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БАЯНДАМАЛАРЫ

ДОКЛАДЫ
НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН

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ASSESSMENT OF QUALITY AND FOOD SAFETY OF VEGETABLE OILS PRODUCED IN VARIOUS REGIONS OF KAZAKHSTAN

Abstract. Vegetable oils are a valuable multivitamin raw material for the food and pharmaceutical industry due to the content of effective biologically active organic components and mineral substances. To assess the quality and food safety of unrefined vegetable oils obtained from sunflower seeds and flax of Kazakhstan agro-formations by the method of «cold pressing,» we studied the basic indicators of their qualitative and quantitative composition. It was found that the organoleptic characteristics (transparency, color, smell and taste) of the tested vegetable oils correspond to unrefined sunflower oil of the highest grade, unrefined linseed oil of the first grade. Studies of the physicochemical parameters of sunflower and linseed oils: colored number (14,40 mg of iodine), acid number (1,4-1,5 mg KOH/g), weight fraction of phosphorus-containing substances (0,18% and 0,5%), humidity (0,13%; 0,17%), peroxide number (6,7 and 9,0 mmol of active O_2/g), iodine number (132 and 176 gJ2/100) and saponification number (188 and 187 mg/g) also correspond the requirements of regulatory documents and standards. In sunflower oil samples, the amount of oleic acid is 52,21%, linoleic acid 28,97% is determined, which are within the normal range, although they are adjacent to the levels of higher limits. Analysis of linseed oil fatty acids showed that samples contain linolenic acid 50,1%, All other fatty acids are noted within the permissible limits, and oleic acid 14,13%, linoleic acid 17,9% are close to the upper limits of permissible limits. NMR spectroscopy confirmed that the optimal ratio of ω-6 andω-3 of polyunsaturated fatty acids in the composition of the studied vegetable oils correspond to their name in the ratio of monounsaturated and polyunsaturated fatty acids.

Key words: sunflower oil, linseed oil, unrefined oils, physicochemical parameters, fatty acid composition, NMR spectroscopy, spectra, proton signals.

Introduction. Food safety for each state is ensured by providing the availability of environmentally sound and affordable products to the population. Vegetable oils take a significant place among important food products in the human diet. They are a valuable multivitamin product for the food and pharmaceutical industry due to the content of effective biologically active organic components and mineral substances [1].

The oil and fat industry of Kazakhstan is currently demonstrating intensive development processes. In the period from 2009 to 2019, the area of oilseeds increased from 1,2 million hectares to 2,9 million hectares. Kazakhstan produces various species of oilseeds (sunflower, flax, safflower, rapeseed, mustard) and their processing products. There are about 52 enterprises for the processing of oilseeds with a total capacity of 2 million tons per year [2].

Some publications provide information on the impact of natural and climatic conditions on the quality of vegetable oil [3, 4]. Due to the anthropogenic disturbance of the natural environment in the places of natural phytocenoses` growth, representatives of which are used for the production of vegetable oils, there are data on the accumulation in fruits and other organs of heavy metals and radionuclides [5, 6].

Traditionally use chemical and physical methods in analytical studies that are cost-based by time and raw materials, as they require multi-stage analyses and large volumes of samples. There is an active search among specialists for methods of analysis and identification of the fatty acid composition of vegetable oils for wide application [7]. The method of nuclear magnetic resonance spectroscopy (NMR spectroscopy) has been widely used among non-destructive methods for the study of vegetable oils. This method makes it possible to quickly determine the content of fatty acids and their ratio, including unsaturated fatty acids [7]. NMR spectroscopy based on known chemical shifts of carbon atoms of acid groups of fatty acid glycerides makes it possible to quantify the composition of unsaturated fatty acids of glycerides (oleic, linoleic and linolenic acids) and determine the type of vegetable oil without additional

sample preparation [8-12]. Given the high demand for vegetable oils among the population and industry, the growing anthropogenic pressure on various components of the natural environment, the goal of the research was to assess the quality and food safety of vegetable oils.

