

Pilot Plant Extraction of Oil from *Vernonia galamensis* Seed

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Vernonia galamensis seed containing 40–42% oil and 30–34% epoxy acid, (*cis*-12,13-epoxy-*cis*-9-octadecenoic) was processed to oil and meal. Seed conditioning, pressing and solvent extraction research were conducted in pilot facilities at the French Oil Mill Machinery Co. (Piqua, OH). The robust lipase system was successfully inactivated by treating 200 lb. batches of *V. galamensis* seed in a cooker/conditioner at 195–200°F and >10% moisture. Conditioned seed was mechanically pressed and the press discharge cone setting was varied during operation from 1/32" to 3/32" to demonstrate the feasibility of both full pressing and prepressing. Prepressing successfully reduced oil level in the press cake to ca. 20%. Press cake was extracted with hexane in a 1.5-ft³ batch-type, four-stage percolation unit with a 6" square extraction cross section. Solvent extraction reduced oil level in the defatted meal to 1–2%. The defatted meal was desolventized and toasted. Excessive foaming of the vernonia oil extract made complete solvent stripping in the oil stripping unit difficult.

KEY WORDS: Epoxy oil, lipase inactivation, mechanical pressing, pilot plant, seed oil, solvent extraction, *Vernonia galamensis*, vernolic acid.

Vernonia galamensis (Cass.) Less. seed yields 40–42% oil containing 75–80% vernolic (*cis*-12,13-epoxy-*cis*-9-octadecenoic) acid, making this native African species an excellent source of naturally epoxidized triglyceride oil. Even in its genetically unimproved state this species appears to be a promising crop for semiarid tropical areas (1), although the range of its climatic and geographic adaptability is still relatively untested (1–3). Use of vernonia oil for coatings (4) and in the preparation of a variety of polymeric materials, from hard resins to elastomers (5–7), is feasible. The oil or its derivatives also function well as plasticizer-stabilizers in PVC resins (8–12). Chemical epoxidation of the oil yields a product that is primarily a hexaepoxy triglyceride (ca. 80%). That product is easily converted to alkyl diepoxy esters of high purity (>90% 9,10,12,13-diepoxy stearates) by simple transesterification (13). This "purity" of epoxidized vernonia oil products makes them unique relative to other commercial epoxy oil products.

Germplasm collection and evaluation, agronomic research, and small-scale seed increases have made it possible to produce small quantities of oil to encourage both academic and industrial interest in *V. galamensis* oil (2,14–16). Laboratory and small pilot-scale processing studies (4) provided information needed to prepare these oil samples for interested parties. However, much larger quantities of oil are needed to meet growing industry interest and to take advantage of significant opportunities to encourage research on important new uses of vernonia oil. Therefore, the pilot-scale studies reported in this paper supplied oil for immediate needs and also advanced

knowledge of how *V. galamensis* seed can be processed in commercial oil extraction equipment. Our objectives of this pilot-scale study were five-fold: (i) to obtain a quantity of vernonia oil to meet research needs and requests for evaluation samples; (ii) to establish conditions for inactivating the robust lipase enzyme in vernonia seed; (iii) to study prepress extraction of vernonia oil; (iv) to explore full press extraction of vernonia oil; and (v) to study solvent extraction of vernonia press cake.

