

## Study of the relationship between oxidation kinetics and deterioration of sensory characteristics of vegetable oils

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**Abstract.** The oil and fat industry is a branch whose development is rapidly gaining momentum in the food industry of Ukraine. However, it is currently facing new challenges that require solutions at the expense of both increasing production volumes and finding new mechanisms for technological solutions. The purpose of the article is to establish the relationship between the appearance of rancidity in vegetable oils and the composition of the main oxidation products in them - hydroperoxides and aldehydes, that is, to find a way to predict the moment of rancidity of oils using the peroxide number and/or anisidine number. A tasting commission consisting of 10 people was formed to carry out the organoleptic evaluation of vegetable oils for the appearance of bitterness. A sensory panel with a scale of taste variation was developed to study the nuances of bitterness. Oil samples for the experiment were placed in transparent glass (Petri dish). The article examines the possibility of predicting the appearance of rancidity by the main indicators of oil oxidation – peroxide and anisidine numbers. During oxidation at 28°C and access to oxygen, the oils accumulated significant amounts of hydroperoxides (up to 160-180 mmol<sub>1</sub>/2O/kg), but there was no feeling of rancidity. It has been proven that the moment of deterioration of organoleptic indicators of oils can be predicted based on the kinetics of oil oxidation based on the data of anisidine numbers. The practical value of the research is to contribute to the transformation and improvement of the products of the oil and fat industry, including through awareness of the changes that occur in fats during their extraction, processing and storage

**Keywords:** oxidation, rancidity, vegetable oils, anisidine number, aldehydes, peroxide number, hydroperoxides

### INTRODUCTION

Currently, the oil and fat industry faces fundamentally new tasks that are solved not only by increasing production volumes, but also require qualitatively new approaches and new technological solutions. The main task can be called the production of high-quality and safe products. Accomplishing these tasks requires a

deep understanding of the changes that occur in fats during their extraction, processing and storage.

One of the main problems of deterioration of the quality and safety of fats is their ability to undergo oxidative deterioration [1]. This problem is also related to the appearance of the so-called rancidity in fats that

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have been oxidized for some time. Primary oxidation products – hydroperoxides do not make a negative contribution to changing the taste and smell of oils [2], but their transformation products – secondary oxidation products, including aldehydes, ketones, spits, oxyacids, epoxy compounds, etc., have a specific taste and aroma of their own [3]. Odorous (aromatic) compounds should be distinguished from volatile compounds, because the latter are not always characterized by their own specific smell. Food products of various types have about 12,000 volatile compounds, but only about 5% of them play a significant role in the formation of the aroma of these products [4]. They can be perceived by the human olfactory system if present in concentrations exceeding their odor threshold.

Degradation of edible oil by oxidation during extraction, processing, storage, and use has long been recognized as a major problem in the edible oil industry [5]. Oxidation of cooking oil leads to undesirable taste, smell, loss of nutrients and formation of toxic compounds. These toxic compounds cause mutations, aging, stroke, emphysema, heart disease and cancer in the human body [6]. A particularly low odor threshold is characteristic of aldehydes, they are the main secondary oxidation products for vegetable oils presented on the Ukrainian market – sunflower, soybean, rapeseed, linseed [7]. Differences in the volatile substances of edible oils lead to the characteristic taste and smell of oils and differ significantly depending on the fatty

acid composition and other characteristics inherent in different oils. Essential polyunsaturated fatty acids are particularly susceptible to oxidation, and their presence in food products is associated with reduced shelf life and changes in their sensory perception [8;9]. At low temperatures, peroxides break down with the formation of secondary oxidation products slowly, and there may be a correlation between their content and the appearance of a feeling of bitterness. Most secondary products have a low molecular weight and are volatile [10].

