Technical

Crambe Processing: Glucosinolate Removal by Water Washing on a Continuous Filter[1]

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ABSTRACT AND SUMMARY

Crambe seed was dehulled and screw pressed to remove approximately two-thirds of the oil, and then it was hexane-extracted to remove the rest. The defatted meal was toasted in the presence of moisture to form a crisped meal possessing fast drainage characteristics required for continuous filtration. The crisped meal was slurried with four parts of water, filtered, and washed on a continuous pilot-plant filter. Water washing removed about one-fourth of the meal solids, which contained 92-96% of the glucosinolates. Estimated processing costs for water-washing crambe meal are 22-23 dollars per ton of unwashed defatted meal, in addition to the cost of crushing the seed to oil and meal.

INTRODUCTION

Crambe abyssinica Hochst ex. R.E. Fries, a member of the Cruciferae family, is a valuable new oilseed crop because of the high erucic acid content of its oil. Erucic acid can be used in a variety of applications. Erucamide, a nitrogen derivative of erucic acid, is much in demand as a slip and antiblock agent in the manufacture of plastic films. Two new nylons (13 and 1313), which are characterized by moderate melting points and low water absorption, are derived from the products of erucic acid ozonolysis (1).

Crambe oil is recovered from the seed either by hexane extraction or by a combination with screw pressing, which removes about two-thirds of the oil, followed by hexane extraction. Although the defatted meal is high in protein (45-50%), it contains significant quantities of glucosinolates (8-10%) (2). These are sources of toxic compounds and must be either inactivated or removed if crambe meal is to be fed to nonruminants. Previous work at the Northern Laboratory has improved palatability and nutritional quality by chemically modifying crambe meal with soda ash (3) or ammonia (4). Many reports have appeared in the literature on extraction of glucosinolates from Cruciferae oilseeds. The methods can generally be classified as (a) water extraction of the glucosinolates, followed by hexane extraction of the oil (5-7), and (b) hexane extraction of the oil, followed by extraction of the glucosinolates with water (8-10), aqueous acetone, or aqueous methanol (11).

Comparing these two procedures, we must consider the adverse effect of moisture on oil quality if extraction is done before the oil is removed. Much of the data reported centers around extraction efficiency of glucosinolates, and filtration rates are rarely mentioned. Also, solids loss to the extract must be considered by each method. With water extraction we can expect to lose 25% or more of the solids (12). When other investigators used aqueous acetone or methanol, solids loss was reduced to 10% or less by manipulation of the water to solvent ratio (13). However, when solvents are used, one must consider the solvent cost, solvent recovery, and increased chemical oxygen demand (COD) in the extract.

In our study, we considered crambe oil to be the main product of an oilseed processing plant and the defatted meal to be the by-product. We expanded previous work with batch extractions (8) to a pilot-plant process for the continuous water extraction of glucosinolates from defatted crambe.

EXPERIMENTAL PROCEDURES

Materials

Crambe seed, grown in Illinois in 1972, was the Prophet variety released by the Agricultural Experiment Station of Purdue University, Lafayette, Indiana. Analyses of whole seed, dehulled seed (pericarp removed), and dehulled, defatted meal are shown in Table I.

Methods

Total glucosinolates were determined on the meals by enzymatic conversion to goitrin by a modified procedure of Wetter (14). Meal (1 g) was extracted with boiling water to remove all the glucosinolates. A 2-ml sample of the water extract was enzyme-converted to goitrin in 4 ml of pH 7 buffer containing 16 mg of myrosinase and held 2 hr at 55 C. The enzyme-converted solution was extracted twice with methylene chloride to remove goitrin; 50 ml of solvent was used for each contact, and final solvent volume was adjusted to 100 ml. Optical density of the methylene chloride extract was read on a Beckman DB spectrophotometer at 5-mμ intervals from 210-280 mμ.

