

THE SOLVENT EXTRACTION OF WAX FROM SORGHUM GRAIN BRAN

by

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INTRODUCTION

It was the purpose of this project¹ to obtain experimental data on the extraction of wax from the bran of sorghum grain and to devise a method of predicting the extraction. Rate and equilibrium relations must be considered for the competent design of extraction equipment. These relationships govern the economical operation and determine the capacity of such equipment.

In order to determine the decrease in wax content with extraction time for the bran of sorghum grain, it was necessary to observe, in batch extractions, the increase in wax content in the wax-solvent solution (miscella). Bran of various screen sizes was extracted at several temperatures and solvent-bran ratios. Separation of the wax from the miscella was effected by crystallizing and filtering at a low temperature. The general extraction rate curves derived provided a method of attacking batch or continuous process design problems.

PREVIOUS WORK

Foveaux (1) has presented operating data for a continuous countercurrent whole grain solvent extraction pilot plant; Trichloroethylene was used as the solvent. It was reported that the maximum amount of wax extracted at 100 to 142° F. was 0.28 percent of the whole grain. Melting points ranged from 74° C. to 77° C. Foveaux discussed the advantages of extracting the

¹This work was performed under the auspices of The Kansas Industrial Development Commission.

wax from the bran or hull fraction. Much less weight of the grain would have to be handled. The grain would be subjected to a dry milling process wherein the grain would be separated into its component parts; the hull, the endosperm, and germ. This would result in a great saving of labor, smaller scale equipment, less solvent loss, and would eliminate the possibility of damage to the starch due to action of the solvent. It was stated that the method awaited a machine which would remove the hull cleanly.

MATERIALS

The whole sorghum grain, the Westland milo variety, was de-branned by an abrasive type machine in the laboratory of the Department of Chemical Engineering at Kansas State College. In order to effect a size separation, the bran was screened in specially designed shaker fitted with screens similar to the Tyler type. In this manner, individual batches of bran of larger than 16, 16 to 20, and smaller than 20-mesh screen size were obtained.

Technical grade trichloroethylene, C_2HCl_3 , which was used, contained a stabilizer to avoid possible decomposition by oxidation in the presence of air. Trichloroethylene meets most of the requirements for a commercial solvent. These requirements include solubility for extracted wax, low cost, chemical stability, volatility, noninflammability, and freedom from corrosion and toxicity.

EQUIPMENT AND PROCEDURE

The batch extraction equipment comprised a water tank, heating coils, agitators, and a thermostat for controlling the temperature of the bran-solvent mixtures. A bran sample was placed in a 350 cc flask containing solvent at the bath temperature. Solvent-bran mixtures in ratios of 6, 10, and 15 parts solvent to one part bran were maintained at temperatures of 68°, 86°, 104°, and 122° Fahrenheit. The predetermined time periods were 30 seconds, 5, 15, and 30 minutes, 1, 2, and 8 hours. Samples were prepared for initial wax content determinations and extracted several times with hot solvent.

At the end of the extraction period the bran-solvent mixture sample was withdrawn and filtered in a Buchner funnel under a slight vacuum. The wax content of the miscella was determined by crystallizing and filtering a miscella sample at -30° Fahrenheit.

In a refrigerator capable of maintaining a temperature of -30° Fahrenheit, a bank of four Buchner flasks was placed. The flasks were attached to a water aspirator and stoppered with Gooch filters and their holders. After allowing two hours for crystallization of the wax in the miscella, 30 cc miscella samples were filtered. All equipment was precooled before use.

The wax samples were dried at 130° Fahrenheit under a vacuum of 29 inches of mercury for two hours. In none of the determinations did the amount of wax collected on a Gooch filter

exceed 200 milligrams. The specific gravity of trichloroethylene at -30° Fahrenheit, as reported by Mellan (2), is 1.555. The highest concentration of wax in the miscella was, therefore, 0.43 percent.

The wax obtained by the above method was quite hard, and had a yellow-orange cast. Preliminary solubility determinations indicated that, within the experimental error of about 5 percent, the wax was insoluble in trichloroethylene at -30° Fahrenheit.

The low temperature crystallization and filtration method was established after investigating other possible methods of removing the wax and determining the concentration in the miscella. The problem was complicated by the presence of extracted oil in the miscella. It was found that three to four times as much oil as wax was extracted from the bran. A differential cloud point method was devised and tested without success. The success of methods utilizing specific gravity, refractive index, and colorimetric measurements was deemed improbable because of the complexity of the miscella.

The wax content of the bran at infinite extraction time or in equilibrium with the miscella was required. With the wax concentration of the miscella, s , the ratio of solvent to bran, R , and the initial wax content of the bran, W_0 , expressed as pounds wax per pound of wax-free bran, known the extent of the extraction was calculated and expressed as pounds wax extracted per pound of wax-free bran, w' .

$$w' = R(1 + W_0)s \quad (1)$$

EXPERIMENTAL RESULTS

The sorghum grain bran extraction data are given in Table 1. Graphs of the results are presented in Figs. 1, 2, 3, and 4. The equilibrium wax content of the bran is a function of the wax concentration of the miscella. Data necessary for the calculation of the equilibrium wax content values have been included in Table 1. Plots showing the distribution of wax between the bran and the solvent at equilibrium are given in Figs. 5, 6, and 7.

APPLICATION OF THE EXPERIMENTAL RESULTS

The General Extraction Equation

In the form presented the experimental results are not, in general, applicable to extraction calculation. In solvent extraction the difference, $W - W_e$, where W is the concentration of wax in the bran and W_e is the equilibrium concentration, is commonly known as the driving force. Consider a bran-solvent mixture at any instant. Extend the miscella without bound so that further extraction will not change the wax concentration in the miscella appreciably. When a steady state is attained, the wax concentration in the bran is the equilibrium concentration, W_e . W_e is clearly a function of the wax concentration in the miscella at any instant. It follows that, in batch extraction, W , W_e , and the driving force, $W - W_e$, all change with time. To bridge the gap between batch extraction data and general extraction rate curves applicable to both batch and continuous

Table 1. Solvent extraction data.

Run	Extraction fraction	Temp. °F.	Solvent bran ratio	Extraction time, hours	Wax from 50 cc miscella at -10° F., grams	Wax extracted, lbs. per lb. wax-free bran
1	F	68	6	0.0083	-	-
2	F	68	6	0.083	0.0514	0.0068
3	F	68	6	0.25	0.0733	0.0097
4	F	68	6	0.50	0.0884	0.0118
5	F	68	6	1.00	0.0907	0.0120
6	F	68	6	>1.00	0.0921	0.0122 ²
7	F	68	10	0.0083	0.0227	0.0050
8	F	68	10	0.083	0.0391	0.0086
9	F	68	10	0.25	0.0502	0.0111
10	F	68	10	0.50	0.0618	0.0136
11	F	68	10	1.00	0.0659	0.0145
12	F	68	10	>1.00	0.0668	0.0147 ²
13	F	68	15	0.0083	-	-
14	F	68	15	0.083	0.0284	0.0093 ²
15	F	68	15	0.25	0.0379	0.0124
16	F	68	15	0.50	0.0473	0.0155
17	F	68	15	1.00	0.0500	0.0164
18	F	68	15	>1.00	-	-
19	M	68	6	0.0083	0.0153	0.0020
20	M	68	6	0.083	0.0229	0.0030
21	M	68	6	0.25	0.0382	0.0050
22	M	68	6	0.50	0.0574	0.0075

Table 1. (cont.)

