Prepress-Solvent Extraction of Crambe: First Commercial Trial Run of New Oilseed[1]

G. C. MUSTAKAS, Northern Regional Research Laboratory,[2] Peoria, Illinois, and
G. KOPAS and N. ROBINSON, Pacific Vegetable Oil Corporation, San Francisco, California

Abstract

Commercial-scale equipment was used to process experimentally 36 tons of *Crambe abyssinica* seed grown in the western part of the United States to obtain information on the processing of this new oilseed and to determine characteristics of the oil and meal products. The run was carried out for USDA at the Pacific Vegetable Oil Corporation plant at Sidney, Nebraska, February 1964.

Process objectives in the study included dehulling, primary oil removal by expeller prepress, secondary oil removal by solvent extraction and control of thioglucosides to obtain good oil quality. A continuous plant operation yielded crude oils and toasted meal that will be compared with similar products from other commercial oilseed processes.

Characteristics of the crude oils that have been determined are composition, refining losses and hydrogenation ability. Organoleptic data on the refined, bleached and deodorized oils have been obtained, as well as compositional data on the desolventized-toasted meals. More than 13 tons of meal and 10 tons of oil were prepared.

Introduction

CRAMBE ABYSSINICA, a member of the family Cruciferae, is an oilseed that has been selected under the USDA screening program as having excellent potential for development as a new industrial oil crop. The purpose of this program is to uncover seeds containing unique oils suitable for industrial use and noncompetitive with those now in production. Crambe, an annual herb, has already proved suitable as a crop in North Central and Northwestern States. Limited plantings have been carried out and good yields reported by the Crops Research Division, ARS, USDA (10).

Crambe oil is similar to rapeseed oil in composition but contains more erucic acid. A typical composition of crambe oil is 54–59% erucic, 16–20% oleic, 8–10% linoleic, 4–8% linolenic and miscellaneous fatty acids. Erucic acid oils are useful as lubricants or for conversion to rubber additives by crosslinking with sulfur. Research on erucic acid and its derivatives should lead to numerous other expanded applications in the fields of plasticizers, resins, fibers, coatings, waxlike materials and others. Crambe oil meal shows promise as a protein supplement in animal feeds.

For several years, development of a suitable process to recover oil and meal from crambe seed has been underway at the Northern Regional Research Laboratory. Information obtained from these studies was used to establish conditions for the commercial processing of 36 tons of crambe seed in a prepress-solvent plant. During February 1964, the trial run was carried out at Sidney, Nebraska, for the Northern Laboratory by the Pacific Vegetable Oil Corporation. The processing operations are detailed in this paper as well as an evaluation and the characteristics of oil and meal products obtained.

Experimental

Materials

Crambe seed was grown by the Crops Research Division, ARS, USDA, under contract during the 1963 season in the States of Oregon, Idaho, Montana, Wyoming and Colorado. Whole seed from the various accessions was blended and analyzed in percent: moisture 8.1, oil 32.5, protein (N ×

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[2] A laboratory of the No. Utiliz. Res. & Dev. Div., ARS, USDA.

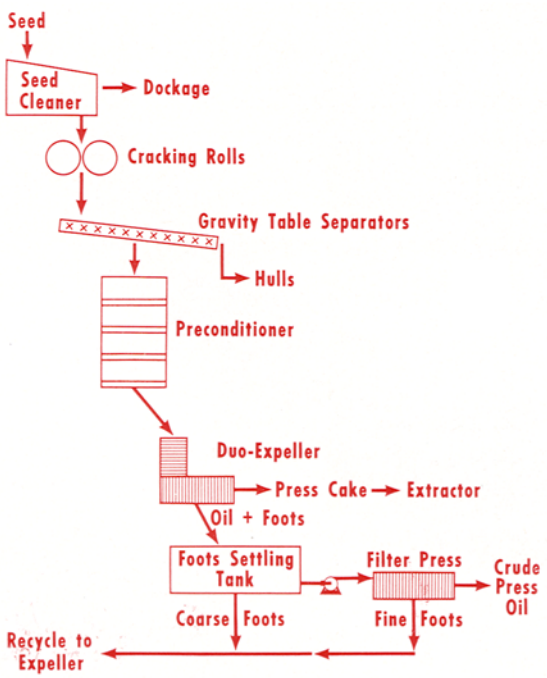

FIG. 1. Phase 1 of the commercial trial run: cleaning, dehulling and prepressing crambe seed.

