

THE SOLVENT EXTRACTION OF LIPID MATERIAL
FROM SORGHUM BRAN

by

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INTRODUCTION

The purpose of this project was to study the extraction of wax and oil from sorghum bran and germ. The phase investigated was the solvent extraction of the wax and the oil from the bran, in particular the selective extraction possibilities of trichloroethylene for the wax portion of the bran.

Work by Kummerow (7) has shown that the majority of the wax lies in the hull of the grain, and although the percentage of wax is small by weight, the concentration is approximately fifteen times the amount in the allied grain, corn. The economic demand for the wax shows that if this source of wax is utilized, sorghum grain will be on a better basis for competing with corn which has the higher oil content, but which is not as suitable to the climatic and growth conditions in Kansas.

The development of a continuous, easily operated piece of extraction equipment is the desired outcome of this project. Useful information in the design of any extraction equipment is the rate of extraction as a function of the operating variables. This information, with the possible occurrence of selective extraction, was the goal of this work.

PREVIOUS WORK

Foveaux (3) investigated the various possible solvents for wax and oil extraction. After a comparison of properties he concluded that trichloroethylene would be a suitable solvent, with the major drawback being the high cost per gallon. He also built

and operated a screw type of conveyor for extraction of the whole grain. Wax was his main product.

French (4), following the recommendation of Kummerow and Foveaux that the wax extraction be carried out on the bran fraction only, obtained data on the equilibrium values of wax and solvent. The data thus gathered were on a batch process with extraction temperatures from 68 to 122°F. He also presented a method for transposing these data to the design of a continuous extraction unit.

Medlin (9) built and operated a pilot plant that used the bucket type of conveyor for the extraction mechanism. He found, through a short series of runs operating at a temperature of 160°F over small time intervals, that the main product was wax. The recommendation was made that work be initiated on the possibility of obtaining a commercial wax, without further refining by the selection of optimum operating conditions.

MATERIALS

The solvent used in this extraction was technical grade trichloroethylene, as obtained from the Dow Chemical Company. In the operation of the equipment as designed by Medlin, it was used directly from the drum. However, for the small scale rate equipment, it was distilled to remove all the color due to the ferric chloride formation during storage.

The bran for this experiment was obtained from Dodge City Industries Plant. Medlin gave the following description:

(it) was processed from country run milo graded as No. 2 yellow. The bulk of this milo was of the Westland variety. It included bran from both the debranning and cracking operations. The debranner bran was cleaned by aspiration and screening over 30 mesh screen. The fines from the cracking bran were not removed.

EQUIPMENT

Bucket Type Extractor

The bucket type of extractor was reassembled to give much the same operation as originally designed by Medlin. The major exception was that spray nozzles were constructed to do the main extraction, whereas formerly the greatest amount of the extraction was obtained in the passing of the buckets through the hot liquid in the bottom of the tank. Minor changes were made in the pumping system. One pump plus gravity flow were used to circumvent the use of two pumps. The heating of the liquid was done by steam rather than by the former electrical method. Pictures of the equipment and a more complete description are given in the unpublished thesis of Medlin (9). A schematic diagram, Fig. 1, is included in this thesis.

Rate Extraction Apparatus

For the runs made to obtain data on the rates of extraction the equipment consisted of liquid reservoir, water bath, extraction tube, and collector. The reservoir held 750 c.c. of trichloroethylene and was heated by means of an electrical knife type heater. The constant temperature water bath that surrounded the extraction tube was temperature controlled by a mercury thermostat

in the electrical heating circuit. The extraction tube contained an asbestos pad to filter the miscella and a wire gauze to distribute the solvent. In order to overcome the resistance offered by the filter pad, the collection flask was connected to an aspirator. Stirring occurred in the liquid reservoir and the water bath. A schematic diagram, Fig. 2, shows the overall assembly.

PROCEDURE

Bucket Type Extractor

In the operation of the bucket conveyor, the steam was first turned on to bring the water surrounding the coils up to the desired temperature. Fresh trichloroethylene from the storage tank was allowed to run through the flat spray nozzle into the bottom of the tank of the conveyor proper. The amount was sufficient to cover the drain leading to the recirculating pump to a depth of one inch. The blower was started to remove all the noxious fumes. The twin stage centrifugal was then adjusted to the desired recirculation rate by means of a mercury flow manometer. Twenty gram samples were then dumped into the passing buckets of the conveyor. The input of fresh solvent was controlled by means of a flow manometer that had cadmium nitrate and cobaltous chloride solution as the heavy liquid. The miscella was removed on the output side of the recirculation pump. The extracted bran was dumped on each complete cycle into the draining bin.