Run	frac- tion ¹	Temp. °F.	Sol- vent- bran ratio	Extrac- tion time, hours	Wax from 30 cc miscella at -30° F., grams	Wax extracted, lbs. per lb. wax-free bran
23	M	68	6	1.00	0.0666	0.0087 ²
24	M	68	6	1.00	-	-
25	M	68	10	0.0083	0.0115	0.0025
26	M	68	10	0.083	0.0161	0.0035
27	M	68	10	0.25	0.0275	0.0060
28	M	68	10	0.50	0.0376	0.0082
29	M	68	10	1.00	0.0459	0.0100
30	M	68	10	>1.00	-	-
31	M	68	15	0.0083	0.0098	0.0032
32	M	68	15	0.083	0.0123	0.0040
35	M	68	15	0.25	0.0208	0.0066 ²
34	M	68	15	0.50	0.0282	0.0092 ²
35	M	68	15	1.00	0.0337	0.0110
36	M	68	15	>1.00	-	-
37	C	68	6	0.0083	-	-
38	C	68	6	0.083	-	-
39	C	68	6	0.25	0.0231	0.0030
40	C	68	6	0.50	0.0369	0.0048
41	C	68	6	1.00	0.0446	0.0058
42	C	68	6	>1.00	0.0462	0.0060 ²
43	C	68	10	0.0083	0.0046	0.0010
44	C	68	10	0.083	0.0074	0.0016

Table 1. (cont.).

Run	: : Bran : fract : tion	: : Temp. : °F.	: Sol- : vent- : bran : ratio	: Extrac- : tion : time, : hours	: Wax from : 30 cc : miscella : et : -30° F., : grams	: Wax : extracted, : lbs. per : lb. : wax-free : bran
45	C	68	10	0.25	0.0171	0.0037
46	C	68	10	0.50	0.0263	0.0057
47	C	68	10	1.00	0.0323	0.0070
48	C	68	10	>1.00	-	
49	C	68	15	0.0083	0.0040	0.0013
50	C	68	15	0.083	0.0065	0.0021
51	C	68	15	0.25	0.0129	0.0042 ²
52	C	68	15	0.50	0.0194	0.0063
53	C	68	15	1.00	0.0246	0.0080 ²
54	C	68	15	>1.00	-	
55	F	86	6	0.0083	0.0938	0.0124
56	F	86	6	0.083	0.1232	0.0163
57	F	86	6	0.25	0.1390	0.0184
58	F	86	6	0.50	0.1490	0.0197
59	F	86	6	1.00	0.1496	0.0198 ²
60	F	86	10	0.0083	-	
61	F	86	10	0.083	0.0805	0.0177
62	F	86	10	0.25	0.0918	0.0202
63	F	86	10	0.50	0.0900	0.0198
64	F	86	10	1.00	0.0897	0.0197 ²
65	F	86	15	0.0083	0.0433	0.0142

Table 1. (cont.).

Run	: Bran : :frac ¹ : :tion :	Temp. : OF. :	Sol- : vent- : bran : ratio:	Extrac- : tion : time, : hours :	Wax from : 30 cc : miscella : at : -30 ^o F., : grams :	Wax : extracted, : lbs. per : lb. : wax-free : bran
66	F	86	15	0.083	0.0512	0.0185 ²
67	F	86	15	0.25	0.0631	0.0207 ²
68	F	86	15	0.50	0.0680	0.0223
69	F	86	15	1.00	0.0683	0.0224 ²
70	M	86	6	0.0083	0.0382	0.0050
71	M	86	6	0.083	0.0634	0.0083 ²
72	M	86	6	0.25	0.0780	0.0102
73	M	86	6	0.50	0.0864	0.0113
74	M	86	6	1.00	0.0887	0.0116
75	M	86	10	0.0083	-	-
76	M	86	10	0.083	0.0395	0.0086
77	M	86	10	0.25	0.0496	0.0108
78	M	86	10	0.50	0.0560	0.0122
79	M	86	10	1.00	0.0588	0.0128
80	M	86	15	0.0083	0.0193	0.0063
81	M	86	15	0.083	0.0300	0.0098 ²
82	M	86	15	0.25	0.0352	0.0115
83	M	86	15	0.50	0.0398	0.0130
84	M	86	15	1.00	0.0392	0.0128
85	C	86	6	0.0083	-	-
86	C	86	6	0.083	0.0254	0.0033

Table 1. (cont.).

Run	: : Bran : frac- : tion ¹ :	: : Temp. : °F. :	: : Sol- : vent- : bran : ratio:	: : Extrec- : tion : time, : hours	: : Wax from : 30 cc : miscella : at : -30° F., : grams	: : Wax : extracted, : lbs. per : lb. : wax-free : bran
87	C	86	6	0.25	0.0485	0.0063
88	C	86	6	0.50	0.0552	0.0072
89	C	86	6	1.00	0.0600	0.0078 ²
90	C	86	10	0.0083	0.0078	0.0017
91	C	86	10	0.083	0.0175	0.0038
92	C	86	10	0.25	0.0309	0.0067
93	C	86	10	0.50	0.0406	0.0088
94	C	86	10	1.00	-	-
95	C	86	15	0.0083	-	-
96	C	86	15	0.083	0.0129	0.0042 ²
97	C	86	15	0.25	0.0229	0.0071 ²
98	C	86	15	0.50	0.0277	0.0090
99	C	86	15	1.00	0.0296	0.0096 ²
100	F	104	6	0.0083	-	-
101	F	104	6	0.083	0.1573	0.0208
102	F	104	6	0.25	0.1746	0.0231
103	F	104	6	0.50	-	-
104	F	104	6	1.00	0.1746	0.0231 ²
105	F	104	10	0.0083	0.0610	0.0134
106	F	104	10	0.083	0.1023	0.0225
107	F	104	10	0.25	0.1092	0.0240

Table 1. (cont.).

Run	: Bran : frac- : tion ¹	: Temp. : °F.	: Sol- : vent- : bran : ratio	: Extrac- : tion : time : hours	: Wax from : : 30 cc : : miscella : : at : : -300 F., : : grams :	: Wax : extracted, : lbs. per : lb. : wax-free : bran
108	F	104	10	0.50	0.1105	0.0243
109	F	104	10	1.00	-	-
110	F	104	15	0.0083	0.1133	0.0150
111	F	104	15	0.083	0.1760	0.0233
112	F	104	15	0.25	0.1910	0.0253
113	F	104	15	0.50	0.1895	0.0251
114	F	104	15	1.00	0.1918	0.0254 ²
115	M	104	6	0.0083	0.0428	0.0056
116	M	104	6	0.083	0.0873	0.0114
117	M	104	6	0.25	0.0964	0.0126
118	M	104	6	0.50	-	-
119	M	104	6	1.00	0.0996	0.0130 ²
120	M	104	10	0.0083	0.0276	0.0060
121	M	104	10	0.083	0.0537	0.0117
122	M	104	10	0.25	0.0596	0.0130
123	M	104	10	0.50	-	-
124	M	104	10	1.00	0.0624	0.0136
125	M	104	15	0.0083	0.0248	0.0081
126	M	104	15	0.083	0.0383	0.0125
127	M	104	15	0.25	-	-
128	M	104	15	0.50	-	-

Table 1. (cont.).