Goitrin was analyzed by a method similar to that of Appelqvist and Josefsson (15). Organic nitriles were determined by IR absorption (16). Glucosidase activity was tested by the method of VanEtten et al. (17). Available lysine was determined by the method of Rao et al. (18). Nonprotein nitrogen was analyzed by the method of Becker et al. (19). Other analyses were conducted according to Official AOCS Methods (20).

TABLE I

Analysis (Dry Basis) of Crambe Seed and Meal

Constituent	Whole seed[a] (%)	Dehulled seed (%)	Dehulled defatted meal (%)
Crude fat	32.6	44.5	0.7
Protein (N x 6.25)	20.9	26.1	51.3
Crude fiber	13.0	4.4	7.0
Ash	6.1	5.1	8.8
Carbohydrates (by difference)	27.4	19.9	32.2

[a]Hull content, 29%.

[1]Presented at the AOCS Meeting, Dallas, April 1975.

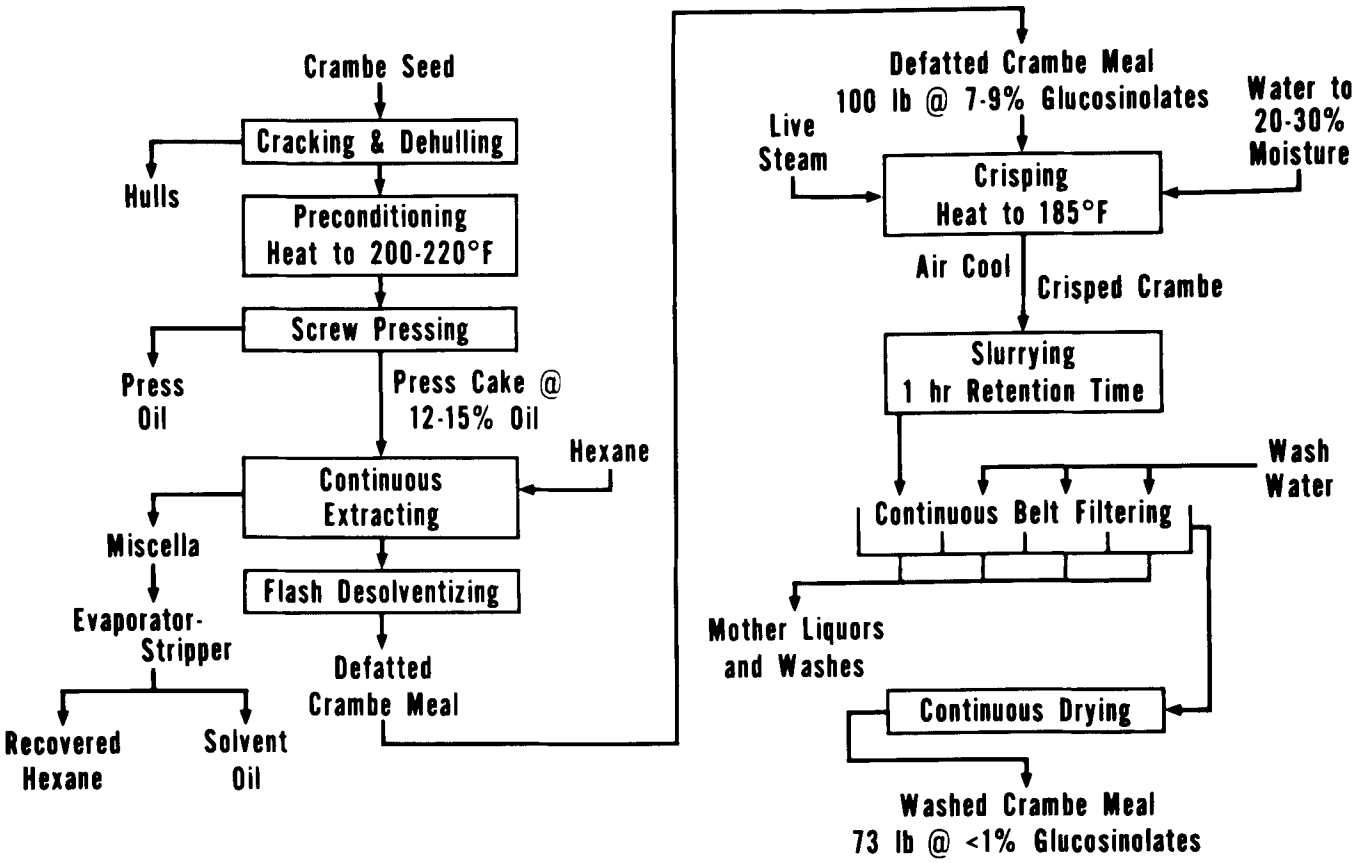

FIG. 1. Flow sheet – continuous water extraction of crambe glucosinolates.

Equipment and Procedure

Crambe seed was cracked on 6-in. diameter rolls with 10 corrugations per inch set at 0.025-in. clearance (Fig. 1). Hulls were aspirated from a 0.1065-in. round hole vibrating screen. The dehulled seed was metered to a two-stage jacketed paddle conveyor, which served as a preconditioner for the screw press. The seed, heated to 210 F, entered a continuous screw press, manufactured by Fuji Bunka Company Ltd., Tokyo, Japan. The press removed about two-thirds of the oil. The press cake containing about 15% oil was fed to a 20-stage countercurrent Kennedy extractor. The cake was extracted with hexane at 140 F to a residual oil content of less than 1%. The defatted meal was flash desolventized.

The defatted meal was toasted in 15- or 150-lb charges in either a 1- or a 10-cu ft blendor. The jacketed, horizontal blendor with double ribbon flights had a removable cover; the variable speed agitator shaft had sparge holes for live steaming. For crisping, the meal was heated in a stirred ribbon blendor with indirect steam to 185 F and sprayed with hot water to increase the moisture to 20 to 30%, followed by live steaming and cooling. Continuous filtrations were run in an Ametek 6-sq ft horizontal cloth belt filter. The self-cleaning belt was driven by a variable speed motor. The filter had four separate vacuum sections separated by adjustable dams – one for dewatering and three for washing and dewatering. Each section had its own vacuum receiver and filtrate pump.

