Development of a plant for continuous counter-current extraction of soybeans with trichloroethylene

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DEVELOPMENT OF A PLANT FOR CONTINUOUS COUNTER-CURRENT EXTRAC TION OF SOYBEANS WITH TRICHLOROETHYLENE

by

Charles Edmund Kircher, Jr.

A Thesis Submitted to the Graduate Faculty for the Degree of

DOCTOR OF PHILOSOPHY

Major Subject Chemical Engineering

Approved:

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In charge of Major work

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Dean of Graduate College

Iowa State College
1940
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by gravity out of the tube R into the meal drier, as is the case when
the tube is inclined slightly from the vertical. Some form of mechanical
device would therefore have to be used to transfer the material from
the extractor to the meal drier.

Still another arrangement of the tube R with respect to the tube
Q should be considered. In this case the tube R is inclined slightly
from the vertical but in a direction away from tube Q. Figure 5 shows
the junction point of the two tubes in detail.

As the material leaving tube Q passes point B, the tendency is to
float up through the solvent in the tube R. However, due to the in-
clination of the tube, the solid particles rising vertically from the
point B impinge almost immediately against the inner wall of the tube.
This results in an accumulation of material in the tube R at a point
just above B. It can be seen that by inclining the tube R, as shown in
the sketch, practically no advantage is taken of the natural tendency
of the solids to float, in securing movement of material from the tube
Q into the tube R. Furthermore, with this arrangement there is no
chance for the material to float naturally in between the flights of the
conveyor in tube R. Instead, the material must be pushed out of tube Q
and into the conveyor in tube R. By comparing the arrangement shown
in Figure 5 with that shown in Figure 3 it is easily seen that in the
former case there is definitely more chance for stoppage and binding
of the conveyors at the junction point of the tubes Q and R. Therefore,
the arrangement as shown in Figure 3 is preferred. However, it should
not be inferred that the other arrangements shown are not workable.
SECTION OF EXTRACTOR

Fig. 5.
The configuration shown in Figure 6 where the tube R takes the position KL is one which is old in the extraction art. The defects inherent in this system are the same as those discussed in the preceding section in connection with the types of extraction apparatus used commercially. It can be seen by referring to Figure 6 and Figure 3 that the tube R would have to be considerably longer in the former case in order to maintain the same column of liquid in the extraction tube Q. Furthermore, the tube R would have to be longer than the tube Q because above the liquid level a zone must be provided for draining solvent from the extracted meal before it leaves the extractor. A further serious disadvantage in this arrangement is that difficulty may be experienced in securing movement of the solid material from the tube Q into the tube R, since the material will not tend to float from the one tube into the other as is the case when the tube takes the position shown in Figure 5.

The actual construction and operation of the extractor used in the pilot plant was as follows: The unit consisted of a relatively long inclined pipe connected at its lower end to a nearly vertical riser pipe. The inclined tube made an angle of 16 degrees with the horizontal, while the riser tube made an angle of 14 degrees with the vertical. Both legs of the extractor were in the same plane, and the angle between them was 60 degrees. Material movement through the unit was effected by solid flight conveyors mounted in each leg. The general arrangement of the extractor and its relationship to the other parts of the plant is shown in Photographs 5, 7, 8, 9, 10, 14, 15, and 17 of the Appendix.
SECTION OF EXTRACTOR

Fig. 6.
A detailed description of the various elements of the extractor follows. The tubes were extra heavy 6-inch pipe (I.D. = 5.76 inches). The overall length of the inclined leg was 25 feet and of the riser leg 13.5 feet. The approximate length of the extraction zone in the inclined leg was 22 feet and in the riser leg 7 feet.

The solid flight conveyor in the inclined leg had a pitch of 6 inches, while that in the riser leg had a pitch of 3 inches. If the conveyor had been centered in the inclined leg, the clearance between the conveyor and the tube would have been 1/16 inch. However, as no bearings were provided inside the tube, the shaft of the conveyor deflected so that at its lower end all of the clearance was above the top of the flight. In the beginning a spider bearing was provided for the conveyor shaft at the lower end of the inclined tube. This was removed after the first trial run, since it was found that plugging could occur as a result of material gradually accumulating around the bearing. Also the presence of the bearing prevented the conveyor flighting from extending to the end of the inclined tube.

The conveyor flighting was 3/16 inch thick, and was tack-welded to 1/2 inch standard pipe which formed the shaft. All driving gears and/or sprockets were mounted on 1 7/8 inch solid shafting which formed an extension to the pipe conveyor shaft. All packing glands and roller thrust bearings were mounted externally. The conveyors were operated by means of bevel gear and roller chain drives connected, in all cases, to a single power supply. Photographs 3, 4, 5, 6, 7, and 9 in the Appendix show the location of the power supply and the arrangement of the drives.
With the solvent flowing from B toward P and the solid material moving in the opposite direction, it is possible for fine particles of material to become suspended in the miscella leaving the extractor (Figure 2). This condition should be avoided as much as possible, as the processing of the miscella to recover the oil is somewhat complicated if solids are present. A method of removing the miscella from the extractor was therefore developed which tended to reduce the amount of solid entrainment in the miscella to a minimum. Narrow tapered slots were cut on the lower side of the extractor tube at the point where it was desired to remove the product miscella. These slots were cut so as to make a very narrow opening (approximately 1/32 inch) at the inside face of the tube and a wider opening (approximately 3/8 inch) at the outside face of the tube. The advantage of this type of miscella outlet was that it practically never clogged, as any particle small enough to enter the slot was washed through by the flow of miscella. However, in case the lowest slot did become temporarily clogged, the miscella could flow out of the next higher adjacent slot, without at the same time increasing appreciably the height of the liquid level in the tube. Actually little or no difficulty was experienced with solids plugging the outlet ports. In actual operation it was found that the flow of miscella through the slots, in conjunction with the wiping action of the conveyor flights across the inside face of the slots, sufficed to keep solids from lodging permanently in the outlet ports.

Loss of solvent either as liquid or vapor was prevented by placing
a housing around the miscella outlet. This housing was welded to the extractor tube, and was provided with a removable side which made possible easy access to the chamber. This was desirable for two reasons. (1) The slots could be easily and quickly cleaned by passing a thin metal spatula or similar instrument through the openings from the underside of the tube. (2) A wire screen suspended on a metal frame could be placed inside the chamber in such a way that the miscella leaving the extractor tube had to pass through the screen before going to the preheater. This proved to be an advantage during the time that filters were not used, as the screen removed an appreciable portion of the fine solids carried out of the extractor by the miscella. The screen could be cleaned without interrupting operations.

Two openings were provided at the upper end of the inclined extractor tube for admission of soybean flakes. One opening was located below and one above the miscella outlet described above. Both openings were on the top side of the tube, and were provided with flanges so that the flake hopper could be attached at either point. By experimentation it was found that the flakes could be fed in at either point; however, some irregularity in the rate of feed was experienced when the flakes were admitted below the miscella outlet. This was because of the fact that the tube was partly filled with miscella at that point. Because of the buoyant action of the liquid the flakes had to be fed in under a slight pressure, which was provided by maintaining a head of several feet of packed flakes above the inlet point. This column of flakes also acted as a very effective vapor seal, preventing the loss of solvent.
vapor. More satisfactory results were obtained in the pilot plant runs when the soybean flakes were fed into the extractor at a point above the miscella outlet. In this case the rate of feed from the flake hopper into the extractor was regulated by merely changing the diameter of the conveyor shaft at the point where the flakes entered. This was easily done by wrapping a piece of leather around the shaft and fastening the ends of the strip to the shaft. Using flakes prepared in the laboratory, the capacity of the conveyor when operated at 1.83 R.P.M., with no wrapping on the shaft, was approximately 105 pounds per hour. During most of the pilot plant runs, however, the conveyor shaft was wrapped so that when rotating at 1.83 R.P.M. only 85 pounds of flakes could enter the extractor per hour. The reason for the lower rate was that when the unit was operated at the rate of 105 pounds per hour, the resistance to flow of solvent through the extractor was such as to require a higher liquid head to be maintained in the riser leg than the construction of the unit would permit. The solvent level in the riser tube had to be kept several feet below the meal outlet spout in order to provide a drainage zone for the extracted meal. Based on pilot plant operations, a safe figure for the capacity of an extractor of the type described above is 15 pounds of soybean flakes per cubic foot of free extractor space.

The two tubes forming the legs of the extractor were connected at their lower ends by means of a flanged "Y"-shaped section. Lead gaskets were used in these flanged joints at the time the unit was assembled, and they were never changed, although later experience indicated that asbestos gasket material such as "Granite" would have been satisfactory. All
flanged joints except the two just mentioned were made up using "Granite" or similar asbestos-type gasket material. For sight glasses, etc., a composition-cork gasket was found to be satisfactory.

An outlet was provided in the flange forming the bottom of the riser leg, so that all solvent could be drained from the extractor if desired. This outlet was so constructed that it formed a small sump where tramp iron and foreign matter could accumulate without causing any harm. Periodically such material was removed from the trap.

