Crambe Seed Processing. Improved Feed Meal by Soda Ash Treatment[1,2]

G. C. MUSTAKAS, L. D. KIRK, and E. L. GRIFFIN, JR.,
Northern Regional Research Laboratory,[3] Peoria, Illinois
D. C. CLANTON, North Platte Station, University of Nebraska, North Platte, Nebraska

Abstract

Crambe seed, like rapeseed, is characterized by having thioglucosides and perhaps other anti-growth factors that diminish feed value and palatability. A soda ash cooking process was developed that modifies the thioglucosides in crambe meal and significantly improves its feeding value.

Destruction of the undesirable thioglucoside fraction of the meal was demonstrated, not only by paper chromatographic changes but also by negative results in tests which were based on conversion of the thioglucoside to thiooxazolidone. Sodium carbonate, added at a level of 1.4% (whole seed basis), destroys both the goitrin precursor, *epi*-progoitrin thioglucoside, and the ultraviolet-absorbing compounds in the meal, at least one of which is associated with bitterness. Animal-feeding tests demonstrated the improved palatability and nutritional quality of the meal.

Introduction

As a result of cooperative research between government and industry, *Crambe abyssinica* is emerging as a new industrial crop. Crambe, a member of the Cruciferae family, was selected as having excellent potential for development in a USDA-screening program for new oilseeds. During 1965 and 1966 approximately 1,500 acres were harvested and commercially processed.

Primary interest in crambe oil is based on its high erucic acid content. Rapeseed oil, also an erucic acid-bearing oil, is used in lubricants, greases, and other industrial applications. Because crambe oil has a higher erucic acid content than rapeseed oil, it should therefore have many potential uses (10). Crambe oil has proved to be a superior mold-release agent in the continuous casting of steel, and a strong demand for the oil is expected for this application alone.

One of the major problems with crambe is the feeding quality of the raw meal, or of untreated meal toasted like soybean meal, so that processing economics are adversely affected by the low value of this by-product. The untreated meal, whether raw or heated, is not palatable to animals and contains thioglucosides which need to be removed or destroyed for non-ruminants (4,12). In addition, sinapine, a bitter substance, and one other fluorescent compound are present in the untreated meal and may be related to a palatability problem noted in the feeding of ruminants.

Heat treatment, as employed in the processing of oilseeds, only partially destroys thioglucosides and does not make the meal palatable to cattle (3). Ammonia treatment of crambe meal, reported previously by Kirk et al. (5), somewhat improved the acceptability and usefulness of crambe meals. Unfortunately the degree of acceptability in preliminary tests with mature cattle was not duplicated in long-term feeding of calves (3).

This paper reports the use of a soda ash (sodium carbonate) process that deactivates unpalatable and growth-inhibitory factors in untreated crambe meal. This treatment was used commercially for the first time in the fall of 1965.

Experimental Section

Materials

Crambe seed used in this study was grown by private contract in 1964 in cooperation with the Crops Research Division, ARS, USDA. Analyses for whole seed, dehulled (pericarp removed) seed, and dehulled prepress solvent-extracted meal are given in Table I.

Methods

Total thioglucoside was determined by the sulfate method of McGhee (7); *epi*-progoitrin thioglucoside, the major thioglucoside, was measured by enzymatic conversion to thiooxazolidone, as illustrated in Fig. 1. For this determination, a modified Wetter procedure (12) at pH 7.0 was used, which measured both free and enzymically produced thiooxazolidone.

In an alternative chromatographic procedure, thioglucosides were isolated by a hot-water extraction of crambe meals; the extracts were then chromatographed on paper by the descending technique discussed previously by Kirk et al. (5). Fluorescent spots were observed by exposure of the chromatogram to a "long-wave" ultraviolet lamp before spraying with silver nitrate was done.

Sinapine was determined by the method of Tzagoloff (9) with the modifications suggested by Austin et al. (2). Crude fat, moisture, ash, and protein analyses were done according to AOCS official methods (1).

Equipment

The seed was cracked on 6-in. diameter rolls with 10 corrugations per inch. The flaking rolls were smooth-faced and 12 in. in diameter. Dehulling equipment consisted of a shaker screen with provision for aspiration at the feed and discharge ends.

