

INVESTIGATION OF OIL EXTRACTION FROM THE CANOLA AND SOYBEAN SEEDS, USING A MICROWAVE INTENSIFIER

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ДОСЛІДЖЕННЯ ЕКСТРАКЦІЇ ОЛІЇ З НАСІННЯ РІПАКУ ТА СОЇ ПРИ ВИКОРИСТАННІ МІКРОХВИЛЬОВОГО ІНТЕНСИФІКАТОРА

Prof. PhD. Bandura V.¹⁾, Prof. Ph.D. Eng. Bulgakov V.²⁾, Prof. Ph.D. Eng. Adamchuk V.³⁾, Ph.D. Eng. Ivanovs S.⁴⁾

¹⁾ Vinnytsia National Agrarian University/ Ukraine; ²⁾ National University of Life and Environmental Sciences of Ukraine / Ukraine, ³⁾ National Scientific Centre "Institute for Agricultural Engineering and Electrification" / Ukraine;

⁴⁾ Latvia University of Agriculture / Latvia

Tel.+37129403708, E-mail: semjons@apollo.lv

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ABSTRACT

Intensification of the technological processes in the production of vegetable oils is a topical scientific and practical task. It is assumed that extracts from the plant raw materials obtained by exposure to the electromagnetic microwave field possess qualitatively new biochemical and biological properties in comparison with similar extracts obtained by means of the conventional extraction methods. The article deals with possibilities to reduce the duration of the extraction process of the soybean and canola seeds, to achieve great output of the target component, to increase the number of valuable components (tocopherols) in the finished product. It has been established that in the microwave intensifier, in contrast to the classic method of extraction, the extraction time of the soybean and canola seeds is reduced to 70%, but the output of the target component increases within the limits of 30%.

РЕЗЮМЕ

Інтенсифікація технологічних процесів при отриманні рослинних олій є актуальною науково-практичною задачею. Передбачається, що екстракти з рослинної сировини, отримані з використанням впливу мікрохвильового електромагнітного поля, набувають якісно нові біохімічні та біологічні властивості у порівнянні з аналогами, отриманими традиційними методами екстракції. У роботі досліджувалися можливості скорочення тривалості процесу екстракції насіння сої та ріпаку, одержання більшого виходу цільового компонента, збільшення кількості цінних компонентів (токоферолів) в готовому продукті. Встановлено, що в мікрохвильовому інтенсифікаторі, порівняно з класичним методом екстрагування, час проведення екстрагування насіння сої та ріпаку зменшується до 70%, а вихід цільового компонента збільшується в межах 30%.

INTRODUCTION

Vegetable oils are an important food product and a raw material for the chemical, machine-building, metallurgical industry, as well as for the production of biodiesel fuel (Thiyam-Holländer et al., 2016). Owing to the presence of fatty acids and absence of cholesterol, vegetable oils have an ability to reduce the risk of cardiovascular diseases. The properties of the oils and the ability to preserve their valuable qualities depend on many factors, including the biologically active components, particularly tocopherols, which are antioxidants preventing and protecting polyunsaturated fatty acids from oxidation (Loganes et al, 2016; Burdo, 2013).

Canola seeds contain 40-45% of oil and, in contrast to other vegetable oils, it has a series of advantages from the point of view of the physiology of human nutrition. It also contains 18-22% of crude protein and other physiologically important acids in an optimal ratio, as well as α and β tocopherols. By the volume of production, rapeseed oil became the third in the world after palm and soybean oils.

Soybean seeds are also used to produce very valuable edible oil, which belongs to the group of linoleic-oleic oils. A very important group of compounds in soybean seeds are phosphatides, as well as tocopherols and pigments. These substances play an active role in metabolic processes, serving as one of the best sources of the natural antioxidant – vitamin E. Soybean seeds contain from 14 to 25% of oil and belong to the group of low-oil crops.

