A MULTISTAGE HYDROCYCLONE - STIRRED-TANK SYSTEM FOR COUNTERCURRENT EXTRACTION OF CANOLA OIL.

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INTRODUCTION

A novel approach for rapeseed processing has been developed in our laboratory (Rubin et al., 1986; Diosady et al., 1985 and 1987). The process uses methanol containing 10% NH_3 and 5% H_2O ($CH_3OH/NH_3/H_2O$) to suspend the seed which is then ground as a slurry in a Szego mill, a unique orbital mill (Trass, 1980). The slurry is passed through the mill a number of times so as to achieve sufficient size reduction of the seed particles. To prevent excessive grinding of the seed particles and also to reduce the amount of recirculating slurry, the slurry is passed through a hydrocyclone prior to the regrinding in the Szego mill (Adu-Peasah, 1990). After the grinding, hexane is added to the slurry to form a second phase in which most of the oil dissolves. The meal is separated by vacuum filtration, washed with methanol, and extracted once again with hexane. In this process almost all the glucosinolates and up to 88% of the polyphenols were removed. The meal contained about 50% of crude protein, and the oil extracted from the meal contained 50 ppm or less of phosphorus.

Large-scale extraction of oil from the finely ground canola meal obtained from the process using a conventional percolating-bed extractor is however impractical, as these fine particles could easily plug the bed, reducing its permeability to solvent. The purpose of this work, therefore, was to design a multistage extraction system, consisting of stirred tanks and hydrocyclones for countercurrent extraction of oil from finely ground canola meal, using hexane as the solvent. An empirical model was developed and tested for the prediction of the performance of such a multistage system during extraction at equilibrium, and the process was then optimized for the maximum oil recovery.

The hydrocyclone was selected for the miscella/meal separation because in the food and other industries, hydrocyclones have been used effectively to separate fine particles from liquids. Hydrocyclones are very compact, inexpensive, and easy to operate continuously. They generally have very short residence time and require very little or no maintenance (Svarovsky, 1984).

MATERIALS AND METHODS

Prior to the design of the multistage extraction unit, the effect of solids (marc) and size of underflow opening of the hydrocyclone on recovery of solution (miscella) in the overflow were studied (Fig. 1). A Bauer model 500 hydrocyclone (CE Bauer Co., Brantford, ON, Canada) was used for the miscella/meal mixture separation. A 1.0 hp moyno pump was used to pump the mixture through the hydrocyclone at 45 psi (3.10×10^5 Pa).

Using a ball valve, it was possible to regulate the size of the underflow opening of the hydrocyclone by throttling. The hydrocyclone was operated at three different overflow-to-underflow ratios (A_r); (A_r) = 1.00; A_r = 0.93; A_r = 0.85. The A_r's were determined by measuring the volumetric split ratio of the overflow-to-underflow streams, using water at a pressure drop of 45 psi. The A_r's were then calculated using an empirical equation (Svarovsky, 1984).

$CH_3OH/NH_3/H_2O$ solution (Rubin et al., 1986) was used as a solvent for grinding the canola (Westar) seed throughout the study. The seed was ground using a 2-pass grinding through a Szego mill-hydrocyclone unit (Adu-Peasah et al., 1989). The ground seed was washed twice with methanol at a solvent-to-seed ratio of 2:1 (v/w) to produce meal "A", which contained 46.9% (w/w) oil. A second meal, "B", which contained 13.7% (w/w) oil was prepared by contacting the ground seed with hexane at a solvent-to-seed ratio of 3.5:1 (v/w). The partially exhausted meal was then washed twice with methanol at a solvent-to-seed ratio of 2:1 (v/w). Both meals "A" and "B" contained between 30% and 45% (w/w) methanol. This methanol concentration was found to be the optimum level to produce solids-free overflows.

Batch Simulation of a 4- Stage Continuous Countercurrent Extraction

The scheme followed during the simulation (Fig. 2) was similar to extraction techniques described elsewhere (Scheibel, 1954; Treybal, 1980). Exact details of how the simulation was carried out is described by Adu-Peasah (1990).

Development of a Model for the Prediction of the Performance of a Multistage Extraction Unit

The goal of the model is to be able to predict the performance (i.e., oil recovery) of any generalized multistage hydrocyclone-stirred-tank unit (Fig.3) operating under equilibrium conditions, knowing the composition of the feed suspension entering the unit at the first stage, the amount of feed hexane entering the last stage, the size of the underflow opening, and the number of extraction stages.

