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Abstract. Processing oil seeds with a modified fatty acid composition requires the development of new ways to control quality indicators. One of the solutions is express control of the mass fraction of oleic acid in oil of sunflower seeds using the NMR method. Production tests of this method showed the necessity for temperature control of samples before analysis for a long time, if the sample of temperature is less than 15 ° C, which significantly increases the analysis time. The article presents data of changing in the weighted average time of spin-spin relaxation of protons contained in sunflower seeds with different mass fractions of oleic acid in seed oil from their temperature. It was found that a decrease in the sample temperature by 1 ° C leads to a decrease in the weighted average time of spin-spin relaxation of protons seeds with a different mass fraction of oleic acid by 2.3 mc. To reduce the time of thermostating of the sample, we proposed the method of SHF-treatment of sunflower seeds. During the tests we identified the optimal energy of SHF-treatment (in our case it was 360 W) for the test sample of 100 g weight. The selected weight of the sample can significantly reduce the influence of the factor of different quality of samples and is minimal to obtain comparable results. SHF-treatment allows uniform heating of samples without evaporation of moisture from their surface and without changing the internal structure of the sample.

1. Introduction

Modern trends of healthy nutrition and modern technologies in the field of food production put forward new requirements for the characteristics of plant materials. This stimulates the work on the creation of modern varieties and hybrids of agricultural plants, significantly differing in chemical composition and properties. These differences may be quantitative and qualitative in nature. So, an example in the oil and fat industry is researching works aimed at increasing the content of oleic acid in sunflower seed oil [1-4].

Depending on the method of formation of consignments, conditions and natural features in oil seeds, the content of the main fatty acid can vary significantly which affects its technological properties and, accordingly, the price.

With this in mind, the processors are faced with the issue of introducing new methods for monitoring the quality indicators of oil seeds, which are subject to the following basic requirements: ease of measurement, accuracy and elimination of the use of chemicals.

The specified requirements are fully consistent with the methods for determining quality indicators based on the pulsed NMR method. They are operational, non-destructive, easy to implement, do not require the use of toxic organic solvents, do not depend on the subjective characteristics of the operator, and also do not require complex sample preparation. The methods for assessing quality indicators developed when using the pulsed NMR method allow determining the quantitative and qualitative composition of heterogeneous systems [5, 6].

A team of authors of the All-Russian Scientific-Research Institute of Oilseeds, Krasnodar, developed a comprehensive system for determining the quality indicators (oiliness and moisture) of oilseeds and their processed products based on the pulsed NMR method. An integrated system for determining quality indicators is operated at more than 330 enterprises of the oil and fat industry in Russia and the CIS countries. The development of an integrated system is aimed at improving existing methods for determining the quality indicators of oilseeds and development of the new ones.

Currently, the method for determining the content of oleic acid in oil of sunflower seeds has been developed and is being introduced [7].

The developed method is based on the dependence of the NM-relaxation characteristics of protons in the analyzed sample on the content of oleic acid in seed oil, while the measurement of these characteristics is carried out at a temperature of 23 ± 0.2 ° C. However, the main acceptance of sunflower seeds at oil and fat enterprises is carried out in the autumn-winter period, when the temperature of the seeds is much lower that leads to an increase in the error in measuring the content of oleic acid in seed oil. To eliminate the measurement error, thermostating of the seed sample is required for at least 2 hours. This, in turn, significantly reduces the efficiency of the process of controlling the quality of seeds received for processing and does not allow purposefully regulating the technological regimes of their processing.

One of the solutions to this problem is thermostating of samples of the analyzed sunflower seeds before measurements by processing them with ultra-high frequency currents (microwave processing), which is widely used in the food and processing industries [8, 9].

The aim of the study is to develop a method of thermostating a sample of sunflower seeds using microwave processing before measuring the oleic acid content in oil of sunflower seeds using a pulsed NMR method.

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2. Materials and methods

As objects of the research we selected samples of sunflower seeds grown in the Krasnodar Territory, Rostov and Voronezh Regions.

The moisture content and oil content of the studied seed samples were determined according to the State Standard (GOST) 8.597-2010 [10], and the oleic acid content in the seed oil (average value of 10 samples taken from one sample) due to the developed method [7] using the impulse AMV-1006M (VNIIMK, Russia).

Table 1 shows the quality indicators of the studied samples of sunflower seeds.

We identify the effectiveness of temperature control of sunflower seed samples using SHF-treatment in SHF-oven with maximal 700 W (brand LGMS2021N).

