Technical

Design of Oilseed Extractors. I. Oil Extraction

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A calculation method is presented that predicts, from laboratory data, retention time and miscella concentrations in commercial oil extractors. The method is based on the finding that rate of solution of oil largely determines retention time, and that resistance to diffusion of oil at the boundaries of the flakes is relatively small.

Continuous extraction of oil from seeds was first practiced in Europe in the early 1920's, and in this country about 1936. Design of early extractors was based on just about any conceivable method of bringing particulates and solvent into countercurrent contact in a vapor-tight apparatus. These methods can be classified as immersion, where the particulates are moved through the solvent as by a screw conveyor, or percolation, where the solvent drains by gravity through moving beds of particulates. The superiority of the percolation method was established early when, in the late 1930's, Central Soya, Archer Daniels Midland and Procter and Gamble jointly studied German oilseed extraction practice and chose for their first plants the Hansemuhle vertical basket extractor. Extractor design is still based largely on experience.

Consideration of a calculation basis for designing commercial solvent extractors for recovering oil from oilseeds is somewhat academic, because retention time needed for the countercurrent extraction of soybeans is less than 1.1 times the retention time required in the laboratory to reach the desired residual oil content, using a method such as that of Wingard and Shand (1). The multiplier for slower-extracting seeds, such as cottonseed and rapeseed, is even smaller. Nevertheless, plant operators should find useful a method that predicts the distribution of miscella concentrations in a commercial extractor. When they are experiencing mysterious operation problems, comparing their distribution with that predicted may give the clue to the source of trouble.

From the standpoint of extraction, vegetable oils may be regarded as a single component, because all of the glycerides are strongly soluble in hexane. The only other components that are extracted in any amount, phosphatides, have limited solubility. Since phosphatides in the cell are located at interfaces, they block access of hexane to the oil, making the extraction slow compared with washing. Extracted flakes into which oil is reintroduced extract much more rapidly the second time. The apparent slow solubility of oil to be discussed is undoubtedly the consequence of slow solution of the phosphatides.

EXTRACTION OF OIL IN THE LABORATORY

Extensive laboratory extractions were performed by King (2) and Coats (3), using solvents and their miscellas of several concentrations. In these experiments a small batch of flakes was extracted with a large excess of solvent or miscella, so that the oil concentration of the extraction solvent was zero or the oil content of the extraction miscella was constant during the entire extraction.

The results were best correlated by plotting "undissolved oil" vs time, as shown in Figure 1 (4). "Undissolved oil" was defined as the oil content of the partly extracted flakes minus the calculated oil in the miscella held up in the flakes, assuming that the miscella in the flakes had the same oil concentration as the miscella or solvent used for extraction. It was found that "undissolved oil" depended only on extraction time, and was independent of miscella concentration. Consequently, oil should go into solution at the same rate in a countercurrent extractor as in the laboratory. This explains the low multiplier.

The purpose of this paper is to broaden the "undissolved oil" concept to include the effect of resistance to diffusion of oil from the miscella held in the flakes into the miscella surrounding the flakes, and to apply the result to design of commercial extractors.

Figure 2 shows schematically a flake immersed in solvent or a weak miscella whose constant concentration is a. The liquid in the flake comprises undissolved oil and miscella phases. Total liquid volume in the flakes is a con-