TABLE I

Analyses of Crambe Seed and Meal

Assay	Whole seed[a,b] %	Dehulled seed[c] %	Dehulled prepress, solvent-extracted meal[d] %
Moisture	6.8	3.9	7.4
Crude fat	34.8	47.0	1.0
Protein (n × 6.25)	18.4	21.9	43.0
Crude fiber	12.8	2.8	6.1
Ash	5.0	4.2	7.8
NFE	22.2	20.2	35.7

[a] Hull content = 30%.
[b] Thioglucoside content = 4.8%.
[c] Thioglucoside content = 5.6%.
[d] Thioglucoside content = 10.5%.

[1] Presented in part at the AOCS Meeting, Philadelphia, October 1966.
[2] Journal Series, Nebraska Agricultural Experiment Station.
[3] No. Utiliz. Res. Dev. Div., ARS, USDA.

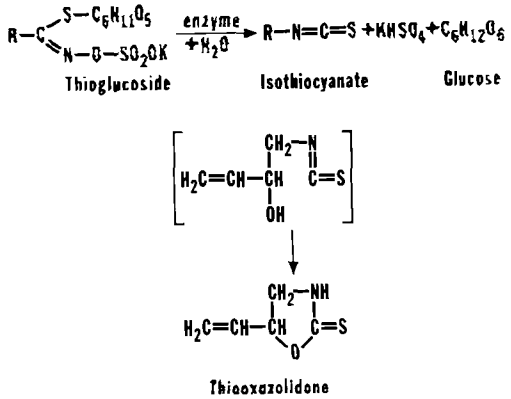

FIG. 1. Enzymatic hydrolysis of *epi*-progoitrin.

Solvent extraction was carried out continuously in a 20-stage counter-current Kennedy extractor or batchwise in a steam-jacketed, 50-gal tank equipped with a screen bottom.

Prepressing was conducted in a continuous screw-press, manufactured by Fuji Bunka Company Ltd., Tokyo, Japan. A two-stage, jacketed paddle conveyor served as a preconditioner for the screw press.

Equipment used for atmospheric cooking was a 1 cu ft ribbon blender (Fig. 2) or a 10 cu ft ribbon blender for the larger-scale experiments. Both units were steam-jacketed, had double-ribbon agitators, were provided with steam sparge systems, and were vented through condensers and receiver tanks.

Procedure

Two methods of cooking crambe with sodium carbonate were evaluated (Fig. 3 and 4): One was conducted on full-fat crambe; the other, on defatted crambe meal. The two approaches were selected as those which most closely simulated oilseed processes now used commercially.

Procedure A. In sodium carbonate cooking of full-fat meals, whole seed was cracked on corrugated rolls, and hulls (pericarp) were removed by air aspiration. This step was by-passed when no dehulling was done. The meats were then flaked and charged to the batch cooker, and carbonate was added to the mixture. The charge was then preheated to 180F, moistened, steamed, dried, and discharged from the cooker. Fig. 5 shows a time/temperature profile of the cooking operation. After cooling, the meal was extracted with hexane at 140F and desolventized by exposure to air.

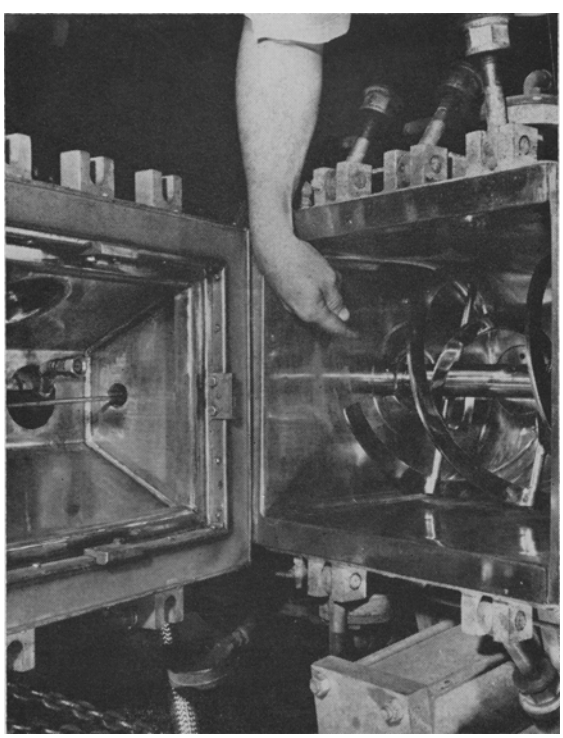

FIG. 2. Ribbon blender-cooker for soda ash treatment of crambe seed.

