

Distillation and Solvent Recovery for Soybean and Other Oilseeds Plants¹

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ABSTRACT

Features which a modern, well designed distillation and solvent recovery system should contain in order to permit safe and profitable operation will be discussed. The technology has advanced rapidly during the past 10 years in the development of more efficient and profitable distillation and solvent recovery systems. These systems operate with less down-time and far more efficiency than they did 20 years ago. The application of good chemical engineering practice, together with a long background of plant operations, will permit continued improvements of these systems in the future. To permit continuous development in this area it is essential that design and operating companies have a two-way interchange of information.

Distillation is one of the oldest arts practiced by mankind. It started as an art long before scientific principles were applied. But only in the past half century has mankind really applied scientific principles to under-

¹Presented at the AOCS Meeting in Minneapolis, October 1969.

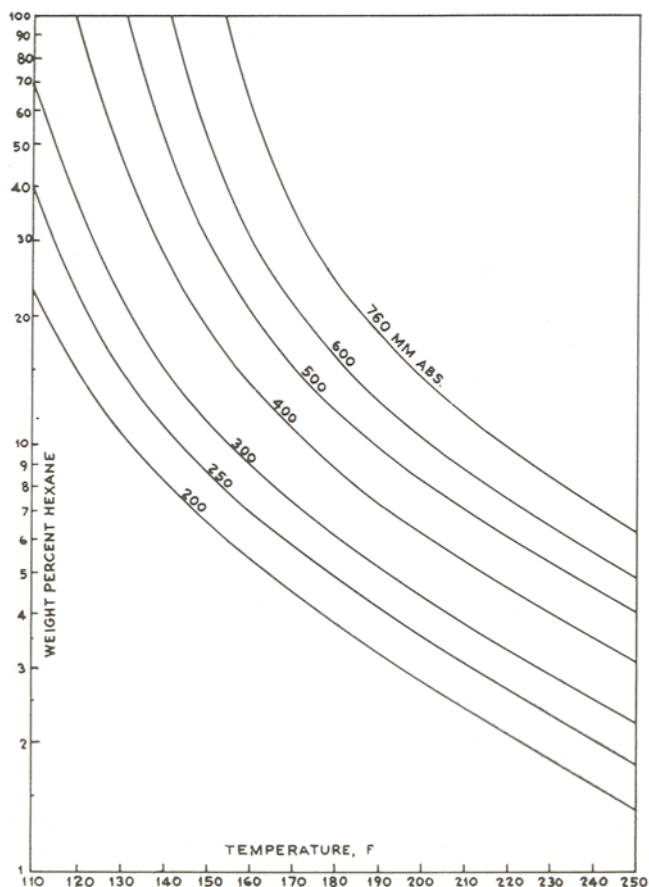


FIG. 1. Boiling point curves for soybean oil-hexane (Skellysolve B) solutions.

stand and solve the relatively complex design problems of distillation. During the past decade, there has been significant acceleration of design know-how in the field of distillation leading to the solution of complex technical and economic problems. The demand for solutions to the problems and the availability of computers have facilitated this rapid progress. Recently, computers have been employed to design considerably improved distillation facilities for the petroleum, chemical and fatty acid industries.

In chemical engineering practice, the term distillation is normally applied only to those operations in which vaporization of a liquid mixture yields a vapor phase which contains more than one component and from which one or more components can be recovered in nearly pure state or in controllable composition. For the oilseeds industry, the term distillation is used rather loosely to describe operations which are primarily evaporation, steam stripping, vapor condensing and decanting water from solvent.