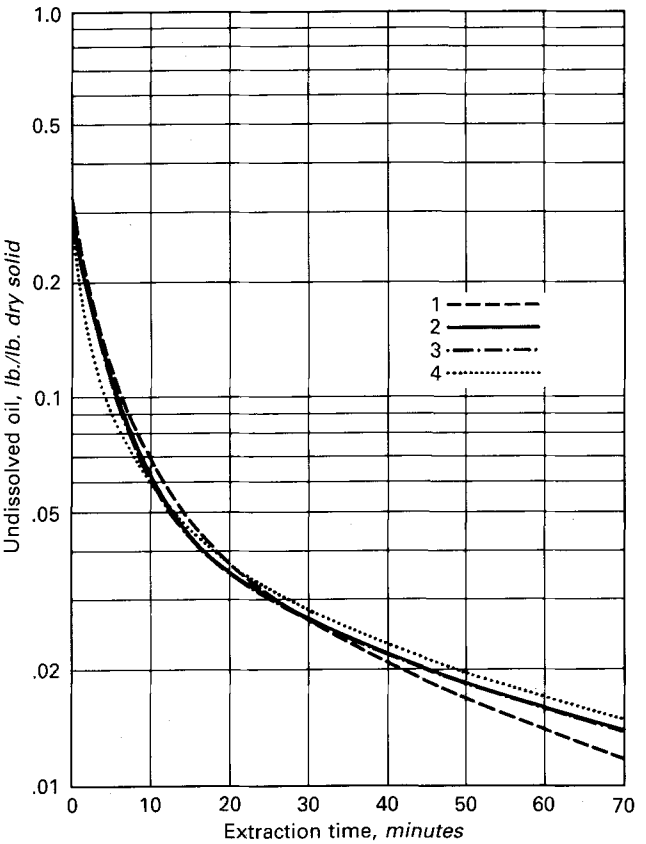

FIG. 1. "Undissolved oil" vs. extraction time (4) at several miscella concentrations. Data of King (2). Curve 1, 15.3% oil; curve 2, 10.4% oil; curve 3, 5.2% oil; curve 4, 0.3% oil.

stant H, designated the holdup. For soybeans, H = 0.788 l/kg meats (oil-free moisture-free flakes). The miscella phase is designated "miscella in the holdup"; its oil concentration is y vol oil/vol miscella. The concentration of undissolved oil in the holdup is z vol oil/vol holdup. Oil is extracted into a miscella whose oil concentration is constant, a vol oil/vol miscella. Then y(1 − z) is the volume of dissolved oil/vol holdup, and

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

where r is the residual oil concentration (derived from extraction rate data), vol oil/vol holdup. Note that all concentrations are volumetric, since extraction is from a constant volume of holdup.

Equation 1 may be solved for z:

$$z = (r - y)/(1 - y) \quad [2]$$

Since oil dissolves throughout each flake into miscella in the holdup, it is reasonable that y is uniform throughout that miscella and the extraction rate may be expressed as derived in Figure 2:

$$dr = -k(y - a)dt \quad [3]$$

where k is a rate constant whose dimension is minutes^{-1}. But dr/dt is the slope (negative) of the extraction rate curve, so (y − a) is simply the slope divided by −k. There is no way of estimating k. To illustrate the calculation method, k = 10 will be used, because it gives plausible numbers for y and z.

The purpose of the calculation outlined in Table 1 is to find z vs t for application to the calculation of continuous extraction, in which z is the same function of t. This is the recommended procedure:

- Plot r vs t, from an extraction rate experiment, on a scale large enough that the values of r read from the smooth curve that best fits the data can be read to three significant figures. Exemplifying this, residual oils read from curve 1 of Figure 4, derived from an extraction rate experiment using 0.20 mm thick soybean flakes, were converted to volumes; readings of r at suitable time increments were recorded in Table 1, down to r corresponding to the residual oil specified for the commercial product, say 0.5 wt. %, equivalent to r = 0.008, v/v.
- Record in Table 1 dt and dr.
- Calculate dr/dt and divide by −k to get y.
- Calculate z from Equation 2 and interpolate as in Table 1.

TABLE 1

Tabulated Calculations for Batch Laboratory and Continuous Immersion and Percolation Extractions