These molecules form the aroma of oils and can be identified using solid-phase microextraction combined with gas chromatography and mass spectrometry (HS-SPME/GC-MS) [11-13]. It should be remembered that the method is not exhaustive, that is, it does not reflect the actual composition of volatile compounds in a certain product, but their relative contribution. Provides important information regarding the presence of odorants. Determining the content of volatiles in fats is a difficult task due to their low concentrations, the simultaneous presence of compounds in ppm (10-6%) and ppb (10-9%) concentrations, and the fact that odorant molecules belong to different chemical classes and are characterized by a wide range of molecular weights [14].

Data on the main odorous vegetable oils, including those formed in the oxidation process, are given in the table. 1.

**Table 1.** Main volatile vegetable oils and their perception thresholds

Volatile compound (ROAV)	Threshold of perception, ng/L	Content in oxidized oil, ng/L			Sensory characteristics
		Soybean	Linseed	Rapeseed	
1	2	3	4	5	6
Pentanol	38	11.5	1.96	1.98	Spicy, almondy, malty
Hexanol	51	11.4	23.7	10.17	Grassy
Heptanol	48	3.3	-	0,84	Rancid, greasy, citrusy
Octanol	9.3	41	42.8	40.37	Soapy, green, oily, fresh
Nonanol	12	100	100	100	Soapy, green, oily, fresh
Decanol	4.3	19.1	40.75	17.1	Floral, orange peel, soapy, greasy, green, fresh
2- butanol	350	-	0.6	0.11	Spicy
(E)-2-pentenol	1500	-	0.14	0.13	Strawberry, fruity, apple

Table 1, Continued

Volatile compound (ROAV)	Threshold of perception, ng/L	Content in oxidized oil, ng/L			Sensory characteristics
		Soybean	Linseed	Rapeseed	
1	2	3	4	5	6
(E)-2-heptenol	88	3.6	1.2	2.46	Greasy, soapy, almond, fatty
(E)-2-octanol	20	4.6	-	nd	Green, nutty, fatty
(E)-2-decanol	3,2	10.3	-	2.42	Orange
Benzaldehyde	186	1.8	2.65	1.91	Almond, burnt sugar
1-heptanol	3590	0.11	0.05	nd	balsamic
1-hexanol	360	-	0.5	nd	Green, floral
1-octanol	73	0.52	-	0.53	Chemical, metallic, annealed
Acetic acid	1384	-	0.26	nd	Sour
Hexanoic acid	3000	-	0.11	0.02	Sour, cheesy
Neanoeva acid	12	10.96	-	9.35	Green, fat
Dodecanese	5300	-	0.15	0.064	Gasoline
D-лімонен	718	2.75	1.06	0.38	Lemon, orange

**Source:** Adapted from [15]

All volatile substances are characterized by different perception thresholds. Aldehydes are the main volatile components of most vegetable oils [16]. According to the data in the table. 1, octanal, nonanal, decanal, (E)-2-decanal, nonanoic acid exert the greatest main influence on the feeling of rancidity of vegetable oils. Thus, mainly aldehydes affect the specific taste and aroma of various oils, as well as the change in their sensory characteristics as a result of oxidation [17]. The low level of hexanal (approximately 5 ng mL<sup>-1</sup>) as a result of its low perception threshold is easily distinguished by smell [18].

Among the other volatiles of sunflower oil, which are present in noticeable quantities, are alcohols, hydrocarbons, acids and ketones. Alcohols are usually formed as a result of the decomposition of unsaturated fatty acids [16]. 1-octen-3-ol is the main alcohol of sunflower oil. However, in general, alcohols are characterized by high perception thresholds, it is unlikely that they significantly affect the sensory characteristics of the oil [19]. Unsaturated alcohols have a lower odor threshold compared to saturated alcohols and, thus, their contribution to the organoleptic properties of oils is greater [20].

Ketones are formed as a result of the enzymatic

decomposition of polyunsaturated fatty acids during the degradation of amino acids or the Maillard reaction. The content of ketones increases in the process of oxidation [8]. For fats, autoxidation and photooxygenation, which are caused by the presence of oxygen in the air, are practically inevitable [21]. Fats always contain a certain amount of hydroperoxides (already in the composition of oilseeds), which are susceptible to decomposition into secondary oxidation products, including at the stage of extracting oil from seeds, for example, when obtaining the pulp before pressing, etc.