Filtration rates were determined on a bench-scale test unit similar to that described by Graci et al. (21); it consisted of a stainless steel filter funnel, a 4000-ml vacuum flask, a Cartesian manostat for vacuum control, and a mercury manometer (Fig. 2). The filter medium was made up of belt filter cloth flanged between two rings. The effective filtering area was 0.15 sq ft. All bench-scale filtrations were carried out at 4-in. Hg, a pressure which approximated the vacuum obtained on the belt filter at steady state during a continuous run.

RESULTS AND DISCUSSION

Several processing steps are needed in defatting and water washing the crambe meal (Fig. 1). A water slurry of the powdery defatted meal behaves like a thin paste. When such a slurry is filtered, it quickly plugs the filter medium and causes the filtration rate to drop rapidly to zero. Cooking the defatted meal in the presence of added moisture and live steaming followed by air cooling agglomerates the fines and improves filterability. This crisping step, which had proved adequate in our previous work with batch filtrations (8,22), had to be optimized for continuous filtrations. Unless the meal slurry had a filtration rate of at least 2000 lb/hr-sq ft, flooding occurred on the belt and sequential washing was impossible. In scanning electron microscope photographs (Fig. 3), we can see how the small fuzzy particles of defatted crambe are fused into large, glazed, spongy, cellular structures when crisped. Trial crisps were made in 15-lb charges in a 1-cu ft ribbon blendor identical in all respects, except size, to the 10-cu ft ribbor blendor.

Processing Variables

Crisping: Defatted crambe meal in 15-lb charges was crisped in the Day 1-cu ft ribbon blendor to determine the effect of moisture, agitation speed, live steaming time, and dry-out time on bench-scale filtration rates. The conditions in run 3 were selected as the base line.

