaru oil (*Dipteryx alata Vogel*) [12], and many more. All these studies have been carried out for the researchers' local raw materials.

Because of the structural features of plant raw materials of the Bukhara region (fruit stones, grape seeds, pumpkins, muskmelons, etc), the technological extraction modes developed for them should involve using liquid and supercritical carbon dioxide, kinetics and dynamics of the extraction process, determining the effect of the extraction process on the yield and quality of the resulting product. As to the content of biologically active substances, the valuable materials are muskmelon seeds [14], pumpkins [15], leaves and fruit of the oleaster tree [16], peppermint [17], rose petals [18], mulberry [19], liquorice root [16,20]. A promising direction is the development of parameters and methods of extracting biologically active components from these objects. Currently, there is no equipment and industrial technologies for supercritical processing of raw materials in Uzbekistan. This being so, there is a need to develop CO_2 extraction equipment and technologies designed for local raw materials.

The purpose of the work is to study the extraction process and obtain extracts from valuable local plant materials by CO_2 extraction under subcritical and supercritical conditions.

Research objectives: to determine the optimal conditions of obtaining extracts from various plant materials.

Research materials and methods

Melon varieties such as Obinovvot and Buriy Qal'a, Alacha and Roxat (Rokhat), which grow in the Bukhara Region of the Republic of Uzbekistan, were selected for the experiments. The seeds of these melon varieties are characterised by a high seed coat content and high mechanical strength (with the average moisture content 9–10%). Unlike other oilseeds, they have a significant carbohydrate content: up to 8–9% of starch and up to 1.0% of monosaccharides.

For the experiments, the local pumpkin varieties Osh Qovoq, Nosqovoq, and Buxoro Kadi, which are widespread in Uzbekistan, were selected.

The mulberry variety was Uzbekistan. The oleaster cultivars were Non Jiyda and Kandak Jiyda. The liquorice variety was *Glycyrrhiza glabra* L.

For extraction, a laboratory installation was used. It allowed extraction by means of a heat pump, with sub- and supercritical CO_2 being supplied with a high-pressure plunger pump [7].

The pressure range was 3–15 MPa, the temperature 295–330 K, the volumetric flow rate of $sCCO_2$ was 800–900 g. CO_2/g/cm 10 litres of CO_2.

To determine the parameters of sub- and supercritical CO_2 extraction in the installation, the experiments were carried out with oleaster and rose flowers, mint and mulberry leaves, ground pumpkin and muskmelon seeds, and with liquorice roots.

Ground pumpkin and muskmelon seeds, weighing 1 kg, with the moisture content 9% and particle size 0.5–2 mm, were placed in a basket made of a thin-walled pipe, 25 mm in diameter and 420 mm high, which was closed with netted covers.

Before processing, the leaves of mint and mulberry had been prewilted.

Results of the research and their discussion

In accordance with the republican scientific and technical project A-9-1, a laboratory unit was created. On it, the methodology of sub- and supercritical CO_2 extraction was developed, which allowed determining the operating parameters of the technological process for various local raw materials of plant origin [13].

Tables 1 and 2 present the results of experiments with ground muskmelon seeds and ground pumpkin seeds.

When under subcritical conditions (300 K, 6.0 MPa), 4 g of a yellow-green extract was extracted in the collector, which in appearance differed from the oil extracted under supercritical conditions (Table 1).

The results of the experiments with ground pumpkin seeds are presented in Table 2. When the pressure in the extractor was 7.5 MPa, the extracted substance was only released in collector 2 and had 2 phases: liquid and solid, of the yellow-cinnamon colour.

