Processing of *Crambe abyssinica* Seed in Commercial Extraction Facilities

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ABSTRACT

Crambe abyssinica seed was processed in four commercial oilseed crushing facilities, two utilizing prepress solvent extraction and two utilizing straight solvent extraction techniques. Mill capacities ranged from several T/day to 200 T/day. Crambe throughput in the larger facilities ranged from 30 to 150 T/day. Seed, press cake, flakes and finished meal samples were collected and analyzed during and following the runs. On-site testing included measurements of moisture, oil, glucosinolate and temperature and estimates of thioglucosidase enzyme activity. Three to 7 T of defatted meal were produced for each of four beef cattle feeding studies, and oil produced was blended into commercial erucic acid production streams. Except for one run, thioglucosidase inactivation had to be completed in the desolventizing/toasting (DT) unit, and the high temperatures required resulted in destruction of glucosinolate and the formation of aglucon product, 1-cyano-2-hydroxy-3-butene, and glucose in the finished meals. Protein solubility and lysine levels decreased with excessive heat. Regression analysis was used to examine some of the data for relationships between temperature, moisture, glucosinolate, nitrogen solubility and aglucon products. The results of these runs further demonstrate the feasibility of processing Crambe in commercial oilseed facilities.

INTRODUCTION

Crambe abyssinica Hochst ex R. E. Fries is a good source of an industrial oil having high erucic acid content and of a seed meal with good quality protein (Table I). Over the past 20 years, as research by USDA and others explored the potential of Crambe as a domestic oilseed crop, significant quantities of Crambe seed were produced and opportunities arose for investigating oil extraction and processing characteristics of Crambe seed in cooperation with industry and academia. Problems associated with these opportunities, however, were 4-fold: (a) finding suitably sized, equipped and available facilities; (b) applying scientific techniques to the processing runs; (c) producing marketable oil and meal, and (d) maintaining some semblance of an economical process during limited operations.

Few oilseed mills were sized (50-200 T/day) or equipped for preparing Crambe seed for oil extraction, and limitations in equipment and overall plant design meant that establishing conditions for steady-state processing would be difficult in short runs. The relatively small quantities of seed available (<1000 T) meant that the mills would be operated at sub-optimum levels, which diminished the chances for economical operations. Nevertheless, over the years commercial production of Crambe was achieved, and seed was processed on at least 8 occasions in 5 different mills. Only the earliest run at Sidney, Nebraska, has been reported (1). Although highly successful, this first run was conducted in two separate stages (pressing later followed by solvent extraction) on a small quantity of seed (36 T).

In this paper we present information obtained in the other, later runs, define important processing objectives in terms of laboratory or pilot-plant experience, and conclude with some specific recommendations toward a protocol for commercial processing of Crambe seed. Such a processing protocol is required to ensure inactivation of enzymes responsible for conversion of glucosinolates in the seed to toxic materials in the finished seed meal during oil extraction.

TABLE I

Average Reported Composition of Crambe Seed and Defatted Meals (Dry Basis)[a]

Constituent	Whole seed		Dehulled seed	
	%	Defatted %	%	Defatted %
Oil	35.3	–	46.5	0.9
Protein (N × 6.25)	20.1	31.1	25.8	48.7
Crude fiber	14.3	22.1	3.6	6.7
Ash	4.8	7.4	4.5	8.6
NFE[b]	25.4	39.3	19.6	35.6
Glucosinolates, μMoles/g	–	105-164	–	187-234

[a]Carlson and Tookey (10) (Adapted from Mustakas and coworkers [1,29,31]; Kirk and coworkers [28,30]; Baker et al. [32]).
[b]Nitrogen free extract by difference.

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EXPERIMENTAL PROCEDURES

Inactivation of Thioglucosidase in Whole Crambe Seed

The following are illustrations of many experiments that have been conducted to define conditions for inactivating thioglucosidase in Crambe seed. The major glucosinolate in Crambe is *epi*-progoitrin [(S)-2-hydroxy-3-butenylglucosinolate], sometimes abbreviated *epi*-PG. In the laboratory, whole Crambe seed was equilibrated at ambient temperatures for 65 hr in constant humidity chambers to moistures of 6.1, 8.3, 9.2, 11.0 and 13.6%. Equilibrated seed samples (10 g) were sealed in test tubes and heated at 180 F for exactly 30 min. Seed was then quickly cooled, air dried, crushed and defatted with petroleum ether. The defatted meals were then quantitatively tested for thioglucosidase activity by measuring HSO_4^- release from *epi*-progoitrin according to the method of Tookey (2). The results are shown in Figure 1.

In the pilot plant, whole Crambe seed was tempered at the rate of 65 lb/hr in a 2-stage horizontal paddle conveyor. The upper stage fed the lower stage, and both were fitted for jacket and/or sparge steam. Total residence time was ca. 20 min. Seed leaving the unit was conditioned at different temperatures and moistures, then sampled and analyzed for moisture and glucosinolate contents and for thioglucosidase activity. The TesTape® method of VanEtten et al. (3) (glucose-sensitive TesTape®, Eli Lilly and Co., Indianapolis, Indiana) was used to check for thioglucosidase activity by wetting a piece of TesTape® with a slurry made from 40-60 mg meal sample and 1 ml pH 7 phosphate buffer. Results are given in Table II.

Sampling and Analytical Procedures

Samples of seed, flakes, press cake, solvent meal, DT (desolventized and toasted) meal and oil were collected for analyses in two ways during each commercial scale run. Grab samples, collected intermittently as appropriate, represented process status after condition changes or as spot checks during "steady state" portions of the runs.