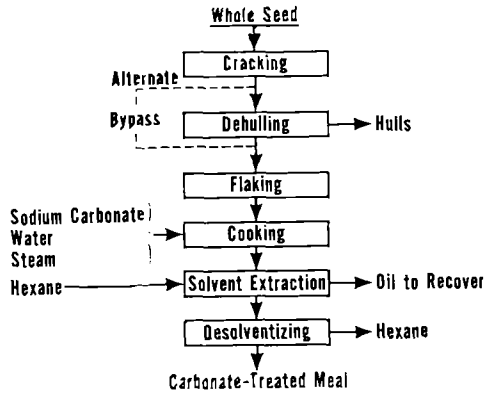

FIG. 3. Procedure A: sodium carbonate cooking of full-fat crambe meal.

Procedure B. In sodium carbonate cooking of defatted meals, whole seed was cracked and dehulled as in procedure A. The dehulled meats were then heated to 200F in a two-stage, jacketed screw conveyor and fed to a screw press to reduce the residual oil content to approximately 20%. The remaining oil was hexane-extracted by either a batch process or by continuous extraction in a Kennedy countercurrent extractor. The defatted meal, containing 0.5–1% residual oil, was then cooked with sodium carbonate by the same method used in procedure A.

Heat-Treated Control Meals. Control meals were prepared for each procedure, A and B, by duplicating the heat-processing treatment but eliminating the addition of soda ash.

Results and Discussion

Processing data for a series of 11 runs are given in Table II.

Destruction of Thioglucoside

Crambe contains relatively large amounts of thioglucosides of which *epi*-progoitrin is the major one

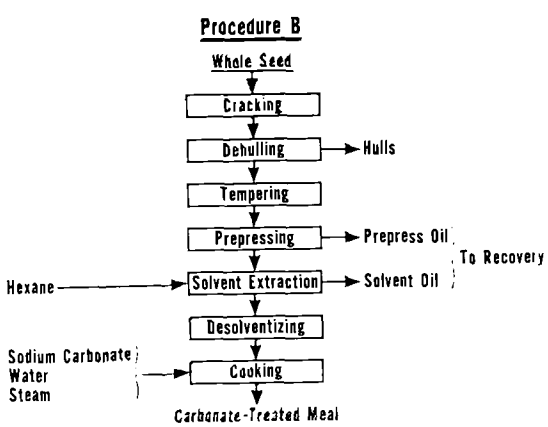

FIG. 4. Procedure B: sodium carbonate cooking of crambe meal defatted by prepress extraction.

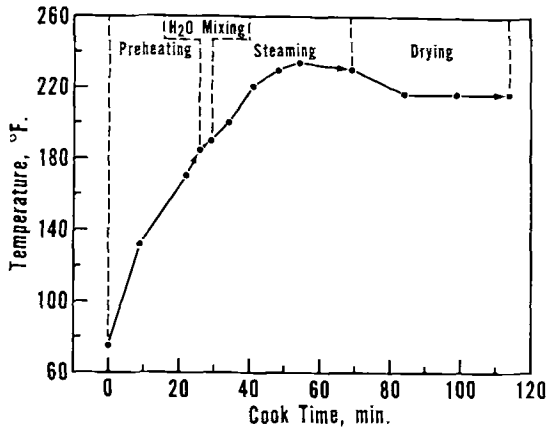

FIG. 5. Time/temperature profile of cooking process.

(90% of total) (12). This major component yields thiooxazolidone on hydrolysis with mustard myrosinase. The destruction of *epi*-progoitrin during sodium-carbonate treatment was demonstrated by paper chromatograms of meal (Fig. 6), made with and without carbonate treatment. Chromatographic spots and R_f values of the pure *epi*-progoitrin, as well as glucose and sucrose, are also shown. The thioglucoside spot at R_f 0.1 (Fig. 6), observed in "heat-treated-only" crambe meal, was not present in the heat- and carbonate-treated meal. The lighter sucrose spot, $R_f = 0.12$, visible in the carbonate-treated meal chromatogram was nearly masked in the chromatogram of the heat-treated-only meal by the partially overlying thioglucoside spot. There was also a distinct color difference between the two spots; sucrose appeared as a light brown spot whereas the thioglucoside was black.