EXPERIMENTAL PROCEDURES

Material and equipment. *Vernonia galamensis* seed was grown in Zimbabwe at the Chipinge and Cheredzi Research Stations. O'Haus moisture meters were used for rapid moisture determinations in the pilot plant. Commercial-grade hexane was used in the solvent-extraction phase. In the laboratory, moisture and oil contents were determined by standard AOCS methods (Ac 2-41 and Ac 3-44). Seed was conditioned in a French 1-deck, 40" × 30" cooker/conditioner with steam-jacketed (150 psig) bottom for heating, a hinged top for loading, and a side door for emptying. The unit had a thermometer well 9" above the floor, and sweeps for agitation of material driven by a 10 HP motor. Seed was pressed in a French 3.5" mechanical press with two-speed shaft powered by two 7.5 HP variable-speed drives. 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 0.020, 0.010, 0.007, and 0.005" 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 self-contained system has a 4" screw for conveying feed into and spent meal out of a 1.5-ft³ extraction column (6" × 6" × 72" h), a four-stage and final-rinse solvent-extraction system (six 40-L tanks), a meal desolventizer-toaster unit (three-deck DT), a miscella (oil in hexane) distillation-vacuum stripper unit for solvent and oil recovery, and associated pumps, heaters, condensers, valving and piping. The solvent-extraction system permits cycling of the contents of any of the six solvent/miscella tanks through the extraction column, pumping from one tank to another, or movement of full miscella to the oil-recovery system. The extraction column can be used as a shallow-bed extractor (ca. 30" depth) or as a deep-bed extractor (ca. 72" depth). Two views of the modular pilot plant are shown in Figures 1 and 2. This equipment permits a variety of extraction conditions to be used, including a four-stage miscella and final rinse solvent extraction procedure on 40–50 lb. batches of seed material. The extraction column is turned upside down to feed spent meal (marc) directly into the DT (Fig. 2).

Seed conditioning. Two batches of vernonia seed (215 lb. and 199 lb.) were heat-and-moisture conditioned in the cooker/conditioner described above. Seed depth in the cooker was 9–11" (ca. half capacity), and stirring rate was 13.5 rpm. Ambient seed moisture was 6.6%. For the first

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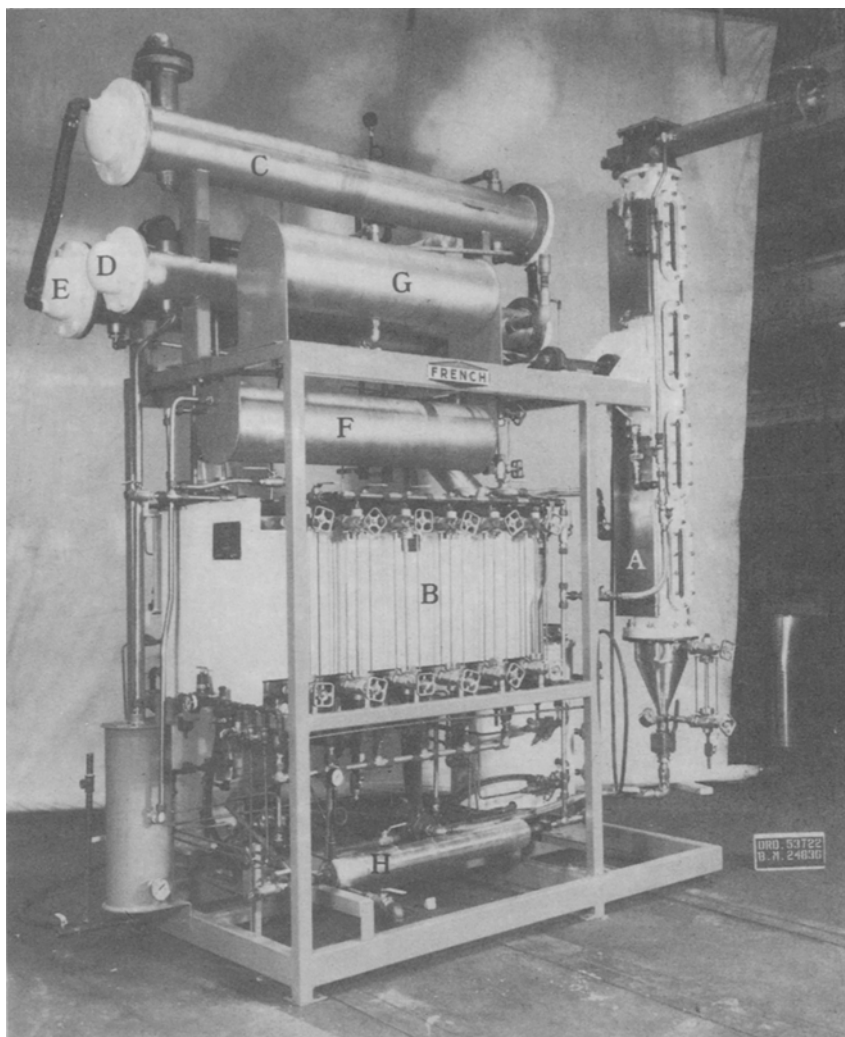


