Analysis of the Oil Extraction Process in Soybeans: A New Continuous Procedure

Kurt L. Wiese and **Harry E. Snyder***
Department of Food Science, University of Arkansas, Route 11, Fayetteville, AR 72703

Soybean oil extraction techniques were studied in which solvent was recirculated or pumped once through a suspension of soybean tissue. Both refractive index and ultraviolet absorbance were used to monitor the extraction continuously. Slices of soybean tissue showed rapid extraction from damaged tissue, followed by slow extraction from intact tissue. When soybean flakes were extracted, a continuously decreasing rate was noted. When solvent was forced through flakes, extraction was more rapid than when solvent was allowed to diffuse in and out of flakes. Reextraction of partially defatted flakes showed that the last soybean oil to be extracted was not inherently resistant to extraction. The adsorption of soybean oil to defatted flakes may account for slow removal of small quantities of oil at the end of the extraction.

Hexane extraction of soybean oil from soybean flakes has been the preferred method of oil extraction for about 40 yr. About 40 yr ago studies had been completed on important factors governing oil extraction by hexane, factors such as flake thickness, temperature, tissue disruption and solvent properties.

The bulk of oil is easily extracted, but the rate of extraction rapidly decreases; considerable time is required for the final oil concentration in the defatted flakes to reach 0.5%, an acceptable level of residual oil.

Several ideas have been proposed to explain the pattern of oil removal. As the extraction proceeds, the composition of extracted oil changes, with phospholipid making up a larger part of the crude oil at the end of the extraction than at the beginning (1,2). Therefore, decreased solubility of phospholipid in hexane may account for the pattern. The pattern may be explained by time needed for diffusion of solvent into the flake and miscella out of the flake (3,4). The calculation of two diffusion coefficients, one for 70-90% of the oil extraction and a second, smaller constant for the remaining 10-30%, can account for the extraction pattern (4). Because phospholipid makes up only 2-3% of crude soybean oil, it is unlikely that the slower rate of extraction is due to the phospholipid.

One continuing idea is that the first, rapid extraction is due to oil from broken cells, and the second, slower rate is due to diffusion of oil from unbroken cells. Othmer and Agarwal (5) postulated that cell walls of unbroken soybean cotyledon cells were impervious to hexane. They found that when intact soybeans or cotyledons are soaked in hexane, essentially no oil diffuses out, even with prolonged soaking. Viscosity of the extracting solvent and of oil miscella is an important factor, and Othmer and Agarwal (5) emphasized that viscous flow through capillaries controlled the rate of extraction.

The early studies were done by periodically sampling

*To whom correspondence should be addressed.

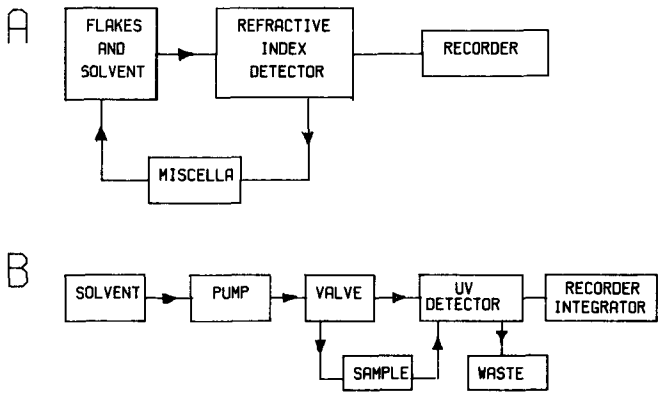

FIG. 1. Block diagram of flow patterns for continuous extraction monitoring. A, Recirculation of miscella with RI detector; B, Single pass extraction with UV detector. Filter was used in the line to debubble the solvent.

