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Rapid Extraction of Canola Oil

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ABSTRACT

The simultaneous size reduction and solvent extraction of canola seeds were studied using a laboratory blender and a small, pilot-scale Szego mill. The laboratory tests established that over 95% of the oil may be removed from the seed in a single contact stage. The effects of contact time and solvent-to-seed ratio were investigated. The extraction equilibrium favored the extraction of the oil at higher solvent-to-seed ratios. In all cases the extraction reached some 90% of the equilibrium value after 3 min. Runs in the Szego mill, which is a unique orbital mill developed by one of us (O. Trass), confirmed that solvent grinding is an efficient extraction technique. In this equipment, contact times as short as 30 sec give significant extraction, with the system approaching equilibrium in one minute. The Szego mill appears to be suitable for the rapid extraction of edible oil seeds such as rapeseed.

BACKGROUND AND OBJECTIVES

The current rapeseed extraction technology is based on percolating bed extractors. The oil content of the seed is removed, usually after prepressing, by contact with hexane, which percolates through large voids between "flaked" seed particles. This technology was developed for soy extraction, but unfortunately neither rapeseed nor the new canola varieties form strong flakes. Accordingly, their extraction requires significantly larger equipment than an equivalent soy-crushing plant.

The rate of solvent extraction is controlled by the solid particle size and the solvent-to-seed ratio. The reduction of particle size increases the surface area, and decreases solvent penetration pathlengths, thus resulting in significantly increased oil transfer rates into the miscella. Similarly, increasing the solvent-to-seed ratio provides a larger concentration driving force for extraction. Unfortunately, both of these approaches are energy-intensive: grinding energy must be provided for size reduction, while the increase in solvent-to-seed ratio results in larger heat requirement during solvent recovery.

Solvent grinding is a technique that may take advantage of the high mass transfer rates characteristic of small particle sizes without prohibitive energy expenditure. Complete extraction may be achieved in shorter time resulting in a more compact and, possibly, significantly less expensive equipment. Grinding also eliminates the need for breaking down the cellular structure of the seed by cooking. Thus it may be possible to balance some or most of the grinding energy requirements against the energy required in the cooking. Thus it may be possible to balance some or most of the grinding energy requirements against the energy required in the cooking step. The technique could be applied to some of the recently developed techniques of seed preparation, resulting in improved oil and protein products from rapeseed, such as the FR1-75 process developed by Jones and Holme (1).

The novel slurry mill (the Szego mill) developed by Trass and his group at the University of Toronto (2) seems especially well suited to the solvent grinding of rapeseed.

EXPERIMENTAL TECHNIQUES

Laboratory Blender

A 2-L Waring blender was used to determine equilibrium extraction data and the best solvent-grinding conditions. Canola seeds were first ground with a Philips' coffee grinder, then an aliquot was placed in the blender. Freon 113 (200 mL) was added and the mixture blended at low speed for up to 2 min. In runs requiring longer blending times, the vessel was cooled under tap water for one minute after each minute of blending beyond the initial two minutes, in order to remove the heat generated by the blending process.

The resulting slurry was 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 (3). Particle size distributions were obtained on a Rotap shaker using standard Tyler sieves.

Blending time and solvent-to-seed ratio were varied. The data obtained were used to derive the equilibrium conditions for wet extraction.

Throughout these runs Freon 113 (1,1,2-trichloro-1,2,2 trifluoroethane) was used as a solvent, since it is similar in polarity and boiling point to hexane, yet it is not flammable, and therefore may be safely used without extensive explosion proofing of equipment.

Szego Mill

The Szego mill is a unique orbital mill developed by one of us (O. Trass) in cooperation with the late Mr. L.L. Szego. As illustrated in Figure 1, the mill consists of a stationary cylindrical grinding surface (stator), inside of which a number of helically grooved rollers rotate suspended on individual flexible shafts.

