Continuous Abrasive Method for Mechanically Fractionating Flaxseed

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ABSTRACT: Both the flaxseed hull, which is rich in the lignan secoisolariciresinol diglucoside (SDG), and the embryo, which is rich in oil with high α-linolenic acid content, are of interest for functional food use. A mechanical process for preparing hull- and embryo-rich fractions was developed and characterized. The process consisted of three pearlers, a sifter that yielded fines, and a gravity table that yielded final hull and embryo fractions. The SDG contents of fractions correlated inversely with oil content, showing that oil content indicated purity of both embryo and hull fractions and that the fines were essentially hull particles. Process performance depended on seed variety, moisture content, and feed rate; the best yields were 285 g hull + fines/kg for low-moisture Omega flaxseed, and 470 g embryo/kg seed for low-moisture Neche. Corresponding oil contents of those fractions were 28.8 and 47.4%, respectively. This process appears to be commercially feasible, provided it can be scaled up. A single stage was used to identify features that should be incorporated into a scaled-up unit, for example a 7-mesh screen in the pearler chamber rather than 6- or 8-mesh, and use of an overhead feed inlet to the chamber to direct seed into the disk-plate gap.

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Flax seeds are rich in the n-3 FA α-linolenic acid and the lignan secoisolariciresinol diglucoside (SDG), and apparent health benefits are associated with these and other flaxseed components (1). Both components are therefore of interest as sources of functional foods. The separation from one another and concentration of these two components should allow them to be used in food products in controlled amounts and in ways that are most compatible with the desired attributes of the food.

A flax seed has two flattened cotyledons that constitute most of the embryo, and the embryo is enveloped by a seed coat that consists of true hull (testa) and an adherent layer of endosperm. The terms "embryo" and "hull" are used hereafter to denote these two main products of flaxseed fractionation. Studies using hand-dissected seed showed that the hull constitutes 40 to 41% of the seed weight (2). SDG is mainly in the hull (3). Oilseed hulls typically have little or no oil, but the oil content of flaxseed hull is not low because of the adherent endosperm tissue. The hull also is particularly rich in water-soluble mucilage or fiber (4). The embryo accounts for the remaining 59 to 60% of the seed, and this tissue contains up to 57% oil. Thus, flaxseed dehulling produces a lignan-rich fraction (hull) and an oil-rich fraction (embryo). The embryo is low in fiber and thus would be an excellent alternative to water-washed flaxseed used to boost n-3 FA in farm-raised fish (5).

The flaxseed hull does not readily detach from the embryo; however, partial dehulling of flaxseed using flaking rolls and sieves was reported more than 50 yr ago (6). Solvent-based methods for concentrating flaxseed protein in defatted cake also have been reported, but recent studies have focused on purely mechanical methods. The use of mechanical methods is compatible with products to be labeled "organic" under the U.S. National Organic Program (7). Use of a batch, abrasive unit called a TADD (Tangential Abrasive Dehulling Device) was initially tested using 5 × 40-g batches of flaxseed (8) and later adapted to 4-kg batches (9). Drying the seed to very low moisture content (<3.5%) using microwave drying appeared necessary to get good results using the TADD. Moisture at this range is tightly bound in flaxseed; thus, energy could be a significant cost. An alternative process used an Urschel Comitrol mill to shear flaxseed into coarse pieces, followed by aspiration to collect a portion of loose flakes of hull (3). This process is continuous, can be easily scaled up, and does not require a drying step; however, hull yield is low (10%), and a pure embryo fraction is not produced.

Exploratory tests showed that the abrasive approach could be adapted to a continuous operation. This approach offered the potential for a process that combined advantages of the alternatives described above. Thus, the objectives of this study were to develop a continuous abrasive process, to evaluate the performance of this process, and to identify key factors for future scale-up.

EXPERIMENTAL PROCEDURES

Source of materials. Neche flaxseed (brown-seeded variety) was from Werth Certified Seed (Lehr, ND) with 7% moisture content. Omega flaxseed (yellow-seeded variety) was from Reimers Seed Company (Carrington, ND) with 9% moisture content, except Omega seed used in a latter investigation of a single-stage process was from Red River Commodities

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(Fargo, ND) with 6.8% moisture content. This original seed, termed "As Is," was stored in plastic bags at 4ºC and then equilibrated to room temperature 18 to 24 h prior to use. "Dry" flaxseed (4 and 5% moisture content for Neche and Omega, respectively) was prepared by placing seed in burlap bags in a 50°C walk-in dryer for 72 h.