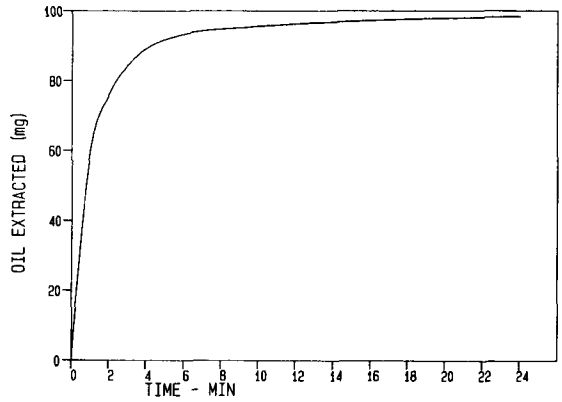

FIG. 2. Extraction of 590 mg full fat soybean flakes in 50 ml hexane with RI detector and recirculation of miscella at 2 ml/min.

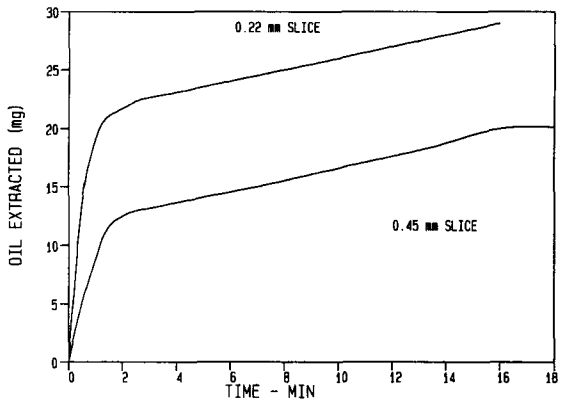

FIG. 3. Extraction of 0.22-mm slices (616 mg) and of 0.45 slices (549 mg) of soybean tissue in 50 ml hexane with RI detector and recirculation of miscella at 2 ml/min.

either the flakes or the extract to determine residual oil. We decided to reexamine the changing rates of extraction by using a continuous process and monitoring system. A better understanding of the extraction process could lead to a more efficient extraction.

MATERIALS AND METHODS

Materials. Soybean flakes, both full fat and defatted, were obtained from a local commercial processor and were stored at 4 C in polyethylene bags until used. Full fat flour was prepared by grinding dehulled, degermed whole soybeans in a U/D Cyclone Sample Mill. The flour was sieved through a 40-mesh screen to remove large particles.

The hexane used for extractions and for zeroing the refractive index (RI) or ultraviolet (UV) detectors was HPLC grade. Soybean oil used to study adsorption was crude soybean oil commercially extracted.

Extractions. The arrangements for recirculating solvent and miscella using the RI detector and for single pass with the UV detector are shown in Figures 1A and 1B, respectively. The RI detector was Altex model 150, and a reciprocating Altex pump (model 110A) was used. The refractive index of the hexane used was 1.375 at 25 C, and that of the crude soybean oil was 1.4705-1.5026 (6). A standard curve relating refractive index response on a recorder to known amounts of crude soybean oil was prepared and used to measure amounts of oil extracted.

Oil extraction measurement by the UV detector (Hitachi model 100-10 with 20-μl flow cell) was done at 225 nm or at 300 nm depending on the sensitivity needed. At each wave length a standard curve was obtained by injecting known amounts of crude soybean oil into the system and by-passing the sample holder. Oil peaks were integrated, and integration values were plotted vs amounts of oil. The integrator used was Varian CDS 111, and parameters were signal/noise ratio = 1, initial peak width = 64, tan % = 0. Perpendicular areas, rather than tangential areas, were calculated. A filter was inserted in the line to debubble the solvent.

RESULTS AND DISCUSSION

To obtain a continuous record of extraction of soybean tissue, we made use of a refractive index detector and pumping arrangement as shown in Figure 1A. The hexane solvent was recirculated, so that the first part of the extraction was due to solvent whereas later extraction was due to a soybean oil miscella.

