# **Distillation and Solvent Recovery**

EDWARD D. MILLIGAN and DAVID C. TANDY, EMI Corporation, Des Plaines, Illinois, 60018

# ABSTRACT

The historical development of various miscella distillation systems used in solvent extraction of oilseeds is reviewed, and their economics are compared. In addition, the design of solvent recovery systems and their economic justification are reviewed.

# INTRODUCTION

An oilseed solvent extraction plant consists of several distinct, different processing systems, each of which must be designed and operated for maximum efficiency for the complete plant to operate at minimum processing cost. Two of the significant systems are the miscella distillation system and the solvent recovery system. They are treated separately because they involve different considerations of economic evaluation, design, and operation. In the first case, minimizing steam consumption required for solvent evaporation, and in the second, maximizing recovery of valuable solvent, are the primary objectives.

# **HISTORY OF DISTILLATION**

Miscella distillation systems have developed over the years into three basic designs. The first is simple distillation without steam savings, generally utilized in the small capacity plant of ca. 100-200 tons per day (TPD), where investment economy is the paramount consideration and the steam savings is not sufficient to result in acceptable payout time. The second is steam saving evaporation, using desolventizer-toaster (D-T) vapors to vaporize part of the solvent in the miscella and using single stage evaporation for obtaining fully concentrated miscella (90% or more by wt) in those cases where the cooling water temperature available is sufficiently low to allow operation at high vacuum. This is followed by single stage oil stripping for final removal of solvent. The third also utilizes desolventizer-toaster vapors but two-stage using evaporation, followed by two stage oil stripping and oil drying in those cases where the cooling water temperature is high or air-cooled condensers are used. This latter design case is becoming more prevalent world wide because of increasing practice of water conservation and also because of the simple unavailability of cold cooling water in many parts of the world, both of which result in increasing reliance on cooling towers or air-cooled condensers.

Other systems can be designed but have not been used to any great extent. For example, mechanical or steam jet chilling of water can be used in the absence of cold water to obtain higher vacuum in the evaporation system, but these systems require sufficient energy in themselves to reduce the energy saving in the overall system dramatically. The required investment also is increased with the result of greatly lengthening the payout period.

Another such system which has been used in a few special cases is multiple-effect evaporation, which utilizes the successive stages of solvent vapor evaporated for the heat source for the earlier stages. This system has been used with good economy in alcohol extraction systems, where there is no high temperature D-T vapor available as a heat source. However, the same limitations on vacuum are imposed, as in the previous system, by available cooling water temperature.

# DESCRIPTION OF DISTILLATION

The three types of miscella distillation systems are

described below. Each process begins with the full miscella from the extractor and ends with a finished oil meeting the trade's flash point specifications of 0.1% moisture and volatile +250 F flash point. The evaporated solvent is condensed and recovered from the solvent water separator for reuse. For simplifying comparisons, the solvent-water separator and accessories are omitted in the following discussions.

Figure 1 illustrates the simple miscella distillation system which utilizes only indirect steam for evaporation. The miscella is pumped from the miscella tank into the evaporator; most of the solvent is evaporated; and the concentrated miscella flows by pressure difference into the vacuum oil stripper. The concentrated miscella, at 90% or more of oil, enters the head of the stripper and flashes to ca. 1% solvent by wt under high vacuum. This miscella then is stripped of the 1% solvent in the lower portion of the oil stripper by descending over a series of disc and donut contact trays countercurrent to a rising stream of live steam. The stripping steam and solvent are condensed in the oil stripper condenser and pumped to the decanter.

The hot solvent vapor from the evaporator is condensed but in some plants is piped through the extractor first before going to the evaporator condenser. This vapor supplies the heat necessary to maintain the extractor temperature high, resulting in improved extraction and minimizing corrosion in the extractor.

The system described above can be used efficiently with cooling water temperatures up to ca. 80 F. Since the cooling water temperature determines the maximum vacuum attainable in the oil stripper, higher water temperatures may require addition of an oil dryer, as described below, to achieve specification flash point and moisture and volatile in the final crude oil. The evaporator and condenser system is basically the same, however, except a larger condenser is used with higher water temperature.

In some installations, with particularly high water temperatures, a preheater is included between the evaporator and the oil stripper to ensure that enough solvent will flash in the head of the oil stripper to ensure an acceptable oil product.

The second system for miscella distillation involves the use of some of the D-T vapors for evaporation of the solvent. Figure 2 illustrates the system which is used for a cooling water temperature from very low up to ca. 80 F. With this water temperature, it is possible to obtain a sufficiently high vacuum in the miscella evaporator to enable concentration of the full miscella to 90% or more by



FIG. 1. Simple distillation system. LC = level control, FI = flow indicator.



FIG. 2. One stage distillation with desolventizer-toaster vapors for low cooling water temperatures. FI = flow indicator, LC = level control.

wt in a single vacuum unit. This is because the miscella boiling point is lower under high vacuum than the dew point or condensing temperature of the D-T vapors.