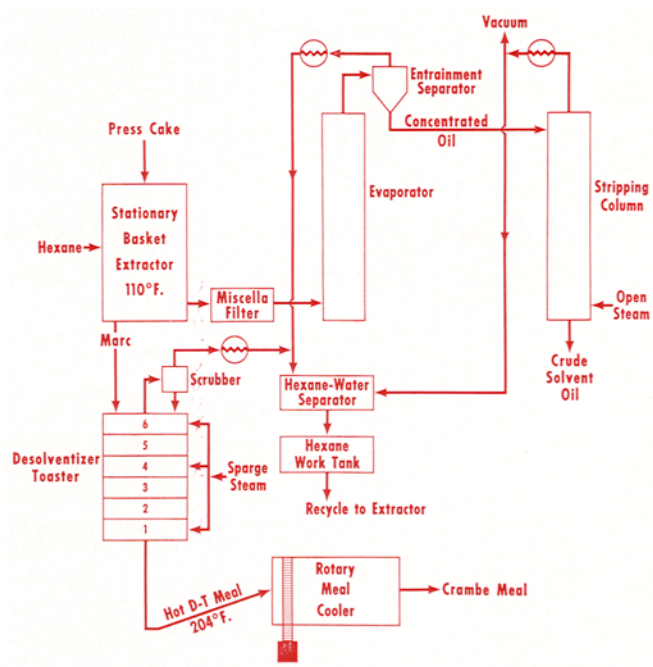

FIG. 2. Phase 2 of the commercial trial run: secondary oil recovery from crambe seed.

TABLE I

Total Sulfur and Thioglucoside Content of Crambe Seed Fractions During Processing

Material	Moisture-free, fat-free basis, %			
	Total sulfur	Total thioglucoside	Conversion products after enzymatic hydrolysis[a] of total thioglucosides	
			R-5-vinyl 2-thiooxazolidone	Volatile isothiocyanate[b]
Original seed	1.4	7.7[c]	1.47	0.1
Dehulled meats	2.0	10.2[c]	2.13	0.1
Hulls	0.33	2.7[c]	0.00	Trace
Press cake	...		2.18	0.1
Toasted meal	2.0	9.2[d]	1.78[e]	0.1

[a] Wetter analytical procedure (3).
[b] Calculated as allyl isothiocyanate.
[c] By sulfate ion analysis (4).
[d] By UV absorption (3).
[e] No free (unbound) thiooxazolidone found by analysis.

6.25) 19.7, crude fiber 14.3, ash 5.0 and nitrogen-free extract 20.5. Sieve analysis of crambe seed and meal was:

Cumulative percentage retained	U.S. standard sieve number								
	3	4	6	8	10	14	20	30	Pan
Seed			5.0	70.8	86.5	97.5	99.8	100.0	
Meal	13.0[a]	20.5[a]	27.8[a]		39.0		75.8	88.3	100.0

[a] Oversize material arising from ball agglomerates produced in the desolventizer-toaster unit.

Standard commercial-grade hexane was used to extract the press cake.

Methods of Analyses

A Beckman Spinco MS amino acid analyzer was used for determination of amino acids by the method of Spackman, Stein and Moore (9); fatty acid compositoin by gas-liquid chromatographic (GLC) analysis of methyl esters (6); total sulfur by the method of Shaw (7); volatile isothiocyanate by the method of Wetter (12); total thioglucosides by sulfate ion and UV absorption (5); thiooxazolidone by a modified Wetter procedure (5); nitrogen solubility index (NSI) for the measurement of water-soluble protein by a modified method of Smith and Circle (8). Other tests unless otherwise designated were analyzed by standard AOCS procedures (1).

Procedures and Equipment

Seed cleaning, cracking, dehulling, preconditioning and expelling were carried out continuously as the first phase of operations over a period of approximately 22 hr. The second and last phase consisted of solvent extraction of press cake, miscella evaporation, oil stripping and oil meal desolventizing-toasting. This last phase was run continuously over an 8-hr period.

