

Design of Oilseed Extractors I. Oil Extraction (Supplement)

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This supplements an earlier article (1) which presented a method of calculating retention time required for countercurrent extraction of oilseeds from laboratory extraction rate data. The method is based on the empirical observation that extraction rate is governed largely by the rate at which undissolved oil goes into solution, resistance to diffusion from the flakes becoming of consequence only in thick flakes. This article introduces the concepts of parabolic concentration gradient in the flakes and of an apparent diffusivity which is an inherent property of the seed. The method is applied to the solution of several commercially important extractions.

This is a supplement to an earlier article (1) with which it is assumed the reader is familiar. The article disclosed a method for design of equipment for extracting oilseeds with hexane based on the supposition that the major impediment to extraction was slow solution of phosphatides, which hindered solution of the oil.

The method was, perhaps unconvincingly, demonstrated in application to soybean flakes 0.22 mm thick, while it is now the commercial practice to extract flakes 0.25–0.3 mm thick. It was demonstrated that the time needed for countercurrent extraction of 0.22-mm flakes is little more than the time needed to extract in the laboratory by successive applications of hexane to a batch. As flakes are thickened, resistance to diffusion from the flakes becomes relatively greater, and the comparative time may increase.

In the method disclosed earlier, diffusion of oil was handled by postulating, at any given time after the beginning of extraction, a miscella of uniform concentration within each flake. Oil diffused from this miscella into the miscella surrounding the flake according to the equation

$$dr/dt = -k(y - a) \quad [1]$$

(see Nomenclature footnote), a plausible, though inexact, assumption that suffices for engineering design so long as the diffusion rate is large compared with rate of solution of oil. This supplement introduces a more plausible, though still inexact, postulate that incorporates diffusion of oil through the miscella in the flake; it also demonstrates how the design method can be applied commercially.

Nomenclature

a, concentration of miscella used in extraction rate experiment, v/v; e, voids fraction in extracted flake; r, residual oil in laboratory extracted flakes, v/v holdup; s, half-thickness of flakes, mm; t, extraction time, min; w, parameter in equation of parabola; x, distance from center of flake, mm; y, average oil concentration in flake at time (t) in laboratory extraction, v/v miscella; y', oil concentration in flake at x at time t in laboratory extraction, v/v miscella; z, undissolved oil concentration, v/v holdup; D, diffusivity, mm/min; R, residual oil in flakes at time (t) in continuous extraction, v/v holdup; X, concentration of miscella at time (t) in continuous extraction, v/v; Y, average oil concentration in flake during continuous extraction, v/v miscella.

PARABOLIC DISTRIBUTION OF y'

The new postulate is that a plot of the concentration of oil, y', in the miscella in the holdup at a distance x from the center of a flake, is a parabola, and that y' at the edge of the flake is the same as the concentration of the surrounding miscella. Since a flake is porous, oil in the miscella held in the pores should diffuse toward the flake surfaces approximately in accord with diffusion theory, which suggests a miscella concentration gradient from center to surfaces that is qualitatively parabolic. Certainly, a parabola is in accord with the readily apparent requirements that the concentration gradient is zero at the center and a maximum at each surface. Other assumptions relating to undissolved oil are the same as those introduced in (1).

Figure 1 shows a section of a flake, containing undissolved oil and miscella phases, surrounded by miscella of concentration a. Superimposed is a graph, y' vs x, showing the instantaneous parabolic distribution of the miscella concentration. The equation of the parabola is:

$$y' = a + w(s^2 - x^2) \quad [2]$$

where w is a parameter that decreases with increasing time. The integrated average concentration y is:

$$y = 1/s \int_0^s y' dx = a + 2s^2 w/3$$

Solving for w:

$$w = 3(y-a)/2s^2 \quad [3]$$

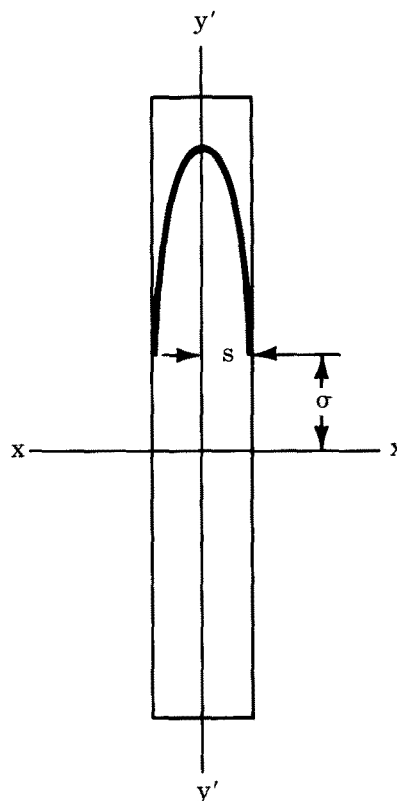


FIG. 1. Oilseed flake with superimposed parabola representing y' vs x.

The rate of flow of oil (vol oil/sq mm) from both flake surfaces, based on the supposition of diffusion in a porous solid, is $-2Ddy'/dx$ at $x = s$; i.e., the flow rate is proportional to the concentration gradient. D is the apparent diffusivity, a property of the seed, designated "apparent" to emphasize that it is the consequence of a theoretically imperfect concept. Differentiating Equation 2 gives $dy'/dx = -2wx$; so dy'/dx at $x = s$ is $-2ws$.

The rate of change of r must match flow through the surfaces. Consider a portion of the flake in which the area of each surface is one sq mm:

$$\begin{aligned} -2sedr/dt &= 4Dws \\ dr/dt &= -2Dw/e \end{aligned} \quad [4]$$

where e is the fraction of volume occupied by miscella in an extracted flake. Substituting for w from Equation 3:

$$dr/dt = -3D(y - a)/es^2 \quad [5]$$

Let

$$k = 3D/es^2 \quad [6]$$

Equation 5 is now identical with Equation 1. Consequently, the entire calculation procedure and the extractor design of (1) are applicable. Also, the rationality of Equation 1 is better established.

THE MISCELLA HOLDUP

In (1) the miscella holdup in soybean flakes was stated, without explanation, to be 0.788 l/kg of meats. Actually, this is the holdup to which flakes drain in commercial extractors, which is greater than the holdup within the flakes. Some miscella undoubtedly adheres to the surface of the flakes within the extractor. To have used a different holdup within the flakes than in the drained flakes leaving the extractor would have required an explanation that seemed unnecessary, since the holdup used in the calculation had little effect on the result. However, the holdup is now required by the derivation of Equation 6 to be the miscella in the voids.

A soybean flake consists by weight of 70% meats, 20% oil and 10% water. A drained, hexane-wet, extracted flake contains 0.33 g hexane/g solvent-free flakes, equivalent to $0.33(70 + 10) = 26.4$ g/100 g unextracted flakes. Converting to volumes: meats = $70/1.43 = 49$; water = $10/1 = 10$; oil = $20/0.9 = 22$, and hexane = $26.4/0.64 = 41$. Consequently, $e = 41/(49 + 10 + 41) = 0.41$; and the holdup in l/kg meats is $41/70 = 0.59$. To convert from g oil/g meats to the units of r , vol oil/vol holdup, multiply by $70/(0.9 \times 41) = 1.88$.

RESIDUAL AND UNDISSOLVED OILS AS FUNCTIONS OF s AND t

Extraction rate curves determined by the method of Wingard and Shand (2) for flakes of four different thicknesses made from the same beans are shown in Figure 2 (3). It is doubtful that the curves have any validity for the first minute of extraction. The first measurement in the rate experiment was made at t greater than 1; a smooth curve was drawn to the initial oil content at

zero time. Yet Frampton et al. (4), who measured residual oil during the first 50 seconds of extraction of oil from cottonseed grits of several diameters by the hexane-acetone-water azeotrope, found that the oil concentration dropped practically instantly to a value which then remained constant for the rest of the 50 seconds. The concentrations, decreasing with decreasing grit diameter, plotted as a straight line against the reciprocals of the diameters.