The weight and thrust of the conveyor in the riser leg of the extractor were carried by a roller bearing located at the upper end of the shaft. The conveyor was centered at the lower end by means of a pin placed in the center of the bottom flange. A clearance of one inch was left between the lower end of the conveyor flight and the inside face of the bottom flange.

The fresh solvent entered the riser leg of the extractor at a point corresponding in elevation with the miscella outlet located at the top of the inclined extractor tube.

The outlet for the extracted flakes was located near the top of the riser leg, and was so constructed that the drained meal fell by gravity from the extractor tube into a short nearly vertical spout which connected directly with the No. 1 drier.

c. Meal drier and steamer units. These units consisted of horizontal steam jacketed tubes through which the meal was moved by means of a modified type of ribbon conveyor. Photographs 1, 2, 12, 13, 14, and 15 in the Appendix show the principal construction details of the
each conveyor. This was to avoid possible accumulation of solids at the
end of solid flighting with reverse thread was put at the discharge end of
the chain in photographs 12 of the appendix, approximately one turn
added to the ribbon flighting of the conveyor in the No. 2 dryer section.
With the same object in mind, short horizontal conveying bends were
added to the conveyors in the No. 1 dryer and steam dryer sections.
By the single ribbon, unaxially flighting of the pitch pitch
meant. In order to obtain more turning of the meal then would be reached
with type of flighting preferred only a narrow face for contact with the
meal. Flotation and starched to the contact sheet by means of tender coke.
Ribbon flighting was made from 1/8 inch red forming into a continuous
ribbon flighting. When the roll of material movement were
the theoretical rate calculated from the known pitch speed of the con-
veyor. However, the actual input rate of travel of meal was approximated one third of
the calculated input rate. The type of flighting used, it was found that the
speeds that were used had to be adjusted to meet the theoretical rates at 6.2 P.M. The type of flighting used, it was found that the
speeds that were used had to be adjusted to meet the theoretical rates at 6.2 P.M. Therefore, the conveyor were operated from a single power
after which the conveyor were operated from a single power
in order to reverse the direction of material movement
conveyor in the No. 2 dryer rotated in a clockwise direction but had a
conveyor in the No. 1 dryer rotated in a clockwise direction but had a
osmazone and conveyors in the No. 1 dryer and in the steam dryer
are presented below. The conveyors in the No. 1 dryer and in the steam dryer
information on the operation and construction of these units is
enough need in the pot plant, and Figure 7 is a diagrammatic draw-
Fig. 7.

AMES PILOT PLANT
MEAL DRYER & STEAMER - ASSEMBLY

NOTES:
ALL CONVEYORS MOUNTED IN STANDARD 6" STEEL PIPE. ALL STEAM JACKETS MADE OF STD 8" STEEL PIPE. ALL CONVEYORS RIBBON TYPE EXCEPT AS FOLLOWS:
5' OF SOLID FLIGHT WHERE MEAL ENTERS 1ST DRYER; 1'-6" OF SOLID FLIGHT WHERE MEAL ENTERS 2ND DRYER & STEAMER. RIBBON FLIGHT MADE OF 3/8" ROD.

ALL CONVEYORS 6" PITCH & OPERATED AT 5.7 R.P.M. - ACTUAL LINEAR RATE OF MOVEMENT THRU UNITS WAS APPROX. 1/3 OF THEORETICAL.
MEAL OCCUPIED APPROX. 30.0% OF THE FREE VOLUME OF 1ST DRYER. THE DRY MEAL OCCUPIED ABOUT 20.0% OF THE FREE VOLUME OF THE STEAMER.
end of the conveyor tube. Actual operation of the plant showed that
this special flighting was not essential.

The bolt holes in the end flanges of the drier and steamer sections
were made appreciably larger than the bolt diameter. In this way the
position of the conveyor in the tube could be adjusted by merely shift-
ing the end flanges slightly. In actual operation the conveyor just
touched the bottom of the tube. No hanger bearings were used inside of
the tubes as they would have interfered with material movement. External
packing glands were mounted on the end flanges. Graphited asbestos pack-
ing was used. Maintaining a vapor seal at the ends of the tubes was no
problem, as all units operated at approximately atmospheric pressure.

The thrust of each conveyor was taken by a Timken roller bearing
mounted externally at the drive end of the conveyor.

Steam pressure used in the jackets of the drier and steamer sections
during operation of the plant was 45-50 pounds per square inch gauge.
Wet meal entered the No. 1 drier at a temperature of about 25° C. and
the stripped meal left the steamer at a temperature of 115-118° C.

d. Miscella filters. During the preliminary test runs the miscella
from the extractor was not filtered before it was passed to the preheater
and still. Consequently, trouble was experienced due to accumulation
of solids in these units. In order to obtain satisfactory operation of
the preheater and still it was found necessary to process a solid-free
miscella. Bag filters were therefore installed and the suspended solids
removed from the miscella by filtration through 8 ounce canvas cloth.
Details of the construction and operation of these units follow.

Photographs 18 and 19 in the Appendix show the relation of the filters to the other pieces of equipment.

The filter shell consisted of a 2.5 foot length of 8 inch standard steel pipe. The filter bag was held at the top by the flanged head piece and rested at the bottom on a ring of 5 inch pipe. A liner made of coarse screen fitted inside the 8 inch pipe and kept the filter bag from sealing against the sides of the pipe. The miscella flowed by gravity from the chamber below the miscella outlet through a one inch line into the top of the filters. Flow through the filters was also by gravity, the filtrate collecting in a tank located directly below the filters. From here it was pumped at a controlled rate into the top of the preheater. Pressure on the outlet side of the filters and in the filtrate storage tank was always kept at atmospheric. No filter aid was used. At the end of each six to eight hour cycle the filter bag was one third to two thirds full of solids, and a head of 8 inches to 10 inches of miscella was held up in the top portion of the bag. This liquid level was never allowed to rise above the top flange of the filter. The sludge removed from the filters was weighed and analyzed to determine the amount of solvent, oil, and dry, fat-free meal present.

b. Miscella preheater and still. These units were purchased from the J. P. Devine Company. The tube sections of the preheater and still were identical and consisted of 19 brass tubes (3/4 inch O.D., #20 B.W.G., 5 feet long) mounted in a shell of 5 inch standard pipe. Steam at 15-25
pounds per square inch gauge was used on the shell side of these units. The only difference between the preheater and the still was that the latter had a short section of steam jacketed 5 inch pipe attached to the bottom flange of the tube section. The two units were placed one above the other and a vapor outlet was located in the spool piece which connected them. In the top section or preheater the miscella was heated from room temperature to about 100° C, by filming it down the inside of the heated tubes. Solvent was vaporized in this zone, and the vapors left through the outlet mentioned above. At the vapor outlet point a 3 inch line was provided, but this was reduced down to a 2 inch line which rose approximately 20 feet to the top of the condenser. This vapor line was jacketed to prevent condensation of steam and consequent refluxing of water back into the still.

A special type of baffle was placed above the tube section of the still in order to direct the flow of miscella from the preheater onto the tube sheet, which acted as a distributing plate. In the bottom section or stripping still the hot solvent-oil mixture from the preheater was again filmed down the inside of the heated tubes. However, in order to sweep the solvent vapors out of the tubes, dry steam was blown up through the filming zone counter-current to the flow of liquid. The dry steam entered the still at the bottom, bubbled through the oil which was about to leave the still, and then passed up through the filming section of the still. The lower end of the still consisted of a jacketed piece of 5 inch pipe which extended about 6 inches into a large open container filled with soybean oil. This container acted as a
settling basin for the solids carried through the preheater and still by the miscella. Being open at the top, the settled solids could periodically be removed from the trough without shutting down the still. The coil for the purging steam was located inside the extension of the still which dipped into the oil filled tank. An overflow line from the tank maintained the oil level constant. The tank could be moved up or down in order to vary the actual depth of immersion of the still bottom in the oil reservoir. During operation, as oil flowed into the tank from the still a corresponding amount of oil flowed from the tank through the overflow line. In this way the liquid level in the tank remained constant.

The oil in the tank, as well as the oil inside the steaming zone at the bottom of the still, was heated by the steam jacket placed around the pipe which dipped into the oil reservoir. The open type of still bottom was necessary in order to make possible the continuous removal of solids from the system.

