# Purification of Cottonseed Oil Using A Sorbent Obtained from the Fibrous Waste of Natural Silk

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**Abstract:** Natural silk is a very expensive material for the textile industry. Silk mills produce large amounts of fibrous waste from natural silk. It is very important to prepare the necessary materials for industry from the fibrous waste of silk. We obtained a multifunctional sorbent from silk fiber waste by various methods to purify vegetable oils. The resulting sorbent was used to purify cottonseed oil instead of bentonite-based sorbents. As a result of the purification of cottonseed oil using sorbent samples from silk fiber waste, we were able to reduce the oil peroxide value from 11 mmol/L to 3,9 mmol/L. Reducing the peroxide value of vegetable oils increases the shelf life of vegetable oils. It is possible to reuse 2-3 times in the purification of vegetable oils by reactivating the sorbent obtained from the fibrous waste of silk, and the sorbent does not affect the composition of the oil, even if it is in the oil environment for a long time. A sorbent derived from the fibrous waste of silk we recommend is used to reduce the waste of silk mills and oil mills.

**Keywords:** Natural silk, hydrolyzed fibroin ("HF"), very high frequency rays, vegetable oil, bentonite, peroxide value

#### Introduction

The vegetable oils industry is one of the leading sectors of the food industry. The vegetable oils industry accounts for a large share of the world's food production. The main products of the industry are cotton, flax, sunflower, soybean oil and others. The main task of the vegetable oils industry is to produce environmentally friendly, competitive high-quality products.

Various adsorbents, mainly soils and silicates, are currently used to purify vegetable oils. They use a lot of energy and cost to clean the vegetable oil. To purify vegetable oils using bentonites, they are activated at high temperatures (250-300°C) with acids. However, if the oil stays with the adsorbent for more than the specified time, the oil will oxidize and take on an earthly odor, changing the structure of the oil [1]. Solving this problem is one of the most pressing issues. To solve this problem, we used sorbents derived from silk fiber waste.

It is important to study the physical and mechanical properties, chemical properties and application of natural silk fibroin. Today, various preparations based on natural silk fibroin have been developed and used in many fields[2-6]. Natural silk fibroin has sorbent properties, and its sorbent properties have been studied. Hydrolyzed fibroin ("HF") from fibrous waste of natural silk can be used in the purification of vegetable oils, in the food industry, in pharmacology.

In the process of obtaining silk fiber from cocoons, 10-28% of silk fibers are converted into waste. Depending on the production capacity of the silk spinning mill, one silk spinning mill in Uzbekistan produces an average of about 60 tons of silk waste per year. It also takes a lot of energy to separate the waste fibers from the additives and separate the silk fibers. Finding ways to use this fibrous waste is one of the key issues. Silk fiber waste is considered unsuitable for the textile industry in terms of mechanical and quality characteristics. However, the chemical composition of silk waste and silk fibers are the same, and their physicochemical properties are similar. This allows to obtain hydrolyzed fibroin with high sorption properties from the fibrous waste of silk and to solve various problems in its practical application[7].

Silk contains fibroin and sericin proteins, the amount of which depends on the diet and living conditions of silkworms. As a result of our research, we found that silkworm cocoons grown in the Khorezm region of Uzbekistan contain 67.5% fibroin, 32.5% sericin and other substances. 25% of silk cocoons are used in yarn quote suitable for the textile industry, 28% are converted into fibrous waste, 47% are composed of other types of waste.

# **Materials and Methods**

# **Required materials and equipment**

Fibrous waste of silk (Cleaned of additives. Khorezm ipagi LLC, Urgench, Uzbekistan), Sodium carbonate (purity 99,9%), HCl(chemically pure), KOH(purity 99,9%) was purchased from Chimreaktivinvest (Uzbekistan), unrefined oil obtained from cotton seeds. Bidistilled water is obtained from the "GFL 2104 Double distillation water still" device (Germany). Optical microscope ("Optika\_B-150 DBR"), Lovibond® Tintometer Model F.