Moreover, to determine the optimal temperature control parameters, the time and power of the SHF-treatment varied.

Operational control of the temperature of the seed sample was carried out using an IR pyrometer (OPTRIS MS, Germany).

The weight of the seed sample used for SHF-treatment corresponded to 100 g that ensures uniform temperature control, eliminates the effect of seeds of different quality.

Table 1. Quality indicators of the studied samples of sunflower seeds

Sample of sunflower seeds	Quality traits, %		
	oil content	moisture	oleic acid content
1	53.5	5.5	38
2	43.3	12.3	45
3	45.7	11.4	92
4	49.2	7.0	78
5	38.7	12.7	30
6	51.4	7.8	82
7	46.3	6.5	87
8	48.6	5.9	67
9	50.0	6.7	73
10	47.1	7.2	60
11	39.9	5.9	53
12	52.4	6.6	41
13	48.3	8.4	89
14	47.6	8.1	85
15	50.2	7.7	79

3. Results and Discussion

The analytical parameter for determining the oleic acid content in sunflower seed oil in accordance with the developed method is the measured value of the weighted average time of spin-spin relaxation of proton oils ($T_{2av.wei}$) [7].

One of the main factors influencing the value of the analytical parameter is the temperature of the seeds. With this in mind, we studied the effect of temperature on $T_{2av.wei}$ of protons in oil of seeds with different oleic acid contents – 38, 67 and 92%.

The results are presented in Figure 1.

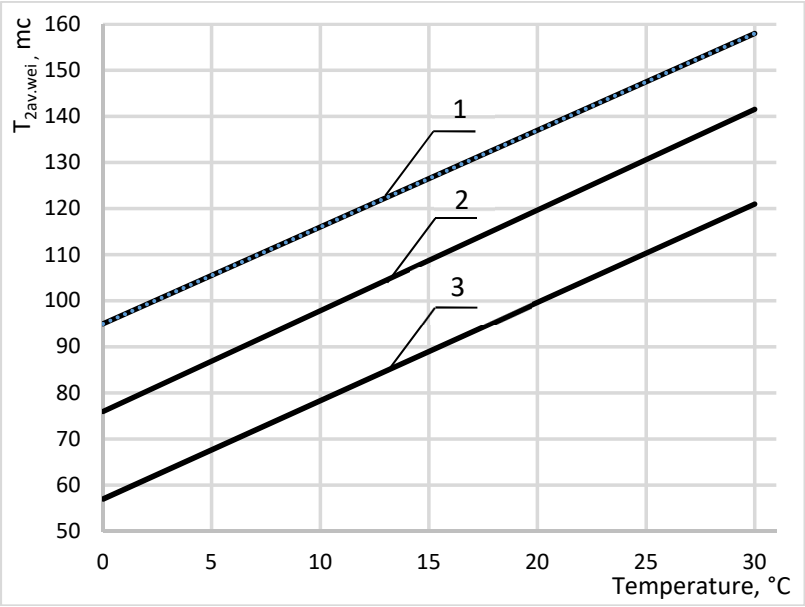

Figure 1. The effect of temperature on the $T_{2av.wei}$ value of protons of seed oil with different oleic acid contents: 1 - 38%; 2 - 67%; 3–92%.

From the data presented in Figure 1 that showed a direct proportional dependence is observed between the temperature of the sample and the value of $T_{2av.wei}$ protons of oil. This explained by the temperature of the seed sample decreases, the mobility of the molecules contained in it decreases. A change in sample temperature by 1 ºC leads to a change in the value of $T_{2av.wei}$ by 2 mc. To exclude the influence of temperature on the result of measuring the oleic acid content, it is necessary to analyze a sample of the sample with a temperature of 23 ± 0.2 ºC.

Figure 2 presents data in the form of a diagram in order to determine the time required for temperature control of a seed sample to a temperature of 23 ° C, depending on its initial temperature.