Estimates of residual *epi*-progoitrin content were made by determining the amount of thiooxazolidone produced by enzymatic hydrolysis. Since no thiooxazolidone was produced from the carbonate-treated meal, quantitative destruction of the parent thioglucoside was demonstrated (Table III).

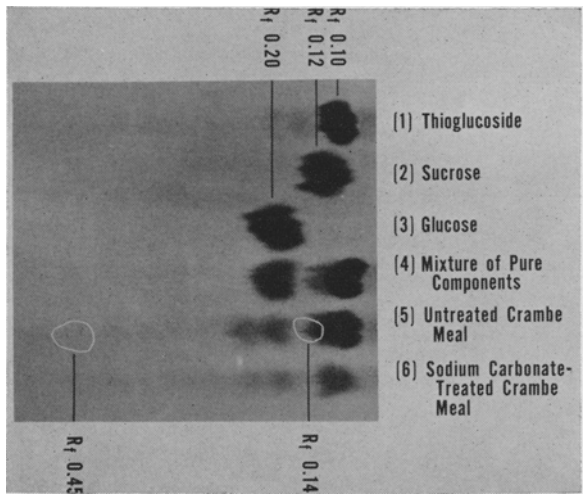

FIG. 6. Paper chromatograms of (1) thioglucoside, (2) sucrose, (3) glucose, (4) mixture of pure components, (5) untreated crambe meal, and (6) carbonate-treated crambe meal.

Destruction of Sinapine

Sinapine and another fluorescent compound were also destroyed by carbonate treatment. By chemical assay, sinapine was reduced from values of 0.5% in the original seed to below 0.05% in the treated meals (Tables II and III).

This compound (R_f 0.45) was readily detected on paper chromatograms of untreated meal extracts because of its fluorescence under ultraviolet light (Fig. 6) whereas the spot was no longer visible after the chemical treatment. A fluorescent spot at R_f 0.14 (Fig. 6) was also removed by the treatment; however, since this compound has not been identified, no significance can be given to its removal.

Sinapine has a bitter taste for human beings. It may therefore contribute to unpalatability to animals, but this has not been demonstrated by feeding tests.

TABLE II

Tabulated Data for Soda Ash Cooking of Crambe Meal

Item	Full-fat series[a]			Defatted series[b]							
Test No.	1[c]	2	3	4[d]	5	6	7	8	9	10[e]	11[f]
Soda ash level											
Lb/100 lb original seed basis	2.0	1.4	0.7	0.0	0.74	1.4	1.4	1.4	1.4	1.4	0.0
Lb/100 lb material treated	2.0	2.0	1.0	0.0	2.0	3.8	3.8	3.8	3.8	3.8	0.0
Cooking											
Preheating period, min	32	24	34	35	27	25	29	30	34	36	23
Mixing											
Moisture level after adjustment, %	22.4	20.0	20.6	21.4	28.6	16.4	23.8	24.4	22.2	21.4	18.1
Steaming											
Pressure, psig	6	6	6	6	6	6	6	0	6	6	6
Period, min	40	40	40	40	40	40	40	40	20	40	40
Temperature, °F	230	230	230	230	230	230	230	212	230	230	230
Moisture, %	23.0	22.0	22.6	25.2	32.2	16.0	30.0	25.8	27.4	28.4	19.0
Drying											
Period, min	45	45	45	45	45	45	45	45	45	45	45
Final temperature, °F	220	220	220	218	213	216	217	212	214	220	226
Final moisture, %	13.4	15.0	12.6	15.8	28.0	11.5	25.4	22.0	24.4	24.2	16.4
Analysis of treated meals											
Thiooxazolidone yield, %[g]	0.05	0.00	0.00	1.05	0.20	0.00	0.00	0.14	0.17	0.00	1.06
Sinapine, %	<0.05	<0.05	0.13	0.37	0.11	<0.05	<0.05	<0.05	<0.05	<0.05	0.48
Crude fat, %	1.4	4.4	3.9	2.7	1.29	2.6	1.1	1.1	1.1	1.2	0.9
Moisture, %	10.1	8.4	10.2	9.2	10.3	7.3	7.9	10.5	8.9	8.6	8.8
Ash, mf, ff basis	12.87	12.84	12.00	8.57	10.66	12.94	12.55	12.35	12.40	12.66	8.70
pH of aqueous slurry	7.6	7.0	6.1	5.3	5.9	6.9	7.0	7.3	7.4	7.0	5.2