Run	frac- tion ¹	Temp. °F.	Sol- vent- bran: ratio	Extrec- tion time, hours	Wax from: 30 cc miscella at -30° F., grams	Wax extracted, lbs. per lb. wax-free bran
129	M	104	15	1.00	0.0425	0.0139 ²
130	C	104	6	0.0083	0.0177	0.0023
131	C	104	6	0.083	0.0492	0.0064
132	C	104	6	0.25	0.0652	0.0085
133	C	104	6	0.50	0.0652	0.0085
134	C	104	6	1.00	0.0684	0.0089
135	C	104	10	0.0083	0.0083	0.0018
136	C	104	10	0.083	0.0351	0.0076
137	C	104	10	0.25	0.0411	0.0089
138	C	104	10	0.50	0.0448	0.0097
139	C	104	10	1.00	-	-
140	C	104	15	0.0083	0.0100	0.0032
141	C	104	15	0.083	0.0247	0.0080
142	C	104	15	0.25	0.0327	0.0103
143	C	104	15	0.50	0.0314	0.0102
144	C	104	15	1.00	-	-
145	F	122	6	0.0083	0.1263	0.0167
146	F	122	6	0.083	0.1836	0.0243
147	F	122	6	0.25	0.1875	0.0248
148	F	122	6	0.50	0.1822	0.0241
149	F	122	6	1.00	0.1865	0.0247 ²

Table 1. (cont.).

Run	: Bran : :frac- :tion ¹ :	: Temp. : °F. :	: Sol- : vent- : bran : ratio:	: Extrac- : tion : time, : hours	: Wax from : 30 cc : miscella : at : -30° F., : grams	: Wax : extracted, : lbs. per : lb. : wax-free : bran
150	F	122	10	0.0083	0.0791	0.0174
151	F	122	10	0.083	0.1133	0.0249
152	F	122	10	0.25	0.1155	0.0254
153	F	122	10	0.50	0.1160	0.0255
154	F	122	10	1.00	0.1161	0.0256 ²
155	F	122	15	0.0083	0.0589	0.0193
156	F	122	15	0.083	0.0784	0.0257 ²
157	F	122	15	0.25	0.0814	0.0267 ²
158	F	122	15	0.50	-	-
159	F	122	15	1.00	-	-
160	M	122	6	0.0083	0.0550	0.0072
161	M	122	6	0.083	0.0894	0.0118
162	M	122	6	0.25	0.0940	0.0123
163	M	122	6	0.50	-	-
164	M	122	6	1.00	0.1047	0.0137 ²
165	M	122	10	0.0083	0.0404	0.0088
166	M	122	10	0.083	0.0592	0.0129
167	M	122	10	0.25	0.0658	0.0139
168	M	122	10	0.50	0.0652	0.0142 ²
169	M	122	10	1.00	-	-
170	M	122	15	0.0083	0.0282	0.0092

Table 1. (cont.).

Run	: : Bran : fract : tion ¹ :	: : Temp. : of F. :	: : Sol- : vent- : bran : ratio:	: : Extrac- : tion : time, : hours	: : Wax from : 30 cc : miscella : at : -30° F., : grams	: : Wax : extracted, : lbs. per : lb. : wax-free : bran
171	M	122	15	0.083	0.0414	0.0133
172	M	122	15	0.25	0.0441	0.0144
173	M	122	15	0.50	-	
174	M	122	15	1.00	0.0435	0.0142 ²
175	C	122	6	0.0083	0.0185	0.0024
176	C	122	6	0.083	0.0600	0.0077
177	C	122	6	0.25	0.0746	0.0097
178	C	122	6	0.50	-	
179	C	122	6	1.00	0.0777	0.0101 ²
180	C	122	10	0.0083	0.0129	0.0028
181	C	122	10	0.083	0.0383	0.0083
182	C	122	10	0.25	0.0466	0.0101
183	C	122	10	0.50	-	-
184	C	122	10	1.00	0.0464	0.0105
185	C	122	15	0.0083	0.0154	0.0050
186	C	122	15	0.083	0.0268	0.0087
187	C	122	15	0.25	-	-
188	C	122	15	0.50	-	-
189	C	122	15	1.00	0.0329	0.0107 ²
190	F	175	-	-	-	0.0280 ²

Table 1. (concl.).

Run	frac- tion ¹	Temp. °F.	Sol- vent- bran ratio	Extrac- tion time, hours	Wax from : 30 cc miscella -30° F., grass	Wax extracted, lbs. per lb. wax-free bran
191	M	175	-	-	-	0.0152 ²
192	C	175	-	-	-	0.0111 ²

¹ F = smaller than 20 mesh.
M = 16 to 20 mesh.
C = larger than 16 mesh.

² Average of two or more determinations.

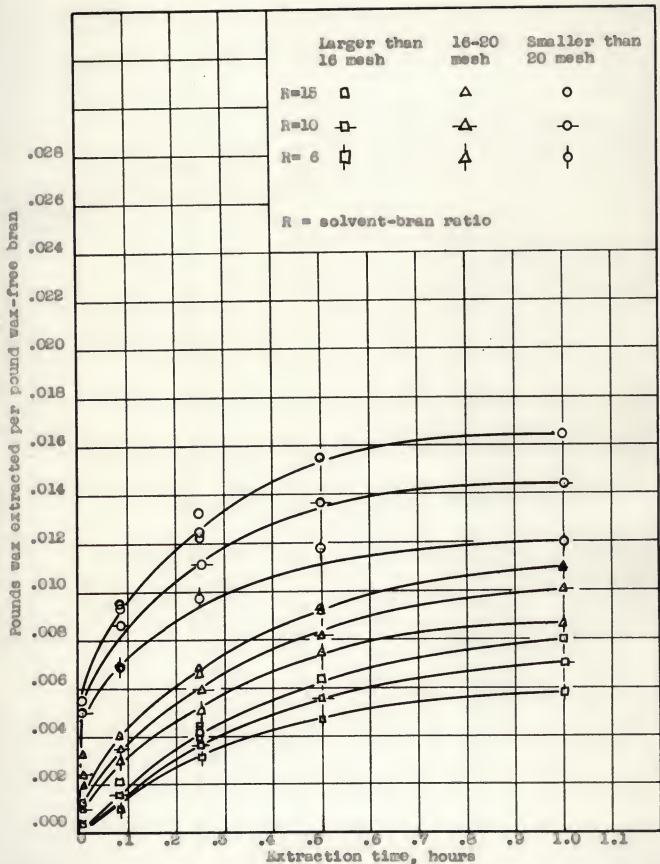


Fig. 1. Extraction curves for sorghum grain bran in trichloroethylene at 68° Fahrenheit.

