



Food Science and Applied Biotechnology

e-ISSN: 2603-3380

Journal home page: www.ijfsab.com
<https://doi.org/10.30721/fsab2025.v8.i2>



Research Article

New technology for low-temperature deodorization of sunflower oil

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Abstract

Using sunflower oil as an example, the possibility of deodorizing oils using a new method has been proven - by chemically transforming aldehydes and ketones that are part of their composition. As a result, the taste and smell of oil is depersonalized. Rational conditions of low-temperature deodorization were established: temperature - 90°C, duration - 16 minutes, amount of ethyl alcohol - 2%, amount of catalyst (concentrated sulfuric acid) - 0.05%. The impact of low-temperature deodorization on the levels of oxidation products in sunflower oil has been investigated. A significant decrease in the content of peroxides and aldehydes, as well as the disappearance of rancidity, has been proven. It has been proven that esters of 3-monochloropropane-1,2-diol and esters of glycidyl are not formed during low-temperature deodorization. Also, in the proposed method there is no need to heat fats to $\geq 200^{\circ}\text{C}$, no hot steam is used.

Keywords

Deodorization of oil, aldehydes, peroxides

Abbreviations

3-MCPD-E – esters of 3-monochloropropane-1,2-diol; TFA – trans fatty acids; GE – esters of glycidyl; VOCs – volatile organic compounds

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Article history:

Received 12 December 2024

Reviewed 14 April 2025

Accepted 15 August 2025

Available on-line 16 September 2025

<https://doi.org/10.30721/fsab2025.v8.i2.493>
2025, UFT Academic publishing house, Plovdiv

Introduction

At the beginning of the development of the fat-and-oil industry, oils and fats were used in unrefined form, their specific taste and smell were an attractive characteristic (Lee et al. 1937). However, the popularity of margarines in the late nineteenth century led to the development of the process of depersonalization of fats - their deodorization. And today, reducing the content of odorous components of fats – one of the tasks of production that will be in demand. Deodorized vegetable oils are used in cooking, as frying fats, in the production of cooking fats, sauces and the like. The only common approach is the use of deodorization or physical refining, which removes odorous as more volatile components of fats compared to triacylglycerols.

Deodorization is a stripping process in which a given amount of a stripping agent (usually steam) is passed for a given period of time through hot oil at a low pressure. This leads to three different effects on fat (Greyt 2020):

- 1 - removal: elimination of volatile components such as light fatty acids, odorous compounds, tocopherols, sterols, and contaminants, including pesticides and light polycyclic aromatic hydrocarbons (PAHs);
- 2 - the targeted removal of various substances responsible for specific odors;
- 3 - exposure to high temperature - thermal destruction of pigments (so-called thermal bleaching), the formation of unwanted toxic substances - trans fatty acids (TFA), esters of glycidyl (GE) and esters of 3-monochloropropane-1,2-diol (3-MCPD-E), polymerization and conjugation reactions.

Traditional deodorization technology leads to a deterioration in the quality of the oil (oxidation, polymerization, removal of antioxidants, formation of toxic compounds). In order to reduce the negative impact on the deodorized oil, a number of new technologies have been developed today. For example, the recently proposed multi-flash auto-evaporation technology “MFA” makes it possible to significantly reduce the deodorization temperature and its duration while maintaining the efficiency of removing odorous components (Jamoussi et al. 2022). Its idea is to combine two consecutive stages – an active stage using pressure and high

temperature treatment and a passive stage of tempering, which takes place under vacuum. The duration of each stage is short, effective deodorization occurs as a result of several repeated cycles, each of which combines the two specified stages. Inlet oil pressure: 200-450 kPa, temperature: 120-148°C, vacuum level (absolute pressure of 4 kPa) and a total time not exceeding 2 min (Jablaoui et al. 2020), 7 treatment cycles are proposed. The drop in total pressure induced an autovaporization of the volatile molecules. Recent studies (Jamoussi et al. 2022; Yang et al. 2021) show that the use of MFA technology allows for the production of high-quality deodorized oils with an increased content of tocopherols and tocotrienols.

Also promising is the method of oil deodorization, where the extractant for odorous substances is ethyl alcohol vapor. Deodorization is carried out at a lower temperature than the traditional method (140 to 220°C), the results of studies with rapeseed oil (Yang et al. 2021) showed lower response values of electronic nose, free fatty acid (0.03% to 0.07%), and peroxide value (0.00 to 0.67 meq.kg⁻¹), but higher retention of tocopherols (554.93 to 551.59 mg.kg⁻¹), total phenols (43.36 to 45.42 mg GAE.kg⁻¹), total carotenoids (65.78 to 67.85 mg.kg⁻¹). This method also expands the possibilities for obtaining higher quality deodorized oils. Although, it should be noted that the increase in the residual antioxidant content is not significant and the deodorization temperature remains high - 180°C is recommended as optimal conditions. However, ethanol has been proven to be an effective extractant for the odorous components of oils.

Volatile organic compounds (VOCs) are an important individual characteristic of vegetable oils, as they are responsible for the aroma and taste of a particular type of oil. They are characterized by low molecular weight (less than 300 Da) (Putri et al. 2022), easily evaporate at room temperature.

Odorizing (aromatic) compounds should be differentiated from volatile compounds, as volatile compounds do not always possess a distinct odor. Various food products contain approximately 12,000 volatile compounds, but only about 5% significantly contribute to their aroma profile (Jeleń et al. 2012). These compounds can be detected by the human olfactory system only when their concentrations surpass their odor threshold.