FIG. 1. Front view of Modular Extraction Pilot Plant. A = 1.5 ft³ extraction column positioned for extraction; B = six solvent/miscella tanks (40 L capacity); C = Oil stripper condenser; D = Final vent condenser; E = Steam jet/DT condenser; F = Solvent/water separator; G = Vacuum condensate tank; H = Solvent/miscella heater; I = Desolventizer/toaster (DT); J = Inlet/outlet conveyer.

batch, 23 lb. of water (calculated to raise seed moisture to 15%) was sprinkled over the 215-lb. seed bed heated to 150°F, and the cooker was closed. When the temperature reached 195°F, a moisture sample was taken (16.8%). Cooking continued for 1 hr at 195–200°F. Another moisture sample was taken (9.1%) and the cooker top was opened to allow the seed to dry to 3.5% moisture (ca. 1 hr at 195–200°F) in preparation for pressing. The second batch of seed (199 lb.) was conditioned similarly, except 35 lb. of water was added at 150°F to bring the seed moisture to 20% (calculated). Moisture after 1 hr of conditioning at 195–200°F was 13.2%. Conditioned seed was then dried to 3.5% moisture. Conditioned seed was transferred to a heavily insulated, wheeled cart, from which manual transfer of the seed was made to the press.

Mechanical pressing. The press shaft and cage jackets were preheated with steam to 200°F. Hot (175–200°F) batch 1 conditioned seed was added through the feed screw on top of the screw press at a steady rate. The speed ratio of the press feed screw/main shaft was set at 3.2:1

(58/18 rpm) for all four runs. The initial cone setting (orifice) was adjusted to 1/4" to provide back pressure and to form the press cake. The press began oiling and forming cake immediately, but the cake was crumbly so the cone was adjusted to 1/16" to increase back pressure (500 psig). Conditions stabilized within minutes and steady-state press cake (99 lb/hr) and oil (40 lb/hr) rate samples were collected (10 min) at a feed rate of 139 lb/hr (Test 1). Oil drainage was heaviest in the mid-section of the press with very little solids (foots) being extruded through the screen bars with the oil. The cone was adjusted to 1/32 in. (cone back pressure 850 psig) to further examine full-press conditions on the remainder of batch 1 conditioned seed. Feed rate during a second steady-state period (Test 2, 10 min) was 152 lb/hr. A firmer press cake was collected (101 lb/hr). Oil drainage (51 lb/hr) was still heavy in the middle of the press although more drainage was observed nearer the feed end.

For conditioned seed batch 2, two-steady-state collections (Test 3 and Test 4) were made at a cone setting of

