Pilot Plant Extraction of Oil from Vernonia galamensis Seed


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 diepoxide esters of high purity (≥90% 9,10,12,13-diepoxystearates) 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” x 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” x 6” x 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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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...