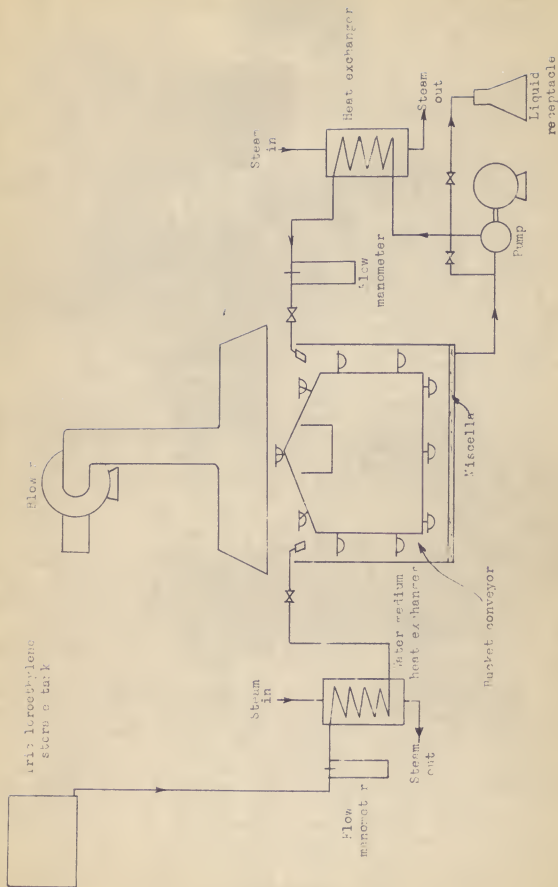


Fig. 1. Pilot plant bucket type extractor

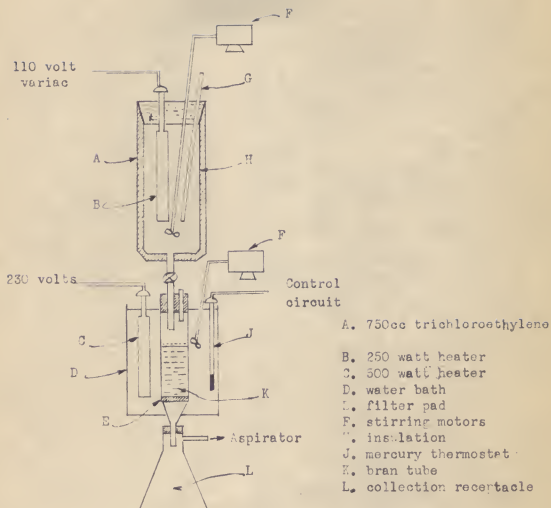


Figure 2. Soxhlet extraction apparatus

Rate Extraction Unit

The bran for the rate extraction unit was obtained from a single sampling of barrel number two. Twenty gram samples, $\pm .01$ gram, were weighed out in lots of ten or fifteen previous to the runs.

The trichloroethylene reservoir was filled and heated slowly as was the water bath. When the water bath was within 10°F. of the operating temperature, bran was placed in the extraction tube. The temperature of the bath was allowed to overshoot the operating temperature by two or three degrees. During this heating time the temperature of the bran was noted. When the bran and the bath reached the same temperature the trichloroethylene was admitted to the tube. The temperature of the trichloroethylene was slightly higher than the bran, depending upon the temperature at which the extraction was taking place and the flow rate. The time for this resistance type flow was measured from the start of the liquid flow into the bran tube until there was a sudden drop in the flow to the liquid receptacle.

Miscella from the extraction was concentrated by distillation and then allowed to evaporate by passing a stream of air over the container at room conditions. Repeated weighings were carried out until a constant weight occurred; this was called the crude.

In order to find the percent wax and oil in the crude, acetone was added and the solution was placed in a refrigerator where the temperature was -15°C. ; this resulted in the crystalliza-

tion of the wax fraction. The solution was filtered cold with a gooch crucible. The acetone was evaporated and the weight of oil and wax recorded.

The above was the general procedure; however, a "free flow" method was used whereby the asbestos filter pad was removed and the only resistance encountered was the resistance of the bran. In this case the time of extraction was recorded as the time between the first drop and the break off of the steady flow from the extraction tube. The resulting miscella was filtered to remove the bran fines. This method by the rapid change of receptacles readily gave the fraction of crude extracted during the different time increments.

RESULTS

The following tabulation sheets and graphs give the numerical results and the trend of the data. In general the increase in temperature increased the crude yield. The range of temperatures was from 130° to 182°F. The crude extracted varied from 1.37 to 3.24 percent of the total dry bran. The total extractable crude was 4.89 percent (Table 3), as determined by Dr. H. N. Barham of the Chemistry Department. The wax content of the bran was 1.00 percent. Contact times varied from 10 seconds to 15 minutes. Two solvent-to-bran ratios were used: 14.6 to 1 and 7.3 to 1 by weight.

Table 1. Solvent extraction data with asbestos pad as filter resistance.