For example, one commercial unit supplied with 70 F water, operated under an absolute pressure of 200 mm mercury equal to ca. 22 in. of vacuum. The concentrated miscella left this evaporator at 91.8% oil, and its boiling point under vacuum was 141 F. The D-T vapor temperature at its inlet was 165 F. This resulted in sufficient temperature difference to transfer the required heat quantity. The miscella is pumped from the miscella tank into the miscella evaporator tubes under vacuuum. D-T vapors are piped to the shell, where a portion is condensed in evaporating the same quantity of solvent as in the simple atmospheric steam evaporation system. The uncondensed or excess D-T vapors return to a D-T condenser for recovery while the condensate returns to the decanter. The concentrated miscella then is pumped to a vacuum oil stripper which performs the same function as before.

In this case, a pump is necessary because of the low pressure differential between the two vessels, and a preheater must be used to raise the miscella temperature sufficiently to realize the proper amount of flashing in the head of the oil stripper. The steam and solvent vapors from the oil stripper are condensed and pumped to the decanter as before.

Condensing of the vacuum evaporator vapors has been found to be most economically accomplished in two stages. The vapors and cooling water pass through the two condensers in series and countercurrently, with the final vapors exiting vacuum condenser no. 2 in contact with the coldest cooling water. Most of the condensing load is carried by vacuum condenser no. 1 in which no attempt is made to obtain a close approach to the water temperature, and the presence of air has little effect upon the overall heat transfer rate. These two factors are important in vacuum condenser no. 2, but its load is a small part of the total and the overall effect is a saving in total area for the two exchanger systems and improved vacuum performance over a single condenser system. This system requires a solvent preheater to be used ahead of the extractor to compensate for the lack of hot solvent vapor heating from a simple distillation evaporator, as in the first system.

The low vapor temperature obtained in the vacuum condenser no. 2 results in a small enough load so that only a small single stage ejector is needed for the vacuum evaporator.

Figure 3 illustrates the third system for miscella distillation which also uses D-T vapors in the first stage of evaporation but is used with cooling water temperature of 85 F and higher. This temperature limits the vacuum attainable in the evaporator and thus the concentration of



FIG. 3. Two stage distillation with desolventizer-toaster vapors for high cooling water temperature. LC = level control.

#### TABLE I

1000 TPD Soybear	Plant Steam Consumption and Savings
	Distillation Systems

	Simple distillation	One stage steam saving	Two stage steam saving
Concentrated miscella (%)	90%	90+	70
Steam consumption (lb/hr)			
Evaporator	8.750	0	1,130
Solvent heater	1,450	2,670	2,530
Miscella heater	1,450	1,070	770
Evaporator ejector	0	250	250
Total	11,650	3,990	4,680
Savings (lb/hr)	0	7.660	6.970
(lb/ton beans)	ō	184	167

miscella which can be obtained. Full miscella is pumped as before to the tubes of the first stage evaporator operating under a lesser vacuum than in the second system. D-T vapors again are used in the shell for heating; but, because the vacuum is limited, the miscella boiling point is higher, and, thus, the temperature difference to the condensing D-T vapors is lower. This results in a lower concentration of miscella produced, typically 70% oil, instead of 90% oil.

This system requires a second stage of evaporation to concentrate fully the miscella, this one heated by indirect steam as in the first system. Concentrated miscella from the second stage evaporator flows by pressure difference to the oil stripper. Second stage evaporator vapor flows to the extractor for heating it prior to being condensed, eliminating the need for a solvent preheater.

Alternatively, the second stage evaporator can be operated with indirect steam under the same vacuum as the first stage evaporator and the oil stripper, where it is desired to minimize the exposure of the oil to high temperature. However, in this case a preheater is required ahead of the oil stripper, which minimizes the effect of operating the second stage under vacuum.

The high cooling water temperature also limits the vacuum obtainable in the oil stripper, and it is frequently necessary to install an oil dryer in the system to achieve specification flash point and moisture and volatile in the oil. This system is considered more economical of steam than a high capacity vacuum system on the oil stripper. This is because the small quantity of solvent and water requiring removal in the oil dryer is handled easily with low steam consumption required for the vacuum system, even at the high vacuum of 27 in. normally used for the oil dryer design.

## **RESULTS, SAVINGS, AND ECONOMICS**

A comparison of the three systems is shown in Table I, which is a tabulation of steam consumption and savings for the one stage and two stage steam saving system compared with the simple distillation system. For a 1000 TPD soybean plant, the single stage system saves the most steam, a total of 7660 lb/hr compared to the two stage savings of 6970 lb/hr, or 184 lb steam/ton of beans compared to 167 lb/ton.

The comparative investment and dollar savings estimates are shown in Table II. The installed cost for a typical one stage steam saving system is ca. \$60,000 higher than a simple distillation system, and a two stage system is ca. \$90,000 higher. The steam savings translated into dollars/day, of 275 and 250 respectively, result in a simple payout period of 31-51 weeks for the two systems.

