A Multistage Hydrocyclone/Stirred-Tank System for Countercurrent Extraction of Canola Oil

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Ground canola seed containing 46.9% oil (A) and partially extracted meal, similar to pre-pressed meal containing 13.7% oil (B), were ground in a methanol/ammonia/water solution, filtered to remove antinutrients and extracted countercurrently with hexane in a multistage hydrocyclone/stirred-tank extraction unit. An empirical model was developed for predicting the yield (*i.e.*, oil recovery) from the process. Based on the model calculations, a six-stage unit operating at a hexane-to-meal ratio (R) of 6.2 L/kg was required for processing meal A. The calculated oil recovery was 98.3%, resulting in a meal containing 0.7% residual oil. Meal B required a five-stage unit operating at R = 5.7 L/kg. The calculated oil recovery was 99.2% with 0.6% residual oil in the meal. The calculations were confirmed experimentally with two- and four-stage crosscurrent extraction processes.

KEY WORDS: Canola, canola meal, canola oil, extraction, food engineering, hydrocyclones, modelling, slurry grinding.

A novel approach to rapeseed processing has been developed in our laboratory (1–5). The process uses methanol containing 10% w/w NH_3 and 5% vol/vol H_2O ($CH_3OH/NH_3/H_2O$) in which the seed is ground as a slurry in a Szego mill (General Comminution, Inc., Toronto, Canada). The slurry is extracted with hexane in a three-phase system to recover the oil and to produce a high-quality meal, which is essentially free of glucosinolates and low in polyphenols. The oil typically contains less than 50 mg/kg phosphorus.

Conventional percolating-bed extractors are unsuitable for the extraction of oil from the finely ground solids from the above process. The purpose of this work was to design a countercurrent oil-extraction process based on hydrocyclones that could be readily scaled up for industrial use. An empirical model was developed based on the performance of a single hydrocyclone. The model was used to determine the optimum process design for maximum oil recovery. The model predictions were tested with two- and four-stage crosscurrent extraction processes.

Hydrocarbons were selected for the miscella/meal separation because they have been used effectively to separate fine solid particles from liquids in a number of industrial applications (6–8). They are very compact and have a low capital and maintenance cost (9,10). They generally have a small liquid hold-up and are easy to operate continuously.

MATERIALS AND METHODS

Meal preparation. Certified Westar canola seed (1988) with an oil content of 46.9 ± 0.5% (moisture-free) was used throughout the study. A meal slurry was prepared by two-pass grinding of canola seed slurried with $CH_3OH/NH_3/H_2O$ (~85/10/5) at a solvent-to-seed ratio R = 6.0 L/kg in a Szego mill/hydrocyclone system as described by Adu-Peasah *et al.* (5). After grinding, two processing approaches were followed in an effort to investigate the effect of prepressing on the extraction efficiency of the system. In the first case, B, the meal slurry was pretreated to reduce the meal's oil content to 13.7 ± 0.5% by mixing it with hexane at R = 3.5 L/kg. In the other series, A, the seed slurry was not pretreated. Both slurries were then vacuum-filtered in a Buchner funnel with Whatman No. 41 filter paper (Maidstone, England), and washed twice with methanol at R = 2.0 L/kg.

The effect of the residual methanol content of the pretreated meal on the extraction was investigated. The final methanol content of the meal was adjusted by the addition of methanol to a predetermined level (20–60%), based on the gravimetrically determined methanol content of the meal.

Hydrocyclone apparatus. A Bauer model 500 hydrocyclone (CE Bauer Co., Brantford, Ontario, Canada) was used to separate the miscella from the meal. It was made of #316 stainless steel and had the following dimensions: chamber diameter 25.4 mm; inlet diameter 4.76 mm; underflow diameter (fully open) 4.76 mm; cone angle 10.2°. The hydrocyclone/stirred-tank apparatus is illustrated in Figure 1.

In a typical hydrocyclone run, 1 kg of pretreated meal was slurried with hexane in the stirred tank. The slurry was agitated at 1200 rpm and recirculated through line 4 for 4 min. At this point, Valve a was closed and Valve b opened, pumping the slurry into the hydrocyclone. Both the overflow and underflow were recirculated to the stirred tank for approximately 30 s, allowing the system to

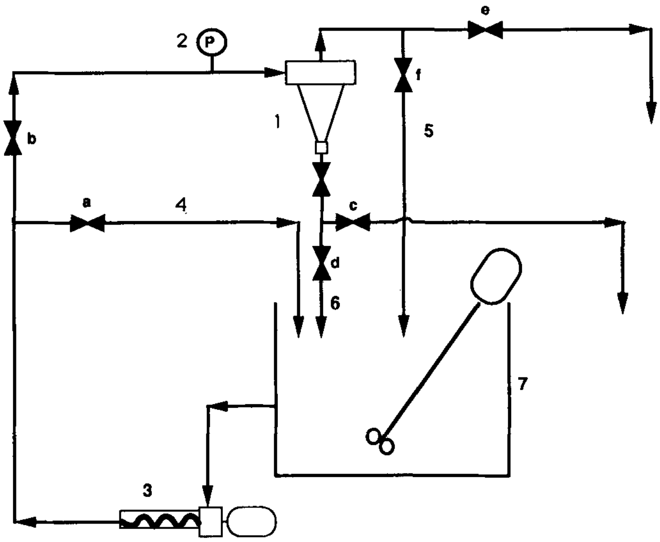

FIG. 1. Schematic diagram of the hydrocyclone/stirred-tank extraction apparatus. 1: hydrocyclone; 2: pressure gauge; 3: Moyno pump; 4: feed recirculation line; 5: overflow recirculation line; 6: underflow recirculation line: 7: feed tank; a, b, c, d, e and f: Valves. P indicates a pressure gauge.