The purging steam passed up through the tube section of the still and then to the condenser. Both the preheater and still were operated at atmospheric pressure.

f. Condenser. A tubular condenser, mounted in a vertical position, was used to condense all the vapors from the several solvent recovery units. The condenser was greatly over capacity, but was used as no smaller one was available. The construction was as follows: Shell 1 foot 7 inches I.D. x 1/4 inch thick x 6 feet 7 1/4 inches long; tubes – 48, 1 inch O.D. x #13 gauge x 6 feet 1 inch long, 75 square feet external
area. It was operated with water on the shell side and vapors in the tube. The advantage of this method of operation was that the condenser could be easily and quickly cleaned in case it became fouled as a result of entrainment of solids in the vapors from the meal driers.

g. Absorber. The noncondensable gases from the condenser were vented through an oil scrubber to remove solvent vapors. This scrubber consisted of a 10 foot length of 4 inch pipe mounted in a vertical position and packed with 3/4 inch Raschig rings. Soybean oil was fed in at the top of the absorber and the oil-solvent mixture from the bottom flowed by gravity into the preheater and still. In this way both oil and solvent were continuously recovered. During regular operation the flow of noncondensable gases through the scrubber was too small to be measured. However, during the starting up period the condenser was vented directly to the atmosphere until the air in the system was displaced by solvent vapors. The pressure drop through the absorber under normal conditions was less than one inch of water.

h. Power supply. The whole plant with the exception of the miscella pump, was operated by a 5 horse power motor. The drive arrangement was as follows: The 1700 R.P.M. motor was connected to a speed reducer (467 to 1 reduction) whose low speed shaft rotated at 3.72 R.P.M. Power from this jack shaft was transmitted to all the conveyors and the barrel valve by means of roller chain drives. For further details refer to photographs 3, 4, 5, 6, and 7 in the Appendix.
C. Results

1. General considerations

Approximately 20,000 pounds of soybeans were processed in the pilot plant during the course of the investigation. Actual operation of the plant showed that the basic design features of the extractor were correct. Due to the effective counter-current extraction realized in the unit, when using trichloroethylene, 95 per cent to 98 per cent of the oil present in the soybean flakes was removed in approximately 45 minutes contact with the solvent. This meant that the soybean meal produced contained about one per cent residual oil. The meal was of good quality and proved to be acceptable to animals as a feed. The product oil was comparable to commercial crude soybean oil, and on refining gave a clear, light yellow oil having the physical and chemical properties characteristic of commercial refined oil.

Total solvent losses during test runs averaged approximately 20 pounds per ton of soybeans processed, or one per cent of the weight of the material extracted.

2. Equipment design and operation

A complete description of the separate items of equipment used in the pilot plant is presented in the section on "Apparatus and Method of Procedure." In this section specific data will be given on the material processed, the products obtained, and the operation of the various pieces of equipment.
a. Characteristics of the soybean flakes.

(1) The individual flakes were 0.015 inch to 0.020 inch thick, and varied in size from 0.25 to 2 square inches in area.

(2) The bulk density of the loosely packed flakes was 25 pounds per cubic foot.

(3) The flakes contained from 3 to 5 per cent of fines passing 80 mesh screen.

(4) The density of the soybean is such that bean particles float in pure trichloroethylene (specific gravity 1.47) but sink in a 20 per cent solution of soybean oil in trichloroethylene (specific gravity 1.3).

(5) Soybeans are made up essentially of protein, carbohydrates and glycerides. The glycerides, or oil, constitute approximately 20 per cent of the dry weight of the bean. Small amounts of phosphatides, sterols, and mineral salts are also present.

b. Solubility of soybean oil in trichloroethylene.

(1) Soybean oil and trichloroethylene are miscible in all proportions.

(2) Solutions of soybean oil in trichloroethylene have a lower specific gravity than the pure solvent. Table 1 gives the specific gravity of solutions of various composition.
Table 1

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<th>Weight % oil in solution</th>
<th>Specific gravity $20^\circ/4^\circ$ C.</th>
<th>Weight % oil in solution</th>
<th>Specific gravity $20^\circ/4^\circ$ C.</th>
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<td>1.243</td>
<td>100</td>
<td>0.93 $15^\circ/15^\circ$ C.</td>
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</table>

a. Data on the extractor.

(1) During most of the test runs the input of flakes to the extractor was 35 pounds per hour. The maximum operating rate was 105 pounds of flakes per hour. Based on these figures the bulk density of the flakes as they passed through the extractor was approximated 15 pounds per cubic foot of free space.

(2) The limiting factor which determined the maximum loading of the conveyors in the extractor was the resistance to flow of solvent through the extractor tubes. This resistance was kept low enough so that gravity flow of solvent through the extractor could be realized.

(3) The time of contact between the soybean flakes and the solvent was about 45 minutes.

(4) By varying the weight ratio of solvent to flakes fed to the extractor, the oil content of the miscella was varied from 12 per cent to 28 per cent by weight.
(5) The miscella leaving the extractor contained from 3 to 5 per cent of solids in suspension, depending on the amount of "fines" in the flakes being processed.

(6) The meal leaving the extractor and entering the drier carried about 90 pounds of trichloroethylene per 100 pounds of dry, oil-free meal.

d. Data on the meal drier.

(1) Composition of the meal entering the drier. The following figures give the weight of water, oil and solvent associated with 100 pounds of dry, extracted meal.

Weight of water ——— 17.0 pounds (corresponds to 12% H₂O in the prepared flakes)
Weight of oil ——— 1.25 pounds (95% extraction)
Weight of solvent —— 90.0 pounds

(2) Composition of the meal leaving the drier. It was estimated that 90 per cent of the solvent and 50 per cent of the water present with the meal was vaporized during passage through the drier unit. The composition based on 100 pounds of bone dry, oil-free meal would then be as follows:

Weight of water——— 8.5 pounds
Weight of oil——— 1.25 pounds
Weight of solvent——— 90.0 pounds

(3) Temperature of the meal entering the drier. Extraction was carried out at room temperature so a value of 25° C. may be taken for the temperature of the meal entering the drier unit.
(4) Temperature of the meal leaving the drier. The meal leaving the drier was heated to about 100° C. The drier operated at atmospheric pressure.

(5) Amount of solvent and water vaporized in the drier.

Based on 100 pounds of bone dry, oil-free meal the approximate weight of solvent and water vaporized in the drier was as follows:

Trichloroethylene = 72 pounds
Water = 8.5 pounds

(6) Thermal data used in making calculations.

(a) Trichloroethylene:

Boiling point at 760 mm. = 86.7° C.
Specific heat at 20° C. (liquid) = 0.223 B.T.U./lb./° F.
Latent heat of vaporization (at B.P.) = 103.1 B.T.U./lb.
Steam distillation temperature = 73° C.

(b) Soybean oil:

Mean specific heat (25° C. – 100° C.) = 0.5 B.T.U./lb./° F.
Non-volatile at temperatures used.

(c) Soybean meal:

Mean specific heat (25° C. – 100° C.) = 0.4 B.T.U./lb./° F.

(7) Heat transfer coefficients.

Type of drier used:

Horizontal steam jacketed tubes.
Ribbon flight conveyors.
Jacket steam pressure 45-50 pounds per square inch gauge.
Meal occupied 25-30 per cent of the total free volume of the drier tube.

Under these conditions an overall heat transfer coefficient of 3-4 B.T.U./hr./sq. ft./° F. (based on total inside heating area) was realized.
(8) Capacity of the drier.

(a) Wet meal entering:
Due to the fact that the solvent wet meal as it entered
the No. 1 drier was bulky and did not tend to pack down,
the loading of the drier at this point was about 6
pounds of meal (calculated on the dry, oil-free basis)
per cubic foot of total free volume.

(b) Dry meal leaving:
As a result of changes in bulk volume as the meal
dried, the No. 2 drier operated so that about 8 pounds
of meal (dry, oil-free basis) could be put through per
cubic foot of total free volume, without overloading
the conveyor.

(9) Vapor velocity in the drier.

The linear velocity of vapors in the drier did not exceed
2 feet per second. Even under these conditions there was
some entrainment of very fine particles in the vapors
leaving the unit.

(10) Temperature limitations.

Soybean meal is not injured by being heated well above
100° C. so no problem was created by operating the drier
at atmospheric pressure and heating the meal to 100° C.

e. Data on the meal steamer.

(1) Composition of the meal entering the steamer. This is the
same as the composition of the meal leaving the drier. For
data see section d-(2) above.
(2) Composition of the meal leaving the steamer. The meal was free of solvent and had the following approximate composition based on 100 pounds of dry, oil-free meal.

- Weight of water = 3 to 4 pounds
- Weight of oil = 1.25 pounds

(3) Temperature of the meal entering the steamer. The meal entered from the drier at about 100°C.

(4) Temperature of the meal leaving the steamer. In passing through the steamer the meal was heated to 115-118°C.

(5) Amount of solvent and water vaporized in the steamer. Based on 100 pounds of bone dry, oil-free meal the weight of the solvent and water vaporized in the steamer was as follows:

- Trichloroethylene = 8 pounds
- Water = 5.5 pounds

(6) Thermal data used in making calculations. Refer to section 1, item (6).

(7) Heat transfer coefficients. Type of steamer used:

- Horizontal steam jacketed tube.
- Ribbon flight conveyor.
- Jacket steam pressure 45 to 50 pounds per square inch gauge.
- Meal occupied 20 per cent of the total free volume of the tube.

Under these conditions an overall heat transfer coefficient of one B.T.U./hr./sq. ft./° F. (based on total inside heating area) was realized.
(8) Capacity of the steamer. The steamer can be loaded so that 10 pounds of meal (dry, oil-free basis) can be put through for each cubic foot of free space in the steamer.