Today, the method of sampling volatile substances HS-SPME in combination with identification by gas chromatography in combination with quadrupole mass spectrometry GC-MS is common for the analysis of volatile fats (Putri et al. 2022). Other modern instrumental methods - Electronic Nose, Liquid Chromatography-Mass Spectrometry (LC-MS) (Bi et al. 2024). When used, it is possible to identify and determine the concentration of more than 100 volatile compounds in oils. Most vegetable oils contain aldehydes, carbohydrates, alcohols, ketones, esters, furans, sulfur compounds and terpenes. The composition of volatiles depends primarily on the fatty acid composition of oils and their oxidative status. The number of volatile oils also varies greatly. According to various studies (Tao et al. 2023; Ozcan-Sinir et al. 2020), sunflower oil contains relatively low amounts of volatiles. The composition of the main volatile sunflower oil is given in Table 1.

Variations in the composition of volatile compounds observed across different studies, influenced by numerous factors, result in inconsistent data regarding both the quantitative and qualitative profiles of volatile compounds in

sunflower oil (columns 3 and 4 of Table 1). The presence of aldehydes with low thresholds of perception and an increase in their quantity during the oxidation process is undoubted.

All odorants are characterized by different thresholds of perception. Aldehydes are the main volatile components of sunflower oil, characterized by low thresholds of perception. For example, for pentanal - 38 ng.L⁻¹, hexanal - 51 ng.L⁻¹, heptanal - 48 ng.L⁻¹, octanal - 9.3 ng.L⁻¹, nonanal - 12 ng.L⁻¹, decanal - 4.3 ng.L⁻¹, etc. (Scortichini et al. 2020). Also, aldehydes are mainly responsible for changes in the sensory characteristics of sunflower oil as a result of oxidation (Table 1). Most sunflower oil contains the following aldehydes: octanal, hexanal, pentanal, nonanal, 2-hexenal, 2-pentenal, 3-hexenal, 2-methylbutanal, 3-methylbutanal, decanal. Sunflower oil also contains volatile acids, the main ones being the following: hexanoic, pentanoic, nonaic, octanoic. Medium-chain fatty acids (C8-C12) were rated as mainly irritating with long-chain fatty acids (C18) described mostly as bitter. The latter may reflect the lack of “fatty” lexicon to describe the sensation (Cheon et al. 2021).

Table. 1. Main odors of unrefined fresh and oxidized sunflower oil and sensory characteristics of odors

VOCs ^a	Sensory threshold, µg.L ⁻¹ (Flavor description) ^b	The content of volatile compounds		
		µg.kg ^{-1c}	µg.L ^{-1d}	After 40 h of frying at 180°C, µg.kg ^{-1c}
Aldehydes				
Hexanal	11 (grass, green, rancid, tallowy, fat)	343.07	720.9	1489.7
Heptanal	85 (rancid, fatty, citrus)	60.19	142.52	248.28
(E)-2-Hexenal	25 (green, banana, fatty, cheesy)	106.72	147.86	204.95
Octanal	170 (aldehydic, waxy)	107.84	168.42	303.69
(E)-2-Heptenal	88 (soapy, almond, oily, fatty)	1621.67	2133.85	3105.86
Nonanal	12 (citrus, fruity, fatty, oily)	664.50	1255,5	1574.58
(E)-2-Octenal	20 (fatty, soapy, green, oily, fresh)	185.44	617.4	1157.68
Decanal	430 (floral, fresh, soapy, tallowy)	217.7	300.7	426.09
(E)-2-Nonenal	10 (green, soapy, cucumber)	67.9	167.4	247.14
(E)-2-Decenal	3,2 (orange-like, tallowy)	75.0	288.6	338.51
(E, E)-2,4-Nonadienal	(fatty, chicken)	19.54	68.0	115.71
2-Undecenal	5 (fresh, fruity, citrus)	54.22	187.44	207.2

Table. 1. - Continuation

VOCs ^a	Sensory threshold, $\mu\text{g.L}^{-1}$ (Flavor description) ^b	The content of volatile compounds		
		$\mu\text{g.kg}^{-1\text{c}}$	$\mu\text{g.L}^{-1\text{d}}$	After 40 h of frying at 180°C, $\mu\text{g.kg}^{-1\text{c}}$
2,4-Decadienal	10 (orange, sweet, fresh citrus, fatty, green)	258.84	805.99	1044.71
(E, E)-2,4-Decadienal	5 (aldehydic, fatty chicken, green, fried and potato)	813.0	2648.23	3646.54
Alcohols				
1-Octen-3-ol	2,7 (green, mushroom, fungal)	353.36	557.2	795.82
1-Pentanol	400 (balsamic)	63.64	142.24	295.77
1-Undecanol	(fresh, waxy, rose)	67.74	95.63	162.38
2-Dodecanol	(characteristic)	40.32	58.34	98.45
2-Butyl-1-octanol	(characteristic)	ND	25.52	43.01
1-Nonen-3-ol	(earthy, mushroom, green)	ND	19.65	31.54
Ketones				
2,4-Heptanedione	140 (ND)	15.0	189.8	244.8
3-Hexen-2-one	ND	ND	47.6	218.1
3-Methyl-3-buten-2-one	ND	94.73	121.38	147.40
4-Methyl-3-heptanone	ND	22.19	72.7	109.32
3-Nonen-2-one	(pleasant fruity)	28.6	67.35	94.53
Acids				
Hexanoic acid	3000 (sour, fatty, sweaty, cheese)	71.2	313.05	711.87
Pentanoic acid	100 (acidic, dairy-like with milky and cheese nuances)	ND	9.2	120.60
Nonanoic acid	12 (green, fatty)	ND	ND	57.65
Heptanoic acid	5 (waxy, cheesy, fruity, dirty and fatty)	ND	ND	26.37
Hydrocarbons				
Undecane	30 (fruity, ketonic)	365.37	429.8	377.45
Hexadecane	ND	ND	171.6	138.80
Tetradecane	(mild waxy)	46.6	70.48	87.29
Furan				
2-Pentyl-furan	181 (butter, green beans)	149.83	339.62	820.43