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stabilize. At that point, Valves c and e were opened and d and f closed, and the overflow and underflow streams were collected.

The size of the underflow opening of the hydrocyclone is an important parameter, which determines the amount of the feed material discharging through it. To modulate this parameter, the underflow outlet of the hydrocyclone was connected to a ball valve, which could be set at three positions corresponding to the following underflow-to-overflow area ratios: $A_r = 1.00$, 0.93 and 0.85. The A_r values were determined experimentally as described by Adu-Peasah (11) by using empirical equations developed by Svarovsky (10).

Preliminary studies. Prior to the design of a multistage extraction unit, the effects of solids (*i.e.*, oil-free solids or marc) concentration in the feed suspension, methanol content of the meal, pressure drop in the hydrocyclone and the size of the underflow opening on the composition of the overflow and underflow streams were determined.

The final step in meal preparation was a methanol wash, resulting in meals that typically contained 40–65% methanol. The initial methanol content of the meal was varied between 0 and 55 wt%. The meal was contacted with hexane in the stirred tank at a solvent-to-seed ratio (R) = 3.5, and the mixture was separated by the hydrocyclone at 3.1×10^5 Pa with the underflow value at position $A_r = 1.00$.

The effect of pressure drop was investigated. The meal was mixed with hexane at R = 3.5 in the stirred-tank and separated in the hydrocyclone at varying pressures between 1.38×10^5 Pa (20 psi) and 3.45×10^5 Pa (50 psi) at $A_r = 1.00$.

Because preliminary studies indicated that 45% was the maximum initial methanol content that produced a solids-free miscella in the overflow, 45% methanol was used to determine the effects of oil-free solids (marc) concentration in the feed suspension and the size of the underflow opening on the separation. Approximately 1 kg of solvent-ground meal was mixed with varying amounts of hexane in the stirred tank to produce slurries with solids concentration between 0 and 23%. The hexane slurry was pumped to the hydrocyclone at 3.1×10^5 Pa (45 psig) and split by using underflow valve settings corresponding to $A_r = 1.00$, 0.93 and 0.85. At steady state, both the overflow and underflow streams were sampled, and their flow rates were determined. The samples were vacuum-filtered, and the solids were dried overnight and then weighed. The oil content of the miscella was determined.

Analytical methods. The flow rates of the overflow and underflow streams were determined by taking samples over a 10–15-s period. Each sample was weighed and vacuum-filtered.

The oil content of the filtrate (miscella) was determined gravimetrically by evaporating the hexane in a vacuum rotary evaporator.

The solids recovered by filtration contained a significant volume of entrained liquid, consisting of solvent and dissolved oil. To determine the true content of undissolved oil in the solids, the sample was weighed (wet) and dried overnight in a vacuum oven. A portion (about 5 g) of the dried meal was defatted in a Soxhlet apparatus with hexane (~12 h) as the solvent to determine the total oil content. Based on the oil content of the filtrate (miscella), the true amount of undissolved oil in the solids was then calculated. All analytical measurements were performed in triplicate.

Simulation of a four-stage continuous countercurrent extraction system. The performance of a multistage countercurrent extraction process can be determined in a single-stage extraction apparatus by a series of crosscurrent extractions that reproduce the feed composition of each stage (12,13). The process is laborious, and the number of experiments required increases exponentially with the number of extraction stages simulated. In this work, we modelled a four-stage countercurrent extraction system by means of the scheme illustrated in Figure 2. In the diagram, each circle represents an extraction stage. The two input streams are thoroughly mixed, and then split into an underflow stream by the hydrocyclone. Each run, representing an approximation of a four-stage countercurrent extraction, is enclosed in dotted lines. In each stage, the feed suspension, S, consisted of 1 kg ground seed in 2 L hexane. The hexane feed, H, consisted of 8 L hexane. Although an infinite number of runs are required to give the exact reproduction of a true countercurrent system, Run 4 already represents a close approximation to the true countercurrent system.

Model development. The objective of the program was to predict the performance of multistage hydrocyclone-based extraction systems, based on the performance of our small single-stage apparatus. An empirical model was developed based on the equilibrium distribution of oil between the solid and liquid phases and the performance of the hydrocyclone. It predicts the oil recovery and residual oil content of the meal for a hydrocyclone/stirred-tank system based on the number of stages, the amount and composition of the feed suspension entering the unit at the first stage, the amount of feed hexane entering at the last stage and the size of the underflow opening of the hydrocyclones. A generalized hydrocyclone/stirred-tank system with the nomenclature of the streams and units is shown in Figure 3.