(9) Vapor velocity in the steamer. Dry steam was used as the purging vapor, and it passed through the unit counter-current to the movement of the meal. The minimum amount of steam which resulted in complete removal of solvent from the meal was used. The linear vapor velocity was not more than one foot per second.

f. Data on the miscella filters.

(1) Weight of miscella filtered per hour. The maximum flow of miscella to the filters was about 130 pounds per hour, and the minimum was about 75 pounds per hour, depending on the concentration of the miscella produced.

(2) Solid entrainment in the miscella. Refer to section e, item (5) above. The amount of solids carried out of the extractor by the miscella was primarily determined by the condition of the flakes fed to the extractor. For a definite amount of fines in the flakes, the concentration of solids in the miscella depended on the weight of miscella produced per 100 pounds of flakes extracted. In other words the entrainment of solids in the miscella was almost entirely determined by the physical condition of the material being extracted, and not by the rate of flow of miscella from the extractor.
(3) Bulk density of the filter cake as formed. The wet cake, saturated with a 20 per cent miscella, weighed approximately 70 pounds per cubic foot.

(4) Nature of the filter cake. The solids filtered out of the miscella consisted of fine bean particles which still contained an appreciable amount of oil. The cake before washing with fresh solvent contained 16-30 per cent oil and 45-55 per cent volatiles, depending on operating conditions. The washed cake held up 80-100 pounds of trichloroethylene per 100 pounds of dry solids.

g. Data on the miscella preheater.

(1) Amount of miscella processed per hour. The maximum flow to the preheater was about 130 pounds per hour, and the minimum was about 75 pounds per hour.

(2) Composition of the miscella entering the preheater. This was determined by the ratio of solvent to flake input to the extractor, and varied from 12-20 per cent oil depending on operating conditions.

(3) Temperature of the miscella entering the unit. In general this was room temperature or about 25° C.

(4) Temperature of the miscella leaving the unit. The temperature of the concentrated miscella leaving the preheater was about 110° C. At this temperature it had the approximate composition of 60 per cent oil, 40 per cent trichloroethylene.
(5) Vapor velocity in the preheater. It was important to prevent entrainment of liquid in the vapors leaving the unit, and therefore linear velocities were kept below 5 feet per second in the tubes and at the vapor outlet.

(6) Steam pressure used. Jacket steam pressure was 15-20 pounds per square inch gauge.

(7) Temperature limitations for the preheater. No critical temperature is recognized. However, it is known that the color of soybean oil may be darkened by prolonged heating, even at temperatures of 100° C., if in the presence of air or in contact with fine particles of soybean. Therefore it is desirable to use the lowest working temperature which will give good operation.

h. Data on the miscella stripping still.

(1) Temperature of the oil leaving the still. The essential requirement in the still was that no steam be allowed to condense in the unit. The oil temperature was kept between 100° C. and 110° C.

(2) Solvent vaporized in the still. Approximately 67 pounds of trichloroethylene were vaporized for each 100 pounds of oil. The heat required for this was supplied by indirect heat, and not by the steam used to displace the solvent vapors from the unit.

i. Data on the condenser.

(1) Operating pressure in the condenser. The unit was operated at atmospheric pressure.
(2) Temperature of the condensate leaving the condenser.

With cooling water available at about 15°C, the condensate left the condenser at a temperature of 20-25°C.

(3) Noncondensible gases entering the condenser. In starting up the plant solvent vapors displaced the air present in the system. Once the plant was in regular operation the only noncondensible gases entering the system came in with the steam, and therefore, the amount of such gases was negligible. Provision was made, however, to vent all off-gases from the condenser through a small oil scrubber to recover the solvent vapors.

(4) Heat transfer coefficient. For a unit in which the vapors condense inside vertical tubes with cooling water on the shell side, an overall coefficient of 120 B.T.U./hour/sq. ft./°F. can be realized.

j. Data on the solvent-water separator.

(1) Temperature of the liquid entering the separator. This was the same as the temperature of the condensate leaving the condenser, and in general was 20-25°C. Trichloroethylene, being heavier than water, formed the bottom layer in the separator.

(2) Solubility data. Solubility of trichloroethylene in water at 25°C.

0.17 pounds per 100 pounds.
Solubility of water in trichloroethylene at 20° C.

0.03 pounds per 100 pounds.

(3) Solids carried into the separator. Under normal operation few solids entered the separator with the condensate. However, if soybean flour was carried out of the meal drier and steamer by the vapors leaving these units, such solids could pass through the condenser and into the solvent separator. This caused no little trouble.
The rate of evaporation from the extractor was not as

B. Experiments

...the ether are removed and the extractable fraction are removed...

...the experiments indicate the problem involved in removing the ether before fractionation....

Laboratory experiments showed that no extractable was removed by

...because for each operation a solution is made to control the moisture content and temperature of the

...the solution is needed to form the thicken... (approached maturity in the thirteenth...)

...large diameter smooth... into the thirteenth thirteenth... large diameter smooth roll... to be 12 inches diameter... small diameter corrugated roll... (9 inches)

...equipment for commercial use. This equipment differs from that described

in the large-scale production or so many times and have developed

...many manufacturers have made a thorough study of the problems involved...

...fundamental... important step in the extraction process... several... 

...in the industrial and no equipment was bought for this specific... experimental procedure was determined by the type of equipment available...

The method of preparing so many times described in the section on

A. Plant Operations

II. DISCUSSION
well regulated and controlled as the input of flakes. The reasons for this were as follows: (1) In order to use gravity flow of solvent to the extractor, the condenser and solvent-water separator were placed at a higher elevation than the solvent inlet to the extractor. All vapors from the miscella preheater and still, and the meal driers and steamer, passed upward to the condenser, and the condensed solvent flowed by gravity from the condenser to the separator and then directly into the extractor. Any irregularity in the flow of solvent vapor to the condenser showed up immediately as a change in the rate of solvent flowing back into the extractor. (2) The solvent-water separator was integral with the condenser, and operated under the same pressure as the condenser. If for any reason this pressure suddenly increased, as it did at times, the rate of flow of solvent from the separator to the extractor would momentarily increase. (3) The gravity head for flow of solvent from the separator to the extractor was only about 2 feet. When at times the level of solvent in the riser leg of the extractor rose 1 to 2 feet above the solvent inlet point, (as a result of variation in flow resistance through the extractor) the rate of flow of solvent from the separator would fall almost to zero.

The irregularity in flow of solvent to the extractor, discussed above, created several other operating problems. The composition of the miscella is largely determined by the ratio of solvent to flake input, and as the rate of input of solvent varied considerably, the concentration of the miscella also varied over a wide range. This variation in turn influenced the rate of extraction as well as the overall per cent extrac-
tion of oil from the soybean flakes. The action of the conveyors in
moving solids through the extractor was also influenced by the irregular-
ity of solvent input to the unit. During periods when the input of
solvent was so low that the miscella actually stopped flowing for a period
of 15 to 30 minutes, trouble usually followed due to packing of the dry
flakes in the upper end of the inclined leg of the extractor.

C. Meal Driers and Steamer

When fabricating the meal driers and steamer, provision was made
for vapor outlets at the meal discharge end of each drier section and at
the meal entrance end of the steamer section. Operation of the plant
showed that with the condenser located about 6 feet above the top drier
most satisfactory results were obtained when only the vapor outlet on
the No. 1 drier was used. This meant that the section designed to be
the No. 2 drier was in effect both a drier and a steamer. The original
1\(\frac{1}{2}\) inch vapor line on the No. 1 drier was replaced by a 3 inch line and
later by a 5 inch line when it was found that best operation was realized
when all vapors were removed at that point. It was important to keep
vapor velocities low in the tubes and at the vapor outlet in order to
avoid serious entrainment of fine meal particles in the vapor stream.

Provision was originally made to introduce purging steam to the
steamer through small ports spaced at about 3 foot intervals along the
bottom of the conveyor tube as shown in photograph 16 of the Appendix.
This method of operation was soon discontinued in favor of one using a
single inlet for superheated steam located at the meal discharge end of
the steamer. The disadvantages of the first method were: (1) Continual plugging of the small inlet ports by meal flour. (2) Difficulty in cleaning the ports during operation. (3) High vapor velocity at the inlet ports increased the tendency to solid entrainment in the vapors leaving the steamer. The purpose of the purging steam was to maintain a nearly solvent-free vapor phase in the steamer. This allowed the last traces of solvent to vaporize out of the meal. The steam continually displaced the solvent vapors and carried them over to the condenser.

During the first test runs it was found that plugging of the conveyor in the No. 1 drier could occur at the point where the wet meal entered, due to packing of the meal between the conveyor shaft and the ribbon flighting. To prevent any accumulation of wet meal at this point and insure positive and uniform movement of material, a short section of solid flighting was substituted for the ribbon flight. When this proved to be effective in eliminating the trouble, the same change was made in the No. 2 drier and the steamer so that in all cases the conveyors had a short section of solid flighting at the point where meal entered the conveyor. Beyond this point, the ribbon flighting proved to be entirely satisfactory for obtaining material movement.

