Prepress Solvent Extraction of Cuphea Seed

Kenneth D. Carlson[a,*], **Robert Kleiman**[a] and **Robert P. Pavlik**[b]
[a]USDA, ARS, NCAUR, New Crops Research, Peoria, Illinois 61604 and [b]The French Oil Mill Machinery Co., Piqua, Ohio 45356

Simulation of commercial processing of Cuphea seed to obtain needed quantities of oil and meal was accomplished with pilot facilities. Cuphea seed was conditioned in a single-deck cooker. Cooked seed was pressed with a mechanical screw with two-speed shaft, variable-speed drives and a four-section cage with cored sleeves. A feed rate of 22 kg seed/h and a feed screw-to-main shaft ratio of 2:1 gave good press cake with 8.1% residual oil. Press cake was extracted in a batch-type modular extraction pilot plant. Miscella stages were sequentially pumped through the beds, followed by hexane rinses. Spent cake was desolventized and toasted, and full miscellas were stripped to recover the crude oil. The finished Cuphea meal had only 0.30–0.55% residual oil. Thus, conditioned Cuphea seed was easily pressed without prior flaking to acceptable cakes, and conditions simulating commercial solvent extraction efficiently removed residual cake oil.

KEY WORDS: *Cuphea lanceolata*, Cuphea oil, *C. viscosissima*, *C. wrightii*, defatted meal, GC of methyl esters, GC of triglycerides, pilot-scale processing, prepressing, solvent extraction.

Cuphea species are major producers of triglyceride oils with medium-chainlength fatty acids. Chemical characterization of numerous Cuphea seed oils has revealed remarkable fatty acid diversity and species-specific predominance of single fatty acids in these oils (1–11). Examples are *C. pulcherrima* Foster (94%, 8:0), *C. schumannii* Koehne (94%, 10:0), *C. melanium* R.Br. (86%, 12:0) and *C. salvadorensis* Stand. (66%, 14:0). More than 70% of the characterized species produce dominant levels of capric and lauric acid, *e.g.*, *ca.* 33 and 40% of the species, respectively (4). Predominant levels of caprylic and myristic acid are produced in *ca.* 6 and 8% of the species, respectively (4).

Markets for lauric and capric acid are large and well established. The United States imports more than a billion pounds of coconut and palm kernel oils each year and consumes more than 500 million pounds of lauric acid in the soaps and detergents industry. Petrochemicals supply large quantities of capric and other medium-chain fatty acids to the plasticizer and lubricants markets (12).

Among the Cuphea species that have received breeding and agronomic attention are *C. wrightii* Gray (*ca.* 50–65%, 12:0), *C. viscosissima* Jacq. (*ca.* 65–70%, 10:0) and *C. lanceolata* Ait. f. (78–86%, 10:0) (9–11). The latter two species have been crossed, and from that germplasm have come the first Cuphea selections with nonshattering seed production characteristics, extremely important for crop commercialization (S.J. Knapp, private communication). Interest in studying the nutritional value of both Cuphea oil and defatted meal necessitated that a significant quantity of Cuphea seed be extracted (13). Since bulked samples of *C. viscosissima* and *C. lanceolata* seed from breeding programs were available, an opportunity was seized to conduct pilot processing studies while obtaining the needed oil and meal for the nutritional studies. We report here the results of this first pilot-scale processing of Cuphea seed.

*To whom correspondence should be addressed at USDA, ARS, NCAUR, 1815 North University St., Peoria, IL 61604.