Empirical equations were first determined to relate the concentration of marc in the feed to that in the underflow, as illustrated in the hydrocyclone performance plots (Fig. 4). These plots were developed by mixing 1-kg samples of pre-treated meal A with hexane at various R values. Each slurry was separated at each of the three A_r settings, the flow rates of each stream were measured and the oil and solid concentrations in the underflow streams were

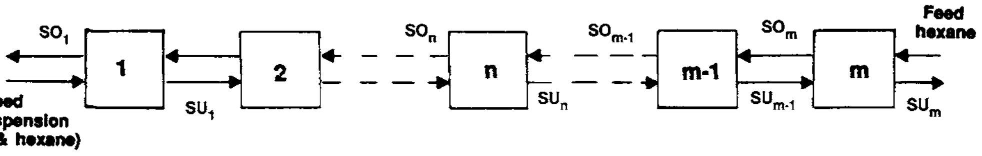

FIG. 2. Crosscurrent simulation of a four-stage continuous countercurrent extraction process.

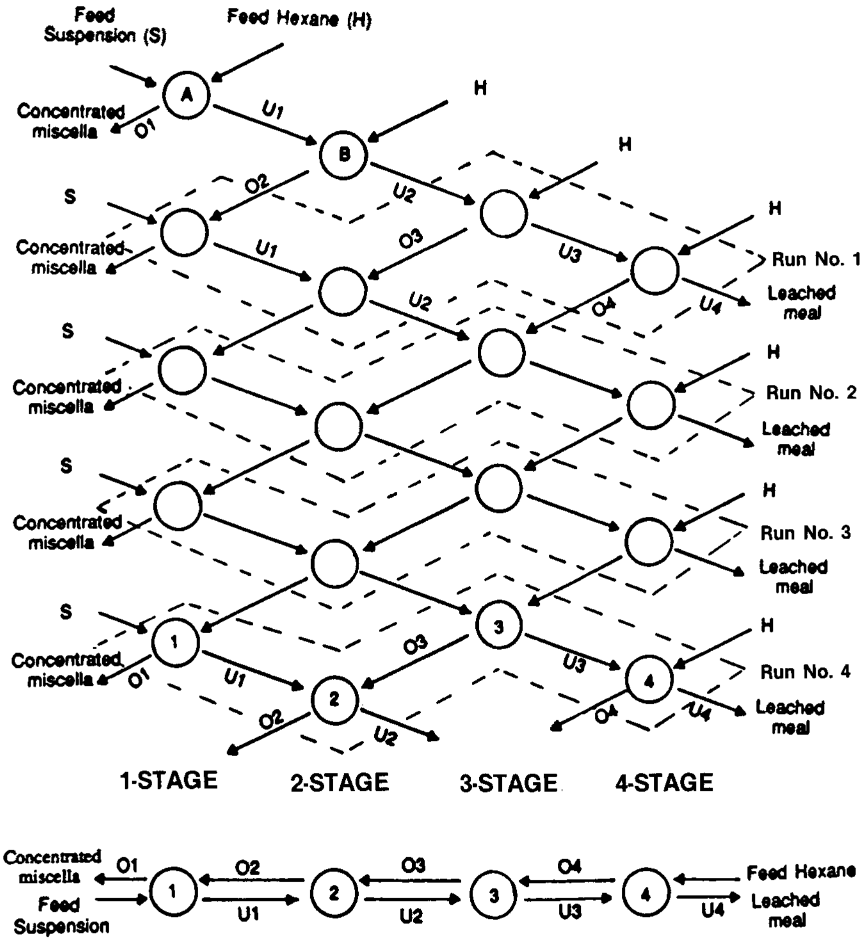

FIG. 3. A generalized multistage continuous extraction system for countercurrent extraction of oil from a finely ground canola meal.

