Analytical Control of Experimental Extraction of Oilseed

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ABSTRACT AND SUMMARY

A study was made of methods for measuring relevant components at different stages of an experimental process for mixed solvent extraction of raw cottonseed flaked meats. The principles discussed should apply to other complex oilseed separation procedures.

INTRODUCTION

Experimental studies on extracting cottonseed with mixed solvents to produce improved meals and oils necessitated development of analytical techniques that could be used to follow reapportionment of soluble components of the complex mixture among the various intermediate and final products of countercurrent extraction. Although the stoichiometric outline presented is useful for other types of solvent mixtures and extractions, the system described is the countercurrent extraction of raw cottonseed flaked meats with a mixture of acetone, hexane, and water in the proportions 53:44:3 v/v/v. This extraction procedure produced improved meals and oils under practical, reproducible conditions (1-4).

MATERIALS AND METHODS

A block diagram of the experimental extraction apparatus is shown in Figure 1. The freshly flaked, raw cottonseed kernels from decorticated prime seed were fed into the hopper at step 1. Miscella which had been used countercurrently in the last nine steps was simultaneously fed into the ribbon screw conveyor of step 1 from step 2. The conveyor mixed the miscella and flakes while moving the marc to its exit and to the upper deck of the vibrating screen separator. The separated marc passed to the conveyor-mixer of step 2, and the process was repeated nine more time until the finally extracted marc (spent flakes) flowed from step 10. The full miscella from step 1 flowed into a settling tank, where the flocculent fines which settled out were recirculated into the stream at the halfway point (step 5).

[1]Presented at the AOCS Meeting, Philadelphia, October 1974.

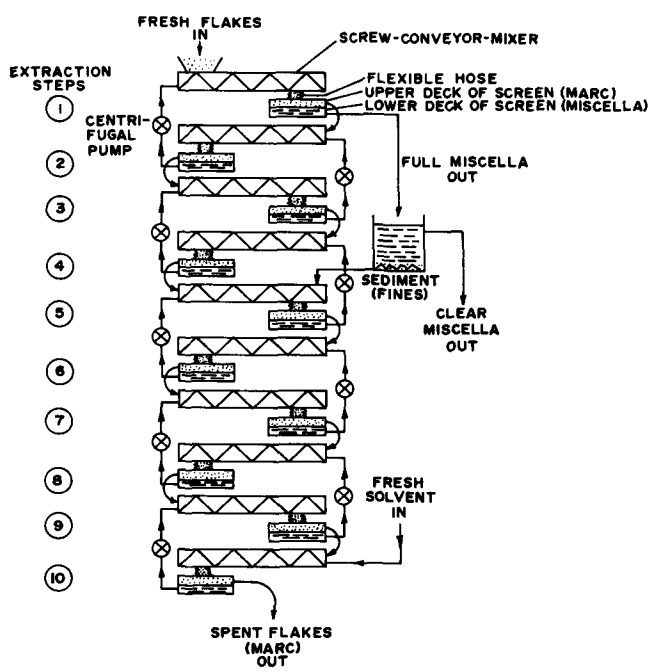

FIG. 1. Experimental extraction apparatus.

The components of the relevant marcs and miscellas are shown in Table I. For the experimental extraction described, 10-lb batches of raw flakes were charged to the system. Representative samples were taken from the raw meats used as starting material and from the marcs and miscellas at each stage of the process, after the extraction was stabilized.

When available, official AOCS methods were used. All quantitative values are expressed in percent by weight.

For determining residual solvent in the marcs and the solvent content of the miscellas, total volatile matter was measured by first removing the inflammable solvents on a steam bath, then oven-drying to constant weight at 105 C.

The original flakes contained 12.0% moisture. Calculation of solvent in the marc samples was made by using equation I:

$$S = V - XM/100 \qquad (I)$$

S is percent solvent in the marc, V is percent total volatile matter in the marc, M is percent whole meal with its unextracted oil and moisture (X).

M of equation I is calculated from equation II:

$$M = \frac{100 - V - V(100 - Y)/100}{1 - X/100} \qquad (II)$$

where Y is total volatile matter (TVM) in the corresponding miscella.

Crude oil, $\bar{O}$, in the solvent adhering to the meal in the marc is calculated from equation III:

$$\bar{O} = S(100 - Y)/Y \qquad (III)$$

T, the total crude oil in the miscella adhering to the marc, is calculated from equation IV:

$$T = \frac{P(100 - V)\,[100/(100 - W)]}{100} \qquad (IV)$$

In this equation, P is percent total crude oil in the air-dried marc (meal), V is TVM in the miscella, and W is the total volatile matter in the air-dried marc.

