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INCREASING THE OXIDATIVE STABILITY OF LINSEED OIL

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The object of the research is the process of oxidation of linseed oil at elevated temperature.

Linseed oil is a valuable raw material for the chemical, food, cosmetic and other industries. The use of linseed oil is complicated by intensive oxidation processes associated with a high content of unsaturated fatty acids. Therefore, an important task is to develop and improve methods of oxidative stabilization of linseed oil.

The oxidation process of unrefined linseed oil (CAS Number 8001-26-1) at a temperature of 110 °C in its original form and with the addition of antioxidants was studied. The effect of antioxidant concentrations (tocopherol, butylhydroxyanisole and butylhydroxytoluene) in the mixture on the induction period of linseed oil was found. The induction period was determined by differential scanning calorimetry. The total concentration of the mixture of antioxidants in each experiment was 0.02 %. The induction period of the initial oil was 155.31 min. Rational ratios of antioxidants in the mixture were found: tocopherol: butylhydroxyanisole (50:50) %; tocopherol: butylhydroxytoluene (50:50) %; tocopherol: butylhydroxyanisole: butylhydroxytoluene (33.33:33.33:33.33) %. At the same time, the oil induction periods are 295.7 min., 290.1 min. and 290.2 min., respectively.

The quality indicators of the initial linseed oil and with the addition of the determined rational ratios of antioxidants after 2 hours of aging at a temperature of 110 °C were determined. The peroxide values of the samples were 8.5, 3.2, 3.6, 3.7 ½ O mmol/kg, respectively.

The research results make it possible to introduce antioxidants in the production of linseed oil in rational concentrations. This will help increase the production of linseed oil resistant to oxidation at elevated temperatures, which will provide various industries with high-quality raw materials

Keywords: linseed oil, free-radical process, oxidative stability, induction period, oxidation inhibitor

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1. Introduction

Linseed oil is used in chemical, pharmaceutical, cosmetic and other industries [1]. Linseed oil is used in the production of surfactants, paints, composite materials, protective and decorative coatings, biofuel, etc. [2, 3]. The use of biofuel with the addition of linseed oil reduces emissions of toxic components by (12–15) %, which reduces the harmful impact on the environment [4, 5]. In the food industry, linseed oil is used to create blended oils with a balanced ratio of essential linolenic and linoleic acids (from 1:5 to 1:10) [6, 7].

The peculiarity of the linseed oil composition is the high content of polyunsaturated fatty acids – (65.5–71.1) %. For sunflower oil, this indicator is (54.8–58.1) % [8]. This causes low oxidative stability and limits the use of linseed oil during heat treatment. In some cases, particularly, in the production of paint and varnish coatings, the increased ability of linseed oil to oxidize and polymerize is important [9, 10]. But for many directions, the appropriate quality of oil without oxidative degradation products is necessary, since oil acts as a reagent for obtaining products of a given composition [11, 12].

Peroxide oxidation of oils is a complex multi-stage free-radical process, as a result of which primary (peroxides and hydroperoxides) and secondary (alcohols, aldehydes, etc.) products of oxidation and oxidative destruction are formed [13, 14]. The physical and chemical properties of oils change, which complicates their use and worsens the quality of finished products based on oils [15, 16].

The oxidation resistance of oils depends on the fatty acid composition, the quality of the initial seed, temperature, light, etc. [1]. To inhibit oxidation processes, antioxidants (process inhibitors) are used. Common synthetic antioxidants are propylgallate (n-propyl ester of 3, 4, 5-trioxybenzoic acid), phenolic compounds: butylhydroxyanisole, butylhydroxytoluene, tert-butylhydroquinone. Natural antioxidants are tocopherols and tocotrienols, which are used in the amount of (0.02–0.06) %. At elevated temperatures, phenolic antioxidants are the most effective [17].