Run	Temp. (°F.)	Weight of sample (grams)	Sol- vent to bran (gm/gm)	Time of contact (minutes)	Weight of crude (grams)	Percent crude extracted (dry basis) † $\frac{\text{gm crude}}{\text{gm sample}} \times \frac{100}{.881}$
1	144	19.78	7.3	4.50	0.4060	2.33
2	144	19.99	7.3	15.00	.4780	2.72
3	144	20.17	7.3	10.25	.4473	2.52
4	144	20.00	7.3	2.50	.3052	1.73
5	144	20.02	7.3	2.25	.3110	1.75
6	144	20.02	14.6	9.50	.3741	2.12
7	144	20.03	14.6	5.35	.3675	2.08
8	144	19.98	14.6	13.00	.4754	2.70
9	144	19.98	14.6	7.00	.4310	2.45
10	144	20.00	14.6	4.15	.3770	2.14
11	144	20.02	7.3	1.65	.3560	2.02
12	158	19.96	7.3	8.35	.4360	2.48
13	158	20.03	7.3	4.50	.3860	2.19
14	158	19.99	7.3	11.75	.4380	2.49
15	158	20.03	7.3	1.50	.3105	1.76
16	158	20.03	14.6	15.00	.4544	2.57
17	158	20.00	7.3	6.50	.4110	2.33
18	158	19.98	7.3	1.15	.3918	2.22
19	158	20.04	7.3	13.25	.4630	2.63
22	182	20.00	14.6	12.35	.5703	3.24

Table 1. (concl.)

Run	Temp. (°F.)	Weight of sample (grams)	Sol- vent to bran (gm/gm)	Time of contact (minutes)	Weight of crude (grams)	Percent crude extracted (dry basis) $\frac{\text{gm crude}}{\text{gm sample}} \times \frac{100}{.881}$
23	188	40.00	7.3	60.0*	1.5890	4.51
24	158	20.01	14.6	8.00	0.4506	2.56**
25	158	19.99	14.6	2.50	0.3998	2.27***
26	158	19.99	14.6	7.65	0.4995	2.84
27	158	20.00	14.6	4.75	0.4316	2.45
28	158	19.96	14.6	2.50	0.4092	2.32
29	158	20.01	14.6	4.75	0.4607	2.61
30	158	20.00	7.3	1.15	0.3624	2.06
31	158	20.00	7.3	5.10	0.4254	2.41
32	174	20.00	14.6	6.65	0.5045	2.86
33	174	20.00	14.6	3.50	0.4692	2.67
34	174	19.99	14.6	4.50	0.4828	2.74
35	174	20.00	7.3	1.40	0.3925	2.23
36	174	20.00	7.3	7.65	0.4003	2.27
37	174	20.00	7.3	4.00	0.3822	2.22

† Moisture content 11.9% as received

* Run 23 reflux one hour

** Run 24 moisture content 14% as received

*** Run 25 moisture content 13.6% as received

Table 2. "Free flow" extraction data.

Run	Temp. :(°F.)	Weight of sample :(grams)	Sol- vent to bran :(gm/gm)	Vol- ume of CHCl ₃ :(cc)	Time of outlet flow :(minutes)	Weight of crude :(grams)	Percent crude extracted (dry basis) : <u>gm crude x100</u> : <u>gm sample .881</u>
20	158	20.03	7.2	100	3.50	0.412	2.34 *
				60	0.0-1.80	0.381	2.16
				23	1.8-3.50	0.031	0.18
21	158	20.01	14.6	200	7.50	0.5183	2.94 **
				41	0.0-1.00	0.3279	1.86
				33	1. -2.00	0.0708	0.40
				28	2. -3.00	0.0379	0.22
				54	3. -5.00	0.0860	0.32
				21	5. -7.50	0.0257	0.15
38	174	19.99	7.3	100	10.00	0.4527	2.57
				24	0.0-2.00	0.3167	1.80
				20	2. -4.00	0.0458	0.26
				12	4. -6.00	0.0365	0.21
				14	6.-10.00	0.0537	0.30
39	174	20.00	14.6	200	4.50	0.4439	2.52
				43	0.0-0.50	0.3277	1.86
				47	.5-1.50	0.0441	0.25
				40	1.5-2.50	0.0369	0.21
				39	2.5-3.50	0.0237	0.13
				11	3.5-4.50	0.0115	0.07

Table 2. (concl.)