This suggested reading from Figure 2 the ordinates of each curve at $t = 1$. Thus, for $s = 0.115$, $r = 0.16$; for $s = 0.18$, $r = 0.24$; for $s = 0.215$, $r = 0.30$; for $s = 0.28$, $r = 0.38$. Plotting these r 's against the reciprocals of s gives a straight line, suggesting that hexane, too, instantly extracts available oil, and that most of the first minute of each curve of Figure 2 should be replaced by a horizontal line.

From their respective definitions:

$$z = (r - y)/(1 - y) \quad [7]$$

Substituting for y from Equation 1, assuming $a = 0$:

$$\frac{z}{r} = \frac{(k + 1/rdr/dt)}{(k + dr/dt)} \quad [8]$$

When useful values for k , r and dr/dt are substituted in Equation 8, z/r increases rapidly with time and is more than 0.9 for most of the duration of the extraction. Such high ratios are a measure of the validity of the undissolved oil concept: solution rate is slow compared with rate of transfer from the flakes.

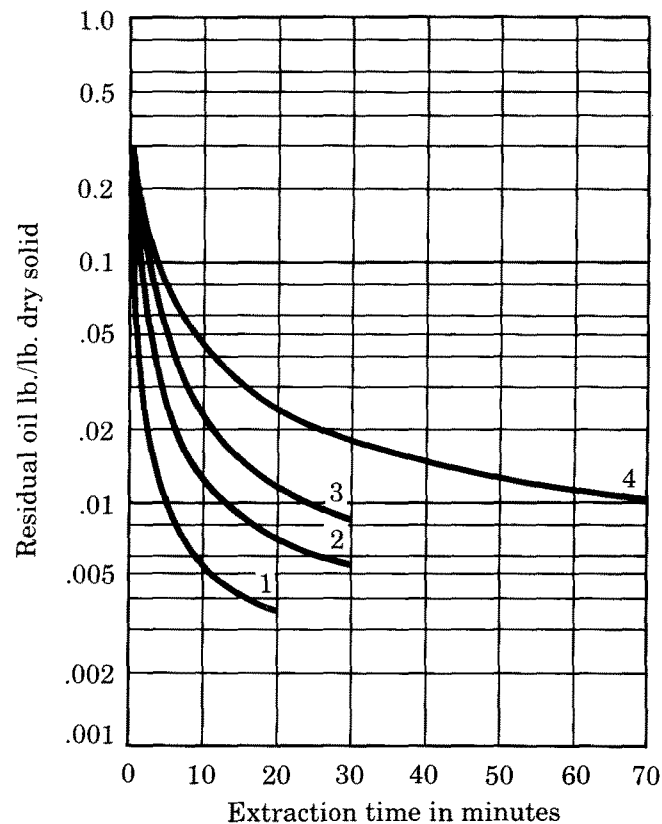


FIG. 2. Extraction of soybean flakes of different thicknesses by percolation with hexane. 1, 0.23 mm; 2, 0.36 mm; 3, 0.43 mm; 4, 0.56 mm.

VALIDITY OF THE UNDISSOLVED OIL CONCEPT

The undissolved oil concept originates from the following experiences:

- It best predicts experimental extraction rates (3).
- Extracted flakes into which oil is reintroduced extract much more rapidly the second time (3).
- Phosphatides are found in the miscella only after most of the oil is extracted (3).
- Extraction of oil from soybeans and cottonseed by the hexane-acetone-water azeotrope, in which phosphatides are very soluble, is rapid (5).
- The vertical basket extractor, in which flow is cocurrent during a little more than half of the total extraction time, was widely used until it was superseded in the 1950's by horizontal extractors such as the Rotocel, in which flow is cocurrent only about 10% of the time. The Rotocel did not extract to a residual oil lower than the basket extractor did.