The term "rancidity" is used to characterize two different processes: oxidative rancidity caused by the formation of lipid oxidation products and hydrolytic rancidity caused by lipid hydrolysis [22]. Oxidative rancidity occurs in fats and oils that contain almost no water [21].

There is quite a lot of data on the relationship between rancidity and the content of oxidation products [23; 24]. The impact on organoleptics of the low-temperature oxidation process has not been sufficiently studied due to its duration and the greater complexity of such studies. The main volatile oxidizing compounds formed during oil storage at ambient temperature are partially different from those formed at

high temperatures [25]. The purpose of the research is to establish a connection between the formation of a feeling of rancidity in vegetable oils and the content of the main oxidation products in them – hydroperoxides and aldehydes, that is, the search for the possibility, using the indicators of peroxide number and/or anisidine number, to predict the moment of rancidity of oils.

## MATERIALS AND METHODS

The most widespread in Ukraine oils belonging to different fatty acid types were chosen as the object of the study, and thus, they can differ significantly in terms of oxidation kinetics and the time of rancidity. The following oils were studied and the initial amount of oxidation initiators – hydroperoxides (value of peroxide number – PM) was determined: unrefined sunflower oil (DSTU 4492:2005), PM=1.7 mmol 1/2O/kg, unrefined corn oil (DSTU 8808:2003) PM=1.4 mmol 1/2O/kg, unrefined rapeseed oil (DSTU 8175:2015), PM=1.5 mmol 1/2O/kg.

The anisidine number was studied according to DSTU ISO 6885-2002. The peroxide number was studied according to DSTU 4570:2006. The fatty acid composition of oils was studied by gas-liquid chromatography according to DSTU ISO 5509-2002.

To conduct an organoleptic evaluation of vegetable oils in order to determine the appearance of a feeling of bitterness, a tasting commission was formed in the number of 10 people (5 men, 5 women, aged 23-60 years), voluntarily included in the group. Commission members underwent at least 10 hours of training to determine variations in organoleptic changes in the process of oil oxidation. A sensory panel was developed to investigate the nuances of rancidity: “rancid” was defined as associated with the aroma of long-life fats with soapy-metallic aromas (minimum fried

potato chips, maximum linseed oil stored for 1 month with access to oxygen). A 5-point scale was used, with a variation of 1 – absent (characteristic of sunflower or other oil without extraneous odors and a taste of bitterness), 2 without extraneous odors, satisfactory taste, but slight bitterness, 3 – slightly sour smell and taste, 4 – sour smell and taste with notes of oxidized oil, 5 – the strongest (oxidized, musty smell of oxidized oil or olive oil). The first manifestations of bitterness were understood to mean “2” on the scale. Oil samples (fresh without rancidity and those stored for different times) were placed in a transparent glass (Petri dish), closed before serving and marked with coded numbers. The samples were kept at room temperature and analyzed in daylight. Samples were randomized and tested on different days.

## RESULTS AND DISCUSSION

The most destructive, from the point of view of oxidation, lipids are unsaturated fatty acid components. For example, the rate of self-oxidation of oleic, linoleic and linoleic methyl esters is 1:40:100 [26]. Linoleic acid is an important precursor of volatile compounds in edible oils and is readily oxidized to form hexanal, pentanal, heptanal, and trans-2-heptenal. Oleic acid is also an important precursor for the oxidation of volatile compounds, especially nonanal and octane compounds. The rate of oxidative degradation of linoleic acid is faster than that of linoleic acid and oleic acid, and linolenic acid is an important source of trans, trans-2.4-heptadienal, and trans-2-hexenal in the oxidation products of edible oils [27]. Thus, for the correct interpretation of the obtained data on the rancidity of oils, it is necessary to establish their fatty acid composition. The results of the research are given in Table. 2.

