ISOLATION OF RAFFINOSE FROM COTTONSEED MEAL.

II. UTILIZATION OF PRODUCTION WASTES

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The course of production has been monitored over the stages of the technological process with the aim of identifying the main losses of raffinose. The fractions were analyzed by a chromato-photometric method. It was established that the main losses of raffinose took place at the stage of purifying the crude extract with gasoline and in crystallization. The composition of the wastes from the production of raffinose has been studied and possible methods for utilizing them have been found.

A method which we have developed for obtaining raffinose from cottonseed meal [1] and some losses during its production [2] have been reported previously. In the present paper we give a quantitative estimate of the losses over the various stages of the technological process and possible methods for utilizing raffinose production wastes.

As the raw material we used cottonseed meal from the 1983 harvest with a raffinose content of 4.5% obtained in the Kokand oils and fats combine. The raffinose was obtained from the meal by the following scheme: The meal after being ground in a hammer mill was extracted with aqueous ethanol by the steeping method, and the extract was evaporated. The viscous residue was treated with gasoline to eliminate hydrophobic impurities and was purified, and then acetone was added and the mixture was left for crystallization. The technical raffinose that deposited was separated off and, after being washed with aqueous acetone and dried, it was recrystallized from aqueous ethanol.

A check was carried out over all the stages of the technological process with the aim of identifying the losses of raffinose. The method of analysis was based on a procedure for determining raffinose in the raw material [3], which permitted its concentrations in the extracts and mother liquors to be determined:

Material analyzed	Amount of raffinose	
	g	%
Meal	4.52	100.0
Bagasse	0.25	5.52
Total extraction	4.20	92.22
Gasoline extraction	0.47	10.38
Technical raffinose	2.87	63.59
Mother liquor after the isolation of the technical raffinose	0.87	19.24
Purified raffinose	2.59	56.45
Ethanolic mother liquor	0.25	5.53

As we assumed [2], the main losses of raffinose take place at the stage of purifying the crude extract with gasoline and during crystallization, which is due to the increase in the solubility of raffinose as the result of the mutual influence of the accompanying substances.

In the production of raffinose, the following wastes are formed:

the insoluble residue of the meal — bagasse;

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the residue after the regeneration of the solvent in the stage of purifying the crude extract with gasoline;

the mother solution after the isolation of the raffinose (technical) from a mixture of ethanol, acetone, and water; and

the mother solution after the recrystallization of the raffinose from 80% ethanol.

The bagasse can be used as a source of food protein and of phytin and also of a protein-rich fodder product in combined feeds. To evaluate the food quality of the bagasse, it was analyzed. It contained (% of the absolutely dry weight): proteins (N × 6.25) – 50; water-soluble carbohydrates – 0.2-0.3; oil – traces; phospholipids (total) – 0.3-0.5; total gossypol – 1.5; free gossypol – absent; phytin – 7.2.

Thus, the elimination of the gossypol in the extraction process considerably improves the food value of the bagasse as compared with the meal, which contains 0.15% of free gossypol. In the extraction of the raffinose from the meal, the free fatty acids, the accompanying pigments, the carbohydrates, and part of the phospholipids are also removed. As a result of this, the bagasse becomes a relatively rich source of protein and phytin.

The temperature regime of the process for regenerating the ethanol in the drying of the bagasse has an appreciable influence on the extraction of the protein. To determine the optimum regime for drying the bagasse we performed a series of experiment in which 500-g portions of the bagasse were dried at various temperatures. Phytin and protein were obtained from the dried raw material by methods described in the literature [4, 5]. The results of the experiments are given below:

Drying temperature, °C	Phytin (tech.)		Protein	
	g	% of the weight of the bagasse	g	% of the weight of the bagasse
20	25.5	5.1	82.5	16.5
50	26.0	5.2	85.5	17.1
80	25.0	5.0	85.5	17.1
90	24.0	4.9	77.0	15.4
100	23.5	4.7	26.5	5.3
110	22.0	4.4	11.0	4.2

With a rise in the temperature at which the bagasse was dried, the yield of protein and, to some extent, that of phytin fell. In veiw of the fact that phytin is fairly resistant to the action of heat, it may be assumed that in this case a complex compound of protein and phytin is formed which has a lower solubility in acid media, while the protein undergoes far-reaching denaturation at a high moisture content and a high temperature. Thus, the drying of the bagasse to obtain protein and phytin must be carried out at a temperature not exceeding 80°C. When the bagasse is used as a fodder, the drying process can be carried out at between 80 and 110°C.

The residue after the regeneration of the solvent in the stage of purifying the viscous extract (the gasoline is sent for re-use) forms a dark resinous mass. As analysis has shown, it includes 10.4% of the raffinose present in the raw material. Although raffinose is practically insoluble in gasoline, its passage into the gasoline fraction can be explained, on the one hand, by the fact that part of the raffinose forms a complex with phospholipids and other components and, on the other hand, by the fact that it can pass over in the form of an emulsion. We performed a series of experiments to isolate an additional amount of raffinose at this stage. For this purpose, the gasoline fraction was dried, and the raffinose was extracted from the viscous solution with water. Analysis showed that the aqueous extract contained raffinose (40% of the sum of the carbohydrates), and also mono- and disaccharides. In view of the comparatively low concentration of raffinose in the aqueous extract and the complexity of isolating it from aqueous solutions, we proposed to use this extract to prepare the extractant (85% ethanol) from 95% ethanol.

The mother solution after the isolation of the raffinose (technical) from the ternary mixture of ethanol, acetone, and water consisted of a transparent dark red solution. After the regeneration of the ethanol and the acetone, the residue contained mainly water and the combined carbohydrates. According to the results of analysis, the combined material, in addition to traces of raffinose (0.16%), also contained sucrose (0.28%) and some other sugars (0.06% on the weight of the initial meal). Because of the presence of a large amount of other sugars, it is impossible to isolate the raffinose from various solutions by crystallization. The chromatographic separation of such a mixture is economically unfavorable.

EXPERIMENTAL

The extraction of the raffinose was carried out with 85% ethanol under static conditions at room temperature. Five steepings were carried out. The liquor ratio in the process was 1:3.

Elimination of Resinous Substances from the Ethanolic Extract. The ethanolic extract obtained was dried to a ratio of 1:5 in relation to the initial meal in a vacuum evaporating apparatus. The hydrophobic impurities were eliminated from the dried extract by two treatments with an equal volume of gasoline in a separatory funnel.

Isolation and Recrystallization of the Raffinose. The raffinose was precipitated from the purified extract with acetone in a ratio of 1:2. The technical product was filtered off, washed on the filter with aqueous acetone, and dried in the air and was then purified by recrystallization from 70% ethanol.

Analysis of the fractions in the various stages of the technological scheme was carried out by a chromato-photometric method [3]. Depending on the concentration of raffinose in the sample being analyzed, for each case a suitable dilution was selected before the material was deposited on the chromatogram.

SUMMARY

1. A method of monitoring the production of raffinose in the various stages of the technological process have been developed.

2. The distribution of raffinose and its losses in the various stages of the production process have been determined.

3. The component parts of the wastes from the production of raffinose have been studied and possible ways of utilizing them have been found.

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