Research materials and methods. The material for the study was sunflower oil from seeds of highly productive hybrids of sunflower «Kazakhstan-5» breed, grown in the East Kazakhstan region and linseed oil from high-yielding breeds of flax-curly seeds «Kostanay-11» from farms of Kostanay region, selected during ripening and mass harvests by the population. Cold oil squeezing was prepared under laboratory conditions for analysis according to «SST 30418-96. Vegetable oils received unrefined sunflower and linseed oils according to standard requirements [13].

The quality characteristics of sunflower and linseed oil were determined following the requirements [14, 15]. Determination of the fatty acid composition of selected oils is carried out according to [16]. To determine the suitability of the oil for food purposes and characterized by the free fatty acid content in the oils, the acid number was studied by titration of the oil sample with an alkali solution in the presence of the phenolphthalein indicator according to [17]. The values of the peroxide number of samples according to [18] were determined by the iodometric method. To analyze the saponification number [19] was used. To evaluate the degree of unfavorability of fat, its ability to oxidize, dry, and hydrogen addition, an iodine number was determined under [20]. Determination of vegetable oils chromaticity is carried out according to [21]. Moisture and volatile substances content are studied by drying method at temperature 100-105°C to constant weight according to [22]. Qualitative and quantitative composition of polyunsaturated fatty acids of vegetable oils with different content ω-6 and ω-3 fatty acids on NMR spectrometer is studied. Ranges of nuclear magnetic resonance ^{1}H and ^{13}C were removed on a spectrometer of JNM-ECA Jeol 400 (frequency 399.78 and 100.53 MHz respectively) with $CDCl_3$ solvent use. Spectra were taken in ampoules 5 mm wide, the total volume of solution 0,6 ml, the concentration of the substance in deuterated solvent 5-10%.

Chemical shifts are measured concerning signals of residual protons or deuterated chloroform`s carbon atoms. For quantitative analysis of the samples, 0,1 ml of vegetable oil was dissolved in 0,3 ml $CDCl_3$. Chemical shifts of the compounds`proton signals were determined by the chloroform signal ($CHCl_3$, δ = 7,27 p.p.m.) that was present in the deuterated solvent. The spectral recording was performed taking into account the relaxation of protons of all compounds. When recording spectra ^{13}C, a solvent signal (δC = 77,7p.p.m.) was also used as a comparative signal. For quantitative analysis, spectra were recorded with suppression of interaction with protons and using pulse sequences that excluded the manifestation of the Overhauser effect. Chromium tris-acetylacetonate was added to the solutions to reduce the time of spin-lattice relaxation. Since the relaxant slightly changes the chemical shifts of the nuclei ^{13}C components of essential oils, spectra of available individual acids present in these oils were recorded for control: oleic, linoleic, linolenic, palmitic and stearic. The signal assignment was made using the Polarization Transfer Spectrum Recording (DEPT) technique.

All studies on the production and analysis of vegetable oils were carried out based on laboratories at S. Seifullin Kazakh Agro Technical University; NMR spectrometry analysis was carried out in the laboratory of NMR spectroscopy engineering profile at Sh.Ualikhanov Kokshetau State University. Statistical processing of the obtained results of laboratory-field experiments was carried out using Microsoft Excel. Taking into account the Fischer-Student test, recorded changes in indicators were considered reliable at $p \leq 0,05$.

Results and discussion. Realization of quality assessment of completion of production (technological) operations, as a rule, is evaluated at the production stage according to organoleptic and physicochemical indicators. The results of the analysis of unrefined sunflower and unrefined linseed oil by organoleptic and physicochemical indices are given in Table 1. As can be seen in Table 1, the organoleptic characteristics, transparency, color, smell and taste of the test vegetable oils correspond to unrefined sunflower oil of the highest grade, as well as unrefined linseed oil of the first grade. Studies of physicochemical indices of sunflower and linseed oils: color number, acid number, a mass fraction of phosphorus-containing substances, humidity, peroxide number, iodine number and saponification number also meet the requirements of regulatory documents [13-22].

