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COLOURING PIGMENTS FROM CRUDE FORPRESS OILS FROM COTTON SEEDS

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Abstract

Vegetable oils contain accompanying substances and impurities of various compositions and properties that affect the quality of the finished product and further processing processes. To remove these substances, a refining process is carried out, in particular adsorption refining using activated carbon, silica gel, zeolite, etc.

This scientific article presents the results of a study on the bleaching processes of cottonseed oils and the fact that the amount of coloring substances in cottonseed oil, namely carotenoids and gossypol, is higher in oils obtained from grades III–IV. It was also found that the mass fractions of chlorophyll and bound gossypol in the oil obtained from a mixture of grades III–IV were higher than in the cottonseed oil obtained from a mixture of grades I and II.

Keywords: *refining, chigiti buttermilk, moya buttermilk, okartirish, caratinoid, gossypol, chlorophyll*

Introduction

Currently, the process of liberalization is being carried out consistently in all sectors and branches of the economy of our country, and economic reforms are being deepened. The absence of adsorbent residues in the oil composition during the bleaching process using adsorbents indicates that the bleaching process has been perfected (Kopeikovsky V. M., Danilguk S. I., Garbuzova G. I., et al., 1982).

The adsorption process is carried out by the absorption of molecules from a liquid or gas onto the surface of a solid substance (adsorbent). The most effective adsorbents for vegetable oils are materials such as activated carbon and silica gel.

Development of new adsorbents: The purity and quality of the oil can be improved by using nanomaterials and bioadsorbents.

Process optimization: Process efficiency can be improved by properly adjusting parameters such as temperature, pressure, and time.

Oil purification technologies: New technologies can be used to purify oil and separate foreign substances from oil during the adsorption process.

The republic processes approximately 1 million tons of cotton seeds annually, from which up to 30% of oil is extracted (of the total production of vegetable oils in the country) and about 1.5 million tons of valuable protein feed meal is produced. Complex processing of cottonseed oil ensures the production of high-quality products – salad oil and palmitin, which can replace sunflower, olive and coconut oils in the country. Salad oil is close in quality and fatty acid composition to refined and deodorized sunflower oil. Palmitin is used in the production of margarine products and toilet soap instead of coconut oil (Korostelev V. M., Maznyak I. E., 1986; Holapek M., Jandera P., Zderadika P., Hruba L., 2003; Khodjaev A. A., Ataullaev A. Kh., 1986).

The color of cottonseed oils obtained from seeds of grades III–IV is also significantly influenced by the protoplasmic pigment localized in plastids – chlorophyll, which, when saponified with alkali, produces reaction products colored brown or yellow (Akhmedov A. N., Abdurakhimov S. A., Azimov Yu. Kh.).

A distinctive feature of cottonseed oils from other vegetable fats is the presence of coloring pigments: gossypol and its derivatives, which are localized in the seed kernel in gossypol glands impregnated with pectin, hemicellulose and other unidentified substances (Akhmedov A. N., Erkaeva N. Ch., 2021).

In order to save expensive highly active bleaching earths for adsorption refining of cottonseed oil containing gossypol, as well as for oils containing significant amounts of chlorophyll, activated carbon is added to the bleaching earth (Tyutyunnikov B. N., Naumenko P. V., Tovbin I. M., Faniev G. G., 1970).

Currently, oil extraction plants use expensive imported adsorbents. Replacing them with local raw materials is a pressing issue.

Thus, cottonseed oils contain a significant amount of free fatty acids, phospholipids and pigments: derivatives and modified forms of gossypol, chlorophylls and coloring agents, unsaponifiable lipids, which worsen the consumer properties of oils during alkaline treatment. The quantity and composition of these accompanying substances depends on the quality of cotton seeds, methods of extracting oils from them, duration and methods of their storage. In oils obtained from low-grade seeds, their quantity is somewhat higher.

The variety of accompanying substances and foreign impurities, the heterogeneity of their chemical composition and properties determines the variety of methods for their removal.

Materials and methods

Qquantitative determination of gossypol was carried out by HPLC (Karishma R., Lakshmi Sahithya U., Suneetha P., et al. 2016); – the content of free gossypol (Abou–Donia S.A., Laches J. M., Abou–Donia M.B., 1981) was determined by high-performance liquid chromatography. A liquid chromatograph Agilent Technologies 1200 series (USA) with a DAD detector was used. Column Ultropac Columen Lihroprep RP18, 5 µn, 2.6 × 100mm. Gossypol was injected in 1.0–1.5 µn acetonitrile and eluted from the column isocratically with a solution consisting of acetonitrile – 0.1% phosphoric acid in water (80: 20) at room temperature. Detection was performed at 254 nm.

Also, in some studies, the mass fraction of free gossypol in oil was determined using the method certified by Certificate No. 188 of the State Standard (Technical Requirements 88.06–27: 2011).

This method for measuring the mass fraction of free gossypol applies to unrefined cottonseed oil and unrefined high-sypol cottonseed oil in the range from 0.50% to 1.5% and uses the aniline method.

The method is based on the reaction of gossypol with aniline and the formation of dianiline-gossypol, which is insoluble in oil and in some organic solvents.

Conducting measurements. A sample of filtered unrefined high-sypol cottonseed oil (10 g) was placed in a conical flask with a capacity of 200–250 cm^3, with a gossypol content in the oil of 0.5–1.5%, and 20–30 g with a gossypol content below 0.5%.

A double amount of petroleum ether, 2 cm^3 of aniline and 2.0–2.5 cm^3 of pyridine were added to the flask. The contents of the flask were shaken, covered with a small funnel and left in a thermostat at a temperature of 40–60 °C for 1 hour.

