EXTRACTION OF COTTONSEED OIL

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Continuing investigations of the isolation of gossypol in the complex processing of cotton seeds by the direct-extraction method [1], we have studied the influence of the nature of the solvents on the extraction of gossypol. As the solvents we used hexane and acetone and mixtures of them both with the addition of water and without it. Begow we give the characteristics of the extracted meal so obtained (%):

Solvent	Number of extractions	Oil content	Free gossypol	Moisture content
Hexane, 70% aqueous acetone	4+4*	2,5	0,03	9,8
Hexane, 80% aqueous acetone	4+4	1,7	0,02	8,6
Hexane, 70% aqueous acetone, dry acetone	4+1+3	1,5	0,04	9,4
Mixture of acetone and hexane (15:85 by volume)	8	0,3	0,25	9,5
Mixture of acetone, hexane, and water (42.5 :55: 2.5)	8	0,5	0,04	9,5
Mixture of acetone, hexane, and water (73:25:2)	8	0,4	0.02	8.5

*The first figure shows the number of extractions by the first solvent, the second figure by the second solvent, and so on.

The extracted oil was characterized by the following indices:

Solvent	Acid No., mg KOH	Color in a 1-cm layer at 35 yellow units	Yield of oil after refining, %	Tocopherols, mg-%	Phosphatides calculated as stearo-oleolecithin, %	Unsaponifiable substances, %
Hexane	1,6	6	94,5	50,9	0,6	0,8
Acetone–hexane (15:85)	7,0	25	88,5	58,9	2,2	0,8
Acetone–hexane–water (42.5:55: 2.5)	8,1	29	85,4	42,4	2,9	0,9
Acetone–hexane–water (73:25:2)	8,7	28	84,8	42,2	3,4	0,8

As the figures given above show, the amount of free gossypol in the extracted meal is practically the same after double extraction first with hexane and then with 80% aqueous acetone as after the use of a ternary azeotropic mixture. However, the quality of the oil in the first case is considerably higher than in the second. Of the solvents that we investigated, the binary mixture of acetone and hexane (15:85) is the worst in relation to the degree of the extraction of free gossypol from cotton-seed flakes. Other workers have come to the same conclusion [2].

The results of experiments performed in order to determine the sequence of the use of solvents showed that on extraction first with 70-80% aqueous acetone and then with hexane the oil content of the extracted meal was higher than with the reverse order of use of the solvents. The amounts of free gossypol were almost the same in the two cases.

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The characteristics of the meal after eight extractions of 30 g of cottonseed flake for 20 minutes each time at a ratio of solvent and material of 20:1 ml/g were as follows (%):

Solvent	Free gossypol	Total gossypol	Oil content	Moisture content
Hexane, 80% acetone	0,08	0,09	2,4	9,3
80% acetone, hexane	0,07	0,09	3,5	9,0
Hexane, 70% acetone	0,08	0,10	3,1	8,9
70% acetone, hexane	0,07	0,2	7,2	9,4

The cottonseed meal obtained after extraction with aqueous acetone followed by drying had a color ranging from cream to light brown, which is connected with the chemical transformations taking place under the action of heat and moisture [3].

To obtain a meal with a low content of free gossypol it has been proposed [4] to cover the hexane-defatted flakes with 70% aqueous acetone for 5 min and to perform extraction with dry acetone. In this way the authors succeeded in obtaining a protein-rich (63%) meal with a light cream color containing 0.02% of free gossypol.

However, the results of the analogous experiments that we have performed have shown that even after 20-min steeping of the flakes with 70% aqueous acetone the amount of free gossypol in the meal was fairly high (0.21%). Apparently, to decompose the gossypol glands with the thickness of the flake that we used (0.3 mm) requires longer steeping than for a flake 0.07 mm thick [4].

The longer the treatment of the meal with anhydrous acetone the lighter did it become. After five treatments with aqueous and dry acetone (length of extraction 10 min, ratio of solvent to material 15:1 ml/g) the meal had the following indices:

Concentration of acetone (%) in extraction No.					Free gossypol content, %	Color
1	*2*	*3*	*4*	*5*		
80	80	80	80	80	0,09	Light brown
80	100	100	100	100	0,27	Light cream
80	80	100	100	100	0,20	Light cream
80	80	80	100	100	0,13	Light cream
70	70	100	100	100	0,21	Cream

In giving our preference to the method of double extraction of the cotton flakes first with hexane and then with 70-80% aqueous acetone, we investigated the extractability of the oil and the gossypol as functions of the method and duration of the process.