The rotor assembly consisting of three rollers is attached to a central, rigid shaft, driven from either below or above the mill. The feed enters the mill at the top and it is repeatedly crushed between the rollers and the stator. The crushing force is generated by the radial acceleration of the rollers. These grooved rollers facilitate the transport of material down the mill, by a screw-conveying effect, thus increasing the throughput of the equipment. The length, mass and number of the rollers and the shape, size and number of starts of the helical grooves may be varied, allowing for wide flexibility in the operation of the mill. The mill is compact, and has surprisingly high throughput capacity at low power consumption (2, 4).

The mill has been tested on the dry grinding of wheat, sand and limestone (4). The mill was shown to be highly effective in slurry grinding of coal, using oil or water as the liquid phase (5-8).

In the present study a small mill with an internal capacity of 4.5 L was used in the batch mode, since the mill capacity is too large to permit continuous operation in a laboratory situation.

Batch Extraction with the Szego Mill

Contact time, solvent-to-seed ratio, rotational speed and hold-up were varied in this series of tests. Hold-up refers to the percentage of the mill volume filled by the seed-solvent slurry. Solvent-to-seed ratio, denoted R, is expressed as volume of solvent/weight of seeds.

A measured quantity of Freon was introduced into the top of the mill, followed by the addition of whole canola seeds. The mill cooling system, consisting of an external cooling jacket, was turned on to prevent excessive loss of solvent through evaporation. The mill was operated at constant temperature for a preselected time period. The resulting slurry was discharged through the bottom opening of the mill, and its volume was measured. A 100-mL sample of this slurry was filtered, and the dried solids were analyzed for oil content and particle size distribution. The filtrate was dried in a rotary evaporator, and the soil residue was weighed.

The bulk of the slurry was returned to the mill for further grinding. At the completion of an experiment the solvent was recovered and the residual meal and oil were discarded.

As stated earlier, the residual oil content of the meal was determined by Soxhlet extraction, using the AOCS Standard Method Da 28-39 (3). All oil extraction efficiencies were expressed as a percentage of the total oil present in the seed. Therefore 0% represents nonextracted seed, while 100% is obtained when the seed contains no oil. Particle size analysis of oil-free solid was performed using Tyler sieves down to 45 μm.

RESULTS AND DISCUSSION

Laboratory Blender

Blending time was varied in the range of 1-9 min. As expected, the fraction of oil recovered initially increased with contact time. After an initial period of less than 9 min, further grinding did not result in additional oil extraction (Fig. 2). Both the time required to reach extraction equilibrium and the final oil recovery were functions of the solvent volume-to-seed weight ratio. High solvent-to-seed ratios resulted in rapid, almost complete, oil extraction. With a solvent-to-seed ratio of 6.6 the extraction was 94% complete in 3 min.

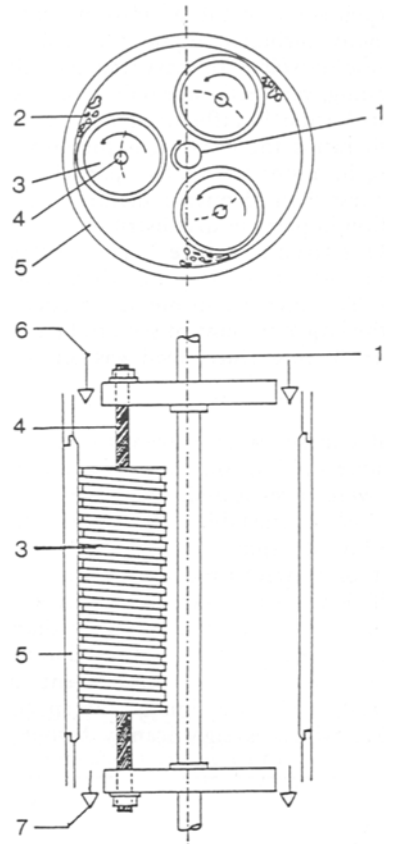

FIG. 1. Schematic diagram of the Szego Mill. (1) Rigid shaft, (2) material to be ground, (3) helically grooved roller, (4) flexible shaft, (5) stator (6) entrance port, (7) exit port.