Figure 2 shows the results of extraction of soy flakes. The soybean oil was removed rapidly at the beginning, with approximately 80% extracted at 2 min and 95% at 4 min. The extraction continued at a decreasing rate for the 24 min of the experiment.

The effect of flaking the soybean tissue can be seen by comparing Figure 2 with Figure 3, in which extraction curves are shown for two samples of thinly sliced soybean tissue with minimal tissue disruption. The amounts of tissue were approximately the same as in Figure 2, but only about 15-25% of the oil was rapidly extracted. After the initial extraction there was a constant rate of extraction for about 14 min. At the end of the 18-min extraction period, there was a decrease in rate for the 0.45-mm slices. Fan et al. (3) found that rates of extraction of oil from peanut slices are diffusion controlled, and depend after the initial rapid extraction on the thickness of peanut slices. If monitoring had been extended for 100 min, as Fan et al. (3) did, different rates for different slice thicknesses probably would have been found.

There is support in the literature (5) for the idea that intact soybean tissue cannot be extracted. Figure 3 shows that a slow extraction of intact tissue proceeded, but during the time period shown, 25% or less of the total oil was extracted. Also, Figure 3 shows that the thickness of slice influenced the rate and amount of oil extracted during the initial time period. By doubling the thickness of the slice, approximately 1/2 as much oil was extracted initially. One would expect that for the same weight of tissue there would be 1/2 as much disrupted tissue with 0.45-mm slices as with 0.22-mm slices, due to 1/2 as many cuts. Hence, the amounts of oil extracted initially in Figure 3 corresponded to the amount of tissue disrupted.

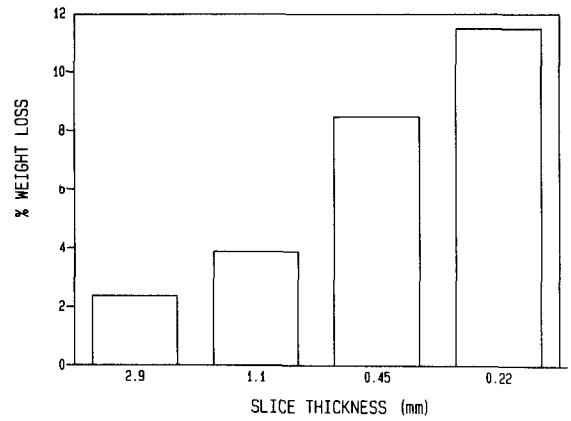

FIG. 4. Sliced soybean tissue extracted for 20 hr (Goldfisch) with hexane.

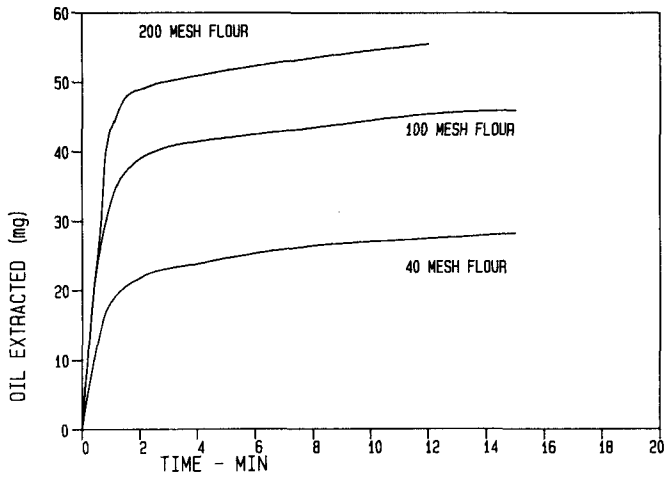

FIG. 5. Extraction of various full fat soybean flours (250 mg) in 50 ml hexane with RI detector and recirculation of miscella at 2 ml/min.