When processing low-oil raw materials, direct extraction of oil is applied (without preliminary extraction of oil by pressing).

The extraction process is based on the ability of vegetable oils to dissolve in organic solvents. The extraction rate depends on the condition of the oilseed material, its temperature, the degree of crushing, etc. In the process of extracting oil from the crushed raw material, a miscela (an oil solution in a solvent) and a defatted residue (meal, solvent cake) is obtained. In order to extract oil, the solvent is subsequently evaporated from the miscela. The resulting vapours of the solvent are condensed and their recovery is conducted to convert the solvent into a liquid state.

Extraction of oil from seeds (considered in the process of these studies) is an important process which determines the degree of extraction of valuable components from the raw materials and the quality of the finished product. However, at the moment, in all industries, including the food industry, the classical extraction is a rather labour-intensive and inefficient process (*Thiyam-Holländer et al., 2016; Burdo et al., 2016*). Among the existing problems, one can note the low output of the target component, the long duration of the process, the use of high pressure, large overall dimensions and the high metal intensity of the apparatus.

The search for new technologies to improve the extraction process allowed discovering methods by which intensification of the extraction process is achieved using electro-pulse technologies characterized by high impacts, specific by their power, upon the biomass placed in the reactor. They may include the shock wave, ultrasound, the electromagnetic field, etc. Application of these impacts allows significant improvement in the efficiency of the process even at room temperatures, reduction of the mass-transfer characteristics of the equipment, dramatic decrease in the electricity consumption.

Ultrasound has a destructive, crushing effect on the plant cells. A negative aspect of this impact is also the relatively high-power consumption. Besides, it is recommended to add surfactants to the extracting agent retarding the formation of cavitation, which is not always consistent with the production technology. When extracting, it is necessary to consider an increase in the extracting agent temperature due to the absorption of the ultrasonic energy and to ensure that the temperature of the extracting agent did not exceed the allowed values (*Burdo, 2015; Orlov, 2002*).

The positive aspects of the CO₂ – extraction technology are (*Salgin et al., 2006; Eggers, 1996, Bozan and Temelli, 2003*) production of native extracts, elimination of high temperatures, raising the quality of the target products, versatility of the CO₂ solvent (not combustible, not explosive, with a low cost).

To increase the extraction rate, catalysts, surfactants, chemical modifiers are used. Despite the fact that supercritical extraction has significant advantages, its application in the food production is still insufficient. This is because the solution of each specific extraction task requires an individual approach to the optimization of a certain technological process, its productivity specifying its raw material, extracting agent and the final product. Naturally, all these tasks are solved if the necessary unification level of the equipment is ensured.

Application of microwave heating makes it possible to intensify considerably the thermal treatment process of the raw material and extraction of stable soluble substances by an aqueous medium. Positive results from the application of electromagnetic pulsed radiation were obtained: in the production of food grade dyes from beets, fruit and berry raw materials, in the scheme of accelerated ripening (aging) of cognac spirits, when extracting cedar oil from the seeds of the Siberian pine, to accelerate the extraction of fungicides from wood materials under laboratory conditions, when extracting the oil from the mint leaves, rosemary, the tea tree (manuka), sandalwood and other plants, when extracting nicotine from the tobacco raw material, when extracting coffee beans (*Rekas et al, 2017; Burdo et al., 2007*).

The aim of the work is to study possibilities of intensifying extraction of the soybean oil and rape seed oil by using microwave exposure and increasing the number of valuable components (tocopherols) in the finished product.

MATERIALS AND METHODS

The extraction process was carried out on the test bench which is described in more detail in works (*Bandura and Kaljanovska, 2011*). Extraction of oil from the miscela to determine the concentrations was carried out arbitrarily by evaporating the solvent. The contents of tocopherols and other components in the oil samples were carried out under the factory laboratory conditions in accordance with accepted standards

and compared with current standards. The methods of investigation are based on a thermophysical analysis of the structure of the material and the solvent.

