On the Extraction of Oil from Raw Comminuted Cottonseed Kernels with the Acetone-Hexane-Water Azeotrope[1]

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Abstract

The effects of particle size on the rate of extraction of oil from raw comminuted cottonseed kernels with the acetone-hexane-water azeotrope were examined. Five samples of cottonseed of different average particle sizes and three extraction times were used in the investigation. The particles were comminuted in such a manner as to be, as a first approximation, essentially spherical in shape; the concentration gradient of oil in the particles was found to follow the relationship $L = \dfrac{-A}{r} + B$ where L is the oil content of the extracted seed, r is the particle radius, and A and B are constants. The establishment of the concentration gradient was extremely rapid (within 10 seconds), and the constants A and B assume the same values for each of the three extraction times employed. These two facts indicate that mass oil movement in the particles is a dilatation and occurs too rapidly for diffusion phenomena to be evident.

Introduction

EVIDENCE HAS BEEN REPORTED (1) that the counter-current extraction of oil from raw cottonseed and soybean flakes by the ternary azeotrope formed by acetone, hexane, and water is a washing extraction, that the oil is rendered rapidly, and that the extraction follows the dilution law. A further study of the phenomenon of oil extraction by the azeotrope is reported here.

Experimental Section

Cottonseed kernels were comminuted in a Waring Blendor, and the resulting material was classified according to size by means of a series of standard screens, with openings of 0.0550, 0.033, 0.023, 0.0165, 0.0117, and 0.0098 in.

A 20-g aliquot of the particles that passed through a 14-mesh screen and were retained on a 20-mesh screen was weighed into the basket of a basket centrifuge, together with 60 g of the azeotrope. The contents of the bowl were commixed gently for 10 sec, then the marc and miscella were separated centrifugally. The miscella was decanted, and the oil content of the air-dried marc was determined in accordance with the method of the American Oil Chemists' Society (2). The experiment was repeated; a second aliquot of the comminuted kernels was commixed with the azeotrope for 20 sec before the marc and miscella were separated centrifugally. This same pattern was followed in extracting aliquots of each size class for 10, 20, and 50 sec.

The data obtained in the series of extractions are reported in Table I. Class size was taken as the median between the openings of two screens used in successive screenings of the comminuted material.

TABLE I

Oil Contents of Marcs Prepared on Exposure of Raw Comminuted Cottonseed Kernels to the Acetone-Hexane-Water Azeotrope

Mean[a] particle size in inches	Percentage of oil content of marc after exposure for		
	10 sec	20 sec	50 sec
0.044	24.2	23.0	24.3
0.028	22.2	20.9	21.7
0.020	20.6	19.4	19.3
0.014	17.9	16.9	16.1
0.011	15.0	14.4	14.3

[a] Corresponding approximately to an average value diameter of the particles.

Thus particles in the 0.044-in. class were those particles that passed a 14-mesh screen but were retained on a 20-mesh screen; those in the 0.028-in. class were the particles that passed a 20-mesh screen but were held back on a 30-mesh screen, etc.

Discussion

Comminuted cottonseed kernels were separated into size classes so that analysis of oil contents for each particle size for each time interval would supply integrated values for oil content over the time intervals of 0–10, 0–20, and 0–50 sec and over mean radii of 0.0055 to 0.022 in. The data should provide for a description of the flow of oil from the comminuted kernels during the first minute of oil extraction.

Two assumptions were made in the analyses of the data, namely, the particles could be treated, as a first approximation, as spheres; the oil distribution within the particles was initially homogeneous so that the only possible mass movement of oil would be radial.

It can be shown, on the basis of these two assumptions, that the time rate of change in oil content of an element of kernel volume $dV = 4\pi r^2 dr$ is described by the relationship

$$r^2 \frac{d^2L}{dr^2} + 2r \frac{dL}{dr} = \frac{1}{4\pi} \frac{dL}{dt} \qquad [1]$$

where L is the percentage of oil, r is the radius of the particle, and t is the time. Actually this is a general expression of Fick's law of diffusion in polar coordinates.