^a VOCs – Volatile organic compounds.^b (Van Dongen 1963-2021; Xu et al. 2017; Laska et al. 2010).^c (Liu et al. 2019).^d (Ozcan-Sinir 2020).

The influence of alkanes on the change of organoleptic parameters is also considered insignificant due to the high thresholds of perception of their odor (Goicoechea et al. 2014).

The largest amounts of undecane and nonadecane in sunflower oil. The main furan in fried sunflower oil - 2-pentyl furan is also one of the oxidation products of linoleic acid.

The process of deodorization of vegetable oils is very energy-consuming (heating the oil to temperatures of 200 - 240°C), the use of direct steam (0.5 - 3%) etc. requires sophisticated equipment that operates using a high vacuum (2-4 mbar).

3-MCPD esters and glycidol esters are generated during deodorization processes conducted at high temperatures. They belong to the group of toxic compounds that have been actively studied in recent years and raise concerns about the safety of consumption of deodorized fats (European Commission 2018). What is the toxic effect of these esters? The primary concerns associated with these compounds include their genotoxic effects, as they can negatively impact cellular genetic material or DNA, potentially leading to mutations; carcinogenic effects, which increase the risk of cancer; nephrotoxicity, contributing to kidney damage; and their harmful impact on male fertility. Notably, glycidol is classified as a probable human carcinogen (Group 2A) (Yabani et al. 2024).

The task of searching for other methods of deodorization that do not require the use of such high temperatures is difficult due to the high affinity of odorizing compounds for oils, their low concentration, and the heterogeneity of their composition. However, most of the odorous compounds are aldehydes and some of the ketones. Thus, it is likely that the deodorizing effect of vegetable oils can be achieved by removing aldehydes and ketones from the oils, or by modifying them in some way.

In our research aimed at reducing the content of secondary oxidation products in oils, we observed a decrease in the anisidine value when oils were treated with alcohols in the presence of acid catalysts. This led to the assumption that aldehydes are converted into acetals. A similar reaction, resulting in the formation of ketals, is also characteristic of ketones (Ouellette et al. 2014).

Obtaining acetals in chemical synthesis technologies is typically carried out by treating the carbonyl compound with alcohol and/or a suitable orthoformate in the presence of an acid catalyst (Keydel et al. 2024).

The use of ethanol (renewable raw material) and strong liquid inorganic acid as sulfuric and phosphoric acid as a catalyst is considered promising for the industrial production of acetals (Capeletti et al. 2000).

Hemiacetals are formed by the simple addition of alcohol to an aldehyde (or hemiketals in the reaction of alcohol and ketone). Like all carbonyl-neutral additives with neutral nucleophiles, the reaction occurs more rapidly in the presence of an acid, but this is not an absolute requirement: hemiacetals are fairly well formed under neutral conditions. However, in order for hemiacetal to reach full acetal, it is necessary to add an initiator - an acid catalyst for the reaction (Capeletti et al. 2000). The reaction is equilibrium, so all steps can be reversed. To switch from acetal back to aldehyde or ketone, you just need to heat the acetal in water in the presence of acid.

Our previous studies demonstrated the effectiveness of deodorizing sunflower oil by incorporating ethyl alcohol and an acid catalyst. This approach is particularly intriguing due to its potential to serve as an alternative deodorization method operating at relatively low temperatures (below 100°C), warranting further investigation.

The purpose of the research is to test the hypothesis of the possibility of sunflower oil deodorization at a temperature below 100°C and establish the optimal conditions for this process.

Materials and Methods

The studies were conducted using pressed sunflower oil of Ukrainian origin. The oil was used after degumming.

The treatment of sunflower oil with alcohol in the presence of a catalyst was carried out in a 500 mL round-bottomed flask, thermostatically controlled and connected through a ground joint to an air condenser. The reaction mixture was stirred at 250 rpm, and the temperature was maintained within the range of 60-90°C (with a control error of $\pm 0.1^\circ\text{C}$)

using a laboratory magnetic stirrer equipped with a thermocouple (RIVA-04.4, RIVA-STAL, Ukraine).

Since the acetal formation reaction can be reversible in the presence of water, anhydrous magnesium sulfate was added to the reaction vessel to remove water. The magnesium sulfate was separated from the oil prior to the deodorization process. Before carrying out the reaction, the oil sample was deaerated by using a vacuum pump (pressure 3 - 5 mbar). The reaction was carried out under inert-gas flushing conditions (nitrogen, purity 99.99%, Tekhgaz-Kharkov Ltd.) to prevent oil oxidation. After treating the sunflower oil with alcohol and acid, the oil was washed in a separatory funnel to eliminate residual acid until a neutral reaction was achieved. Subsequently, the water-alcohol mixture was removed from the oil using a rotary laboratory evaporator (IKA RV 10 digital V-C) for 30 min at a pressure of less than 5 mbar. Distillation temperature - 140°C. Oxidation of degummed sunflower oil was performed by storing it with access to oxygen from the air and the sun for 6 months.