**Table 2.** Fatty acid composition of the studied oils

Oil	Fatty acid, %					
	C <sub>16</sub>	C <sub>18</sub>	C <sub>18</sub> <sup>=</sup>	C <sub>18</sub> <sup>2=</sup>	C <sub>18</sub> <sup>3=</sup>	C <sub>20</sub>
Soybean	4.5	3.4	28.2	54.9	9.0	
Corn	7.3	5.3	44.5	42.9		–
Linseed	5.9	3.8	22.6	37.8	28.8	8.5
Sunflower	6.2	3.5	20.1	64.2	5.4	0.7

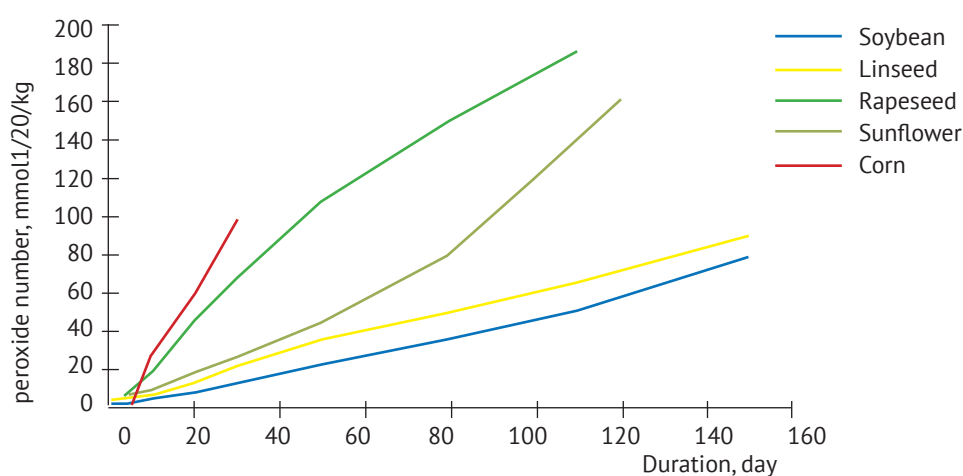
Table 2, Continued

Oil	Fatty acid, %					
	C <sub>16</sub>	C <sub>18</sub>	C <sub>18</sub> <sup>=</sup>	C <sub>18</sub> <sup>2=</sup>	C <sub>18</sub> <sup>3=</sup>	C <sub>20</sub>
Rapeseed	3.8	1.2	67.2	18.7	8.3	0.8

Unrefined sunflower and linseed oil contain the maximum number of unsaturated fatty acids among the studied oils – sunflower oil linoleic, linseed oil – linolenic (Table 2).

In order to establish the influence of the number of hydroperoxides on the organoleptic parameters of oils, the kinetics of the accumulation of peroxide number in the

samples under conditions of access to oxygen at a storage temperature of 28°C were studied. Measurement of hydroperoxides is a general assessment for determining the oxidation status of oils [28], and significantly more correct results are obtained at low oxidation temperatures. On Fig. 1 shows the graph of the kinetics of oil oxidation according to the change in the peroxide value (PH).



**Figure 1.** Kinetics of oil oxidation according to the change in peroxide values (under conditions of air oxygen access, storage temperature 28°C)

A correlation was observed between the rate of oxidation and the content of unsaturated fatty acids (see Table 2 and Fig. 1). Linseed oil accumulates hydroperoxides the fastest, corn and soybean oil the least, which is connected not only with the high content of linoleic acid (soybean oil), linoleic and oleic acids (corn oil), but also with the naturally high content

of antioxidants in these oils.

During the study of the kinetics of oxidation of oils based on the values of peroxide numbers, the organoleptic commission established changes in the smell and taste of oils and looked for the first manifestations of the feeling of bitterness. The results are shown in Table 3.