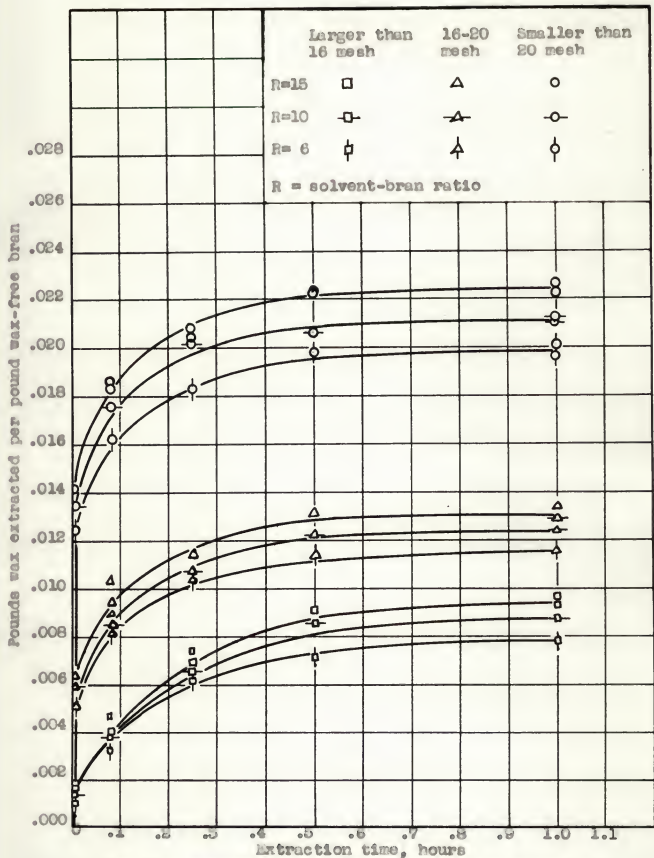


Fig. 2. Extraction curves for sorghum grain bran in trichloroethylene at 86° Fahrenheit.

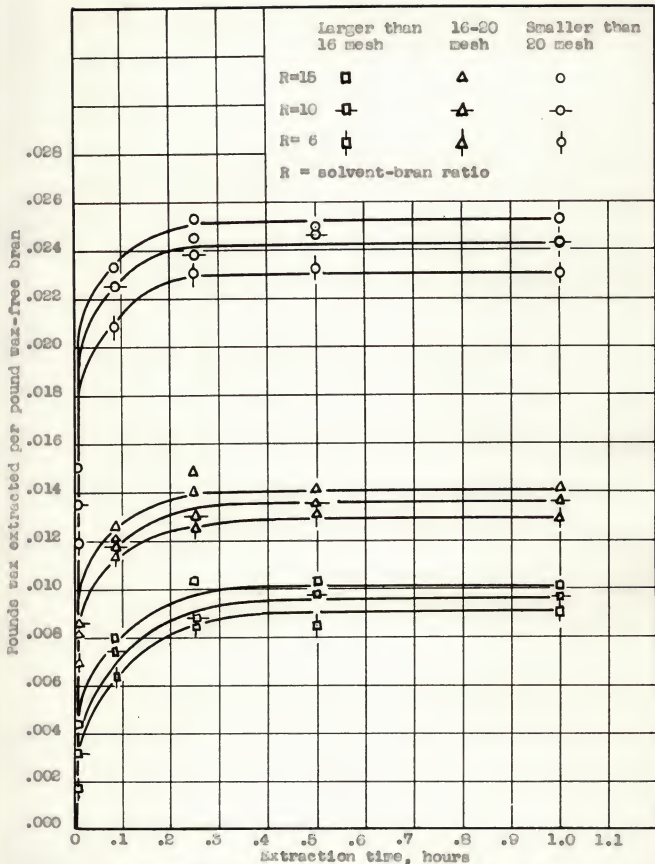


Fig. 3. extraction curves for sorghum grain bran in trichloroethylene at 104° Fahrenheit.

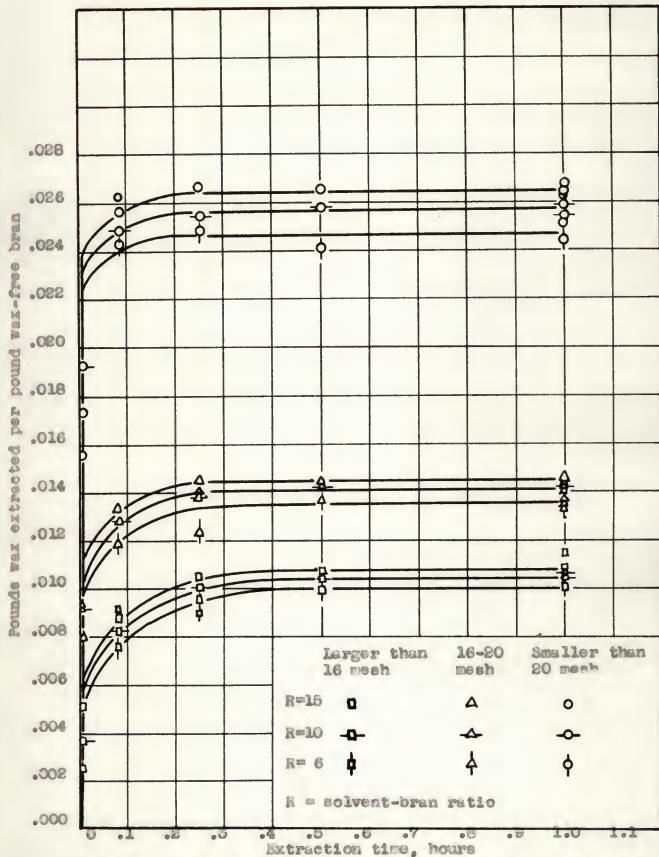


Fig. 4. Extraction curves for sorghum grain bran in trichloroethylene at 122° Fahrenheit.

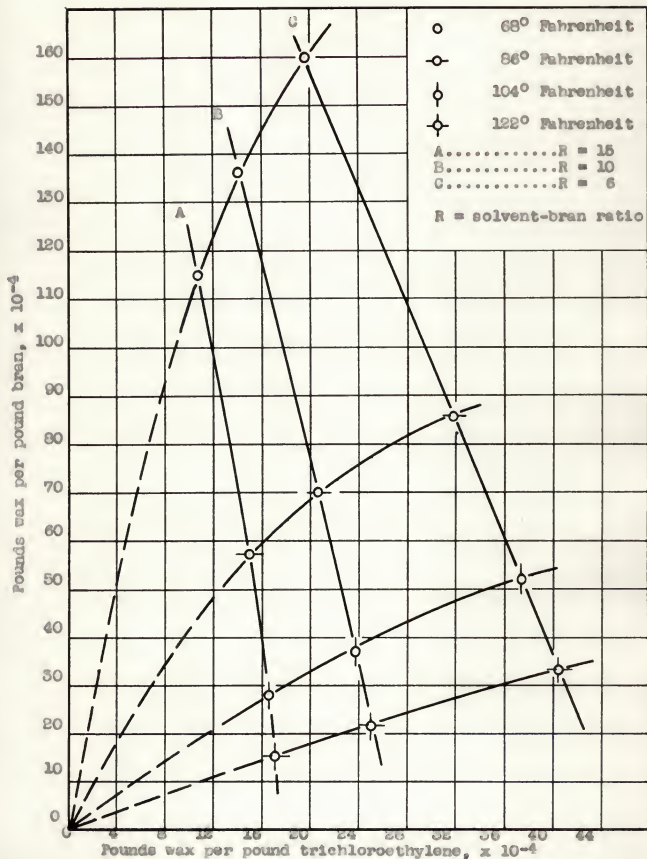


Fig. 5. Equilibrium distribution of wax between trichloroethylene and bran smaller than 20 mesh.