After this, the flask was closed with a stopper and left in the dark until the dianilingossypol precipitate fell out. Two days after the precipitate fell out, it was separated from the mother liquor by filtration through a crucible with a porous plate No. 3 or No. 4, previously dried at 100–105 °C.

The sediment and the flask were washed with small portions of a mixture of 96% ethyl alcohol and petroleum ether in a ratio of 1: 2.

The crucible was dried in a drying cabinet at a temperature of 100–105 °C to a constant weight and weighed.

The first weighing was carried out after 1 hour, the subsequent weighings after 30 minutes.

Processing of measurement results. The mass fraction of free gossypol in percent (X) was calculated using the formula:

$$X = \frac{P_1 \times 0{,}775 \times 100}{P}; \qquad (1)$$

where: P_1 – mass of dianilingossypol sediment in g;

0.775 – coefficient characterizing the ratio between gossypol and dianilingossypol.

P – weight of unrefined high-sypol cottonseed oil in g.

Results

It is known that the color of dark-colored cottonseed oils, along with the presence of gossypol and its derivatives, is also determined by the content of carotenoids and chlorophylls (Kopeikovsky V. M., Danilguk S. I., Garbuzova G. I. et al. 1982). Moreover, carotenoids appear in the red colors of the resulting cottonseed oil, and chlorophylls – in blue units of oil color.

Taking this into account, we conducted analyses to determine the content of carotenoids and chlorophylls in the composition of raw forepress oils obtained from mixtures of cotton seeds of grades I–II and III–IV. The results of the analyses are presented in Table 1.

Table 1. *Indicators of coloring pigments of raw forepress oils obtained from mixtures of cotton seeds of I–II and III–IV varieties*

Name of oil coloring pigments	Unit of measurement	Cottonseed Oil: I and II grades	Cottonseed Oil: III and IV grades
mass fraction of carotenoids	$\times 10^{-4}$,%	4,8–5,0	6,0–7,2
mass fraction of chlorophylls	$\times 10^{-4}$,%	0,5–0,7	0,8–1,3
mass fraction of total gossypol	%	0,38–0,51	0,25–0,35
mass fraction of bound gossypol	%	0,11–0,15	0,21–0,28

Discussion and Conclusion

It is evident from Table 1 that the content of mass fractions of carotenoids and free gossypol in oil obtained from a mixture of cotton seeds is higher than from a mixture of grades III and IV. And vice versa, the mass fractions of chlorophyll and bound gossypol in oil obtained from a mixture of grades III–IV are higher than from a mixture of grades I and II. This is also confirmed by the high color of oil obtained from a mixture of cotton seeds of grades III–IV (Table 1).

Данные результаты были получены при соблюдении в опытно-производственных условиях соотношения сортов семян хлопчатника в смеси равным 50: 50 (%).

These results were obtained under experimental production conditions when the ratio of cotton seed varieties in the mixture was 50:50 (%).

Analysis of the annual receipts of cotton seeds to the oil and fat enterprises of the Republic shows that approximately 2/3 of the seeds are grades I and II, and 1/3 are grades III and IV. The trend over the last 5 years shows an increase in the supply of grades III and IV cotton seeds to the country's oil and fat enterprises, which reduces the yield of the oils obtained and their quality. Refining of crude oils obtained from low-grade and non-standard cotton seeds requires improvement of the existing technology.

References

Technology of production of vegetable oils / (V. M. Kopeikovsky, S. I. Danilguk, G. I. Garbuzova et al.): edited by V. M. Kopeikovsky – M.: Light and food industry, 1982. – 416 p.

Korostelev V. M., Maznyak I. E. Results of the oil and fat industry for 1985 // Oil and fat industry. – Moscow, 1986. – No. 6. – P. 16–22.

Holapek M., Jandera P., Zderadika P., Hruba L.: Characterization of triacylglycerol and diacylglycerol composition of plant oils using high-performance liquid chromatography-atmospheric pressure chemical ionization mass spectrometry. Journal of Chromatography A, 1010, – 2. 2003. – P. 195–215.

Khodjaev A. A., Ataullaev A. Kh. Improving the quality and reducing losses of cotton seeds // Oil and fat industry. – Moscow, 1986. – No. 12. – P. 12–13.

Akhmedov A. N., Abdurakhimov S. A., Azimov Yu. Kh. Bentonite adsorbents modified with urea solution. Universum: technical sciences, – P. 59–62.

Akhmedov A. N., Erkaeva N. Ch. Improving the process of primary purification of vegetable oils. Journal of innovative technologies. – Karshi, – Special issue. 2021. – P. 35–39.

Akhmedov A. N. Study of the indicators of cottonseed oil obtained by the method of forpressing from low-grade cotton seeds. Universum: Technical sciences. Issue: – 4 (61) April, 2019. – Moscow, – P. 23–26.

Tyutyunnikov B. N., Naumenko P. V., Tovbin I. M., Faniev G. G. Technology of fat processing. – Moscow: Food industry, 1970. – 652 p.

Karishma R., Lakshmi Sahithya U., Suneetha P., et al. Determination of Total Gossypol and Free Gossypol Content in different varieties of Bt and Non Bt Cotton seed extracts by High- Performance Liquid Chromatography (HPLC) // Research journal of biotechnology. 2016. – Vol. 11. – No. 2. – P. 70–74.

Abou-Donia S. A., Laches J. M., Abou-Donia M. B. High performance liquid chromatography analysis of gossypolum. J. Chromatography. 1981. – 206 (3). – P. 606–610.

Technical Requirements 88.06–27: 2011. Unrefined high-gossypol cottonseed oil. Appendix "B". Method for determining the proportion of free gossypol. – Tashkent. Gosstandart. 2011. – 9 p.

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