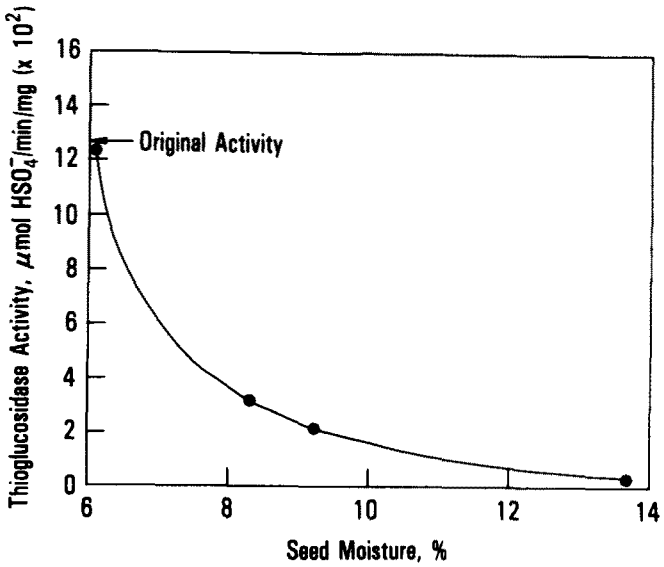

FIG. 1. Thioglucosidase activity as a function of seed moisture when seed is heated for 30 min at 180 F.

Other samples of the various products, representing significant portions of the runs, were prepared by compositing regular hourly or bihourly on-stream collections during 4 to 20 hr "steady state" sequences. These included periods when DT meal was being collected for later feeding experiments. These latter meals also were sampled and analyzed at the time of formulation into feeds (4).

Moisture, oil, fiber, ash and protein (N x 6.25) contents of the various Crambe seed and meal products were determined by official AOCS methods (5), and are reported on a moisture and fat free basis (mffb) unless otherwise noted. Total glucosinolate (by glucose release and calculated as *epi*-PG), free glucose and thioglucosidase activity were determined according to Van Etten et al. (3). 1-Cyano-2-hydroxy-3-butene (nitrile) was determined by gas liquid chromatography (GLC) (6). Amino acid analyses were determined on a Glenco MM-100 amino acid analyzer after 6N HCl hydrolysis. Nitrogen solubility in water, 0.5 M NaCl, or 0.03 M NaOH was determined by measuring Kjeldahl N solubilized by these extractants as a percent of total sample N. Oil fractions were refined, bleached and hydrogenated by the method of Baker et al. (7). Fatty acid composition and erucic acid content of the oils were determined by GLC (8). Other oil analyses followed standard AOCS methods (5). On-site checks for sulfur in the Crambe oil were made by observing whether or not freshly cleaned Cu coils tarnished with time when placed in the heated oil. Briefly, pieces of No. 22 Cu wire were brightened with fine sandpaper and then fashioned into two coils (1/4 in. diam x 1/2 in. separated by 1 in.). One or 2 ml of oil was placed in a small test tube, and the Cu coils were inserted so the bottom coil was fully immersed in oil and the top coil was above the oil but still within the test tube neck. The oil was then heated on a steam bath (212 F). Oil known to contain sulfur rapidly gave a light-reddish to black tarnish to the immersed coil (10-60 min), the color and time apparently depending upon sulfur content of the oil. The upper coil, serving as a control within the test tube, remained bright. Though not examined quantitatively, this method was found to correlate well with the AOCS silver dime test, and appeared to require less time and lower temperatures. Frequently, a fresh Cu coil inserted into the previously tested hot oil remained bright, indicating that reactive sulfur was removed by the initial coil.

Commercial Processing Facilities

Table III shows processing locations and years, type of processing facilities, quantities of Crambe seed processed and cooperating organizations. The two Culbertson runs were carried out under a USDA contract, and the other runs involved mutually arranged and beneficial informal cooperation between USDA and the indicated organizations. The Cleveland run is described in most detail since it encompasses all steps in a prepress solvent extraction process. Processing objectives were similar for all runs. Commercial grade hexane was used.

Cleveland, Ohio, 1972 (Prepress Solvent Extraction). Whole Crambe seed from storage was fed to a double-screen cleaner for separation of dockage. For two short test periods (each 4-5 hr), cleaned seed was cracked in corrugated rolls (2 high, 10 in. diam x 42 in. width, top and bottom clearance 0.040 and 0.200 in., respectively) and passed over a 2-deck Bauer dehuller (3/32 in. top screen to feed and 1/16 in. bottom screen to tailings). The two short test periods enabled operation of the mill on largely dehulled

TABLE II

Thioglucosidase Inactivation Tests in the Pilot Plant

	Tempering conditions[a]				Tempering results[b]		
	Jacket or sparge steam		Seed temperature, F				
Test number	PC-1	PC-2	PC-1	PC-2	Seed moisture, %	Thioglucosidase activity[c]	Glucosinolates, μMoles/g
Composite seed[d]	–	–	–	–	8.02	Yes	167
1	J	J	179	195	7.24	Yes[e]	153
2	J&S	J	192	208	8.05	Yes[e]	121
3	J&S$^+$	J	215	202	13.69	No	126
4	J&S^{++}	J	220	220	11.89	No	113
5	J&S^{++}	J	220	220	13.63	No	111
6	J	J&S^{++}	190	220	13.78	No	138

[a]Two stage horizontal paddle conveyor, upper (PC-1) stage feeding lower (PC-2) stage, fitted for jacket (J) and sparge (S) steam. Residence time of seed was 16 to 23 min at feed rates of 62-71 lb/hr. S, S$^+$ and S^{++} = light, medium and high sparge rates, respectively.

[b]Seed exiting PC-2 sampled and composited for analysis. Mean oil content was 32.2%. Glucosinolate and oil analyses on fat- and moisture-free basis.

[c]TesTape® method for glucose release (VanEtten et al. [3]).

[d]Untempered seed composited from all tests.

[e]Weak but detectable activity.

TABLE III

Facilities Used for Processing Crambe

Location and year		Process type	Plant capacity Tons/Day	Seed processed Tons	Seed processed Tons/Day
Cleveland, Ohio	1972[a]	Prepress solvent	200	905	50-150
Angola, Indiana	1973[b]	Solvent extraction	3	6	3
Culbertson, Montana	1974[c]	Prepress solvent	100	452	48
Culbertson, Montana	1975[c]	Prepress solvent	100	279	66
Champaign, Illinois	1978[d]	Solvent extraction	100	85	30

[a]Crambe Enterprises, Inc. and Vincennes University, Vincennes, Indiana.

[b]Angola Soya Mill, Angola, Indiana.

[c]Agricom International, San Francisco, California, with P.J. Anderson & Sons (1974) and Continental Grain Corp. (1975), Culbertson, Montana.