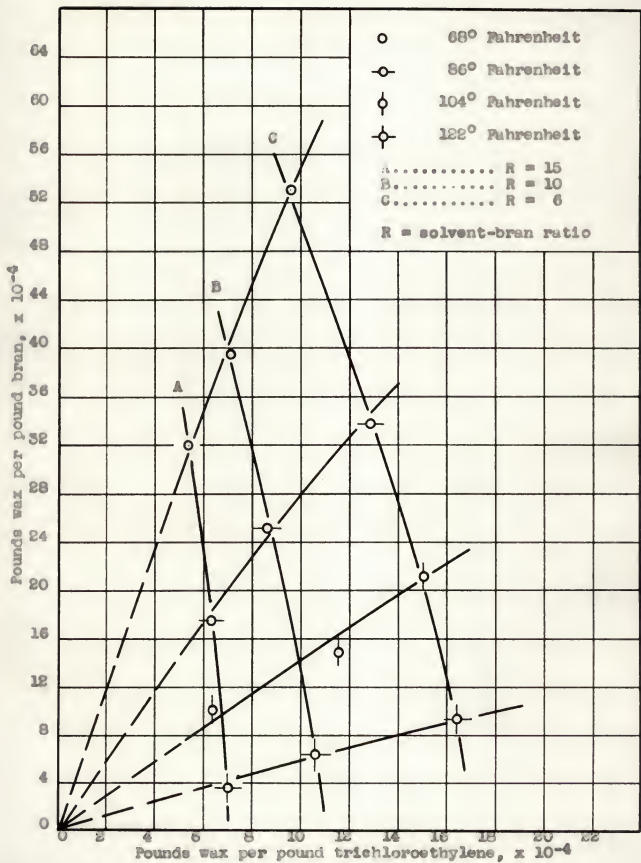


Fig. 6. Equilibrium distribution of wax between trichloroethylene and bran 16 to 20 mesh.

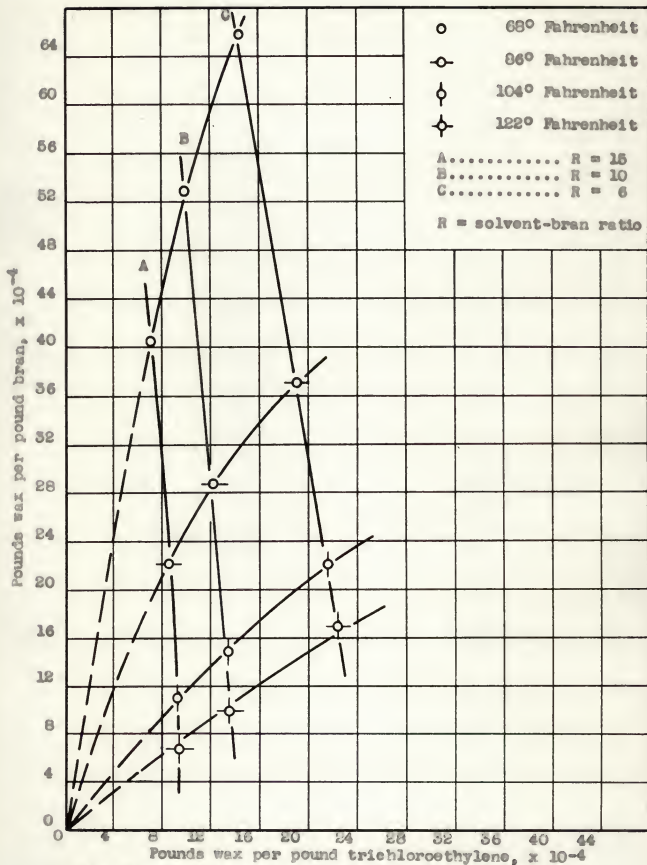


Fig. 7. Equilibrium distribution of wax between trichloroethylene and bran larger than 16 mesh.

extraction process problems, it is common procedure to assume that

$$-\frac{dW}{dt} = f(W - W_e) \quad (2)$$

obtaining dW/dt by graphical differentiation and determining the time of a specific extraction by evaluating

$$t = \int_{W_1}^{W_f} \frac{dW}{f(W - W_e)} \quad (3)$$

by graphical integration.

For the extraction of a bran fraction at a specific temperature, Figs. 1, 2, 3, and 4 show a distinct extraction curve for each of the three solvent-bran ratios. In order to apply the above method, each of these curves, 36 in all, would be differentiated graphically, an operation resulting in inaccurate slope values because of the difficulty in drawing representative curves through the points corresponding to the experimental data. Also, the values of $W - W_e$ would be average ones. A method considered more accurate was devised.

Sherwood (3) has given and King (4) has discussed a theoretical diffusion equation for the fraction of extractable solute, E , retained within an infinite slab of thickness, $2d$, as a function of the diffusion time, t , and diffusion constant, D . The equation is

$$E = \frac{W - W_e}{W_0 - W_e} = \frac{8}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{(2n-1)^2} \exp \left[-\frac{D(2n-1)^2 \pi^2 t}{(2d)^2} \right] \quad (4)$$

Other similar functions express diffusion within solids of various shapes.

Since the shape of the solid was incapable of description, the equation in its general form

$$E = \frac{W - W_e}{W_0 - W_e} = F(t) \quad (5)$$

was used.

The plots of E , the fraction of extractable wax, as a function of time are presented in Figs. 8, 9, and 10. It may be noted that the function, E , merges extraction curves for a particular bran fraction at a specific temperature into one curve.

The derivation of the general extraction curves required the graphical differentiation of E with respect to time. Remembering that, in general, both W and W_e change with time as extraction proceeds

$$E' = \frac{dE}{dt} = \frac{(W_0 - W_e) \left[\frac{dW}{dt} - \frac{dW_e}{dt} \right] + (W - W_e) \frac{dW_e}{dt}}{(W_0 - W_e)^2} \quad (6)$$

Simplifying,

$$E' = \frac{(W_0 - W_e) \frac{dW}{dt} - (W_0 - W) \frac{dW_e}{dt}}{(W_0 - W_e)^2} \quad (7)$$

In order to put the latter equation in a usable form, consider the operating curve, W , as a function of s , the wax concentration in the miscella. If the operating curve is a straight line,

$$W = m_0 s + b \quad (8)$$

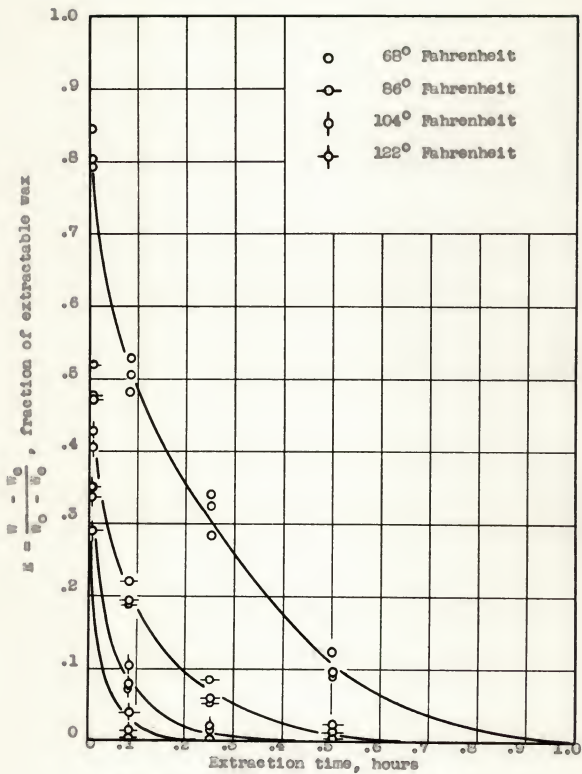


Fig. 8. K , fraction of extractable wax, versus extraction time.