[a] Soda ash added to full-fat (ff) crambe meal.
[b] Soda ash added to defatted crambe meal.
[c] Not dehulled; all other runs dehulled.
[d] No soda ash added, otherwise same heat treatment as Nos. 1–3.
[e] Soda ash added as wet solution; all other runs, dry granular Na_2CO_3.
[f] No soda ash added, otherwise same heat treatment as Nos. 4–9.
[g] Thiooxazolidone yield after enzymatic hydrolysis.

TABLE III

Destruction of Thioglucosides and Sinapine by Sodium-Carbonate Treatment

Component	Defatted untreated meal %	Prepress, solvent-extracted, heat-treated meal		Destruction by carbonate treatment %
		No carbonate %	Carbonate-treated %	
Thiooxazolidone yield	2.3	1.1	0.00	100
Sinapine	0.6	0.5	0.05	90–100

Process Variables

Sodium-Carbonate Concentration. In the full-fat series Nos. 2 and 3, soda ash was added to the dehulled full-fat meal whereas in Test No. 1 it was added to a nondehulled full-fat meal. A level of 2.0% (dehulled meal basis) or 1.4% (seed weight basis) was required to reduce thiooxazolidone and sinapine below 0.1%. For the defatted series Nos. 5–10, soda ash was added to the dehulled, defatted meal. In this series a level of 3.8% (dehulled, defatted meal basis) or 1.4% (seed weight basis) was required for the same effect.

Effect of Dehulling. The presence of loose hull in the seed during processing made little difference as shown in the comparative analysis of Tests 1 and 2 (Table II). However more soda ash was used to compensate for the presence of the hulls.

Moisture in Cooking. In Tests 6 and 7 high- and low-moisture levels were compared during cooking; however, under the conditions used, no significant difference in thioglucoside or sinapine destruction occurred.

Temperature, Pressure, and Time. The effect of a higher temperature and pressure during steaming was demonstrated in Tests 7 and 8. A pressure of 6 psig and 230F for 40 min effectively reduced thioglucoside, but when pressure was reduced to atmospheric and steam temperature to 212F, the resultant thiooxazolidone value of 0.14%, after processing for the same period, indicated residual thioglucoside. A reduction of steaming time (Test 9) to 20 min gave incomplete thioglucoside destruction.

Dry vs. Wet Addition. No differences were found in adding carbonate as a dry solid or as an aqueous solution. In Test 10, carbonate was introduced as a 14% solution whereas dry granular carbonate was used in Test 7 under identical processing conditions. Thioglucoside destruction was achieved under both conditions.

Processing Full-Fat vs. Defatted Crambe Meals. Carbonate was equally effective in reducing thioglucosides, whether it was added to full-fat or to defatted crambe meals. Therefore a processor could add the carbonate either before or after oil extraction.

Cattle Feeding

In initial cooperative cattle feeding experiments with the University of Nebraska (6) commercial meals (prepared under contract by the Pacific Vegetable Oil Corporation, Sidney, Neb.), which had been processed by conventional heat toasting methods, had a serious palatability problem. Slight but inadequate improvements in palatability were made by the addition of molasses or by pelleting the meal. Animal growth was poorer than with soybean meal. This deficiency was believed to be a result of the palatability problem.

More recently, carbonate and other treatments of crambe were evaluated in four palatability trials (6) with steers and compared with soybean meal as sources

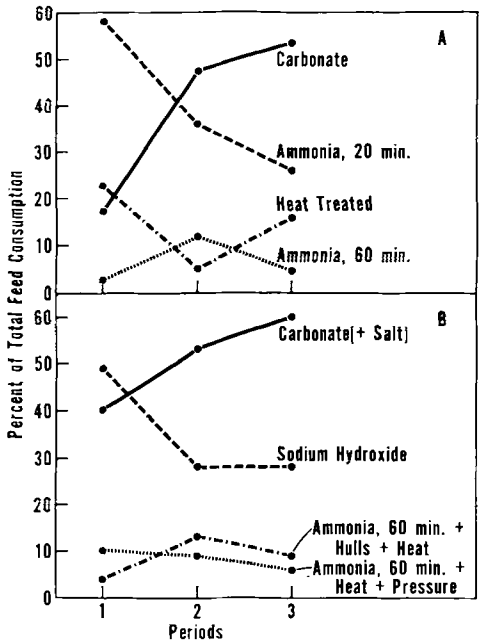

FIG. 7. Palatability trial on steers: percentage of total feed consumption coming from rations which contained differently treated crambe meals.

of supplemental protein. The treatments included sodium carbonate, ammonia, sodium hydroxide, and straight heat without additives.

