THE ISOLATION OF OLITORISIDE FROM JUTE SEEDS

M. T. Turakhozhaev, L. P. Zubkova,
M.-R. I. Shamsutdinov, G. L. Genkina,
and T. T. Shakirov

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The cardiac glycoside olitoriside, which is used in medical practice [1], has been isolated previously from the seeds of Corchorus olitorius [2].

We have developed a method for isolating this glycoside with a yield of 0.2% (on the weight of the raw material) [3]. Control analyses performed on the stages of this method have shown that the bulk of the loss of the olitoriside takes place at the crystallization stage [4]. Because of the inadequate purification of the extract in the preceding stages, a large amount of ballast substances remain in the mother liquor which prevent the crystallization of about 30% of the olitoriside [4].

In order to increase the yield of the glycoside, an aqueous solution of the extract was purified on ion-exchange resins. The best results for the selective adsorption of the ballast substances from an aqueous solution of jute seeds were achieved on ÉDÉ-10p anion-exchange resin (in the OH form) and on the KU-2 cation-exchange resin (in the H form). The ÉDÉ-10p anion-exchange resin adsorbed about 98% of the ballast substances from an aqueous solution of the extract while absorbing only a small amount of the glycosides (5%); the corresponding figures for the KU-2 cation-exchange resin were 97 and 23%. As can be seen, it is preferable to use ÉDÉ-10p anion-exchange resin (in the H form) for the purification of the extract.

The ballast substances (a considerable part of which consists of neutral substances) are adsorbed mainly by molecular adsorption. However, in the purification of an aqueous solution of the extract on the anion-exchange resin, the pH of the purified solution changes, which is impermissible for the isolation of the cardiac glycosides. The pH of the purified solution apparently changes because of the partial adsorption of the ballast substances on the anion-exchange resin through ion exchange.

To neutralize the medium, a mixture of anion-exchange and cation-exchange resins was used. A purified aqueous extract remained neutral on mixtures of anion-exchange and cation-exchange resins in ratios of 1:2, 2:1, and 3:1.

Considering the higher capacity of the cation-exchange resin for the adsorption of the glycoside, we selected a mixture of the anion-exchange and the cation-exchange resins in a ratio of 3:1 by weight.

An ethanolic extract obtained from the plant and treated with acetone and ether by the method described previously [3], was dissolved in water and passed through a mixture of ÉDÉ-10p anion-exchange resin and KU-2 cation-exchange resin. The olitoriside was extracted from the purified aqueous solution with a mixture of chloroform and isopropanol (1:1 by volume).

The alcoholic–chloroformic extract containing mainly olitoriside was concentrated at a temperature not exceeding 45°C, and the glycosides were precipitated from the viscous solution with diethyl ether. The precipitate that deposited was separated off and washed with ether. The technical product was dissolved in ethanol and the solution was diluted with an equal amount of water. The crystals that deposited were filtered off with suction and dried in the air. In the precipitation of the olitoriside with ether, the less polar glycosides remained in the solution and the more polar ones were retained in the aqueous ethanolic mother

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liquor. When ion-exchange resins were used to purify the glycosides from ballast substances, the yield of the preparation increased to 0.3% (on the weight of the raw material).

The stagewise monitoring of the isolation of olitoriside by this method was performed in a similar manner to the known method [4].

The results of determinations of the amounts of olitoriside in the stages of the technological process are given below.

Stage of Isolation	Amount of Olitoriside, %	
	of the amount in the total extract	of the weight of the seeds
Total extract	100	0.82
Extract after precipitation of sugars	85.4	0.70
Aqueous extract before ion-exchange purification	73.2	0.60
After purification on ion-exchange resins	62.2	0.51
Chloroformic extract	6.2	0.05
Ethanolic-chloroformic extract	52.4	0.43
Technical product after precipitation with ether	47.5	0.39
After recrystallization	36.6	0.30

EXPERIMENTAL

The defatting of the seeds, the extraction of the glycosides with ethanol, the precipitation of the sugars from the viscous extract, and the precipitation of the glycosides from the extract freed from sugars were performed in a similar manner to the first method [3]. The viscous extract obtained by the method described above from 100 kg of seeds was dissolved in 35 liters of water.

Preparation of the Ion-Exchange Resins. A mechanical mixture of 4.5 kg of ÉDÉ-10p anion-exchange resin (OH form) and 1.5 kg of KU-2 cation-exchange resin (H form) was charged into a column and washed with distilled water.

Purification of the Aqueous Solution of the Extract. The aqueous solution of the extract (35 liters) was passed through the column containing the mixture of ion-exchange resins from the bottom to the top at a rate of 5-6 liters/h. The temperature of the solution was not below 20°C and not above 30°C. The purified solution was clear and slightly yellowish.

Extraction of Corchoroside. In an apparatus for liquid-liquid extraction, 3.5 kg of common salt was added to the purified aqueous solution, and it was washed with 5 liters of chloroform. After stirring for 10 min and settling, the lower, chloroform, layer was run off. The operation was repeated four times. The chloroform extract contained mainly corchoroside.

Isolation of Olitoriside. The olitoriside was extracted from the purified aqueous solution with a mixture of chloroform and isopropanol (1 : 1, by volume; 8×5 liters). The last seven extracts were combined and were concentrated in a vacuum evaporator to 3 liters.

Precipitation of the Olitoriside. The olitoriside was precipitated from the 3 liters of concentrated extract with 12 liters of ether. The pulverulent technical product was filtered off with suction and was washed with 3 liters of ether.

Crystallization of the Olitoriside. The technical olitoriside was dissolved in the minimum amount of ethanol, filtered through a hot filter, diluted with an equal volume of water, and left to crystallize. The crystals that deposited were filtered off with suction and washed with water. The yield of olitoriside was 300 g, or 0.3% on the weight of the seeds.

SUMMARY

1. The possibility of using ion-exchange resins for the purification of extracts in the isolation of cardiac glycosides has been shown.

2. A method is proposed for obtaining olitoriside by using a mixture of anion-exchange and cation-exchange resins to purify the extract, this method giving an increased yield of the preparation.

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