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Solvent Extraction of Soybean Oil by Mixtures of Trichloroethylene and Ethyl Alcohol

S. G. MEASAMER, O. R. SWEENEY AND L. K. ARNOLD

Various solvents have been used for extracting vegetable oils such as soybean oil. Certain petroleum fractions are widely used commercially. They have the advantage of low cost, but the disadvantage of being highly flammable and their vapors being explosive in a wide range of mixture with air. Certain of the chlorinated hydrocarbons are excellent solvents but have had only limited use because of high cost and lack of suitable processing methods and equipment. Process and equipment for the use of trichloroethylene, one of these chlorinated hydrocarbons, have been developed in Engineering Experiment Station Laboratory, Iowa State College. Trichloroethylene is a colorless liquid boiling at 86.7°C. with a specific gravity of 1.46 at 25°C. It is an excellent solvent for soybean oil, is non-flammable, and of a toxicity of the order of the usual petroleum fractions. Various alcohols such as ethanol and methanol have had limited application.

Anhydrous ethanol and methanol are good solvents for the oil at temperatures close to their boiling points but poor solvents at room temperature. It is thus possible with the alcohols to dissolve the oil at the higher temperature and then cool the mixture until it separates into two solutions, one rich in oil and the other containing very little oil. As the alcohols take up water they become poor solvents thus making it necessary to fractionally distill them after each use as a solvent unless extracting material very low in moisture. The alcohols are also flammable although not as hazardous in this respect as gasoline.

Preliminary work in this laboratory by Ferguson (1) indicated that a mixture of ethanol and trichloroethylene combined certain advantages of the former together with decreased fire hazard resulting from the latter.

To determine whether mixtures of either methanol or ethanol and trichloroethylene would be suitable for commercial soybean extraction, it was first necessary to secure data on the solubility of the oil in various solvent mixtures at different temperatures. The equilibrium solubility relationships for trichloroethylene-methanol-soybean oil and trichloroethylene-ethanol-soybean oil systems were determined as follows: Mixtures of the alcohol and trichloroethylene of known compositions varying from 0 to 60 percent trichloroethylene were placed in a 3-necked balloon flask. An air driven stirrer was operated in the flask through a mercury seal in the center neck. A thermometer was mounted in a stopper in a second neck. Soybean oil was added in small amounts from the burette. After each addition the temperature was raised until the oil dissolved. The temperature was then lowered until turbidity or cloudiness was observed and then raised until the mixture was clear. The arithmetic average of the clearing and cloud-

ing temperatures were taken as equilibrium temperatures. Data for absolute methanol is given in Fig. 1 and absolute ethanol in Fig. 2.

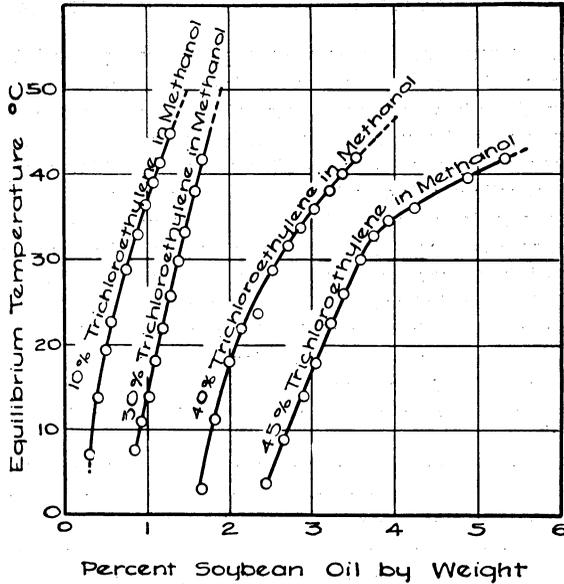


Fig. 1—Solubility of soybean oil in absolute methanol.