TABLE 1

Extraction With Hexane of Different Particle Sizes of Soybean Flour by Recirculation of Solvent[a]

Flour size	Weight of tissue (g)	Initial oil extracted (%)	Initial extraction rate (g/min-ml)	Extraction rate after 7.5 min (g/min-ml)
40-mesh	0.25	8.3	3.16×10^{-4}	4.88×10^{-6}
100-mesh	0.25	14.8	1.03×10^{-3}	1.65×10^{-5}
200-mesh	0.27	16.8	8.69×10^{-4}	1.05×10^{-5}

[a]Flow rate 2 ml/min through RI detector and total volume of 50 ml.

Othmer and Agarwal (5) used the equation $-dC/dt = KS^{-3.97} C^{3.5}$ to describe the extraction of oil from flaked soybean tissue where $-dC/dt$ is the rate of extraction, S is the flake thickness, C is the residual oil concentration and K is a proportionality constant. We applied the above equation to the initial rate of extraction for the two thicknesses of soybean tissue and found that the rate was only 3 times greater for the thinner tissue (0.46 mg/min ml vs 0.15 mg/min ml). The equation predicts that the rates should be 16× different for a doubling of thickness; however, Othmer and Agarwal experimented with flaked soybeans whereas our results were with the damaged tissue from sliced soybeans.

Samples of soybean tissue of various thicknesses were extracted in a Goldfisch apparatus for 20 hr. The results (Fig. 4) indicated that there is tissue in soybeans that is resistant to oil extraction. One would have expected about 20% weight loss in these samples after total extraction, whereas actual weight loss ranged from about 2-12%.

In another series of experiments, we investigated the effect of particle size of full fat soybean flours on extraction rates and total extraction of oil. Figure 5 shows the extraction patterns for 40-, 100- and 200-mesh flour. The amounts of soybean tissue were about 250 mg per experiment; therefore, complete extraction would have given about 50 mg of oil. For 200-mesh flour the extraction appeared to be completed in about 2 min, but a continual slow increase in refractive index showed further extraction was taking place over the 15-min duration of the experiment. As particle size increased, the amount of oil extracted during the initial rapid phase decreased, and the subsequent slow extraction was at essentially the same rate for the three particle sizes. Table 1 shows extraction rates calculated for the initial phase and subsequent phase of the extraction.

The data for the 200-mesh flour indicated that if the particle size of the flour was small enough (tissue was totally disrupted), the extraction occurred very rapidly, but when the proportion of intact tissue increased, the amount of oil extracted initially decreased with 100-mesh flour and more dramatically with 40-mesh flour.

Extraction of these three samples of soybean flour was also done by Goldfisch for 5 hr (Fig. 6). The 100- and 200-mesh samples appeared to be fully extracted (more weight loss than expected was obtained with 200-mesh flour), but 40-mesh flour yielded only a little over half of the expected 20% weight loss.

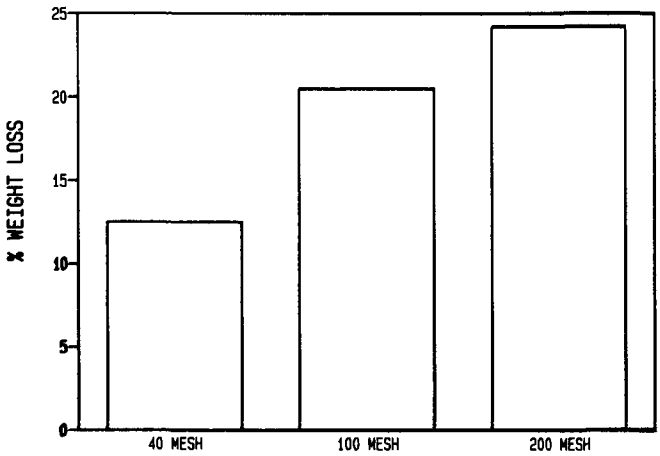

FIG. 6. Various mesh sizes of soybean tissue extracted for 5 hr (Goldfisch) with hexane.