EXPERIMENTAL PROCEDURES

Materials and methods. Available Cuphea seed was a blend of 20% *C. viscosissima* (13 kg) and 80% *C. lanceolata* (54 kg) that had been accumulated as bulked seed from various plot studies. Commercial-grade hexane was used in the solvent extraction phase. Seed was conditioned without cleaning in a French single-deck, 102 cm dia. × 76 cm ht cooker/conditioner with a steam-jacketed (1.035 MPa) bottom for heating, a hinged top for loading and a side door for emptying (The French Mill Machinery Co., Piqua, OH). The unit's thermometer well was located 23 cm above its floor, and sweeps for agitation of seed were driven by a 10-hp motor. Conditioned seed was pressed in a French 8.9-cm mechanical screw press with a two-speed shaft powered by two 7.5-hp variable-speed drives (The French Oil Mill Machinery Co.). The press was equipped with a four-section cage with cored sleeves for water cooling or steam heating on all four sections. All sections were lined with screen bars for oil drainage with spacings of 0.51, 0.25, 0.18, 0.13 mm from auxiliary to discharge. A variable-speed standard screw feeder was used for uniform feed rate to the press. Press cake was extracted in a French Modular Extraction Pilot Plant (The French Oil Mill Machinery Co.). The self-contained unit has a 10.1-cm screw for conveying press cake into and spent meal out of the extraction column (15 cm × 15 cm × 183 cm ht; 42.5 × 10^3 cm^3), a four-stage and final-rinse six-tank (40 L each) solvent extraction system, a meal desolventizer–toaster unit (3-deck-DT), a miscella distillation–vacuum stripper unit for solvent and oil recovery, and associated pumps, heaters, condensers, valving and piping. The operation of this solvent extraction system is described in detail elsewhere (14).

Seed conditioning. The cooking unit described above was preheated, and the blend of Cuphea seed (67 kg) was heat-and-moisture conditioned in the unit at *ca.* 8% moisture and *ca.* 102°C for 90 min (stirring rate about 13.5 rpm). Near the end of the cooking time, the cooker was opened to reduce seed moisture level. Cooked seed was transferred from the cooker at about 3% moisture to an insulated cart, from which it was loaded to the feed screw on the preheated mechanical press during pressing.

Mechanical pressing. The press shaft and cage jackets were preheated to 77 and 85°C, respectively. The feed screw-to-main shaft speed ratio was set at *ca.* 2:1 (38:18 rpm). As hot, conditioned seed (79–93°C) was added through the feed screw, the press discharge cone was set at *ca.* 0.16 cm to develop a cone pressure in the range of 4.83 to 5.87 MPa. Oiling and cake production occurred readily and required only a fine spray of water to be added to the seed feed as needed to maintain visual cake quality. During this initial pressing phase (PC-1), oil and cake were sampled for analyses, and the feed rate through the press was *ca.* 22 kg/h. The feed screw-to-main shaft speed ratio was then adjusted to 4:1 (80:20 rpm), and oil and cake were

sampled for analyses. During this pressing phase (PC-2), the feed rate through the press was *ca.* 61 kg/h. Oil and cake were sampled during pressing of the remainder of the seed (PC-3, feed screw/main shaft speed ratio = 80:24) where the feed rate was *ca.* 75 kg/h. The discharge temperature of the press cake during the entire run ranged from 99–107°C. Temperatures of the screw shaft (*ca.* 79°C) and the cages (99°C) were monitored during the pressing operation.

Solvent extraction. Press cake from the pressing operation was divided into four equal quantities for separate extraction in the modular extraction pilot plant. Further description than given here of the extractor and extraction process is given elsewhere (14). Press cake (13 kg) was loaded into the extraction column through the unit's screw conveyor. Initial bed depths ranged from 97–109 cm. Extractions were conducted at 57–60°C by pumping 30 L of hexane or miscella through a heat exchanger during cycling through the press cake in the extraction column. For press cake batch 1 (PC-b-1), two stages (30 L each) of hexane were cycled through the extraction column. For batch 2 (PC-b-2), two stages of batch 1 miscella (30 L each) and one stage (30 L) of fresh hexane were cycled through the extraction column. For batch three cakes (PC-b-3), three stages of batch 1 and 2 miscellas (30 L each) and one stage of fresh hexane were cycled through the extractor. Finally, for the fourth batch of cakes (PC-b-4), four stages of the previous miscellas (30 L each) and one stage of fresh hexane were cycled for 30 min through the column of press cake. For each batch, total extraction times were 30 min with a 15-min drain time. As extraction of each batch of press cake was completed, the spent Cuphea meal was discharged from the extraction column into the DT unit, where hexane was stripped by jacket heat and sparge steam, and the desolventized meal was toasted at 93–104°C (*ca.* 45-min total). The DT meals were sampled for storage and analyses. Miscellas were pumped to the oil recovery system, where hexane was first distilled at < 77°C, and the concentrated oil was then stripped of residual hexane under vacuum (−72 kPa). Crude oil was filtered and further stripped of residual solvent in the laboratory before being degummed, dried and bleached as described elsewhere for another oil (Filtrol Nevergreen, Engelhard Corp., Edison, NJ) (15).