(a) *Moisture:* In the first four runs moisture was varied. Filtration rates were best at 20% moisture. Both higher and lower moistures gave lower rates (Table II).

(b) *Live steaming:* We found the filtration rates were fastest for 20 min of steaming. Increasing the time of live

steaming from 20 to 40 min (run 6) had a slightly negative effect on filtration rate. When live steaming was eliminated (run 5), the filtration rate was reduced by 95%.

(c) *Agitation:* Slow agitation is important in the agglomeration process. The slowest speed setting was 4 rpm. In run 9 which was made at the next lowest setting (16 rpm), the rate was reduced by ca. 85%.

(d) *Dry-out time:* Filtration rate is not significantly affected by dry-out time in the blendor if moisture loss is minimized by restricting the amount of venting to the atmosphere. When the cover was removed during dry out (run 10, Table II), the filtration rate was approximately halved.

The optimal conditions for crisping to give the best filtration rates were slow agitation, 20% moisture, 20-min live steaming, and 60-min dry-out with the blendor cover on.

Extraction efficiency: Since the quantity of crambe seed available for the experiments was limited and since a pilot-plant run consumed roughly 500 lb of seed, many of the variables were explored on the bench-scale setup.

In a series of bench-scale experiments, effects of slurry time and of temperature on solids extraction were studied. Crisped crambe meal was slurried with four parts of water, filtered, and washed with nine volumes of water. Temperatures between 60 and 120 F were found to be not too critical – solids loss varying by only 1.6%. Pilot-plant runs were made at ambient temperature (80 ± 5 F) (Table III). Doubling the slurry time from 0.5 to 1.0 hr increased solids loss slightly (1.4%), but a second doubling from 1 to 2 hr had only a negligible effect. Slurry time in the pilot plant was set at 1 hr. Glucosinolate removal was 98% or more in all experiments.

Belt filter operation: At 3:1 water:meal ratio, the slurry was too thick and plugged the flow splitter on the belt filter feeding sytem. Slurry consistency was generally good at a 4:1 water:meal ratio. Slurry feed rate to the belt filter was kept constant, retention time was varied by belt speed, and the ratio of wash water to the meal on the belt was varied from 3:1 to 6:1. Retention time on the belt made no significant difference on residual glucosinolates in the washed meal (Table IV). The 6:1 wash water:meal ratio was the maximum that could be used without flooding. Reducing the wash water to a 3:1 ratio increased residual glucosinolates in the washed meal from 0.3 to 0.6%.

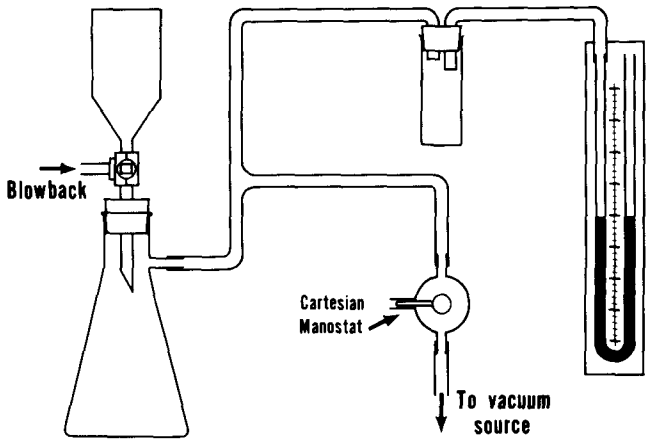

FIG. 2. Bench-scale filtration setup.

Quality of Water-Washed Crambe

Quantities of meal sufficient for rat and chick feeding studies have been prepared, and the analyses of these meals for the feeding trials are shown in Table V. Protein content increases from ca. 46% in the unwashed to 49% in the washed meals. About 85% of the sucrose and dextrose are removed by the extract. Total glucosinolates are reduced by 92 to 96% in the washed meals. Available lysine, a good indicator of heat damage during processing, remained fairly high in the range 4.9 to 5.0% compared to 5.5% in the starting defatted meal. Free nitriles are low (0.04% or less) in the processed meals. Free goitrins varied from 0.06% to zero in processed meals. Myrosinase activity, which was positive in defatted crambe, is negative in the crisped and water-washed meals. The most heat in the crisping step effectively inactivates the enzyme. About half of the undenatured nitrogen in the defatted meal is denatured in the crisping step as indicated by the NSI values. The water-washed meals have less than 4% soluble nitrogen.