The acid value of sunflower oil was determined according to the AOCS method Cd 3a - 63 (AOCS, 1997), the peroxide value - AOCS Cd 8b-90 (AOCS, 2017). The levels of 3-MCPD and glycidol were analyzed using gas-liquid chromatography on an Agilent 7890V/5977B gas chromatograph-mass spectrometer equipped with an automated sample preparation system based on MPS Robotic, following a modified ISO 18363-1:2015 method.

Degumming of sunflower oil was carried out by adding 1% water and 0.2% citric acid using a magnetic stirrer with a thermocouple with constant stirring at a speed of 160 rpm. The sample volume was 500 mL the temperature control accuracy was $\pm 0.1^\circ\text{C}$. A magnetic stirrer (RIVA-04.4, RIVA-STAL, Ukraine) and a centrifuge (RC-6, Dastan, Russia) operating at 1000 $\times g$ were used in all experiments. The process duration was 15 min at 60°C, after which the mixture was cooled to room temperature and left for 60 min. Then the mixture was separated in a centrifuge for 10 min, the resulting degummed oil was used in further experiments.

Neutralization of degummed sunflower oil was performed at 60°C for 15 min when using sodium hydroxide 0.1 N. Bleaching was performed with

degummed neutralized deodorized oil at a temperature of 60°C for 15 min in the presence acid activated clay Tersil Z. After each refining step, centrifugation (1000 $\times g$ for 10 min) and separation of by-products of the stages were performed.

The analysis of the taste and odor profiles of sunflower oil samples was conducted following the method proposed by (Güner et al. 2019). A panel of 10 tasters (4 men, 6 women, aged 20-60) participated in a voluntary 10-hour training session aimed at establishing the organoleptic parameters of sunflower oil. The study focused on the taste and odor intensity of various sunflower oil samples available on the Ukrainian market. After the training, the panel evaluated the sunflower oil samples produced through the reaction described in the article.

To assess the effectiveness of deodorization, a sensory panel was employed, with a scale ranging from 1 (indicating no taste or odor) to 5 (indicating the taste and odor typical of unrefined sunflower oil, without added flavors). The oil samples were placed in transparent glass containers (Petri dishes), covered before serving, and labeled with coded numbers. The samples were stored at room temperature and analyzed in daylight.

Analysis of variance (ANOVA) was used to determine the statistical significance of the effects of ethanol content (X_1), deodorization duration (X_2), and sulfuric acid content (X_3) on the sensory properties of sunflower oil. 16 experiments were conducted in a given factorial space: X_1 : from absence to 10% relative to oil; X_2 : from 1 to 20 min; X_3 : from absence to 0.2% relative to oil. The range of variation was chosen based on previous experiments.

The data were analyzed using three-way ANOVA to assess: the effect of each independent variable (X_1 , X_2 , X_3), two-factor interactions (X_1X_2 , X_1X_3 , X_2X_3) and the three-factor interaction ($X_1X_2X_3$). The significance level was $p < 0.05$.

The results of the experiment are a regression equation of the form:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 + b_{123}X_1X_2X_3,$$

Y - sensory result of deodorization on a 5-point scale, where 1 - complete impersonalization of the

taste and smell of oil, 5 - characteristic taste and smell of raw sunflower oil without changes;

b_0 – intercept;

$b_1, b_2, b_3, b_{12}, b_{23}, b_{13}, b_{123}$ - interaction coefficients.

At 4 points, the calculated response value (Y - sensory result of deodorization) was determined according to the obtained regression equation. Experimental studies were conducted at the same points (with the same values of the parameters X_1, X_2, X_3). The obtained values of the experimental results and the theoretical ones (according to the regression equation) were compared and the coefficient of determination (R^2) value was established. In this way, it was assessed how well the model describes the obtained results. In scientific and technical research, $R^2 > 0.70$ is usually considered acceptable. Mathematical modelling was carried out using the Python programming language and the Sklearn libraries to determine the regression equation and Scipy for optimization.

The samples were randomized and tested on different days, with all measurements repeated three times. The statistical significance of differences between the mean values of all measurements was assessed at a significance level of $p = 0.05$ (5%).

Results and Discussion

5% ethyl alcohol (96%), 0.5% sulfuric acid (98%) were added to degummed unrefined sunflower oil, and the reaction of formation of acetals, ketals from aldehydes and ketones of sunflower oil was carried out. A mixture of alcohol, water, and reaction products was distilled off from the oil under reduced pressure. The results are presented in Table 2. As a result, at a reaction temperature of 90 there was a deodorizing effect - the specific smell and taste of sunflower oil disappeared.

In order to confirm the working hypothesis about the depersonalization of the taste and smell of sunflower oil by the formation of acetals and ketanals, sunflower oil was treated separately with ethyl alcohol under the same conditions, an aqueous solution of sulfuric acid (0.5% 98% sulfuric acid), acetone in the presence of sulfuric acid. The deodorization effect did not occur in any of the samples.

A decrease in the efficiency of deodorization was observed with an increase in the number of OH groups in alcohols, which is probably due to the difficulty of the reaction for stoichiometric reasons.

It was decided not to study other monohydric alcohols for the purpose of deodorizing oils, as the safest food liquid among them is ethyl alcohol.