Analyses. O'Haus moisture meters were used for rapid moisture determinations on seed and press cake in the pilot plant. In the laboratory, oil and meal analyses for ash, free fatty acids (FFA), iodine value (IV), moisture, oil, nitrogen and crude protein (N × 6.25), phosphorus (P), peroxide value (PV), and unsaponifiables followed official AOCS methods (16). Gas chromatography (GC) was performed as follows. Fatty acid methyl esters (FAME) were prepared by saponification of Cuphea oil (*ca.* 200 mg) in 5 mL of 0.5 *N* KOH in methanol, followed by reaction with 5 mL of 10% BF_3 in methanol. For each analysis, a 1-μL sample (10 mg/mL in hexane) of FAME was injected into a SP-7100 GC (Spectra Physics, San Jose, CA) equipped with a 30 m × 0.32 mm SP2340 capillary column (Supelco, Inc., Bellefonte, PA), helium carrier gas and flame-ionization detector (FID). Oven temperature was programmed from 100–250°C at 7.5°C/min with an initial 3-min hold. Reference samples of FAME ($C8_8$–C_{24}; Supelco, Inc.) and of *C. wrightii* FAME were used for routine identification of the Cuphea oil esters. The Cuphea triglycerides were analyzed by injecting 1-μL samples (10 mg/mL in hexane) into a HP 5890A GC (Hewlett Packard Co., San Fernando, CA) equipped with a 2.5 m × 0.32 mm GB-1 capillary column (Foxboro/Analabs, North Haven, CT), helium carrier gas and FID. Oven temperature was programmed from 170–350°C at 15°C/min with an initial hold of 1 min. Triglyceride references (tridecanoin, trilaurin, trimyristin, tripalmitin, triolein) aided in identifying triglycerides by carbon number (CN).

RESULTS AND DISCUSSION

Cuphea seed is currently a rare commodity. The amount of seed available to us was small and prevented more than cursory pilot-scale study. Even so, the results obtained in this research are satisfying, showing that traditional prepress–solvent extraction processing of Cuphea seed should offer few, if any, obstacles to further scale-up. Also, our main objectives of obtaining low residual-oil meal and sufficient crude oil for our cooperators were satisfied.

Sixty-seven kg of a mixture of *C. lanceolata* (80%) and *C. viscosissima* (20%) seed was cooked at *ca.* 102°C. The cooker was vented so that the initial seed moisture of *ca.* 8% was reduced to *ca.* 3% after 90 min, and the cooked seed was then fed to the mechanical press. The seed was too dry initially for oil and press cake to form, so a fine spray of water was added to the hot seed at the feed screw to initiate and maintain good pressing operations. Further study of the relationship between seed moisture and good oil and press cake formation will reveal optimum moisture levels for pressing Cuphea seed. As with other oilseeds we have processed, our experience in this study suggests that 4–6% moisture in the cooked seed will be appropriate (see below).

Table 1 shows material output during pressing. In all instances, heaviest oil drainage occurred in the feed section of the press, and few solids extruded through the screen bars. Conditions for generating press cake-1 (0.16-cm cone spacing, 5.87 MPa cone pressure, feed screw/main shaft speed ratio = 3.3:1) represent the best operation of the press in our experiments. These conditions approximated good prepress operation, giving the highest oil yield (18.4%) and lowest residual oil in the press cake (8.5%) (Table 1). Full pressing of Cuphea seed should be possible with tighter main shaft tolerances and smaller spacer bar and cone spacings. When the feed screw/main shaft speed ratio was adjusted to 4:1 (80 rpm/20 rpm), a 2.8-fold increase in material throughput occurred but a 33% reduction in press oil yield (12.3%) resulted, and press cake-2 with nearly 14% residual oil was obtained (Table 1). Increasing the main shaft speed to 24 rpm (feed screw/main shaft ratio of 80:24 = 3.3:1) to further increase material throughput reduced oil yield another 50% (to 6.1%) and gave press cake-3 with *ca.* 17% residual oil. The data in Table 1 show a high negative correlation between press cake oil and moisture contents ($r = -0.987$). This result may be fortuitous, but it supports the observation noted above that proper pressing of the whole seed required seed moistures above 3%. Conditions for generating press cakes-2 and -3 represented inefficient use of the screwpress for recovering Cuphea oil, but did yield press cake that was efficiently extracted in the solvent extractor. Our results suggest that flaking of Cuphea seed is not necessary for successful prepressing.