Inspection of the data in Table I reveals that the time rate of change in the oil content of the particles in the time interval of 10–50 sec, which was selected for this investigation, is small. The homogeneity of the regression coefficients for these data was established through an analysis of covariance, and the regression coefficient for the pooled data was determined to be −0.005. This is the time rate of change in the oil content of the particles where the time is expressed in seconds (cf. data in Table I). This value is considered to be negligibly small, and therefore, as a first approximation, the equation [1] may be written as

$$r^2 \frac{d^2L}{dr^2} + 2r \frac{dL}{dr} = 0 \qquad [2]$$

TABLE II
Regression Analyses and Test of Homogeneity of Regression Coefficients

Sample	Corrected sum of squares of oil contents	Corrected sum of products	Corrected sum of squares for 1/d	Regression coefficient	Sum of squares due to regression	DF	Sum of squares about regression	DF	F ratio
10 sec	52.25	−395.14	2994.8	−0.1319	51.13	1	0.12	3	1278
20 sec	45.13	−367.79	2994.8	−0.1228	45.17	1	0.14	3	969
50 sec	65.88	−440.49	2994.8	−0.1470	64.69	1	1.19	3	163
Sum	163.44	−1203.42	8988.4		161.99	3	1.45	9	
Pooled				−0.1339	160.82	1			
					1.17	2			

This is Laplace's equation in polar coordinates. A solution to this equation is

$$L = -\frac{A}{r} + B \qquad [3]$$

which may be verified by simple differentiation.

A and B are constants of integration, and their values are determined by the boundary conditions of the experiment.

The requirement of equation [3] is that the oil content of the particles be proportional to $\frac{1}{r}$. The data plotted in Figure 1 indicate that the oil content
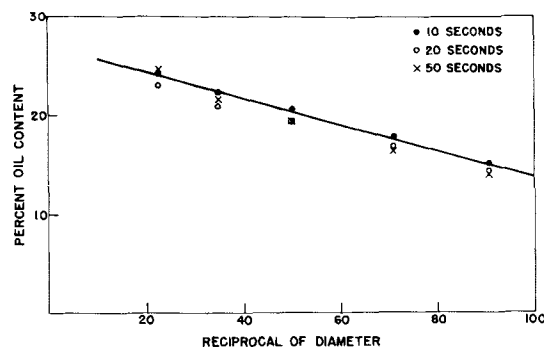

Fig. 1. Relationship between the reciprocal of the diameter and the oil content of the meal particles.

of the kernel particles is indeed proportional to $\frac{1}{r}$

Regression analyses were carried out with the data to determine the degree_of_linearity between $\frac{1}{r}$ and the oil content and to determine the homogeneity of the regression coefficients obtained for each time interval. The analyses are shown in Table II. The ratios of the mean square due to the regression to that about the regression for the time intervals 10, 20, and 50 sec were found to be 1278, 969, and 163 respectively. These F ratios for 1 and 3 degrees of freedom are highly significant, and it is obvious that the deviation from linearity for each of the curves is insignificant. The F ratio of $\dfrac{\frac{1.17}{2}}{\frac{1.45}{9}} = 3.6$ in the test

for homogeneity of the regression coefficients for 2 and 9 degrees of freedom is not considered to be significant, and it is concluded that there are no statistical differences in the slopes of the curves obtained for the different time intervals, that is, A has the same numerical value for each of the curves.

The evidence deduced from these analyses seems conclusive that the oil movement in comminuted cottonseed kernels in contact with the azeotrope during the initial contact must be described as a dilatation, and the space distribution of oil within the particles is similar to the space distribution of the gravity potential in space about a celestial body. The establishment of the oil concentration gradient described by equation [3] is very rapid; it is established within a period of about 10 sec.

The phenomenon may possibly be accounted for on the basis of imbibition forces that are generated because of a rapid penetration of the solvent into the cells of the kernel particles, with the subsequently rapid expulsion of oil-rich miscella. The cells on the surface of the particle receive the full impact of the solvent, but the cells in the second layer are contacted with the solvent that has been diluted with oil from the first layer. The cells in the third layer are contacted with the solvent that carried oil from the first two layers, etc. Thus the oil concentration gradient is established, with the oil concentration increasing as one approaches the center of the particle.

Diffusion phenomena are not evident in these first seconds of contact between the kernel fragments and the solvent. The space distribution of oil within the speed particles at any selected period of time would be a function of e^{-kr^2}, where e is the Napierian constant, if the space distribution of oil were due to diffusion processes. There is no evidence that the oil distribution is a function of e^{-kr^2}. Obviously the concentration gradient within the particles occurs too rapidly to be accounted for on the basis of diffusion.

REFERENCES

1. "Countercurrent Extraction of Raw Cottonseed Flakes with the Acetone-Hexane-Water Azeotrope," by V. L. Frampton, A. B. Pepperman, Jr., Joseph Simmons, and W. H. King, presented at Southwestern Regional Meeting, Am. Chem. Soc., November 29—December 2, 1966.
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