Run	Temp.	Weight of sample	Sol- vent to bran	Vol- ume of CHCl ₃	Time of outlet flow	Weight of crude	Percent crude extracted (dry basis)
(°F.)	(grams)	(gm/gm)	(cc)	(minutes)	(grams)	$\frac{\text{gm crude}}{\text{gm sample}} \times 100$	
40	130	20.01	14.6	200	5.50	0.4269	2.42
				20	0.0-.50	.2422	1.37
				43	.50-1.50	.0992	0.56
				53	1.50-3.00	.0452	0.26
				42	3.00-4.50	.0303	0.17
				11	4.50-5.50	.0100	0.06
41	130	20.00	7.3	100	2.50	.3980	2.24
				17	0.0-0.33	.2562	1.45
				16	.33-.66	.0814	0.46
				16	.66-1.00	.0295	0.16
				21	1.0-1.50	.0242	0.14
				6	1.5-2.50	.0065	0.04
42	130	20.01	7.3	100	1.00	.3637	2.05
				22	0.0-.16	.2356	1.33
				21	.16-.33	.0758	0.43
				17	.33-.50	.0296	0.17
				17	.50-1.00	.0227	0.13
43	138	13.57	14.6	135	1.25	.3113	2.34 ***
				35	0.0-0.25	.2320	1.75
				31	.25-.50	.0404	0.30
				52	.50-1.00	.0346	0.26
				7	1.0-1.25	.0043	0.03

* Starch in sample corrected

** Starch in sample uncorrected

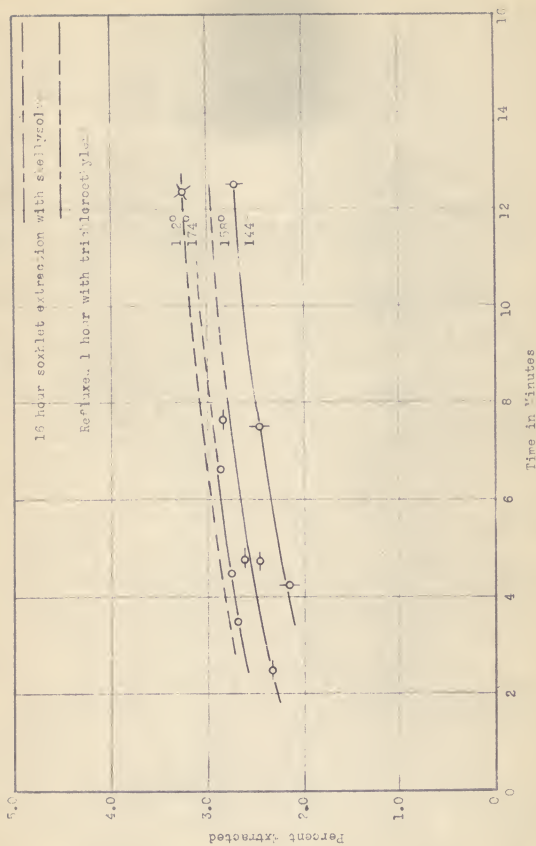
*** Moisture content 2% as received

Table 3. Bran analysis of barrel #2 (dry basis).

Crude percent	Pure oil percent	Pure wax percent	Melting point of wax °C.
4.58	3.57	1.01	79.7-80.7
5.24	4.25	.99	80.0-80.8
4.84	3.85	.99	
Av. 4.89	3.89	1.00	

Table 4. Screen analysis of barrel #2.

Sample	Weight of sample grams	Percent on 16 mesh	Percent on 20 mesh	Percent thru 20 mesh
Top	165	17.6	29.1	53.3
Middle	156	21.8	30.1	48.1
Bottom	178	25.8	23.6	50.6
Av.		21.7	27.6	50.7
Overall	153	17.6	28.7	53.6



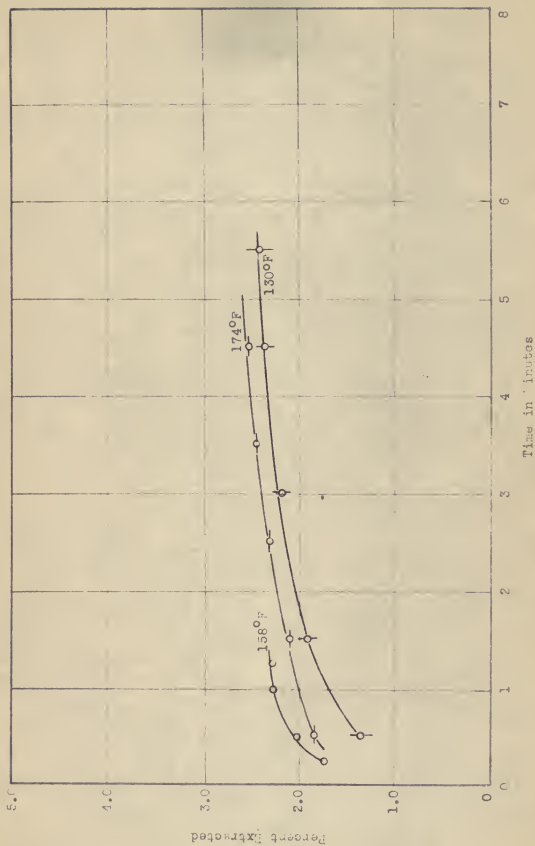


Fig. 4. Percent exchanged, organic, versus contact time in minutes with a xylene-toluene mixture of 14. to one at various temperatures. Free flow diffusion.