TABLE 1

Results of Cuphea Prepressing Phase[a]

Sample	Material output: Press cake (kg/h)	Material output: Oil (kg/h)	Material output: Oil yield (%)[b]	Press cake: Moisture (%)	Press cake: Residual oil (%)[c]
Press cake-1[d]	18.0	4.1	18.4	4.85	8.5
Press cake-2[e]	53.4	7.5	12.3	4.08	13.8
Press cake[f]	70.3	4.5	6.1	3.23	17.1

[a]The 67.5 kg of seed into processing at 7.63% moisture and 18.7% oil (db). Seed was a mixture of *C. lanceolata* (80%) and *C. viscosissima* (20%).
[b]Oil yield, % = oil (kg/h)/[press cake (kg/h) + oil (kg/h)] × 100.
[c]Dry-weight basis (db).
[d]Feed screw/main screw speeds = 38:18 (2.1:1).
[e]Feed screw/main screw speeds = 80:20 (4:1).
[f]Feed screw/main screw speeds = 80:24 (3.3:1).

Table 2 clearly shows how efficiently the Cuphea press cake was extracted in the shallow bed configuration (102 cm) of the modular solvent extractor. The three press cakes discussed above were distributed evenly into four batches (PC-b-1,2,3,4) of 13 kg each for hexane extraction. All four extracted, desolventized and toasted meals (DT-1,2,3,4) had residual oil levels well below 1%.

Recovery of oil from Cuphea was evenly distributed between the mechanical pressing (5.95 kg, 8.8% recovery) and solvent extraction (6.08 kg, 9.0% recovery) phases. Total press and solvent oil was 12.03 kg (17.8% recovery) or 95.3% of the oil in the starting uncleaned Cuphea seed (18.7%). [Note: The processed seed contained a significant amount of foreign matter. A sample of the seed cleaned in the laboratory before processing had *ca.* 25% oil, typical of cleaned Cuphea seed samples analyzed from breeding programs (S. Knapp and W. Roath, private communications)]. Crude press and solvent oils were both dark greenish black, which was due at least partly to immature seeds and to foreign matter. Degumming followed by bleaching had little effect on the oil color (too dark for color measurement). Table 3 shows that degummed and bleached solvent oil had a high level of P, a low IV and a significant PV. Unsaponifiables were plentiful, and the level of FFA (1.2%) was not excessive. When degummed oil was alkali-refined and then bleached, an acceptably light-colored Cuphea oil was obtained (Gardner 3-4). Alkali-refining removed FFAs and significant color and enabled the bleach (Filtrol Nevergreen) to work effectively.

Table 4 compares the analyses of a composited defatted meal from this study with a laboratory-prepared meal from *C. wrightii*. Both Cuphea meals had significant ash levels, but no mineral analyses have been made. These Cuphea meals had good levels of crude protein (N × 6.25). The much higher level of crude fiber in the *C. lanceolata/viscosissima* meal, compared to the *C. wrightii* meal, is probably explained by the amount of foreign matter that was processed with the former, rather than a species difference.

Figure 1 compares the fatty acid distributions of oil produced in the pilot study from *C. lanceolata/viscosissima* seed and oil extracted in the laboratory from *C. wrightii* seed. The dominant amount of capric acid in the former (78%) and the predominance of lauric acid (51%) in *C. wrightii* illustrates the species specificity for fatty acids in the genus. Capric acid is also abundant (35%) in *C. wrightii*. Both GC curves illustrate the breadth of available chainlengths (from 8:0 to 18:2) occurring in Cuphea species.