In experimental studies, control instrumentation, modern methodologies and devices were used, among which there are the authors' developments. For analytical research and processing of experimental research results, a PC and the corresponding software packages MathCAD and Excel were used.

Studies on the oil extraction of the canola seeds, the "Champion" variety, and the soybean seeds, varieties "Vinnichanka", using ethyl alcohol, were carried out on elaborated experimental equipment with a microwave intensifier. In order to study the process, the solution concentration was determined depending on the extraction time, etc. For determine oil concentration in the miscela, a HP 1100 chromatograph (Agilent Technologies (USA) was used.

After a series of preliminary experiments were conducted on the extraction of the micromodel (Koljanovska and Bandura, 2012), a larger installation was created (Fig. 1).

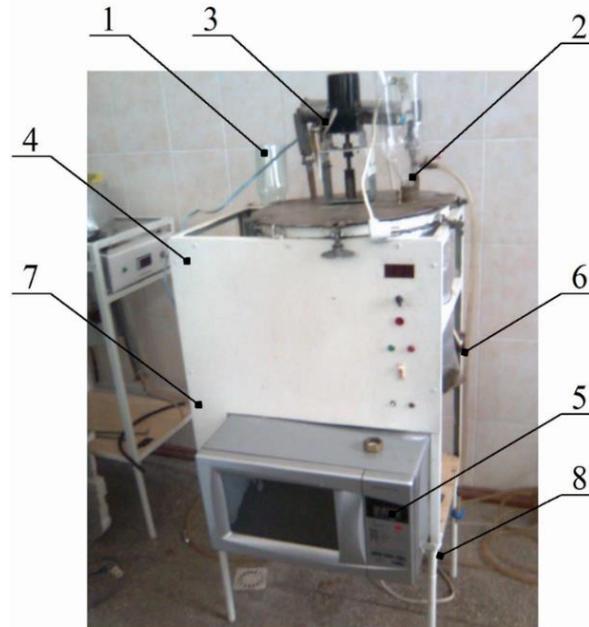


Fig. 1 - Laboratory equipment (extractor with a microwave intensifier)

1 – an orifice with a vessel for filling the extractor with the solvent; 2 – an orifice with a vessel for filling the reaction vessel with a solid phase; 3 – a reflux condenser; 4 – the extractor; 5 – the electromagnetic intensifier (MWI); 6 – a sensor for taking the temperature of the intermediate heat carrier; 7 – a sensor for taking the temperature of the product at the inlet into the microwave intensifier; 8 – a sensor for taking the temperature of the product at the outlet from the microwave intensifier

In the laboratory installation, the power of the microwave intensifier can be varied within the limits from 0.4 to 1.6 kW, the volume of the solvent is from 0.008 to 0.015 m³, the mass of the canola and soybean seeds was from 2 to 5 kg. The frequency of the microwaves was 2450 MHz.

In the preliminary investigations, we estimated the impact of the power of the microwave intensifier 0.4; 0.6; 0.8; 1.0; 1.2; 1.4; 1.6 kW upon the oil output. The results of the extraction studies with the help of a microwave intensifier at different capacities showed that the concentration of the canola and soybean oil increased in the range from 0.4 to 0.8 kW, and then, starting from 0.8 kW and higher, practically did not change. Therefore, in order to save energy, the applied capacity of the intensifier in the following experiments was 0.8 kW. In this paper, there are presented the results of research on the extraction process of the canola seeds, the "Champion" variety (the oil content – 43%) and soy bean, the "Vinnichanka" variety (the oil content - 21%), with ethyl alcohol, using the self-developed experimental equipment on the basis of a microwave intensifier (MWI). The laboratory equipment works as follows: the seeds of the investigated oil crops are fed into the extractor 4 through the orifice 2, and the solvent – through the orifice 1. The solvent is condensed in the reflux condenser 3. The extraction intensification takes place in the electromagnetic intensifier (MWI) 5.