The loose usage of the terms distillation and solvent recovery can be a source of poor communication between engineering contractors and oilseeds processors unless the scope of work and particular processes are precisely defined. In general, oilseeds processors use the terms distillation and solvent recovery together, broadly to describe all processes in the solvent handling area other

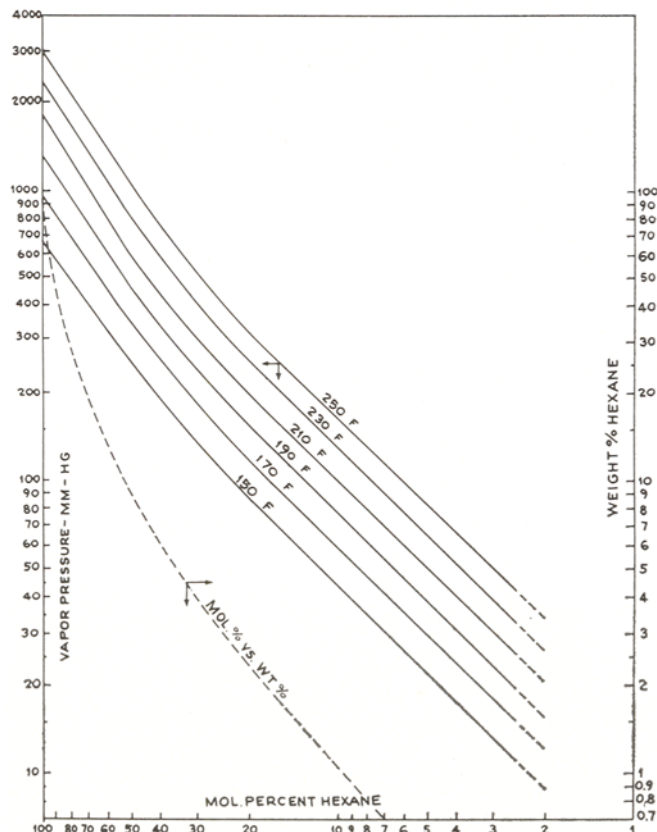


FIG. 2. Vapor pressure isotherms, soybean oil-hexane (Skellysolve B) solutions.