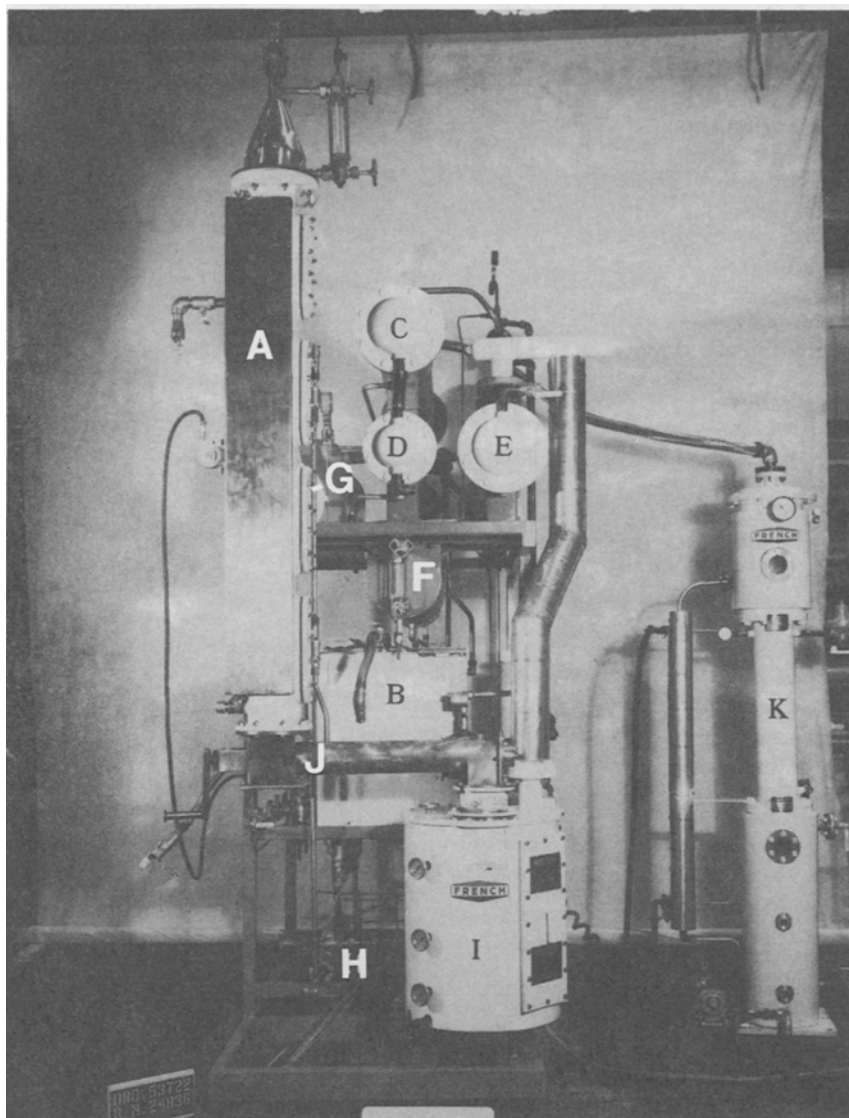


FIG. 2. Side view of Modular Extraction Pilot Plant. A = 1.5 ft³ extraction column positioned for conveying spent cake into DT unit; B = Solvent/miscella tanks; C = Oil stripper condenser; D = Final vent condenser; E = Steam jet/DT condenser; F = Solvent/water separator; G = Vacuum condensate tank; H = Solvent/miscella heater; I = Desolventizer/toaster (DT); J = Inlet/outlet conveyor; and K = Oil stripper.

3/32" and back pressure of 300 and 200 psig, respectively. Feed rates were 125 (Test 3) and 155 lb/hr (Test 4). Press cake collection rates (10 min) were 93 and 111 lb/hr with oil-drainage rates of 32 and 44 lb/hr, respectively. Tests 3 and 4 simulated prepress conditions suitable for preparing press cake for subsequent solvent extraction. Test conditions and results are summarized in Table 1.

Solvent extraction. Press cake obtained in the pressing operation (220 lb.) was divided into five equal quantities (44 lb.) for separate deep-bed extraction in the Modular Extraction Pilot Plant. Press cake was loaded into the extraction column using the screw conveyor (Fig. 1, J). Initial bed depths ranged from 63–68". Extractions were conducted at 130–140°F by pumping the hexane/miscella through a heat exchanger. For batch 1 press cake, the full miscella tank was empty and stage 4, 3, 2, 1, and rinse tanks each contained 30 L of hexane (See Fig.

3). Hexane from stage 4, 3, 2, 1, and the rinse tank was pumped for 10 min each in sequence through the cake bed. These extracts were stored, in order, in the full miscella, stage 4, 3, 2, and 1 tanks, respectively. The "full" miscella was stripped and the recovered hexane was returned to the rinse tank for the next batch extraction. The extraction column was turned upside down and spent cake 1 was discharged by the screw conveyor (Fig. 2, J) into the DT unit for desolventizing and toasting. The extraction column was righted, reloaded with batch 2 press cake, and extracted by cycling (10 min) stage 4, 3, 2, and 1 miscellas (obtained from cake 1) in sequence. Hexane in the rinse tank was then pumped through the bed (10 min) (Fig. 3). These five extracts were stored as full miscella, stage 4, 3, 2, and 1 miscellas for the next batch of cake. The "full" miscella was stripped in the solvent/oil recovery system. Spent cake 2 was then desolventized and toasted, and the