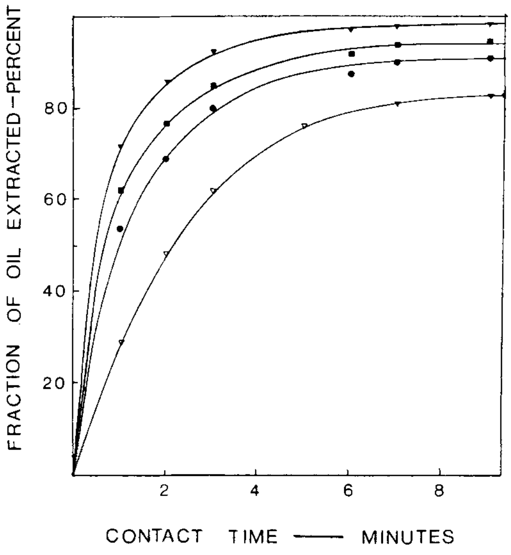

FIG. 2. Effect of contact time and solvent-to-seed ratio on extraction efficiency in the Waring blender. ▼ R = 6.66; ■ R = 3.33; ● R = 1.67; ▽ R = 1.25.

During blending, two processes occur simultaneously: the blender reduces the average particle size, while the extraction proceeds. The impact of the blade may also mechanically expell some of the oil into the solvent. Blending provides a highly turbulent liquid phase, which minimizes the liquid phase resistance to mass transfer. After the initial 3 min of blending, very little size reduction occurs. The particle size distribution is influenced by the solvent-to-seed ratio. At high ratios the grinding is somewhat more effective, resulting in a smaller median particle size and more particles with less than 45 micron diameter.

The variation in particle size distribution with solvent-to-seed ratio (R) is given in Figure 3. The median particle size increased from 140 µm to 180 µm when R decreased from 3.33 to 1.25. This increase in median particle size is probably due to the larger amount of seed to be ground at lower R values, since the total slurry feed was kept constant.

Szego Mill

The effect of contact time, solvent-to-seed ratio, rotation rate and volume hold-up on the extraction efficiency and particle size were investigated. Since the rotation rate was not continuously adjustable, all experiments were performed at 680 or 860 rpm.

The effect of contact time and solvent-to-seed ratio on extraction efficiency is illustrated in Figure 4.

Throughout this work, Freon rather than hexane was used, in order to minimize the fire hazard in the laboratory. Although the polarities and the solvent properties of hexane and Freon 113 are similar, the specific gravity of Freon is 1.57, which is significantly higher than that of hexane, which is 0.66. Rapeseed readily floats on Freon, an effect that is not significant in the highly turbulent Waring blender, but it may influence the performance of the Szego mill. The density differential between the seed and the solvent tends to force the seed away from the grinding zone. This effect is overcome by the turbulent flow in the mill and viscous drag. As the extraction proceeds, oil with a density of 0.9 enters the solvent and the average solvent density decreases, while the residual meal density increases, thus reducing the tendency of the meal to leave the grinding zone.

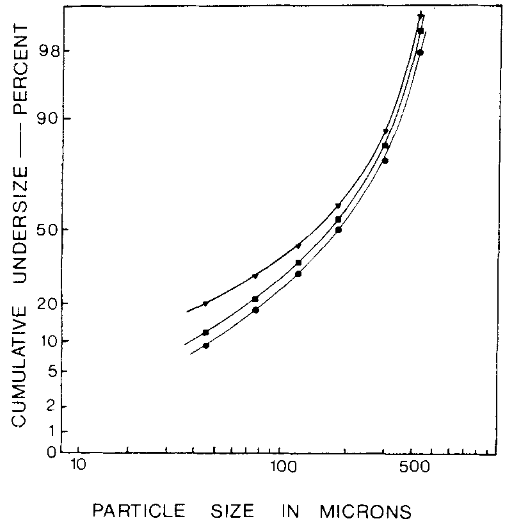

FIG. 3. Effect of solvent-to-seed ratio on grinding efficiency in the Waring blender; 3 min contact time. ♦ R = 3.33; ■ R = 1.67; ● R = 1.25.