Temperature measurements by the sensors of the intermediate heat carrier, of the miscela at the inlet into and the outlet from the microwave intensifier were carried out with a 2 minutes' interval; after every 2 minutes samples were taken to determine the concentration of the miscela. To increase the validity of the results, the experiments were conducted in 6-fold replicates. Where the results were obtained with significant

deviations (with a coefficient of variation of more than 10%), the number of measurements was increased to 12.

During the study, temperature indicators of the product were taken using of sensors at the inlet 7 and the outlet 8 from the microwave intensifier, as well as temperatures of the intermediate heat carrier 6. The characteristics of the laboratory equipment during the experiments were as follows (Table 1).

The processing of the experimental data about the change in the concentration of the solid and the liquid phases under different condition parameters in the ethyl alcohol medium was carried out using the MathCad system.

Table 1

Characteristics of the extractor with a microwave intensifier		
Characteristics of the equipment	Unit of measurement	Quantity
Volume of the solvent (V)	m ³	0.008
Mass of the canola seeds (M _{Cp})	kg	2
Mass of the soybean seeds (M _{Cc})	kg	2
Duration of extraction (t)	min	32
MWI efficiency (N)	kW	0.8

RESULTS

During the research, the temperature indicators of the miscela (the solution of oil in the solvent) at the inlet 7 and at the outlet 8 from the microwave intensifier, as well as the temperature of the intermediate heat carrier 6 were taken with the help of sensors. The graph (Fig. 2) shows experimentally obtained dependences of the temperature of the miscela on time in the process of extracting the canola seeds and soybean seeds using ethyl alcohol in the extractor with a microwave intensifier.

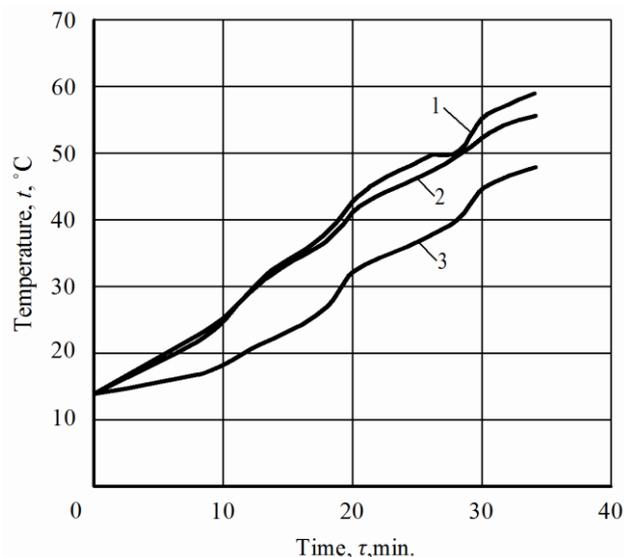


Fig. 2 - The value of the miscela temperature depending on the time of the canola and soybean seeds using ethyl alcohol, in the extractor with a microwave intensifier
 1 - temperature at the outlet; 2 - temperature at the inlet; 3 - temperature of the heat carrier

As it can be seen from Fig. 2, the temperature of the miscela at the outlet of the microwave intensifier was high, while during the investigation its maximum value was 59°C, the maximum value at the inlet was 56°C and the temperature of the intermediate heat carrier at the end of the study was not more than 48°C. The graph was created on basis experimental data (the obtained dependences do not have a strictly linear form), and in the present paper its further approximation has not been fulfilled.