Therefore, an actual task is to develop and improve methods for increasing the oxidative stability of linseed oil, which is a raw material for various industries. Oxidative stabilization of linseed oil at elevated temperatures is of particular importance, since an increase in the temperature of storage and use of the oil significantly intensifies the processes of oxidative deterioration.

2. Literature review and problem statement

There are various ways to increase the oxidative stability of linseed oil, which involve the use of various antioxidants, as well as special conditions of storage and use of the oil. Thus, in [18], an oxygen-absorbing film based on the catalytic system with palladium was used to prevent the oxidation of lipids in linseed oil. Linseed oil was packaged in the normal or modified (with 2 % oxygen by volume) atmosphere with or without the catalytic system and stored at 45 °C for 6 months. During storage, peroxide and anisidine values were determined. The use of such a system significantly reduced lipid oxidation in linseed oil. However, there are no data on the effect of temperatures above 45 °C on oil storage under these conditions.

The method of oxidative stabilization of linseed oil is the use of antioxidants from vegetable raw materials. The authors [19] showed an increase in the oxidative stability of linseed oil using rice protein hydrolyzate obtained by the alcalase method. This was revealed by the reduction of the peroxide value by (95–96) %. The disadvantage is the complexity of the technology and protein instability at elevated temperatures. The work [8] shows the effectiveness of lycopene as an antioxidant for linseed oil. The presence of 80 mg/kg of lycopene in the oil resulted in the same induction time at 110 °C as the use of 200 mg/kg of the artificial antioxidant butylhydroxytoluene. But darkening of the oil was detected when lycopene was added. According to [20], plant antioxidants are effective in inhibiting the oxidation of linseed oil at a temperature of 60 °C. Antioxidant activity was characterized by the percentage of formation of conjugated dienes. But plant antioxidants have a limited temperature range of use. The effect of antioxidant concentrations on the peroxide value is also not shown.

To increase the oxidative stability of linseed oil at elevated temperatures, synthetic antioxidants are used. The authors [21] determined the effectiveness of various types of antioxidants at 100 °C. Ascorbic acid esters have been shown to effectively reduce the induction period of linseed oil. The work [22] presents data on increasing the oxidative stability of linseed oil using synthetic antioxidants tert-butylhydroquinone and butylhydroxytoluene with the addition of garlic extract. But these works do not provide data on the effect of antioxidant concentrations on the oil induction period.

One of the ways to produce oils with increased oxidative stability is blending. The authors [23] investigated the oxidative stability of mixtures of linseed and sesame oil at 110 °C, 120 °C and 130 °C. Sesame oil has significantly higher oxidative stability, contains compounds with antioxidant action, in particular, lignin. But this method does not solve the problem of increasing the oxidative stability of linseed oil if it is necessary to use the oil individually.

Thus, it is necessary to find the dependence of the oil induction period on the concentrations of antioxidants. Existing scientific studies show ways to increase the oxidative stability of linseed oil using various types of antioxidants. But an unsolved issue remains to determine rational concentrations of antioxidants at elevated temperatures, which will allow efficient use of resources and predicting the oxidative stability of the oil. Therefore, it is appropriate to study the effect of concentrations of individual antioxidants on the oxidative stability of linseed oil.

3. The aim and objectives of the study

The aim of the study is to determine the rational concentrations of antioxidants (tocopherol, butylhydroxyanisole and butylhydroxytoluene) in the mixture for oxidative stabilization of linseed oil. This will make it possible to obtain linseed oil with increased oxidative stability for various industries, as well as predict the induction period of linseed oil depending on the concentration of introduced antioxidants.

To achieve the aim, the following objectives were accomplished:

– to find the dependence of the induction period of linseed oil on the concentration of antioxidants in the mixture and determine the ratio of antioxidants under which the oxidation resistance of the oil is maximum;

– to determine the effect of antioxidant mixtures in rational ratios on the quality indicators of linseed oil at elevated temperature.

