

## SOLVENT GRINDING AND EXTRACTION OF RAPESEED

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### ABSTRACT

Slurry grinding of rapeseed promises many advantages: the cracking, cooking, pre-press and flaking operations may be eliminated, and a well controlled meal drying and desolventizing step may replace the harsh treatment of the desolventizer toaster. The Szego mill, a unique orbital mill developed in our Department, was used for the solvent grinding of canola. Laboratory results indicate that a counter-current system consisting of two mills, with residence times of eight seconds each, and three solvent mixing stages will recover approximately 99% of the oil content of the seed.

### BACKGROUND

Rapeseed crushers use the "classical" pre-press solvent extraction system adopted from the soy crushing industry. Since rapeseed forms very weak flakes, the extractors operate at much lower rates with rapeseed than with soybeans, primarily due to reduced penetration rates. The extractors cannot handle dehulled rapeseed or ground seed.

The simultaneous grinding and extraction of canola seeds is a very attractive approach for improving the conventional extraction process. If the extraction is carried out rapidly, at low temperatures and moisture levels, the quality of the resulting oil and meal may be improved, while significantly reducing the complexity of the conventional process. Presently the seed is cracked and then cooked to inactivate myrosinase, and to break down the cellular structure of the seed, thus resulting in easier oil extraction. If the seed is slurry ground and extracted by the solvent medium in the mill, the myrosinase activity will be

minimized by the low moisture level, and the extraction will be very rapid due to the large solvent-contact surface produced by the grinding. This approach can replace the cracking, cooking and extraction steps with a single operation.

Previous work by our group (1) demonstrated that simultaneous grinding and extraction using the Szego mill in a batch mode can achieve extraction equilibrium in two to three minutes. The mill extracted more than 80% of the oil available under equilibrium conditions within 30 seconds of operation, however, industrial application of this approach will require the development of a multistage counter-current process utilizing an industrially available, edible grade solvent.

The present study was initiated with the aim of further exploring the feasibility of the simultaneous grinding and extraction technique, using the Szego mill. The work consists of: the optimization of the continuous grinding of canola seed; the investigation of the effect of solvent density on the operation of the mill; and modelling of a counter-current extraction system by a cascade of co-current extractions.

#### EXPERIMENTAL TECHNIQUES

##### Szego Mill

The Szego mill is an orbital mill developed by one of us (O. Trass) in cooperation with the late L.L. Szego. The mill consists of a stationary cylindrical grinding surface (stator), and a rotor assembly, which contains typically three grooved rollers supported on individual flexible shafts. The rollers are free to rotate around their flexible shafts, and therefore they are in rolling contact with the stator. The crushing force is generated by the radial acceleration of the rollers.

The grooves on the rollers facilitate the movement of material down the mill by a screw conveying effect in the normal operation of the mill. The mill is compact, and has a remarkably high throughput in its normal configuration, at a low power consumption (2,3). It has been tested in the dry grinding of wheat, sand, and limestone (3), and in the slurry grinding of coal (4,5). In the first stage of this project the mill was effective in grinding rapeseed in Freon 113 (1) in the batch mode. In the present study the operation was modified to permit continuous grinding for short periods of time.

Contact time, solvent-to-seed ratio, rotational speed and hold-up were varied. Tests were performed primarily with Freon 113, although several runs were done using aqueous methanol solutions. Work with hexane was not possible since the laboratory was not equipped with explosion proof wiring and adequate ventilation.

##### Simulation of Counter-current Extraction

Most commercial extractors operate in the counter-current mode. This is difficult to reproduce in the laboratory, due to the

high throughput and great capital requirements, however, the process may be modelled by a cascade of cross current extractions that require only a single mill. The process is presented schematically in Figure 1. Stages a through h may be carried out consecutively in the same equipment.

Stages a, b and c constitute the cross-current extractions, as the feed is repeatedly extracted by fresh solvent. In stage c of both the counter- and cross-current extractions the most extracted meal is in contact with fresh solvent. Similarly in stage f the most concentrated miscella contacts fresh seed. Therefore the sequence of c g e h f represents reasonably well a five stage counter-current extraction process.

#### Analytical Techniques

Slurry samples were vacuum filtered on a Buchner funnel. The filtrate was collected, and the solvent removed by a rotary vacuum evaporator. The weight of oil and cake were measured, and corrected for the weight of miscella entrained in the filter cake. Residual oil in the meal was measured by the AOCs standard method Da28-39 (4). Particle size distributions were obtained on a Rotap shaker using standard Tyler sieves down to 45  $\mu\text{m}$ , in the presence of the solvent.

#### RESULTS AND DISCUSSION

##### Continuous Solvent Grinding

Previous work with batch extractions in the Szego mill demonstrated that a solvent-to-seed ratio of 1.67 produces the best results. The optional grinding conditions were found to be 50% hold-up, at a rotor speed of 680 r.p.m. (1). These conditions were used in the initial stage of optimizing the continuous operation of the Szego mill. During continuous operation the seed spent approximately eight seconds in the mill. The effective grinding time in contact with the grinding surfaces was approximately two seconds, which is a factor of fifteen shorter than the residence time during the batch extraction reported earlier (1).

The process was optimized in the continuous mode. Increased mill hold up resulted in significant reduction in both extraction and grinding efficiency. Since the density of the solvent is 1.5 times higher than that of the seed, at high hold-up and limited turbulence the seeds tend to move away from the grinding surfaces, and pass through the mill without grinding, resulting in the presence of whole seeds in the effluent.