		Laboratory				Immersion			Percolation		
t	dt	r	−dr	y	z	R	Y	X	R	Y	X
0		.408		0.158	.297	.512	.306	.176	.454	.223	.077
0.05	0.1	.342	.134	.134	.240						
0.1		.274			.206	.389	.230	.133			
0.15	0.1	.236	.078	.078	.171						
0.2		.196			.155	.298	.169	.101			
0.3	0.2	.158	.078	.039	.124					Cocurrent	
0.4		.118			.107	.189	.092	.063			
0.5	0.2	.104	.029	.0145	.091						
0.6		.0890			.083	.138	.060	.045			
0.75	0.3	.0781	.0218	.0073	.071						
0.9		0.672			.063	.100	.039	.032	.229	.177	.077
1.05	0.3	.0597	.0150	.0050	.055						
1.2		.0522			.050	.078	.029	.024	.093	.045	.030
1.4	0.4	.0473	.0098	.0030	.044						
1.6		.0424			.040	.059	.020	.018	.061	0.22	.0185
1.85	0.5	.0377	.0094	.0019	.036						
2.1		.0330			.031	.048	.0175	.014	.048	.0175	.014
2.4	0.6	.0291	.0078	.0013	.028						
2.7		.0252			.025	.035	.0102	.0094	.035	.0102	.0094
3.05	0.7	.0210	.0071	.0010	.021						
3.4		.0181			.0187	.025	.0095	.0059	.025	.0095	.0059
3.8	0.8	.0165	.0033	.00041	.0161						
4.2		.0148			.0145	.018	.0036	.0035	.018	.0036	.0035
4.65	0.9	.0133	.0029	.00032	.0130						
5.1		.0119			.0115	.0145	.0030	.0023	.0145	.0030	.0023
5.6	1.0	.0180	.0022	.00022	.0106						
6.1		.0097			.0095	.0106	.0011	.0009	.0106	.0011	.0009
6.6	1.0	.0091	.0014	.00014	.0090						
7.1		.0083			.0082	.0089	.0006	.0003	.0088	.0006	.0003
7.35	0.5	.0080	.0005	.00010	.0079						
7.6		.0078			.0077	.0078	.0001	.0001	.0078	.0001	.0001
7.7		.0077			.0076						
7.8		.0076			.0075						

Time t in min, all other terms dimensionless.

The values of y in Table 1 demonstrate again that the rate of dissolution of oil controls extraction rate. (After 5.0 min of extraction, y is only 2% of r.) For the soybeans whose extraction rate is represented by curve 1 of Figure 4, at least, k cannot go much below 5 without having the values of z, calculated as in Table 1, go through a minimum, which is impossible. Even when k = 5, y is still only 4% of r after 5.0 min of extraction.

The residual oil data of curve 1 of Figure 4 was derived from an experiment in which hexane (a = 0) was the solvent. Suppose the initial value of y was y_0. Let the same soybeans be extracted with a miscella of concentration a. The initial concentration of the miscella in the holdup is $y_0 + a(1 - y_0)$. The initial driving force for extraction is therefore $y_0 + a(1 - y_0) - a = y_0(1 - a)$, compared with y_0 when hexane is the solvent. Close examination of Figure 1 will indeed show that initial rate of extraction was greatest for a = 0.3% and least for a = 15.3%. However, the initial additional driving force does not persist, since the rate of dissolution of oil quickly becomes controlling, and y adjusts itself so that in each extraction y − a is the same function of t.

CONTINUOUS EXTRACTION

To design a continuous extractor, a rate equation is combined with an oil volume balance. Typical balances for soybeans are shown in Figure 3, one for a countercurrent immersion extractor, the other for a percolation extractor. The balances are based on 100 kg meats from soybeans containing 20% oil and 10% water, extracted to 0.5% residual oil with hexane at 1:1 solvent to flakes ratio. The only new parameter is X, the concentration of oil in the miscella surrounding the flakes at the section of the extractor where the flakes retention time is t. R and Y replace r and y to distinguish from laboratory extraction.

In percolation extraction the flakes are slurried with miscella from a section of the extractor downstream in the direction of flakes flow. As shown in Figure 3, there is a cocurrent zone, preceding the countercurrent zone, in which Y decreases with time, and approaches equality with X, which increases to 0.176 in the full miscella.