4. Materials and methods of research

4. 1. The object and hypothesis of the research

The object of the research is the process of linseed oil oxidation at elevated temperature. The main hypothesis of the research is that the concentrations of individual antioxidants affect the oxidative stability of linseed oil. The study suggests that the combined use of antioxidants has the greatest effect on increasing the induction period of linseed oil. A simplification is adopted that the initial physicochemical parameters of the oil do not affect the dependence of the oil induction period on the concentration of antioxidants. Standard research methods were applied.

4. 2. Examined materials and equipment used in the experiment

The following reagents and materials were used (CAS Number is a unique numerical identifier assigned to chemicals for a consistent method of identification by the Chemical Abstracts Service):

– unrefined linseed oil (CAS Number 8001-26-1);

– butylhydroxyanisole, purity 98.0 % (CAS Number 25013-16-5);

– butylhydroxytoluene, purity 99.0 % (CAS Number 128-37-0);

– α-tocopherol (CAS Number 1406-18-4).

The equipment used in the work is a differential scanning calorimeter (model Q20).

4. 3. Methods of studying the quality indicators of linseed oil

Physicochemical indicators were determined using generally accepted methods presented in international documentation on the analysis of oils and fats. The mass fraction of moisture and volatile substances is determined by the standard method intended for animal or vegetable fats and oils according to ISO 662:2016. Method A is used, which is applied for liquid and solid fats and oils (with different acid values). Acid value and peroxide value are determined according to the international standards ISO 660:2020 and ISO 3960:2017, respectively.

4. 4. Methods of determining the induction period of linseed oil

The induction period of linseed oil was investigated by differential scanning calorimetry (DSC) in the isothermal mode at a temperature of 110 °C in accordance with ISO 11357-1:2016. Research by the DSC method is carried out using a differential scanning calorimeter. The device has two measuring cells: one is intended for the test sample, in the other (comparison cell) an empty crucible is placed. The dependence of the heat flow difference between these two cells on time is measured. During the course of heat release or absorption processes (chemical reactions, phase transitions, etc.) in the sample, deviations from the monotonic change of the signal dependence on time (anomalies) are observed on the DSC curves. Thus, the oxidation process is characterized by a slow, and then a sharp decrease in the difference in the heat fluxes of the cells over time. Thus, the time of a sharp decrease in the signal value is determined. This is the time during which the slow phase of the chemical oxidation reaction (induction period) took place. The DSC method is a reliable and efficient method for measuring the degree of oxidation of oils and fats.

4. 5. Planning of experimental studies and processing of the data obtained

Scheffe's three-factor simplex lattice design was used to plan research and calculate mathematical dependence. Data processing was performed in the environment of the Stat Soft Statistica v6.0 package (USA). Each experiment was repeated twice. Scheffe's simplex lattice designs provide a graphical visualization of the results in the form of "composition-property" diagrams, which allow you to efficiently determine the rational concentrations of the three components and analyze the simultaneous effect of the components on the response function. Thus, the use of Scheffe's design meets the objectives of the study. In the Statistica package, calculations were made using the "3 Factor mixture design" module, which uses the following tabs:

– "Coeffs" (calculation of equation coefficients, standard error, 95 % confidence interval);

– "Observed, Predicted, and Residual Values" (determination of estimated values of the response function);

– "ANOVA" (analysis of variance).

5. Results of determining rational concentrations of antioxidants for oxidative stabilization of linseed oil

5. 1. Determining the dependence of the induction period of linseed oil on the concentration of antioxidants in the mixture

Unrefined linseed oil was used in the study. The determined quality indicators of the oil are presented in Table 1.

Table 1

Physicochemical indicators of initial linseed oil

Indicator	Value
Mass fraction of moisture and volatile substances, %	0.2
Acid value, mg KOH/g	1.6
Peroxide value, ½ O mmol/kg	2.3

According to the international standard for vegetable oils (CODEX STAN 210-1999), oils must have a mass fraction of moisture and volatile substances of no more than 0.2 %, acid value of no more than 4.0 mg KOH/g, peroxide value of no more than 10.0 ½ O mmol/kg. Therefore, the indicators of linseed oil correspond to the standard values.