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TABLE 1

Mechanical Pressing of *Vernonia galamensis* Seed: Conditions and Results

	Conditioned seed Batch 1		Conditioned seed Batch 2	
	Test 1	Test 2	Test 3	Test 4
Cage coolant in, °F	200	195	198	198
Cage coolant out, °F	180	182	180	179
Shaft coolant in, °F	215	208	208	208
Shaft coolant out, °F	202	195	190	191
Seed moisture in, %	3.5	3.5	3.5	4.4
Cake moisture out, %	2.3	1.6	5.6	4.2
Cone setting, in.	1/16	1/32	3/32	3/32
Cone pressure, psig	500	850	300	200
Feed screw/main shaft, rpm	58/18	58/18	58/18	58/18
Cake:				
Discharge temp., °F	198	204	178	180
Rate, lbs./hr	99	101	93	111
Residual oil, %	20.2	15.8	20.1	19.9
Bulk density, lb/ft ³	—	—	26.3	
Oil:				
Temp. in pan, °F	161	—	152	161
Rate, oil & Fouts, lb/hr	40	51	32	44
Free fatty acid, % by GC ^a	<0.3	<0.3	<0.3	<0.3
Density, g/mL	0.891	0.891	0.891	0.891
Fines, %	5.2	4.6	6.4	5.6
Material Balance:				
Total wt. of cake,		135		129
Total wt. of oil + fines, lb.		60		51
Initial wt. of seed, lb.		216		199
Material lost, %		9.9		9.7

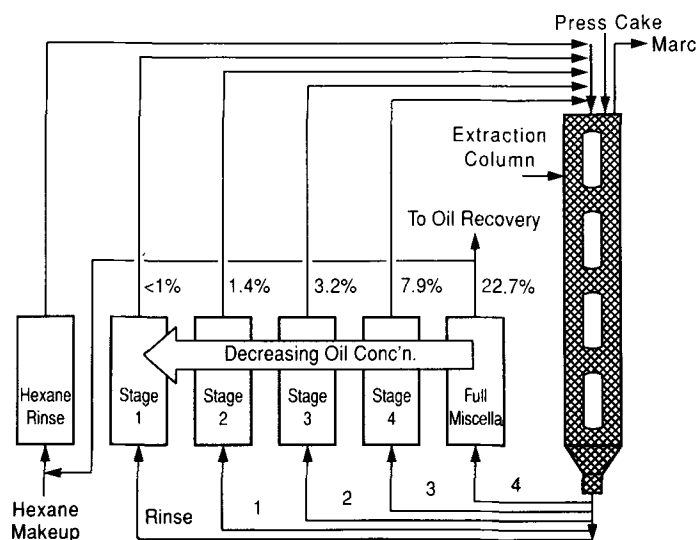
^aReference 14.

FIG. 3. Four-stage miscella and final-rinse extraction scheme with Modular Extraction Pilot Plant. After sequential passage through the extraction column, stages 4, 3, 2, 1 miscellas and hexane rinse become full miscella, stages 4, 3, 2, and 1 miscellas, respectively, for the next batch of press cake.

extraction column was loaded with press cake 3. From this point on, press cakes 3–5 were subjected in similar fashion to this standard four-stage miscella and fresh hexane rinse extraction process (Fig. 3). Miscella concentrations were determined after batch 5 by evaporating known volumes of miscella and weighing the recovered oil.

Initial drainage rates through the press cake beds were 9–10 L/min and fell as low as 6–7 L/min near the end of the extractions. With the 10-min cycle times, contents of each tank passed through the extraction column 2–3 times. After completion of each extraction, spent press cake (marc) was allowed to drain for 25–30 min. Flexible hoses were disconnected by means of quick couplers and the column was turned upside down and coupled to the desolventizer/toaster (DT) unit (Fig. 2). The spent cake was conveyed into the DT, where hexane was stripped by jacket heat and sparge steam. The desolventized meal was toasted at 200–220°F.