Table 2. Change in organoleptic characteristics of sunflower oil

Initial oil, characteristics	Process temperature, °C	Changes of characteristics
Unrefined degummed sunflower oil, taste and smell are inherent in sunflower oil, without extraneous flavors	60	Taste and smell inherent in sunflower oil
	70	Taste and smell inherent in sunflower oil
	80	Reducing the intensity of taste and smell inherent in sunflower oil
	85	Significant reduction in the intensity of taste and smell inherent in sunflower oil, but the oil is identified
	90	Lack of taste and smell inherent in sunflower oil

To determine the optimal conditions for deodorizing sunflower oil, an experimental study was conducted with three varying factors. The outcome was assessed based on the sensory effect of

deodorization using a 5-point scale, where 1 represents complete depersonalization of the taste and odor of the oil, and 5 indicates the taste and odor typical of raw sunflower oil without any changes.

Intervals of variation: the amount of ethyl alcohol X_1 : 0 – 10 % ; duration of deodorization X_2 : 1 – 20 min; the amount of sulfuric acid (X_3 : 0 – 0.2 %). It was previously found that at temperatures below 90°C the full effect of deodorization does not occur. It was decided not to raise the temperature above 90°C to prevent oxidative processes in the oil.

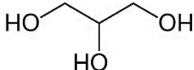
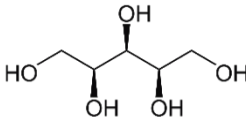
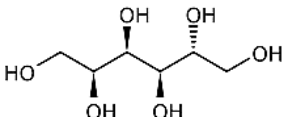
The possibility of deodorization of vegetable oils with food polyols (polyhydric alcohols) in the presence of sulfuric acid was investigated. It was assumed that with an increase in the number of OH-groups, that is, active centers of the reaction, the

efficiency of deodorization will increase. All experiments used 5% polyol, 0.5% sulfuric acid 98%. The results are presented in Table 3.

As a result of processing the data of the factorial experiment, a regression equation was obtained that describes the influence of the parameters of low-temperature deodorization:

$$Y = 8.64 - 4.09 \cdot X_1 - 6.95 \cdot X_2 - 1.24 \cdot X_3 + 0.045 \cdot X_1^2 + 0.021 \cdot X_2^2 + 95.5 \cdot X_3^2 + 0.008 \cdot X_1 \cdot X_2 - 0.820 \cdot X_1 \cdot X_3 - 0.403 \cdot X_2 \cdot X_3$$

Table 3. Deodorization of sunflower oil by polyols

Polyol used	Polyol formula	Initial oil for deodorization	Result
Glycerin			The intensity of taste and smell decreased significantly, but the smell of sunflower oil could be identified
Xylitol		Unrefined degummed sunflower oil	The intensity of the smell has decreased; the smell and taste of sunflower oil are felt
Sorbitol			The taste and smell are almost identical to unrefined sunflower oil

The correlation coefficient of the equation $R^2 = 0.74$. From the obtained regression equation, it can be concluded that an increase in all selected factors has a positive effect on the deodorization of sunflower oil (the best is the minimum response value). Regarding the mutual influence of factors: a simultaneous increase in the amount of alcohol and catalyst, as well as the duration of deodorization and the amount of catalyst leads to an increase in the efficiency of deodorization. All other vapors reduce the effectiveness of deodorization. In our study interval, only the duration of deodorization has an extremum at point 16.3, an increase in other factors leads to an improvement in the deodorization result, but the presence of quadratic values of all factors indicates the presence of function extremes that are outside the study range. From the point of view of determining the rational conditions of deodorization of sunflower oil, it was decided to narrow the

intervals of variation - for the amount of ethyl alcohol - 2%, for the amount of sulfuric acid - 0.05%. The resulting function reaches a minimum at the point:

$$X_1 = 2; X_2 = 16.3; X_3 = 0.05$$

In this case, the minimum response value is $Y = -1.16$. Thus, the rational conditions for low-temperature deodorization are as follows: ethyl alcohol concentration – 2%, reaction time – 16.3 min, sulfuric acid (98%) concentration – 0.05%, and deodorization temperature – 90°C.

The results of changes in the intensity and sensory profiles of sunflower oil following low-temperature deodorization (at the points $X_1 = 2$, $X_2 = 16.3$, $X_3 = 0.05$) are presented in Fig. 1.

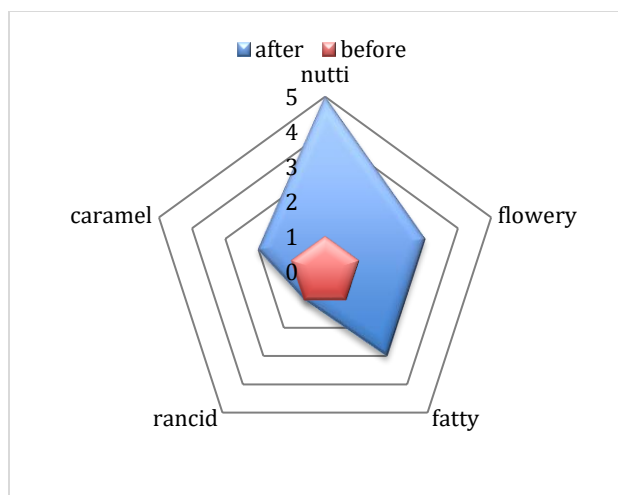


Figure 1. Changes in the sensory characteristics of sunflower oil as a result of low-temperature deodorization

Thus, as a result of low-temperature deodorization, the characteristic shades of smell (nut, fruit, fatty,

caramel) and taste disappear, the oil becomes depersonalized. The intensity of the aroma also corresponds to the samples of the traditional deodorized sunflower oil.