APPLICATION OF THE DESIGN METHOD

It will be assumed, as in (1), that the flakes are extracted to 0.5% residual oil with hexane at 1:1 solvent to flakes ratio. Figure 3 shows a volume balance for a percolation extractor based on 1 kg of meats, equivalent to the balance of (1) but reflecting the new miscella holdup. On this basis oil in the exiting flakes is $0.005(80/70)(0.59)(1.88) = 0.0063$ l; R is $0.0063/0.59 = 0.0107$ l oil/l holdup.

The volume balance from Figure 3 is:

$$0.59R = 2.0407X + 0.0063 \quad [9]$$

The iteration equation, equivalent to Equation 9 of (1), is:

$$R_2 = \frac{z/(1-z) + 2R_1/kdt - 0.0031}{0.5/(1-z) + 1/kdt - 0.1446} - R_1 \quad [10]$$

To demonstrate their commercial application, these equations were applied in the solution of several problems.

Flake thicknesses between 0.254 mm and 0.304 mm are more likely to be used in commercial practice than the 0.22-mm flakes described in (1). It was demon-

strated there that, for 0.22-mm thick flakes, $k = 5$ was probably a conservative guess. From Equation 6, assuming that D is independent of flake thickness, corresponding k 's are: for 0.254 mm flakes, $k = 5(0.22/0.254)^2 = 3.75$; for 0.304 mm flakes, $k = 5(0.22/0.304)^2 = 2.6$.

Data compatible with the curves of Figure 2 for the extraction rates of 0.254-mm and 0.304-mm flakes were converted to the units of r by multiplying by 1.88. Values of r vs t read from large scale plots and R 's calculated by substituting in Equation 10 are listed in Table 1. The columns headed I list t vs r vs R for 0.254-mm flakes extracted in a Rotocel with a retention time of 13 min in the extraction zone, the first 1.3 min in cocurrent flow. The columns headed II list t vs r vs R for 0.254 mm flakes extracted cocurrently for nine min prior to countercurrent extraction. The columns headed III list t vs r vs R for 0.304-mm flakes extracted in a Rotocel with a retention time of 18 min in the extraction zone, the first 1.8 min in cocurrent flow. The conclusion to be reached in each case is that when the time is reached at which the residual oil r measured in the laboratory is the desired 0.0107, R likewise approaches 0.0107. From columns II, this appears to be true even though nine min of a total of 11.7 are in cocurrent flow.

The columns headed IV examine the extraction of flakes in an extractor too small to provide the extraction time needed to achieve a low residual oil. Flakes 0.304 mm thick are extracted in a Rotocel which provides only 10 min. The solution required trial and error, since the number 0.0031 in Equation 10 is proportional to residual oil, which is no longer 0.0107. When 0.0052 was substituted, corresponding to a residual oil of $0.0107(0.0052/0.0031) = 0.018$, the calculated R at 10 min matched.

The reason that commercial extraction times are so close to those in the laboratory, particularly when the desired residual oil is very low, can be ascertained by examining Table 1. As dr/dt becomes very small at the end of extraction, diffusion from the flake continues at a relatively high rate, so R catches up with r . Even when there is a short countercurrent time after long soaking, the initially high R is very rapidly reduced.