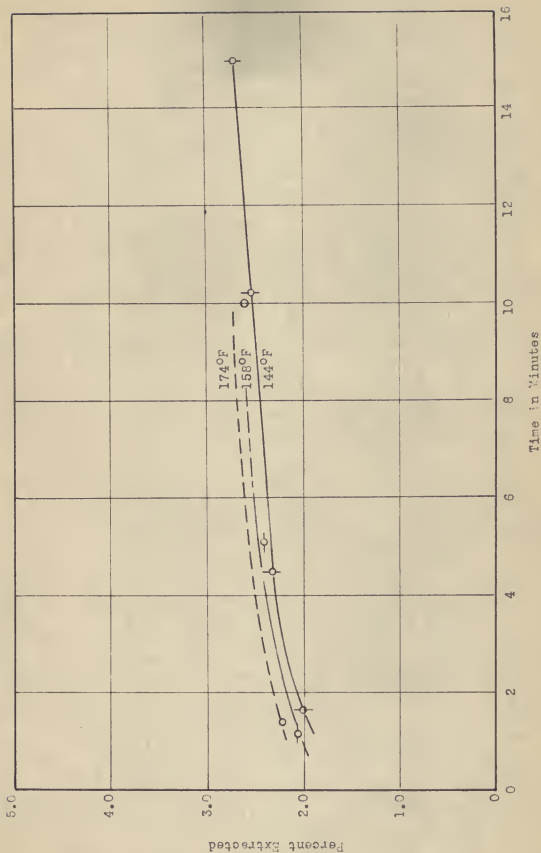


Fig. 5. Percent extracted, crude, versus contact time in minutes with a solvent-to-bran ratio of 7.3 to 1 at various temperatures using an asbestos filter pad.

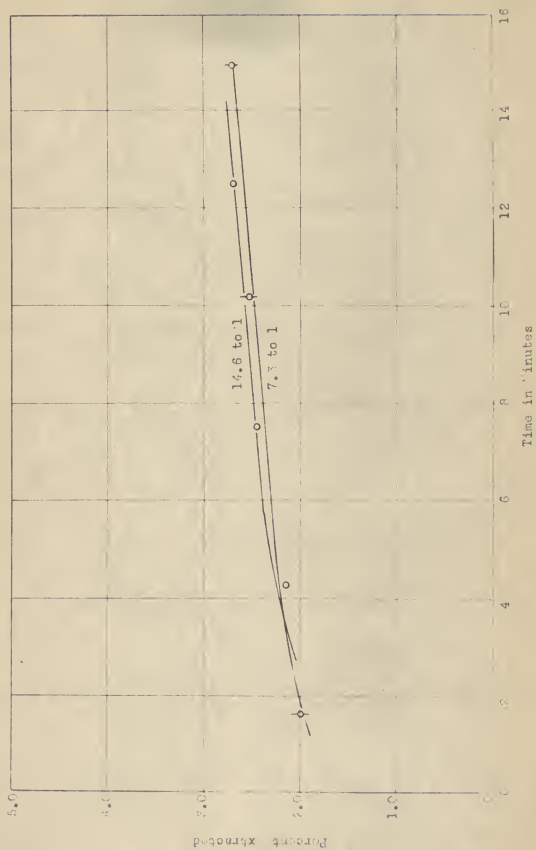


Fig. C. Comparison of solvent-to-ran ratios at 144°F using asbestos filter pad.

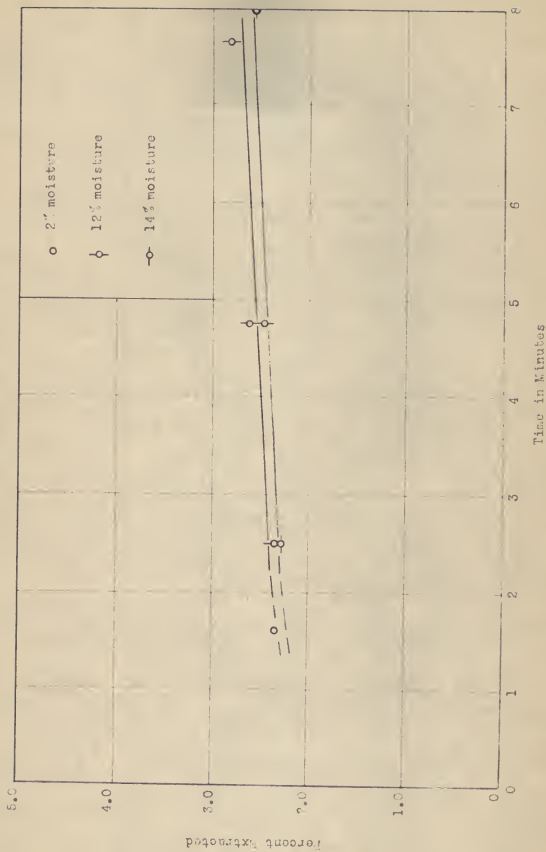


Fig. 7. Influence of moisture content on extraction rate at 150°C with solvent-to-bran ratio of 1.6