[d]National Protein Corp., Champaign, Illinois.

material and permitted sampling of the process stream for later comparison with whole seed processing.

From these preparation steps, cracked whole seeds (or dehulled meats) were conveyed to a 6-kettle French cooker (72 in.) situated above 3 French screw presses. Seed was tempered with jacket steam only and exited the cooker at 190-205 F before entering the screw presses. Press cake (1/8 to 3/16 in. thick) was conveyed to a French vertical basket extractor (22 baskets, 52 × 40 × 25½ in.) where hexane extraction occurred counter- and cocurrently in the up and down transits, respectively. Extraction temperature was 130 F, solvent to feed ratio ca. 1.6:1, and residence time ca. 45 min. Miscella was pumped through a pressure leaf filter before transfer to the bottom of the evaporator unit where indirect heat concentrated the miscella to 95% oil as it exited the top through a vapor-entrainment separator. Oil concentrate passed downward through a packed stripping column (14 in. Hg vacuum) where remaining hexane was stripped with sparge steam. Solvent- and press-oil were combined and degummed by processing through DeLaval centrifuges and a vacuum dehydrator.

Marc (spent defatted meal from the extractor) entered the top of a 7-deck French desolventizing-toasting (DT) unit, which utilized both sparge (sections 1, 3) and indirect heat (sections 1-3, 5-7 off). DT meal, discharged at 190-230 F, was conveyed to a ribbon screw type meal cooler and then to storage. Several tons of meal were bagged for beef cattle feeding studies. Material balance: In, 905 T seed with 130 T screenings (14.3%). Net seed in, 775 T. Out, 172 T oil (22%), 558 T DT meal (72%) and 45 T (6%) lost. Running time, 7 days.

Angola, Indiana, 1973 (Straight Solvent Extraction). This small facility was owner-fabricated with a capacity of ca. 3 T oilseed/day. Whole, clean seed was metered into a heated tempering conveyor where it was rapidly heated to ca. 170 F before dropping into smooth flaking rolls. Flakes (0.005-0.010 in. thick) were conveyed to a rotating basket extractor (18 in. sectors, 36 in. flake depth); residence time ca. 45 min; solvent-to-flake ratio of 1.8:1. Spent flakes were conveyed to a 3-high screw conveyor DT unit, feeding consecutively from top to bottom, each with individually controlled jacket steam and with sparge steam in the middle unit. Meal entered unit 2 at ca. 185 F and exited unit 3 at ca. 225 F (moisture 8.5-10%). DT meal was cooled, dried and conveyed to storage. Meal was bagged for beef cattle feeding experiments. Miscella was desolventized in two steps through a rising-film evaporator and a disk- and donut-type stripping column. Residual hexane was removed in a separate vacuum stripping unit. Material balance: In, 5.9 T seed; out, 4.2 T meal (71.7%), 1.6 T oil (26.5%) and 0.1 T loss (1.8%). Running time, 58 hr.

Culbertson, Montana, 1974 (Prepress-Solvent Extraction). Whole seed was conveyed from storage to a two-deck shaker-screen, which removed foreign matter over the top (18 mesh) and collected seed at the blanked-off lower level. Seed passed through a Shanzer drier, over corrugated cracking rolls (10 in. diam × 42 in.), through a short preconditioning unit (steam jacketed conveyor, sparge steam added), and then was fed to two Anderson expellers. Press cake conveyed to the solvent building was fed via a two-flight auger to the Anderson oval, horizontal, moving basket extractor (21 baskets, extraction temperature 128-136 F). Miscella was filtered on an 80-mesh screen strainer on its way to the evaporator and bubble-cap stripping units. Marc from the extractor entered the top of the DT unit, a five-high bank of Schneckens steam-jacketed screw conveyers, sparge steam entering at units 3 and 4 as required. Toasted meal (210-225 F) was cooled in a rotary cooler, ground in a Prater knife mill (5/32 in.), and conveyed to storage. Seven T of DT meal was bagged for beef cattle feeding studies. Material balance: In, 452 T seed; out, 313 T DT meal (69.3%), 70.7 T press oil (15.7%), 51.0 T solvent oil (11.3%), 3.2 T dockage (0.7%) and 13.6 T loss (3%). Running time, 10 days.

Culbertson, Montana, 1975 (Prepress-Solvent Extraction). The same facilities were used in 1975 as in 1974. Two 3-T lots of DT meal were collected for beef cattle feeding experiments. Material balance: In, 279 T seed; out, 202 T DT meal (72.4%), 31.6 T press oil (11.3%), 23.7 T solvent oil (8.5%) and 21.6 T dockage and loss (7.7%). Running time, 5 days.

Champaign, Illinois, 1978 (Straight Solvent Extraction). Crambe seed (85 T) was processed in two 3-day segments a week apart. Cleaned seed entered a preconditioning unit (3 ft diam × 10 ft long steam jacketed paddle conveyor) where sparge steam could be introduced with entering seed

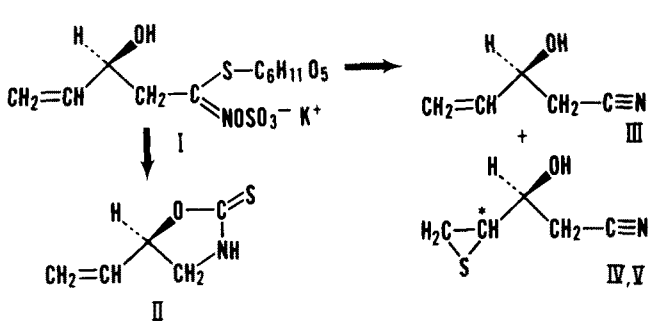

FIG. 2. Aglucon hydrolysis products (II-V) of *epi*-progoitrin (*epi*-PG, I) in Crambe meal. Glucose and HSO_4^- ion are also products.

(max. T ca. 165 F). Tempered seed passed through smooth flaking rolls (flakes 0.010-0.015 in. thick). A Redler conveyor transported flakes to the French stationary basket extractor (solvent to flake ratio 1.8-2.0:1, residence time 90-120 min). Miscella passed through a Dorrclone Separator before entering the plant's evaporator-stripper system where hexane was recovered and hexane-free oil was then pumped to storage. Spent solvent meal was conveyed to the 4-kettle French DT unit arranged for sparge steam in the top kettle and jacket-steam in all four. Finished DT meal was cooled, screened and conveyed to storage. No feeding studies were conducted on this meal. Material balance: not available. Running time, total of 7 days.