Table 1 - Quality characteristics of unrefined sunflower and linseed oil

Indicator name	Sunflower oil	Linseed oil
Transparence, color	Transparent, golden	Transparent, rich golden
Smell and taste	Characteristic of sunflower oil, free of foreign smell and taste	Characteristic of linseed oil, free of foreign smell and taste
Color value, mg of iodine	14	40
Acid index, mgKOH/g	1,4	1,5

Mass fraction of phosphorus-containing substances,%; in terms of stearo-oleo-lecithin	0,18	0,5
Mass fraction of moisture and volatile substances,%, maximum	0,13	0,17
Peroxide number, active oxygen mmol/kg	6,7	9,0
Iodine value, гJ_2 /100	132	176
Iodine absorption value, mg KOH/g	188	187

The production of high-quality vegetable oils is due to the applied technology, which was based on the method of «cold» pressing. Due to the gentle regimes of processing oil-containing raw materials, this method is effective for producing oils from sunflower and flax seeds and provides the release of the highest quality oil with a minimum amount of related substances, which eliminates the need for refining.

To study the resistance of oils to oxidation, we investigated the composition of fatty acids of the oils understudy, obtained by the method of «cold» pressing, which is considered perspective from preserving oils` native properties. The main initial criterion for the quality of food fats is fatty acids. Fatty acids of natural oils and fats differ significantly in the length of the carbon chain, the number and position of double bonds in it, and spatial configuration. This causes physical, chemical and biological properties. The results of the fatty acid composition of vegetable oils are summarized in Table 2.

From the data given in Table 2, it can be seen that the sunflower oil samples contain an increased amount of oleic acid 52,21%, linoleic acid 28,97%. However, it should be noted that these types of acids are within normal limits, although they are adjacent to levels of higher limits. Analysis of linseed oil fatty acids showed that samples contain an increased content of linolenic acid 50,1%, All other fatty acids are noted within the permissible limits, and indexes of oleic acid 14,13%, linoleic acid 17,9% are close to the upper limits of permissible limits.

Table 2–Indexes of the compound of fatty acids in vegetable oils

Acids` name	Norms, %	Sunflower oil, %	Norms, %	Linseed oil, %
	-	9,01	-	7,01
C 4:0 Oleic	до 0,1	0,05	-	0,06
C 14:0 Myristic	4,0-5,5	5,25	3,2-7,2	6,0
C 10:0 Palmitic	до 0,1	0,03	до 0,2	0,07
C16:1 Palmito oleic	до 0,1	0,07	-	-
C 17:0 Heptade coic	2,1-5,0	4,04	2,5-5,5	3,63
C 18:0 Stearic	43,1-71,8	52,21	11,3-24,0	14,13
C 18:1 Olein	18,7-45,3	28,97	10,4-18,7	17,9
C 18:2 Linoleic	-	0,1	до 1,5	50,1
C 18:3 Linolenic	0,2-0,4	0,09	до 0,3	0,28
C 20:0 Arachic	0,6 – 1,1	0,1	до 0,2	0,64
C 22:0 Behenic	до 0,4	0,08	-	0,18
C24:0 Lignoceric				

By the NMR spectroscopy method, we supplemented the characteristics of the qualitative and quantitative composition of polyunsaturated fatty acids of vegetable oils with different contents of ω-6 andω-3 fatty acids. Carbons at double bonds had the most characteristic signals, which made it possible to easily identify chemical mixtures. The chemical shifts of the region of double bonds acyl chains of the combination of the fatty acid of the vegetable oils on the proton spectra are shown in (Figures 1-2).

