# Ethanolic Extraction of Sunflower Oil in a Pulsing Extractor

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**ABSTRACT:** Oil extraction by ethanol from partially defatted prepressed sunflower seeds in pulsed and nonpulsed extractors was compared. The oil yield was increased by 8.7% after short extraction periods (up to 6.06 residence times) with a pulsing flow, which was probably due to reduction in the axial dispersion that induces a greater concentration gradient between the miscella surrounding the solid and the bulk miscella.
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**KEY WORDS:** Alcohol, extraction, oil, pulsed extractor, sunflower.

The last few decades have seen a growing interest in replacing hexane as an extracting solvent for vegetable oils. Thus, potential solvents and processes have been reviewed (1–3). The most promising biorenewable solvents for oil extraction are water and alcohols, which have lower flammability and toxicity than hexane, and which often have the ability to remove antinutritive factors (4–6). Both ethyl and isopropyl alcohols have been studied for oil extraction from soybeans (7) and cottonseed (4–6).

Oil extraction from seeds involves two different mechanisms, washing and diffusion. It is generally assumed that the oil from broken cells is first extracted by washing, whereas that in intact cells is slowly extracted by diffusion. Wiese and Snyder (8) reported that soybean oil was extracted much more rapidly when the solvent was forced through the flakes in a packed bed. Forced flow increases the mass transfer rate from ruptured cells in a washing step, whereas the extraction of oil from unruptured cells is controlled by the diffusion of solute through the solid (9). The oil concentration in the miscella surrounding the solid is always greater than the concentration in the bulk miscella (10). This fact is noted especially when the oil is not totally miscible with the solvent, as observed by these authors when ethanol was used instead of isopropanol or hexane. The lower miscibility of the oil and the solvent leads to a greater oil concentration in the retained miscella and that surrounding the solid than in the bulk miscella (4). Concentration gradients in the bulk miscella are opposite to the flow of solvent and favor the transport of solute (oil) in axial direction (dispersion), reducing the concentration gradient between the solid and the bulk miscella and consequently the extraction rate. The pulsing flow reduces the axial dispersion and increases the mass transfer into the solid and the bulk miscella (11).

This communication presents the effect of pulsing flow on the extraction of oil from prepressed sunflower cake in an immersion extractor, with ethanol as solvent. The beneficial effect of the pulsing flow has been previously demonstrated in the analysis of sunflower polyphenolic extraction (12).

## MATERIALS AND METHODS

Partially dehulled sunflower seed was kindly supplied by ALCO, S.A. (Maia, Portugal). The seed was prepressed in a laboratory screw press, between 85 and 92°C, to obtain a semidefatted cake, whose characteristics were previously reported (12). Extractable oil from the cake, determined by Soxhlet extraction for 8 h with petroleum ether as solvent (Probus, Vigo, Spain), was 32.73% (w/w dry basis).

*Column extraction.* Sunflower cakes were extracted in laboratory immersion extractors (4.5 cm inner diameter, 10 cm height), kept at 50°C by a thermostated external water bath and fitted to a condenser to avoid solvent loss. The extraction of oil from the cake was carried out with reagent-grade 96% (vol/vol) ethanol (Analema, Vigo, Spain). Fresh solvent was pumped upward through the cake bed at 0.048 mL/s. The outlet miscella was periodically sampled, and the oil concentration was determined. A detailed description of the equipment was reported by Sineiro *et al*. (1996). One extractor was operated at continuous flow and the other with pulsed flow. The latter flow was achieved by means of a flexible tube, where the solvent accumulated until a timer-controlled electrical valve was opened (13). The bed void volume corresponding to the initial value was calculated from the residence time in the bed and the solvent flow during the initial filling of the extractor. The residence time was 9 min, and in the void space over the bed it was 10 min.

*Oil content in the extracts*. Oil content in the effluent from the extractors was determined by evaporating ethanol from the miscella in tared tubes.

## RESULTS AND DISCUSSION

Figure 1 shows the oil concentration in the miscella from the pulsed and nonpulsed extractor. The slope of extracted oil

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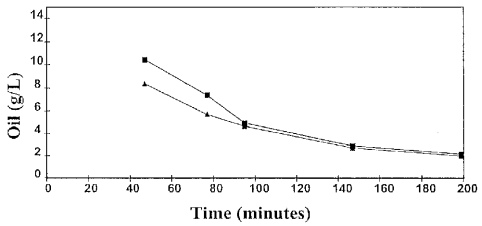


**FIG. 1.** Oil concentration in the outlet miscella in pulsing (-■-) and nonpulsing (-▲-) extractors

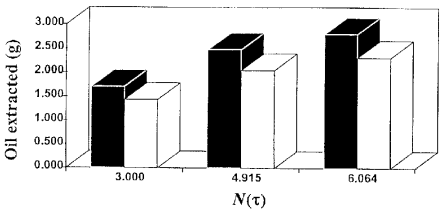


**FIG. 2.** Oil extracted as function of number of residence times $N(\tau)$ in the pulsing (■) and nonpulsing (□) extractors.

plotted vs. time decreases abruptly for the prolonged periods; this effect shows the nature of washing extraction of oil from ruptured cells. The remaining oil is slowly extracted by diffusion, and this mechanism is independent of the flow pattern. Moreover, at more prolonged extraction times, the methodology employed did not give reliable measurements of the miscella concentration. However, after 95 min (6.06 residence times), the residual oil in the cake was the same in both extractors.

The effect of pulsing flow was notable at relatively short extraction periods, corresponding up to six residence times in the whole extractor and 10 residence times in the packed solid. Higher outlet oil concentration could be attributed to the concentration gradient between the solid material and the bulk miscella. To compare the differences in extracted oil caused as a result of the pulsing flow, we estimated the oil extracted from the cake only during the initial extraction periods according to the equation:

$$\text{Extracted oil } (g) = Q_{\text{Et}} \left(\frac{\text{L}}{\text{min}}\right) \cdot \int_0^t C_{\text{Et}} \left(\frac{\text{g}}{\text{L}}\right) \cdot \text{d}t$$

where $Q_{\text{Et}}$ is the ethanol flow, $C_{\text{Et}}$ is the oil concentration in the miscella, and $t$ is the extraction time.

The value was calculated by assuming a linear behavior of oil concentration in the miscella with respect to time for residence times below 3. The number of residence times $N(\tau)$ was obtained as $N(\tau) = t/\tau$, where $t$ is the extraction time and $\tau$ the residence time (extractor volume/flow rate).

In Figure 2, the amount of oil extracted for both extractors up to 6.06 residence times is shown. The increase in oil extracted by means of the pulsing flow was achieved at relatively few residence times.

The pulsation induced an 8.7% increase in oil extracted; thus, after 95 min, 2.800 g oil (50.3% of total extractable oil) was extracted with the pulsed extractor, whereas only 2.313 g (41.6% of total extractable) was extracted in the nonpulsed mode.

Because the solvent flow was pulsed every 31.25 s with an amplitude of 0.8 mm, 17 pulses were provoked during one residence time in the bed (540 s), and the displacement provoked was 13.8 mm, which is almost one half of the bed height (30 mm). Thus, this fact, which reduces the axial dispersion against the flow, could increase the driving force responsible for the oil extracted from the cells.

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