EXTRACTION OF COTTONSEED MEAL

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A method has been developed previously for obtaining food protein from industrial cottonseed meal in a weakly acid medium [1]. As the extractant we have used aqueous solutions of ammonium chloride.

The extraction of protein with solutions of ammonium chloride has a number of advantages in comparison with methods that have been developed which are based on extraction in an alka-line medium [2-4].

When the meal is extracted in a weakly acid medium, in addition to protein and some other soluble components of the meal, a well-known medicinal preparation — phytin — which is isolated from the serum of the protein, is obtained [5].

The possibility of using the mother solution obtained by extraction with 5% NH₄Cl from cottonseed meal as a fertilizer for technical crops is of no little importance, since the utilization of a large amount of mother solution containing more than 5% of other inorganic salts and 3% of organic substances is a problem that is difficult to solve on the industrial scale.

In order to study the influence of the concentration of the extractant and the ratio of meal and extractant on the protein-extraction process, we have performed a series of experiments on a large laboratory apparatus for obtaining 50 kg of protein per day.

Batches of 50 kg of the meal ground in a hammer mill and sieved (through a 1-ml sieve) were extracted with 2, 5, 7, and 10% solutions of ammonium chloride under the same conditions. For extraction we used the meal of the Andizhan oils and fats combine, 1974 harvest. The yields of protein were, respectively, 7, 14, 14.5, and 15% of the weight of the meal. In view of the economic undesirability of using a 10% solution of ammonium chloride, with which the yield of protein increases by only 1% (on the weight of raw material) as compared with extraction with a 5% solution, as the extractant we selected the 5% solution of ammonium chloride.

Extraction with a 5% solution of ammonium chloride at various ratios of meal and extractant showed that an increase in the ratio to 1:15 leads to some rise in the yield of protein, but the bulk of the protein is extracted even at a ratio of meal and extractant of 1:8.

We also studied the effect of the intensity of stirring on the protein-extracting process, since the performance of the extraction process (on the industrial scale) in reactors with anchor type stirrers was envisaged. As the experiments showed, within a range of 50-200 rpm the speed of the stirrer has practically no effect on the extraction process, which is explained by the good structure of the ground meal for extraction.

In order to study the influence of the temperature and the time on the process of extracting protein with a 5% solution of ammonium chloride at a ratio of meal and extractant of 1:8, we performed a series of experiments in which 50 kg of milled and sieved meal was extracted under similar conditions with variations in the time and temperature of extraction (Fig. 1).

As can be seen from Fig. 1, the soluble fraction of the protein passes into the extract fairly rapidly. With an increase in the time of extraction the yield of protein rises mainly in the first 30 min, and a further increase in the time has little influence on the extraction process. At temperatures above 25°C extraction is intensified fairly slowly, and at temperatures above 70°C the yield of protein falls as the result of denaturation.

Institute of Chemistry of Plant Substances, Academy of Sciences of the Uzbek SSR, Tashkent. Translated from Khimiya Prirodnykh Soedinenii, No. 6, pp. 849-952, November-December, 1977. Original article submitted June 24, 1977.

UDC 664.762



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In order to study the influence of the pH of extraction on the yield of protein and phytin, we performed a series of experiments in which portions of the meal were extracted under the same conditions except for variations in the pH of extraction. The pH of extraction was established by using 5% solutions of hydrochloric acid and of ammonia. The results of the experiments are given below:

Yield of pro- tein, % on the weight of the meal	Amount of bound gossypol in the protein, %	Yield of phytin, % on the weight of the meal
3,5	0,1	4.5
10,0	0,12	4.5
13,5	0,12	4,5
14,5	0,20	4,7
15,0	0,20	4,3
15,0	0,20	4,3
15,0	0,23	0,5
15,1	0,25	
15,1	0,30	
15,2	0,3)	
	Yield of pro- tein, $%$ on the weight of the meal 3.5 10,0 13,5 14,5 15,0 15,0 15,0 15,1 15,1 15,2	$\begin{array}{c} Yield of pro-tein, $\%$ on the weight of the mean of t$

They show that with a rise in the pH to 6.0 the yield of protein increases appreciably but with a further rise in the pH the amount of ballast substances extracted rises, which imparts to the protein an undesirable color and smell after drying in a spray dryer. In addition to this, at pH 7 and above practically no phytin is extracted.

In the extraction of the protein with a 5% solution of ammonium chloride part of the free gossypol passes into the protein extract and, accordingly, into the precipitated protein. However, after washing and drying in a spray dryer only traces of free gossypol are found in the protein, which is due to its decomposition in the protein-drying process.

One must take into account the fact that in the extraction of the meal at pH 7 and above practically no phytin is extracted. The initial 5% ammonium chloride solution has pH 5.7-6.0, which is also the optimum for extracting protein and phytin.

The protein obtained after the optimum conditions with a yield of about 15% on the weight of the meal correspond to the main requirements set for food proteins, apart from the amount of assimilable lysine — the limiting amino acid of cottonseed protein.

EXPERIMENTAL

Preparation of the Meal for Extraction. Cottonseed meal (100 kg) was ground in a hammer mill and sieved through a 1-mm vibrosieve, and 50 kg was weighed out for the extraction of the protein and phytin.

Preparation of the Extractant. A 620-liter reactor (extractor) with an anchor type stirrer was charged with 400 liters of water and, with the stirrer working, ammonium chloride of "kh. ch." ["chemically pure"] grade was added in 20-kg portions. After the complete dissolution of the salt, the pH of the solution was checked. Where the pH deviated from the norm (5.7-6.0) it was brought to 5.7-6.0 by the addition of ammonia solution or hydrochloric acid. Extraction of the Protein. With the stirrer working (60 rpm), 50 kg of milled and sieved meal was added to the extractor through an opening, and extraction was performed for 30 min.

Separation of the Extract from the Pulp (Insoluble Residue). After the lapse of 30 min, the slurry was passed from the extractor to a filter funnel where the extract was separated from the pulp in vacuum through a cloth filter fabric. If suspended particles were observed in the extract, it was clarified in a SGO horizontal continuous centrifuge at 10,000 rpm.

Precipitation and Separation of the Protein. The clarified extract (340-350 liters) was pumped into a settling vessel (capacity 500 liters, stainless steel, fitted with a stirrer) and, with the stirrer working (50 rpm), a 5% solution of hydrochloric acid was added to the extract to give a pH of 3.5. The pH was checked by means of a remote-reading pH-meter. The protein suspension formed was transferred to the vacuum drum filter where the precipitate of protein was separated from the serum under continuous working conditions.

<u>Washing and Drying of the Protein</u>. The protein paste (37.5 kg; protein content 20%) was mixed with 50 liters of water and was milled in an homogenizer. The milled protein suspension was diluted with 300 liters of water and transferred to a vacuum drum filter where, with the drum working continuously, an additional 50 liters of water was added in order to displace the residues of salts.

The washed and separated protein paste was diluted to a 10% concentration of protein in the slurry and was dried in a spray dryer of the "Angidro" type and a RSL-10 with an inlet air temperature of 200°C and an outlet temperature of 75°C. The dried protein powder (7.5 kg) contained about 7% of moisture.

SUMMARY

A method for extracting protein in a weakly acid medium has been developed which ensures, apart from a satisfactory yield and a good-quality main product, the isolation of a valuable medicinal preparation — phytin.

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