Table 1 – Obtaining extracts from ground muskmelon seeds

Experiment	Process parameters	Extractor	Collector 1	Collector 2	Extraction time, hours	Mass of the extract (oil), g	
						Collector 1	Collector 2
1	T, K	305	308	293	0.5	4.0	4.0 (oil + wax)
	P, MPa	15	7.0–8.0	3.0–4.0			
2	T, K	320	323	293	1	12.0	20.0 (cream-coloured oil)
	P, MPa	15	7.0–8.0	3.0–4.0			
3	T, K	330	7.0–8.0	293	2	12.0	18.0 (cream-coloured oil)
	P, MPa	15	325	3.0–4.0			
4	T, K	290	292	313	2	3.0	3.0 (cream-coloured oil)

Table 2 – Obtaining extracts from ground pumpkin seeds

Experiment	Process parameters	Extractor	Collector 1	Collector 2	Extraction time, hours	Mass of the extract (oil), g	
						Collector 1	Collector 2
1	T, K	305	308	293	0.5	4.8	4.8 (oil + wax)
	P, MPa	15	7.0–8.0	3.0–4.0			
2	T, K	330	323	293	1	11.6	20.8 (yellow oil)
	P, MPa	15	7.0–8.0	3.0–4.0			
3	T, K	330	7.0–8.0	293	2	11.4	18.2 (yellow-green oil)
	P, MPa	15	325	3.0–4.0			
4	T, K	290	292	313	2	3.2	3.0 (green oil)
	P, MPa	4.0	4.0	3.0			

The extracts differ in their fatty acid composition, which manifests itself in their colour: each of the components (fatty acids) has a specific colour, and this ultimately determines the overall colour [2]. At low pressures and low temperatures, the first molecules extracted are those with a relatively low weight and containing more double bonds (omega2 ÷ 4). As the temperature and pressure increase, fatty acids and some fat-soluble vitamins start being extracted. This will effect on the final colouring of the extractant [2].

Four consecutive one-feed extraction cycles, with supercritical CO_2 parameters (315–330 K; 3–7.5 MPa), resulted in a decrease in the mass of the muskmelon seeds by 90 g (the pumpkin seeds lost 80 g of their total weight). This coincides with the total content of extracted substances [9]. During the total extraction time (2.5 hours), 20 kg of CO_2 were pumped through the reactor (25 litres at 290 K and 6.8 MPa), with the average oil content in the extract being 4 g per 1 kg of CO_2 (3.0 g per 1 litre of $sCCO_2$).

The experiments with ground pumpkin seeds have shown that the moisture content of the raw materials decreased by 2%, i. e. about 20 g of water was extracted from the raw materials, but in collector 2, only 12 g of liquid was collected. The moisture content of the raw material after extraction also decreased by almost 2%, but there was little liquid in the collector. Obviously, water vapour had escaped into the condenser.

Table 3 shows the results of the experiments with oleaster flowers. As seen from Table 3, different extraction modes resulted in accumulation of green paste in collector 2, although the extraction time differed.

The pressure decrease to 10 MPa in the reactor and to 6 MPa in collector 1 resulted in the release of the extracted substance in collector 1 in the form of a yellow liquid phase (2 g) and a solid phase (3 g) consisting of approximately equal amounts of two components coloured white and green. In general, the maximum of the extracted solid substance (2% by weight of the raw material) was obtained at the temperature in the extractor 308 K and the pressure 7.5 MPa. Upon subcritical extraction, there was no liquid phase in collector 2: only yellow-green paste was released in it.

Let us turn now to the results of experiments with rose flowers (Table 4). Here, accumulation of green liquid was observed under all extraction conditions, but the weights of the liquid were different. The highest yield of yellow liquid was obtained in variants 1 and 4 of the experiments.

The results of the experiments with mint leaves are presented in Table 5. In this case, the maximum yield of greenish liquid was observed at T=315 K and P=4 MPa (variant 1).

The extraction of mulberry leaves (Table 6) was also carried out in 4 modes. The maximum yield of light yellow liquid was observed in collector 2 at T=306 K and P=6.0 MPa.

Table 7 summarises the results of the experiments with liquorice roots. Here, in different experimental variants, the colour of liquids ranged from yellow-green to yellow-brown, and the maximum yield was in 2 extraction modes (variants 1 and 2).