The influence of antioxidant concentrations in the mixture on the induction period of linseed oil at a temperature of 110 °C was determined. The induction period is a slow phase of the chemical oxidation reaction, after which a rapid chain process begins. This parameter characterizes resistance to oxidation and reflects the shelf life of oils and fats. The induction period of the initial oil was 155.31 min. The following factors were studied:

– x_1 – concentration of tocopherol: (0–100) %;

– x_2 – concentration of butylhydroxyanisole: (0–100) %;

– x_3 – concentration of butylhydroxytoluene: (0–100) %.

The total concentration of the antioxidant mixture in each experiment was 0.02 % by mass of oil. The response function (y) is the induction period of linseed oil, determined in the isothermal mode at a temperature of 110 °C by the DSC method, min. In the package Stat Soft Statistica v6.0 (USA), the coefficients of the regression equation, the significance of the coefficients according to the p-criterion (p>0.05), which represents a 95 % confidence probability, are determined. The estimated values of the induction period in each experiment and the coefficient of determination (0.986) were determined. Table 2 shows the experimental design matrix, experimental (y_e) and calculated (y_c) values of the response function (induction period of linseed oil).

Table 2

Design matrix and response function values

Experiment number	Factors of variation			Induction period, min. (y_e)	Induction period, min. (y_c)
	Mass fraction of tocopherol, %	Mass fraction of butylhydroxyanisole, %	Mass fraction of butylhydroxytoluene, %		
1	100.00	0	0	283.20	281.69
2	0	100.00	0	250.70	251.05
3	0	0	100.00	270.80	271.96
4	50.00	50.00	0	295.70	293.61
5	50.00	0	50.00	290.10	288.82
6	0	50.00	50.00	273.30	273.88
7	66.67	16.67	16.67	288.30	293.77
8	16.67	66.67	16.67	278.70	278.58
9	16.67	16.67	66.67	286.50	283.92
10	33.33	33.33	33.33	290.20	291.19

The dependence of the induction period of linseed oil (y) on the mass fraction of antioxidants in the mixture, in its natural form, is as follows:

$$y = 266.215 + 0.692 \cdot x_1 - 0.005 \cdot x_1^2 + \\ +0.401 \cdot x_2 - 0.006 \cdot x_2^2 + 0.001 \cdot x_3^2. \quad (1)$$

A graphical dependence of the induction period of linseed oil on the concentration of antioxidants in the mixture was constructed (Fig. 1).

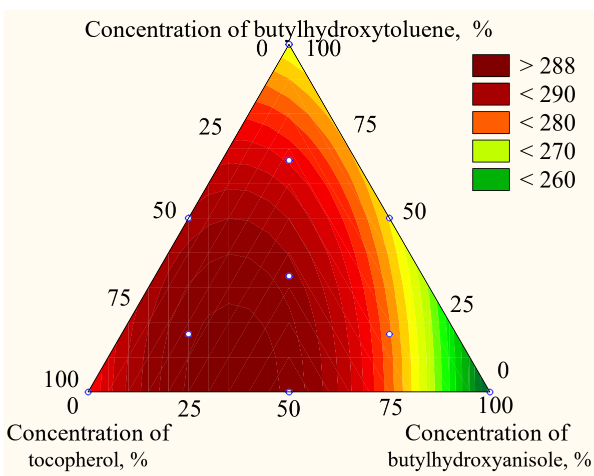

Fig. 1. Dependence of the induction period of linseed oil on the concentration of antioxidants in the mixture

As evidenced by analysis of equation (1), Table 2 and Fig. 1, when using individual antioxidants, the greatest increase in the induction period of linseed oil was demonstrated by tocopherol, the least by butylhydroxyanisole. In all experiments, when two or three antioxidants are used, higher values of induction periods are observed than the maximum possible for each of the individual antioxidants. This indicates a synergistic effect of antioxidants. The maximum values of induction periods are observed for the following ratios of antioxidants: tocopherol: butylhydroxyanisole (50:50) %; tocopherol: butylhydroxytoluene (50:50) %; tocopherol: butylhydroxyanisole: butylhydroxytoluene (33.33:33.33:33.33) %. The corresponding induction periods of linseed oil were 293.61 min., 288.82 min. and 291.19 min. Therefore, it is rational to use these concentrations of antioxidants in the mixture for oxidative stabilization of linseed oil.