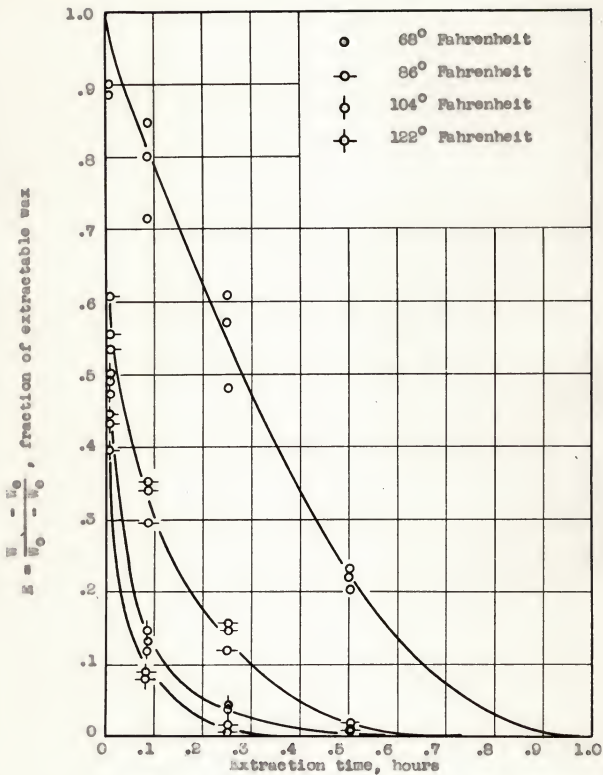


Fig. 9. E , fraction of extractable wax, versus extraction time.

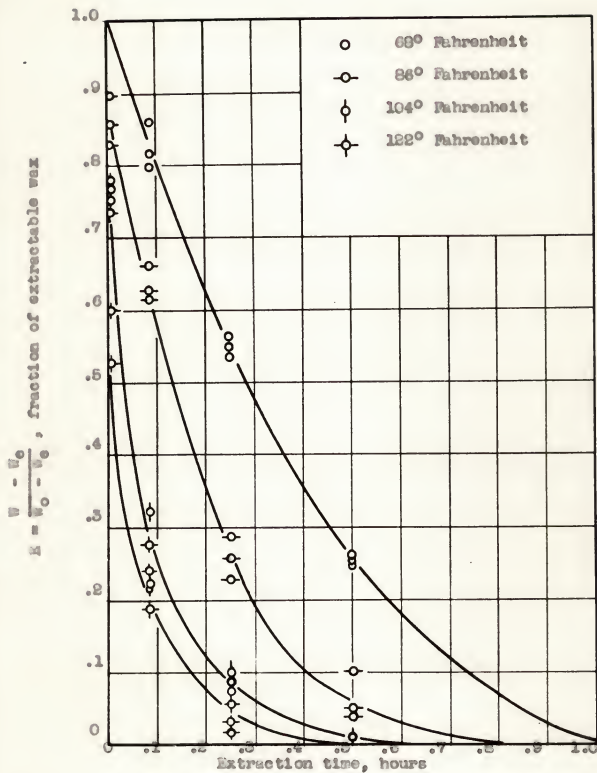


Fig. 10. K , fraction of extractable wax, versus extraction time.

Differentiating with respect to time,

$$\frac{dW}{dt} = m_o \frac{ds}{dt} = m_o \frac{ds}{dW_e} \frac{dW_e}{dt} \quad (9)$$

Rearranging,

$$\frac{dW_e}{dt} = \frac{1}{m_o} \frac{dW}{dt} \frac{dW_e}{ds} \quad (10)$$

The expression,

$$\frac{dW_e}{ds} = m_e \quad (11)$$

is the slope of the equilibrium curve. Rewriting equation (9),

$$\frac{dW_e}{dt} = \frac{m_e}{m_o} \frac{dW}{dt} \quad (12)$$

Substituting this result in equation (7), a usable form is obtained:

$$E' = \frac{(W_o - W_e) - (W_o - W) \frac{m_e}{m_o}}{(W_o - W_e)^2} \frac{dW}{dt} \quad (13)$$

Two operations are required before the latter equation can be applied to general extraction problems. The results of the first, a graphical differentiation of E with respect to time, were plotted versus E . These plots are given in Figs. 11, 12, and 13. Figures 14, 15, and 16 show plots of m_e , the slopes of the equilibrium curves, versus s .

For convenience, let

$$G = \frac{(W_o - W_e) - (W_o - W) \frac{m_e}{m_o}}{(W_o - W_e)^2} \quad (14)$$

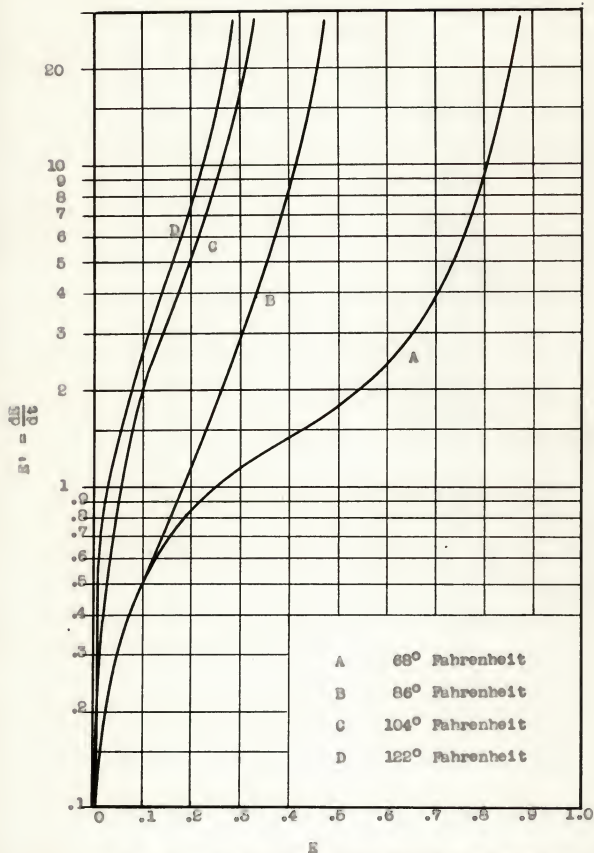


Fig. 11. E' versus E ; bran smaller than 20 mesh.

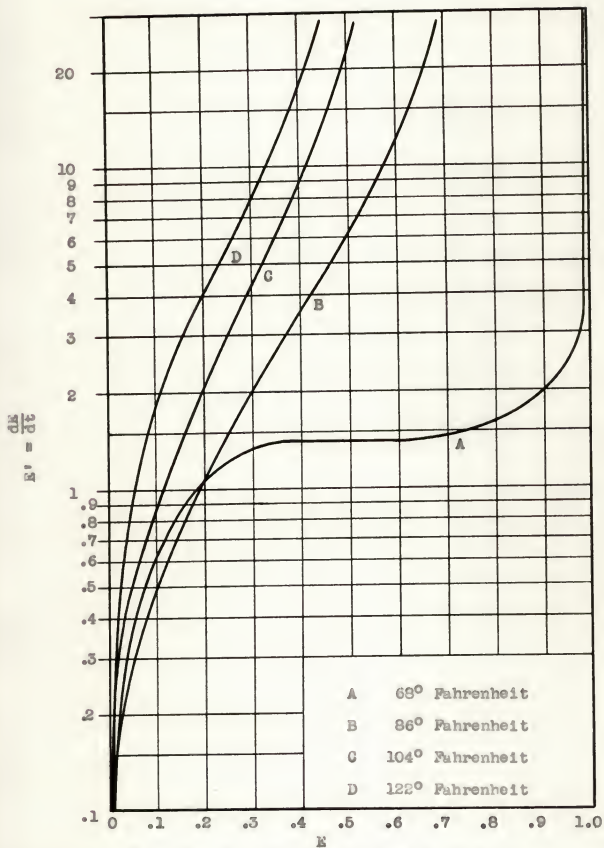


Fig. 12. E' versus E ; bran 16 to 20 mesh.