In order to accomplish this, empirical equations were determined relating i) the concentration of marc in the feed to the concentration of solids in the underflow, i.e., the "Hydrocyclone Performance Equations" (Fig. 4), and ii) the concentration of oil in the miscella to the concentration of undissolved oil remaining in the meal at equilibrium, i.e., the "Equilibrium Equation" (Fig. 5).

In addition to the "Equilibrium" and "Hydrocyclone Performance" equations, the following material balance equations 1 and 2, and a "steady state" equation 3 were also required for the calculation.

Material balance equations for n^{th} Stage:

Solution Balance

$$SO_{n+1} + SU_{n-1} = SO_n + SU_n \qquad (1)$$

Hexane Balance

$$HO_{n+1} + HU_{n-1} = HO_n + HU_n \qquad (2)$$

Steady state condition:

$$OO_n/HO_n = OU_n/HU_n \qquad (3)$$

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

Details of the algorithm and the computer program used for the computation are given by Adu-Peasah (1990).

RESULTS AND DISCUSSION

Miscella-Meal Separation

In Figure 6, the effect of the concentration of solids in the feed suspension and the size of the underflow valve opening (characterized by the A_r) on the recovery of solution (miscella) in the overflow is illustrated.

On increasing the solids concentration in the feed from 0.0% (i.e., using only hexane) to 20%, and keeping the underflow valve fully opened (i.e. at $A_r = 1.0$), the recovery of the feed solution in the overflow increased from 47.5% to 63.2%. The reason for this increase in the solution recovery is probably due to the increased amount of solids in the underflow where all of the feed solids are discharged. Increasing the solids in the underflow, increased the viscosity also and this, in turn, increased the flow resistance in the underflow, causing more solution to exit through the overflow. On increasing the solids concentration beyond 20%, the hydrocyclone could no longer produce a solids-free overflow miscella. As a result, the hydrocyclone was not, and should not to be operated with feeds containing more than 20% solids (i.e. X_{crit}, whereby X_{crit} represents the maximum or critical wt. % of solids that can be in the feed and still obtain solids-free miscella in the overflow), when the throttling valve is fully opened ($A_r = 1.0$).

Throttling the underflow orifice to the $A_r = 0.93$ position, and increasing the solids concentration in the feed from 0.0% to 17.7%, increased the recovery of the feed solution in the overflow from 55.5% to 70.3%. Compared to the situation where the hydrocyclone was operated with its underflow orifice fully opened ($A_r = 1.0$), there is a substantial increase in the solution recovery. This increase in the solution recovery at a reduced underflow opening was expected, as a decrease in the size of the underflow orifice also increased the flow resistance in the underflow, causing more solution to be discharged in the overflow. Because of the reduced size of the underflow orifice, X_{crit} decreased to 17.7%.

As expected, a further decrease in the underflow opening to position $A_r = 0.85$, caused more solution to be recovered in the overflow, and the X_{crit} also decreased to 13.1%.

Based on these observations, it can be said that the hydrocyclone recovers more miscella when operated with concentrated feed suspensions at a reduced underflow opening. However, there is a limit to how small the orifice size and how concentrated the feed suspension can be. The use of very concentrated slurries at very small underflow size opening readily leads to either contamination of the overflow with solids or complete plugging of the underflow orifice. It is therefore essential to know during the operation of the hydrocyclone, the X_{crit} value.

Batch Simulation of a Four-Stage Continuous Countercurrent Extraction Process

The results of four-stage countercurrent extraction of oil from the ground meal "A" is summarized in (Table 1). The extraction was carried out at hexane-to-dry-meal ratio of 10 (L/kg), and the hydrocyclone was operated with its underflow orifice fully opened ($A_r = 1.0$).

As can be seen in Table 1, the oil content of the miscella remained fairly constant after the third set of simulation. As a result of this, it will be appropriate to assume that the results obtained from the third and the fourth sets of simulation approached very closely those of a steady state continuous operating unit (Scheibel, 1954; Treybal, 1980). Using these steady results, it can be said that about 83.7% of the oil was recovered from the meal. The remaining 16.3% of the oil, which is not recovered, is contained in the miscella/meal mixture, exiting from the last stage underflow.