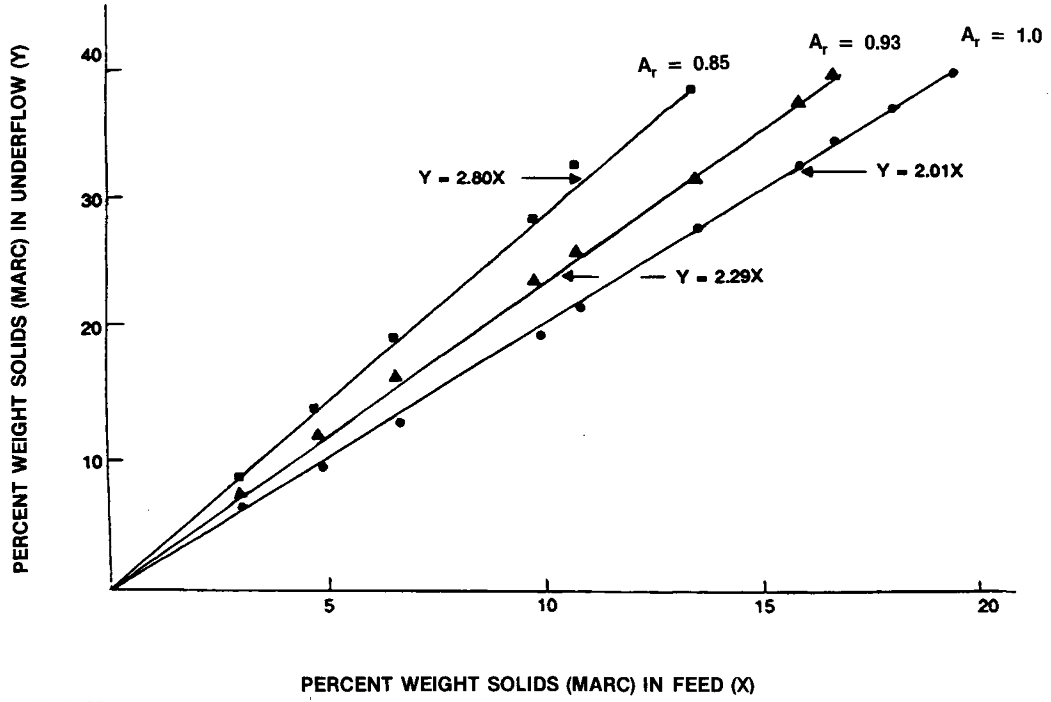

FIG. 4. Hydrocyclone performance plots.

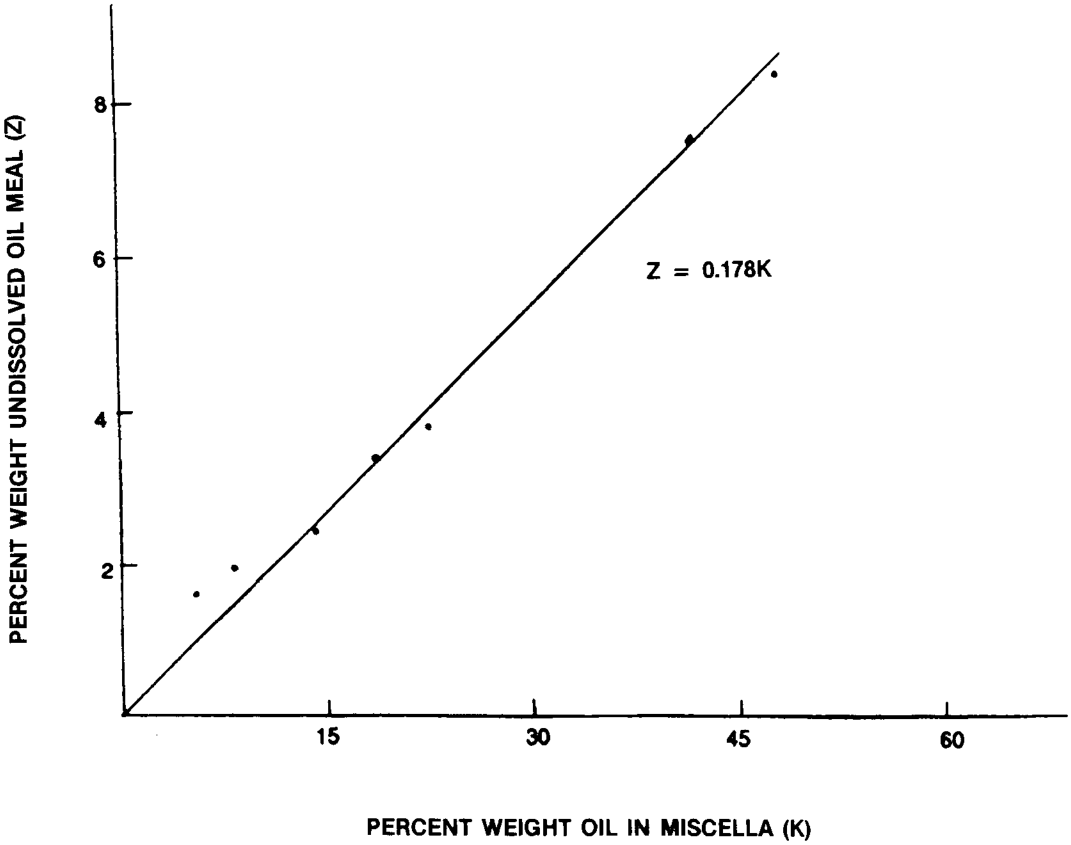

FIG. 5. Equilibrium diagram.

determined as described previously. The data were fitted to the hydrocyclone performance equations with the LOTUS 1-2-3 database program.

The equilibrium distribution of the oil between the liquid phase (*i.e.*, the miscella) and the solid phase (*i.e.*, the unextracted residual oil in the meal) was measured over a wide range of concentrations. The results are presented in the form of an equilibrium diagram (Fig. 5). The equilibrium equation

$$Z = 0.178\ K \qquad [1]$$

was determined by contacting 100 g of meal A with hexane at various R values in an Osterizer blender for 4 min. The slurry was then filtered, and the oil content of the two phases was determined as described above. The equilibrium data were fitted by linear regression with the LOTUS 1-2-3 database program.