Process overview. Duplicate 10-kg lots of flaxseed were processed on separate days by abrasive dehulling, sifting, and fractionating. Product fractions were weighed, recorded, labeled, and stored in plastic bags at 4°C for further analysis. Hull, embryo, and fines fractions were analyzed for moisture and oil contents. A subset of samples was analyzed for SDG content.

Abrasive dehulling step. The abrasive dehulling unit consisted of a three-stage unit, except where noted otherwise. Three Seedburo barley pearlers (Seedburo Equipment Co., Chicago, IL) were aligned vertically on a steel frame, such that product from the first, uppermost pearler flowed by gravity into the second pearler, and product from the second pearler flowed into the third pearler. Each pearler contained a 30-grit stone disk that was 25.4 mm thick × 165 mm diameter. The curved surface of the disk had 18 notches (4 mm deep × 4 mm wide) that spanned the width of the disk and were spaced at 25-mm intervals. The disk was direct driven at 1730 rpm by a ¼ hp (190 W) fixed-speed 110 VAC electric motor. The curved surface of the disk was surrounded by a 7-mesh screen spaced 10 mm from the disk. The two flat surfaces of the disk were opposed by parallel plates having unfinished, sand-cast surfaces. The gap between disk and plate was 8 mm on each side of the disk. Flaxseed entered the pearling chamber just above the axis of rotation of the disk through an external hopper on the plate opposite the motor, except where noted otherwise. A screw feeder (Model E-2; Zero-Max Co., Minneapolis, MN) was calibrated to deliver flaxseed to the first pearler at either 75 or 150 g/min. The actual average feed rate was determined by measuring sample run time for each 10-kg lot. Pearlers were modified to permit continuous, steady-state operation by (i) replacing the timer with a toggle switch, and (ii) replacing the sample collection drawer below the pearling chamber with a funnel to direct product to the subsequent stage. The funnel consisted of two flat slides inclined 35° relative to horizontal for directing product into a square tube (57 mm i.d.) at the front center of the pearler base. The tube then directed product vertically into the hopper of the next stage.

Sifting step. Flaxseed discharged from the third pearler stage dropped into the base of a screw-conveyor that elevated product to a continuous-flow, flat-bed sifter. The screw conveyor consisted of a 1.9 m long × 0.1 m o.d. steel tube encasing a mild steel helicoid flight screw with a 5.4-cm diameter and 5.5-cm pitch that was inclined 45° relative to horizontal. The conveyor screw was turned at 12 rpm. The sifter (Rotex, Style No. 12; The Orville Simpson Co., Cincinnati, OH) shook the 1.0 m long × 0.4 m wide 18-mesh screen at a frequency of 140 cycles/min with a 5-cm amplitude.

Fractionation step. The coarse product from the sifter was separated into three fractions—hull, embryo, and intact plus cracked seed—using a gravity table with a 12-mesh deck (Model 10-M2; Forsberg, Thief River Falls, MN). The trapezoidal-shaped deck measured 47 cm at the back base, 82 cm at the front base or discharge end, and 43 cm at the feed edge (low side). Deck oscillation was 120 cycles/min with an 11-mm amplitude. The coarse fraction from the sifter was fed to the deck *via* vibratory feeder at a rate of 650 to 700 g/min. Airflow through the deck mesh was 0.7 to 2 m/s as measured by an electronic anemometer (Turbo Meter; Davis Instruments, Hayward, CA) when no seed was present. The feed edge was inclined 2 to 4° (side slope) upward from the front base. The pitch of the front base was 4 to 9° (end slope). Airflow, side slope, and end slope were adjusted by the operator as needed to improve separation. A typical fractionation required up to 5 min time to achieve steady separation, during which time all product was recombined and recycled to the feed. The hull fraction was collected from the zone of the front base that extended from its left end to a point 8 to 12 cm from that end. The embryo fraction was from a zone of the front base between two points located at 18 and 32 cm, as measured from the left end. The fraction between these two zones consisted of a mixture of hull and embryo pieces that was recycled to the feed.