RESULTS AND DISCUSSION

Glucosinolates and Processing Objectives

As a crucifer, Crambe seeds contain glucosinolates and an associated enzyme system, thioglucosidase (thioglucoside glucohydrolase [EC 3.2.3.1]), which in concert can release toxic aglucon products into oil and meal during processing (Fig. 2). Except for palatability, ingestion of glucosinolates independently of thioglucosidase is not particularly harmful to beef cattle, but this is not true for other animals, especially for monogastric animals (9,10). We recently reviewed the factors involved in thioglucosidase reactions with *epi*-progoitrin (I, Fig. 2) and how the products (II-V) of these reactions adversely affect the quality of the byproduct Crambe meal as a protein source for feeds (10). Further, release of sulfur into extracted oil during these reactions leads to catalyst poisoning when hydrogenation of the oil is attempted.

Our approach to processing Crambe seed for quality oil and meal has been two-fold: (a) to inactivate thioglucosidase at the earliest stage of processing, and (b) to prevent both enzymatic and thermal degradation of *epi*-progoitrin (I) during all processing stages, thus preventing the release of the more toxic aglucons (II-V, Fig. 2) into the meal and of sulfur into the oil. This basic approach has been used in the rapeseed processing industry (11), and has been discussed from various perspectives by numerous rapeseed researchers (12-20). However, even before the introduction of the low glucosinolate Canola varieties, the rapeseed glucosinolate problem was different because glucosinolate levels in traditional rapeseed varieties were about half those of Crambe.

Thioglucosidase Inactivation

Figure 1 shows results of a laboratory study where heating whole Crambe seed at 180 F for 30 min was only marginally effective at enzyme inactivation unless seed moisture was above 10%. Under dynamic conditions in the pilot plant, whole seed retained thioglucosidase activity even after tempering for 20 min at 190-208 F and 7-8% seed moisture (Table II, Tests 1-2). Indeed, dynamic moisture levels above 12% and seed temperatures above 200 F were required for complete thioglucosidase inactivation (Tests 3-6). Some decrease in glucosinolate content also is observed. Other experiments have shown, however, that enzyme inactivation can be accomplished more easily in ground or flaked seed than in whole seed.

In commercial processing runs, seed, press cake, flakes, solvent or DT meal samples were checked for thioglucosidase activity using glucose sensitive TesTape®. The intensity of color developed in the TesTape® gave an indication of the glucosinolate content of the sample. Where no color developed (enzyme inactive), addition of 10 mg of purified mustard mynosinase (13,21) to the meal slurry gave a quick check on residual glucosinolate as glucose was released. Frequent in-plant checks using this technique provided insight into the fate of *epi*-progoitrin (I) as processing advanced from stage to stage.

Only in the Cleveland run was full enzyme inactivation achieved at an early stage of processing. The French preconditioner had sufficient capacity to maintain whole seed at 190-205 F (moisture ≥7%) so that thioglucosidase was reduced significantly and then fully inactivated in the hot expellers, a result similar to the Sidney, Nebraska, run (1). Ironically, the general overcapacity of the Cleveland plant resulted in excessive temperatures elsewhere with resultant production of relatively poor-quality meal and crude oil.

In the Angola and Champaign mills, tempering at < 165 F adequately prepared seed for flaking but did not inactivate thioglucosidase, and the DT units were relied on to eliminate enzyme activity. The small DT at Angola was efficiently controlled (185-225 F, 8.5-10% moisture) for full enzyme inactivation without excessive loss of glucosinolate and produced a high quality meal. At Champaign, moisture input to the DT was limited by condenser capacity, which meant that higher temperatures were needed for full enzyme inactivation resulting in greater destruction of glucosinolate.

At Culbertson, the preconditioner could not provide the right combination of heat and moisture for destroying thioglucosidase there or in the expellers, and inactivation was completed in the DT unit at temperatures > 210 F.

Process Fractions and Oil Extraction

Table IV lists proximate analyses on composite samples from the different stages of processing. The oil content of the seeds appeared remarkably uniform (30.5%, moisture free basis [mfb]), although considerable variability was observed in grab samples collected during each run. Protein content of the Culbertson seeds was notably lower both years compared with the other three runs (28.2% vs. 33.6%, grouped means, moisture and fat free basis [mffb]). Crude protein content between seeds and DT meals was not different (mffb), except in the Cleveland experiment where the DT-meal data are for a test period on dehulled seed during that run. Dehulling upgraded both oil (to 40% mfb) and crude protein (to 42% mffb) contents of the seed furnished to the presses, and ultimately the protein level of the DT meal (to 46%, mffb). The fiber content of the finished meal also was significantly lowered. Although technically simple, dehulling presents added costs to the mill operator and produces a relatively innocuous waste fraction (hulls).

The expellers at Cleveland removed only 16% of the dehulled-seed oil, in contrast to the Culbertson presses, which removed 40% of the whole-seed oil. Oil removed by pressing dehulled seeds at Sidney, Nebraska, was similar to the Culbertson runs (1). Crambe press cake usually had 18-20% residual oil. Choking the presses to reduce this level generally produced scorched press cake, because whole Crambe seed has a large hull content (30% by wt, 60% by vol.) and friction from the hulls created high heat in the expellers.