# **Obtaining fibroin fiber from fibrous wastes of natural silk**

Studies have focused on the use of silkworm waste as a sorbent in the purification of vegetable oils. The fibrous waste of silk was found to contain up to 88% fibroin, up to 12% sericin and other substances. Conditions were selected for the obtaining of fibroin from the fibrous waste of silk: sericin was dissolved in a Na<sub>2</sub>CO<sub>3</sub> solution at 90°C for 40 min. Fibroin fibers were obtained from the solution and washed with distilled water.

# **Obtaining "HF" from silk fibroin**

Subsequent studies have been performed on the conversion of fibroin fibers from silk fibers into a powder to increase their sorbent properties. Silk fiber fibroin was

hydrolyzed under acidic and alkaline conditions, thermally (90°C) and very highfrequency rays (340-850W) to obtain a "HF" in the form of a powder. Conditions for obtaining "HF" in alkaline and acidic media by thermal methods were studied [9]. In this case, the thermal method took 12 min to obtain "HF" from silk fibroin in a 3% KOH solution in an alkaline environment. Under the influence of very high-frequency rays, "HF" was obtained in an alkaline medium for 7 min.

The formation of cracks and pores on the surface of "HF" particles has been studied. The fact that "HF" has ampholytic properties and the presence of multifunctional groups allows it to be used in a variety of environmental conditions. In order to further increase the cracks and porosity in the "HF" particles, the "HF" powder was immersed in an alkaline environment and exposed to very high-frequency rays for 5 min in the wet condition. After the process, the sample was neutralized and washed with hot distilled water until no salt remained. As the cracks and pores in the "HF" increase, its sorption properties increase.

# Purification of cottonseed oil with "HF"

Cottonseed oil was purified to test the sorption properties of "HF" samples obtained by alkaline and acidic conditions under thermal and very high-frequency rays. 100 g of purified vegetable oil was poured into 5 flasks numbered (I-V) and immersed in a water bath. When the temperature reached 60°C, 0,5 g of bentonite and hydrolyzed fibroin ("HF") samples obtained by various methods were added to the flasks and mixed at a speed of 250 rpm. Continue stirring for 20 min, bringing the oil temperature to 90°C. At the end of the process, the oil is filtered. The color level of the filtered oil was measured on a Lovibond device. Peroxide count, acid count, and moisture were measured by the method [10].

#### **Results and Discussion**

#### Average size of "HF" particles

"HF" was obtained from natural silk under the influence of very high frequency rays of different powers. The average size of the obtained "HF" particles was determined under the microscope "Optika\_B-150 DBR".

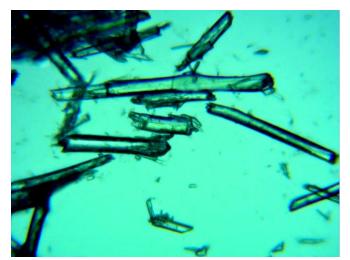


Figure. 1. HF obtained in an acidic environment by acidic method

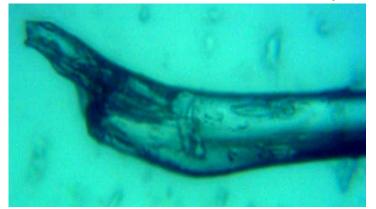


Figure. 2. HF obtained under acidic conditions under the influence of very high frequency rays

# Table 1. Dependence of time and yield on obtaining "HF" powder under theinfluence of very high frequency rays

Nº	Power (W)	Hydrolyzing reagent	Time spent on the	Product yield (%)
1	340	3%	process 20 min	75
2	510	3%	15 min	72
3	850	3%	10 min	60

 Table 2. Changes in the average size of particles of "HF" under the influence of very high frequency rays

No	Power (W)	The smallest particle size (µm)	The largest particle size (µm)	The average size of the particles (µm)	Product yield (%)
1	340	22,89	179	90	75

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2	510	20	214	81	72
3	850	18	207,5	80	60

We selected 510W as the optimal very high-frequency rays power, taking into account the "HF" formation time and process efficiency.