The most important factor in obtaining good operation of the conveyors, and consequently uniform movement of material, was to prevent the meal from becoming wet with water. Soybean meal when wet with trichloroethylene will not stick to the conveyors or tube walls, but when wet with water it becomes pasty and on drying sets up to a hard mass. It is therefore necessary in designing equipment of this type to avoid
"cold spots" where condensation of water vapor might occur with consequent wetting of the meal. In nearly every case where failure of the barrel valve (located at the meal outlet end of the steamer) occurred it was due to steam condensing in the valve and wetting the meal.

Experiments showed that all vapor outlets should be located either at the end of the conveyor tubes or in the vertical pipe connecting adjacent tubes. The main objection to placing a vapor outlet at some point between the ends of a conveyor tube is that such an outlet could easily become plugged if for any reason the conveyor was overloaded even for a short period of time. The action of the conveyor is such that solids would be forced up into the vapor outlet if the level of material in the tube became abnormally high due to a temporary overload.

In the section describing the pilot plant equipment it was pointed out that, in the initial construction of the conveyors for the meal drier and steamer sections, special auxiliary flighting was provided for turning the meal and thereby increasing the rate of drying. While this action was realized to a certain extent, the effect was not great, and it was decided that for a commercial installation the auxiliary flighting would not be necessary. A standard ribbon type conveyor is recommended for use in the meal drier and steamer sections, and in actual practice the ribbon flighting should be made from flat rather than round stock. The latter was used in fabricating the pilot plant equipment only because it simplified construction.
D. Miscella Filters

In order to use gravity flow of miscella from the extractor to the preheater and still, thereby avoiding the use of liquid pumps, the installation of a miscella filter was deferred until after a number of experimental runs had been made with the pilot plant. The results of these runs may be briefly stated as follows: (1) When the miscella going to the preheater and still contained entrained solids, gradual fouling of these units resulted due to settling out of the solids. (2) Periodic shut-downs were required in order to clean out the preheater and still. (3) The color and quality of the oil appeared to be adversely affected by being heated in contact with partly charred bean material.

The amount of solid entrainment in the miscella was greater than had been anticipated in designing the pilot plant because of the use of small-scale equipment for preparing the soybean flakes. (See section on flaking equipment above.)

When it was observed that the operation of the preheater and still was impaired as a result of processing a miscella containing 3 - 5 per cent of solids, the experiment was tried of installing a settling tank between the preheater and still. While this helped some it did not permit successful operation to be realized. After several trial runs the settling tank was removed. Finally two bag filters were installed in such a way that one could be cleaned while the other was in operation. The filtered miscella was practically free of solids and was pumped at a uniform rate into the preheater. The installation of the filters and
In the section on filters, the experiment was not particularly successful when the paper was to insert a settling tank between the two units in an attempt to remove the bulk of the suspended solids from the mixture as mentioned.

However, a slight addition into the top of the still, the greatest change made and the hot mixture from the bottom of the separator flowed by gravity.

The first indication of the separator and still were outlined separately.

When the concentration of oil was as low as 12 per cent by weight, it was apparent that the flow of mixture from the extractor was still not completely separated to handle the flow of mixture from the extractor even.

Despite these defects, the units had not been perfectly vertical. Despite these defects, the units had not been perfectly vertical.

2) The pressure through the ethylene glycol was so that they would move uniformly throughout the mixture and made the mixture almost uniform.

1) Accumulation of solids on the top tube sheet and in the tubes pre-

According to the test condition was not realized for the following reasons:

The mixture of the mixture was supposed to flow by gravity into a.

Filter, Preheater and Still

Because throughout the preheater and before pumping it into the separator, the mixture might be realized in certain cases. By utilizing the mixture entered from the oil. Some improvement in operation and quality of product that these may have been through out solution of the solvent was stripped however, still appeared in the product and Important experiences indicated the mixture pump improved the operation of the plant. Some solids.
ful and the tank was removed after several test runs. Finally, the pre-
heater was placed directly above the still and connected to it by means
of a short section of pipe flanged at both ends. The vapor outlet for
both units was located in this connecting spool piece. Solvent vapors
from the preheater flowed down while vapors from the still flowed up to
the common vapor outlet. With this arrangement, it was impossible to
study the operation and efficiency of each unit separately. This fact,
in conjunction with the general uncertainty of operation due to accum-
ulation of solids in the tubes of the preheater and still units, made
calculation of overall heat transfer coefficients almost impossible.
Based on pilot plant experience, a filming type of miscella preheater
and still is not recommended unless a special type of unit is designed
which will work effectively even though conditions of operation vary
widely.

It should be pointed out that under the conditions of operation
discussed above traces of solvent were present at times in the product
oil. However, all solvent can be removed from the oil if adequate and
proper counter-current steaming is realized in the stripping still.
V. CONCLUSIONS

A. Equipment Required for Preparing Soybeans for Extraction

In general one or more of the following operations must be performed in order to put the beans in suitable condition for extraction.

1. Removal of all foreign matter from the beans.
2. Mechanical treatment to reduce the beans to the required particle size.
3. Adjustment of the moisture content of the beans by adding or removing water.
4. Adjustment of the temperature of the beans in order to carry out any of the above operations or in preparation for the extraction.
5. Addition of any activating agents required.

As a specific example the equipment used commercially in preparing soybeans for extraction is listed below. The order given corresponds to the position of the equipment in a flow sheet.

1. Cleaning unit for removing foreign material from the beans.
2. A magnetic separator for removing tramp iron from the beans.
3. One, or in some cases a two pair high, set of corrugated rolls (9 inches - 12 inches diameter, Le Page cut) for breaking the whole bean into pieces suitable in size for flaking. In general each bean should be divided into 4 to 6 pieces.
4. A unit for adjusting the moisture content of the cracked beans to obtain a product containing 10 - 13 per cent moisture.
5. A unit for heating the cracked beans to 120° F. without at the same time lowering the moisture content below 10 per cent.

6. A pair of large diameter smooth rolls for preparing flakes having a thickness between 0.010 inch and 0.015 inch. It is essential that conditions be adjusted so that little or no bean flour is formed during the flaking operation. In general the larger the diameter of the rolls, the better the type of flake produced.

B. Equipment for Carrying out Continuous Counter-Current Extraction

A complete discussion of the type of extractor developed for use with high specific gravity solvents such as trichlorethylene has been presented. In principle it consisted in a long inclined tube joined at its lower end to a shorter nearly vertical tube. Material was moved through the tubes by means of screw conveyors, while at the same time solvent was made to flow through the system in a counter-current direction, thereby effecting the desired extraction. A drawing of this type of unit has already been presented in Figure 2. Details on construction and operation of the extractor are given below.

1. Movement of solids and solvent through the extractor must be counter-current. The solids should enter near the top of the long inclined leg and the solvent should enter at a point in the riser leg located about 8 feet below the outlet for the extracted meal. The miscella should leave through a special outlet located near the flake inlet on the long extractor leg.

2. The long leg of the extractor should be mounted so that it makes an angle of about 15 degrees with the horizontal, while the shorter
riser leg should be placed so that it makes an angle of about 15 degrees with the vertical.

3. The bean flakes can best be moved through the extractor by means of solid flight screw conveyors. The pitch of the conveyors should be approximately one half the inside diameter of the extractor tube. Clearance between the conveyor and the inside wall of the tube should be 1/16 inch - 1/8 inch, depending on the size of the tube. No intermediate hanger bearings should be used to support the conveyor shaft in either leg of the extractor.

4. The length of the long inclined tube should be such that, for the conveyor speed used, the solids in passing through this section will be in contact with the solvent for the required length of time. In the case of soybean flakes this time is about 50 minutes. With the extraction time specified, the capacity of the unit is determined by the free volume in the liquid zone of the long leg of the extractor.

5. The angular speed of the conveyor in the riser leg should be 1.2 to 1.5 times that of the conveyor in the inclined leg of the extractor.

6. The screw conveyors should be made up of several sections coupled together so that they can be easily removed from the unit.

7. The flake inlet should be located as near as possible to the upper end of the long extractor tube. The vertical spout from the feed bin should connect directly to the extractor tube. The opening in the tube at this point should be 1 to 1.5 times the conveyor pitch in length, and the full diameter of the tube in width. The feed hopper should be placed several feet above the tube so as to maintain a
vapor seal by means of a column of loosely packed flakes. In order
to avoid overloading the conveyor in the long leg of the extractor,
the diameter of the conveyor shaft can be increased, at the point
where the flakes enter, by wrapping it with leather or similar
material. This wrapping should extend to a point just below the
miscella outlet. By changing the diameter of the conveyor shaft at
the feed point, the capacity of the conveyor can be easily varied.
However, it should be borne in mind that the tighter the material
is packed in the flights of the conveyor, the greater the resistance
to flow of solvent through the extractor.