Figure 1 presents a flowsheet of the first-phase operations. Whole crambe seed from storage was fed to a double-screen seed cleaner for separation of dockage. The clean seed was then cracked in corrugated rolls and passed over gravity table separators to separate the hulls.

Decorticated whole and split kernels were conveyed to a five-deck preconditioner situated on a superstructure above the expeller. This unit was used to heat-condition the meats before they passed to the Anderson duoexpeller below for pressing out the oil. Press cake was accumulated and stored temporarily before conveying to the extractor building. Oil

TABLE II

Analyses of Crambe Process Fractions

Constituent	Whole seed	Kernels	Hulls	Meal
	%	%	%	%
Moisture	8.1	6.1	13.5	11.3
Oil	32.5	42.7	1.8	4.0
Protein (N × 6.25)	19.7	25.2	6.9	39.0
Crude fiber	14.2	4.0	40.4	9.1
Ash	5.0	4.5	5.3	7.6
NFE	20.5	17.5	32.1	29.0
	100.0	100.0	100.0	100.0

TABLE III

Physical and Chemical Properties of Crambe Oils

Analysis	Prepress oil		Solvent oil	
	Crude	Refined–bleached	Crude	Refined–bleached
General				
Moisture	0.04	0.02	0.018	0.05
Density, 25C		0.9394		0.9408
Refractive index, 20C		1.4713		1.4719
Melting point, C		6.5		6.0
Viscosity, 25C				
Centipoise		85		85
Gardner		C		C
Ash, %		0.005		0.020
Phosphorus, %	0.005	0.0004	0.075	0.006
Sulfur, %	0.031		0.026	
Unsaturation				
Iodine value		93.0		96.1
IV after hydrogenation		0.9		1.0
Fatty acids				
Free-fatty acids				
% as oleic	0.62	0.29	1.85	0.14
Saponification value				
Mg KOH /g oil		173		173
Nonglyceride constituents				
Unsaponifiable matter, %		0.47		0.59
Break, %	0.14	0.02	0.40	0.01

and foots leaving the expeller were conveyed to a large foots settling tank where a slow moving dragline settled out the coarse foots. The decanted oil from this unit was pumped to a filter press for separating the "fine foots" (dispersed phase) and getting a clean press oil.

Press cake was transferred pneumatically to the solvent extraction building. A flowsheet of the extraction operations is shown in Figure 2. In the French extractor (a round, stationary basket type), 12 sector-shaped cells were filled with solids, one at a time, by a rotating conveyor. The meal was fed continuously as a slurry in miscella. In each cell the meal formed a bed supported by hinged screen doors at the bottom. Four miscella washes followed by pure hexane were sprayed over the solids countercurrently by rotating feed distributors that followed the solids feeder. By this means each cell was flooded with successive miscella washes of gradually decreasing oil concentration. The miscella draining from the cells collected in ring-shaped troughs or compartments from which it was withdrawn and advanced countercurrently by stage pumps. Finally, the flakes were sprayed with fresh solvent and permitted to drain before they were discharged. Extraction for a 360° cycle required approximately 45 min. Spent meal was conveyed directly to a desolventizer-toaster (D-T).

Full miscella leaving the extractor was pumped through a leaf-pressure filter and then pumped to an evaporator. In this step, miscella was fed at the bottom of the evaporator unit to long vertical tubes that were heated indirectly by a steam tube chest. In a single pass miscella reached a concentration of approximately 95% oil content before leaving at the top of the evaporator unit through a vapor-entrainment separator. The oil concentrate was finally passed through a bubble-cap stripping column operated under vacuum. As oil flowed downward by gravity, open steam fed at the bottom of the column stripped the remaining hexane free from the oil.

Marc leaving the extractor entered the top of the D-T unit, which was operated at atmospheric pressure and with both open and indirect steam. Sparge steam was used in three of the trays (Fig. 2). Desolventized and toasted meal

TABLE IV

Fatty-Acid Composition of Refined–Bleached Crambe Oils

Carbon-double bond ratio	Blended oils [a]
14:0	0.3
16:0	2.0
16:1	0.3
18:0	0.7
18:1	15.0
18:2	10.4
18:3	6.6
20:0	0.7
20:1	3.0
20:2	0.1
22:0	1.6
22:1	55.0
22:2	0.2
24:0	0.7
24:1	3.2

[a] Blended, refined and bleached in commercial equipment by Pacific Vegetable Oil Corporation, Richmond, Calif. Ratio of prepress/solvent oils = 1.25:1.