Fig. 3 shows the concentration of the miscela of the soybean seeds, the "Vinnichanka" variety, and of the "Champion" canola seeds, depending on the extraction time using the microwave intensifier. In contrast with the traditional technology, the use of a microwave intensifier contributes to a significant reduction in time and an increase in the amount of the extracted oil (about 30%) (Bandura et al., 2011, Burdo et al., 2016). The output of the extracted canola seed oil with 2 kg of the loaded canola seed grain, crushed to the size of a fraction of 0.5-1 mm with an initial oil content of 43%, was 0.83 kg, i.e. 41.5% (i.e. only 1.5% of the canola oil remains in the residue). The concentration of the soybean oil at the end of the experiment, using the MWI,

was 5.0%. The output of the soybean oil from 2 kg of seeds, crushed to the fraction size of 0.5-1 mm and the initial oil content of 21%, was 0.40 kg, i.e. 20%. This means that only 1.0% of the soybean oil remains in the residue after the seed processing. Intensification of the extraction process by means of the microwave field is more efficient than the classical method of boiling. Intensification of the extraction process, using the microwave field, takes place by increasing pressure (barodiffusion) inside the capillaries of the plant raw material, followed by their destruction and the maximum arrival of the target component into the extracting agent. A barodiffusion current arises which contributes to significant reduction in the duration of the extraction process and substantial increase in the extraction of valuable components from the raw material.

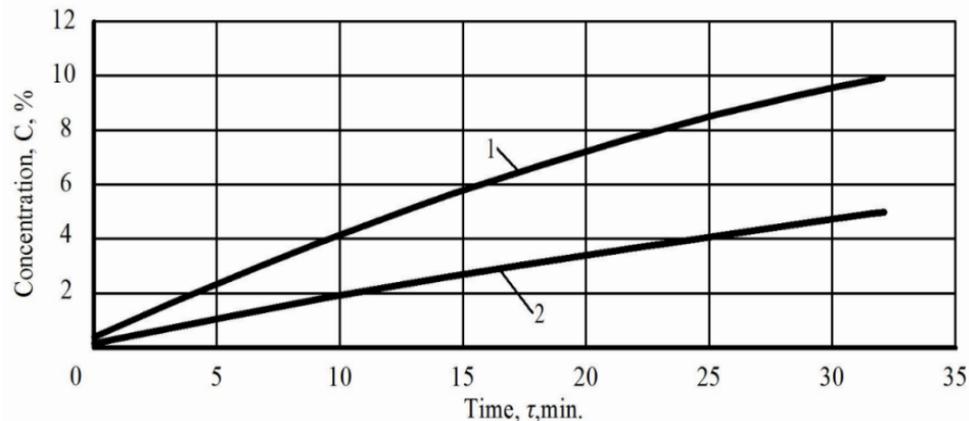


Fig. 3 - Dependence of the oil concentration in the miscela on the time in the extraction process of oil from the canola and soybean seeds using alcohol, in a microwave intensifier

- 1) - colza + alcohol, fraction of grain 0.5-1 mm, hydromodule 1: 4, output power 800 W;
2) - soybean + alcohol, fraction of grain 0.5-1 mm, hydromodule 1: 4, output power 800 W.

As it is known (Bogaert et al., 2018; Bredeson, 1983; Karaj and Müller, 2011; Ward, 1976), increasing temperature raises the intensity of the oil release, both by a mechanical method of obtaining oil from the seeds and by extraction. However, high temperature can adversely affect the quality of the product to be obtained, and therefore the maximum allowed temperature is limited by certain requirements of the standard. Particularly, for the crops under consideration (canola and soybean) the maximum allowed temperature should be not more than 60°C. In order to obtain a compatible comparison for the oil production intensification by means of a microwave field and by raising temperature, research was carried out of both crops.

The experimental research shows that, when extracting crushed canola and soybean grains by alcohol in the conventional way (in a thermostat by infusion) at different temperatures (12°C, 40°C, 50°C, 60°C), the process can also be intensified by moderate increase in temperature to 60°C.