Full miscellas were pumped to the oil-recovery system where hexane was first distilled at $\leq 170^\circ\text{F}$ and the concentrated oil was stripped of residual hexane under vacuum (22"). Here difficulties were encountered because vernonia oil foamed excessively under vacuum and the oil concentrate ended up in the condenser system. This was especially serious after all five full miscellas had been concentrated in the stripper. At this point, final stripping of the solvent oil concentrate (87% oil) was accomplished in a laboratory rotary evaporator. Extraction conditions and results are summarized in Table 2.

RESULTS AND DISCUSSION

Equipment used in this study enabled us to relate the results directly to commercial oilseed-extraction processes. The small amount of seed available did restrict the number of experiments we could conduct, but, even so, significant progress was made toward achieving our

TABLE 2

Solvent Extraction of *Vernonia galamensis* Press Cake: Conditions and Results

	Press cake batch into the extractor				
	1	2	3	4	5
Press cake:					
Weight, lb.	44	44	44	44	44
Oil, %	18.6	16.6	18.2	22.6	18.3
Moisture, %	3.8	3.1	4.3	4.1	4.0
Bed height, in.	68	68	64	63	67
Solvent & miscella:					
Temp., °F	130-40	130-40	130-40	130-40	132-38
Volume:					
Hexane, L	150	30	30	30	30
Miscella, L	0	120	120	120	120
Drainage rate, L/min	—	—	6.4-9.0	6.9-10.0	—
Oil concentration, % (w/w):					
Full miscella	—	—	—	—	21.7
Stage 4	—	—	—	—	10.2
Stage 3	—	—	—	—	3.4
Stage 2	—	—	—	—	1.2
Stage 1	—	—	—	—	0.4
Solvent/cake ratio (w/w):					
Hexane/fresh press cake	5.0	1.0	1.0	1.0	1.0
Cum. hex./cum. press cake	5.0	3.0	2.3	2.0	1.8
DT Meal:					
Temp. out, °F	200	200	200	180	220
Weight, lb.	35.5	35.1	35.3	34.5	34.8
Moisture, %	4.9	3.7	6.3	5.5	6.4
Oil, %	1.8	2.0	1.9	2.0	1.9

TABLE 3

Material Balance from Prepress Solvent Extraction of *Vernonia galamensis*

	Feed to process	Intermediate products	Final products
Raw Seed ^a , lb.	415		
Press cake, lb. (%)		270 (65.1)	
Press oil, lb. (%)		111 (26.7)	111
Press recovery, lb. (%)		381 (91.8)	
Press cake to extractor, lb.	220		
DT meal, lb. (%)		176 (80.0)	176
Solvent oil, lb. (%)		28 (12.7)	28
Extraction recovery, lb. (%)		204 (92.7)	
Press cake not extracted ^b , lb.			49.5
Total recovery, lb. (%)			364.5 (87.8)
Oil recovery, lb. (%)			139 (33.5)

^aOil content of seed = 40-42%.

^bResidual oil = 14.8% (7.3 lb.); if recovered, the final oil yield would be 35.2%.

objectives. We obtained 139 lb. of oil, 111 lb. press oil (Table 1) and 28 lb. solvent oil (Table 3)—enough to pursue current research and evaluation objectives. The vernolic acid content of the oils produced was 79-81%, indicating that processing conditions did not alter the epoxy acid content of the oil.

Seed conditioning. The free fatty acid content (FFA) of extracted oil was very low (0.18-0.28%; Table 1) indicating that conditions used for cooking/conditioning the seed inactivated the lipase system in the vernonia seed. Previous work (4) showed that conditions of ca. 200°F for 90 min at ca. 15% seed moisture were adequate for

inactivating the seed lipase and for keeping the FFA content of the extracted oil below 1%. In this study, successful seed conditioning was achieved at 195-200°F for 1 hr at initial seed moistures of 15-20% (9-13% at end of cook). Seed conditioning on a commercial scale could include flaking if the flakes were immediately heated to 200°F, and then sparge steam is injected to quickly raise the flake moisture to 15% for cooking and lipase inactivation. Alternatively, cooked seed could be flaked prior to pressing.