TABLE V

Refining Data for Prepress and Solvent Crude Oils

Data on crambe oils	Prepress oil	Solvent oil
Free-fatty acid content, %	0.62	1.85
Sodium hydroxide required, g/100 g oil	0.08	0.21
Excess NaOH used, g/100 g oil	0.2	0.2
Degumming loss, %	0.5	3.7
Color index		
Crude oil, Gardner	11.0	11.5
Refined-bleached oil, Gardner	1	3
Refined-bleached oil, AOCS	2.44	4.46
Yield of refined oil, %	98.0	91.8

was discharged at the bottom and was conveyed into a rotary, kiln-type meal cooler. The toasted meal was finally pneumatically conveyed to bin storage before sacking in 100-lb burlap bags.

Control of Thioglucosides for Product Quality

On a moisture-free, fat-free basis, this crambe seed contained 7.7% thioglucosides or 10.2% on a dehulled basis (Table I). Enzymatic hydrolysis of these compounds results in their conversion to aglycones, which are carried into the oil fraction upon solvent extraction of the meal. For this reason, processing variables that contribute to the hydrolysis were controlled in order to suppress thioglucoside hydrolysis and to obtain a sulfur-free oil; also temperatures were minimized to obtain a good protein quality in the feed meal. Even a slight sulfur contamination of the oil can result in poisoning the hydrogenation catalyst.

Results and Discussion

Material Balance

The seed processed and the products obtained from it were as follows:

	In pounds		
Gross crambe seed		71,660 IN	
Hulls (+ dockage)	15,874		
Toasted meal	27,200		
Prepress crude oil	11,350		
Solvent crude oil	9,092		
Foots cleanup	3,240	66,756 OUT	4,904 = 6.8% loss

Dehulling for a High-Protein Meal

Seed cleaning removed empty hulls, large stems, large foreign seeds, small weed seeds and fines. Setting the clearance between the corrugated rolls at 0.063 resulted in splitting the loose outer pericarp from the whole kernel. Figure 3 is a photograph of the whole seeds, kernels and partially cracked hulls.

Operation of the gravity table separators went smoothly to give a good separation of hulls and meats in a single pass. Approximate analyses of the whole seed, kernels and hulls are presented in Table II. Hull content of the clean whole seed, estimated by cracking and dehulling this fraction in a Bates laboratory asperator, ran 30.6%. The low oil content in the hull fraction (Table II) indicated that a good separation was obtained with a minimum contamination of fine meats.

Primary Oil Recovery by Prepressing

In preconditioning dehulled meats for the expeller, the heat treatment was minimized and moisture eliminated. Previous laboratory tests indicated that a wet-cook treatment gave good expeller operation; however, moisture was eliminated in this run to minimize enzymatic hydrolysis as discussed earlier. Without elevated temperatures in the expeller, however, poor pressing of the meats resulted. Consequently, to develop initial heat in the expeller, the feed was switched temporarily from dehulled to whole crambe

TABLE VI

Dried Lecithin Composition [a]

Source of lecithin in crambe oil	Acetone insoluble	Benzene insoluble	Phosphorus
	%	%	%
Prepress	14.7	.....	0.60
Solvent	52.9	0.74	1.54

[a] Lecithin fraction prepared from crude oil by water degumming, centrifuging gums and vacuum drying at 140F for 3 hr.

TABLE VII

Flavor Evaluation of Crambe Oils

Oil	Flavor score [a]					
	Initial			After 4-day storage [b]		
Prepress crambe [c]	7.7	7.2		6.9	6.3	
Solvent crambe [c]	7.4		7.2	6.5		5.3
Soybean		8.1	8.1		6.5	5.2
Significant difference		x				

[a] Range 1–10 units with 10 rated best.
[b] At 140F in presence of air.
[c] Deodorized with 0.01% citric acid.

seed. The presence of the hulls gave better expeller operations and built up frictional heat in the unit rapidly. In addition, indirect steam was introduced in the lower trays of the preconditioner so that the feed material entered the expeller at 125F. These modified conditions greatly improved expeller operation as demonstrated by a clean break between the oil and foots leaving the expeller. After approximately 1 ton of whole seed passed through the unit, the feed was switched back to the original dehulled material.