In the first trial (Fig. 7A) treatments included processing dehulled crambe with sodium carbonate, ammonia over a 20-min period, ammonia over a 60-min period, and a heat control without ammonia or carbonate additives. For the second trial (Fig. 7B) the treatments included an ammonia cook under 20 psig pressure, an atmospheric ammonia cook with hulls, sodium hydroxide, and sodium carbonate plus salt. Carbonate heat-treated meal was compared with a heat-treated-only control meal in the third trial and then compared with soybean meal in the fourth trial.

In the series of four trials, steers were randomly assigned to pens where experimental rations were placed in individual feeders in each pen. Since the feeders were available at all times, the steers could select the ration they wanted. Each trial was divided into a 9-day preliminary period and a 15-day test period. During the preliminary period the crambe meal in each ration was gradually increased until all

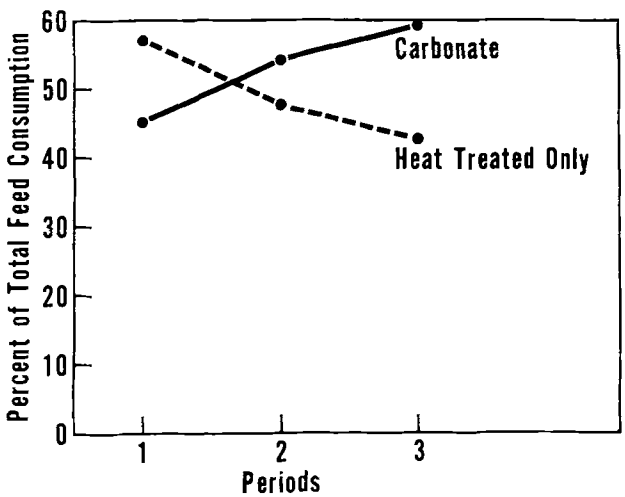

FIG. 8. Palatability trial: comparison of heat-treated vs. heat-treated plus carbonate meals.

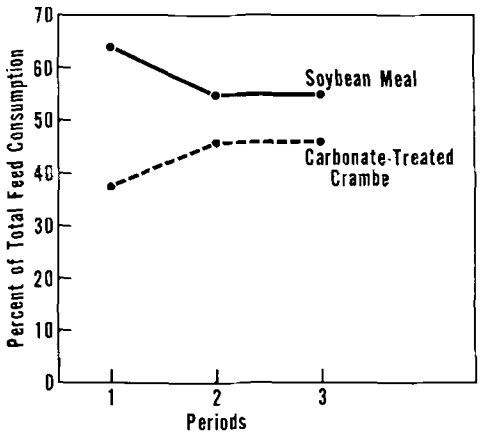

FIG. 9. Palatability trial: comparison of carbonate-treated crambe meal vs. soybean meal.

the supplemental protein was coming from the different crambe meals. The rations were rerandomized to the feeders at given intervals. The 15-day test period was divided into three 5-day periods, and the feed consumption was measured for each.

Fig. 7A shows the first trial in which animals were allow to select among the four meals. The intake of rations containing carbonate meal increased gradually and had the best total consumption at the end of the trial. In Fig. 7B intakes for the carbonate and sodium hydroxide treatments were both significantly higher ($P < 0.01$) than those for the other two meals fed.

Fig. 8 shows results from a separate trial which was conducted to compare the acceptability of carbonate-treated meal with heat-treated meal. Although over-all consumption of the ration containing carbonate-treated meal was not significantly different from the heat-treated, there was a decrease during the 15-day test period in consumption of the heat-treated while consumption of rations containing carbonate-treated meal increased. This behavior suggests that adjustment to the meals takes some time and that the carbonate meal would be more palatable after animals become used to it.