Qualitative indicators of unrefined degummed sunflower oil before and after low-temperature deodorization were established (Table 4). The process of low-temperature deodorization, in addition to the reaction of oil with alcohol, also includes washing the oil from acid residues and distilling off residual water and alcohol.

Carrying out low-temperature deodorization does not negatively affect the quality of sunflower oil. There is a decrease in the value of the peroxide value. It is possible that hydroperoxides are destroyed under the influence of sulfuric acid with the formation of secondary oxidation products, for sunflower oil is characterized by the formation of primarily aldehydes.

Table 4. Quality parameters of sunflower oil before and after low-temperature deodorization

Parameters	Before deodorization	After deodorization
Acid value, mg KOH.g ⁻¹	1.42 ± 0.15	1.39 ± 0.17
Peroxide value, mmol½O.kg ⁻¹	4.5 ± 0.12	2.2 ± 0.08
P-anisidine value	1.8 ± 0.08	2.0 ± 0.19
TOTOX	10.80	6.46
Mass fraction of phosphorus-containing substances, % in terms of stearooleolecithin	0.065 ± 0.0074	0.063 ± 0.0065
Mass fraction of moisture and volatile substances, %	0.10 ± 0.05	0.13 ± 0.45
Color, mg of iodine	15	15
Taste and odor	Inherent in degummed sunflower oil without foreign odor, taste and bitterness	Taste of impersonal oil, odorless

One of the indicators of oil quality is the profile of fatty acids, especially polyunsaturated ones, the content of which decreases as a result of the oxidation process. The results shown in Table 5 demonstrate the absence of significant changes in the content of essential fatty acids in sunflower oil.

It was decided to monitor the change in aldehyde content with anisidine number. After 10 min of reaction (under pre-deaeration and inert-gas flushing to prevent oil oxidation), the P-anisidine value (PAV) decreased from 2.06 to 1.31.

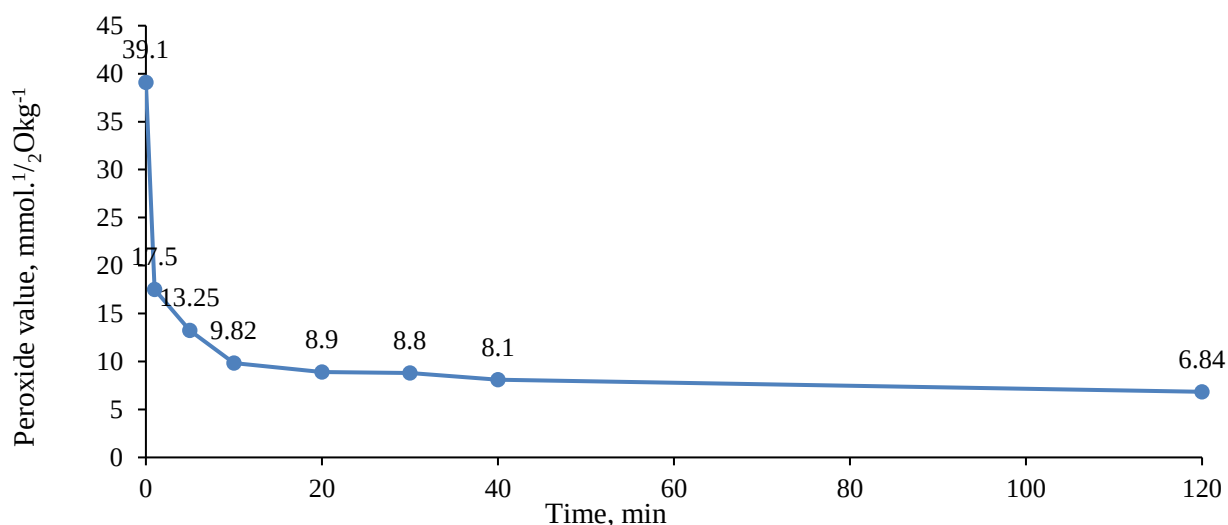
Table 5. The composition of fatty acids in sunflower oil before and as a result of low-temperature deodorization

Sunflower oil	Mean $\pm$ SD									
	C _{8:0}	C _{10:0}	C _{12:0}	C _{14:0}	C _{16:0}	C _{18:0}	C _{18:1}	C _{18:2}	C _{18:3}	C _{20:0}
Degummend oil	-	-	-	0.09 $\pm$ 0.05	6.3 $\pm$ 1.5	3.77 $\pm$ 0.8	27.3 $\pm$ 3,5	59.8 $\pm$ 5.2	-	2.3 $\pm$ 1.0
After 8 min of deodoriz.	-	-	-	0.11 $\pm$ 0.08	5.9 $\pm$ 0.7	4.02 $\pm$ 1.2	28.8 $\pm$ 4.3	59.0 $\pm$ 4.6	-	1.8 $\pm$ 0.08
After 16 min of deodoriz	-	-	-	0,07 $\pm$ 0.04	7.0 $\pm$ 0.9	4.65 $\pm$ 1.0	27.0 $\pm$ 4.7	58.2 $\pm$ 6.8	-	2.1 $\pm$ 1.1

With increasing reaction time, the PAV value decreases insignificantly (up to 1.19 after 40 min), which also confirms the above-defined rational duration of deodorization of 10 min.

