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# INDUSTRIAL TRIALS OF THE REFINING TECHNOLOGY FOR LONG-TERM STORED SUNFLOWER OIL

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**Abstract:** The article discusses the results of a refining technology for processing long-stored sunflower oil. After adding a filtering agent in the amount of 0.1-0.3% to the same oil samples, an increase in the oil filtration rate from 0.42 to 1.63 kg s/m<sup>2</sup> was achieved during filtration. Simultaneously, the color of the oil decreased from 14 mgJ to 8 mgJ. It can be observed that the acid value of the oil decreased from 0.17 to 0.15 mg KOH/g, while the yield increased from 98.1% to 99.2%. The results indicate that the developed technology and filtering agent are highly effective in processing oils that have been stored for a long time. In our research, based on this technology, we conducted pyrolysis of sunflower husk at 200°C in an oxygen-free environment for 120 minutes. The experiment was carried out using laboratory equipment for the thermal pyrolysis of sunflower husks.

**Keywords:** refining, polished, sunflower, vegetable oils, glycerin, pyrolysis, concentration, filtering agent, acid value, and others.

**Introduction.** The oil and fat industry is one of the main branches of the food industry in the Republic of Uzbekistan, supplying the population and national economy with refined vegetable oils, fats, as well as products made from them such as margarine, mayonnaise, glycerin, fatty acids, and soap. It is known that the oil and fat industry accounts for nearly 40% of the food products manufactured in our country.

Nowadays, new technologies for packaging products into convenient and visually appealing containers for production, processing, consumption, and storage are being widely implemented. Work continues automating technological processes at oil and fat enterprises and equipping them with machinery from foreign companies.

The primary objective of our republic's oil and fat industry is to produce environmentally friendly, competitive, high-quality products for the population. [1].

Vegetable oils are mainly obtained from sunflower, cottonseed, mustard, oilseed flax, hemp, sesame, poppy, safflower, walnut, almond, bean, peanut, soybean, olive fruit, and others. Among these, the most important edible oils include sunflower oil, cottonseed oil, olive oil, sesame oil, and others.

Valuable products such as glycerin, carboxylic acids, and salts of carboxylic acids – soap, as well as technical salomas, are obtained from fats. [2].

As we know, sunflower is the most widely distributed oilseed crop in the world and belongs to the Asteraceae family. On a global scale, sunflower oil ranks second as an edible vegetable oil, following soybean oil. However, one of the main drawbacks of sunflower seeds is the difficulty of storage. In particular, it is necessary to dry the moisture in the seed coat, as it contains 3-4% more water than the kernel.

Thus, sunflower seeds contain 29-56% oil and 15% protein. The oil composition includes up to 62% biologically active linoleic acid, as well as vitamins A, D, E, K, and phosphatides. In addition to being used as food, sunflower oil is also applied in medicine, cosmetology, perfumery, and the construction industry. It is a clear, pale yellow liquid, with sunflower seeds containing 29-56% oil and 15% protein.

Based on this, during the processing of various types of oilseeds and oil-bearing fruits, their shells and seed coats are separated from the main oil-containing tissue – the kernel. This process increases the oil content of the raw material being processed, enhances the productivity of technological equipment and devices, and improves the quality of both oil and protein. [3]

Thus, each type of oil obtained from high-quality raw materials has a distinct taste and aroma, which may change over long-term storage. Such changes usually occur due to the formation of new specific compounds from acylglycerols and the partial loss of natural flavor and aroma compounds. These alterations in the taste and smell of oils can sometimes render them unfit for consumption. This process is known as the deterioration of edible oil (or fat). Nutritional deterioration typically develops gradually: initially, the oil may cause a slight scratching sensation in the throat when tasted, and in some cases, it may produce a characteristic “burning” sensation. Over time, an unpleasant taste develops, which is characterized by certain distinct features. This off-flavor is usually perceived slightly earlier than the emergence of an unpleasant odor.

There are several main and widespread types of oil deterioration, which can be chemically identified based on the accumulation of free fatty acids, peroxides, as well as rancidity and solidification. The deterioration of oils is primarily characterized by the accumulation of specific chemical compounds in their composition.