In addition to the equilibrium and hydrocyclone performance equations, the model calculations included material balances and a steady state equation for the n^{th} stage:

$$\text{solution balance: } SO_{n+1}H + HSU_{n-1} = SO_n + SU_n \qquad [2]$$

$$\text{hexane balance: } HO_{n+1} + HU_{n-1} = HO_n + HU_n \qquad [3]$$

$$\text{steady state conditions: } OO_n/HO_n = OU_n/HU_n \qquad [4]$$

where SO is the weight of solution (miscella) in the overflow; SU is the weight of solution in the underflow, including the weight of undissolved oil in the meal; HO is the weight of hexane contained in SO; HU is the weight of hexane contained in SU; OO is the weight of oil in SO; and OU is the weight of dissolved oil in SU.

The calculation was carried out by initially estimating the amount of solution exiting the last stage (SU_m). The distribution of overflow and underflow was then calculated for each stage by using the hydrocyclone equation for the appropriate A_r value and Equation 4. At the end of the cycle, the assumed and calculated values of SU_m were compared, the input value was adjusted and the calculations were repeated. The iterations were terminated when the difference between the assumed and calculated values was <0.5%. At that point the composition of each stream was calculated from the equilibrium correlation (Equation 1).

Details of the algorithm and the computer program used for the computation are given by Adu-Peasah (11).

RESULTS AND DISCUSSION

Preliminary studies. The effect of methanol content on the fraction of the solids recovered in the overflow is summarized in Table 1. At low methanol concentrations (<25%), a significant amount (8.5%) of the feed solids was discharged in the overflow. With the meal containing between 30 and 45% methanol, less than 1% of the feed solids was lost to the overflow. This indicates that the presence of higher levels of methanol in the meal helps

TABLE 1

Effect of Methanol Content on the Recovery of Feed Solids in the Overflow

Methanol content (%)	Hexane-to-dry meal ratio, S (L/kg)	Recovery of solids in the overflow (%)
25 ± 0.2[a]	3.5	8.5 ± 1.5
30 ± 0.3	3.5	<1%
45 ± 0.2	3.5	<1%
50 ± 0.2	3.5	emulsion

[a] Mean value ± SD for three replicates.

in agglomerating the fine meal particles, causing them to be removed from the overflow.

As the methanol content was increased to >50%, the local methanol concentration exceeded its solubility in hexane, and an emulsion was formed, probably as a result of combined effects of the low interfacial tension between hexane and the methanol/meal phases (14) and the high shear stress in the hydrocyclone (9,10). Because the goal of the process is to produce a solids-free miscella in the overflow, the optimum methanol content of the meal is between 30 and 45 wt%.

The effect of pressure drop on solids recovery in the underflow is illustrated in Figure 6. Increasing the pressure drop from 1.38×10^5 Pa (20 psi) to 3.10×10^5 Pa (45 psi) increased the solids recovery from ~80 to 99%. This increase was expected because an increase in pressure drop corresponds to an increase in rotational flow in the hydrocyclone and the energy available for the separation (9,10). Further increase in the pressure drop beyond 3.1×10^5 Pa, however, did not result in any increase in the solids recovery due to the detrimental effects of increased turbulence. As a result, 3.10×10^5 Pa was selected as the optimum operating pressure drop.

Figure 7 shows the effect of the feed solids concentration on miscella recovery in the overflow at different A_r values. Increasing the solids concentration in the feed from 0% (using only hexane) to 20%, and keeping the underflow valve fully opened ($A_r = 1.00$), increased the solution recovery in the overflow from 47.5 to 63.2%. This increase in solution recovery stemmed from the increased amount of solids in the underflow where all of the feed solids were discharged. The increased solid content resulted in an increased viscosity in the underflow, which in turn increased the flow resistance, causing more solution to exit through the overflow.

On increasing the solids concentration beyond 20%, the hydrocyclone could no longer produce solids-free miscella