The results with the refractive index detector and recirculating miscella showed that the degree of disruption of soybean tissue was an important factor in the rate and amount of oil extracted. With soybean flakes or 200-mesh flour, a rapid, complete extraction was followed by a slow, additional extraction. When soybean tissue was less disrupted, as with sliced tissue or 40-mesh flour, only a portion of the oil was extracted rapidly, and subsequent extraction was very slow.

The refractive index detector gave useful results, but there also were some difficulties. We did not have precise temperature control on the system, and the sensitivity of RI to temperature caused fluctuations that made comparison of results difficult. Also, we wanted to experiment with the effect of different solvent systems on the extraction, but the recirculation arrangement caused the solvent to constantly change as the miscella became more concentrated. Consequently, a change was made to an UV detector with no recirculation of extract (Fig. 1B).

The continuous extraction is plotted in Figure 7. In this plot the ordinate gave the concentration of oil being extracted at a particular time. The total amount of oil extracted was obtained by integration of the area under the curve. The integration value was dependent on the flow rate of solvent; consequently, a doubling of the flow rate meant that the integration value had to be doubled to give the correct amount of oil extracted.

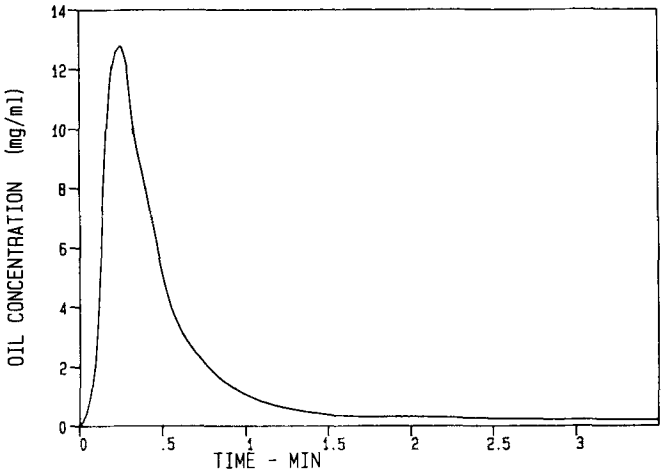

FIG. 7 . Extraction curve for full fat flour (50 mg) using the UV detector (300 nm) with hexane as solvent (2 ml/min).

Figure 7 shows that the extraction under these circumstances was quite rapid, being essentially complete in 1.5 min. There was little indication in this plot of a "hard to extract" fraction, although the rate of extraction continually decreased.

Table 2 shows the results of increasing the polarity of the extraction solvent by adding 1% IPA to hexane. Significantly more oil was extracted from full fat flour with 1% IPA in hexane than with hexane. The same trend was observed when full fat flakes were extracted with the two solvents, but the difference was not significant. The additional extraction may have been due to increased phospholipid extraction, but samples were not analyzed for phospholipid. The greater percentage of oil extracted from flour than from flakes by both solvent systems was due to a fixed time used for the extraction, and because oil is extracted more rapidly from flour than from flakes, more oil was extracted over the fixed time.

Usually the extraction pattern was similar to that shown in Figure 7, but occasionally a greatly decreased peak was observed with prolonged extraction (Fig. 8). Upon opening the sample holder after the extraction, usually the flakes or flour were packed at the end of the sample holder toward which the solvent was flowing. But when a decreased peak with a prolonged extraction pattern was observed, the flakes or flour were evenly suspended in the sample holder and not packed at one end. This observation led us to the conclusion that oil was extracted much more rapidly when solvent was forced to flow through the flakes than when flakes were suspended, and the extraction mechanism was due mainly to diffusion.