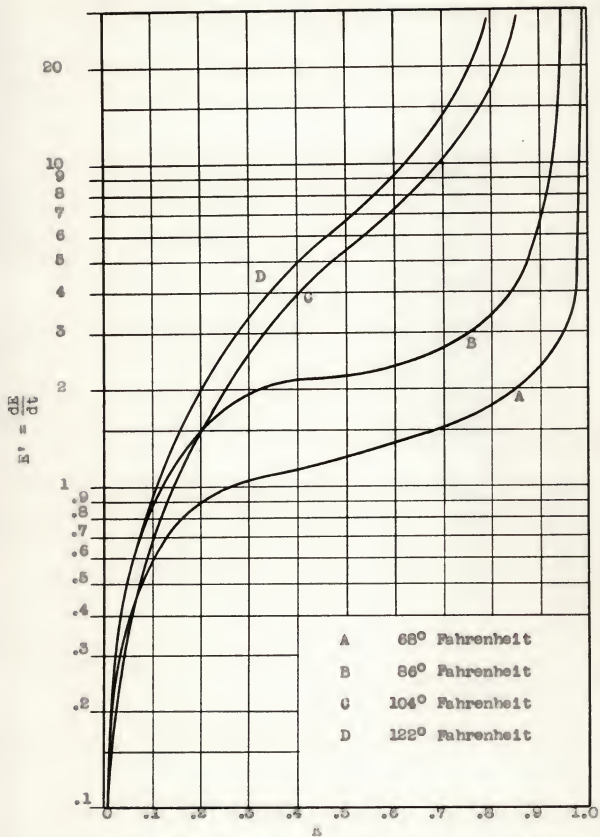


Fig. 13. E' versus S ; bran larger than 16 mesh.

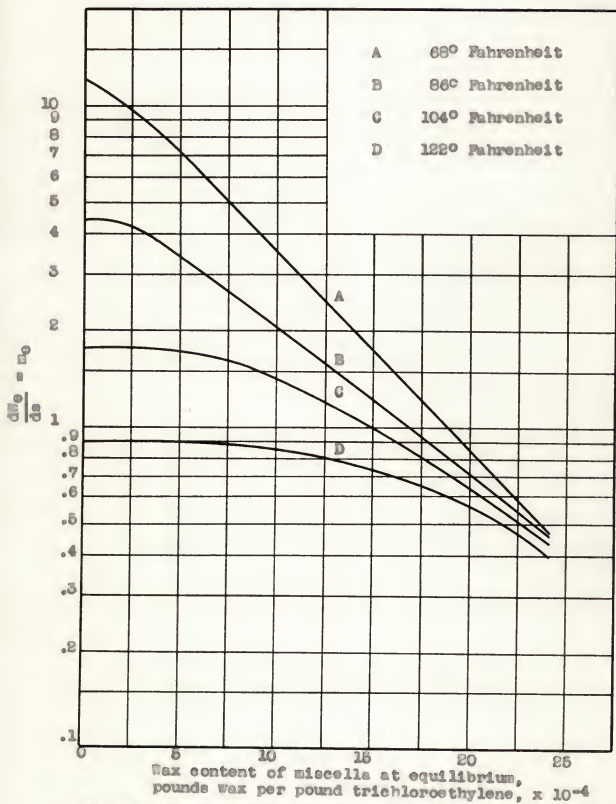


Fig. 14. $\frac{dW_0}{ds}$ versus the wax content of the miscella at $\frac{dW_0}{ds}$ equilibrium; bran smaller than 20 mesh.

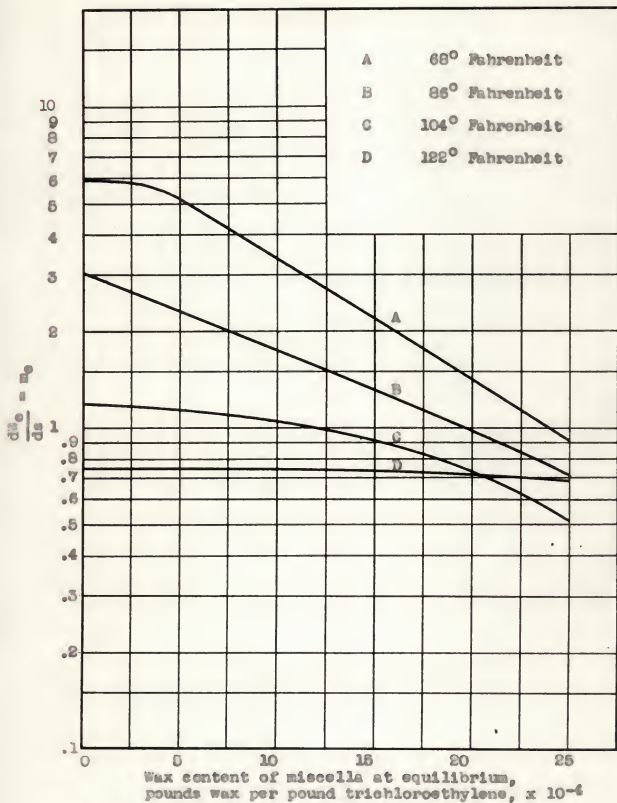


Fig. 15. $\frac{dw_e}{ds}$ versus the wax content of the miscella
 $\frac{dw_e}{ds}$ at equilibrium; bran 16 to 20 mesh.

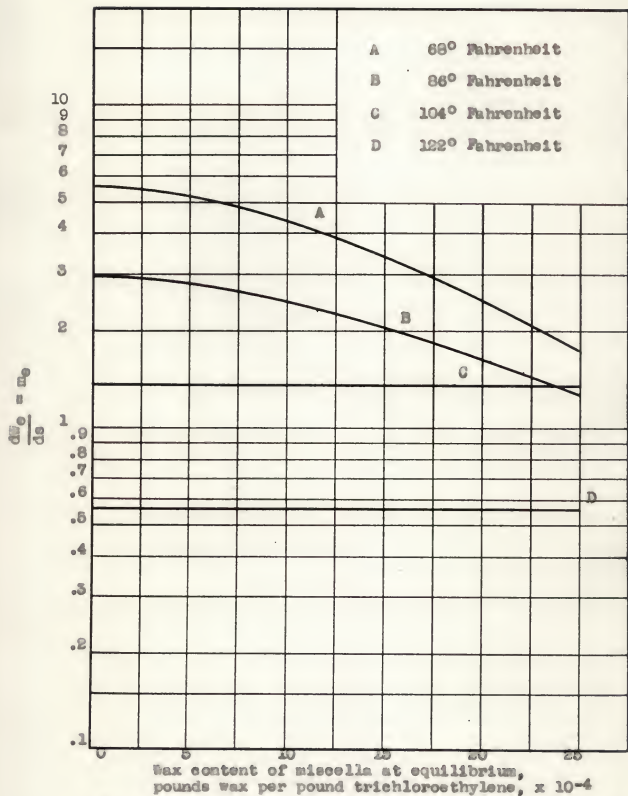


Fig. 16. $\frac{dW_0}{ds}$ versus the wax content of the miscella at $\frac{dW_0}{ds}$ equilibrium; bran larger than 16 mesh.