Table 3 – Obtaining extracts from flowers of oleaster

Experiment	Process parameters	Extractor	Collector 1	Collector 2	Extraction time, hours	Mass of the raw materials, g		Extracted substance, g, collector 2
						before	after	
1	T, K	330	320	315	3	450	438	10 (green paste)
	P, MPa	15.0	10.0	4.0				
2	T, K	330	320	315	2.5	448	434	5 (green paste)
	P, MPa	10.0	8.0	6.0				
3	T, K	293	298	308	2.5	476	464	2,5 (green paste)
	P, MPa	6.5	6.5	6.5				
4	T, K	330	320	310	3.5	474	446	10 (green paste)
	P, MPa	10.0	8.0	6.0				

Table 4 – Obtaining extracts from rose flowers

Experiment	Process parameters	Extractor	Collector 1	Collector 2	Extraction time, hours	Mass of the raw materials, g		Extracted substance, g, collector 2
						before	after	
1	T, K	330	318	312	3.0	448	430	8 (yellow liquid)
	P, MPa	15	9.0	4.4				
2	T, K	330	318	312	1.5	441	432	4 (yellow liquid)
	P, MPa	10	8.0	5.0				
3	T, K	293	296.2	306.0	1.5	480	466	2.5 (yellow liquid)
	P, MPa	6.0	6.2	6.0				
4	T, K	330	318	300	3.5	476	442	8 (yellow liquid)
	P, MPa	9.0	7.0	5.0				

Table 5 – Obtaining extracts from mint leaves

Experiment	Process parameters	Extractor	Collector 1	Collector 2	Extraction time, hours	Mass of the raw materials, g		Extracted substance, g, collector 2
						before	after	
1	T, K	330	320	315	180	440	436	8 (greenish liquid)
	P, MPa	15.0	10.0	4.0				
2	T, K	330	320	315	160	428	424	4 (green liquid)
	P, MPa	10.0	8.0	6.0				
3	T, K	293	298	308	120	496	490	2 (green liquid)
	P, MPa	6.5	6.5	6.5				
4	T, K	330	320	310	200	490	470	2 (green liquid)
	P, MPa	10.0	8.0	6.0				

Table 6 – Obtaining extracts from mulberry leaves

Experiment	Process parameters	Extractor	Collector 1	Collector 2	Extraction time, hours	Mass of the raw materials, g		Extracted substance, g, collector 2
						before	after	
1	T, K	330	318	312	140	702	758	5 (yellow liquid)
	P, MPa	15	9.0	4.4				
2	T, K	330	318	312	120	690	682	5 (light yellow liquid)
	P, MPa	10	8.0	5.0				
3	T, K	293	296	306	180	678	666	8 (light yellow liquid)
	P, MPa	6.0	6.2	6.0				
4	T, K	330	318	300	160	650	660	2 (yellowish liquid)
	P, MPa	9.0	7.0	5.0				

Table 7 – Obtaining extracts from liquorice roots

Experiment	Process parameters	Extractor	Collector 1	Collector 2	Extraction time, hours	Mass of the raw materials, g		Extracted substance, g, collector 2
						before	after	
1	T, K	306	293	300	150	950	926	6 (yellow-brown liquid)
	P, MPa	16.0	9.6	5.5				
2	T, K	300	294	300	180	926	908	6 (yellow-brown liquid)
	P, MPa	19	10.8	5.5				
3	T, K	310	298	300	120	998	980	3 (yellow-green liquid)
	P, MPa	21	12.8	5.8				
4	T, K	303	295	302	90	998	990	2 (yellow-green liquid)
	P, MPa	21	11.8	5.8				
5	T, K	303	290	300	90	926	912	2 (yellow-green liquid)
	P, MPa	19	10.6	5.6				
6	T, K	305	295	304	90	890	880	1.5 (yellow-green liquid)
	P, MPa	21	10.2	5.6				

At the subcritical CO_2 parameters, the amount of substance extracted from the liquorice root decreased by about 2 times. However, the moisture content of the raw material did not change, which indicates that water does not dissolve in liquid CO_2.

The results of the extraction processes with muskmelon and pumpkin ground seeds, liquorice roots, mint and mulberry leaves, and oleaster flowers confirm that the maximum yield of the extracted substance is achieved with supercritical CO_2 parameters in the extractor (310 K, 7.5 MPa). During liquid CO_2 extraction (300 K and 6–8 MPa), up to 2% of a yellow substance is extracted, and it looks the same as SC extract.