For all zones the rate equation is:

$$dR = -k(Y - X)dt \quad [4]$$

DESIGN OF THE IMMERSION EXTRACTOR

The volumetric oil balance from Figure 3 is:

$$0.779R = 2.23X + 0.006 \quad [5]$$

or

$$Y - X = Y - 0.3492R + 0.0028 \quad [6]$$

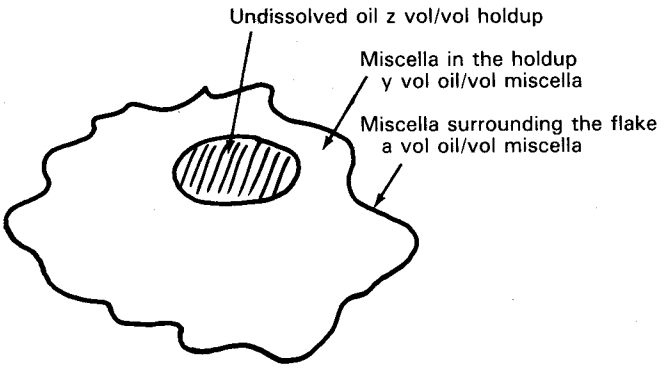

FIG. 2. Schematic flake with derivation of extraction rate equation. Let r be the total oil in the flake, vol oil/vol holdup. ∴ r = y(1 − z). For 1 kg flakes, oil content is 0.788r l. In time dt: 0.788dr = $-k_1(y - a)dt$ where k_1 is a constant whose dimensions are l/kg min^{-1}. Let $k = k_1/0.788$. ∴ $dr = -k(y - a)dt$.

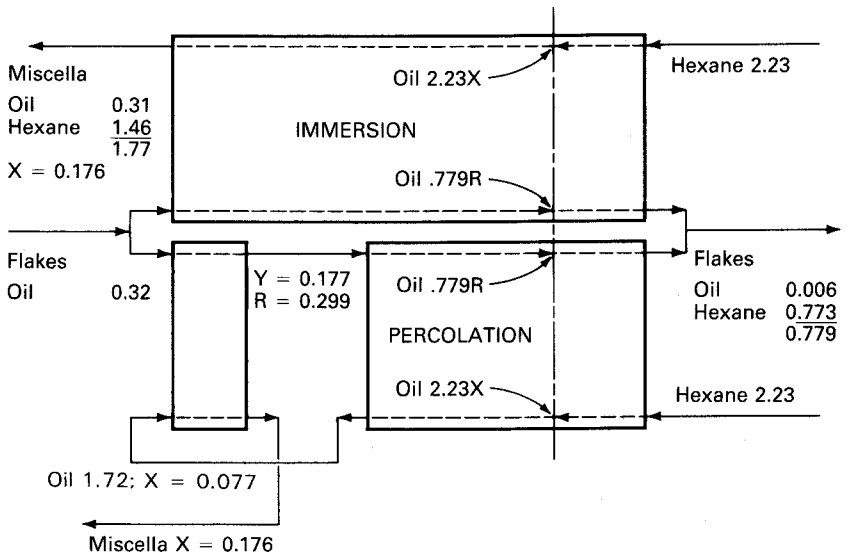

FIG. 3. Oil and hexane volumes balance in continuous extraction. Basis, 100 kg meats. Volumes in liters.

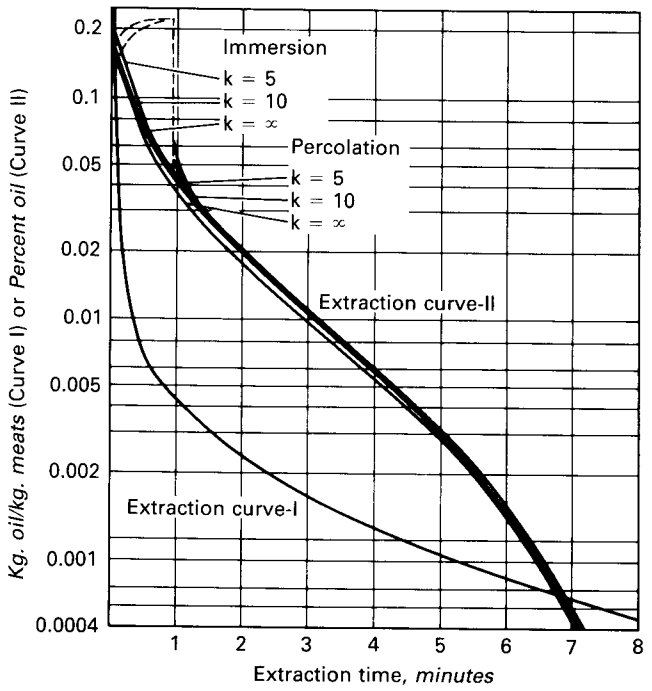

FIG. 4. Curve 1, residual oil vs. extraction time from batch laboratory extraction of 0.008″ thick soybean flakes with hexane. Curve 2, predicted miscella concentrations vs. extraction time in continuous extractors.