Moisture and oil contents. Intact seed, hull, and embryo samples were milled to pass through a 1-mm screen in an ultracentrifugal mill (model ZM1; Brinkmann Instruments Co., Westbury, NY) before analysis. Moisture content was determined with an IR moisture analyzer (Mettler LJ 16 Dryer; Mettler-Toledo, Inc., Hightstown, NJ). Duplicate 3- to 4-g samples were heated in a 7.5-cm diameter aluminum dish at 120°C. The instrument was programmed to stop automatically when the rate of weight loss fell to less than 2 mg/30 s.

Duplicate 2.5 to 3.5 g samples were extracted at 70°C with hexane for 16 h using a Soxhlet apparatus. The solvent was evaporated gently under vacuum pressure at 40°C using a rotary evaporator and the residual oil weighed for calculation of oil content. The resulting defatted meal was air dried at room temperature in a fume hood, labeled, and stored in plastic bags at 4°C for SDG analysis.

SDG content. Defatted fractions from the oil content analysis were milled to pass through a 0.25-mm screen. Extracts for HPLC analysis were prepared as described by Westcott and Muir (10) with minor alterations (3). A 0.5-g sample and 10 mL aqueous methanol (700 mL/L) were added to a screw-capped test tube (16 × 100 mm). Tubes were vortexed for 30 s, then incubated in a circulating water bath for 3 h at 60°C. Tubes were again vortexed during incubation for 10 to 15 s at 30-min intervals. After incubation, tubes were cooled to room temperature (22°C) under tap water. The contents were centrifuged for 20 min at room temperature and 3000 × *g*. Supernatant (2 mL) was transferred to a second screw-capped test tube (16 × 100 mm), to which was added 0.5 mL of 20 g/L aqueous sodium hydroxide. Contents were vortexed 15 s then held 3 h at room temperature before being neutralized with 0.5 mL 30 g/L aqueous acetic acid. About two-thirds of the neutralized extract was transferred into a 3-mL

latex-free syringe fitted with a 0.45-µm nylon Acrodisc membrane microfilter (Gelman Science, Ann Arbor, MI), filtered directly into a 2-mL amber vial, and stored at 4°C before HPLC analysis.

HPLC was performed using a Hewlett-Packard model 1090 (Waldbronn, Germany) fitted with an autosampler, 0.2-µm in-line filter, photodiode array detector, and integrator. The column was a Lichrosphere 100 RP-C18 column (E. Merck, Darmstadt, Germany) with 5-µm particle size and 4 mm i.d. × 250 mm length. Injection volume was 10 µL. Solvent A was water containing 10 mL/L acetic acid and solvent B was methanol. The following five-step linear gradient profile was run at 1 mL/min over 60 min: A = 5% and B = 95% at 0 min; A = 60% and B = 40% at 44 min; A = 60% and B = 40% at 48 min; A = 5% and B = 95% at 55 min; and A = 5% and B = 95% at 60 min. The column temperature was held at 40°C, and the column effluent was detected at 280 nm. A scan of the SDG peak was performed at 190 to 600 nm. A series of five different standard solutions of SDG ranging from 0.0625 to 1.0 mg/mL aqueous methanol (500 mL/L) was used. The relationship between peak area at maximal absorbance and concentration was linear in this concentration range.

Statistical analysis. ANOVA and Duncan's Multiple Range Test were performed using the General Linear Model Procedure (SAS System for Windows, release 8.2, SAS Institute, Cary, NC). Regression analysis and calculation of SD were performed using Microsoft Excel (Redmond, WA).

RESULTS AND DISCUSSION

Evaluation of the three-stage process. Pearler performance was compared for both Omega and Neche flaxseed at two seed moisture contents ("As Is" and "Dry") and two feed rates ("As Is" and "High"). Omega yielded less embryo and fines but more hull compared to Neche (Fig. 1A). The yield of intact plus cracked seed is not shown, but this fraction accounted for nearly all of the remainder of the flaxseed. The lower proportion of hull fraction to embryo fraction from Neche suggested that the fines fraction consisted mainly of hull particles and that a higher proportion of hull was retained in the intact plus cracked seed. The difference in the proportion of hull to embryo in these two varieties probably is small. Oil contents of Omega fractions were higher than for the corresponding Neche fractions in all instances (Fig. 1B), partly reflecting the higher oil content of the intact Omega seed (40.9 and 40.2% for Omega and Neche, respectively). Analysis of seed samples by the State Seed Department (Fargo, ND) showed similar seed purity, seed count, and test weight [99.4%, 184,000 seeds/kg, and 51.0 lb (= 23.1 kg), respectively, for Neche vs. 99.4%, 180,000 seeds/kg, and 50.5 lb (= 22.9 kg) for Omega].