**Table 3.** The relationship between the amount of hydroperoxides and the formation of the rancid feeling of oils

Oil	The first signs of bitterness		Significant changes in taste and smell	
	PM, mmol/20/kg	Storage time, days	PM, mmol/20/kg	Storage time, days
Soybean	78	80	161	120
Corn	79	150	-	-
Linseed	45	20	120	50

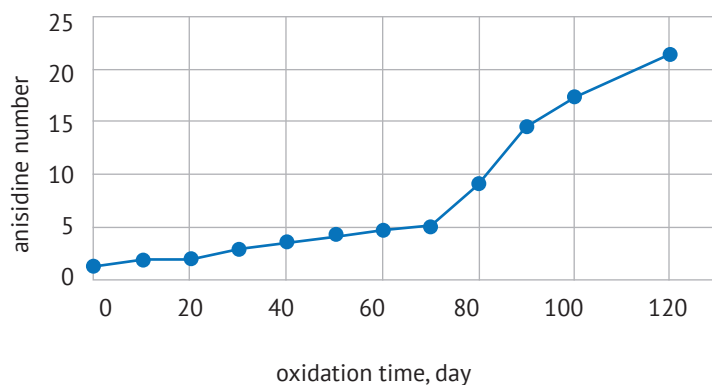
Table 3, Continued

Oil	The first signs of bitterness		Significant changes in taste and smell	
	PM, mmol1/2O/kg	Storage time, days	PM, mmol1/2O/kg	Storage time, days
Sunflower	65	110	90	150
Rapeseed	18	7	59	20

In our opinion, no correlation was found between the values of the peroxide number and the onset of rancidity of oils. Even with high values of peroxide values, the oils did not have a characteristic rancid smell (soybean, rapeseed oil, etc.), and linseed oil with low values of peroxide values already significantly changed the sensory characteristics – the feeling of rancidity was intense (see Table 3). At the selected research temperature (28°C), when oxygen is present in the oils, significant amounts of hydroperoxides accumulate (more than 100 mmol1/2O/kg), which is a rather rare condition for oils. In most cases, during the processing or use of oils, high temperatures are used, which leads to the rapid destruction of hydroperoxides with the formation of other oxidation products [29]. In the case of low-temperature oxidation, it is convenient to monitor the effect of hydroperoxides on the formation of the feeling of bitterness. Thus, soybean oil did not significantly change its taste and smell, despite

the high concentration of hydroperoxides (more than 150 mmol1/2O/kg). These data coincide with the results of other studies [15].

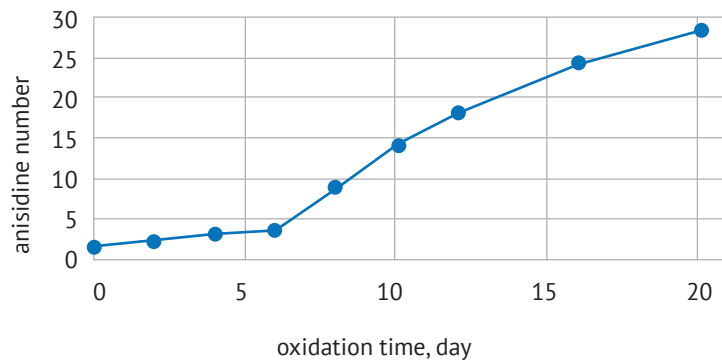
To establish the influence of the number of oxidation products on the feeling of rancidity, the kinetics of changes in the anisidine number (AN) of oils were studied, the results are shown in fig. 2-6. AC is a measure of the content of secondary oxidation products, such as aldehydes,  $\alpha$ -alkenals and  $\beta$ -alkenals and all compounds that can react with p-anisidine [30]. Those aldehydes that affect the result of AH are not volatile, but have an average and higher molecular weight. However, edible oils are considered acceptable for consumption when the pH is below 10 [31]. This indicates the almost absence of non-volatile aldehydes [32]. Therefore, it would be reasonable to find out how the AP values are correlated with the organoleptic evaluation data (when low-molecular volatile aldehydes appear in amounts above the recognition threshold and a feeling of bitterness appears).