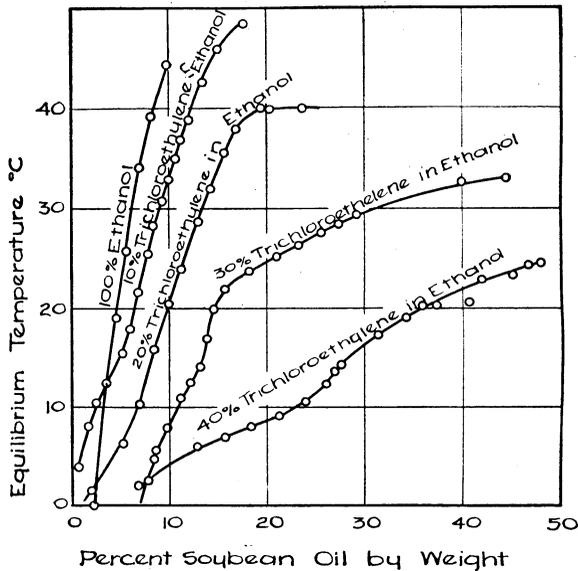


Fig. 2—Solubility of soybean oil in absolute ethanol.

The trichloroethylene-ethanol mixtures are much better solvents than trichloroethylene-methanol mixtures.

Mixtures of trichloroethylene and oil were made over a range from 0 to 60 percent trichloroethylene and absolute ethanol was added to these using the same procedure used in the preceding determinations. The data obtained were combined with the preceding data to plot the solubility curves in Fig. 3.

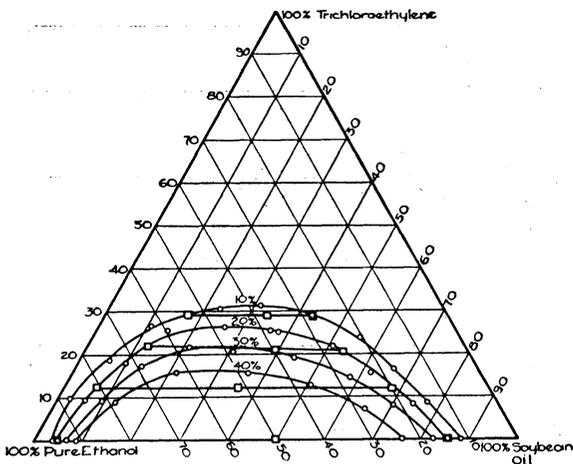


Fig. 3—Ethanol-trichloroethylene-soybean oil solubility diagram.

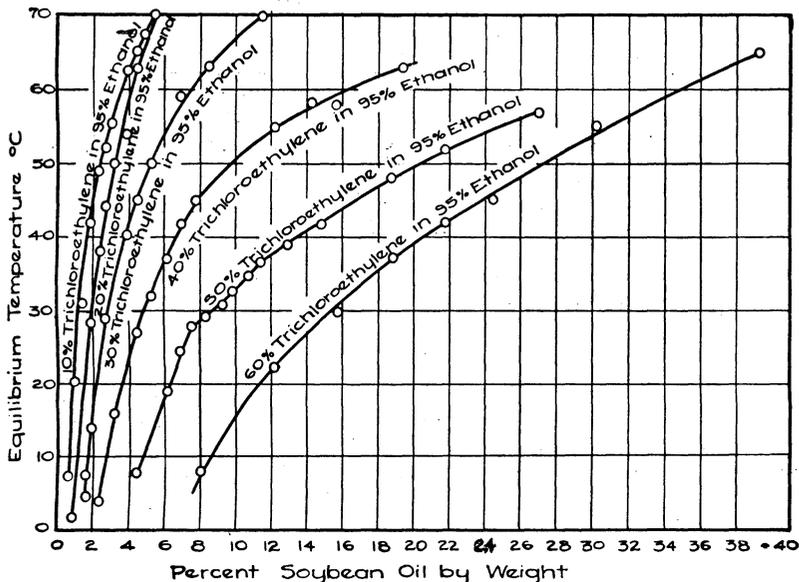


Fig. 4—Effect of temperature on the solubility of soybean oil in various trichloroethylene mixtures.