8. The miscella outlet should be located on the bottom side of the
extractor tube at a point one to two feet below the flake inlet point.
The miscella can flow out of the unit either through narrow "V"
shaped slots, cut on the under side of the tube, or through a wire
screen. These should be so designed that practically all solids are
retained in the extractor and only liquid can pass out. The outlet
should be enclosed in some way to prevent vapor and liquid losses.
This housing should be provided with a removable door, and sight
glasses should be installed for observing the flow of miscella at all
times.

9. The thrust bearing and drive for the conveyor in the long leg of the
extractor should be located externally at the upper end of the unit.
A bearing is not provided inside the tube for the lower end of the
conveyor.
10. The thrust bearing and drive for the conveyor in the riser leg of the extractor should be located externally at the upper end. A guide pin for centering the conveyor shaft at the lower end is located in the bottom flange plate. A clearance of about one inch should be left between the end flight of the conveyor and the flange plate. A trap and drain outlet must also be provided at the lower end of the riser tube.

11. The solvent inlet line should be installed so that when the flow of solvent is stopped solids from the extractor cannot float out into the line and plug it up. Solvent should be pumped into the extractor through a metering device, and the flow carefully regulated. Solvent should enter the extractor at a point about 6 inches below the operating liquid level in the riser tube.

12. A gauge glass must be provided on the riser leg for observing the level of solvent in this side of the extractor during operation.

13. It is important to make provision in the riser leg for a head of solvent sufficient to produce flow of solvent through the extractor. An allowance of 3 to 4 feet should be made for this. A draining zone of about 5 feet must be provided between the operating solvent level and the point where the meal leaves the extractor. The conveyor flight in the riser leg should extend just up to the meal outlet spout and then stop. The outlet from the extractor to the meal drier should be the full diameter of the riser tube. The meal flows by gravity from the extractor to the drier, and the connecting pipe should make an angle of less than 30 degrees with the vertical.
Provide a sight glass for observing the movement of the extracted meal at the point where it leaves the extractor.

14. Sampling outlets should be provided at intervals along the side of the extractor tube.

C. Equipment for Separating Solids from the Miscella

In general, the miscella leaving the extractor will carry some solids in suspension, and these must be completely removed before passing the miscella to the preheater and still. The filter used for this operation should meet the following requirements:

1. Adequate provision for precoating the filter cloth if necessary.
2. Easy and thorough washing of the filter cake with hot or cold solvent at the end of each operating cycle. Make provision to pass hot solvent vapors through the filter cake after washing with hot solvent. It is essential that provision be made to remove the filter cake without losing solvent either as liquid or vapor.
3. The filter, if of the batch type, must have sufficient capacity so that it can be used for at least 8 hours without being cleaned. Maximum cake thickness for a batch filter will, in general, be about 1.0 inch.

D. Equipment for Separation and Recovery of Solvent and Oil from the Miscella

In order to obtain the pure extracted oil, the filtered miscella
is first heated to a temperature of about 100° C. in a unit which will 
be called the miscella preheater. In the preheater the major portion of 
the solvent is distilled out and is passed to a condenser for recovery. 
In the event solids are thrown out of solution as a result of concentrat-
ing the miscella, it may be necessary or desirable to filter again before 
passing the hot miscella to the stripping still. In the latter unit the 
temperature of the miscella is maintained at about 110° C. while it is 
steamed counter-currently to remove all solvent from the product. A 
detailed discussing of the equipment follows:

1. Miscella preheater

   a. The miscella from the extractor should be filtered to remove 
entrained solids, and then it is ready to be treated to recover the pure 
oil and solvent. In the operation of the preheater it is important to 
take into consideration the following facts: If the extract is a liquid 
such as soybean oil, there will be a definite equilibrium concentration 
of solvent in the oil when the solution is heated to say 110° C. in 
the presence of pure solvent vapor. For example, if a dilute solution of 
soybean oil in trichloroethylene is heated to 110° C. in contact with 
pure solvent vapor at atmospheric pressure, the liquid phase at 
equilibrium will have the approximate composition 60 per cent oil, 40 per 
cent trichloroethylene. Therefore, the amount of solvent to be vaporized 
in the preheater is determined by the initial concentration of the 
miscella, since the final concentration is a constant for any given set 
of operating conditions. The preheater must be designed to handle the
most dilute miscella that will be produced. In other words, the preheater
must be calculated on the basis of maximum extractor capacity and minimum
miscella concentration. For a given amount of oil, the heat required in
the stripping still will be a constant, but in the preheater the heat
demand will vary with the concentration of the incoming miscella.

b. In general the miscella from the extractor after filtering will
be stored in a small tank and will be at room temperature (25° C.). From
the tank it should be pumped through a rotometer (or other flow meter)
to the preheater. If desired, a small heat exchanger can be provided
ahead of the preheater for raising the temperature of the liquid from
25° C. to about 90° C.; however, this is not necessary.

c. The preheater must have sufficient capacity to raise the
temperature of the incoming miscella to 110° C. and supply the heat for
vaporizing the solvent which will automatically distill out as the
temperature is raised.

d. Easy escape of the vapors from the liquid in the preheater must
be provided. It is important to keep vapor velocities low (below 6
feet per second) in order to reduce the possibility of liquid entrainment
to a minimum. If necessary an entrainment trap can be placed in the vapor
line leading to the condenser.

e. The preheater should be operated at atmospheric pressure unless
the resulting temperature is considered too high for the oil, in which
case vacuum may be used.

f. A sight glass should be provided in the top section of the pre-
heater for observing its operation. Connections should also be made for
reading the temperature of the vapor and liquid leaving the preheater, and the operating pressure within the unit.

g. In order to avoid heat losses and prevent condensation of vapors, the unit should be well insulated.

h. The concentrated miscella should leave the preheater through a running trap which will act as a vapor seal.

i. The preheater should be so placed in relation to the still that the miscella can flow continuously by gravity from the former unit into the latter. In certain cases it may prove desirable to filter the miscella after it leaves the preheater, in order to remove precipitated solids.

j. Steam pressure required for the preheater is about 15 pounds per square inch gauge.

k. A falling film type of preheater is not recommended, as uniform operation of such a unit is difficult if not impossible to obtain.

l. In order to avoid foaming in the preheater it is essential that the miscella be kept dry (i.e., no free water should be present).

m. Steel construction is recommended.

2. **Stripping still**

a. The miscella from the preheater should either pass continuously by gravity into the still or be filtered and then pumped at uniform rate into the still. It should enter at a temperature of 110° C., and will have a definite and uniform composition corresponding to its temperature. The size of the still is determined by the amount of oil to be processed,
as each pound of oil will have associated with it a definite weight of solvent which must be stripped out.

b. A bubble cap column may be used in order to obtain good counter-current stripping of the oil. Dry or superheated steam should be used as the sweeping vapor. The time required for stripping, and the amount of steam to be used will in general have to be determined by experimentation. In the case of soybean oil it should be steamed counter-currently for about 30 minutes.

c. The construction of the unit should be such that it can be easily opened for inspection or cleaning.

d. Heat for vaporizing the solvent in the still must be supplied by auxiliary heating units and not by the steam used as the purging vapor. Condensation of steam in the product should be avoided. Fifteen pounds steam pressure is sufficient for the heating elements, as in general the oil need not be heated above 110° C. The maximum auxiliary heat requirement will be at the point where the miscella enters the still, as vaporization of the solvent is most rapid in that section of the column.

e. The still should be operated at atmospheric pressure unless a temperature of 110° C. is excessive for the oil being processed, in which case vacuum can be used and the temperature lowered.

f. In order to avoid heat losses and prevent condensation of the purging steam, the unit must be well insulated. In most cases it is essential that the oil be kept dry (i.e., free of water) if satisfactory operation is to be realized. The presence of water in oil will cause foaming.
g. The still should be so placed that the miscella from the preheater can flow by gravity into the still, and the product oil leaving this unit can flow by gravity into the storage tank. In order to prevent loss of vapor a running liquid seal should be provided in the inlet miscella and outlet product lines.

h. Provisions should be made for the following readings to be taken:

(1) Temperature of the miscella entering the still.
(2) Temperature of the product leaving the still.
(3) Temperature of the purging steam entering the still.
(4) Temperature of the vapors leaving the still.
(5) Pressure in the bottom section of the still.
(6) Pressure in the top section of the still.

i. It is important to keep vapor velocities low (below 5 feet per second) in order to reduce the possibility of liquid entrainment to a minimum. If necessary an entrainment trap can be placed in the vapor line leading to the condenser. Place a sight glass in the top of the still in order to observe if foaming or excessive entrainment occurs.

j. Steel construction is recommended.

E. Equipment for Separation and Recovery of Solvent and Product from the Extracted Meal

The extracted meal containing solvent must be heated and then countercurrently steamed in order to produce a desirable product and at the same time recover all the solvent. This can be done in various types of equip-
ment, one type being horizontal steam jacketed tubes through which the material is moved by means of screw conveyors. The unit in which the material is heated from the temperature of extraction up to 100° C. is called the drier, while the unit in which the heated material is counter-currently steamed is referred to as the steamer. An outline of the requirements and specifications on the above mentioned type of equipment is given below.