**Figure 2.** Kinetics of sunflower oil oxidation according to the change in anisidine numbers (under conditions of air oxygen access, storage temperature 28°C)

The period of induction of sunflower oil oxidation according to ACh data is 70 days. The first

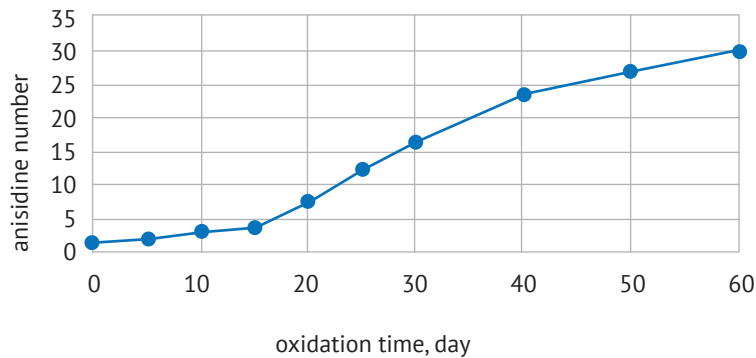
manifestations of bitterness were noticed at AX=8.88 after 80 days of oxidation.



**Figure 3.** Kinetics of oxidation of linseed oil according to the change in anisidine numbers (under conditions of air oxygen access, storage temperature 28°C)

The induction period of linseed oil oxidation according to ACh data is 6 days. The first manifestations

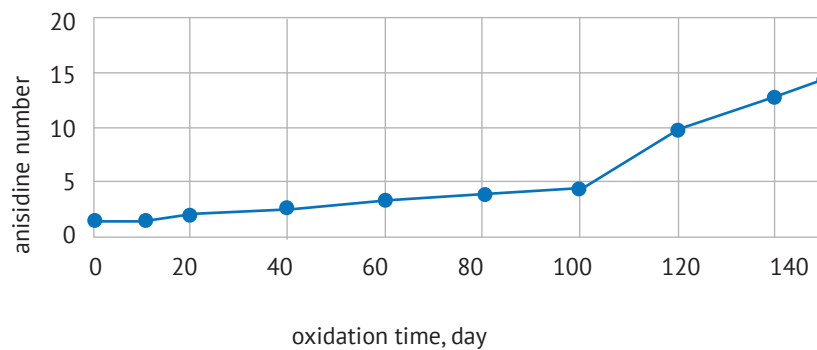
of rancidity were noticed at AX=6.51 after 7 days of oxidation.



**Figure 4.** Kinetics of rapeseed oil oxidation according to the change in anisidine numbers (under conditions of air oxygen access, storage temperature 28°C)

The period of induction of rapeseed oil oxidation according to ACh data is 18 days. The first

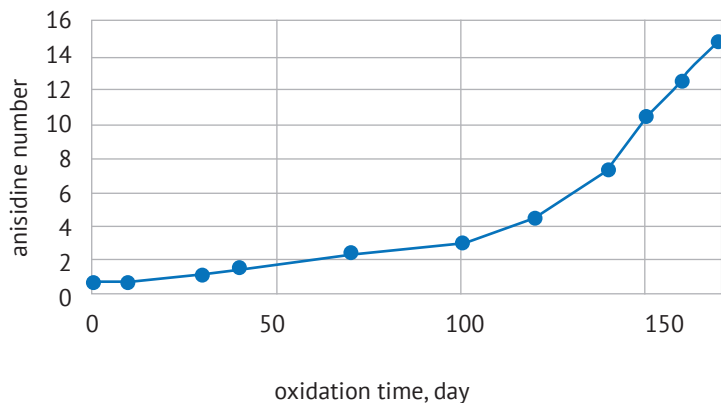
manifestations of bitterness were observed at AX=7.43 after 20 days of oxidation



**Figure 5.** Kinetics of oxidation of corn oil according to the change of anisidine numbers (under conditions of air oxygen access, storage temperature 28°C)

The period of induction of rapeseed oil oxidation according to ACh data is 98 days. The first

manifestations of bitterness were observed at AX=8.48 after 110 days of oxidation.