Solvent extraction at Cleveland was relatively inefficient, 73% of the press cake oil being removed compared with 79 and 87% in the Culbertson runs. In the straight solvent extraction runs, where residence times were long, the Angola and Champaign extractors removed 83% and 86%, respectively, of the flake oil. The total seed oil extracted in the five runs was 77% (Cleveland), 85% (Angola), 86% (Champaign), 87% (Culbertson, 1974) and 92% (Culbertson, 1975). Using 45 min residence times and solvent-to-meal ratios of 1.6-2:1, the Cleveland and Culbertson extractors reduced oil levels in finished meals to ca. 4% overall but as low as 1.5-2% at times. To achieve this result at Angola and Champaign with straight solvent extraction,

TABLE IV

Proximate Analyses on Crambe Process Fractions[a]

	Moisture, %	Oil, %	Protein (N X 6.25) %	Crude fiber, %	Ash, %	NFE,[b] %
Whole seed						
Cleveland 1972	8.8	26.9	22.5	22.5	5.5	13.8
Angola 1973	5.6	28.9	23.6	NA[c]	5.3	–
Culbertson 1974	7.0	29.4	18.7	23.9	4.4	16.6
Culbertson 1975	6.6	28.3	17.6	20.8	5.8	20.9
Champaign 1978	6.6	28.7	21.1	NA	NA	–
Dehulled seed						
Cleveland 1972	5.4	37.9	24.5	12.2	4.8	15.2
Press cake						
Cleveland 1972[d]	4.7	31.9	27.2	20.1	5.6	10.5
Culbertson 1974	6.3	17.7	24.4	24.3	5.1	22.2
Culbertson 1975	6.3	17.4	20.2	24.9	6.6	24.6
Flakes						
Angola 1973	7.1	25.4	25.3	NA	7.2	–
Champaign 1978[e]	6.7	28.2	20.3	NA	NA	–
DT-Meals						
Cleveland 1972[d]	5.1	8.7	40.2	14.8	8.3	22.9
Angola 1973	7.1	4.3	32.2	21.0	7.2	28.2
Culbertson 1974	4.9	3.7	27.6	26.2	6.4	31.2
Culbertson 1975	5.7	2.3	24.3	23.6	7.9	36.2
Champaign 1978[e]	7.0	4.1	28.5	NA	NA	–

[a]Composite samples from runs.
[b]Nitrogen free extract, by difference.
[c]NA = Not analyzed.
[d]From dehulled seed test run.
[e]Mean values for 26 samples distributed throughout run.

TABLE V

Selected Analyses on Commercial Crambe Oils[a]

	Cleveland 1972		Culbertson 1974		Culbertson 1975		Champaign 1978
	Refined and bleached		Refined and bleached		Crude		crude
Analysis	Press	Solvent	Press	Solvent	Press	Solvent	solvent
Moisture, %	0.27	0.41	0.01	0.01	0.05	0.13	0.64
m.p., C	6	6	6	6	NA[b]	NA	NA
Ash, ppm	<10	<10	<10	20	NA	NA	NA
Phosphorus, ppm	NA	NA	3	9	163	500	26
Peroxide value, meq./Kg	NA	NA	5.0	5.7	6.3	4.1	2.1
Iodine value	93	89	93	93	99	99	92
FFA, %	0.07	0.06	0.03	0.05	0.20	0.25	1.3
Sap. value, mg/g	173	180	171	172	NA	NA	NA
Unsaps., %	0.25	0.57	0.7	0.7	NA	NA	NA

[a]Composite oil samples from runs, refined and bleached in the laboratory.
[b]NA = Not analyzed.

long residence times (ca. 2 hr) were required. Otherwise, extractor operation appeared to be similar for the two types of material (cake and flakes). Ideally, residual oil content of finished meals should be ≤1%. Economically, prepress solvent extraction normally is favored over straight solvent extraction for high-oil seeds. However, for straight solvent extraction, operating a more efficient, perhaps oversized, extractor without sacrificing capacity might have cost advantages (22) over running expellers.

Typical analyses on different Crambe oils produced in this study are shown in Table V. Press oils were lighter in color, more easily refined and bleached and generally of higher quality than the solvent oils. As judged from hydrogenation tests and the Cu coil test for sulfur, press oils either had no sulfur contamination or much lower levels than the solvent oils. Refining and bleaching reduced sulfur in the solvent oils, but did not completely eliminate it. At both Culbertson and Champaign, higher sulfur levels in solvent oils were directly related to the presence of meal fines (inadequate miscella filtration) and excessive hexane stripping temperatures (final oil temperature >200 F). Apparently, sulfur is released into the oil when glucosinolates in meal fines decompose at high oil temperatures. A portion of the 1974 solvent oil was refined in the batch refinery associated with the Culbertson facility, but all other oils produced were marketed crude. Erucic acid content of the seed oil ranged from 52-54% at Culbertson to 54-60% at Cleveland and Champaign.

TABLE VI

Selected Analyses for Comparing the Culbertson Processing Runs[a]

Product type[b]	Moisture, %		Oil, %		Free glucose, μMoles/g		Glucosinolate, μMoles/g	
	1974	1975	1974	1975	1974	1975	1974	1975
Seed (2,2)	6.4	8.9	30.4	27.4	8	19	153	127
Press cake (16,22)	7.2	8.0	20.3	19.5	8	20	135	116
Solvent meal (1,8)	9.6	8.0	6.2	3.3	12	28	105	99
DT meal (16,28)	6.6	6.8	4.4	3.7	17	35	26	29

[a]Mean values as determined for samples collected on-stream, including composites; fat and moisture free basis.
[b]Numbers of samples analyzed in parentheses (1974, 1975).

TABLE VII

Selected Analyses on Champaign Crambe Meals[a]

Product type[b]	Moisture, %	Oil, %	Protein (N X 6.25) %	Glucosinolate, μMoles/g	Free glucose, μMoles/g	Nitrogen solubility, %	Nitrile as *epi*-PG,[c] μMoles/g
Seeds (5)	6.6	30.7	30.4	151	8	84.6	–
Flakes (27)	6.7	30.8	31.9	153	8	76.5	0.9
DT meals (26)	6.9	4.8	31.2	20	17	35.2	35

[a]Mean values as determined for samples collected on stream, including composites; fat and moisture free basis.
[b]Number of samples analyzed in parentheses, applies to moisture, oil, protein, glucosinolates and free glucose. Nitrogen solubility (0.03 M NaOH) on 1 seed composite, 4 flake samples and 12 DT meals.
[c]1-Cyano-2-hydroxy-3-butene (III) calculated as *epi*-progoitrin (I) (2 flake and 12 DT meals).