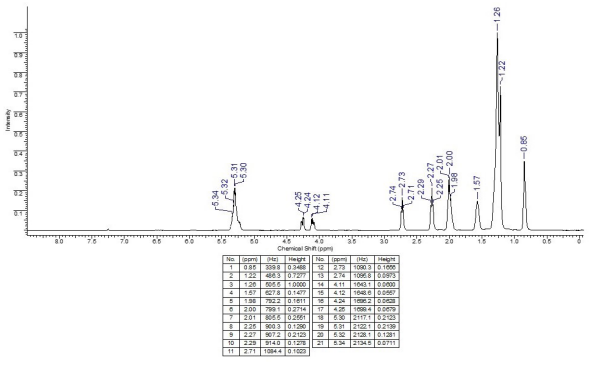
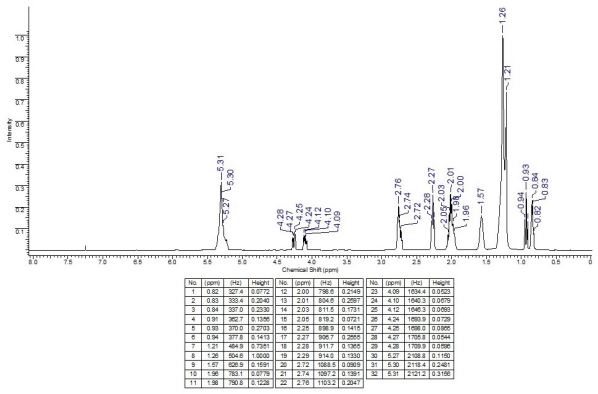


Figure 1 –^{1}HNMR-spectrum of sunflower oil

The range of nuclear magnetic resonance ^{1}H consisted of several multiplets.



No.	(ppm)	(Hz)	Height	No.	(ppm)	(Hz)	Height	No.	(ppm)	(Hz)	Height
1	0.82	327.4	0.0772	12	2.00	798.6	0.2149	23	4.09	1634.4	0.0523
2	0.83	333.4	0.2240	13	2.01	804.6	0.2597	24	4.10	1640.3	0.0679
3	0.84	337.0	0.2330	14	2.03	811.5	0.1731	25	4.12	1646.3	0.0563
4	0.91	362.7	0.1396	15	2.05	819.2	0.0721	26	4.24	1693.9	0.0729
5	0.93	370.0	0.2703	16	2.25	898.9	0.1415	27	4.25	1698.0	0.0995
6	0.94	377.5	0.1413	17	2.27	906.7	0.2855	28	4.27	1705.8	0.0544
7	1.21	484.9	0.7391	18	2.28	911.7	0.1365	29	4.29	1709.9	0.0595
8	1.26	504.6	1.0000	19	2.29	914.0	0.1330	30	5.27	2108.8	0.1150
9	1.57	626.9	0.1591	20	2.72	1088.8	0.0909	31	5.30	2118.4	0.2340
10	1.96	783.1	0.0716	21	2.74	1097.2	0.1391	32	5.31	2121.2	0.3158
11	1.98	790.8	0.1238	22	2.76	1103.2	0.2047				

Figure 2 –^{1}H NMR -spectrum of linseed oil

In the area 5.2-5.4 p.p.m. signals of olefin protons and methine proton of glycerol residue were observed; 4,1-4,3 p.p.m. - a range of chemical shifts of methylene protons of glycerin; 2,6-2,8 p.p.m. - absorbed methylene protons CH=CHCH₂CH=CH residues of linoleic and linolenic acids; about 2,3 p.p.m. - all methylene protons located next to the carboxyl group; approximately 2,0 p.p.m. - all methylene protons near double bonds; about 1,6 p.p.m. - the following methylene protons; 1,2-1,4 p.p.m. - a range of chemical shifts of all remaining methylene protons; about 0,94 p.p.m. - methyl protons of linolenic acid, located next to a double bond; 0,8-0,9 m. - range of chemical shifts of all methyl protons, except linolenic. The NMR ^{13}Cspectrum is much more informative.

As can be seen from sunflower and linseed oil spectrum fragments, the most intense are linolenic acid signals (127,7; 128,3; 128,8; 130,7; 132.4 p.p.m.) - the main ones in this form of oil Linoleic signals (128,5; 130,6; 130,8 p.p.m.) and oleic (130,2; 130,6 m.) of acids are less intense.

The full range of nuclear magnetic resonance ^{13}C of linseed oil included the following groups of signals: in the field of about 173p.p.m of absorbed atoms of carbon of carboxyl groups; the area of absorption of double bonds was in the range of 127-132 p.p.m., and in the area, about 130,4-130,5 p.p.m., C^{-13} and C^{-14} carbons of erucic acid were absorbed. The glycerol residue carbons were absorbed in about 69 p.p.m (CH) and about 62 p.p.m (CH_2). Methylene C-atoms of the molecular chain appeared in the region of 21-34 p.p.m., Methyl carbons - about 14 p.p.m. Due to the similarity of the chemical structure of fatty acids of oils the NMR signals of individual acids were observed close to each other and formed clusters.