**Figure 6.** Soybean oil oxidation kinetics according to the change in anisidine numbers (under conditions of air oxygen access, storage temperature 28°C)

The period of induction of rapeseed oil oxidation according to ACh data is 120 days. The first manifestations of rancidity were noticed at AX=10.59 after 150 days of oxidation. No significant bitterness was detected during the investigated time interval (170 days).

A correlation is observed between the moment of exit from the induction period according to the data of anisidine numbers and the beginning of rancidity of all the studied oils. The period of induction of oils according to the anisidine number ends before the deterioration of taste and smell begins (see Fig. 2-6,

Table 4). For example, for sunflower oil, the first manifestations of rancidity appeared after 80 days of storage (Table 4), its induction period (Fig. 2) was 70 days.

It should also be noted that according to the degree of oxidation resistance, based on the change in the peroxide and anisidine numbers, the oils can be arranged in the following series (as stability decreases): soybean oil>corn oil>rapeseed oil>sunflower oil>linseed oil. There is a correlation between the oxidative instability of oils and their content of unsaturated fatty acids.

**Table 4.** The relationship between the value of the anisidine number and the deterioration of the sensory characteristics of the studied oils

Олія	The first signs of bitterness		Significant changes in taste and smell	
	AP, y.o.	Storage time, days	AP, y.o.	Storage time, days
Sunflower	8.88	80	21.42	120
Soybean	10.59	150	-	-
Rapeseed	7.43	20	26.87	50
Corn	8.48	110	14.53	150
Linseed	6.51	7	24.25	16

It should be noted that in all samples there is no sharp increase in AC, which is associated with the low temperature of oil storage and the absence of rapid destruction of peroxides (Fig. 2).

At the end of the studied oxidation interval, the number of aldehydes decreases (Fig. 2-6), which is probably related to their transformation into other oxidation products.

## CONCLUSIONS

According to the set goal, the article investigates the relationship between the formation of the feeling of rancidity in vegetable oils and their content of the main oxidation products – hydroperoxides and aldehydes.

The level of oxidation of fats and oils, as well as the accompanying formation of off-flavors, is ultimately assessed by the senses. However, sensory evaluations are resource-intensive and cumbersome to perform (need to create tasting panels to rule out subjective errors, etc.). To avoid these drawbacks, an array of chemical tests is often used to monitor the progress of lipid oxidation. These include peroxide content (PC), anisidine value (AC), iodine value (IV), thiobarbituric acid (TBA) value, and levels of conjugated dienes (CD)

and hexanal (Hex) produced during oxidation. In order to reduce the number of necessary studies for predicting the moment of the formation of the feeling of rancidity, the article establishes a correlation between the kinetics of oxidation of oils according to peroxide and anisidine numbers and the moment of deterioration of their organoleptic indicators. It has been proven that the content of hydroperoxides does not affect the formation of the feeling of bitterness - even with high peroxide numbers of the studied oils, they did not change their organoleptic characteristics. It has been proven that using the kinetics of the change in the anisidine number, it is possible to predict the moment of rancidity of oils. Bitterness occurs after exiting the induction-period according to the kinetics of anisidine numbers.

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## Дослідження зв'язку між кінетикою окиснення та погіршенням сенсорних характеристик рослинних олій

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**Анотація.** У статті досліджено можливість передбачення появи відчуття згіркнення за основними показниками окисненості олій – пероксидними та анізидиновими числами. Під час окиснення при 28°C та доступі кисню олії накопичували суттєві кількості гідропероксидів (до 160-180 ммоль/20/кг), однак не з'являлось відчуття згіркнення. Доведено, що по кінетиці окиснення олій за даними анізидинових чисел можливо передбачення моменту погіршення органолептичних показників олій

**Ключові слова:** окиснення, згіркнення, рослинні олії, анізидинове число, альдегіди, пероксидне число, гідропероксиди.