Press-cake grab samples obtained near the end of the run analyzed 17.2% residual oil. In normal operations it would be desirable to reduce the amount of residual oil to a range of 12–15%. A low free-fatty acid (0.6%) in the press oil indicated that lipase activity was under good control during processing.

Secondary Oil Recovery by Solvent Extraction

The first press cake fed to the extractor was somewhat dry (8% H_2O) and crumbled readily to give some fines. Processing this material resulted in poor percolation rates and consequent flooding in the extractor. When the fines were agglomerated by a small addition of moisture to the press cake, the percolation rate was much improved, and a good extractor operation was obtained.

Grab sample analyses of the toasted meals showed that when nearing the end of the run, residual oil value was reduced to 2.8% although the composite average oil content of the meal was 4.0% (Table II).

Characteristics of Oil Products

Composition and properties of the crude and refined-bleached oils from both pressing and extraction operations are shown in Tables III and IV. Refining data are shown in Table V and lecithin compositions in Table VI. Viscosities of the oils are significantly higher than soybean oil. The refined-bleached prepress oil is somewhat lighter in color than soybean oil.

Refining of the oils was accomplished without any special problems. Refining the solvent oil resulted in considerably higher losses (8.2%) than for the prepress oil (2.0%). These losses can be attributed to higher phosphatide and free-fatty acid content, as well as other undetermined factors. Good bleachability of the oils was shown by their low color values.

Composition of the dried lecithins indicates again the higher phosphatide composition of the solvent oil as demonstrated by the acetone insolubles and phosphorus content.

Good hydrogenation ability of both the prepress and solvent oils was demonstrated since both oils were reduced to iodine values of one or less by hydrogenation with 0.1% nickel catalyst for 4 hr at 347F, 100 psig. These iodine values indicate that neither oil was contaminated by sulfur through hydrolysis of the thioglucosides in the crambe seed.

Initial organoleptic flavor scores (Table VII) showed no significant difference between the prepress and solvent oils (7.7 and 7.4) when compared with each other. At zero time when the prepress crambe oil was compared with the soybean oil control, a significant difference (8.1 vs 7.2) was shown in favor of the soybean oil. When the solvent crambe and soybean oil were compared at zero time, a larger test error was obtained (0.33), but no significant difference between the oils was found. After 4 days' storage at 140F there were no significant differences between either of the crambe oils and the soybean oil control. Oxidation indices of the oils are shown in Table VIII. The peroxide values

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TABLE VIII

Oxidation Indices

Product from crambe oils	Peroxide value	AOM stability PV after 8 hr at 100C	Hydroxyl value mg KOH/g oil
Prepress			
Refined, bleached	1.9		1.87
Refined, bleached, deodorized	0.33	2.29	
Solvent			
Refined, bleached	1.0		3.08
Refined, bleached, deodorized	0.10	3.83	

determined after 8 hr under AOM conditions indicate a stable oil, and this property was confirmed by the taste panel.

There seems to be little difference in mouth feel between crambe and soybean oil especially if warmed for flavor evaluation. Many tasters remarked that they thought the crambe oil was soybean oil.

A refined, bleached deodorized crambe oil was prepared in the laboratory subsequent to the commercial run under conditions where none of the processing operations were interrupted to provide intermediate oil or meal storage. With this oil, the excellent organoleptic flavor scores (8.3–8.8) obtained were equal to soybean oil, both at zero time and at 4 days' storage at 140F. On the basis of this study, it appears that a straight (uninterrupted) commercial run should produce a high-quality crambe oil.