The fact that the partially exhausted meal obtained from the last stage underflow contained about 1.0% undissolved oil suggests therefore that most of the unrecovered oil was contained in the miscella. In order to improve the overall oil recovery, the amount of solution exiting from the last stage underflow must be decreased.

Model-Based Calculated Result

The influence of hexane-to-meal ratio (S) and the number of contact stages on countercurrent extraction of oil from meal "A", as predicted by the model is illustrated in Figure 7. All the hydrocyclones were assumed to be operating at A_r = 0.93, and points illustrated on the Figure represent the experimental results.

Depending on the number of extraction stages in the unit, the curve started from either S = 2.9, 4.3, 4.8, 5.6 or 5.9. These starting values (which are also referred to as S_{min}) represent the minimum hexane-to-meal ratio required during the process, so that the multistage unit can successfully produce solids-free miscella overflow products. Should the unit be operated below S_{min}, the critical allowable solids concentration in the feed (X_{crit}) will be exceeded at the first stage, and the unit can no longer produce a solids-free miscella overflow.

S_{min} increased from 2.9 to 5.9, as the number of contact stages was increased from 1 to 6. Since each stage concentrates the underflow solids by removing more hexane from the feed entering from the previous stage, more hexane is needed downstream in the multistage systems so as to prevent the increase of solid concentration to above S_{min} in the first stage. Hence the increase in S_{min} with increasing number of contact stages.

Due to the increase in the number of contacts between the meal and the solution, an increase in the number of extraction stages in the unit also increased the oil recovery.

The amount of oil recovered from a given unit, however, decreased with increasing S in the region of lower S, then gradually leveled off. This was a bit of a surprise as more oil is expected to be extracted from the meal when more hexane is used. The cause of this decrease in oil recovery with increasing S (i.e., with dilution), results from the discharge of a relatively large volume of miscella in the underflow, since the hydrocyclone recovers less solution in the overflow as the feed material is further diluted (Fig. 6).

Calculated results for the maximum oil recoverable during processing of the ground meals "A" and "B" with different extraction units are summarized in Table 2 (meal "A") and Table 3 (meal "B"). Based on the results it can be said that i) the maximum oil recovered during the processing of meal "A" is 98.3%. This occurs at S_{min} = 6.2 (L/kg), using a 6-stage unit consisting of hydrocyclones operating at A_r = 0.85. The process produces a miscella containing 15.8% (w/w) oil, and a leached meal containing 0.7% oil by weight; ii) the best extraction unit for processing of meal "B" is a 5-stage unit operating at 5 min = (L/kg). The unit recovers 99.2% oil from the meal, producing a miscella containing 6.4% (w/w) oil and a leached meal containing 0.6% oil.

CONCLUSIONS

The performance of the hydrocyclone during the separation of the miscella-meal mixtures was influenced by the following factors: i) size of the underflow opening, ii) concentration of solids in the feed suspension, iii) pressure drop in the hydrocyclone, and iv) methanol content of the meal. The smaller the size of the underflow opening and the higher the concentration of solids in the feed, the better is the miscella recovery. The optimum pressure drop for operating the hydrocyclone was established as 45 psi (3.10 X 10^5 Pa). To prevent the formation of an emulsion or contamination of the overflow product with meal particles, the meal should contain between 30% and 45% (w/w) methanol.

The results obtained from the model calculations agreed very well with those obtained experimentally. Based on the model calculations, it is predicted that the optimum multistage system for extracting oil countercurrently from canola treated with $CH_3OH/NH_3/H_20$ containing about 46.9% (w/w) of oil, meal "A", was a 6-stage unit. With the unit operating at a hexane-to-dry-meal ratio of 6.2 (L/kg), about 98.3% of the oil in the meal is expected to be recovered, resulting in a miscella containing 15.8% (w/w) oil. Processing of pre-extracted meal "B" containing 13.7% (w/w) oil required a 5-stage extraction unit, operating at a hexane-to-dry meal ratio of 5.7 (L/kg). The unit recovers 99.2% oil from the meal and produces a miscella containing 6.4% (w/w) oil..

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Table 1. Experimental results of four-stage processing of ground canola seed.

Run Number	Oil content of miscella (%)	Residual oil in meal (%)	Overflow flowrate (Kg/min)	Underflow flowrate (Kg/min)	Oil Recovery (%)
1	9.8±0.4	0.8±0.3	0.046	0.071	82.9±0.3
2	10.9±0.3	1.1±0.2	0.046	0.068	83.7±0.3
3	12.3±0.3	0.8±0.1	0.042	0.072	83.8±0.2
4	12.1±0.2	1.1±0.3	0.048	0.071	83.6±0.4

The results are mean value of five replicates.