TABLE 2

Results of Cuphea Solvent Extraction Phase

Press cake feed[a]	Finished meal[b]	Moisture (%)	Oil (%)[c]
PC-b-1	DT-1	7.69	0.38
PC-b-2	DT-2	4.03	0.55
PC-b-3	DT-3	3.95	0.30
PC-b-4	DT-4	3.61	0.32

[a]PC-b = press cake batches 1–4, from *C. lanceolata/viscosissima* (80:20) seed.
[b]DT = desolventized/toasted meals 1–4.
[c]Dry-weight basis.

TABLE 3

Analysis of Refined Cuphea Oil[a]

Item	Value
Free fatty acids	1.21%
Unsaponifiables	1.81%
Iodine value	14.9%
Peroxide value	14.3 cg/g
Phosphorus	134 ppm

[a]Filtered, degummed and bleached oil from *C. lanceolata/C. viscosissima* (80:20) seed.

TABLE 4

Analysis of Defatted Cuphea Meals[a]

Analysis	*C. lanceolata* + *C. viscosissima*[b]	*C. wrightii*[c]
Moisture %	5.02	7.44
Ash %	7.81	8.38
Crude fat %	0.40	0.16
Crude protein[d] %	27.25	33.15
Crude fiber %	21.76	14.41
Carbohydrate[e] %	37.76	36.46

[a]Dry weight basis.
[b]This study; from *C. lanceolata/viscosissma* (80:20).
[c]Comparison meal from seed extracted in the laboratory.
[d]Nitrogen × 6.25.
[e]By difference.

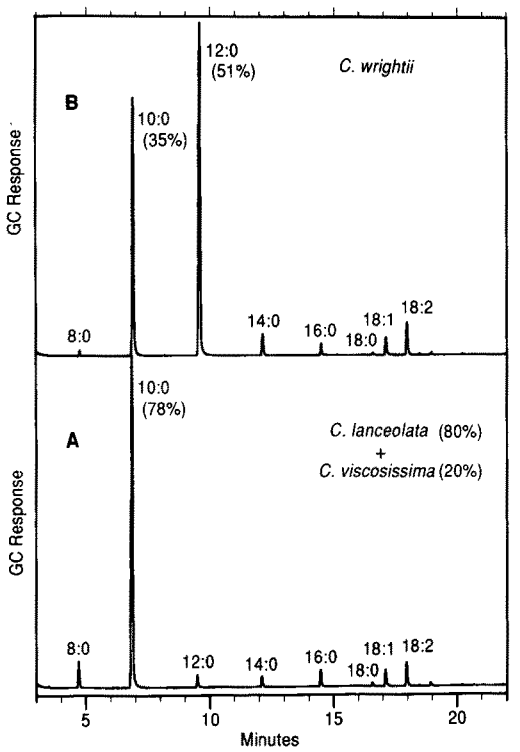

FIG. 1. Gas chromatographic (GC) curves for Cuphea methyl esters. A. Esters (78% 10:0) from oil extracted in the pilot plant from an 80:20 mixture of *C. lanceolata* and *C. viscosissima* seed. B. Esters (51% 12:0, 35% 10:0) from oil extracted in the laboratory from *C. wrightii* seed.