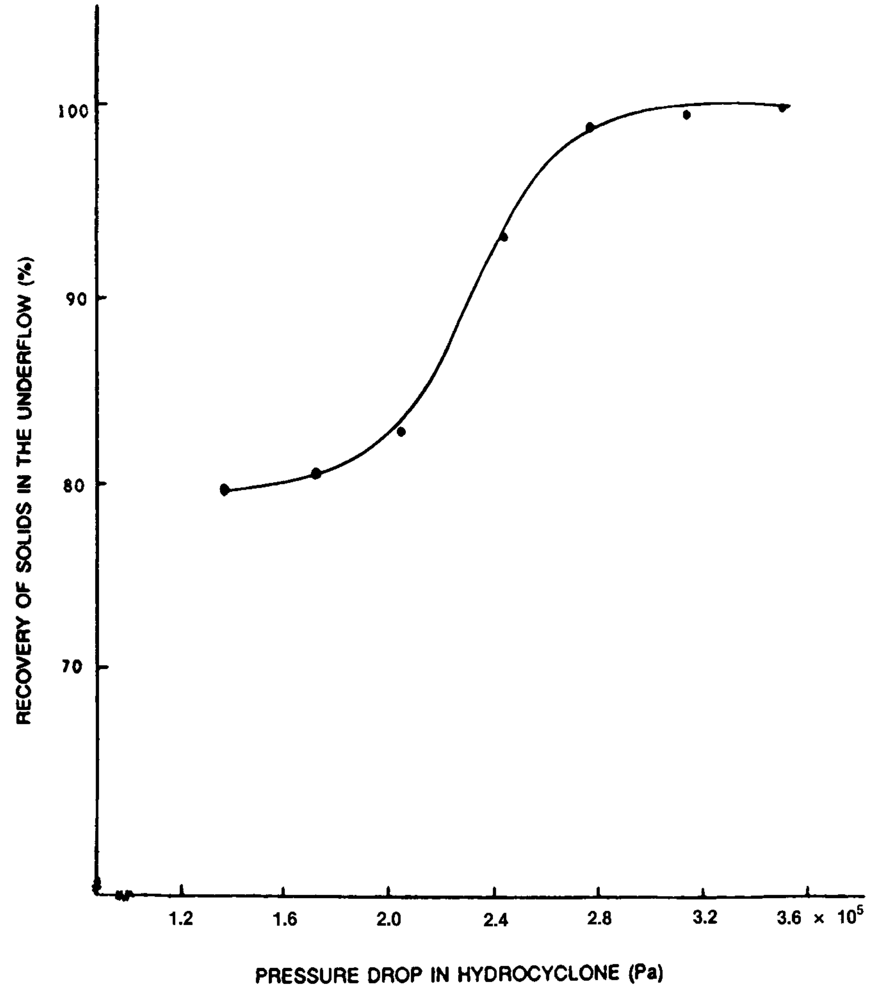

FIG. 6. Effect of pressure drop on feed solids recovered in the underflow.

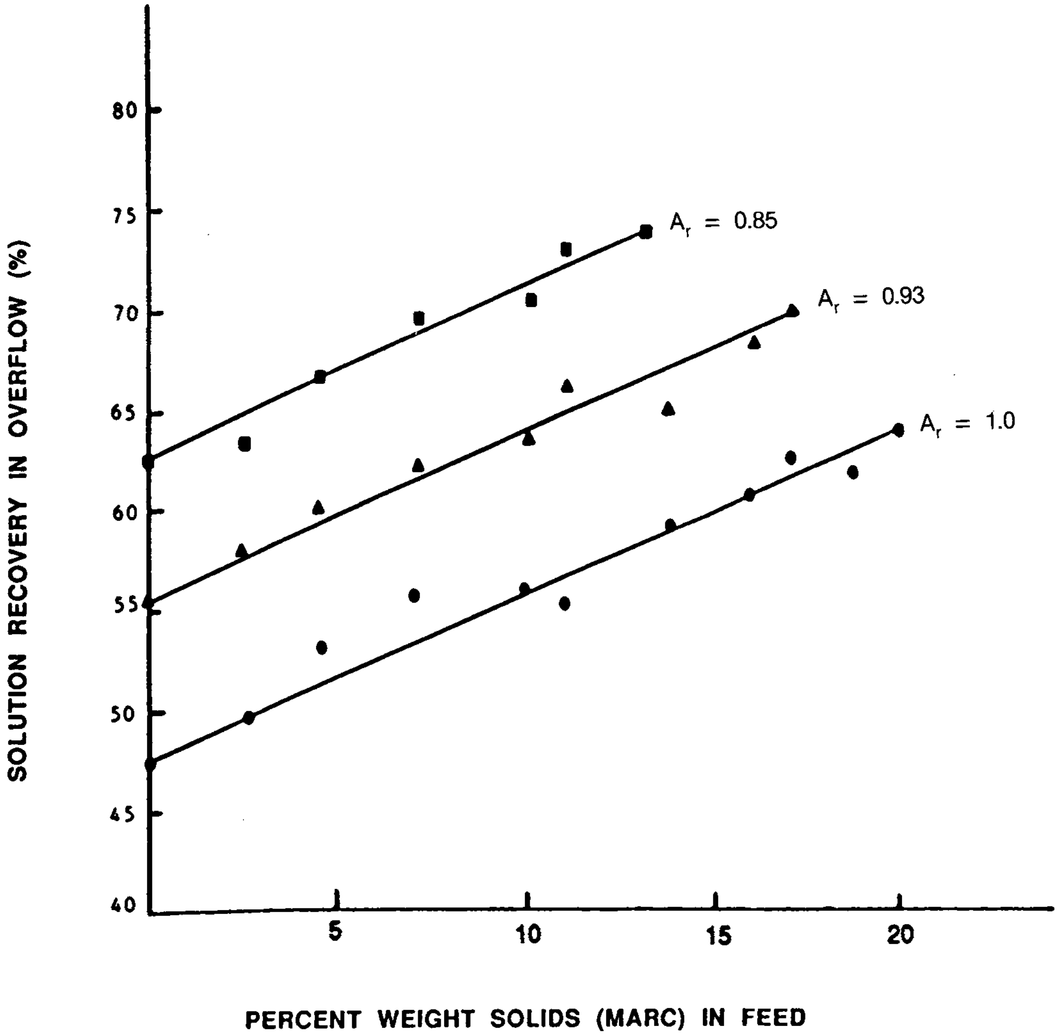

FIG. 7. Effect of solids concentration in the feed on solution recovery in the overflow.

in the overflow and 10% or more of solids were discharged in the overflow. The term "X_{crit}" was defined as the maximum concentration of solids in the feed that could be successfully separated by the hydrocyclone to produce a solids-free miscella in the overflow.