Extracted Solids

Analysis of the freeze-dried extracts shows fairly good agreement in extract composition between bench-scale and

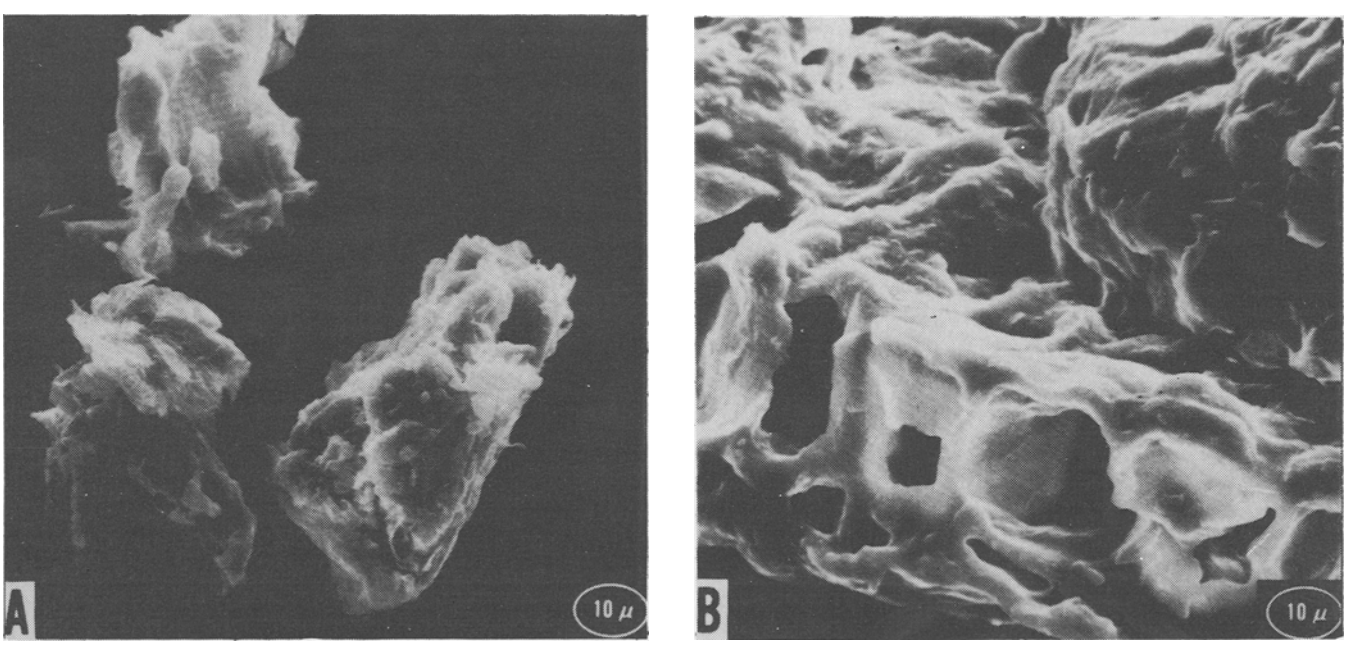

FIG. 3. Scanning electron microscope photographs of defatted crambe meal before (left) and after (right) crisping (1000x).