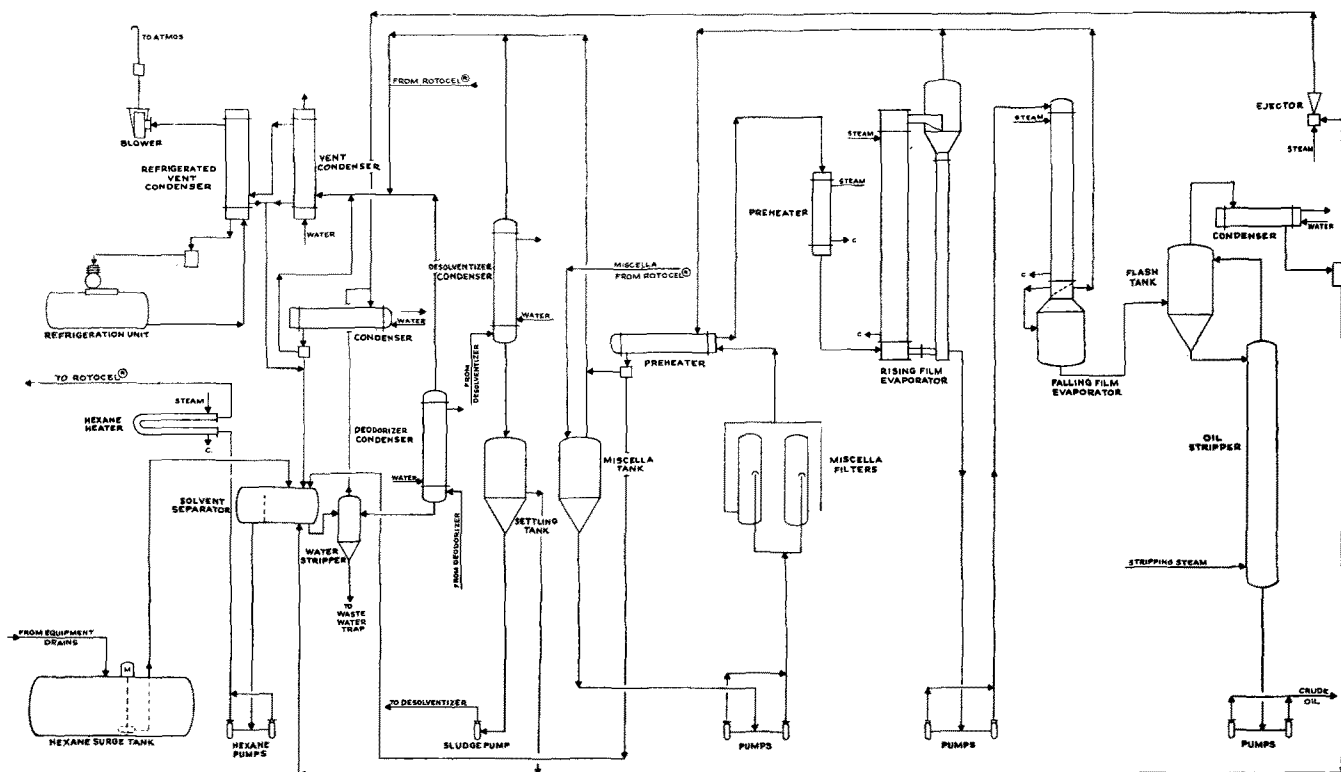


FIG. 3. Distillation and solvent recovery system, late 1940's.

than solvent extraction and meal desolventizing. More precisely, distillation and solvent recovery for this industry describes the following processes:

1. Evaporation of hexane from vegetable oil—single or dual effect—from the miscella to about 90 wt % oil concentration. (A mixture of vegetable oil and solvent is known as miscella.) Generally, the miscella supply tank, which receives miscella from the extractor, is considered as part of the evaporation system.

2. Flashing and low pressure steam stripping to remove hexane from the oil down to commercially acceptable levels (less than 0.15 wt % moisture and volatile content).

3. Heat exchange systems which are often employed to utilize evaporator surface area more efficiently.

4. Steam economizer systems which serve the dual purposes of conserving steam and performing an essential

processing function.

5. Condensers, a solvent separator and a water stripper which are used to recover the hexane and separate it from waste process water in a safe manner. The waste water sump is also normally included as a component part of the process water system.

6. Vent condensers, blowers and ejectors, which are used to recover hexane from the exit gases.

7. A mineral oil absorption and recovery system for final removal of solvent from exit gases. (This topic has been described thoroughly in a previous paper (1).

8. Pumps, piping, flame arrestors, vent seals, instruments and controls, and other accessory facilities which are necessary to make the previously described facilities

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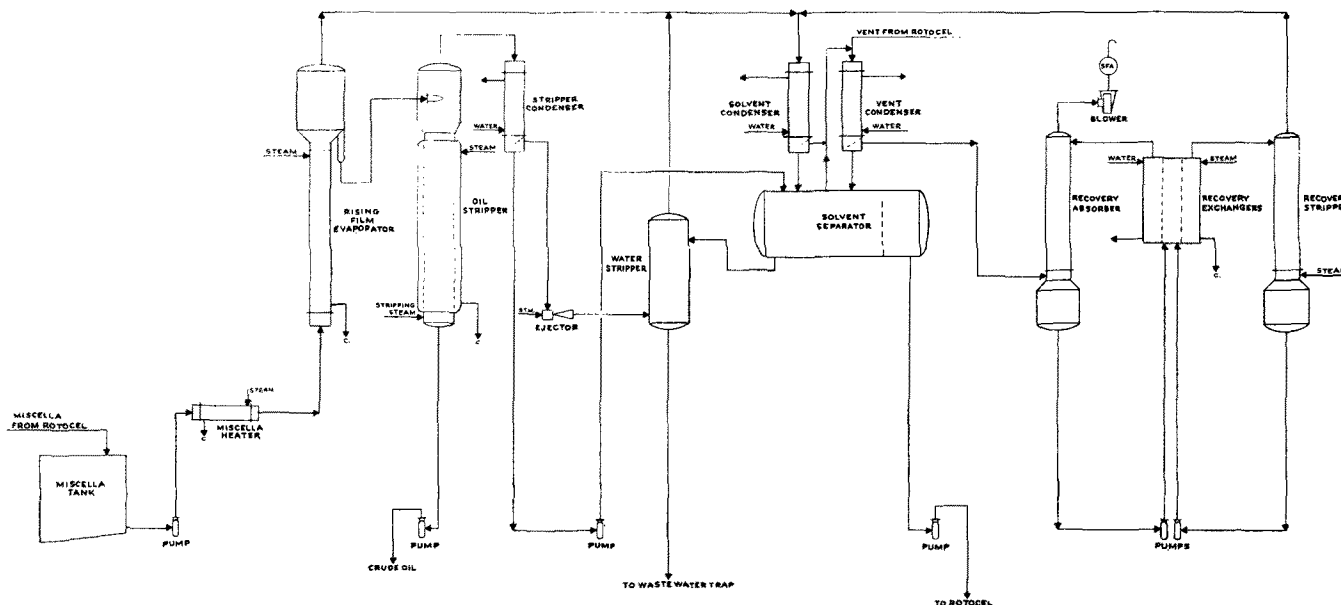