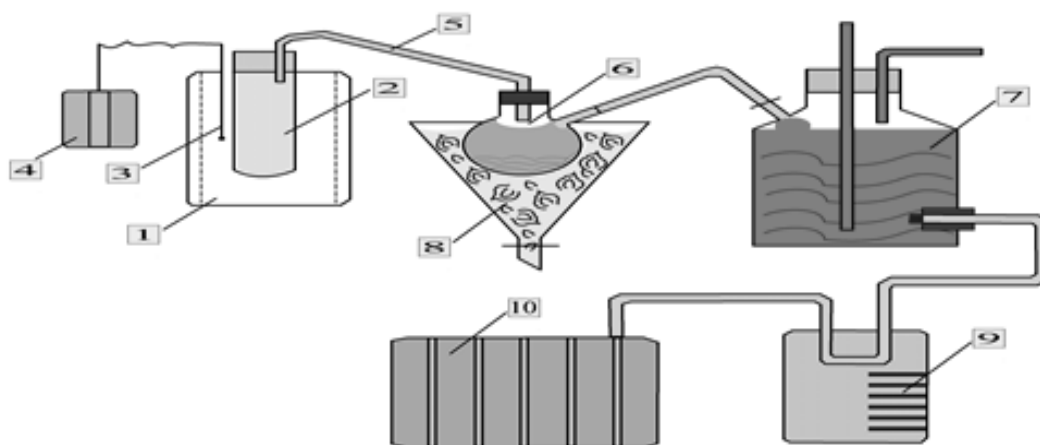
The type of oil deterioration depends on its fatty acid composition and storage conditions. These two factors significantly influence the intensity and rate of oil degradation. If oil deteriorates under the influence of dioxide, the speed of this process will also depend on the presence of oxidation accelerators or antioxidants in the oil.

As we know, the total composition of peroxide compounds includes less active peroxide substances, such as dialkyl peroxides. The reason for the low peroxide value in deteriorating oils is that the active part of peroxide compounds rapidly transforms into other substances that do not contain peroxide oxygen.

The composition of peroxides in rancid oils is not uniform. It includes substances that react almost instantly with hydroiodic acid, primarily hydroperoxides. The importance of determining peroxide compounds in oils lies in the fact that they serve as specific indicators of oil deterioration caused by the oxidation of acylglycerols under the influence of dioxide, as well as other contributing factors.

Research Methods. Although natural sunflower husk has a relatively high porosity (73.2 cm<sup>3</sup>/g), it does not have the ability to fully absorb harmful substances. To achieve the desired results, researchers developed an activation technology primarily based on the pyrolysis of the husk. In our research, we based our methodology on this technology and conducted pyrolysis of the husk at 200°C in an oxygen-free environment for 120

**minutes.** The laboratory equipment used for the thermal pyrolysis of sunflower husk is shown in the following figure. [5].



**Figure 1. Laboratory apparatus for the thermal pyrolysis of sunflower husk.**

(1 - Vertical furnace with electric heater, 2 - Reactor, 3 - Thermocouple, 4 - Millivoltmeter, 5 - Glass tube, 6 - Wurtz glass flask, 7 - Gas collector, 8 - Ice-cooled condenser, 9 - Measuring container, 10 - Gas analyzer)

**Results and Discussion.** In our previous research, we developed a refining technology for polishing long-stored cottonseed and sunflower oils. According to this method, oils that had exceeded standard acidity and peroxide values during storage were refined using a **250 g/L caustic soda solution**, setting the excess alkali coefficient at **0.5 units** to reduce quality indicators to standard requirements. However, during the process, the **fine soapstock particles** formed coagulation centers, leading to challenges in **soapstock precipitation and oil filtration** [5,6].

To validate the results, industrial trials were conducted by applying this technology to **5,000 kg of refined sunflower oil** that had been stored in factory tanks for **270 days** and required re-refining.