To investigate the effect of alcohol containing some water data for the trichloroethylene—95 percent ethanol—soybean oil system was determined with results shown in Fig. 4. Data from the preceding curves together with additional data determined in a similar manner is plotted in Fig. 5 to show the increase in solubility of the soybean oil in various mixtures of the alcohols, water, and trichloroethylene

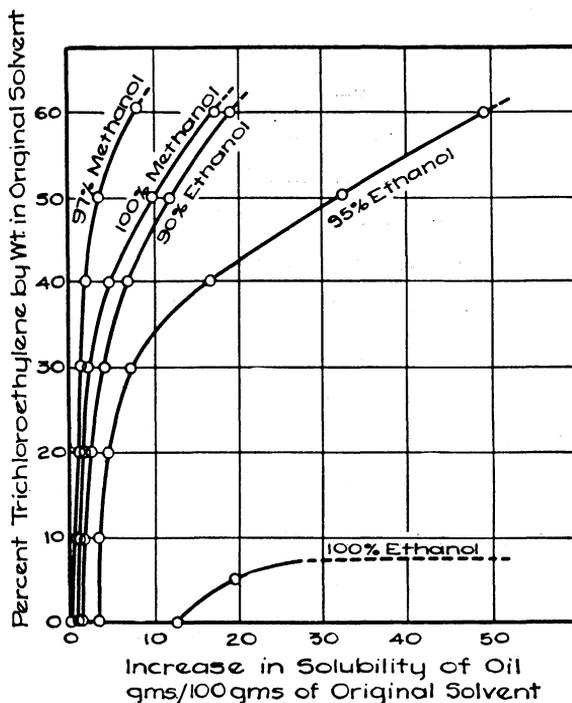


Fig. 5—Increase in the solubility of soybean oil in alcohol-trichloroethylene mixtures with increase in percentage of trichloroethylene.

when the temperature is raised from 10°C. to 60°C. Data is also given in Table 1 showing the distribution of soybean oil in the phases

Table 1.

Distribution of Soybean Oil in the Phases of a Two Phase Mixture of the System Trichloroethylene—95 percent Ethanol—Soybean Oil

Composition of original mixture, percent by weight

Percent by weight of oil in phases after separating at 20°C.

Oil	Tri.	Ethanol	Upper phase	Lower phase
47.7	0.0	52.3	2.87	93.7
45.2	9.53	45.3	2.79	91.8
42.3	16.0	41.7	2.75	75.5
37.7	24.8	37.6	3.78	63.3
35.9	28.6	35.5	4.70	59.1
33.3	33.0	33.7	3.32	60.7

of a two-phase mixture of the system trichloroethylene—95 percent ethanol-soybean oil.

From the data presented it is seen that the greatest increase in solubility of soybean oil with temperature rise is secured when absolute ethanol and trichloroethylene are used. If eight percent by weight of trichloroethylene is present in the original solvent the oil will be soluble in all proportions above 60°C. At 10°C. the solubility decreases to three percent. If it were practical to operate an extraction plant with a mixture of absolute alcohol and trichloroethylene, a mixture containing 10 percent trichloroethylene could be used to extract the beans in a countercurrent process above 60°C. A miscella concentration of 25 to 30 percent could be readily obtained which on cooling to 20°C. would settle into two phases containing 75 percent and 6 percent oil.

Since it would probably be impractical to extract beans containing no moisture the miscella would soon contain some water. It should be possible by drying the beans to a low moisture content to use 95 percent ethanol without taking up water from the beans. As shown in Table 1 mixtures of trichloroethylene, and 95 percent ethanol containing sufficient soybean oil at higher temperatures, separate at 20°C. into two phases one containing the greater part of the oil. If a mixture containing 65 percent trichloroethylene is used oil will be soluble in all proportions above 60°C. Mixtures containing 30 percent trichloroethylene will dissolve about 12 percent oil at 70°C.

The absorption of trichloroethylene and ethanol vapors by the soybean flakes and extracted meal was studied. Thirty-gram portions of both flaked soybeans and extracted meal were placed in evaporating dishes in tight metal containers, the first container containing a dish of trichloroethylene, the second one of alcohol, and the third one containing a mixture of trichloroethylene and alcohol having a specific gravity of 0.920. Care was taken that the liquids did not come into direct contact with the flakes or meal. After standing 3 days the flakes and meal were weighed to determine the amount of solvent absorbed. The extracted meal absorbed 0.064 grams trichloroethylene per gram; 0.246 grams ethanol; and 1.0377 grams of the trichloroethylene-ethanol mixture. The unextracted flakes absorbed 0.495 grams trichloroethylene per gram; 0.228 grams ethanol; and 0.140 grams of the trichloroethylene-ethanol mixture.