Pearling of "Dry" seed resulted in a large increase in embryo and fines yields for both varieties relative to pearling "As Is" seed. However, hull yield was apparently not improved by drying. Again, this suggests that the fines fraction consisted mainly of hull particles; the decreased moisture content may make the hull more brittle. Fractional oil content was not significantly affected by drying ($P \leq 0.05$).

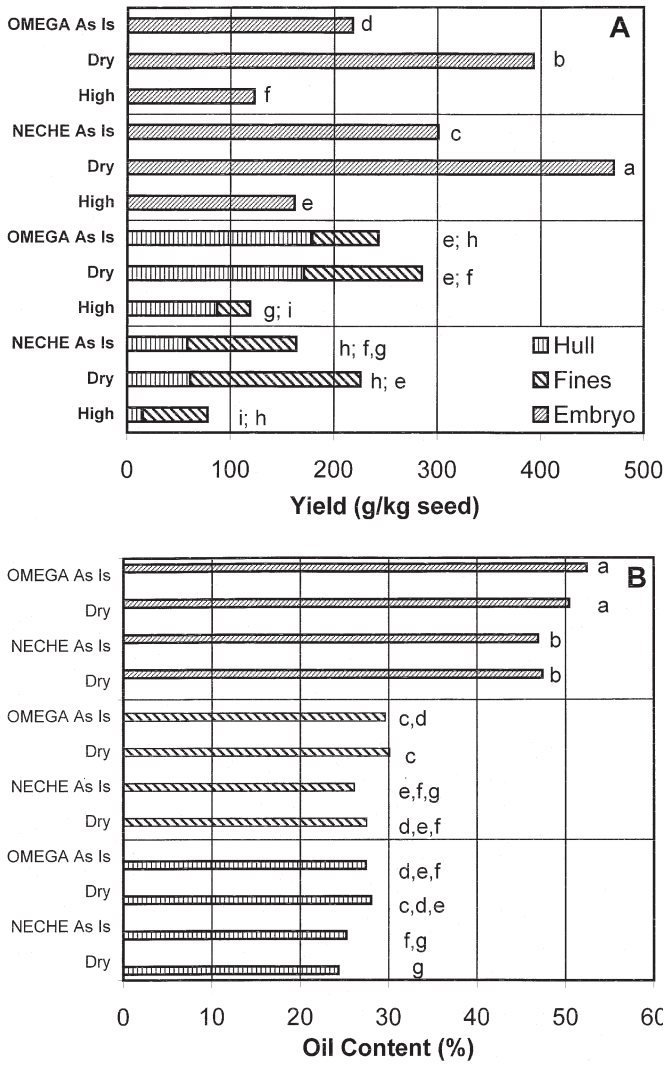

FIG. 1. Yield (A) and oil content (B) of flaxseed fractions produced by an abrasive dehulling process. "As Is" denotes seed that was dehulled with moisture content as received (9 and 7% for Omega and Neche, respectively) and at a baseline feed rate (75 g/min) to the dehulling unit; "Dry" denotes seed that was dried to 5 and 4% for Omega and Neche, respectively, before dehulling at the baseline rate; and "High" denotes seed that was fed as received to the dehulling unit at twice the baseline rate (150 g/min; not shown in B). Yields having the same letter to the right of the bar are not significantly different ($P \leq 0.05$). The letter for the hull in A precedes that of the corresponding fines.

Doubling the pearler feed rate relative to the baseline feed rate of 75 g/min reduced by half the embryo, fines, and hull yields for both varieties (Fig. 1A). Consequently, the productivity (yield × feed rate) of each type of fraction remained about constant; thus, the increased feed rate merely served to dilute the product with intact seed. This feed rate comparison demonstrated the limited capacity of this pearler system. One way to scale up capacity would be to incorporate multiple disks alternating with stationary plates on a single shaft in

each stage, but fabrication of scaled-up equipment was beyond the scope of this study.