Maximum oil recovered during processing of meal "A" at different underflow valve positions.

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

Maximum oil recovered during processing of meal "B" at different underflow valve positions.

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

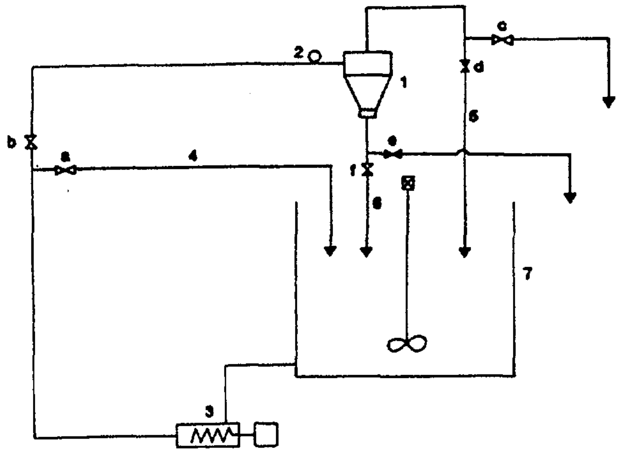

Figure 1. Stirred tank-hycrocyclone unit used for the miscella-meal separation.

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.

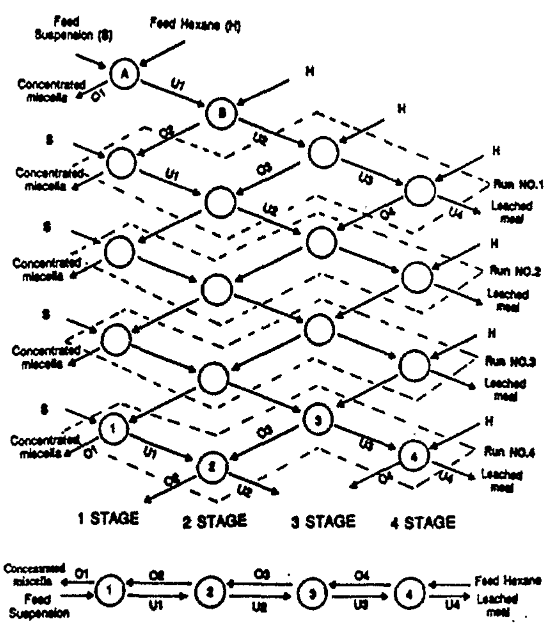

Figure 2. Batch simulation of a 4-stage continuous countercurrent extraction process.

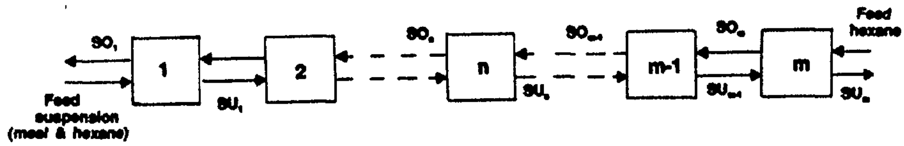

Figure 3. A generalized multi-stage continuous countercurrent extraction system for extraction of oil from finely ground canola.

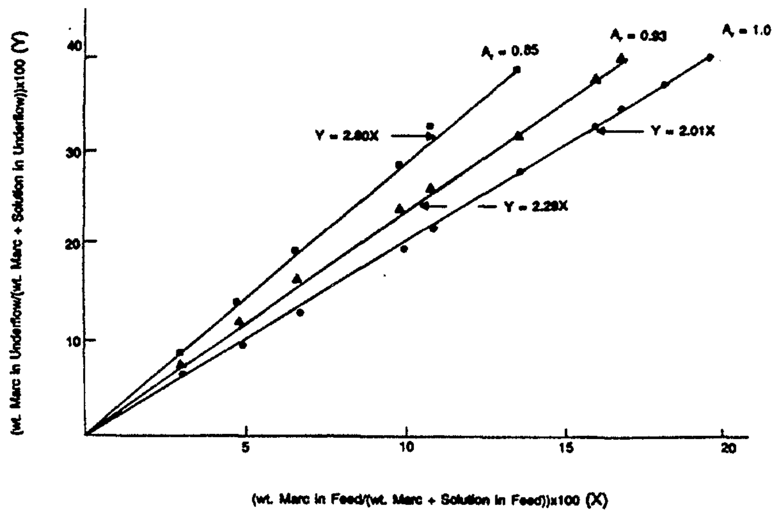

Figure 4. Hydrocyclone Performance Equations.