Increased solvent-to-seed ratios results in higher final miscella densities, thus more effective grinding of seed was expected at lower solvent-to-seed ratios. As the ratio was decreased from 3.33 to 1.67 the median particle size was reduced from 150 to µm to 130 µm. Further decrease of the solvent-to-seed ratio to 1.25 resulted in an increase of the median particle size to 180 µm. Since the mill was operated with a constant slurry volume, the quantity of seed to be ground increased with decreased solvent-to-seed ratio. While this effect was overcome by the large miscella density difference for R = 3.33 and R = 1.67, as the value of R decreases further an equilibrium miscella concentration, and thus an equilibrium density, is approached, and the effect of increased seed mass predominates.

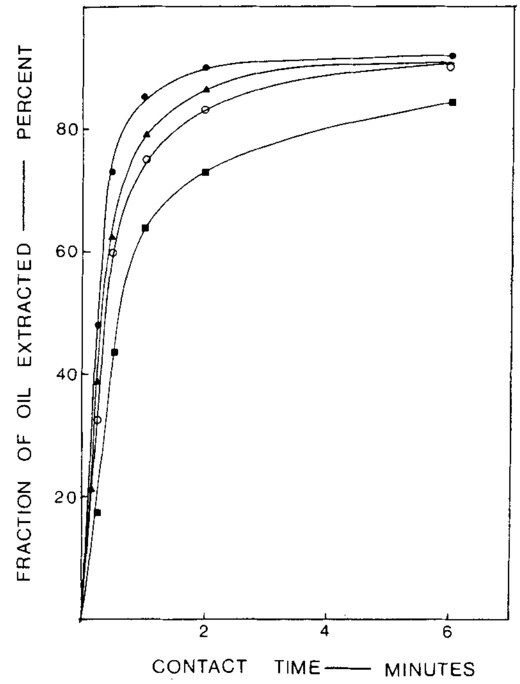

FIG. 4. Extraction efficiency in the Szego mill. ● 50% hold-up, 860 rpm, R = 1.67; ▲ 50% hold-up, 680 rpm, R = 1.67; ○ 75% hold-up, 680 rpm, R = 1.67; ■ 50% hold-up, 680 rpm, R = 1.25.

The extraction efficiency increased from 64 to 82.5% as the solvent-to-seed ratio was increased from 1.25 to 3.33. This was expected, since the concentration driving force increases at higher R values.

Figure 5 illustrates the variation of particle size with hold-up. The extraction efficiency was insensitive to mill

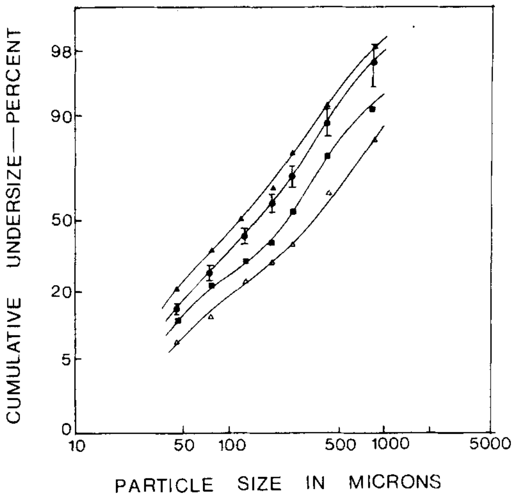

FIG. 5. Effect of hold-up and rotation rate on particle size distribution in the Szego mill; 1 min contact time. ▲ 50% hold-up, 860 rpm; ● 50% hold-up, 680 rpm; ■ 75% hold-up, 680 rpm; △ 25% hold-up, 680 rpm.

hold-up from 30% to 50%. In this range the liquid surface in the mill assumed a parabolic shape, which keeps most of the slurry in the grinding zone. At higher hold-ups a significant portion of the liquid bypasses the rollers through the annular space in the center of the mill, while at values below 30% less material is in actual contact with the rollers. Only the 30-50% hold-up range results in optimal particle size reduction and extraction only in the 30-50% hold-up range.

Extraction and grinding efficiency increased with increased rotation rate (Fig. 4). This effect was expected, since higher rotation rates generate more grinding surface, and provide higher radial acceleration, which in turn forces the material into the grinding zone. The increase, from 78% to 85.1%, in extraction efficiency when the rotation rate was raised from 680 to 860 rpm is offset by higher energy requirements and increased mechanical stresses in the mill at higher rotation rates, and therefore operation at high speeds may not be desirable.