The strong effect of feed rate also suggested that close attention should be given to maintaining a uniform, well-defined feed rate for studies of this process. Our earlier pearler results with a vibratory feeder (not shown) gave poor reproducibility, probably owing to nonuniform feed rate. Feed rate drifted downward with time at a given vibratory feeder setting until the feeder chute was cleaned to remove the coating of grain dust that gradually accumulated. Thus, the screw feeder used to produce the results reported here was first evaluated carefully for uniformity of feed rate, calibrated to achieve the desired feed rate, and then the actual average feed rate was calculated from the run time for that test. The average feed rate was 0 to 8% greater than the target rate in these tests.

Initially, we thought that friction in the pearler chamber would result in a gradual temperature rise of the equipment surfaces and product, thereby causing results to be dependent on time and potentially causing thermal stress to the oil in the products. Thus, the temperatures of the pearler wall and disk and pearled seed were monitored using a noncontact IR thermometer. These temperatures were shown to remain between 24 and 29°C; thus, the concern for friction-induced heating was unfounded. Temperature monitoring did show, however, that the pearler motors became hot after 20 min of operation; thus, regular lubrication and use of cooling fans were incorporated into the protocol to protect the motors.

Fraction purity. The large sizes of embryo and hull pieces permitted very good separation of the two particle types on the gravity table. Exploratory studies showed that the gravity separation produced much better separation than sieves or aspiration. Nevertheless, even using the gravity table, visual inspection showed that some embryo pieces remained in the hull fractions and that some hull pieces remained in the embryo fractions. A small amount of Neche hull and embryo samples that were pure to the eye was prepared for analysis by handpicking pieces of these two components from the gravity table fractions.

Sample analysis showed that SDG content varied inversely and linearly with respect to oil content (Fig. 2). The SDG content of the handpicked hull sample was 46-fold greater than that of the handpicked embryo. The residual SDG detected in the embryo may have been contributed by particles of hull not apparent to the eye. SDG content likely is an indicator of purity of both hull and embryo fractions. A simple measure of purity would be a very useful performance measure of a fractionation process during process optimization. However, we were unable to analyze SDG content quickly and easily. Oil content was much more easily measured than SDG content, and the excellent correlation between SDG and oil content suggested the use of oil content as a routine indicator of hull and embryo purity. The precise relationship between oil content and SDG content likely depends on the seed variety and on growing location and conditions. Thus, the oil contents in Figure 1B can be compared for relative purity between like fractions and variety, but not for relative purity of Neche fractions vs. Omega fractions. The correlation in Figure 2 is not valid for samples that were screw-pressed to reduce oil content.

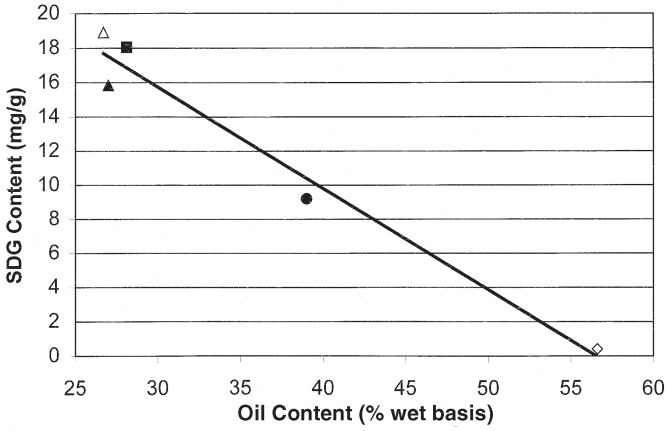

FIG. 2. Inverse linear relationship between secoisolariciresinol diglucoside (SDG) content (SC) and oil content in flaxseed fractions produced mechanically (SC = 33.5 − 0.593OC; r^2 = 0.97). Points denote handpicked hull (△), gravity table hull (▲), fines (■), intact seed (●), and hand-picked embryo (◇).

The oil content of the hull fractions appears high, even in the handpicked fractions. Yet this content was consistent with other reported values for hand-dissected hulls (2) and the hull fraction obtained from the TADD (8,9). As mentioned earlier, the high oil content resulted from adherent endosperm tissue. However, lower hull oil contents have been reported in hulls aspirated from coarsely ground samples (3). The proportion of endosperm tissue that adheres to the hull probably varies from seed to seed and is likely influenced by the method of dehulling. Cutting and impact forces may tend to fracture the seed at a different plane compared to abrasive forces and thereby result in hull that retains less endosperm.