For the studied types of plant materials (muskmelon and pumpkin seeds, oleaster flowers, mint and mulberry leaves, liquorice roots), experimental data were obtained on CO_2 extraction under sub- and supercritical conditions in the extractor (300–310 K, 6–7.5 MPa) and with the parameters in the collector 298–303 K, 5–6 MPa.

Conclusion

We have studied carbon dioxide extraction of biologically active substances from raw materials of plant origin: muskmelon seeds, mint leaves, liquorice roots, mulberry leaves, etc.

It has been shown that the maximum yield of extracted substance for these plant raw materials is observed at pressures and temperatures that exceed critical values for CO_2 (35–45°C, 3–7 MPa).

The extraction time has a significant effect, which is important when developing technological conditions of obtaining valuable components from plant materials. Reducing the extraction time saves energy and other expenses significantly.

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ОТРИМАННЯ ЕКСТРАКТІВ ІЗ РОСЛИННОЇ СИРОВИНИ ДВООКИСОМ ВУГЛЕЦЮ

К.Х. Гафуров [1], к.т.н., доцент, *E-mail*: kgafurov@yahoo.com
Б.Т. Мухаммадієв [2], к.б.н., доцент, *E-mail*: shohistamirzayeva79@mail.ru
Ш.У. Мірзаєва [1], докторант, *E-mail*: shohista.m@rambler.ru
Ф.С. Кулдошева [1],асистент, *E-mail*: tjbakt@mail.ru
[1] Кафедра машин та обладнання харчової та хімічної промисловості
[2] Кафедра хімії
Бухарський інженерно-технологічний інститут, м. Бухара, Узбекистан

Анотація. Унікальні властивості надкритичного двоокису вуглецю як розчинника, знаходять широке застосування для екстрагування. У надкритичних середовищах можливе розчинення молекул різної хімічної природи. Метою цього дослідження було вивчення процесу екстрагування та отримання екстрактів з цінної регіональної рослинної сировини шляхом застосування CO_2-екстракції в до- і надкритичних умовах. Об'єктами досліджень були: мелені насіння дині, гарбуза і коренів солодки, а також листя м'яти, шовковиці і квітки джиди. Для екстракції використовували лабораторну установку, яка дозволяє проводити екстракцію при подачі CO_2 плунжерним насосом високого тиску в до- і надкритичному стані з використанням теплового насосу. Діапазон тиску 3–15 МПа, температури 295–330 К, об'ємні витрати надкритичного CO_2 800–900 г. Досліди з меленими насінням дині та гарбуза показали, що в результаті 4-х послідовно проведених циклів екстракції на одному завантаженні при надкритичних параметрах CO_2 (315–330 К; 3–7,5 МПа) зменшення маси насіння дині склало 90 г, гарбуза – 80 г. За загальний час екстракції (2,5 години) через реактор прокачано 20 кг CO_2 (25дм^3 при 290 К і 6,8 МПа), при цьому середній вміст олії в екстракті становить 4 г на 1 кг CO_2 (3.0 г на 1 дм^3 НК-CO_2). У дослідах з квітками джиди, максимальна кількість твердого матеріалу, що екстрагується, (2% від маси сировини) отримано при температурі в екстракторі 308 К і тиску 7,5 МПа. При екстракції в докритичних умовах в збірнику 2, рідка фаза була відсутня, в ньому виділялася тільки паста жовто-зеленого кольору. За результатами дослідів з листям м'яти, максимальний вихід рідини зеленуватого кольору спостерігався при Т=315 К і Р=4 МПа, шовковиці – при Т=306 К і Р=6,0 МПа. Результати процесів вилучення масел і екстрактів з мелених насіння дині, гарбуза і коренів солодки, а також листя м'яти, шовковиці і квіток джиди підтверджують, що максимальний екстракція речовини досягається при надкритичних параметрах CO_2 в екстракторі (310 К, 7,5 МПа). Під час вилучення рідким CO_2 (300 К і 6–8 МПа) екстрагується до 2% речовини жовтого кольору.

Ключові слова: надкритична екстракція, насіння дині, насіння гарбуза, листя м'яти, коріння солодки, листя шовковиці, квітки троянди.

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