Mechanical pressing. In a pressing operation, there are a number of variables that can be tested. In this study,

we varied discharge cone settings to effect cake thickness and back pressure on the discharge. We did not change shaft arrangement of individual worms and collars, screen bar spacing, temperature of the press shaft and cages, or feed screw and main screw speeds.

Conditions and results are shown in Table 1 for the four short test runs we made on conditioned seed. Tests 1 and 2 were brief attempts to simulate full pressing using cone settings of 1/16" and 1/32" and cone back pressures of 500 and 850 psig, respectively. Press cake discharged at ca. 200°F and at a rate of ca. 100 lb/hr. Residual oil in Test 1 cake (1/16") was 20.2%, and was 15.8% in Test 2 cake (1/32") (Table 1). Oil (and foots) was collected at ca. 160°F at the rate of 40 and 51 lb/hr, respectively. Fines represented ca. 5% of the expelled oil. Oil yields (less fines) were impressive at 27.3% (Test 1) and 32.0% (Test 2).

However, it was apparent that the configuration of the equipment and conditions chosen for Test 1 and 2 did not meet residual oil expectations (5–7%) for a full-press operation. In fact, residual oils in the press cakes (15–20%) were in the proper range for prepressing of a high-oil seed like vernonia (40–42% oil). The following changes would be expected to improve oil removal under full-press conditions. The relatively low fines content of recovered oil indicates that larger screen-bar spacing could be used to allow greater feed-section oil drainage. A modified shaft arrangement with tighter tolerances between shaft parts and inside barrel diameter would create higher pressures, hopefully improving oil removal. Additional seed conditioning such as flaking would rupture oil cells prior to pressing and contribute to lower residual oils. Also, since full pressing is more sensitive to feed moisture content, feeding lower moisture seed or flake to the mechanical press would contribute to improved oil recovery.

To explore prepressing conditions, the second batch of conditioned seed was pressed with a wider cone setting (3/32") and lower cone back pressure (200–300 psig). Residual oil contents of the two press cakes produced (Tests 3 and 4) were essentially the same (20%), although the cake and oil production rates were lower in Test 3 than in Test 4 (Table 1). The lower product rate in Test 3, as compared with Test 4, was partially due to a momentary loss (15–20 sec) of steady state feed during Test 3. Lower cone back pressures and resultant lower cake discharge temperatures (compared with Tests 1 and 2) resulted in higher cake moistures for Tests 3 and 4. Fines contents of the expressed oils were also slightly higher in Tests 3 and 4. Note that the cage and shaft "coolants" exited at lower temperatures, since they served as heat sources for raising the temperature of the press feed (seed). With this high-oil seed, insufficient mechanical energy was generated to raise the press and its contents much above 200°F. The difference in press cake discharge temperatures apparently reflects the difference in mechanical energy generated in the "hard press" runs (Tests 1 and 2) vs the prepress runs (Tests 3 and 4). Bulk density of the cake produced was 26.3 lb/ft³. Oil yield (less fines) for Test 3 was 24.0%, the lowest of the four tests. Oil yield for Test 4 (26.8%) was ca. the same as for Test 1. Material balances (Table 1) show that oil yield (less fines) for batch 1 conditioned seed averaged 29.3%, and for batch 2 conditioned seed averaged 26.6%.

Since longer residence time in the press is important

to oil yield, a soft seed like vernonia could benefit by operating at slower main shaft speeds, although this would result in a trade-off with lower machine capacity. Also, press configurations for common oilseeds are designed so that oil drains most heavily from the feed section of the press and drainage decreases as material approaches the discharge section. With the press configuration used in this study, most oil drainage occurred in the middle section with very little drainage at the feed end. A tighter shaft arrangement and larger spacer-bar spacings would create higher drainage toward the feed section and should result in lower residual oil in the cake.