The fines fraction is represented by the solid square on the upper left side of Figure 2, confirming that the fines are essentially hull particles since they are not very different in SDG and oil contents from the hull fraction. Thus, the sum of hull and fines fractions is a valid indicator of actual hull yield, hence the combination of these two fractions in Figure 1A. The highest combined yield (hull + fines) was 285 g/kg seed for dried Omega. This combined yield was viewed as a very encouraging result considering the inherent difficulty of dehulling flaxseed. The oil content (weighted average) of this combined fraction was 28.8%.

Studies to increase purity and yields of hull and embryo fractions. The initial selection of three pearling stages was somewhat arbitrary. We subsequently compared the use of one, two, and three pearlers in this process and found a nearly proportionate increase in the yields of hull and fines with each added disk (Fig. 3). This linear trend was not necessarily expected; it was thought before this study that either (i) passage through the initial stage might weaken hull attachment, thereby predisposing that kernel to dehulling in a latter stage; (ii) seed that remained intact after the first stage might be inherently resistant to hull removal, in which case the second

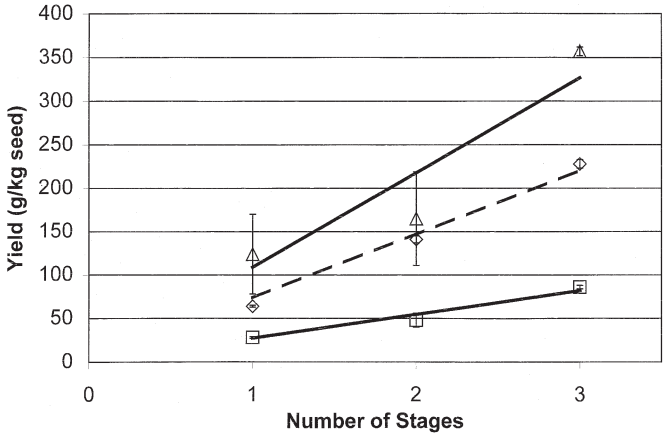

FIG. 3. Proportional increase in yield of flaxseed fractions with number of dehulling stages. Symbols denote gravity table embryo (—△—), gravity table hulls (– –◇– –), and fines (—□—). Error bars represent one SD.

and third stages might accomplish relatively little additional dehulling; or (iii) there might be diminishing return, in terms of incremental hull yield increase, in accord with a first-order model. Considering that the hull yield with three stages was still far below the 40 to 41% maximum achievable yield, one might suppose that addition of one or more pearler stages is desirable. This addition does not appear practical, however, because accumulation of fines in the third stage required stopping the process periodically for cleaning, and this problem would be greater with additional stages.

An alternative approach to increasing hull and embryo yields is to recycle back to the pearlers the fraction from the gravity table containing intact and cracked seed. The recycle step was evaluated in the case of Omega flaxseed at the same two seed moisture contents used above. The yield increase was especially pronounced for "As Is" seed (to 330 and 390 g/kg for hull + fines and embryo, respectively), because this seed had a much higher proportion of intact and cracked seed after the first pass compared to "Dry" seed. The corresponding yields with "Dry" seed were 320 and 465 g/kg for hull + fines and embryo, respectively. This evaluation showed that seed that remained intact after the first pass through the three pearler stages is not especially resistant to dehulling. The relatively small amount of recycled fraction that was available resulted in less accurate gravity table separations compared to the first-pass results. This less accurate separation was reflected in the lower fraction purity of second-pass fractions, as indicated by higher oil content in the hull + fines fractions (28.7 and 29.1% for "As Is" and "Dry," respectively) and lower oil content in embryo fractions (51.5 and 49.9% for "As Is" and "Dry," respectively). Nevertheless, these results probably more accurately reflect the hull and embryo yields that can be achieved in a commercial process than do the single-pass results.

Improvements in hull and embryo yield and purity were also demonstrated using a two-step gravity table separation in which the first step was optimized for hull and the second step for embryo (data not shown). This two-step separation would not likely be needed given a full-size gravity table, as a better separation should be expected relative to the bench-scale gravity table used in this study.