FIG. 4. Single effect distillation and solvent recovery system, mid 1960's.

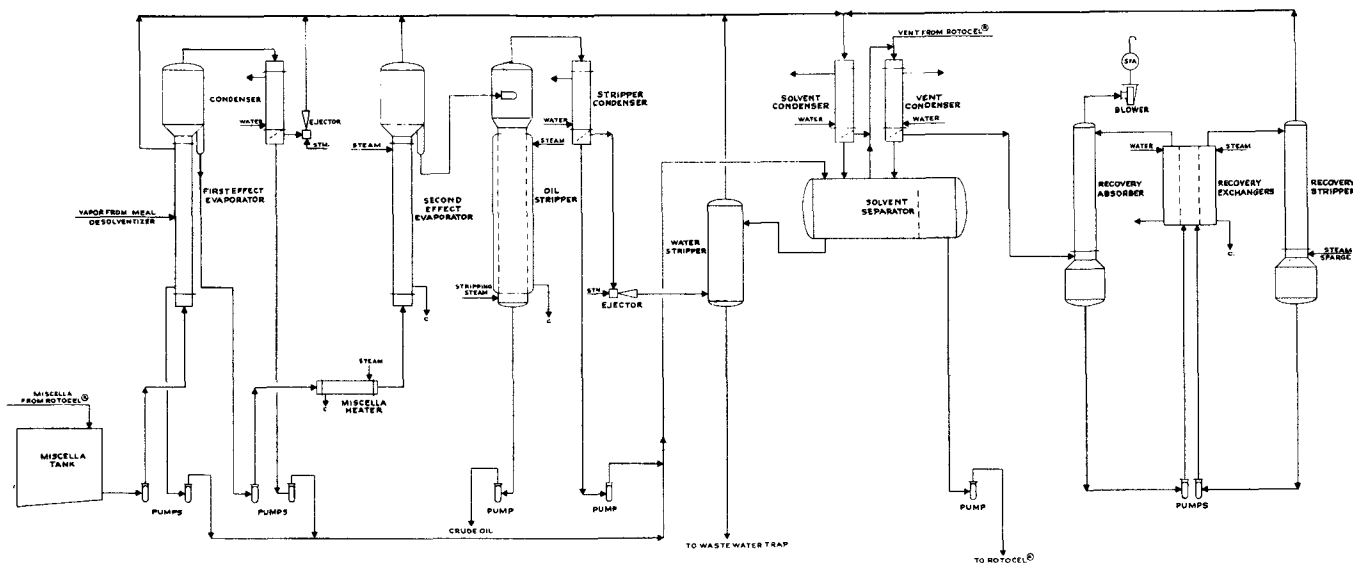


FIG. 5. Dual effect distillation and solvent recovery system, mid 1960's.

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functional from both operational and safety standpoints. Vent seals provide protection by relieving when abnormally high or low pressures occur in a solvent extraction plant.

It is well known that adverse operational conditions such as pressure fluctuations or improper decantation of solvent will have an adverse effect on the solvent extraction and meal desolventizing systems. For a profitable and well designed plant, it is essential to coordinate design considerations of the extraction and meal desolventizing systems with those of the distillation and solvent recovery systems. Unfortunately, this has not been done in numerous cases. Typically, it is often not done when plant modifications and expansions have been carried out on a piece-meal basis without proper consideration of overall plant design. Such practice, if continued for several sequential plant modifications, will usually lead to many operational and safety problems that are very expensive to correct.