Fifty gram lots of soybean flakes were placed on a screen $\frac{3}{4}$ inches above the bottom of 2-inch diameter by 4-inch metal can. Air bubbled through trichloroethylene at a known rate was run into the bottom of the can. Weighings were made at 10-minute intervals to determine the amount of trichloroethylene absorbed. The results (see Table 2) from Test 4 are plotted in Fig. 6, to the relation between amount absorbed and time. To secure further data trichloroethylene vapor and air were blown through a 3-foot by 2-inch in diameter column of flakes for 30, 60 and 90 minutes at rates of flow from 0.03 to 0.07 cubic feet per minute. The results are given in Table 3. From the data in Fig. 6 the coefficient of absorption can be calculated to

Table 2.

Absorption of Trichloroethylene Vapors from Air by Soybean Flakes

	1	2	3	4
Rate of air flow c.f.m.....	0.07	0.06	0.05	0.06
Temperature of evaporator °C.....	24.0	11.0	11.0	11.0
Temperature of absorber °C.....	25.0	15.0	25.0	25.0
Original net weight of flakes in grams.....	50	50	50	50

Time Weight Data

Time Min.	Grams (Gross Weight)			
0	93.06	93.08	93.98	93.89
10	94.85	93.60	95.82	95.55
20	95.75	96.70	96.73	96.49
30	96.32	97.41	97.01	96.71
33	97.42
40	96.73	98.00	97.20
43	97.67
50	96.92	98.40	97.23
53	97.86
60	97.15	98.70	97.64
63	98.00
70	97.70
73	98.03
80	97.86
90	97.82	99.19	97.86
110	99.56	97.96
120	98.15

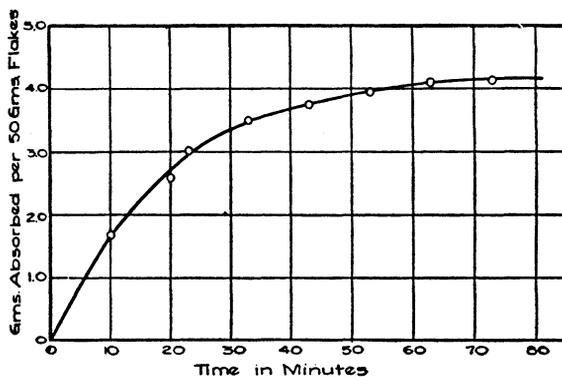


Fig. 6—Absorption of trichloroethylene vapors by soybean flakes.

be 152 pounds of trichloroethylene per cubic foot of soybean flakes per atmosphere partial pressure difference per hour. Since in an extraction plant the quantity of trichloroethylene escaping with the air vented from the condensers will be small compared to the flakes processed it should be possible to vent the condensers into the incoming flakes and completely absorb the solvent vapor.

Table 3.

Absorption of Trichloroethylene from
Air in Absorption Towers at 25°C.

	1	2	3	4
Rate of air flow c.f.m.....	0.06	0.07	0.07	0.03
Time of absorption in minutes.....	90	60	30	60
Loss of weight in evaporator gms.....	20.0	22.0	12.0	14.5
Gain in weight by flakes gms.....	23.2	17.3	6.5	6.7
Total weight of flakes.....	591.5	484.5	475.0	647.1

To secure data for the design of a commercial solvent extractor for soybean oil using a mixture of trichloroethylene and ethanol as a solvent a small pilot plant unit was designed and put into operation. A flow diagram of this plant is shown in Fig. 7. The extractor