Sulfur and Thioglucoside Content of Crambe Fractions

Table I gives the sulfur contents and total thioglucoside content of the original seed, dehulled meats, hulls, press cake and toasted meal fractions. Thiooxazolidone [(R)-goitrin] (2) and volatile isothiocyanates after enzymatic hydrolysis of thioglucosides are given in the last two columns of figures. The higher values for dehulled meats over the original seed reflect the concentration effect due to hull removal. A thioglucoside content of 2.7% for the hull fraction was higher than found in previous laboratory hull samples. Essentially all the thioglucosides remain unchanged in the press cake as indicated by the thiooxazolidone values (2.13 vs 2.18). However, a small decrease in thiooxazolidone precursor was obtained in processing from press cake to the desolventizing-toasting step (2.18 vs 1.78). Total sulfur content in toasted meal remained the same as in dehulled meats (2.0 vs 2.0).

These data show that in the entire process only a small amount of thioglucosides were converted. Based on thioglucoside contents of dehulled meats and toasted meal, this conversion calculates to approximately 10% or by (R)-goitrin analysis, to approximately 16%. Since no free (R)-goitrin was found in the toasted meal it is conceivable that the portion of thioglucoside hydrolyzed was not progoitrin.

Meal Characteristics

The medium brown meal had a good toasted aroma. Good lysine and balance of other essential amino acids were obtained in the assay of the meal (Table IX). These analyses are within experimental error of the average values previously reported from five other accessions of crambe seed meal analyzed at the Northern Laboratory. Sieve analysis of the meal is given in the Experimental Section. The large fractions from 3–6 mesh consisted of balls or lumps formed during the toasting operation. Normally in commercial operations such agglomerates would be screened out, reground and recombined with the remaining fractions. Proximate analyses of the meal are given in Table II. In commercial operations residual oil content should be reduced to under 1%, and this reduction would raise the protein of the meal to over 40%. Nitrogen solubility index of the meal was not greatly reduced in value during processing. Dehulled meats analyzed 52.2 NSI; after processing the meal analyzed 42.5 NSI, and after solvent extraction, desolventizing and toasting the meal was reduced to 32.5 NSI. The relative small decrease in NSI for a toasted meal indicates that the heat treatment was not excessive and probably did little to no damage to the heat-labile amino acids. Previous chemical characterizations of crambe meals have been reported (11).

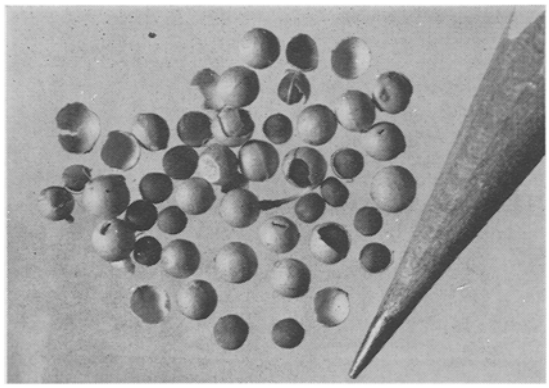

FIG. 3. Whole crambe seeds, kernels and partially cracked hulls. The pencil indicates comparative size.

TABLE IX

Amino Acid Composition of Toasted Crambe Meal

Amino acid	G/16 g nitrogen	Amino acid	G/16 g nitrogen
Lysine	5.3	Arginine	6.4
Methionine	1.6	Glycine	4.9
Isoleucine	3.7	Alanine	4.1
Leucine	6.1	Aspartic acid	6.3
Phenylalanine	3.7	Glutamic acid	15.5
Tyrosine	2.7	Hydroxyproline	0.4
Threonine	4.1	Proline	6.1
Valine	4.7	Serine	3.6
Histidine	2.3		

% Nitrogen as amino acids	72.1
% Nitrogen as ammonia	13.5
% Crude protein (Kjeldahl N × 6.25) air-dry meal	41.3
% Residual oil, as-is basis	4.0

Conclusions

Based on the results of this first commercial run, suitable conditions have been developed to extract oil from crambe seed by a prepress-solvent process. Based on chemical, physical and organoleptic analyses of the crambe oil the quality was good. Amino-acid composition of the crambe meal indicates that it should have potential as a protein supplement in animal feeds, but actual animal feeding tests will be required to assess its practical feeding value. Meals prepared in this run are being evaluated now by ruminant feeding. Further studies at the Northern Laboratory are aimed at developing crambe meals suitable for use as a protein supplement for both ruminants and nonruminants (3,4).

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