The typical design criteria for distillation and solvent recovery systems for soybean oil and most other oilseeds plants are:

1. Oil products specifications: maximum temperature, moisture and volatile content, and fines content.
2. Miscella fed to evaporator: throughput rate and concentration.
3. Quantity of air flowing through system: during normal operation and during start-up operation.
4. Steam at use point: pressure and quality and cost.
5. Cooling water at use point: temperature, quantity available, pressure and tendency to foul heat exchangers.
6. Special: unusual corrosive conditions, installation for existing plant, very frequent start-ups and shut-downs, unusual plant layout, requirements for instruments and controls and anticipated future requirements.

For example, the answers to questions raised by these design criteria may justify entirely different designs for distillation and solvent recovery systems even when handling the same throughput rate of soybeans because:

1. Some plants will produce lecithin so filtration of oil or miscella may be required.
2. Tradition and governmental regulations in some European countries and in Japan may require the miscella to be evaporated under sub-atmospheric pressure. Most of the earlier European and Japanese evaporation systems, because of the nature of their design, permitted considerably more contact time with heated surface than did the American rising film evaporators. Consequently, a

maximum temperature limit was placed on the oil product so that deterioration would not occur. This limitation is sometimes required by some processors on a traditional basis rather than on a rational basis.

3. Miscella concentration can be much higher in a Rotocel extractor than for other extractors such as the Bollman type.

4. Air volumes to be handled during normal and start-up conditions will vary widely if the volumes of major vessels vary.

5. Steam cost of plants in the lower to intermediate range (500 to 750 tons/day) will determine whether or not dual evaporators are justified.

6. The nature of the cooling water, clean or fouling type, will determine the type of heat exchangers and condensers to be used.

Figures 1 and 2 respectively show the boiling point curves and vapor pressure isotherms for soybean oil-hexane solutions. These curves illustrate why most large soybean plants use a dual stage evaporator followed by a low pressure steam stripping system for their distillation facilities. Typically, the following conditions dictate design:

1. Miscella from the extractor varies in concentration from 20 to 30 wt % oil in hexane.
2. Desolventizer-toaster vapor at 170 F to 200 F is utilized to heat the first effect evaporator, concentrating the miscella to a range of 55-70 %. Obviously, sub-atmospheric pressure must be used in this evaporator to carry out the operation.
3. Steam heat at 10-20 psig is used to concentrate the miscella further under atmospheric pressure to about 90-94%.
4. The miscella enters the oil stripper under vacuum and is flashed to about 98% concentration.
5. Low pressure steam stripping is used to remove the final hexane from the oil so that the temperature of the oil product is minimized.

A modern well designed distillation and solvent recovery system at least should contain the following essential features:

1. Design conditions: capacity and functions suitable for extractor and desolventizer, and prudent investment consistent with maximum solvent recovery and minimum steam usage.
2. Operating features: produce specification crude oil, handles necessary excess miscella and solvent, easy to start-up and shut-down, allows uniform control of con-

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ditions and suitable access to valves, controls, etc.

3. Safety provisions: can shut down safety during emergency, automatic venting during abnormal pressures and will discard waste water and exit gases safely

4. Maintenance considerations: easy to inspect and clean out, easy to replace parts and standard equipment for replacement.

The technology—design conditions, operating features, safety provisions, maintenance considerations—for distillation and solvent recovery systems has advanced considerably during the past two decades.

Figure 3 is a flow diagram of a distillation and a solvent recovery system that was designed in the late 1940's. As illustrated comparatively with Figure 4, which is a flow diagram of similar facilities designed several years ago, the following changes have occurred in 20 years: Miscella filters, deleted; falling film evaporators, deleted; rising film evaporator, incorporates integral liquid-vapor separator; oil stripper, combined with flash tank and internals now are disc and donut type; refrigerated condenser systems, deleted and replaced by mineral oil absorption systems; solvent separator, modified to include both surge tank and separator; vent seals, improved; pumps, piping, instruments and controls, improved.