The first two series of experiments were performed by steeping the flake in each of four separatory funnels. The experiments differed from one another only by the time of steeping.

The hexane from the first funnel together with the extracted oil was passed through all funnels. The material was steeped eight times.

In the third series of experiments, the hexane was continuously fed into the first funnel, from which (without steeping) the miscella passed successively into the other three funnels. Then the concentration of the miscella was determined. Below we give the results of an analysis of the miscellas and meal after the extraction of 200 g of cottonseed flake at a ratio of hexane and material of 4.5:1 ml/g (in the second and third series of experiments 50 ml more hexane was used than in the first):

Series of experiments	Duration of the process, min	Concentration of the miscella (%) in extraction No. 1	2	3	4	5	6	7	8	Oil-content of the meal, %
1	150	50,6	25,2	11,5	2,5	2,2	0,9	0,6	0,5	2,7
2	30	35,6	24,3	13,7	7,1	3,6	2,1	1,8	0,9	3,2
3	60	48,6	20,7	6,5	2,4	1,2	0,7	0,3	0,2	2,2

It can be seen that the method of continuous feeding of the solvent is the most effective, although the oil-content of the meal in this case is considerably higher.

With an increase in the thickness of the layer of flake (100 g in each of seven separatory funnels) at a consumption of solvent of 3.7:1 ml/g it was possible to reduce the oil content of the meal to 1.5% on steeping for 2 h 20 min. The subsequent extraction of the dried defatted material with 80% aqueous acetone (6:1 ml/g) enabled us to obtain meals containing 0.02% of free gossypol.

In order to reduce the consumption of solvent and the time of the extraction process, in addition to hexane we used weak miscellas [5]. For this purpose, 100 g of cottonseed flake was steeped six times for different lengths of time at a ratio of solvent and material of 12:1 ml/g (Table 1).

From Table 1 it can be seen that using low concentrations for extraction of the miscellas practically does not affect the oil content of the meal and permits one to reduce the consumption of solvent to a solvent–material ratio of 4:1 ml/g.

TABLE 1.

Time of extraction, h	Amount of oil (g) after extraction with												Indices of the meal after extraction with			
	hexane						miscella						hexane		miscella	
	1	2	3	4	5	6	7%	3%	1%	0,5%	hexane	hexane	oil content, %	free gossypol	oil content, %	free gossypol
0,5	24,0	5,7	1,8	0,8	0,4	0,3	31,3	11,0	4,1	1,9	0,8	0,4	3,1	2 3	3,5	2,1
1,0	26,3	6,3	0,8	0,4	0,2	0,1	36,0	9,4	3,3	1,5	0,5	0,3	2,0	2,2	2,2	2,0
1,5	26,7	6,5	0,8	0,3	0,2	0,1	36,5	9,3	3,5	1,3	0,4	0,2	1,6	2,2	1,7	2,0
2,0	27,9	6,8	0,7	0,3	0,2	0,1	37,9	9,7	2,8	1,2	0,3	0,1	1,0	2,1	1,4	1,9

Experiments were carried out similarly on the extraction of gossypol from meal defatted with a hexane miscella using weak gossypol acetone miscellas. The defatted flake (100 g) was extracted six times with a solution of 80% aqueous acetone, containing gossypol and was treated twice with pure 80% acetone. The meal obtained after extraction for 2 h contained 0.05% of native gossypol.

EXPERIMENTAL

The amounts of free gossypol, phosphatides, and tocopherols and the acid numbers of the oils were determined by known methods [5]. The weights of cottonseed flake (the kernel flattened out on rolls) amounted to 30–100 g, depending on the quantity of solvent used.

SUMMARY

It has been shown that oil of the best quality is obtained on its selective extraction by hexane. Subsequent extraction from the meal of gossypol by 70–80% aqueous, and then dry, acetone permits meal to be obtained with a light cream color containing 0.02% of free gossypol. The omission of dry acetone leads to a darkening of the meal.

It has been established that it is possible to achieve a reduction in the consumption of solvents on the direct extraction of cottonseed flake by using weak oil and gossypol miscellas.

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