Studies related to future scale-up. An important limitation of the process evaluated in this study was low capacity. Obviously, this process should be scaled up in order to be of practical value. The trends in Figure 3 suggested that the performance of a single stage can be used to predict performance of a three-stage process. This knowledge will help streamline the evaluation of pearler modifications. Thus, several pearler modifications were evaluated with a single pearler stage. Only Omega flaxseed was used in these evaluations.

The results in Figures 1–3 were obtained with pearler units that used a 7-mesh screen to retain seed within the pearler chamber. However, this mesh size is less available commercially, such as for equipment fabrication of a scaled-up prototype, than 6- and 8-mesh screens. Also, it was thought that 6- or 8-mesh screens might give results similar to the 7-mesh for flaxseed dehulling. The finer 8-mesh screen actually yielded significantly less embryo fraction ($P \leq 0.05$) and somewhat less hull fraction (Fig. 4) and became plugged in some process tests. Tests with a 6-mesh screen were stopped before completion when it became apparent that a very high proportion of seed passed through the unit intact. Apparently, the openings in 6-mesh screen are too large. Thus, the 7-mesh screen was used in subsequent tests.

The pearlers used in this study introduced the seed into the pearling chamber close to the axis of rotation on one side of the disk. However, in a scaled-up three-stage process with each stage having multiple disks alternating with stationary plates on a single shaft as suggested above, it would be

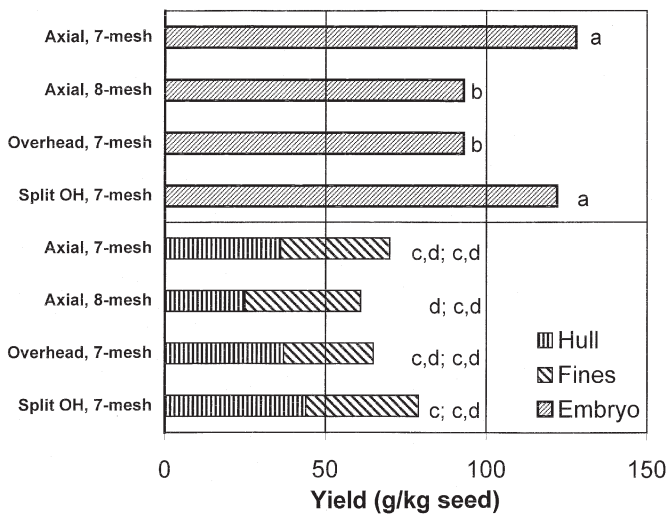

FIG. 4. Yield of flaxseed fractions from a single-stage abrasive dehulling process with various modifications to the pearler chamber. Axial, Overhead, and Split OH (split overhead) refer to the feed inlet into the pearler chamber, and 7-mesh and 8-mesh refer to the screen within the pearler chamber. All tests were performed with 75 to 77 g/min Omega flaxseed having 6.8% moisture content. Yields having the same letter to the right of the bar are not significantly different ($P \leq 0.05$). The letter for the hull precedes that of the corresponding fines.

simpler if seed were introduced at the top of the pearler chamber. It was thought that this also might make better use of both faces of each disk. This inlet location was tested with a single-stage pearler that was modified by creating an opening 49 × 36 mm immediately above the pearler chamber through which feed was funneled; this modification resulted in the same hull yield but significantly less embryo yield compared with the original axial feed configuration (Fig. 4). In a refinement of the overhead feed inlet, feed was split and directed into the two gaps between disk and stationary plate. The reason for this modification was that abrasion between disk and screen was thought less effective than between disk and plate; therefore, feed should be directed toward the disk–plate gap. This method of feeding was shown to be at least comparable to the axial configuration (Fig. 4) and would be simple to implement in a scaled-up system.

Fines fractions from the single-stage process tended to have less oil than the corresponding hull fractions, indicating that they were more enriched in SDG. This fines characteristic was different from the three-stage process but was not surprising since a multiple-stage process increases exposure of embryo particles to abrasion. Unfortunately, conditions that produced fractions with the least oil also were associated generally with the lowest overall yield. For example, the lowest oil content for a fraction in this study (21.6%) was found in fines from the 8-mesh screen study. With the exception of fractions obtained with the 8-mesh screen, oil contents from the single-stage study were similar among like fractions ($P \leq 0.05$).

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