1. Heat drier

a. The function of the conveyors is to move the material uniformly through the steam heated tubes, and at the same time effect sufficient turning of the material to obtain good heat transfer. This is accomplished in the following way. A short section of solid flight conveyor is provided at each point where material drops into a drier tube. This solid flighting extends about 2 feet beyond the inlet opening so that no accumulation of solids can take place at that point. The section of solid flighting joins a section of ribbon flighting which extends to the point where the material drops out of the drier. A maximum length for each drier section is about 25 feet and the number of sections required is determined by the total amount of heating surface needed. The conveyors may be either right- or left-hand thread, depending on the direction of rotation of the shaft; however, right-hand thread is standard. The width of the ribbon flight should not be less than 7 per cent of the inside diameter of the tube. When such a conveyor is used in a tube filled 20 - 40 per cent of capacity, the actual lineal travel of material
through the unit is approximately 30 per cent of the theoretical rate calculated from the pitch and the speed of the conveyor. The bars holding the ribbon to the central shaft should be arranged so that a rod can be passed the entire length of the conveyor between the ribbon and the shaft. No intermediate hanger bearings should be used to support the conveyor shaft. External bearings at each end of the conveyor are sufficient, as the conveyor flighting rests lightly on the bottom of the drier tube.

b. The vertical pipe connecting two drier sections should be approximately the same diameter as the drier tubes. The parallel drier tubes should be spaced as close together as drives, etc., will permit. Inspection or clean-out ports can be provided in the pipes connecting drier sections.

c. The size and number of drying sections required should be based on the following considerations. In the drying zone the soybean meal must be heated from entering temperature (25°C.) up to 100°C., and in so doing the major portion of the solvent will be vaporized. Jacket steam pressure can be varied, but should be 15 to 20 pounds per square inch gauge if trichloroethylene is the solvent used.

d. The vapor outlet to the condenser should be located at the exit end of the meal drier, thereby providing for parallel movement of solids and vapors through this unit. In order to avoid entrainment of solids in the vapors leaving the drier, linear velocities should be kept below 2 feet per second. Vapor outlets should be located at the end, never in the middle, of a tube section. If possible, the vapor line should take
off at a point beyond the spout where the meal drops from the drier into
the steamer. Vapors from the drier should pass down to the condenser and
not up. If necessary, a cyclone separator can be installed in the vapor
line leading to the condenser in order to remove entrained solids. The
pressure drop through this unit should be kept at a minimum, and provi-
sion made to avoid condensation of vapors in the separator.

e. It is important to avoid having "cold spots" in the drier sec-
tions and connecting pipes as condensation of water vapor may cause
trouble. Such spots can be avoided by proper design of the steam jackets,
and by the use of insulation.

f. Provision must be made for measuring the pressure and temper-
perature at the following points inside the drier sections: Temperatures at
the exit end of each horizontal drier section; pressures at the entrance
and exit end of the No. 1 drier, and at the exit end of all other drier
sections. Operating pressure readings should be in the order of 1 to
2 inches of water, and a sudden increase in pressure at a particular point
indicates that a plug has formed in the conveyor between that point and
the vapor outlet. Plugs may also be located, and opened up, by passing
a rod through the conveyor between the ribbon and the central shaft. A
small port in the flange at the exit end of each drier tube should be
provided for this purpose.

g. A pressure regulator should be installed in the steam line to
the drier jackets so that easy and rapid adjustment of steam pressure can
be made.

h. The steam jackets should be tested for a working pressure equal
to full boiler pressure even though they will normally operate at only 15 to 20 pounds per square inch gauge.

i. Each steam jacket should have a small "bleeder valve" for venting noncondensable gases during operation, and for admitting air to the jacket when not in operation.

j. Packing glands for the conveyor shafts should be located externally on the flange plates which close the ends of the tubes.

k. Place a sight glass at the discharge end of the first drier section for observing the movement of the meal and the load of the conveyor at that point.

2. Meal steamer

The construction and operation of the steamer sections is the same as the drier sections except as indicated below:

a. The bulk density of the dry meal entering the steamer will be different from that of the wet material entering the drier. The steamer should be loaded so that the meal occupies about 40 per cent of the free volume of the tube.

b. In order to produce a marketable product the soybean meal should be heated to about 115° C. in the steamer at the same time that it is being stripped of residual solvent by the counter-current steaming. In this way condensation of steam in the meal is prevented, and the moisture content of the meal can be reduced to the required value for storage and shipment. The stripping steam should enter at the meal discharge end of the steamer. The jacket steam pressure can be regulated to give
Never as well as the action of the rotary valve to be checked.

At a point just above the rotary valve permit the operation of the cone.

If a sight glass at the discharge end of the last steam or section

while at the same time providing a tap or seal at the point

decrease and of the last steam or section for removing the finished product

If the rotary valve or similar device should be used at the after

insulated on the steam arrestor.

Temperature of 1150°, some form of compensating control device should be

maximum recommended for use. In order to insure heating the meat to a

oven desired, a pressure of 50 pounds per square inch gauge at the

in the jackets of the steamer tubes in the dryer through this is

flow should be made for the use of higher steam pressure

charge and end of each section.

Pressure readings in the steam should be taken at the meat discharge end of each steamer section, and at the vapor outlet.

d. Temperature readings should be taken at the rotating points.

counter-current movement of solids and vapors is regained.

the path where the meat enters from the dryer section. In this way

the vapor outlet for the steamer should be located adjacent to

temperature of the meat to the desired point

the desired temperature to the finished product. The size of the steam-

79-
F. Condenser Equipment

Two condensers should be provided, one for the vapors from the miscella preheater and still, and one for the vapors from the drier and steamer units. In order to make possible easy cleaning of the condensers, they should be of the vertical tube type with cooling water on the shell side and vapors on the inside of the tubes. Further specifications are given below.

1. Each condenser should be so located that the hot vapors from the solvent recovery units flow down into the condenser.

2. A small cyclone separator should be provided in the vapor line leading from the drier and steamer units to the condenser, to remove entrained solids carried by the vapors.

3. The solvent-water separator should be placed below the condenser so that the condensate can flow into it by gravity.

4. All noncondensable gases should be vented from the condenser through a solvent recovery unit to prevent loss of solvent vapor.

5. A manometer should be installed on the vent line from the condenser for reading the pressure.

6. An adequate vent should be provided on the condenser for purging air from the whole system when starting up the plant. If the air cannot be purged quickly, pressure will build up in the system and the manometers will blow out.

7. Steel construction is satisfactory.
G. Solvent-Water Separator

In an extraction process in which steam is used to recover trichloroethylene from the products, the condensate from the condensers should flow by gravity into a tank where separation of the solvent and water can take place automatically due to the immiscibility and difference in specific gravity of the liquids. It is an advantage that the mutual solubility of the solvent and water is very low, as otherwise provision would have to be made to recover solvent from the water leaving the separator before it could be discharged from the system. In most cases the solubility of water in the solvent is so low that it does not constitute a problem. Some specific points on the construction and operation of this type of equipment are given below.

1. The size of the separator tank should be determined by the input of condensate, and the minimum time required for gravity separation of solvent and water to take place. In other words, the rate of flow of liquid through the unit must be such that ample time is provided for the mixed liquids to separate into two distinct layers. The more dense liquid (trichloroethylene) will form the bottom layer and the lighter liquid (water) the top layer.

2. The condensate should enter the separator at a point corresponding to the level of the solvent-water interface which is set and maintained by the position of the liquid overflow lines from the unit. The heavier liquid flows continuously from the bottom of the unit through a line which forms a "U" with the separator, thereby
position of the solvent-water interface

a gauge glass should be provided on the tank for observing the
temperature

operation

A meter should be made for measuring the width of the interfacial

flow of solvent and water though the separator can be used.

Flow of solvent where vapor can be lost at this point.

Constant pressure is maintained in the separator tank to

in the unit. In general, the separator tank

a vent must be provided in the top of the separator tank so as to

that the required volume hold-up, based on liquid flow, is provided.

the water is kept at enough above the condenser in that point so

maintaining a constant depth of solvent in the tank. The outlet for
VI. SUMMARY

The research investigation discussed in this thesis was carried out during the years 1937 and 1938. The object of the research was to build and operate a pilot plant for extracting oil from soybean flakes using the non-flammable solvent trichloroethylene. After preliminary laboratory experiments, the basic principles underlying the design of a continuous counter-current extractor were worked out. A pilot plant having a capacity of approximately one ton of soybeans per (24 hour) day was installed in the Chemical Engineering Building of Iowa State College. Experiments conducted with this plant over a period of months contributed much in the way of specific information on the design and operation of equipment for an extraction plant using a high specific gravity solvent such as trichloroethylene. Data were obtained on which to base the design of plants for the commercial extraction of oils and fats from animal and vegetable materials using the above mentioned solvents.
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Photograph 1

General View of Meal Drier Assembly during Construction Period (A)

A - No. 1 meal drier.
B - No. 2 meal drier.
C - Meal steamer.
D - Upper end of riser leg of extractor.
E - Meal spout connecting second drier to steamer.
F - Initial vapor outlet from second drier. Note: This line not used in all pilot plant runs.
G - Initial vapor outlet from meal steamer. Note: This line not used in all pilot plant runs.
H - Steam jacketed vapor line running to condenser.
I - Initial vapor line from first drier. Note: When use of outlets "F" and "G" was discontinued, line "I" was increased from 1 1/2 inch to 3 inch and then to 5 inch pipe.
J - Condenser.
K - Water line to condenser.
L - High pressure steam supply line for jackets.
Photograph 2

General View of Meal Drier Assembly during Construction Period (B)

A = No. 1 meal drier.