The effect of solvent-to-seed ratio (R) on particle size distribution is presented in Figure 2. Although, the density difference tends to force the seed away from the grinding zone, this effect is overcome by viscous drag and the turbulent flow in the mill. As the extraction proceeds, oil with a density of 0.9 enters the solvent and lowers its average density, while the residual meal density increases, thus reducing the tendency of the

meal to leave the grinding zone. Increased solvent-to-seed ratios result in higher miscella densities, thus less effective grinding.

The seed through-put increased with decreasing solvent-to-seed ratio, (R) and the combination of throughput and grinding efficiency defined the resulting particle sizes. The mean particle size increased from 110  $\mu\text{m}$  to 205  $\mu\text{m}$  as R decreased from 3.33 to 2.10, but dropped back to 105 at R=1.67 and did not change significantly when R was further decreased to 1.25. The slope of the particle size distribution in Figure 2 gives a good indication of the size uniformity in the ground meal. Higher slope indicates a more uniform meal. The operation was optimized at R=1.67, since at this solvent-to-seed ratio the mean particle size is minimized at 100  $\mu\text{m}$ , while the product has an acceptable particle size distribution. While similar product characteristics can be also attained at R=3.33, at R=1.67 the solid throughput of the mill is doubled, and the solvent use is halved.

Several runs were performed using multiple passes through the same mill. The results demonstrated that multiple passes decrease the mean particle size of the products while maintaining very similar particle size distributions. The results indicate that increasing the contact time by increasing the length of rollers will result in decreased mean particle size without changes in the uniformity of the particle size distribution.

#### Effect of Solvent Density

Most of the preliminary work was carried out with Freon 113, since it has similar polarity and volatility to hexane. However, due to the great density differences between these solvents their behaviour in the Szego mill is not expected to be identical. Since our mill was not equipped for explosion proof operation, the effect of solvent density on particle size distribution was investigated with aqueous methanol solutions. The results, presented in Figure 3 demonstrate that increased densities in the range of 0.8 (pure methanol) to 1.0 (water) result in decreasing mean particle sizes.

The results for Freon 113 were similar to that of methanol. However, the grinding mechanism in Freon 113 is significantly different from the methanol system, since specific gravity differences tend to remove the seed from the grinding zone in Freon 113, while these forces tend to move the seed into the grinding zone in methanol. It would be interesting, therefore to investigate the whole density gradient between 0.7 and 1.6 to determine if the observed trends are symmetrical on either side of the meal density which is 1.02-1.15  $\text{g cm}^{-3}$ .

#### Cross-Current Extraction

The cross-current extraction scheme illustrated in Figure 1 was followed to determine the probable behaviour of the extraction system during counter-current operation. Initial experiments demonstrated that two Szego mills are sufficient for obtaining the

optimal particle size distribution in the meal, and in further contact stages simple stirred tanks were used for solid liquid contact. The results indicate that the five stage counter-current extraction simulated by this cross current schematic is capable of extracting 99% of the oil present in the seed. The oil extraction efficiencies obtained in the five stages were:

Stage	0	1	2	3	4	5
Oil content in meal	100%	72.9%	43.8%	25.2%	8.6%	1.3%

The oil contents were expressed as percentage of the initial, 43% oil content of the seed. The mill was operated in the continuous mode at R=1.67, 20% holdup.

#### Conclusions

The work to date demonstrated that the Szego mill is effective in simultaneously grinding and extracting canola seeds in Freon 113. Continuous runs using a solvent-to-seed ratio of 1.67 resulted in a meal with mean particle size of 360  $\mu\text{m}$ . Approximately 40% of the oil in the seed is extracted within 8 seconds total contact time, equivalent to less than 2 seconds in the grinding zone.

A crossflow model, equivalent to a five-stage counter-current extraction was investigated. The results indicate that a five stage counter-current extraction, consisting of two grinding and three solvent contact stages will remove 99% of the oil content of the seed. This work will have to be repeated with a conventional solvent, such as hexane, in an explosion-proof mill.

While much work on optimization remains, the results to date indicate that the Szego mill can form the basis of an effective, small and probably inexpensive extraction system for canola seeds.

#### REFERENCES

1. Diosady, L.L., Rubin L.J., Ting N., Trass O. 'Rapid Extraction of Canola Oil', American Oil Chemists' Society Annual Meeting, May 1982 Toronto.
2. Sing, V.P., 'Boiler Ignition with Coal/#2 Oil Mixtures', Rept. No. 81-91-K, Research Division, Ontario Hydro, Toronto, Ontario (1981).
3. Hohmann, R.H., 'Grinding of Wheat and Limestone with the Szego Mill', M.A.Sc. Thesis, Department of Chemical Engineering and Applied Chemistry, University of Toronto (1977).
4. Trass, O. and Papachristodoulou, G., 'Grinding of Coal-Oil Slurries with the Szego Mill', Proc. Second Int. Symp. on Coal-Oil Mixtures Combustion, Danvers, MA, (1979).
5. Trass, O., Koka, V.R., Papachristodoulou, G. and Vasquez, ER 'Grinding of Coal-Oil Slurries with the Szego Mill-Effect of Solids Concentration Proc. Fourth Intl. Symp. on Coal Slurry Combustion, Oroudo. Fl. (1982).
6. American Oil Chemists' Society, Official and Tentative Methods of Analysis.