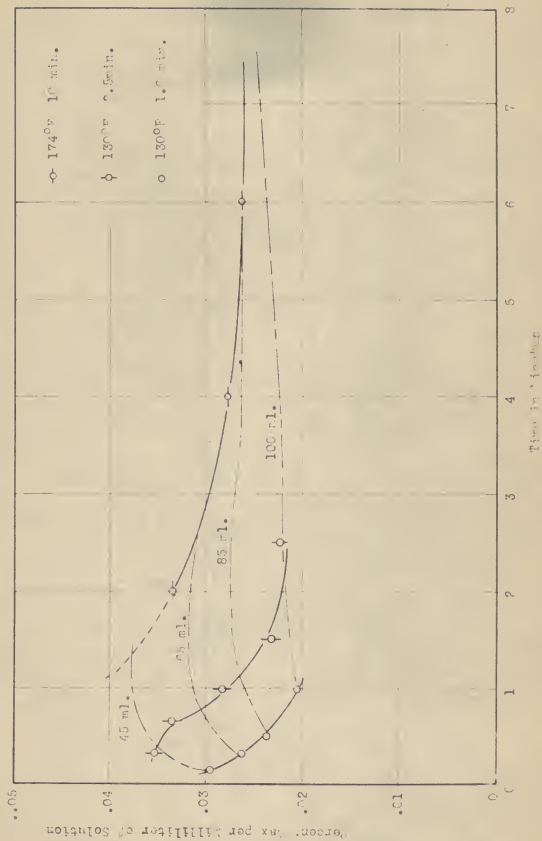


Fig. 1. Percent Hex per milliliter of solution versus reaction time in minutes for three different volumes of a solvent-to-reactant ratio of 1 to 1.0, 1.5, and 2.0, at 174°F, 130°F, and 130°F.

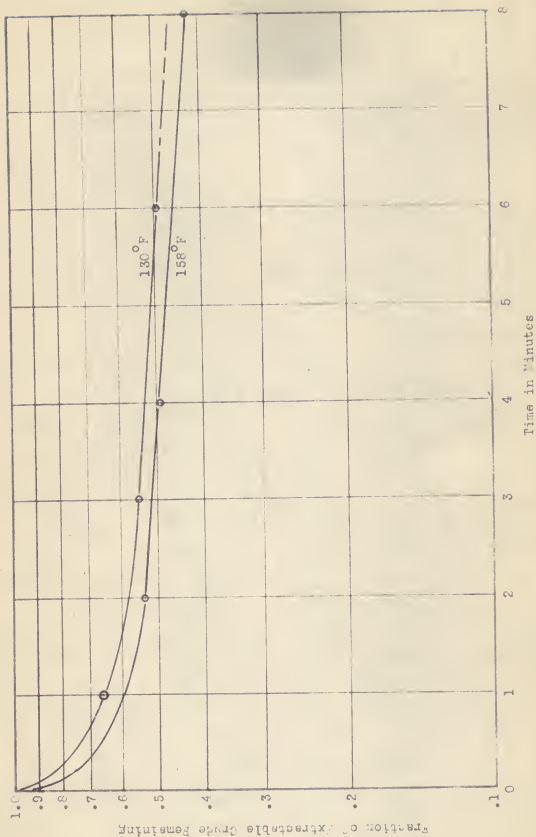


Fig. 9. Fraction of extractable crude remaining in bran versus contact time in minutes with solvent-to-bran ratio of 14.5 to 1. The 130°F run was made by the free flow method. 158°F run was made using the shistos filter pad.