In an attempt to learn what was causing the "hard to extract" oil that had been observed by others, we compared the extraction of full fat flakes with that of partially extracted flakes. Our reasoning was that if the "hard to extract" oil were due to unruptured oil cells or to intact tissue that was difficult for the solvent to penetrate, then partial extraction would not change that situation. "Hard to extract" oil would still be present and would be extracted at the end of the

TABLE 2

Comparison of Oil Extractions With Two Solvent Systems by a Single Pass Through Full Fat Soybean Flour and Full Fat Soybean Flakes[a]

	Percentage Soybean Oil Extracted	
	Hexane	1% IPA in Hexane
Full fat flour	22.50 (1.82)a[b,c]	26.04 (0.28)b
Full fat flakes	17.14 (2.51)a	18.35 (2.07)a

[a]Flow rate 2 ml/min, extraction time 4 min, UV detector at 300 nm.
[b]Figures in parentheses are standard deviations.
[c]Different letters in rows indicate a difference at the 5% level.

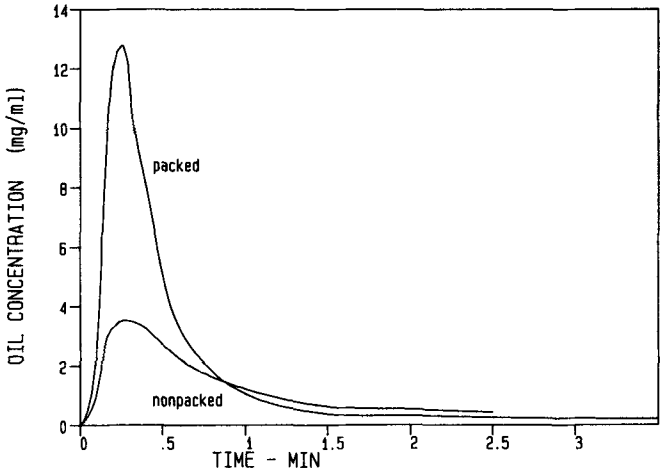

FIG. 8. Extraction curves for two full fat flour samples (50 mg) at 300 nm and hexane as solvent (2 ml/min). The decreased extraction resulted from failure of the sample to pack at one end of the sample holder.

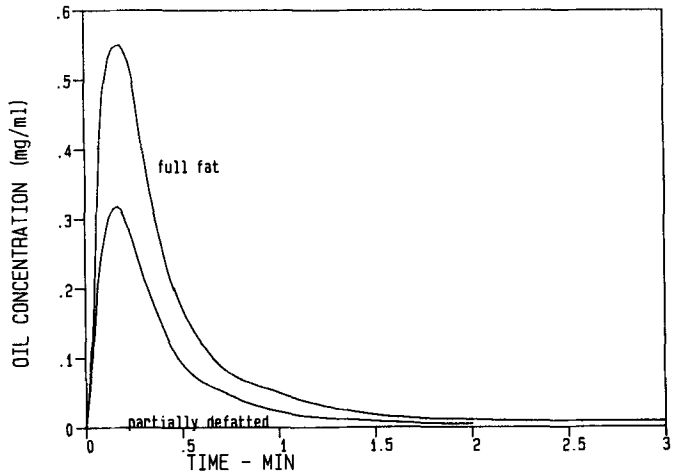

FIG. 9. Extraction with hexane of two soybean flake samples. The full fat sample was 15 mg and the partially defatted sample was 13 mg. Flow rate 2 ml/min and wavelength 225 nm.

extraction period. The results of this experiment are shown in Figure 9. The partially extracted flakes showed a pattern identical to full fat flakes except that the amount of oil extracted had decreased. This result indicated that there was no unique "hard to extract" oil under our extraction conditions. The oil that was extracted during the end of the extraction time was oil that was least accessible. When the readily extracted oil was removed by partially extracting the flakes, then the remaining oil became easily extracted. The results shown in Figure 9 did not support the concept that there was a "hard to extract" fraction of oil due to unruptured cells or intact tissue.

It is well established that soybean protein binds lipid in aqueous systems (7). No similar data have been found for a solvent system such as hexane. During extraction of soybean oil, the extracting solvent usually flows counter to the flow of flakes, so that full fat flakes are contacted by full fat miscella and defatted flakes are washed with fresh solvent. As the content of soybean oil in the solvent increases, the polarity of the solvent increases, and this change may influence any binding of soybean oil to soybean flakes.