Solvent extraction. The 44-lb. press cake batch size utilized 88–94% of the 1.5 ft³ capacity of the extraction column (0.5 × 0.5 × 6 ft). Oil contents of the cakes (Table 2) ranged from a low of 16.6% (Batch 2) to a high of 22.6% (Batch 4), and averaged 18.3%. Moisture contents ranged from 3.1–4.3% (3.9% average).

Each fresh batch of press cake was carried through similar four-stage miscella and final-rinse extractions (Fig. 3). Thus, except for the batch operation, the system simulated a commercial oilseed-extraction process. Table 2 shows that drainage rates during extraction (Batch 3 and 4) ranged from 10 to 6 L/min, with drainage slowing as the column of cake shrunk during oil removal (4–6"). Miscella concentrations were determined only after batch 5 (Table 2) and ranged from 22% oil in the full miscella to ca. 0.4% in stage 1 miscella (w/w). We assume that miscella concentrations reached equilibria by the third or fourth batch of press cake.

For batch 1 cake, 150 L (218 lb.) of hexane extracted 44 lb. of cake for a solvent/cake ratio of 5:1 (w/w). Thereafter, a solvent/cake ratio of 1:1 is calculated on the basis of 30 L (43.6 lb.) of fresh hexane for each 44 lb. of press cake (Batches 2–5, Table 2). On the other hand, if the total hexane + miscella that is pumped through each batch (44 lb.) of cake is used in the solvent calculations, then per-batch solvent/cake ratios of 5:1 are determined. For purposes of this study, a meaningful ratio can be determined from the cumulative amount of fresh hexane used (e.g., 210 L or 305 lb. for Batch 3) and a cumulative weight of cake extracted (e.g., 132 lb. for Batches 1–3). This comparison gives solvent/cake ratios of 5.0, 3.0, 2.3, 2.0, and 1.8 for the five batches (Table 2). This does not take into account the small amount of solvent makeup (1–2%) required due to hexane loss during each full miscella distillation and spent-meal desolventization.

Regardless of how the solvent/cake ratio is calculated, residual oil content of the DT meals are the same (1.9%) for each batch of press cake (Table 2). This result is interesting since it shows that a five-stage hexane wash (Batch 1) is no more efficient at removing vernonia oil than are the following four-stage miscella-and-rinse extractions (Batches 3–5). It is not clear from the results what steps should be taken to reduce residual oil content of the DT meal to <1%. Possibly this residual oil is primarily in the form of less soluble hydrated gums, in which case lower moisture levels at the pressing and extraction phases might help. Also, flaking the seed as part of the conditioning phase might contribute to more efficient oil removal in the solvent-extraction step.

Table 3 summarizes material balances at different stages of processing. Mechanical pressing recovered 111 lb. of oil (26.7%) and solvent extraction recovered another

28 lb. of oil from the press cake (12.17%). Total oil recovery was 139 lb. (33.5%). Foaming in the oil stripper contributed to significant loss of oil (8–9 lb.) in the oil/solvent recovery system. Very careful control of vacuum and temperature in the stripper, using a higher capacity stripper, or using antifoaming agents, would minimize this problem. Material losses of 7–8% in both the pressing and solvent-extraction operations are probably acceptable for these batch operations. Overall recovery was 88% (Table 3).

In summary, this was a successful test of pilot-scale prepress/solvent extraction of oil from *Vernonia galamensis* seed. Improvements could be made in the process by adjusting the configuration of the mechanical press to increase oil expression, especially with regard to a full-press operation. This needs to be examined further, since oil production in developing countries of Africa may utilize only mechanical pressing. Prepress conditions were not difficult to establish. Flaking vernonia seed as part of the seed-conditioning phase would probably increase oil recovery in both the pressing and solvent-extraction steps, and additional reduction of moisture level (to ca. 2%) also is expected to improve oil-extraction efficiency at both steps. It is anticipated that extraction of vernonia oil should be competitive with other high-oil seeds such as cottonseed, rapeseed, and sunflower seed.

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