Equation 1 may be solved for Y:

$$Y = (R - z)/(1 - z) \qquad [7]$$

Combining Equations 4, 6 and 7 gives:

$$(R - z)/(1 - z) = 0.3492R - 1/k\ dR/dt - 0.0028 \qquad [8]$$

For calculation by iteration, let $dR = R_2 - R_1$ and $R = (R_1 + R_2)/2$. Substituting in Equation 8 gives:

$$R_2 = \frac{z/(1 - z) + 2R_1/kdt - 0.0028}{1/2(1 - z) + 1/kdt - 0.1746} - R_1 \qquad [9]$$

Starting at t = 0, apply Equation 9 repeatedly to get the values of R in Table 1. Let the subscript 0 connote t = 0. As earlier demonstrated, $Y_0 = y_0 + 0.176\ (1 - Y_0) = 0.306$, and $R_0 = 0.306 + 0.297 - (.306)\ (.297) = 0.512$. Reading from Table 1, R reached 0.008 when t = 7.5 min, so the multiplier of the laboratory extraction time is 7.5/7.35 = 1.02. Values of X from Table 1 converted to wt % oil are plotted in curve 2 of Figure 4.

Values of k can range from 5 to infinity. Calculations not reported here were made for k = 5 and k = ∞. Calculated values of X converted to wt % oil are plotted in curve 2 of Figure 3 for comparison with k = 10. For k = 5, the multiplier of the laboratory extraction time is 1.04; for k = ∞ the multiplier is 1.0. The curve for k = ∞ cannot be found by iteration, since the term 1/k dR/dt in Equation 8 is zero. However, it is readily understood that when k = ∞, y = 0, z = r, and Y = X. (These are the assumptions of the "undissolved oil" concept.) Hence, R = Y + z − Yz = X + r − Xr. Combining this with Equation 5 gives:

$$X = \frac{r - 0.0080}{1.864 + r} \qquad [10]$$

All extractors extracting these flakes at this solvent ratio should have miscella concentrations lying in the narrow band between k = 5 and k = ∞. Since the band is narrow, it is not possible to determine k from plant data for miscella concentration vs. extraction time. It can now be concluded that for all practical purposes the "undissolved oil" concept (k = ∞) gives the distribution of miscella concentrations expressed by Equation 10, where r is derived from concentrations read from curve 1 of Figure 4. Retention time provided in designing a commercial extractor for soybeans should be at least 1.04 times the laboratory extraction time, to allow the possibility that k is 5.

DESIGN OF THE PERCOLATION EXTRACTOR

The equations derived for the immersion extractor apply also to the countercurrent zone of the percolation extractor. Although similar equations can be derived for the cocurrent zone, there is no need to do so. In the approximately one minute that the flakes are in the cocurrent zone of a percolation extractor, Y approaches X very closely. In this case, X in the miscella leaving the cocurrent zone is 0.176, so it is safe to assume that Y in the flakes entering the countercurrent zone is 0.177.

In Table 1 it was assumed that the retention time in the cocurrent zone is 0.9 min, so the countercurrent calculation begins with Y = 0.177, z = 0.063, hence R = 0.177 + 0.063 − (.063) (.177) = 0.229, compared with R = 0.100 at 0.9 min in the immersion extractor. Nevertheless, R quickly catches up. After 2.1 min, the R's are identical. There is no penalty for cocurrency.

Values of X for percolation extraction from Table 1, converted to wt % oil, are plotted in curves 2 of Figure 4, as are the values of k = 5 and ∞. For k = ∞: at the entrance to the cocurrent zone Y = 0.176 and X = 0.078; but Y and X drop immediately to 0.040, and X then follows the curve for countercurrent extraction, Equation 10.

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