We experimented with the concept of soybean oil being bound to soybean flour by placing defatted soybean flour in the samples holder (Fig. 1B) and pumping hexane through the system to establish a baseline. Then 0.695 mg of soybean oil (20 μl of a miscella) was injected into the line, and the amount of oil coming off the defatted flour was measured by the integrator. The recorded elution pattern is shown in Figure 10 for the oil sample injected with nothing in the sample holder and with defatted flour in the sample holder. These results show that 14.3% of the injected soybean oil was retained on the defatted flour. Based on the defatted flour weight, this amounted to 0.3% soybean oil retained. Subsequent injections showed the same pattern of adsorption of defatted flakes, so the flakes had capacity for more adsorption of soybean oil. When 20 μl of IPA was injected into the hexane stream, the adsorbed oil was eluted from the soybean flour as indicated by a peak emerging from the sample of soybean flour. The peak was not due to IPA being detected.

These results demonstrated that defatted soybean tissue could adsorb soybean oil in the presence of hexane as a solvent. No attempt was made to determine the total capacity of adsorption under these

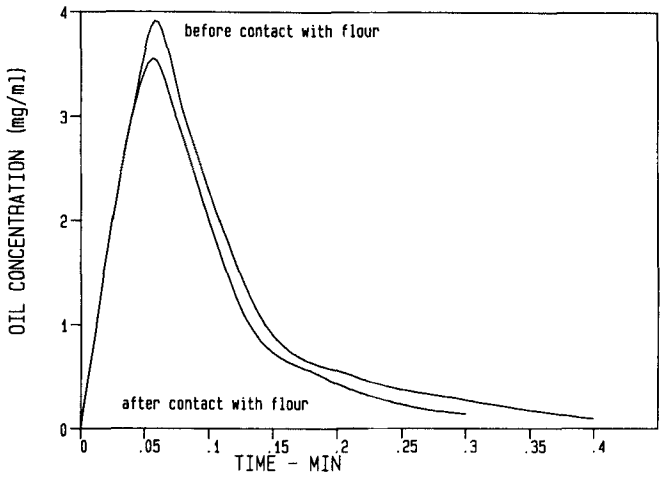

FIG. 10. Decrease in soybean oil detection as a result of being pumped over 26 mg of defatted soybean flour. Detection at 225 nm.

conditions, but the calculation of 0.3% of the defatted flour weight being adsorbed with most of the soybean oil being eluted showed the capacity of soybean tissue for adsorption of soybean oil in the presence of hexane was not great. Still more oil could be adsorbed by the flakes, as shown by repeated injections, and oil adsorption may well be a factor in the long time needed to completely extract flakes.

REFERENCES

1. Bull, W.C., and T.H. Hopper, *Oil and Soap 18*:219 (1941).
2. Karnofsky, G., *J. Am. Oil Chem. Soc. 26*:564 (1949).
3. Fan, H.P., J.C. Morris and J. Wakeham, *J. Ind. Eng. Chem. 40*:195 (1948).
4. Osburn, D.F., and D.L. Katz, *Trans. Am. Inst. Chem. Eng. 40*:511 (1944).
5. Othmer, D.F., and J.C. Agarwal, *J. Chem. Eng. Prog. 51*:543 (1955).
6. Pryde, E.H., in *Handbook of Soy Oil Processing and Utilization*, edited by Erickson, D.R., E.H. Pryde, O.L. Brekke, T.L. Mounts and R.A. Falb, American Soybean Association, St. Louis, MO, and American Oil Chemists' Society, Champaign, IL, 1980, pp. 35-37.
7. Hutton, C.W., and A.M. Campbell, in *Protein Functionality in Foods*, edited by Cherry, J.P., American Chemical Society, Washington, DC, 1981, Chapt. 9.

[Received September 3, 1986]