Throttling the underflow valve to $A_r = 0.93$ and increasing the solids concentration in the feed from 0 to 17.7%, the solution recovery in the overflow increased from 55.5 to 70.3%. At the reduced underflow opening, there was a substantial increase in the solution recovery, as expected. The decrease in the underflow orifice size increased the flow resistance in the underflow, causing more solution to be discharged in the overflow, and X_{crit} decreased to 17.7%. A further decrease in the underflow opening to $A_r = 0.85$ caused still more solution to be recovered in the overflow, while X_{crit} decreased to 13.1%.

Simulation of a four-stage continuous countercurrent extraction. The results for meals A and B are summarized in Tables 2 and 3, respectively. The extraction was carried out at R = 10, and the hydrocyclone was operated at $A_r = 1.00$. The oil content of the miscella remained fairly constant after the third set of extractions (Run 3),

TABLE 2

Experimental Results of Four-Stage Processing of Meal A

Run number	Oil content in miscella (%)	Residual oil in meal (%)	Overflow flowrate (kg/min)	Underflow flowrate (kg/min)	Oil recovery (%)
1	9.8 ± 0.4[a]	0.8 ± 0.3	0.046 ± 0.003	0.071 ± 0.011	82.9 ± 0.3
2	10.9 ± 0.3	1.1 ± 0.2	0.046 ± 0.006	0.068 ± 0.008	83.7 ± 0.3
3	12.3 ± 0.3	0.8 ± 0.1	0.042 ± 0.004	0.072 ± 0.010	83.8 ± 0.2
4	12.1 ± 0.2	1.1 ± 0.3	0.048 ± 0.009	0.071 ± 0.009	83.6 ± 0.4

[a] Mean value ± SD for three replicates.

TABLE 3

Experimental Results of Four-Stage Processing of Meal B

Run number	Oil content in miscella (%)	Residual oil in meal (%)	Overflow flowrate (kg/min)	Underflow flowrate (kg/min)	Oil recovery (%)
1	4.7 ± 0.3[a]	0.7 ± 0.2	0.048 ± 0.007	0.069 ± 0.011	86.2 ± 0.2
2	4.9 ± 0.2	0.9 ± 0.1	0.052 ± 0.010	0.071 ± 0.010	87.2 ± 0.3
3	5.2 ± 0.3	0.7 ± 0.1	0.048 ± 0.004	0.067 ± 0.009	87.5 ± 0.2
4	5.3 ± 0.2	0.8 ± 0.2	0.051 ± 0.007	0.073 ± 0.009	87.8 ± 0.2

[a] Mean value ± SD for three replicates.

confirming that the results obtained from Run 4 nearly reached steady state and the results were representative of the continuous countercurrent operation. In a four-stage system, 83.7% of the oil would be recovered from the meal. The remaining 16.3% of the oil was contained in the underflow from the last stage.

Because the solvent-free meal in the last stage underflow contained only ~1.0% undissolved oil, most of the unrecovered oil was lost as dissolved oil in the miscella. To increase the overall oil recovery, the meal must be recovered after the last stage by a solid/liquid separation step, such as filtration or centrifugation.

When meal B was processed (Table 3), the residual oil in the meal decreased, as expected. The oil recovery increased slightly, but the miscella concentration was low.

Model predictions. The influence of hexane-to-meal ratio (R) and the number of contact stages on countercurrent extraction of oil from meal A was calculated with the model. For each stage, the value of A_r was set at 1.00. The results are presented in Figure 8. The predictions for