Substituting in equation (13), the general extraction equation is obtained:

$$\frac{dW}{dt} = \frac{E'}{G} \quad (15)$$

Batch Extraction of a Bran Fraction

The time required for extracting a particular bran fraction is given by

$$t = \int_{W_1}^{W_f} \left(\frac{dt}{dW} \right) dW \quad (16)$$

By equation (15),

$$\frac{dt}{dW} = \frac{G}{E'} \quad (17)$$

Substituting,

$$t = \int_{W_1}^{W_f} \left(\frac{G}{E'} \right) dW \quad (18)$$

The integration is performed graphically. The procedure is as follows: W versus s plots of the operating line and equilibrium curve are made. For each assumed value of s the values of G, E', and W are determined utilizing the derived general extraction curves. The area under the curve of G/E' versus W gives the extraction time.

Continuous Extraction of a Bran Fraction

If, in the continuous extraction of bran, the bran distribu-

tion and bran velocity are constant, the following equation applies:

$$B = av = a \frac{dh}{dt} \quad (19)$$

in which

B = bran rate, lb. per sq. ft. of extractor cross section per hr.

a = packing factor, lb. bran per cu. ft. of bran-solvent mixture

$v = \frac{dh}{dt}$ = velocity of bran particles through extractor, ft. per hr.

h = distance along length of extractor, ft.

Multiplying both sides by dW , equation (19) becomes

$$BdW = a \frac{dW}{dt} dh \quad (20)$$

By equation (15)

$$\frac{dW}{dt} = \frac{E'}{G}$$

Substituting in equation (20) the equation giving the extraction column height is obtained

$$h = \frac{B}{a} \int_{W_1}^{W_2} \left(\frac{G}{E'} \right) dW \quad (21)$$

The integral is determined graphically in a manner similar to the batch extraction procedure.

Batch Extraction of Bran-Fraction Mixtures

By a material balance

$$b(f_1W_1 + f_2W_2 + f_3W_3) = bW \quad (22)$$

where

b = total pounds bran

f_1 = fraction by weight of bran larger than 16 mesh

f_2 = fraction by weight of bran, 16 to 20 mesh

f_3 = fraction by weight of bran smaller than 20 mesh

W_1, W_2, W_3 = wax concentration in bran fractions,
lbs. wax per lb. wax-free bran

W = wax concentration in bran mixture, lb. wax per
lb. wax-free bran

Dividing both sides of equation (22) by b and differentiating with respect to time, the following equation is obtained:

$$f_1 \frac{dW_1}{dt} + f_2 \frac{dW_2}{dt} + f_3 \frac{dW_3}{dt} = \frac{d\bar{W}}{dt} \quad (23)$$

Utilizing equation (15) and integrating, the equation for the extraction period is obtained:

$$t = \int_{\bar{W}_1}^{\bar{W}_f} \frac{d\bar{W}}{f_1 \frac{E'_1}{G_1} + f_2 \frac{E'_2}{G_2} + f_3 \frac{E'_3}{G_2}} \quad (24)$$

In the above equation, the presence of an E'/G term for each bran fraction requires the establishment of an operating line for each fraction. The material balance

$$f_1(W_0 - W)_1 + f_2(W_0 - W)_2 + f_3(W_0 - W)_3 = R_s$$

suggests three operating lines with slopes, $-R/f_1$, $-R/f_2$, and $-R/f_3$ and intercepts, $(W_0)_1$, $(W_0)_2$, $(W_0)_3$.

The procedure for determining the time of batch extraction of mixtures of bran fractions is as follows: The overall oper-

ating line is plotted, \bar{W} versus s . An operating line for each of the bran fractions is drawn with a slope as indicated above. For various assumed values of s , a plot of \bar{W} versus $1/(f_1E_1^1/G_1 + f_2E_2^1/G_2 + f_3E_3^1/G_3)$ is made. By means of a graphical integration, the extraction time is evaluated.

Continuous Extraction of Bran-Fraction Mixtures

Equation (23) applies and if equation (19) is utilized, which, in this case, becomes

$$Bd\bar{W} = a \frac{d\bar{W}}{dt} dh \quad (26)$$

the column height equation is obtained:

$$h = \frac{B}{a} \int_{\bar{W}_1}^{\bar{W}_f} \frac{d\bar{W}}{\frac{f_1E_1^1}{G_1} + \frac{f_2E_2^1}{G_2} + \frac{f_3E_3^1}{G_3}} \quad (27)$$

The material balance for continuous extraction, which is

$$f_1(W - W_o)_1 + f_2(W - W_o)_2 + f_3(W - W_o)_3 = \frac{S}{B}(s - s_f) \quad (28)$$

suggests operating lines, one for each bran fraction, with slopes, S/Bf_j , ($j = 1, 2, 3$), with intercepts, $(W_o)_j$, ($j = 1, 2, 3$), at s_f , the final concentration of wax in the out-going miscella. The graphical integration is performed in the manner described for batch extraction of bran-fraction mixtures.

SUMMARY

1. Westland milo bran fractions larger than 16-mesh, 16 to 20-mesh, and smaller than 20-mesh were solvent-extracted, using solvent-bran ratios of 6, 10, and 15. The extractions were performed at 68°, 86°, 104°, and 122° Fahrenheit using commercial trichloroethylene as a solvent.

2. It was necessary to observe the increase in wax content in the wax-solvent solution (miscella) as the extractions proceeded. The removal of the wax from the miscella, composed of wax, oil, and solvent, was effected by crystallizing and filtering at -30° Fahrenheit. In this work the wax concentration in the miscella filtered did not exceed 0.43 percent. The wax obtained had a yellow-orange cast, was quite hard, and had a melting point of 75-78° Centigrade.

3. The wax contents of the bran fractions on a wax-free basis were 2.80 percent for larger than 16-mesh, 1.52 percent for 16 to 20-mesh, and 1.11 percent for smaller than 20-mesh. The bran comprised 20 percent of larger than 16-mesh, 38 percent of 16 to 20-mesh, and 42 percent of smaller than 20-mesh. The wax content of the original bran was 1.98 percent on a wax-free basis.

4. General extraction rate equations and curves were derived providing a method of attacking batch or continuous process design problems.

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LITERATURE CITED

- (1) Foveaux, M. T., Jr.
Construction and operation of a pilot plant for the continuous solvent extraction of wax from sorghum grain. Unpublished thesis, Kansas State Col. Agr. and Appl. Sci. 50 p. 1947.
- (2) Mellan, I.
Industrial solvents. New York. Reinhold Publishing Corp. 480 p. 1939.
- (3) Sherwood, T. K.
Ind. Eng. Chem., 21:12-16. 1929.
- (4) King, C. O., D. L. Katz, and J. C. Brier.
The solvent extraction of soybean flakes. Am. Trans. Inst. Chem. Engrs. 40:533-556.