The method of low-temperature deodorization can

also be considered as a method of stabilizing fats for oxidative damage. For the purpose of more careful research the kinetics of change of peroxide numbers of sunflower oil preliminary oxidized with the initial content of hydroperoxides in 39.1 mmol $\frac{1}{2}$ O.kg $^{-1}$ was studied (Fig. 2).

**Figure 2.** Changes in peroxide content in sunflower oil during low-temperature deodorisation

As a result, we have a sharp decrease in the peroxide value already during the 5-min deodorization. At the same time sensory characteristics of oil are completely restored (Fig. 3). Thus, the method of low-temperature deodorization is both an effective method of restoring the sensory characteristics of fats and reducing the content of oxidation products.

It is known that 3-MCPD esters and glycidyl esters are formed during the deodorization stage of vegetable oils at specific temperatures: 3-MCPD esters form at temperatures around 160°C and

higher, while glycidyl esters form at temperatures from 230°C (Greyt et al. 2020). Therefore, in our conditions of low-temperature deodorization, these toxic components of oils should not be formed.

To confirm the results, their content in sunflower oil was determined before and after low-temperature deodorization (Table 6). The level of both 3-MCPD-E and GE in 100-1000 μ g.kg $^{-1}$ of each of the compounds is typical for sunflower deodorized oil (Demydova et al. 2021).

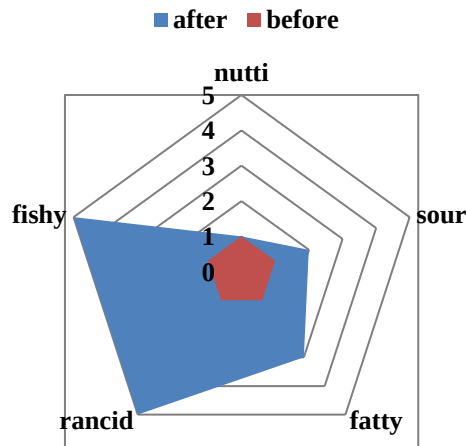


Figure 3. Sensory characteristics of oxidized sunflower oil (after) and after low-temperature deodorization (before)

As can be seen from the data in Table 6, the content of toxic esters of 3-MCPD and glycidol in the oil after low-temperature deodorization is below the detection limit.

Thus, the developed method of deodorization can be recommended as a method of obtaining deodorized

fats free of 3-MCPD-E and GE, which is an urgent task today (Jablaoui et al. 2020; An et al., 2022). However, unlike other numerous studies on reducing the content of 3-MCPD-E and GE, where it is possible to achieve only a partial decrease in their concentration (Yang et al. 2021) in deodorized fats, when using the low-temperature deodorization method, these toxic components of fats are not formed at all. The process of low-temperature deodorization itself is schematically presented in Fig. 4.

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The process of low-temperature deodorization itself is schematically presented in Fig. 4.

Table 6. Analysis of the content of 3-MCPD-E and GE before and after low-temperature deodorization

№ 1 (analysis of degummed sunflower oil)				
1	2	3	4	5
Mass fraction of 3-monochloropropanediol, µg.kg ⁻¹		less (<100)	± 30	ISO 18363-1:2015 (modified)
Mass fraction of esters of glycidyl fatty acids in terms of glycidol, µg.kg ⁻¹	not more than 1000*	less (<100)	± 37	
№ 2 (Analysis of oil deodorized according to the low-temperature method)				
1	2	3	4	5
Mass fraction of 3-monochloropropanediol, µg.kg ⁻¹		less (<100)	± 35	ISO 18363-1:2015 (modified)
Mass fraction of esters of glycidyl fatty acids in terms of glycidol, µg.kg ⁻¹	not more than 1000*	less (<100)	± 31	

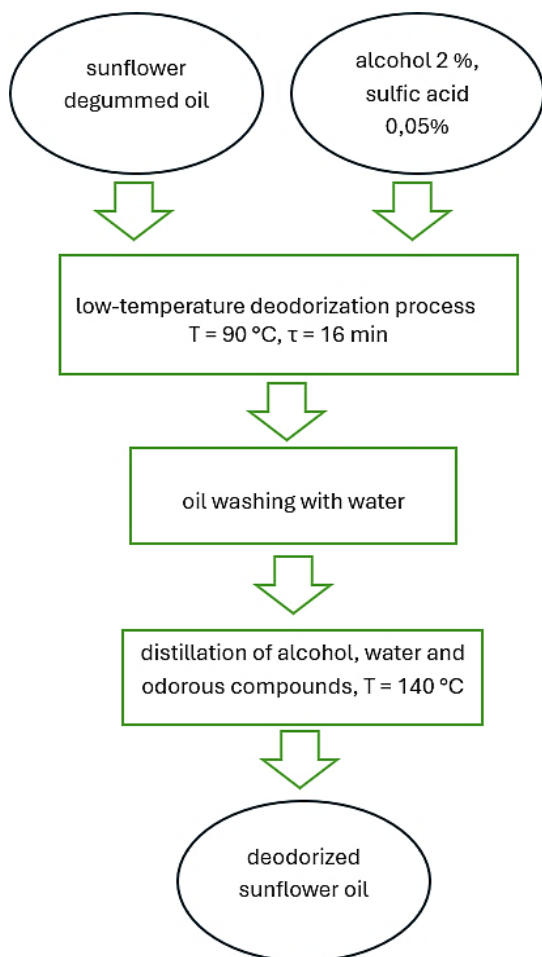


Figure 4. Schematic representation of the process of low-temperature deodorization of sunflower oil