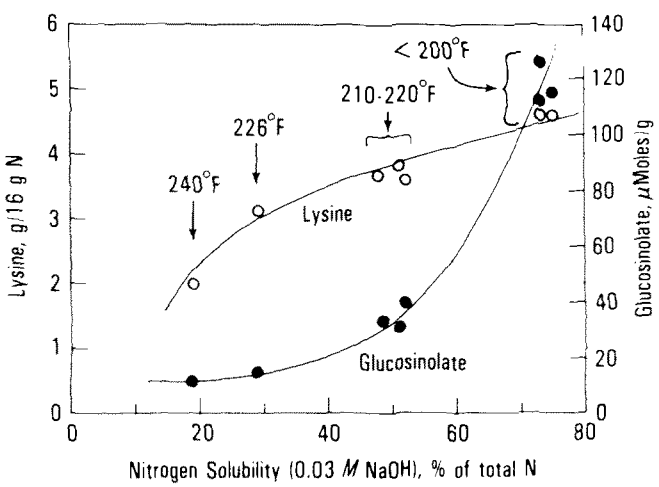

FIG. 3. Relationships of three quality parameters determined for 1975 Culbertson Crambe meals. Analyses on moisture and fat free basis.

Meal Chemistry in Process Fractions and Finished (DT) Meals

A selective comparison of the Culbertson runs is given in Table VI. These samples include, but are not limited to, "steady state" portions of the runs as in Tables IV and V. The free glucose analysis served two purposes: (a) to correct glucosinolate analyses (based on glucose release) for glucose inherently present at low levels in Crambe seed meal, and (b) to detect increases in glucose related to hydrolytic or thermal degradation of glucosinolate during processing.

Note that glucose levels in 1975 fractions are about twice the 1974 levels due to significant amounts of green and immature seed in the former, which probably accounts also for lower glucosinolate content of the 1975 seed. Some loss of glucosinolate occurred in the expellers, but no associated increase in free glucose is detected. Another decrease in glucosinolate, this one associated with an increase in free glucose, occurs in the extractor (solvent meal). Since thioglucosidase was still active, and extractor temperatures were <140 F, these changes may represent hydrolytic loss of glucosinolate. These small glucosinolate losses, however, don't compare with those in the DT. Nearly 3/4 of the glucosinolate in meal leaving the extractor is destroyed in the DT unit, which is again associated with an increase in free glucose. Under these dynamic and rigorous plant conditions the amount of free glucose detected never accounts for all glucosinolate lost, apparently because liberated glucose reacts with meal components (23-26) during processing.

Figure 3 illustrates the relationships among nitrogen solubility, lysine and glucosinolate in finished meals collected at different temperatures in the 1975 Culbertson run. Decreasing solubility of meal nitrogen is indicative of heat damage to, and lower quality of, protein in processed seed meals. VanEtten et al. (27) found that 0.03 M NaOH extracted 96% of the total nitrogen of unheated Crambe meal. Figure 3 shows that over 70% was extractable from meals processed at ≤200 F at Culbertson, but that this drops to <50% for meal processed above 220 F. Since lysine may react with glucose released from the glucosinolate, it is not surprising that as processing temperatures increase, lysine, glucosinolate and nitrogen solubility all decrease. Glucosinolate appears to be more sensitive to temperature than lysine, although reduced levels of either can be viewed as a significant indicator of lower protein quality in the finished Crambe meals.

Table VII further illustrates these relationships in process fractions from the Champaign run. Unlike the expellers at Culbertson, the flaking operation at Champaign

did not affect glucosinolate content, and free glucose levels are typical of lightly or unprocessed Crambe seed and flakes. However, the relatively low temperatures (<165 F) in the preconditioner prior to flaking affected a 10% decrease in nitrogen solubility (seed → flake). Again, more than 80% of the glucosinolate was destroyed in the DT unit, where temperatures were maintained near 220 F to ensure complete thioglucosidase inactivation. Free glucose content of the DT meals is typically double that of the seed (cf. Table VI), and nitrogen solubility (35%) in the Champaign meals is typical of Crambe DT meals processed at ca. 220 F (cf. Fig. 3).

Nitrile (III) content of the Champaign meals also represents hydrolytic and/or thermal degradation of glucosinolate during processing. Flakes contain only a trace, arising either from slight hydrolysis when the seed is crushed or as a result of hydrolytic action in wet and damaged seed furnished to the flaking mill. Nitrile content of the DT meals is typical of Crambe meals where most of the glucosinolate is lost in processing. The amount of nitrile, like the amount of free glucose detected, does not account for all glucosinolate destroyed. Since the enzymatic pathway to episulfides (IV, V) is readily disrupted by mild heat treatments (2), it is not surprising that these aglucon products were not found in any of the analyzed Crambe process fractions. Goitrin (II) is sometimes detected in processed meals, but only at low levels (<0.1%). Temperatures in the DT units would likely destroy these more labile products were they formed at an earlier processing stage.

Figure 4 illustrates the dynamic nature of the Champaign run. Not only were the two segments distinctly different, but considerable variation within each segment is evident. Even so, it is apparent that a direct relationship exists between moisture and free glucose and possibly between glucosinolate and oil contents of these DT meals. Therefore, confirmation of these and other relationships was sought by subjecting the Champaign data to regression analyses. The results are presented in Table VIII. As expected, combining data for seeds, flakes and DT meals

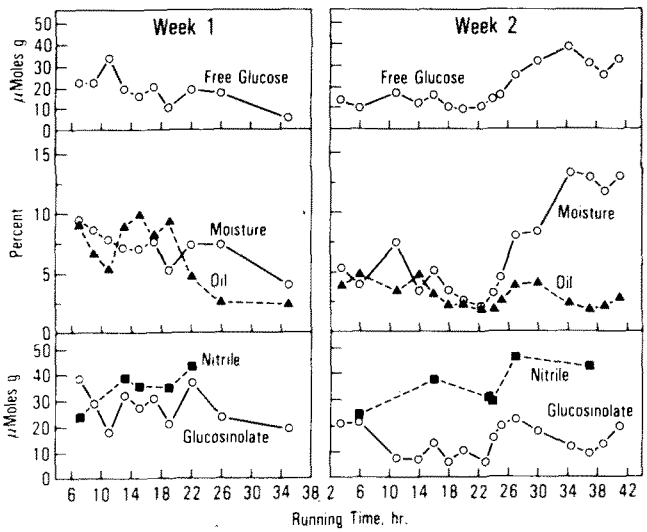

FIG. 4. Analyses as a function of time for Crambe DT meals from the Champaign processing run. Samples represent the two segments and span the entire processing run.