The smallest size of "HF" particles obtained in the very high-frequency rays effect with a power of 510W was 20  $\mu$ m, while the largest one was 214  $\mu$ m and the average size of the particles was 81  $\mu$ m. Due to the transition of silk fibroin from the fibrous condition to the powder-like condition of "HF", on the surface of the particles of "HF", there is an increase in the active polyfunctional micro and nano-sized pores. Due to the presence of active polyfunctional porosity, we will be able to use "HF" as a polyampholyte sorbent.

#### Indicators of refined cottonseed oil

Pakistani bentonite and hydrolyzed fibroin("HF") samples were used in the purification of cottonseed oil ("HF" obtained under I-thermal acid conditions, "HF" obtained under II-thermal alkaline conditions, "HF" obtained under III-very high-frequency rays under alkaline conditions, IV-very high-frequency rays under alkaline conditions. The resulting powder is processed in the wet condition "HF"). The following table shows the cottonseed oil purification performance of "HF" samples.

Parameters	Unrefined oil indicators	Oil indicators refined with Pakistani bentonite used in Urgench vegetable oil production plant	Oil refined with sample I	Oil refined with sample II	Oil refined with sample III	Oil refined with sample IV
	Yellow	Yellow	Yellow	Yellow	Yellow	Yellow
Color level	unit:35	unit:35	unit:35	unit:35	unit:35	unit:35
	Red: 17	Red: 14	Red: 14	Red: 13	Red: 13	Red: 11
	Blue: 5	Blue: 4	Blue: 4	Blue: 4	Blue: 4	Blue: 4
The						
amount of	0,4	0,19	0,26	0,26	0,26	0,27
acid						
Peroxide	11	6	6	6,5	6,5	3,9

Table 3.

value						
Spark tempera- ture	Not available	Not available	Not available	Not available	Not available	Not available
Humidity level	0,1	0,05	0,06	0,06	0,06	0,055

The table shows that the color level of cottonseed oil obtained after purification with "HF" samples from natural silk fiber waste decreased from 17 red units to 11-13 red units. The acid content decreased from 0,4 mmol/L to 0,26 mmol/L, and the peroxide content decreased from 11 mmol/L to 3.9-6,5 mmol/L. The moisture content of cottonseed oil also decreased from 0,055 to 0,06.

In the table, the values of refined cottonseed oil in samples I, II and III of "HF" are almost the same as those of bentonite imported from Pakistan used in Urgench yogmoy JSC. It was found that the refined cottonseed oil in the sample IV had a significant reduction in oil peroxide content from 11 mmol/L to 3,9 mmol/L compared to bentonite imported from Pakistan. The moisture content of the oil dropped to 0,055. The reason for such a high sorption property in the sample of HF in the wet state in the alkaline medium treated with VHF rays is the formation of many multifunctional group pores and cracks on the surface of HF particles under the influence of VHF rays and the increase in the sorption surface area. This can be seen in the images taken with the help of an optical microscope (Fig.1). As can be seen from the figure, the particle size of the thermally treated "HF" samples is 65  $\mu$ m and no pores or cracks are observed on their surface (Fig.1). The particle size of "HF" samples treated with very high-frequency rays is 81  $\mu$ m, and pores and cracks are observed on their surface (Fig.2).

#### Conclusion

In conclusion, in the use of soil-containing sorbents in the purification of oils, that is, bentonite-if the bentonite is held more than the norm with the oil, the oil is oxidized and tastes like earth. When fibrous silk sorbents, ie "HF" derived from silk fiber waste, are used in oil refining, the "HF" sorbent does not affect the composition of the oil, even if it is mixed with cottonseed oil for a long time. This is explained by the fact that the "HF" sorbent reduces the peroxide content of oil. Decreasing the number of peroxides of "HF" sorbent oil leads to an increase in the shelf life of cottonseed oil.

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