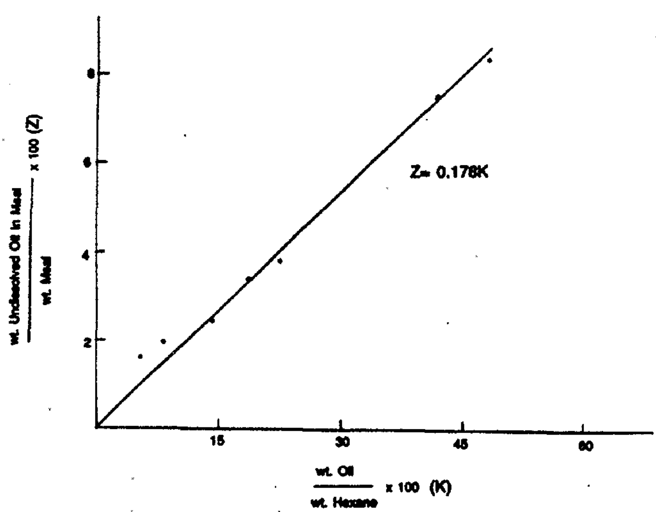

Figure 5. Equilibrium Equation.

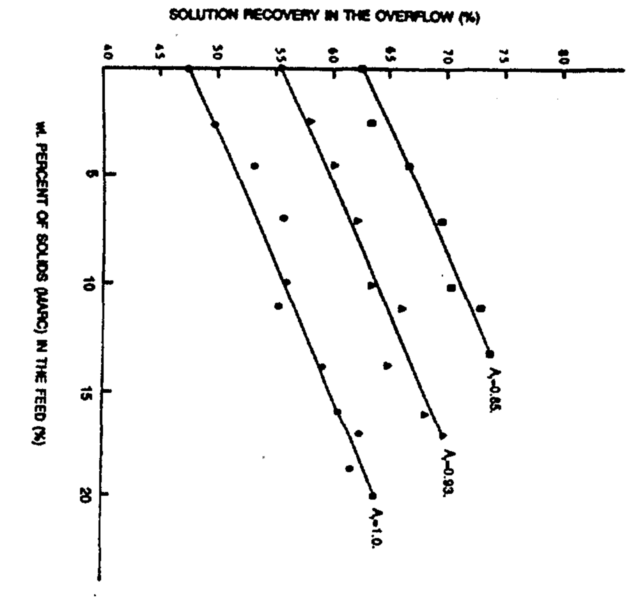

Figure 6. Effect of solids concentration in the feed on solution recovery in the overflow.

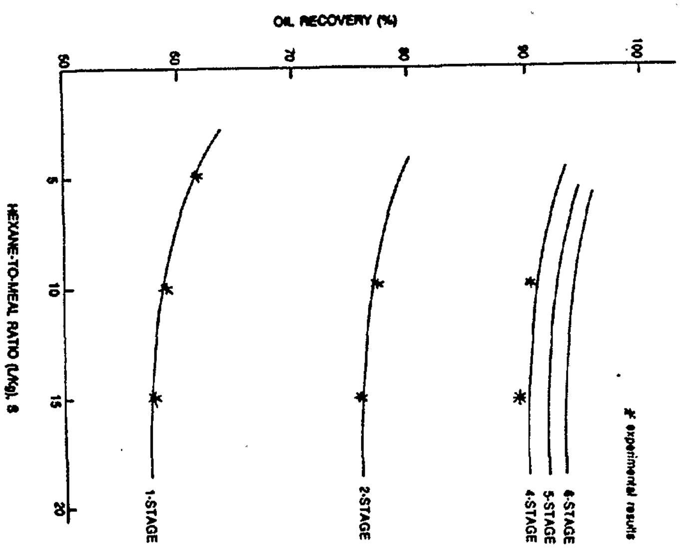

Figure 7. Calculated and experimental values of oil recovery for processing of meal "A", using hydrocyclones operating Ar = 1.0.