revealed a highly significant ($P < .01$) negative correlation between nitrogen solubility and processing temperature. For DT meals only, however, this linear correlation is not significant ($P < .05$). Possibly, significance is masked because processing changes during the run caused excessive variability within the DT meals. Also, temperature and moisture levels of meals exiting the DT unit may inadequately reflect conditions affecting nitrogen solubility within the several compartments of the DT. Note also that, contrary to what might have been expected, glucosinolate content is not correlated ($P < .05$) with either DT temperature or moisture. This, too, may reflect real differences between conditions within the DT unit and in meal emerging from the unit. Correlations of nitrogen solubility with free glucose ($P < .05$), glucosinolate ($P < .01$) and moisture ($P < .01$) are all significant, showing that conditions at

TABLE VIII

Regression Analyses on Data from Champaign Crambe Meals[a]

Product examined	Relationship examined	Correlation coefficient, r	Level of significance, P
Seed, flakes, DT meals[b,c]	Nitrogen solubility * Processing temperature	−0.88	<0.01
DT meals only[c]	Nitrogen solubility * DT temperature	0.29	ns
	Nitrogen solubility * Free glucose	0.68	<0.05
	Nitrogen solubility * Glucosinolate	0.92	<0.01
	Nitrogen solubility * Moisture	0.83	<0.01
	Glucosinolate * DT temperature	0.21	ns
	Glucosinolate * Moisture	0.19	ns
	Glucosinolate * Flake glucosinolate	0.51	ns
	Free glucose * DT temperature	−0.48	<0.05
	Free glucose * Moisture	0.88	<0.01
Flakes only[c]	Oil * Glucosinolate	0.88	<0.05
DT meals, flakes[b,d]	Glucosinolate * Moisture * Nitrile	0.99	<0.01
	Glucosinolate * Free glucose * Nitrile	0.99	<0.01
	Free glucose * Moisture * Nitrile	0.95	<0.01

[a]Analyses made on 5 composite seed, 27 flake and 26 DT samples, except nitrogen solubility and nitrile analyses on 14 samples, uniformly distributed throughout the run.

[b]Combined data.

[c]Linear regression.

[d]Multiple regression.

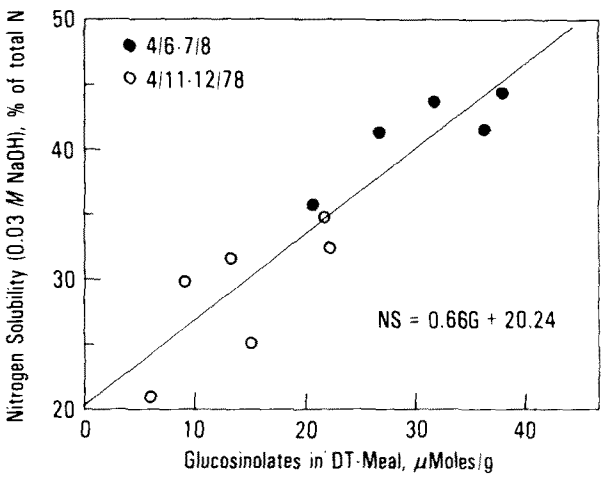

FIG. 5. Relationship between nitrogen solubility and glucosinolate levels in Crambe DT meals from Champaign processing run.

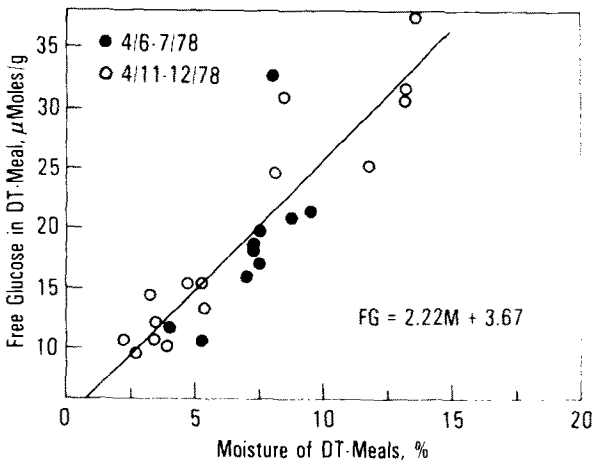

FIG. 6. Relationship between free glucose and moisture levels in DT meals from Champaign processing run.

different stages of processing affect these parameters in a predictable manner, even though our ability to measure conditions within the systems was limited. The highly significant correlation between nitrogen solubility and glucosinolate (Table VIII), implied by Culbertson data in Figure 3, is shown in Figure 5. As has been pointed out above and elsewhere (10), either analysis is a measure of quality of Crambe meals for feed purposes.

Free glucose content of DT meals is negatively correlated with temperature (Table VIII, $P < .05$), probably because glucose reacts with meal components at elevated temperatures (25-26). The positive correlation with moisture ($P < .01$), shown in Figure 6, may reflect hydrolytic release of glucose from glucosinolate. However, since a corresponding negative correlation between glucosinolate and moisture is not observed (not significant, Table VIII), it is conceivable that a protective relationship between moisture and free glucose is involved, which inhibits thermal degradation and/or reaction of glucose with meal components (24).