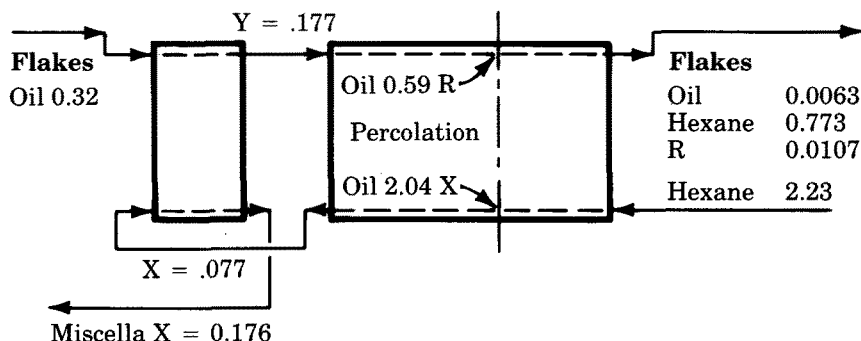


FIG. 3. Oil and hexane volumes balance in continuous extraction. Basis: 100 kg meats. Volumes in liters.

TABLE 1

Iterative Calculation of R by Equation 10

Extraction of 0.254-mm flakes						Extraction of 0.304-mm flakes					
I ^a			II ^b			III ^c			IV ^d		
s = 0.127	k = 3.75		s = 0.127	k = 3.75		s = 0.152	k = 2.6		s = 0.152	k = 2.6	
t	r	R	t	r	R	t	r	R	t	r	R
1.3	.1297	.263	9	.0132	.188	1.8	.1202	.258	1	.1792	.299
1.35	.1245	.2445	9.025	.013175	.1765	1.85	.1169	.2447	1.1	.1700	.2758
1.45	.1137	.2122	9.05	.01315	.1659	1.9	.1139	.2325	1.2	.1616	.2557
1.6	.0978	.1728	9.075	.013125	.1559	1.95	.1109	.2213	1.4	.1461	.2225
1.9	.0799	.1263	9.1	.0131	.1465	2.05	.1052	.2012	1.6	.1325	.1963
2.2	.0677	.1001	9.15	.01305	.1296	2.35	.0905	.1558	1.9	.1142	.1668
2.5	.0564	.0809	9.2	.0130	.1149	2.7	.0780	.1231	2.3	.0943	.1338
2.9	.0479	.0660	9.25	.01295	.1020	3.1	.0669	.0992	2.7	.0789	.1096
3.3	.0414	.0558	9.3	.0129	.0907	3.5	.0575	.0824	3.1	.0669	.0913
3.7	.0352	.0474	9.4	.012825	.0723	3.9	.0508	.0709	3.5	.0575	.0775
4.1	.0306	.0403	9.5	.01275	.0472	4.35	.0441	.0605	3.9	.0508	.0672
4.6	.0269	.0344	9.7	.01257	.0233	4.9	.0374	.0504	4.35	.0441	.0573
5.2	.0226	.0283	10	.0123	.0140	5.5	.0325	.0429	4.9	.0374	.0475
5.8	.0199	.0242	10.5	.0117	.0124	6.15	.0295	.0380	5.5	.0325	.0401
6.4	.0176	.0208	11	.0112	.0116	6.8	.0259	.0330	6.15	.0295	.0352
7.1	.0162	.0187	11.7	.0107	.0108	7.55	.0235	.0293	6.8	.0259	.0301
8	.0147	.0165				8.5	.0205	.0250	7.55	.0235	.0264
9	.0132	.0144				9.5	.0186	.0221	8.5	.0205	.0221
10	.0123	.0131				10.5	.0169	.0196	9.5	.0186	.0192
11	.0112	.0116				11.5	.0156	.0177	10	.0177	.0180
11.7	.0107	.0108				13	.0140	.0155			
						14.5	.0126	.0135			
						16	.0115	.0119			
						17.7	.0107	.0108			

^aSoaking for 1.3 min followed by 10.4 min in countercurrent.

^bSoaking for 9 min followed by 2.7 min in countercurrent.

^cSoaking for 1.8 min followed by 15.9 min in countercurrent.

^dSoaking for 1 min followed by 9 min in countercurrent.

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