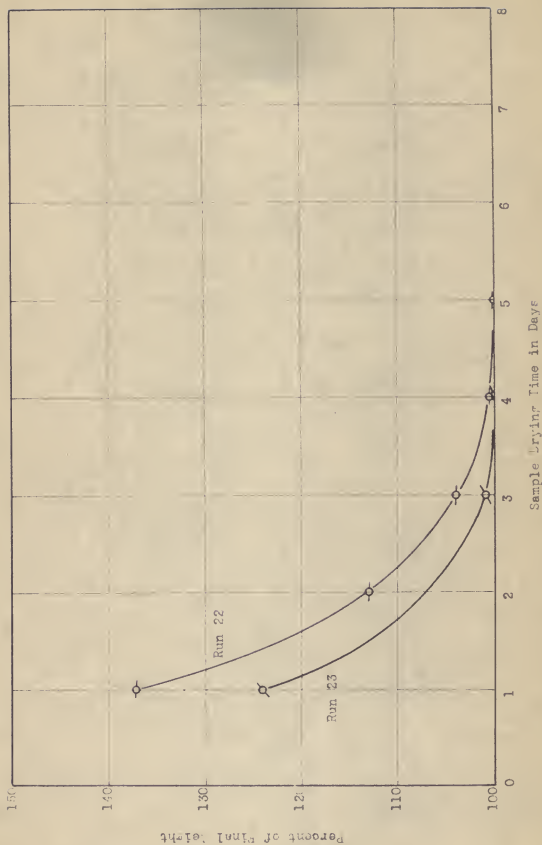


Fig. 10. Correction curves for drying time of the 143° runs.

DISCUSSION

Bucket Extraction Unit

The bucket type extractor was reassembled to give slightly different operation from the original design. The bulk of the extraction was to be done by the flat spray nozzles; however, due to heat losses, the spray was cooled from 170°F. to room temperature before contact with the bran was made, thus nullifying the desired high temperature solvent effect. The equipment would have to be insulated and in a closed system in order to operate the unit under these conditions.

This type of equipment represents the older form of extraction apparatus. In general the bucket system is used for a large tonnage of feed, 100 to 1000 tons per day. This tonnage is much larger than the desired amount for bran extraction. The smaller commercial equipment is of a number of different designs. The main designs are the screw conveyor and the chain conveyor systems. It is of interest to note that the smallest size commercial extractor for this type of work is 25 tons per day; this would indicate the necessity for processing 250 tons per day of sorghum grain. Since the starting and stopping of the extraction operation are inefficient, long periods of operation at rated load are desired. The bran and germ might be extracted on the same piece of equipment at different times, thus effectively reducing the tonnage of whole grain required per day. The economics of bran extraction indicates that it is a marginal operation.

If the germ is prepressed before solvent extraction, which is the indicated economical practice, the bran and germ might be simultaneously extracted. This would require that the oil and wax be separated by some method, such as liquid-liquid extraction. A separation would also be required if the bran only was extracted, providing that no point of operation could be found where the wax alone would be extracted.

The bran and germ are 7 percent and 10 percent by weight respectively of the whole sorghum grain. The amount of extractable material in the bran, with reasonable time limit, is about 3 percent of the bran. The germ, which contains 25 percent by weight of total extractable material, would, when compared to other germs, yield two-thirds of this when prepressed at moderate pressures. There would then remain 8 percent lipid material to be solvent extracted. After the germs were prepressed, they would be sent to the flaker. With the germs in the flaker condition, the extraction time required to remove the remaining lipid material to less than 1 percent is within the same time interval as normal bran extraction. The bran and germ would then be mixed and sent to the solvent extractor. The solvent recovery system with simultaneous extraction method would be simpler than the double system for separate extraction and recovery. For counter-current solid-liquid flow, the washing of the material could be effectively done with a small amount of solvent. The germ contains about 0.5 percent wax, which would be separated in the same step with the wax from the bran.

For the above reasons, work on the prepressing and subsequent solvent extraction of the germ should be initiated.

Rate Extraction

The visible results of the rate extraction showed that only limited selective extraction for the wax portion of the bran occurred over the range investigated. Medlin stated that, "The difference in the extraction rates of the wax and oil indicated that, under suitable operating conditions, satisfactory separation can be made in the extraction step of the process." This selectivity does not appear to happen. However, this bran contained only 1.00 percent wax compared to the former report of 2.00 percent wax. Since the bran contained only 1.00 percent wax and for the shortest time, 10 seconds free flow, and the lowest temperature, 130 F., a crude content of 1.37 percent was reported, no operational point beyond this could result for this type of contact in the obtaining of a quality commercial wax. The free flow time was from 15 to 30 seconds less than the actual contact time. A shorter time period could be obtained by decreasing the bed thickness, which was approximately 4 1/2 inches, however, in a commercial extractor using large quantities of bran, the depth of the bed perpendicular to the flow of the liquid will be at least this great.

The influence of temperature on extraction rates showed a regular increase of rate with increase in temperature. While the amount of crude extracted with a reasonable upper extraction limit of 15 minutes was not as high as desired, the higher temperatures gave an increased yield that might warrant the use of these condi-

tions. But from pump considerations of vapor lock and packing and from heat input to the fluid, 170°F is the normal upper limit at atmospheric conditions. The use of pressure equipment is not feasible from the extra cost entailed and from the possibility of leaks, which would cause the loss of valuable solvent or create a fire hazard.