As a result of continuing improvements, today's solvent extraction plants operate with less down time and far more efficiently than they did even a few years ago. Figures 4 and 5 comparatively illustrate single and dual effect evaporator systems that were used a few years ago. For a typical 1500 ton/day soybean plant, a dual evaporator system will only use 80% of the steam used by a single effect evaporator system (extraction, desolventizing, distillation and solvent recovery systems steam usage only).

It is suggested that the potential buyer of new distillation and solvent recovery facilities can avoid many pitfalls by observing these ground rules:

(a) Obtain complete and accurate information relating to design criteria; (b) List the essential features for the plant; (c) If possible, avoid piecemeal expansions. In any event, be sure that the new facilities are suitable from capacity and functional standpoints to the extraction and desolventizing systems; (d) Define the scope of work precisely. Then discuss design criteria, essential features and scope of work thoroughly with the design-supplier; (e) Be sure that the new facilities you procure include the latest technology and that future overall plant needs are anticipated.

REFERENCES

1. Good, R.D., *Oil Mill Gazetteer* 73:28-32 (1969).

[Received January 2, 1970]



New Phosphate Free Detergents

Dynachim S.A.R.L. of Paris has developed completely workable formulations for detergents which contain little or no phosphates. Other types of chemical builders of the well known silicate, perborate and carbonate salts are utilized, as well as some novel derivatives of citric acid and polymers of maleic acid—a large scale nontoxic petrochemical with many uses, including plastics containers for food.

The chemical development is credited to a team led by M. Viguier, Chemist and formerly laboratory director of the French Institute of Laundering.

The new detergents called "Nophos" and "Sanfos" (the latter formulated for automatic cloths and dishwashing machines) respond to the requirements of some areas concerned with water pollution control.

In some countries there is a real phosphate hunt, which has even brought hopes for a renaissance of soap. (Detergents now control 68% of the U.S. market and 60% of the European demand.) It is known that soap does not perform as well as syndets under all conditions, nor is there a sufficient amount of fats and oils in the world to supply the raw materials needed for the switch. At least ten organizations (including Dynachim) are working on the synthesis of fatty acids in the lauryl and tallow ranges. The USSR has also developed a process; however it is nonselective and gives rise simultaneously to a wide range of products and by-products.

About a billion pounds of sodium tripolyphosphate are used in the U.S. with heavy duty powders. It performs by sequestering calcium and magnesium found in water. The phosphate concern is for sewage installations, rivers and lakes. The oxygen balance can be destroyed, encouraging growth of algae. New sequestering agents used in the Nophos and Sanfos formulations have been partially responsible for solving this problem.

Ampholytics insensible to water hardness in the absence of phosphates are probably the longer term answer. Dynachim is actively field testing new polyelectrolytes as syndet builders.

The new compositions contain a basic detergent salt (other than a basic phosphate), a derivative of a sulfopoly-carboxylic acid and a polymer of a substituted maleic anhydride. The formulations contain a specially compounded mixture of silicates, carbonates and perborates as well as tetra acetyl ethylene diamines, and nonionics. The sulfopoly-carboxylic derivatives can be water soluble esters of an alpha-beta unsaturated polycarboxylic acid whose double bond has been sulfonated. While fermentation citric acid can be used as a base, other acids which can eventually be made by large scale petrochemical routes are preferred.

Some nonionic surfactants derived from starch can also be used as alternates in the formulations but are not included, as they are not yet available commercially. For more details contact Mr. D. Passini, Dynachim s.a.r.l., 5 Avenue de l'Opéra, Paris (1), France.

Detergent Manufacturers to Label Phosphate Products

Household laundry and dishwasher detergents, including all of the leading brand-name products, will soon be labeled nationally as to their phosphate content, E. Scott Pattison, Soap and Detergent Association President, has announced.

Member companies who market the bulk of these products have advised the Association that they will label detergent packages according to the per cent phosphorus in the formula and also its equivalent in grams per recommended use level.

This action has been taken to correct the largely erroneous information about phosphate content of products which appears in the many lists distributed by various organizations and publications to consumers.