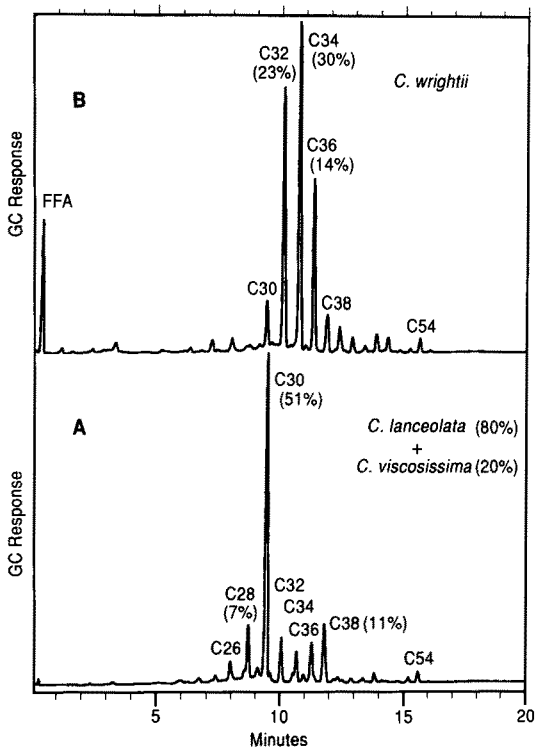

FIG. 2. Gas chromatographic (GC) curves for Cuphea triglycerides. A. Oil extracted in the pilot plant from an 80:20 mixture of *C. lanceolata* and *C. viscosissima* seed [(*ca.* 51% C-30); *ca.* 1% free fatty acids (FFA)]. B. Oil extracted in the laboratory from *C. wrightii* seed (*ca.* 23% C-32, 30% C-34, 14% C-36; *ca.* 9% FFA).

Figure 2 illustrates the marked difference in triglyceride distribution in the two oils. Tridecanoin, CN 30, is in great abundance (51%) in oil from the two-species composite (*C. lanceolata/viscosissima*) processed in the pilot plant, whereas the combinations of capric and lauric acids in *C. wrightii* triglycerides account for significant peaks with CNs 32 (23%), 34 (30%) and 36 (14%). Thus, a narrow or broad range of triglycerides is available in this interesting genus. The marked difference (*ca.* 8%) in FFA levels between the two oils is not understood at this point, but may be related to immature seed in the available *C. wrightii* seed lot (*ca.* 9% FFA).

In summary, pilot-scale prepress solvent extraction of Cuphea seed was accomplished without difficulty with excellent oil recovery achieved at both pressing and solvent extraction stages. Meal with low residual oil and high crude protein level, but high fiber content, was obtained. Degummed and bleached oil has been used in mice feeding studies (13). Alkali-refining and bleaching was required to reach acceptable P, FFA and color levels. We believe, with further optimization in seed preparation stages, including conditioning, flaking and extruding, that direct solvent extraction of Cuphea seed should be possible.

ACKNOWLEDGMENTS

The excellent technical assistance of James McVety, Mark Klokkenga, Paul Hilst, Imelda Schulte and Jack Glover is acknowledged.

REFERENCES

1. Miller, R.W., F.R. Earle, I.A. Wolff and Q. Jones, *J. Am. Oil Chem. Soc. 41*:179 (1964).
2. Graham, S., F. Hirsinger and G. Robbelen, *Am. J. Bot. 68*:908 (1981).
3. Wolf, R.B., S.A. Graham and R. Kleiman, *J. Am. Oil Chem. Soc. 60*:27 (1983).
4. Thompson, A.E., *Hort. Sci. 19*:352 (1984).
5. Graham, S.A., and R. Kleiman, *J. Am. Oil Chem. Soc.* 62:81 (1985).
6. Hirsinger, F., *Ibid. 62*:76 (1985).
7. Thompson, A.E., and R. Kleiman, *Ibid. 65*:139 (1988).
8. Knapp, S.J., L.A. Tagliani and W.W. Roath, *Ibid. 68*:515 (1991).
9. Graham, S.A., and R. Kleiman, *Industr. Crops Products 1*:31 (1992).
10. Roath, W.W., M.P. Widrlechner and R. Kleiman, *Ibid. 1*:5 (1992).
11. Webb, D.M., and S.J. Knapp, *Crop Sci. 31*:621 (1991).
12. Arndt, S., *J. Am. Oil Chem. Soc. 62*:6 (1985).
13. Hendrich, S., C.-K. Lii and R. Myers, *Ibid. 70*:797 (1993).
14. Ayorinde, F.O., K.D. Carlson, R.P. Pavlik and J. McVety, *Ibid. 67*:512 (1990).
15. Carlson, K.D., and R. Kleiman, *Ibid. 70*:579 (1993).
16. *Official Methods and Recommended Practices of the American Oil Chemists' Society*, 3rd edn., American Oil Chemists' Society, Champaign, 1983.

[Received July 14, 1993; accepted October 5, 1993]