A solvent-to-bran ratio of 14.6 to 1 gave a better yield than the 7.3 to 1, Fig. 6, but the increase was slight so that the lower ratio would be desired. In practice the actual ratio would be in the region of 3 to 1, since the bran on leaving the extractor contains from 1 1/2 to 2 times the dry weight of wash solvent. With this solvent-to-bran ratio a recycle ratio of around 3 to 1 would be used. This would be economical and could be easily done since the concentration of the crude in the miscella is only about 3 percent by weight, and therefore the loss in driving force would be small.

In Fig. 7, the moisture contents and their influence upon the rate of extraction show that there was for the two moisture contents, 2 percent and 14 percent, little difference from the rate of extraction compared to the rate with 12 percent of the regular bran. This does not mean that there is no intermediate point where there would be better extraction; for there is, in general, with ordinary oil seed an optimum moisture content. The Skipin process (11) is predicated upon this phenomena. The economics of bran wetting or drying is, of course, a major factor for operating at a given moisture content.

The majority of the lipid material was extracted with the first few milliliters, as is seen from Fig. 8, and from the data, Table 2. In the construction of these curves from free flow data, the amount of liquid remaining on the bran, after the extraction was completed, was added to the amount of liquid through at any time. For a 100 milliliter input, 70 percent of the wax extracted was removed with the first 50 milliliters through.

Mechanism of Lipid Extraction

In the process of a solid or liquid material going into solution, the diffusional step is important. In the case of a solid there is the surface diffusion; however, if it is a porous solid or is contained within a porous solid, there will be intra-solid diffusion. For the conjecture of the possible mechanism of lipid extraction these two diffusions, surface and intra-solid will be considered.

Katz (6) has pointed out that for a porous solid if the rate governing step is the diffusion within the solid the following expression holds:

$$E = \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp \left\{ - \frac{D (2n+1)^2 \pi^2 t}{L^2} \right\}$$

E is the fraction of extractable material remaining unextracted after time, t.

D is the diffusion coefficient.

L is the flake thickness.

t is the time.

n refers to the terms in the series.

This equation has certain restrictions which the bran obeys in general. If E is plotted on semi-logarithmic paper against Dt/L^2 the plot will be a straight line for E less than 0.7. This is evident from the nature of the series when it is expanded. Referring to Fig. 9, the straight line portion of the curve agrees with the equation, but at a lower value of the fraction of extractable material remaining. Among the possible reasons for curving to a lower value of E are two, that may, in this case, be the cause for the variation. The first is due to non-homogeneity of the bran, if this were true the lower portion of the curve would not be straight. Another reason is that there is surface diffusion as well as intra-solid diffusion during the first part of the run. From the physical nature of the bran, the last reason appears to be the plausible one. This would give an explanation for the apparent partial selectivity of the trichloroethylene for the wax. For, by cooling equal portions of wax and oil in this solvent, the wax will crystallize out first showing that the solvent does not appear to have greater solvency powers for the wax portion. Carnauba wax, to which the sorghum wax is closely related, is found on the outer surface of a certain type of palm tree. During the hot, dry weather wax is exuded from the pores on the surface. The sorghum grain shows this same tendency for increased wax content during dry years. One of the reasons that the oil content of this bran is higher than the normal run is that there is a large amount of germ dispersed throughout the bran. This would also hinder any possibility of obtaining only wax by a suitable choice of operating conditions.

Since the semi-logarithmic plot follows a straight line for a major part of the time intra-solid diffusion governs, and it may be concluded that liquid rate and stirring have little effect during this period, so that a smaller liquid rate will be about as effective as the high liquid rate. This fact was also concluded from the effect of solvent-to-bran ratios on extraction rates.

Miscellaneous

The trichloroethylene when in contact with free water or moist air tends to hydrolyze with the resulting formation of hydrochloric acid. An inhibitor may be used to stabilize and reduce this formation rate, but in the construction of any equipment, the use of ordinary steel parts should be avoided for ferric chloride is readily formed, which colors the final product. The use of copper should also be avoided, since small traces of copper increases the rate at which the vegetable oil becomes rancid and darkens.

For the 144°F. runs, the samples did not dry completely but rather were weighed after one day. For this reason curves of representative runs were plotted so that these runs could be put on the same basis as the crude that had complete removal of the trichloroethylene.

CONCLUSIONS

1. The bucket type extractor does not appear to be the best apparatus for small tonnage extraction.

2. Work should be initiated on the germ extraction process.
3. No operational point was found whereby wax alone could be extracted, but this may be due to the sample of bran.
4. The probable mechanism for extraction is surface diffusion and intra-solid diffusion.
5. The upper operating temperature limit is in the region of 170°F.
6. A solvent-to-bran ratio of 7.3 to 1 has nearly the same rate of extraction as a 14.6 to 1 ratio.

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