5. 2. Determining the effect of antioxidant mixtures on the quality indicators of linseed oil at elevated temperature

The induction period of oils characterizes resistance to oxidation, but does not reflect physical and chemical indicators that are important for assessing oil safety, its suitability for use in certain chemical processes. Peroxide value, which characterizes the content of hydroperoxides in the oil, is one of the most important indicators of oil quality when controlling its degree of oxidation. The change in the quality indicators of the initial linseed oil and with the addition of rational mixtures of antioxidants during the oil aging in the drying cabinet at a temperature of 110 °C for 2 hours was studied. The obtained data are shown in Table 3 1 – mixture of tocopherol: butylhydroxyanisole (50:50) %; 2 – mixture of tocopherol: butylhydroxytoluene (50:50) %; 3 – mixture of tocopherol: butylhydroxyanisole: butylhydroxytoluene (33.33:33.33:33.33) %.

Table 3

Quality indicators of linseed oil after heating at a temperature of 110 °C for 2 hours

Indicator	Value			
	Initial	1	2	3
Mass fraction of moisture and volatile substances, %	0.15	0.16	0.15	0.15
Acid value, mg KOH/g	2.3	1.8	2.0	1.9
Peroxide value, ½ O mmol/kg	8.5	3.2	3.6	3.7

Therefore, the peroxide values of oil samples aged with mixtures of antioxidants are significantly lower than those of the initial sample (without antioxidants). The acid values of samples with antioxidants are also lower. The presence of the developed rational mixtures of antioxidants significantly reduces the rate of oxidative deterioration in linseed oil at elevated temperatures.

6. Discussion of the results of determining the rational concentrations of antioxidants for oxidative stabilization of linseed oil

The method of increasing the oxidative stability of linseed oil at elevated temperature using synthetic antioxidants was investigated. According to equation (1), Table 2 and Fig. 1, rational ratios of antioxidants were determined: tocopherol: butylhydroxyanisole (50:50) %; tocopherol: butylhydroxytoluene (50:50) %; tocopherol: butylhydroxyanisole: butylhydroxytoluene (33.33:33.33:33.33) %. At the same time, the induction periods of linseed oil amounted to 293.61 min., 288.82 min. and 291.19 min., respectively. The induction period of the initial linseed oil was 155.31 min.

Oxidation of fats occurs as a chain free-radical process, to slow down which inhibitors – antioxidants of various nature are used. So, phenolic antioxidants butylhydroxyanisole and butylhydroxytoluene are able to react very quickly with peroxide radicals with chain breakage. Inhibitors of different groups, being used together, mutually enhance each other's effect (so-called synergistic inhibitors). The issue of inhibition of oxidation processes is especially relevant for oils with a high concentration of polyunsaturated fatty acids. Such oils include linseed oil, which is an important industrial type of oils.

According to Table 2, the greatest increase in the induction period of linseed oil was demonstrated by tocopherol, the least by butylhydroxyanisole. With the simultaneous use of two or three antioxidants, a phenomenon of synergism is observed – mutual strengthening of the antioxidants action. Rational ratios of antioxidants are as follows: tocopherol: butylhydroxyanisole (50:50) %; tocopherol: butylhydroxytoluene (50:50) %; tocopherol: butylhydroxyanisole: butylhydroxytoluene (33.33:33.33:33.33) %. The addition of antioxidants made it possible to increase the oil induction period by 1.9, 1.86, 1.87 times, respectively.