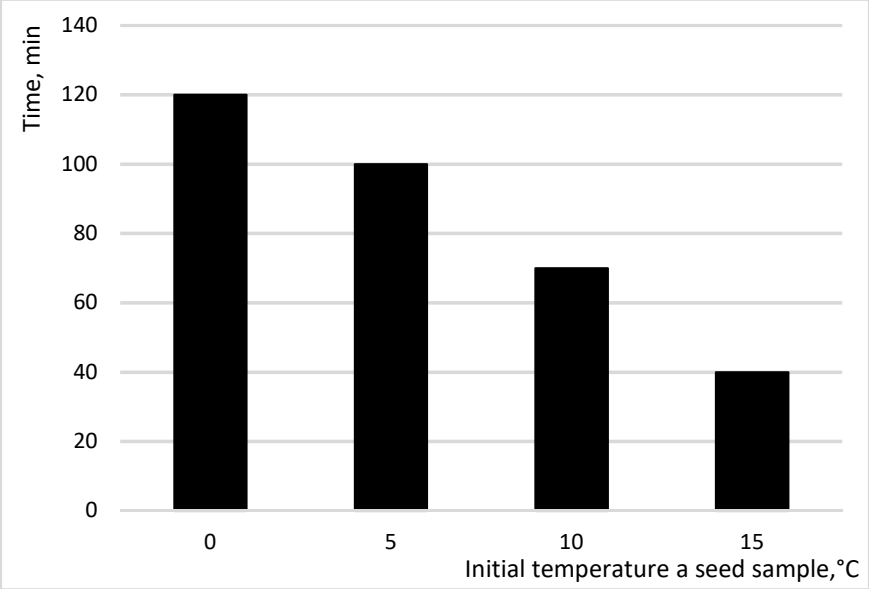

Figure 2. The time required for temperature control of a seed sample to a temperature of 23 ºC, depending on its initial temperature

From the data presented in Figure 2, it can be seen that thermostating of a seed sample using a classical thermostat is a rather lengthy process, which is explained by the low thermal conductivity of the husk of the seeds. So, for example, for thermostating of a sample of seeds weighing 100 g with an initial temperature of 0 ºC, 120 minutes will be required. This makes it difficult to use this method for the purpose of express input control of the content of oleic acid in sunflower seed oil.

To reduce the time of thermostating of the seed sample, it is proposed to use SHF-treatment. The advantage of this method is that the heating is carried out over the entire volume of the sample and is applicable to seed samples with different humidity.

Preliminary experiments found that the most effective is SHF-treatment of a seed sample weighing 100 g at a power of 360 W, since this mode ensures its uniform heating. In addition, there is no loss of moisture in the sample that allows the use of the SHF-treatment regime when preparing seeds to determine moisture and oil content using a pulsed NMR method.

To apply SHF-treatment to determine the oleic acid content in sunflower seed oil, it is necessary to determine the processing time to prevent overheating of the sample. For this purpose, the prepared samples with 100 g seed weight were thermostated to a temperature of 0 ºC. Then they were subjected to microwave processing, determining the temperature and time required to achieve the required temperature of 23 ° C.

Figure 3 shows the dependence of the time of SHF-treatment of the seed sample until the temperature reaches 23 ºC from their initial temperature.

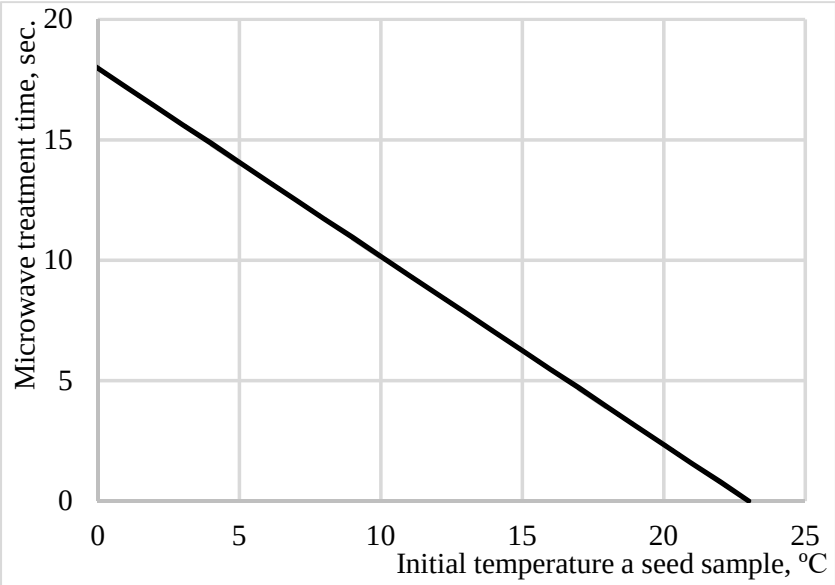

Figure 3. Dependence of the time of SHF-treatment of a seed sample to a temperature of 23 ºC on the initial temperature

It was found that between the time of SHF-treatment of the seed sample, necessary for its heating to a temperature of 23 ° C, and the initial temperature of the seeds, a linear relationship is observed with a high correlation coefficient equal to 0.988.