Sieve analyses of samples of seed entering the flaker at intervals during the Champaign runs revealed that the fraction of dehulled seed ranged from 25% to 35%, while that of whole seed (with hull) dropped accordingly (65% to 55%, chaff remained ca. 10%). Oil and glucosinolate contents of the collected seeds rose or fell with the levels of

TABLE IX

Analyses of Commercial Crambe Meals Prepared for Beef Feeding Trials (Air-Dry Basis)[a]

	Processing plant location			
	Cleveland	Angola	Culbertson	
Component (%)	1972	1973	1974	1975
Protein (N × 6.25)	27	31	28	25
Crude fiber	25	21	24	22
Ash	8.0	7.2	6.2	7.5
Fat	3.8	3.8	4.0	2.1
Moisture	11.0	8.3	6.0	7.6
Glucosinolate (*epi*-PG)[b]	26	87	44	37
Nitrile (as *epi*-PG)[c]	35	14	33	33
N solubility (0.03 M NaOH)[d]	–	72	36	50

[a]Adapted from Perry et al. (4). See also Carlson and Tookey (10).
[b]2-Hydroxy-3-butenyl glucosinolate (*epi*-progoitrin, I), μMoles/g.
[c]1-Cyano-2-hydroxy-3-butene reported as *epi*-progoitrin, μMoles/g.
[d]Percentage of total nitrogen extracted with 0.03 M NaOH.

dehulled seed in the furnish to the flaker. The linear correlation observed between oil and glucosinolate contents of the full-fat flakes (Table VIII, $P < .05$) is undoubtedly related to these observations. The relatively high level of dehulled seed apparently resulted from prior operations such as combining, cleaning, shipping, conveying, etc.

Multiple regression analyses on combined data for flakes and DT meals revealed highly significant correlations ($P < .01$) among: glucosinolate * moisture * nitrile (III); glucosinolate * free glucose * nitrile, and free glucose * moisture * nitrile (Table VIII). These interactions reflect the inherent relationship of glucosinolate to products (glucose, nitrile), and the important role that moisture plays in enzymatic and thermal degradation of substrate and in the stability of the derived products. Since time and temperature, as well as moisture, are involved in these complex relationships, the limited amount of data permit only a cursory interpretation of the results. Glucosinolate and nitrile are negatively correlated as expected, and free glucose and moisture are positively correlated. For a given glucosinolate content it appears that, during processing, nitrile levels increase with moisture as does free glucose, but, unlike glucose, nitrile (III) is relatively stable once formed in the meal.

Disposition of Products

Processing runs at Cleveland, Angola and Culbertson provided several tons of meal for each of 4 long-term beef feeding studies (4,10). Analyses of these meals, sampled at the time of feed formulation, are shown in Table IX. The Angola and Cleveland meals, as reflected both by analyses and animal performance (4,10), represent quality extremes that might be expected for Crambe meal produced in typical small oilseed crushing facilities (<200 T/day). Only in the Angola plant was the target goal of high glucosinolate content and high nitrogen solubility with low nitrile level achieved in the finished meal. Even so, performance of animals consuming any of the meals was adequate, perhaps typically represented by results of feeding the Culbertson meals (4,10), which were intermediate in quality between the other two. Also, the bulk meals produced in these runs were formulated privately into feeds for beef cattle.

A portion of the 1974 solvent oil was refined in a batch refinery associated with the Culbertson facility, but all

other oils were marketed crude. Primarily because of the sulfur content, this was accomplished by blending the oils into rapeseed oils in commercial process streams.

Recommendations for Processing Crambe Seed

Since no equipment has been specifically developed for harvesting or processing Crambe seed, precautions are necessary to adequately handle the material. Crambe seed seldom exceeds 6-8% moisture. However, harvested seed may contain significant trash (up to 20%) in the form of leaf and stem debris and foreign seeds. Some of this may be green and wet, and therefore a mill should have seed cleaning and drying equipment for proper storage and preparation for milling. Common weed seeds generally will pass No. 12 or 14 U.S. Standard Sieves, which retain ca. 98% of the spherical Crambe seeds.

For preconditioning seed, a stack cooker with 4 to 6 kettles is desirable. Seed entering the first kettle should be rapidly heated to 200 F, before being equilibrated in a following kettle to a moisture of ≥10% by uniformly distributed live steam. Subsequent kettles should then maintain temperature and moisture levels sufficiently long to complete thioglucosidase inactivation (15-20 min), and then the last kettles should dry and/or cool the seed before it is fed to the expellers or flaking rolls.

Expellers operating at 190-200 F, with cake thickness ≤¼ in., generally give a clean break between oil and foots leaving the expellers. Although press cake with low residual oil (12-15%) is desirable, experience has taught that 17-20% is more usual. If flakes are to be prepared for straight solvent extraction, flaking rolls should be set for flakes of 0.010 to 0.015 in. thickness, which along with adequate moisture (ca. 8%) to control fines give satisfactory extractor performance.

Extractor design appears to be of secondary importance for oil removal from Crambe flakes or press cake. Cake or flakes should allow adequate percolation of hexane without channeling or flooding. Although we encountered unacceptably high residual oil levels (4%) in finished meals in our studies, lower levels (2%) were interspersed through each run. To consistently achieve 1-2% residual oil probably requires solvent to meal ratios of 2:1, extractor residence time of ≥60 min, and operating temperatures of 135-145 F.

Desolventizing and toasting of spent Crambe solvent meal can be achieved in stack cookers (4-6 kettles), where the bulk of the solvent is flashed from the meal in the top kettles with live steam injected to facilitate solvent removal. Final stripping and drying of the meal is then accomplished in the lower kettles, the finished meal emerging at 8-10% moisture into a meal cooler before grinding and conveying to storage. If thioglucosidase inactivation has been achieved in the preconditioner, then relatively mild conditions can be used in the DT unit where temperature should not exceed 220 F in any event. Ideally, the finished meal will have no thioglucosidase activity, a high glucosinolate level (≥70% of original seed value), more than 50% of the meal N extractable with 0.03 M NaOH, a high lysine content (>4.5%), a low oil level (≤1%) and little or no nitrile.

With proper thioglucosidase control and retention of high glucosinolate levels in spent meal, there should be little sulfur contamination of the oil. Careful attention to miscella filtration and moderate stripping temperatures (final oil temperature <200 F) will prevent undue contamination of oil by sulfur derived from thermal degradation of glucosinolate in any meal fines contained in the oil.

The results presented in this paper show that Crambe seed can be commercially processed for commercial markets. Since some of the facilities used in these studies had been dormant for months prior to the Crambe runs, a significant portion of the problems experienced was associated with equipment failures. In the short runs reported here, downtime for repairs sometimes led to product variability beyond that contributed by willful experimentation. A few more strategically placed thermocouples, moisture probes and sampling ports would have been valuable for monitoring and controlling conditions at the different processing stages. Even so, Crambe seed was processed in several very different facilities with a measurable degree of predictability in the resultant products.

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