Conclusion. Sunflower and linseed oils obtained from seeds by the «cold pressing» method were studied to assess quality and food safety among vegetable oils. Organoleptic characteristics, transparency, color, smell and taste of the tested vegetable oils correspond to unrefined sunflower oil - of the highest grade, unrefined linseed oil - of the first grade. Studies of the physicochemical parameters of sunflower and linseed oils: color number, acid number, a mass fraction of phosphorus-containing substances, humidity, peroxide number, iodine number and saponification number also meet the requirements of regulatory documents and characterize sunflower and linseed oil as valuable food products. The biological value of vegetable oils is due to the content of polyunsaturated fatty acids in them. NMR spectroscopy method confirmed that the optimal ratio ω-6 andω-3 of polyunsaturated fatty acids in the composition of the studied vegetable oils corresponds to their name in the ratio of monounsaturated and polyunsaturated fatty acids.

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ҚАЗАҚСТАННЫҢ ӘР ТҮРЛІ ӨҢІРЛЕРІНДЕ ӨНДІРІЛЕТІН ӨСІМДІК МАЙЛАРЫНЫҢ САПАСЫ МЕН ТАҒАМДЫҚ ҚАУІПСІЗДІГІН БАҒАЛАУ

Аннотация. Мақала авторлар өсімдік майлары туралы биологиялық белсенді органикалық компоненттер мен минералдардың құрамына байланысты тамақ және фармацевтика салалары үшін құнды мультивитаминдік өнімдер ретінде өсімдік майлары туралы ақпаратқа қысқаша талдау жасады. Тұқымдардан «престеу» әдісімен алынған тазартылмаған өсімдік майларының сапасы мен тағамдық қауіпсіздігін бағалау үшін олардың сапалық және сандық құрамының негізгі көрсеткіштері зерттелді.

Зерттелген өсімдік майларының органолептикалық сипаттамалары бойынша (мөлдірлігі, түсі, иісі мен дәмі) өсімдік майларына сәйкес мөлдірлігі мен түсімен жағымды иіс пен дәмге ие екендігі анықталды. Күнбағыс пен зығыр майларының физико-химиялық көрсеткіштерін зерттеу ҚР Мемлекеттік стандарттарының индикаторлары бойынша жүргізілді. Түс саны 14, 40, мг йод, қышқыл саны 1,4-1,5 мг КОН / г, құрамында фосфор бар заттардың массалық үлесі 0,18% және 0,5%, ылғалдылығы 0,13%; 0,17%, пероксид саны 6,7 және 9,0 ммоль белсенді O_2/г, йод саны 132 және

176, gJ_2/100 және сабындалу саны 188 және 187 мг/г зерттелетін үлгілерді жеткілікті сапалы өсімдік майлары ретінде сипаттайды. Күнбағыс майының үлгілерінде олеин қышқылының мөлшері 52,21%, линол қышқылының 28,97% анықталды, олар жоғары шектер деңгейіне жақын болғанымен, қалыпты шектерде. Зығыр майы май қышқылдарының талдауы көрсеткендей, сынамаларда 50,1% линолен қышқылы бар, қалған барлық май қышқылдары рұқсат етілген шектерде белгіленеді, ал олеин 14,13%, линол 17,9% қышқылдардың индикаторлары рұқсат етілген стандарттардың жоғарғы шегіне жақын. Зерттелген өсімдік майларының құрамындағы ω-6 және ω-3 полиқанықпаған май қышқылдарының оңтайлы арақатынасы моноқанықпаған және полиқанықпаған май қышқылдарының қатынасы тұрғысынан олардың атына сәйкес келетіні ЯМР спектроскопиямен расталды.

Түйін сөздер: күнбағыс майы, зығыр майы, тазартылмаған майлар, физикалық-химиялық параметрлері, май қышқылының құрамы, ЯМР спектроскопиясы, спектрлер, протондық сигналдар.