By the way, after extracting canola for 5 hours the oil concentration in the miscel was 8.1% at a temperature of 12°C, 15.2% at 40°C, 20.9% at 50°C and 24.3% at 60°C (fig.4). The process becomes practically stable for all the investigated temperatures after 10 hours of extraction, the oil concentration at 12°C being 12.3%, at 40°C – 21.4%, at 50°C – 24.7%, and at 60°C – 28.3%.

The tendency of increasing the oil concentration, when temperature increases, was preserved for soybeans too. So, when extracting for 5 hours, the oil concentration was at a temperature of 12°C – 3.7%, at 40°C – 13.1%, at 50°C – 14.9% and at 60°C – 21.5%. At temperatures of 40-60°C extraction was carried out for 5 hours and at a temperature of 12°C – 24 hours. At a temperature of 12°C – 24 hours the concentration of the soybean oil reaching only 8.9%, i.e. it was by 6% lower than at a 5-hours' extraction at 50°C (Fig. 5).

However, when comparing the data obtained by the conventional method (extracting in a thermostat) with the data obtained using a microwave intensifier, it is evident that the proposed method allows at the same time increasing the oil concentration by about 30%.

An important parameter is also time consumed for the production of oil. The results of the research show that, in order to achieve a concentration equal to that reached by the conventional method, the time required, when using a microwave intensifier, is reduced almost twofold (1.96 times).

An important indicator of the quality of the canola and soybean oil is the amount of the acid and the peroxide value, which contain common tocopherols, etc. The obtained experimental samples fully corresponded (Table 2) to the requirements of the standards (State Standard of Ukraine No 4534: 2006; State Standard of Ukraine No 46.072: 2005).

The efficiency of the use of a non-standard for the given process polar solvent of ethyl alcohol is confirmed by the results of the gas-liquid chromatography, which show that under the influence of the

electromagnetic field, this solvent intensifies extraction from the canola and soy bean seeds not only fatty acids but also biologically active substances, in particular, tocopherol $C_{29}H_{50}O_2$.

As it is evident from the data in Table 3, in contrast with the classical method of extraction, when using a microwave intensifier, the content of tocopherols increased in the canola oil, on the average, 1.8 times, and in the soybean oil - 2.6 times. This indicates promising prospects for the application of microwave radiation to produce better quality products from the canola and soybean seeds.

Table 2

Compliance of the tested samples of oils with the requirements of state standards of Ukraine

Indicators	Canola oil (a sample)	Requirements of the standard: DSTU 46.072: 2005 "Canola oil"	Soybean oil (a sample)	Requirements of the standard: DSTU 4534: 2006 "Soybean oil"
Acidity number, mg KOH/g	3.9	not more than 6.0	4.0	not more than 6.0
Mass fraction of moisture and volatile substances, %	0.25	not more than 0.25	0.19	not more than 0.2
Peroxide number, 0.5-O mmol.kg ⁻¹	8.9	not more than 10.0	9.1	not more than 10.0
Mass fraction of phosphorus-containing substances in terms of stearo-oleo-lecithin, %	1.9	not more than 2.0	4.0	not more than 6.0
Mass fraction of erucic acid, %, to the sum of fatty acids	0.8	not more than 2.0	-	-

Table 3

Content of tocopherols in the canola and soybean oil samples

Oil	Content of common tocopherols after extraction in the MWI, %	Content of common tocopherols after classical extraction, %
Canola	92	51
Soybean	301.2	137