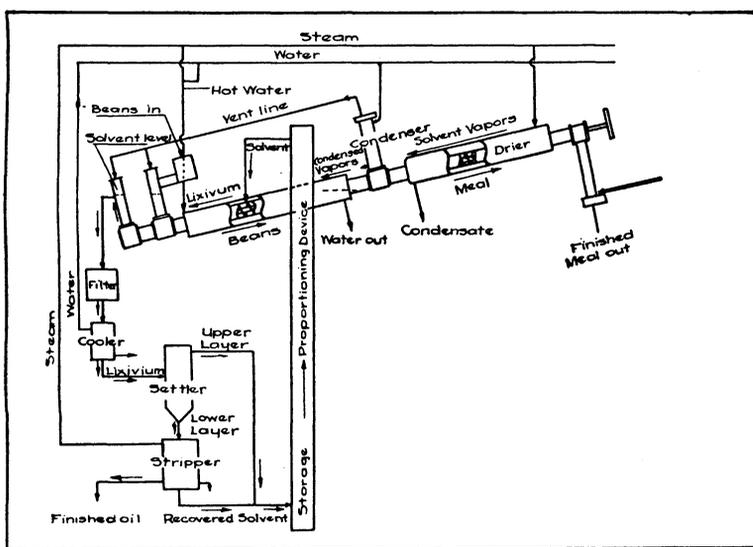


Fig. 7—Flow diagram of pilot plant for soybean oil extraction.

consisted of a six-foot length of jacketed 2-inch pipe connected to another 6-foot length of jacketed pipe which served as a drier. A motor driven screw conveyor extended through both extractor and drier and the whole assembly was mounted at a slight angle from the horizontal. Flaked beans were fed in at the lower end of the extractor and were carried upward through the extractor and drier by the screw. The extractor was heated by hot water to about 70°C. The drier was heated by steam under 25 p.s.i. pressure. The low oil containing phase from a previous run with trichloroethylene added to give a specific gravity of 1.00 was added at about the center of the extractor and flowed in the opposite direction to the beans leaving the lower end of the extractor. Solvent vaporized from the

extracted flakes in the drier passed out of the lower end of the drier into the reflux condenser between drier and extractor. The condensed solvent from the condenser ran into the upper end of the extractor passing down counter current to the flakes thus washing the meal of the oil containing solvent used in the lower half of the extractor and extracting additional oil from the flakes. The non-condensable gases from the condenser were vented into the incoming flakes. The dried meal passed out of the upper end of the drier through a 2-inch pipe and an Everlasting valve. The miscella (solvent-oil mixture) passed out of the lower end of the extractor through a bag type filter and a cooler into a glass jar in which it separated into two layers. The upper layer containing only a few percent of oil ran into the combined storage and proportioning device. The lower layer containing most of the oil ran to the stripper. In the stripper the miscella flowed down over a vertical row of four steam heated pipes. On the two sides of the bank of pipes were two flat sheet metal condensers cooled by water. As the solvent vaporized from the oil on the hot pipes the vapor came in contact with the condenser plates and condensed dropping off the lower end to run into the solvent storage. The oil dripped off the bottom of the pipes into a collecting trough. The proportioning device which fed the recovered solvent back into the extractor was a miniature bucket elevator inside a sheet metal casing. A variable speed drive made it possible to regulate the flow into the extractor as desired.

A series of 10 runs of approximately 24 hours each were run on the pilot plant. The solvent used was a mixture of denatured alcohol and trichloroethylene having a specific gravity of 0.920 to 1.000. The denatured alcohol was of the following composition: 100 parts 95 percent ethyl alcohol, 5 parts menthanol, 5 parts ethyl acetate, 1 part gasoline. The beans were flaked to a thickness of .020 inches thick. Most of the runs were made using flakes dried to 2.0 to 2.8 percent moisture. Two runs were made with flakes having 6.0 percent moisture by regulating the driers to avoid evaporating too much moisture. The extraction time was 20 minutes; drying time, 20 minutes. The solvent flow varied somewhat but averaged about 1380 ml. per hour. The extractor was heated to 70°C. The rate of extraction was 1.5 pounds of beans per hour. The oil in the meal varied from 0.5 to 5.0 percent, most runs producing some meal with 1.0 percent or less soil. While the finished oil showed no trichloroethylene to the taste, chemical tests indicated that it contained on the average of about one percent.

Conclusions

A solvent consisting of denatured alcohol mixed with trichloroethylene to give a specific gravity of 0.910 may be used successfully in a continuous extraction plant to remove oil from soybeans. The extraction is carried out at 70°C. and the miscella cooled to cause it to separate into two phases. The lower phase can be separated and

stripped to remove the solvent while the upper phase can be returned to the system without evaporation to extract more oil. The moisture content in the solvent will not build up if beans having less than 6 percent moisture are used and the drying is done with 10 p.s.i. steam pressure on the drier.

Literature Cited

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