B = No. 2 meal drier.

C = Meal steamer.

D = Spout for meal leaving extractor. Note: Connection not yet made with first drier.

E = Sight glass on riser leg of extractor.

F = Connection for solvent inlet line.

G = Condenser.

H = Motor

I = Speed reducer.
Photograph 3

General View Showing Power Supply, Drives and Arrangement of Equipment

A - Motor (5 H.P., 1700 R.P.M.).
B - Speed reducer (457 to 1 reduction).
C - Drive for conveyor in riser leg of extractor.
D - Drive for conveyors in meal driers and steamer.
E - Drive for conveyor in inclined leg of extractor.
F - Riser leg of extractor.
G - No. 1 meal drier.
H - No. 2 meal drier.
I - Feed hopper for soybean flakes.
J - Condenser.
K - Tri-water separator.
L - Solvent inlet to extractor.
M - Sight glass.
Photograph 4

Power Supply, Drives and Meal Driers

A - Motor.
B - Speed reducer.
C - Drive for conveyor in riser leg of extractor.
D - Drive for conveyors in meal driers and steamer.
E - Drive for conveyor in inclined leg of extractor
F - No. 1 meal drier.
G - No. 2 meal drier.
H - Sight glasses.
I - Drive for barrel valve.
J - Jack shaft from speed reducer.
Photograph 5

Drive End of Meal Drier and Steamer Sections

A - Main drive for conveyors.

B - No. 1 meal drier.

C - No. 2 meal drier.

D - Meal steamer.

E - Packing gland.

F - Tinned roller bearing.

G - Inclined leg of extractor.

H - Riser leg of extractor.

I - Spout connecting extractor to first meal drier.

J - Vapor outlet at meal exit end of second drier. Note: This was only used during the preliminary test runs and was then capped off.

K - Vapor outlet at meal entrance end of steamer. Note: This was capped off at the same time as "J" above.

L - High pressure steam supply line.
Photograph 6

End of Meal Steamer Showing Barrel Valve and Drive

A - Meal steamer.
B - Barrel valve.
C - Drive for barrel valve.
D - Sampling outlet on inclined leg of extractor.
E - No. 2 meal drier.
F - Cap sealing end bearing of conveyor shaft. Note: As the conveyor shafts did not extend through the bearings located in the end flanges no packing glands were needed at this end of the driers or steamer.
G - Support for condenser.
H - Condensate line from steam jackets.
I - Pet cock for bleeding N₂C₆ gases from the steam jacket.
Photograph 7
Upper End of Riser Leg of Extractor Showing Drive and Meal Outlet Spout

A - Spout for gravity flow of meal from the extractor to the first drier.
B - Top end of riser leg of the extractor.
C - Packing gland.
D - Timken roller bearing.
E - Roller chain-bevel gear drive.
Photograph 3

Miscella Outlet and Housing

A - Miscella outlet consisting of tapered slots cut on the under side of the extractor tube.

B - Vapor-tight housing surrounding miscella outlet.

C - Frame for supporting filter screen if desired.

D - Original flake inlet to extractor.

E - Final location of flake inlet to extractor.
Photograph 9
Upper End of Inclined Leg of Extractor

A - Drive for conveyor.
B - Timken roller bearing.
C - Packing gland.
D - Miscella outlet slots.
E - Housing for miscella outlet.
F - Support for metal screen.
A - Feed hopper for soybean flakes.
B - Housing for miscella outlet.
C - Inspection door.
D - Miscella line to filters.
E - Miscella line to top of preheater.
F - Original location of flake inlet.
G - End of No. 1 meal drier.
H - End of No. 2 meal drier.
I - Vapor line from end of No. 1 drier.
J - Thermometer well.
Photograph 11

Conveyor for Inclined Leg of Extractor

A - Solid flight running full length of conveyor (6 inch pitch, tack welded to shaft).

B - Auxiliary flighting originally installed at point of flake inlet.
   Note: It was found that the double flight was not necessary as the auxiliary flight "B" was removed.

C - Joint in conveyor shaft for use in dismantling conveyor.

D - Upper end of inclined leg of extractor.
Photograph 12

Conveyors used in Meal Drier Sections

A - No. 1 meal drier.
B - No. 2 meal drier.
C - Conveyor shaft made of 1/2 inch pipe.
D - Solid shaft turned to fit into capped bearing located in end
    flange of drier tube.
E - Ribbon flighting made of 3/8 inch rod (6 inch pitch).
F - Auxiliary flighting made of 3/8 inch rod (18 inch pitch).
G - Solid flighting having reverse thread.
H - Short lengths of 3/8 inch rod joining consecutive flights of the
    conveyor.
I - Original 1 1/2 inch vapor outlet from No. 1 meal drier.
Photograph 13

General View of Meal Drier Arrangement During Operating Period (A)

A = No. 1 meal drier.
B = No. 2 meal drier.
C = Meal spout connecting extractor to first drier.
D = Vapor line from second drier and steamer.
E = High pressure steam supply line.
F = Condenser.
G = Metering pump for adding make-up solvent. Note: This method used in only a few runs.
H = Roller chain drive for barrel valve.
Photograph 14

General View of Meal Drier Arrangement During Operating Period (B)

A - Flake hopper at upper end of inclined extractor leg.
B - Inclined leg of extractor.
C - Riser leg of extractor.
D - Sight glass for observing solvent level in riser leg.
E - Solvent inlet to extractor.
F - Spout for gravity flow of meal from extractor to first drier.
G - No. 1 meal drier.
H - No. 2 meal drier.
I - Meal steamer.
J - Vapor line from No. 1 drier.
K - Vapor line from No. 2 drier and steam.  
   Note: This line only used in some of the first test runs.
L - Vapor line from preheater and still condenser.
M - Condenser.
N - Absorption tower for vent gases.
O - Oil reservoir for absorption tower.
P - Water manometer for measuring pressure in condenser.
Q - Water manometer for measuring pressure at meal entrance end of No. 1 drier.
R - High pressure steam supply line.
S - Steam pressure gauge.
T - 5 H.P. motor.
U - Speed reducer.
Photograph 15

General View of Meal Drier Arrangement During Operating Period (C)

A = Inclined leg of extractor.
B = Riser leg of extractor.
C = Solvent inlet to extractor.
D = No. 1 meal drier.
E = No. 2 meal drier.
F = Meal steamer.
G = Barrel valve at end of steamer.
H = Spout for meal leaving barrel valve.
I = Miscella preheater.
J = Miscella still.
K = Manometer connection for reading pressure in meal steamer.
L = Sampling outlets on bottom of extractor tube.
M = Trap and drain at bottom of riser leg.
Photograph 16
Meal Steamer and Barrel Valve

A = Meal steamer.
B = Sight glasses.
C = Connection for water manometer.
D = Vent on steam jacket.
E = Line for introducing sparging steam to steamer.
   Note: This method used only in preliminary test runs.
F = Final location of inlet for superheated sparging steam to steamer.
G = Heater for superheating low pressure steam used as sparging vapor in
   meal steamer and miscella still.
H = Barrel valve.
I = Meal spout.
J = Miscella preheater.
K = Steam jacketed vapor line from preheater and still.
L = Steam pressure gauge for preheater and still.
M = Trap on vent line from condenser to absorption tower.
Photograph 17

Meal Steamer, Battered Wafer and Absorption Tower
Photograph 18

Miscella Filters, Preheater and Still

A - Line for gravity flow of miscella from extractor to filters
B, C - Bag filters.
D - Point of entrance of filtered miscella to preheater.
E - Miscella preheater.
F - Flanged section connecting bottom of preheater and top of still.
   Note: Vapor outlet for both units located in this connecting piece.
G - Miscella still.
H - Barrel valve at end of meal steamer.
I - Spout for meal leaving steamer.
Photograph 19

Miscella Filters

A - Miscella line from extractor.
B, C - Bag filters.
D - Storage tank for filtered miscella.
E - Pump for feeding filtered miscella into preheater.
F - Motor for pump.
G - Gauge glass on storage tank.
Photograph 20

Miscella Still

A - Miscella still.

B - Still bottom.

C - Open vessel for collecting oil and solids from still.
   Note: During operation vessel "C" is nearly filled with oil, and is elevated so that section "B" is submerged to a depth of about 6 inches. The oil level in "C" is maintained constant by an overflow line.

D - Spout for meal leaving the steamer.
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