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ОЦЕНКА КАЧЕСТВА И ПИЩЕВОЙ БЕЗОПАСНОСТИ РАСТИТЕЛЬНЫХ МАСЕЛ, ПРОИЗВОДИМЫХ В РАЗЛИЧНЫХ РЕГИОНАХ КАЗАХСТАНА

Аннотация. В статье авторы представили результаты научных исследований по оценке качества и пищевой безопасности растительных масел, производимых из семян подсолнечника сорта «Казахстанский-5» и высокоурожайных сортов льна-кудряша «Костанайский-11» казахстанских агроформирований. Авторы статьи привели краткий анализ имеющейся в литературе информации о растительных маслах как о ценных поливитаминных продуктах для пищевой, фармацевтической промышленности ввиду содержания эффективных, биологически активных органических компонентов и минеральных веществ. Для оценки качества и пищевой безопасности нерафинированных растительных масел, полученных методом «холодного отжима» из семян, были изучены базовые показатели их качественно-количественного состава. Благодаря щадящим режимам переработки маслосодержащего сырья, холодное прессование эффективно для получения масел из семян подсолнечника и льна и обеспечивает выделение самого высококачественного масла с минимальным количеством сопутствующих веществ, что исключает необходимость в рафинации.

Установлено, что по своим органолептическим характеристикам (прозрачность, цвет, запах и вкус) исследуемые растительные масла обладают приятным запахом и вкусом, свойственным растительным маслам, с соответствующей прозрачностью и цветом. Исследования физико-химических показателей подсолнечного и льняного масел проведены по показателям ГОСТ РК. Показано, что цветное число 14,40 мг йода, кислотное число 1,4-1,5 мг КОН/г, массовая доля фосфорсодержащих веществ 0,18% и 0,5%, влажность 0,13%; 0,17%, перекисное число 6,7 и 9,0, ммоль активного O_2/г, йодное число 132 и 176, , гJ2 /100 и число омыления 188 и 187, мг/г характеризуют исследуемые образцы как растительные масла достаточно хорошего качества. Известно, что основным исходным критерием качества пищевых жиров являются жирные кислоты. Жирные кислоты природных масел и жиров значительно различаются между собой по длине углеродной цепи, числу и положению в ней двойных связей, пространственной конфигурацией. Биологическая ценность растительных масел обусловлена содержанием в них полиненасыщенных жирных кислот. В пробах растительных масел изучен качественный и количественный состав полиненасыщенных жирных кислот с различным содержанием ω-6 и ω-3 жирных кислот на ЯМР-спектрометре. ЯМР-спектроскопия на основе известных химических сдвигов атомов углерода кислотных групп глицеридов жирных кислот дает возможность количественно установить состав ненасыщенных жирных кислот глицеридов (олеиновая, линолевая и линоленовая кислоты) и определить вид растительного масла без дополнительной пробоподготовки. В пробах подсолнечного масла определено количество олеиновой кислоты 52,21%, линолевой кислоты 28,97%, которые находятся в пределах нормы, хотя прилегают к уровням высших пределов. Анализ жирных кислот льняного масла показал, что образцы содержат линоленовой кислоты 50,1%, Все остальные жирные кислоты отмечены в пределах допустимых норм, а показатели олеиновой 14,13%, линолевой 17,9% кислот близки к верхним пределам допустимых норм. Методом ЯМР-спектроскопии подтверждено, что оптимальное соотношение ω-6 и ω-3 полиненасыщенных жирных кислот в составе изученных растительных масел соответствуют своему наименованию по соотношению мононенасыщенных и полиненасыщенных жирных кислот. Полученные результаты лабораторных анализов свидетельствуют о том, что образцы подсолнечного и льняного масел также соответствует требованиям нормативных документов и стандартов.

Проведенные комплексные исследования растительных масел позволили заключить, что они соответствуют нерафинированному подсолнечному маслу высшего сорта, нерафинированному льняному маслу первого сорта с удовлетворительными физико-химическими и биохимическими параметрами.

Ключевые слова: подсолнечное масло, льняное масло, нерафинированные масла, физико-химические показатели, жирнокислотный состав, ЯМР-спектроскопия, спектры, сигналы протонов.

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