In addition, during SHF-treatment of a seed sample weighing 100 g during 1 sec, the temperature of the sample rises by 1.3 º regardless of the moisture content, oil content and oleic acid content in the seed oil.

Verification of the developed method for thermostating of sunflower seeds using SHF-treatment was carried out as follows. A 100 g sample was taken from a prepared seed sample, cooled to a temperature of 0 ºC. After that, in accordance with the proposed methodology and schedule shown in Figure 3, SHF-treatment was performed (energy 360 W and time 18 sec). Then, in the prepared sample, the presence of weed impurities was checked and the content of oleic acid was measured using a previously developed and patented dosing device. This allows you to reduce the influence of the volume factor of the analyzed sample [11]. The measurement results are presented in Table 2.

We should note that the samples of sunflower seeds used for the study were selected in the conditions of production laboratories. This explains the large coefficient of variation (different quality) of the mass fraction of oleic acid in the oil of sunflower seeds within one analyzed sample, which reaches 8%. Different quality can be explained by the method of formation of commodity consignments of seeds and their biological features.

From the data presented in Table 2, it can be seen that the developed technique allowed one to significantly reduce the thermostating time of cold samples of sunflower seeds without increasing the measurement error of the analyzed quality indicator. Moreover, the deviations of the measured values of the oleic acid content in the oil of sunflower seeds do not exceed the specified errors for the developed express method for determining the oleic acid content in the oil of sunflower seeds using the pulsed NMR method.

Table 2. The results of measuring the content of oleic acid using the developed method of temperature control of sunflower seeds

Sample	Oleic acid content in testing set, %										Middle meaning	Certified meaning	Δ, %
	1	2	3	4	5	6	7	8	9	10			
1	35	34	40	41	38	37	36	38	37	36	37	38	1
2	45	43	46	47	45	46	46	47	42	43	45	45	0
3	90	93	90	91	92	93	92	90	91	90	91	92	1
4	78	83	80	81	78	75	82	77	80	81	80	78	-2
5	25	30	30	31	32	32	33	34	31	30	31	30	-1
6	76	78	82	89	80	78	76	75	79	80	79	82	3
7	87	88	87	82	86	90	92	88	90	92	88	87	-1
8	70	66	68	70	72	64	65	68	62	65	67	67	0
9	70	75	76	74	74	78	74	71	74	76	74	73	-1
10	62	55	54	60	64	58	56	61	60	54	58	60	2
11	45	53	57	56	59	55	54	58	54	56	55	53	-2
12	38	42	45	42	41	41	40	43	45	41	42	41	-1
13	89	84	89	87	90	91	90	91	90	92	89	89	0
14	85	84	86	84	85	80	82	81	83	81	83	85	2
15	80	79	78	76	74	76	79	82	83	76	78	79	1

4. Conclusion

In the course of the studies, it was found that in order to reduce the thermostating time of seed samples, it is possible to use SHF-treatment. SHF-treatment allows reducing the thermostating time of sunflower seeds from 2 hours to 1 minute. That allows you to save the main advantage of the developed method for determining the content of oleic acid in oil of sunflower seeds, using the pulsed NMR method, its operational nature.

The use of SHF-treatment can reduce the influence of the temperature factor on the results of measuring the oleic acid content in the seed oil with insufficient temperature control of the analyzed samples. In addition, due to the peculiarities of SHF-treatment and the minimum processing time, moisture loss does not occur, which is especially important in the process of a comprehensive determination of the quality indicators of sunflower seeds.

The developed technique consists in following. A sample weighing 100 g is taken from the analyzed seed sample and its temperature is measured. Depending on the initial temperature, the required SHF-treatment time is determined from the calibration schedule (Figure 3) (when using another microwave oven, it is necessary to construct a similar calibration schedule). After that, sample preparation and measurement of oil content and humidity are carried out in accordance with State Standard (GOST) 8.597-2010 while measuring the oleic acid content in sunflower seed oil according to the developed method.

Further research is focused on the development and implementation of metrological support for the developed express method for determining the oleic acid content in sunflower seed oil and its implementation at oil and fat enterprises [12].

Due to the widespread use of SHF-ovens, this method is easy to implement in the conditions of production laboratories. It should be borne in mind that in domestic SHF-ovens, power can vary widely depending on the purpose and manufacturer. Accordingly, it is necessary to pre-calibrate the SHF microwave oven to select the optimal operating mode.

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