2. The sunflower oil used for industrial trials had the following characteristics:

Indicators	Quantity
Acid value of the oil, mg KOH/g	0,52
Peroxide value, $\frac{1}{2}\text{O}_2$ mmol/kg	18,4
Moisture content, %	0,18
Color, according to the iodine scale, mg J	23
Odor	Characteristic of sunflower oil, with the presence of oxidation products' odor and taste.
Taste	

The authors have developed a **refining technology for polishing long-stored cottonseed and sunflower oils**. According to this method, oils that exceeded standard acidity and peroxide values during storage were refined to the required level using a **250 g/L caustic soda solution**, setting the excess alkali coefficient at **0.5 units** to determine the optimal refining conditions. The above-mentioned oils were refined using this method. Based on the **acid value and color** of the



oil, **caustic soda with a concentration of 250 g/t** was used, and the excess alkali amount was calculated with a coefficient of **1.5**, resulting in a consumption of **0.56 kg/t**. Neutralization was carried out under these conditions. The **neutralization temperature** was **27°C at the beginning** of the process and reached **55°C at the end**. To facilitate **soapstock coagulation**, **2 kg/t of water** was added. The obtained results are presented in the following table.

Indicators	Quantity
Acid value of the oil, mg KOH/g	0,17
Peroxide value, $\frac{1}{2}\text{O}_2$ mmol/kg	6,4
Moisture content, %	0,19
Color, according to the iodine scale, mg J	14
Odor	Characteristic of sunflower oil, odorless and tasteless.
Taste	

As can be seen, the indicators of the polished refined oil were successfully reduced to meet standard requirements. However, the color indicator was 4 units higher than the required level. The research conducted by the authors and the results obtained under these conditions showed that the soapstock particles formed were very small and tended to remain suspended, resulting in a lower filtration rate than usual.

The oil that underwent polishing refining was treated with a filtration agent developed by the authors, which possesses bleaching properties.

In this process, the authors proposed a composition consisting of inorganic natural minerals, including opoka-like clays, which exhibit selective effects and possess sorption properties distinct from traditional adsorbents used in the oil and fat industry. The authors thermally activated the opoka-like clays to obtain a fraction composed of meso- and macropores with a relative surface area of up to 120 m<sup>2</sup>/g and an average adsorptive pore size of approximately 120 Å. Additionally, to create a structure with smaller pore sizes, they conducted pyrolysis of crushed sunflower husk fractions (1–2 mm) in an oxygen-free environment at a temperature of 180–200°C. This process yielded an adsorbent composition with micropores having a relative surface area of up to 58.72 m<sup>2</sup>/g and an average adsorptive pore size of less than 33.1 Å. The authors prepared a composite of these adsorbents and proposed it as a filtering material.

To experimentally verify the above-mentioned process, filtration was carried out using a pre-prepared filtration agent. To assess the actual filtration rate in the filter and filter medium, a control sample (alkali-neutralized, newly produced oil) was taken, and at a temperature of 70°C, the filtration rate was determined to be 1.65 kgs/m<sup>2</sup>. Since this condition applied to the initially refined, washed, dried, and adsorbent-treated bleached oil, other parameters were not considered.

The results are presented in the following table:

Experiment	Amount of filtration agent relative to oil mass, %	Oil filtration rate, kgs/m <sup>2</sup>	Oil color, mgJ	Acid value of oil, mg KOH/g	Peroxide value, $\frac{1}{2}\text{O}_2$ mmol/kg	Filtered oil yield, %
Control	-	1,65	-	-	-	-
1	-	0,42	14	0,17	6,4	98,1
2	0,10	0,88	12	0,16	6,1	98,7
3	0,15	1,54	8	0,15	5,6	99,1
4	0,20	1,60	8	0,15	5,1	99,2
5	0,25	1,62	8	0,15	4,9	99,1
6	0,30	1,62	8	0,15	4,8	99,0

As seen from the table, the filtration rate of the oil that underwent polishing refining without adding a filtration agent (Experiment 1) was 0.42 kg/s·m<sup>2</sup> due to the presence of soapstock particles formed during the polishing refining process. However, when filtering the same oil samples with the addition of a filtration agent in amounts ranging from 0.1% to 0.3%, the filtration rate increased from 0.42 to 1.63 kg/s·m<sup>2</sup>. Additionally, the oil's color decreased from 14 mgJ to 8 mgJ. The acid value of the oil decreased from 0.17 to 0.15 mg KOH/g, and the oil yield increased from 98.1% to 99.2%.

**Conclusion.** The results indicate that the developed technology and filtration agent are highly effective for processing long-stored oils. Considering the above, the technology of polishing refining and filtration using filtration agents is accepted for use in the refining process of stagnant and quality-degraded oils in industrial enterprises, based on its proven efficiency.

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