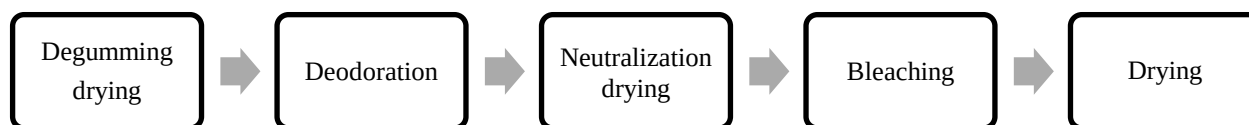


Figure 5. Block diagram of the sequence of stages of refining with the introduction of low-temperature deodorization

It is advisable to carry out low-temperature deodorization after the deaerator due to the negative effect of dissolved oxygen in the oil on its oxidation processes. Crude sunflower oil was refined according to the scheme in Fig. 5.

Quality indicators of refined deodorized sunflower oil were established (Table 7). All quality indicators of deodorized oil are within the requirements of regulatory documents. The only exception is the

Of course, the stage of low-temperature deodorization cannot replace the entire refining cycle. However, it obviously changes him fundamentally. The *degumming* step must be performed before the low temperature deodorization step due to the negative effect of the deodorization temperature and the subsequent evaporation of volatiles on the quality characteristics of phospholipids and oil. It is known that phospholipids at elevated temperatures are subject to melanoidins formation reactions, and will color the oils and add new odors to its sensory characteristics.

After degumming, it is necessary to dry the oil due to the negative effect that water has on the kinetics of reactions of formation of acetals and ketals.

The neutralization step can be performed both before and after low-temperature deodorization. In fig. 3 neutralization is after, due to the absence of the need for washing from the residues of the acid catalyst and the distillation of residues of water and alcohol before neutralization.

The stage of bleaching is mandatory, especially when it comes to modern refining cycles, due to the positive impact on the quality characteristics of oils. The block diagram of the sequence of stages of refining is shown in Fig. 5.

color number. Due to the lack of thermal destruction of pigments during traditional deodorization, the color number of sunflower oil deodorized by alternative technology is equal to the value for bleached oil.

The developed alternative method of sunflower oil deodorization is based on a fundamentally new approach, which differs from traditional deodorization in the following elements:

Table 7. Qualitative parameters of sunflower oil before and after the refining cycle with the introduction of low-temperature deodorization

Parameters	Qualitative indicators of unrefined sunflower oil	Qualitative indicators of refined deodorized oil
Acid value, mg KOH.g ⁻¹	1.9 ± 0.10	0.18 ± 0.05
Peroxide value, mmol ½O.kg ⁻¹	4.8 ± 0.14	1.1 ± 0.06
Mass fraction of phosphorus-containing substances, % in terms of stearooleocithin	0.72 ± 0,012	0.085± 0.005
Mass fraction of moisture and volatile substances, %	0.27± 0.08	0.10 ± 0.04
Color, mg of iodine	15	12
Taste and odor	Inherent in sunflower oil without foreign odor, taste and bitterness	Taste of impersonal oil, odorless

- no need to heat the oil to a temperature of 200°C or higher;
 - no need to use direct steam, which in combination with the first item leads to a significant reduction in the cost of the stage;
 - when using low-temperature deodorization there is a decrease in the content of peroxides and aldehydes, which should stabilize the oil against oxidative damage;
 - esters of 3-MCPD and glycidyl are not formed;
 - there is no distillation or thermal destruction of pesticides, volatile PAHs, other toxic substances that may be present in unrefined oil, ie they must be isolated at the stage of bleaching. Such technologies are known, mainly using activated carbon;
 - there is no distillation of free fatty acids, mono-, di- and triglycerols, which, on the one hand, reduces losses, and on the other hand - there will be no additional reduction in acid value during deodorization;
 - there will be no thermal bleaching of vegetable oils and reduction of oil color;
 - tocopherols and tocotrienols will remain in the oil in larger quantities, which should lead to greater stability of the oil to oxidative damage and theoretically to increase the shelf life;
 - there will be no formation of oxypolymers and other products of deep oxidation;
- there is no formation of trans-isomers. It is known that as a result of deodorization under normal conditions in sunflower oil accumulates 0.5 - 1.5% of trans-isomers of fatty acids.

Conclusions

The possibility of sunflower oil deodorization at temperatures below 100°C has been proven. The determined optimal conditions of low-temperature deodorization (temperature 60°C, amount of ethyl alcohol 2%, amount of sulfuric acid 0.05%, duration of the process 16 min) ensure the production of deodorized sunflower oil. Carrying out this reaction also relieves the oil of rancidity. The content of 3-MCPD esters and glycidyl esters in deodorized oil is below the detection limit.

Low temperature deodorization can provide oils with improved quality and safety. The cost of refining can be reduced due to the use of significantly lower temperatures and simplification of equipment requirements.

Author Contributions

Conceptualization, Demydova A., Aksonova O.; methodology, Aksonova O., Molchenko S., Berezka T., Demidov I., Yevlash V.; formal analysis Demydova A., Molchenko S., Berezka T., Demidov I., Yevlash V.; investigation, Demydova A.; writing, Demydova A., Aksonova O., review and editing, Molchenko S., Berezka T., Demidov I., Yevlash V.; All authors have read and agreed to the published version of the manuscript.

Funding

No funding

Informed Consent Statement

Not applicable.

Data Availability Statement

The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding authors.

Conflicts of Interest

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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