Based on the results, 30 sec contact time, 50% hold-up, 1.67 solvent-to-seed ratio and 680 rpm are the optimal operating conditions for this mill in the batch mode. The mill operates more efficiently than the blender: Figure 6 compares the size distribution and Figure 7 compres the extraction efficiencies of the Waring blender and the Szego mill. The results clearly show that the Szego mill is more effective. At 1 min contact time, 1.67 solvent-to-seed ratio, the average particle size in the Szego mill was 130 μm compared with 260 μm in the blender, while the extraction efficiencies were 80% and 55%, respectively.

These tests demonstrated that the solvent milling of rapeseed can be highly effective in extracting oil from the seed, eliminating the need for the pretreatment steps of cracking, prepress and flaking.

The Szego mill is well suited to the rapid extraction of oil from canola seeds, since up to 85% of the oil present in the seed may be removed by a single grinding stage, with a contact time of 1 min, at a solvent-to-seed ratio of 1.66.

Further work in this program will be carried out to determine the effect of continuous grinding operations on the oil recovery and on the quality of the oil extracted by the Szego mill. Long-range plans for the commercialization of the process must take into account multistage countercurrent operations, and techniques for the separation of the meal and meal fines from the miscella. The use of more conventional solvents will be investigated, after appropriate changes in the equipment to permit safe operation in a flammable atmosphere.

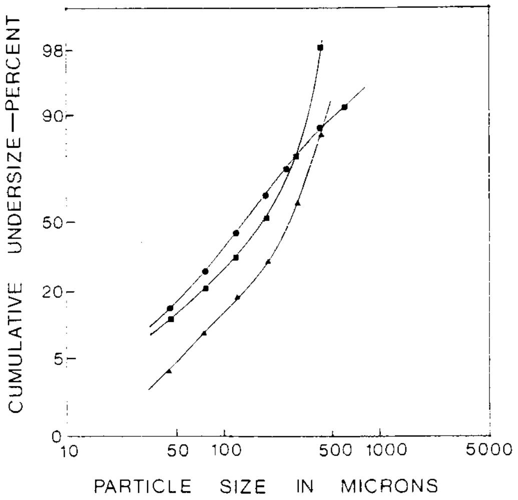

FIG. 6. Comparison of particle size distributions in the Waring blender and the Szego mill. ■ Waring blender, 3 min, R = 1.67; ● Szego mill, 1 min, R = 1.67, 680 rpm, 50% hold-up; ▲ Waring blender, 1 min, R = 1.67.

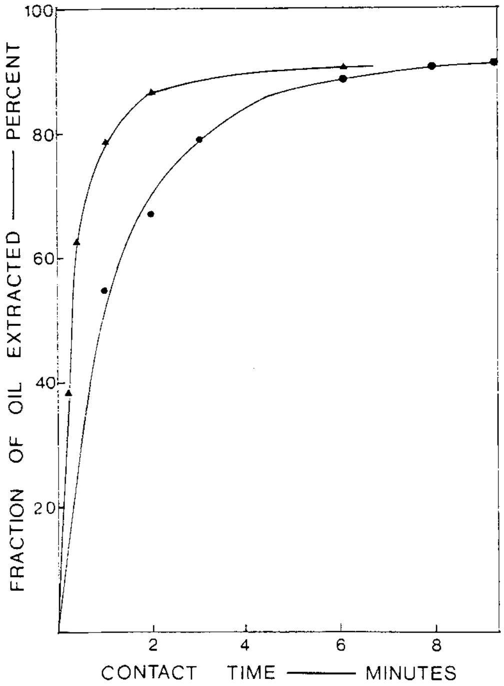

FIG. 7. Comparison of the extraction efficiencies of the Szego mill and the Waring blender. ▲Szego mill, 1 min, R = 1.67, 50% hold-up, 680 rpm; ● Waring blender, 1 min, R = 1.67.

ACKNOWLEDGMENTS

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