TABLE I

Relevant Components

Marcs	Miscellas
Residual solvent	Solvent
Whole meal with unextracted oil	Crude oil
Total oil	Neutral oil
Oil in adhering solvent	Gossypol
	Phosphatides and sugars
Original oil Remaining in meal	Aflatoxins
Gossypol	
Phosphatides and sugars e.g., raffinose	

TABLE II

Analytical Data

Step no.	Total volatile matter in marc (V) %	Total volatile matter in miscella (Y) %	Crude oil in air-dried marc (P) %	Total volatile matter in air-dried marc (W) %
Flakes	12.0	---	30.6	12.0
1	56.2	89.4	16.5	7.3
2	59.2	90.6	14.9	7.4
3	64.2	92.1	12.7	7.3
4	64.8	92.8	11.8	7.1
5	64.7	94.4	9.5	7.2
6	66.1	95.2	8.5	7.5
7	68.4	96.4	6.2	7.7
8	69.1	97.8	4.0	10.0
9	70.8	98.2	3.8	9.8
10	71.3	98.85	2.5	9.4

TABLE III

Composition of Marcs
(calculated from analytical data)

Step no.	% Solvent (S)	% Whole meal with remaining original oil (M)	Total crude oil (T)	Oil in adhering solvent ($\bar{O}$)	Original oil remaining in meal (Q)	% Original oil in air-dried marc (meal) on dry basis (Q:dry basis)
Flakes	0	100	--	--	--	30.6
1	51	43	8	6	2	4.5
2	54	40	7	6	1	2.5
3	60	35	5	5	0	0
4	61	35	4.5	4.5	0	0
5	60	36	4	4	0	0
6	62	35	3	3	0	0
7	64	33	2	2	0	0
8	65	33.5	1.5	1.5	0	0
9	67	32	1.2	1.2	0.0	0.0
10	68	31	0.8	0.8	0.0	0.0

Q, the original unextracted oil remaining in the meal, is calculated from equation V:

$$Q = T - \bar{O} \qquad (V)$$

where T is the total crude oil in the marc and $\bar{O}$ is crude oil in the adhering solvent.

In equation VI:

$$Q(\text{air-dried-meal basis}) = 100(T - \bar{O})/M \qquad (VI)$$

Thus, four conventional, simple, analytical measurements of the marcs and miscellas from each step in the experimental extraction and of the final air-dried marc can be used to obtain the amounts of solvent, whole meal with unextracted oil, total crude oil, oil in adhering solvent, unextracted oil remaining in the meal, and percentage of original oil remaining in the air-dried marc, or meal.

RESULTS AND DISCUSSION

Table II gives analytical data obtained on analysis of the original, flaked meats and of the marcs, miscellas, and air-dried final marc (or meal) from ten steps of the experimental extraction.

In Table III are tabulated the composition of the individual marcs calculated from the analytical data of Table II.

Thus, with the above stoichiometric analysis of the ten-step experimental extraction, the progress of rendering and removal of the oil by contact and extraction with the mixed solvent can be followed. Buildup of solvent in the marc can also be measured.

Analysis of the flocculent fines which settled from the final miscellas after these fines were recirculated at steps 3, 5, and 7 showed that 35%, 12%, and 27% of the weight of original flakes fed to the system appeared as fines. These data led to the use of step 5, halfway in the extraction, as the recirculating point for the fines. Ten additional analyses showed that stabilization of the extraction occurred at this point, and the quantity of fines in the final miscellas, before settling, varied from 8% to 16%, averaging 12%.

Total gossypol was determined on the air-dried marcs (or meal) obtained from each step. This value decreased progressively from 0.9% to 0.2%. Free gossypol was very low (0.03% or less) in the final meal; this was expected from the nature of the mixed solvent used.

Phosphatides (lecithin, etc.) and sugars (mostly raffinose), of secondary interest because of their nonnutritive and otherwise biologically inactive natures, were considered as nuisances to be disposed of. Both were distributed between the oil and final meal products. Their removal from the oil on refining did not present any problems—as a matter of fact, prime oil was obtained by miscella refining, and a slightly higher yield of neutral oil was obtained than was indicated by analysis of the original raw flakes by the official AOCS method (2).

Aflatoxins present in the raw flakes are removed with the oil, where they are inactivated or removed during the refining process (5,6).

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