ISOLATION OF PHYTIN FROM COTTONSEED MEAL

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Phytin — a complex organic phosphorus-containing material — is mainly a mixture of the Ca and Mg salts of inositol hexaphosphate. Like other phosphorus-containing preparations, it is widely used in medicine [1].

A number of methods exists for isolating phytin from hempseed meal and other meals [2]. Attempts have been made to isolate it from the wastes of the starch syrup industry, but because of the low concentration of phytin in the effluents its production proved to be unprofitable [3, 4]. A method has also been proposed for isolating phytin from cottonseed meal [7], but on the industrial scale it is impossible to perform because of the unsuitability of individual stages on this scale. At the present time, the medicinal industry uses a comparatively cheap fodder product, rice bran, as the raw material for the production of phytin [6].

We have developed a method for isolating phytin from the mother solution obtained in the production of fodder protein from cottonseed meal. When the protein is extracted from the meal in a weakly acidic medium (pH 5.7) with 5-10% aqueous solutions of ammonium chloride [7], the phytin passes into the extract together with the protein.

Experiments have shown that in the extraction of a meal containing 4.7-4.8% of phytin by these solutions in a ratio of 10 liters per kg with stirring for 30 min, it is possible to extract about 95% of the soluble part of the phytin. The loss of phytin in its isolation at the stage of the precipitation of the protein from the extract is about 10%, at the stage of the precipitation of the phytin from the serum about 5%, and at the stage of its reprecipitation about 10%.

The bulk of the precipitatable phytin deposits from the serum (the mother solution after the separation of the protein) at pH 7-7.5. To neutralize the mother solutions we used 5-10% aqueous solutions of NaOH, KOH, and NH_4OH, and it was found that the concentration and nature of the alkali do not affect the yield of phytin. As the experiment showed, 5 min is sufficient for the formation of the phytin precipitate, which is then separated from the mother solution in standard separators (about 6000 rpm) or by the automatic filter presses used in the medical industry. One of the indices of the quality of the phytin is its phosphorus content, which is determined in the form of phosphorus pentoxide [8]. The dried phytin precipitate contained about 35% of phosphorus pentoxide, which is 4-5% lower than the required amount.

Analyses showed that the technical phytin contained about 5% of water-soluble protein and about 10% of ammonium chloride. To get rid of the ballast substances, we used the method of the direct washing of the precipitate with water or the reprecipitation of the technical phytin from acid solutions. The reprecipitation of technical phytin proved to be the more effective method, the best result being obtained by dissolving the phytin in dilute solutions of mineral acids, especially hydrochloric acid.

From the filtered solution of technical phytin (pH 2-3), the desired product was precipitated with a 5% aqueous solution of NH_4OH at pH 7-7.5. The reprecipitated phytin contained 40-42% of phosphorus pentoxide.

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EXPERIMENTAL

Precipitate of Phytin. A 120-liter glass reactor with a stirrer was charged with 90 liters of the mother solution (serum) after the precipitation and separation of the protein obtained from 10 kg of cottonseed meal. With the stirrer working (~60 rpm), a fine stream of 5% NH_4OH solution was run into the solution to give a pH of 7-7.5, and the mixture was stirred for 5 min for the formation of the precipitate.

Separation of the Phytin Precipitate. To separate the precipitate of technical phytin we used a suction filter (20 liters). The suspension of the phytin precipitate was filtered through a double filter consisting of a "Belting" fabric filter and a paper filter.

Reprecipitation of the Technical Phytin. In a 20-liter glass reactor, 2 kg of the filtered-off phytin paste (moisture content ~75%) was stirred with 10 liters of water (speed of the stirrer ~90 rpm). Then 10% hydrochloric acid was added in 50-ml portions until the phytin had dissolved completely (pH ~ 3). The phytin solution was passed through a double-layer paper filter on a suction filter. The clarified solution was transferred to a 20-liter glass reactor, and the phytin was precipitated with a 5% solution of NH_4OH (at pH 7.5). The phytin precipitate was separated by filtration and washed with water (one liter).

Drying of the Phytin. The filtered-off and water-washed precipitate of phytin was stirred with 800 ml of water and ground in a colloid mill. The ground phytin suspension was passed through a sieve (0.25 mm) and was dried in a spray dryer of the "Angidro" type. The dried phytin powder (300 g, 3% of the weight of the initial meal) contained about 42% of phosphorus pentoxide and corresponded to the requirements of the State Pharmacopoeia [8].

SUMMARY

A method has been developed for isolating phytin from the waste material (mother solution after the precipitation of protein) obtained in the production of fodder protein from cottonseed meal.

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