A ration containing carbonate-treated meal was compared directly with one containing soybean meal (Fig. 9). Consumption levels were lower for the carbonate, but the difference in feed consumption was not significant at the 1% level of probability.

Conclusions regarding the value of crambe meal in beef cattle rations as a source of supplemental protein will depend on the outcome of further feeding tests. At this writing, preliminary reports of large beef finishing trials with soda ash-processed meals appear highly encouraging.

Poultry Feeding

Chick-feeding studies at the Wisconsin Alumni Research Foundation, Madison, Wis., were begun with commercially prepared crambe meal that had been given the conventional heat toasting during processing. Chicks consuming this meal at a 20% level in the ration developed enlarged thyroids, grew poorly, and suffered high mortality. In subsequent tests with ammonia-treated meals, deaths and thyrotoxicity were eliminated, and antigrowth factors were reduced significantly (5). Later tests show that similar improvements in feeding quality were obtained with carbonate-treated meal and with a pressure-cooked meal. These treatments eliminated acute factors which caused abnormalities in the liver and death. The toxic factors responsible for enlarged thyroids were also largely removed except from the pressure-cooked meal. Untreated meals enlarged thyroids to 15–20 times their normal weight whereas alkali-treated meals resulted in less than a two-fold enlargement.

At the 20% feeding level these meals improved weight gains up to 60–75% of the soybean control. Body-weight gains reflected consumption so that it appeared that palatability was an important factor in increasing growth. The pressure-cooked meal produced good gains in weight but did not overcome the thyrotoxic factor.

Investigations will be continued to develop information on antigrowth factors. Although the carbonate treatment improves the nutritional value of crambe for nonruminants, more information is needed before any recommendations can be made.

Commercial Processing

Crambe was first processed commercially in 1964 by the Pacific Vegetable Oil Corporation (PVO) as a cooperative research venture with this laboratory (8). This operation was carried out in standard prepress-solvent equipment but with no chemical additive. In late 1965 crambe was crushed again by PVO with the use of sodium-carbonate addition. Composition and properties of the crude and refined bleached oils were similar to those reported for the first crush (8). The oil was not contaminated by sulfur through hydrolysis of the thioglucosides in the crambe seed, and it hydrogenated normally.

ACKNOWLEDGMENTS

P. O. Nees, Wisconsin Alumni Research Foundation, conducted the poultry-feeding tests; John Kneeland, Kurt Halseide, and George Kopas, Pacific Vegetable Oil Corporation, Richmond, Calif., processed crambe seed in commercial equipment under a cooperative agreement. R. L. Brown prepared the experimental crambe meals; J. E. McGhee and L. T. Black analyzed the meals for thioglucosides, thiooxazolidone, and sinapine; C. H. Van Etten, H. L. Tookey, and F. L. Austin provided information which was needed in conducting the analyses.

REFERENCES

1. American Oil Chemists' Society, "Official and Tentative Methods," including additions and revisions (1964).
2. Austin, F. L., and I. A. Wolff, Abstracts of Papers, Paper No. 5A, 149th Meeting, ACS, Detroit, Mich., April 1965.
3. Clanton, D. C., University of Nebraska, private communication, November 1964.
4. Hesketh, H. R., C. R. Creger and J. R. Couch, Poultry Sci. *42*, 1276 (1963).
5. Kirk, L. D., G. C. Mustakas and E. L. Griffin Jr., JAOCS *43*, 550–555 (1966).
6. Lambert, John, and D. C. Clanton, M. S. thesis, "The Use of Crambe By-products in Beef Cattle Rations," University of Nebraska, January 1966.
7. McGhee, J. E., L. D. Kirk and G. C. Mustakas, JAOCS *42*, 889–891 (1965).
8. Mustakas, G. C., G. Kopas and N. Robinson, JAOCS *42*, 550A, 552A, 554A, 594A (1965).
9. Tzagoloff, A., Plant Physiol. *38*, 202–206 (1963).
10. U. S. Dept. Agr., Bull. ARS-34-42, September 1962, 9 pp.
11. VanEtten, C. H., M. E. Daxenbichler, J. E. Peters and H. L. Tookey, J. Agr. Food Chem. *14*, 426–430 (1966).
12. VanEtten, C. H., M. E. Daxenbichler, J. E. Peters and I. A. Wolff, J. Agr. Food Chem. *13*, 24–27 (1965).

[Received July 9, 1967]