TABLE II

Effect on Filtration Rate of Processing Variables During Crisping in a 1 cu-ft Ribbon Blendor Agitated at 4 rpm

Run number	Moisture (%)	Live steam (min)	Dry out (min)	Filtration rate (lb/hr-ft^2)
1	15	20	60	860
2	20	20	60	2300
3	25	20	60	1620
4	30	20	60	1180
5	25	0	60	77
6	25	40	60	1320
7	25	20	30	1330
8	25	20	90	1220
9[a]	25	20	60	200
10	25	20	60[b]	1080

[a]Agitation rate for this run only was 16 rpm.
[b]Blendor cover removed for dry out.

TABLE III

Influence of Slurry Time and Temperature on Extracted Solids

Test number	Slurry time (min)	Slurry temperature (°F)	Solids in extract (%)	Glucosinolates extracted (%)
BS-1	60	60	26.7	98
BS-2	60	80	27.5	98
BS-3	60	120	28.3	98
BS-4	30	80	26.0	98
BS-5	120	80	28.1	99

pilot-plant runs (Table VI). Ash content is ca. 14%. Oil and fiber content are less than 1%. Glucosinolates are 13.3 to 14.1%. Dextrose and sucrose account for about one-third of the extracted solids. Protein loss to the extract is in the neighborhood of 18%. The balance of the extracted solids consists mainly of carbohydrates. Glucosinolates might be separated from the extract by ion exchange (unpublished data) to upgrade this fraction to a feed supplement; however, if this should prove impractical, then the cost estimate would have to be revised to include the cost of waste treatment of the extract.

Cost Estimate

A preliminary cost extimate for water-washing defatted crambe meal in a plant processing 200 tons of whole crambe seed daily shows that the processing costs for the washing operation would be in the order of $22-23 per ton of unwashed defatted meal, in addition to the cost of crushing seed to oil and meal. In the estimate, it was assumed that the meal-washing operation would be conducted as an adjunct to an existing crambe processing plant operating 24 hr per day, 300 days per year. Operating procedures in the hypothetical plant upon which the estimate is based follow, in general, those depicted in the flowsheet (Fig. 1).

TABLE IV

Effect of Belt Filter Retention Time and Amount of Wash Water on Extraction of Glucosinolates

Test number	Retention time (min)	Cake thickness (in.)	Wash water:meal ratio	Residual glucosinolates (%)
PP-1	7.2	5/8	3:1	0.6
PP-2	7.0	3/8	3:1	0.7
PP-3	7.2	5/8	6:1	0.3
PP-4	5.0	3/8	6:1	0.2

The estimated fixed capital investment for an installation that includes only the equipment directly associated with producing a dried, defatted, water-washed meal is about $775,000. A breakdown of the processing costs for producing water-washed meal includes utilities ($6.45), labor and supervision ($9.63), maintenance ($1.74), and fixed charges which include depreciation, taxes, and insurance ($4.62). These items add up to $22.44 to process 1 ton of unwashed defatted meal. Selling costs, administrative expenses, and profit are omitted as part of processing costs.

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TABLE V

Analyses of Defatted Crambe and Meals Prepared for Feeding Trials

Item	Defatted crambe	Crisped crambe	Water-washed crambe-1	Water-washed crambe-2
Approximate composition				
Moisture, %	7.1	3.0	5.2	4.4
Ash, %	8.4	8.5	9.6	10.5
Oil, %	0.7	0.7	0.8	0.8
Crude fiber, %	6.5	6.7	8.3	9.0
Protein (N x 6.25), %	45.5	47.9	50.7	48.0
Total glucosinolates[a], %	7.9	5.6	0.6	0.3
Sucrose, %	9.8	9.6	1.2	1.6
Dextrose, %	3.3	2.9	0.5	0.4
Other carbohydrates (by difference), %	10.8	16.1	24.7	23.8
	100.0	100.0	100.0	100.0
Available lysine, %	5.5	5.1	5.0	4.6
Myrosinase activity	Positive	Negative	Negative	Negative
Nitrogen Solubility Index, %	50.5	25.3	2.4	3.8
Free goitrin, %	—	0.06	0.04	0
Free nitrile, %	—	0.04	0.03	0.03

[a]As *epi*-progoitrin sodium salt.

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