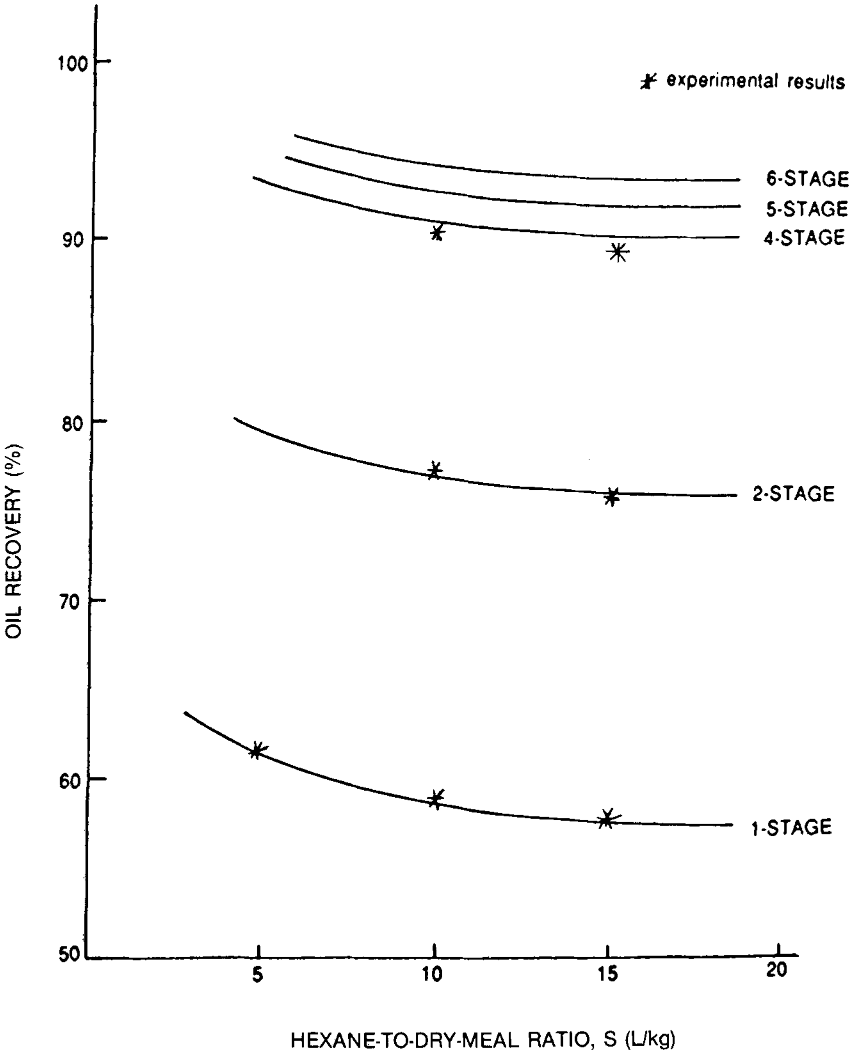

FIG. 8. Calculated and experimental values of oil recovery during extraction of meal A; A_r = 1.00. S, suspension.

TABLE 4

Predicted (maximum) Oil Recovery for Processing Meal A with Hydrocyclones Operating at Different Underflow Valve Positions

Number of extraction stages	Position of underflow valve (A_r)	S_{min} (L/kg)	Residual oil in meal (%)	Oil recovery (%)
1	1.00	2.5	7.9	57.9
2	1.00	3.4	3.1	74.7
4	1.00	4.2	1.2	87.6
5	1.00	4.6	1.1	90.5
6	1.00	5.3	0.9	92.7
1	0.93	2.9	7.0	63.6
2	0.93	4.3	2.2	80.5
4	0.93	4.8	1.1	93.2
5	0.93	5.6	1.0	94.6
6	0.93	5.9	0.8	95.6
1	0.85	3.4	6.3	70.8
2	0.85	4.2	1.9	88.4
4	0.85	5.0	1.0	96.3
5	0.85	5.5	0.9	97.2
6	0.85	6.2	0.7	98.3

TABLE 5

Predicted (maximum) Oil Recovery for Processing Meal B with Hydrocyclones Operating at Different Underflow Valve Positions

Number of extraction stages	Position of underflow valve (A_r)	S_{min} (L/kg)	Residual oil in meal (%)	Oil recovery (%)
1	1.00	2.8	5.2	63.6
2	1.00	3.7	2.1	80.8
4	1.00	4.5	0.9	93.7
5	1.00	5.1	0.8	94.9
6	1.00	5.5	0.7	96.4
1	0.93	3.1	4.9	69.3
2	0.93	4.4	1.5	87.5
4	0.93	5.0	0.8	97.6
5	0.93	5.8	0.7	98.1
6	0.93	6.1	0.7	98.9
1	0.85	3.6	4.2	73.2
2	0.85	4.6	1.3	90.2
4	0.85	5.4	0.7	98.0
5	0.85	5.7	0.6	99.2

one-, two- and four-stage systems were confirmed experimentally by the crosscurrent scheme described earlier. The agreement between the calculated and measured results was good (±1%).

If the value of R is decreased, the solids content of the underflow from the final stage increases. The value that results in the feed solids concentration just to exceed X_{crit} is defined as R_{min}. It represents the practical lower limit for solvent use with the system and produces the maximum oil recovery. The value of R_{min} increased from 2.9 to 5.9 as the number of stages was increased from 1 to 6. As expected, an increase in the number of extraction stages also increased the oil recovery.

The calculated values for R_{min}, residual oil in the meal and oil recovery are presented in Table 4 for full-fat ground seed (meal A) and in Table 5 for pre-extracted meal B. The calculations were not extended beyond six stages because in each case acceptable results were obtained with five or six stages.

Somewhat surprisingly, the amount of oil recovered initially decreased with increased solvent-to-seed ratio and gradually levelled off as R was increased further (Fig. 8). This decrease in oil recovery with increasing dilution results from the discharge of a relatively larger volume of dilute miscella in the underflow, which still contains a significant amount of dissolved oil. As the feed is diluted, the hydrocyclone recovers less of the oil in the overflow, as shown earlier in Figure 7.

The model calculations indicate that a six-stage hydrocyclone/stirred-tank unit would recover 98.3% of the oil contained in full-fat ground seed (meal A) at a solvent-to-seed ratio of 6.2 L/kg. The process would produce a miscella containing 15.8 wt% oil and a meal containing 0.7% oil. For pre-extracted meal (B) a five-stage unit operating at R = 5.7 L/kg would recover 99.2% of the oil, producing a miscella containing 6.4 wt% oil and a leached meal containing 0.6% oil. Although the solvent usage in these systems far exceeds that of conventional percolating-bed extractors, the simplicity and low cost of the hydrocyclone system provides an alternative to the capital-intensive conventional systems in some commercial applications.

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