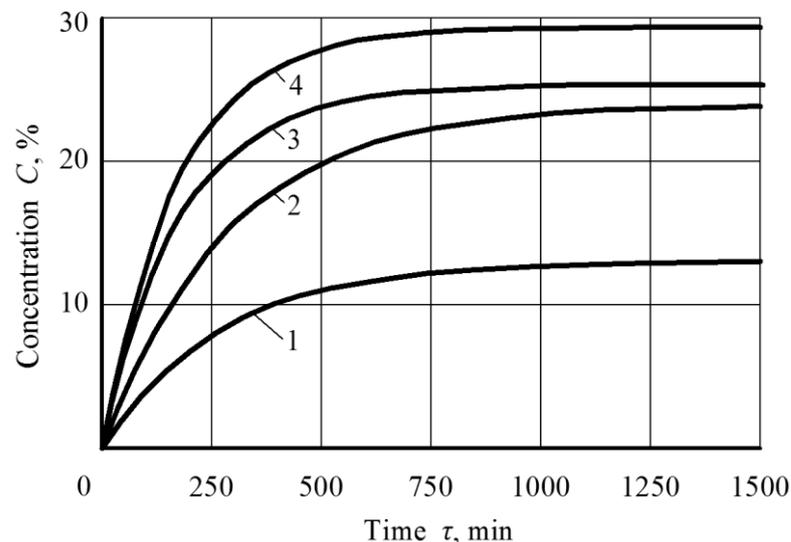


Fig.4 - Dependence of the oil concentration in the miscela on the time in the extraction process of oil from the canola using the conventional method (in a thermostat by the infusion method)

(colza + alcohol, fraction of grain 0.5-1 mm, hydromodule 1: 4)

On the basis of these studies, a technological scheme for extraction of oil from the rapeseed and soybean seeds, using a microwave intensifier was developed and recommended; functional dependences were found that allowed to determine the rational operating conditions of extractors depending on the influence of intensifying factors; the efficiency was established of the use of a more safe, non-toxic ethyl alcohol (as opposed to the toxic explosive hexane, which is used now in production). The proposed

technology will provide an opportunity to obtain a product that meets the requirements of the existing national standards.

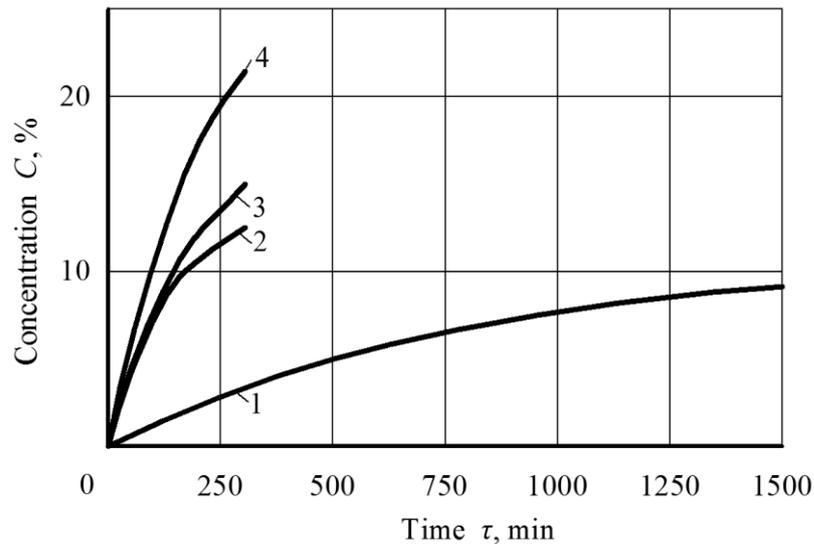


Fig. 5 - Dependence of the oil concentration in the miscela on the time in the extraction process of oil from the soya seeds using the conventional method (in a thermostat by the infusion method)
(soya + alcohol, fraction of grain 0.5-1 mm, hydromodule 1: 4)

CONCLUSIONS

1. In contrast with the traditional extraction method, the use of a microwave intensifier to extract oil from the canola and soy bean seeds makes it possible to accelerate the process and allows obtaining a better-quality product.

2. In experiments using a microwave intensifier, the output of the extracted canola oil was 41.5% and the soy bean oil 20%.

3. In contrast with the classical extraction method, when using a microwave intensifier, the content of tocopherols increased in the canola oil, on the average, 1.8 times, and in the soybean oil – 2.6 times.

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