The effect of mixtures of antioxidants in rational proportions on the quality indicators of linseed oil after exposure at a temperature of 110 °C for 2 hours was studied. The peroxide values of oil samples stored with antioxidant mixtures were, respectively, 2.65, 2.36, 2.30 times lower than the peroxide value of the sample without antioxidants (Table 3). This is due to the action of oxidation inhibitors, which cause chain breakage in reactions with peroxide radicals.

The obtained results make it possible to produce linseed oil with increased stability at elevated temperatures and predict the effect of antioxidants in different concentrations on the induction period of the oil. The results can be used in the production of linseed oil for various industries: chemical, food, pharmaceutical, cosmetic, etc. For example, in the production of cosmetic creams enriched with polyunsaturated fatty acids, it is advisable to use linseed oil. The use of the determined rational concentrations of antioxidants will help to increase the shelf life of the finished product – cream. This will also make it possible to significantly reduce the rate of formation of toxic oxidation products even in the case of using elevated temperatures in production (for example, during the dissolution or melting of individual components of the cream).

The works [8, 18–23] present data on various methods of oxidative stabilization of linseed oil using special storage films that absorb oxygen, as well as various types of antioxidants. Thus, the authors [22] showed the effectiveness of ascorbic acid esters. According to [23], oxidative stabilization of linseed oil using tert-butylhydroquinone and butylhydroxytoluene with the addition of garlic extract is appropriate. The main method of increasing the oxidative stability of linseed oil is the addition of various types of antioxidants, which was used in this work. But there is not enough data on the influence of antioxidant concentrations on the oxidative stability (induction period and physicochemical indicators) of linseed oil at elevated temperatures. This task is solved in the work.

The limitation of using the results of the work is the oxidation temperature and the concentration of antioxidants. Thus, with a decrease in the total concentration of antioxidants and an increase in temperature, the effect of antioxidants on the oxidative stability of the oil can be reduced.

The disadvantage of the study is the lack of data on the storage of linseed oil in the presence of antioxidants under standard conditions. It would be useful to check and compare the shelf life of the original oil and with the addition of antioxidants at standard temperature.

A promising direction of work is the use of a larger list of antioxidants of different nature, a wide range of oxidation temperatures. This will expand the possibilities for the production of linseed oil with increased oxidative stability.

7. Conclusions

1. The dependence of the induction period of linseed oil on the antioxidant concentrations in the mixture was determined. The greatest increase in the induction period of linseed oil was demonstrated by tocopherol, the least by butylhydroxyanisole. With the simultaneous use of two or three antioxidants, a phenomenon of synergism is observed – mutual strengthening of the action of antioxidants. Rational ratios of antioxidants were determined: tocopherol: butylhydroxyanisole (50:50) %; tocopherol: butylhydroxytoluene (50:50) %; tocopherol: butylhydroxyanisole: butylhydroxytoluene (33.33:33.33:33.33) %. At the same time, the induction periods of linseed oil at a temperature of 110 °C were 293.61 min., 288.82 min. and 291.19 min., respectively. The induction period of the initial linseed oil was 155.31 min. Therefore, the addition of antioxidants made it possible to increase the oil induction period by 1.9, 1.86, 1.87 times, respectively.

2. The quality indicators of linseed oil with the addition of rational ratios of antioxidants after 2 hours of aging at a temperature of 110 °C were determined. The peroxide values of the samples were 8.5, 3.2, 3.6, 3.7 ½ O mmol/kg, respectively. Therefore, the peroxide values of oil samples that were aged with mixtures of antioxidants were, respectively, 2.65, 2.36, 2.30 times lower than the peroxide value of the sample without antioxidants.

Conflict of interest

The authors declare that they have no conflict of interest in relation to this study, whether financial, personal, authorship, or otherwise that could affect the study and its results presented in this paper.

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Data availability

The manuscript has no associated data.

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