### SOLVENT RECOVERY SYSTEM

In every solvent extraction plant, it is necessary to minimize the loss of solvent to achieve economic operation. To illustrate, the well operated plant can, with present technology, operate with an average solvent loss of  $\frac{1}{2}$ gal/ton feed material processed and at times can operate with only  $\frac{1}{4}$  gal/ton loss. For a 1500 ton/day plant, if the actual loss is 1 gal/ton, the avoidable loss is an average of 750 gal/day which costs \$150/day at a solvent cost of \$.20/gal, or \$45,000/year with a 300 day operating year. The expenditure in solvent recovery equipment needed to eliminate this avoidable loss usually is small and often results in a payout of 6 months or less based upon solvent saving.

Most of the solvent is recovered for reuse by condensing the vapor from the equipment which has evaporated it, such as the desolventizer-toaster which removes solvent from the extracted flakes or cake, and the miscella distillation system already described, which evaporates solvent from the oil. This is accomplished in either water-cooled or air cooled condensers of both standard and special design. If there were only pure solvent and steam vapor to be condensed and if the extraction equipment were completely pressure tight, the reovery of solvent would be simple. As it is, the plants are processing solids under severe conditions and much of the equipment

	Simple distillation	One stage steam saving	Two stage steam saving
Estimated installed cost of evaporators, condensers, vacuum systems, pumps, and			
instruments	\$40,000	\$100,000	\$130,000
Incremental cost	0	\$+60,000	\$+90,000
Steam savings (\$/Day)	0	275	250
Simple payout (Days)		218	360
(Weeks)		31	51



FIG. 4. Flow diagram of vent solvent recovery system. PI = pressure indicator, FI = flow indicator, LI = level indicator, FA = flame arrestor and STM = steam.

contains motor driven shafts with packing glands and stuffing boxes. In addition, there are sight glasses, cleanouts, and access doors; all these leak vapor or liquid, if not maintained well or if subjected to pressure. This leakage out can result in excessive solvent loss. Also, the vapor to condensed is not pure vapor but contains a be noncondensible gas-air, which is introduced with the feed material and also enters any leak which is under negative pressure. This air eventually must be discharged to atmosphere, with the equilibrium quantity of solvent vapor which accompanies it. This air may be cooled in an indirect water-cooled or refrigerated condenser, or it may be passed through an oil absorption system to recover the maximum possible quantity of solvent from it before it is discharged to atmosphere. Modern extraction plant practice calls for operating the equipment under a definite negative pressure of 2-3 in. water column to minimize loss of solvent out through any leaks, but instead to pull small quantities of air into the system. Adequate vent air condensing surface and oil absorption capacity then is provided to remove efficiently the solvent from the air so pulled in.

A typical vent solvent recovery system as might be installed in a modern solvent extraction plant, is illustrated in Figure 4, which includes only the major process items, for sake of clarity. This sytem consists basically of: (A) water-cooled vent condenser, (B) mineral oil absorption column, (C) mineral oil stripping column, (D) oil heater and cooler, (E) circulating pumps, and (F) vent blower.

The vent blower maintains a slight negative pressure on the extractor and other process tanks and equipment, the level of which is adjusted by the control valve. Any air which enters the extractor with the feed and which enters any of the process equipment through small leaks is exhausted by the vent blower after first passing through the vent condenser. Here as much as possible of the accompanying solvent is removed by cooling in an indirect cooled tubular condenser. The coldest water entering the plant is used first in this condenser to provide the greatest amount of recovery and decrease the load on the mineral oil absorption system.

The cooled vapor is blown into the mineral oil absorption column filled with inert ceramic packing to facilitate intimate contact between the vapor and a



FIG. 5. Solvent savings with mineral oil absorption system vs vent condenser outlet temperature.

descending stream of cooled, lean mineral oil. The vapor flows upward countercurrent to the oil and becomes leaner with solvent as absorption proceeds, finally being scrubbed with cool, fresh, clean oil, and leaving the top of the absorption column with 1% by volume or less of solvent vapor, representing a recovery of 95-98% of the solvent in the inlet vapor, depending upon the equilibrium temperature of the vapor from the vent condenser. The lean vapor is discharged through a flame arrestor to atmosphere. The oil, now rich in solvent, is pumped out of the absorption column, through a steam heated exchanger, to the top of the stripping column. Here, the solvent in the oil is removed by an ascending stream of dry stripping steam added at the column bottom, and rising through ceramic packing. The steam, rich with stripped solvent vapor, leaves the top of the stripper and is condensed in the vent condenser along with the process vent streams. The hot, stripped oil free of solvent is accumulated in a reservoir, then is pumped through a water-cooled exchanger and recirculated back to the absorption column for repeated absorption of solvent. Here, as in the vent condenser, the coldest possible water is used, because the more cool the oil the greater capacity it has for absorbing more solvent from the vent air.

Figure 5 shows the improvement in solvent recovery which can be expected by the use of a mineral oil absorption system as against a simple water-cooled vent condenser, in terms of the potential solvent savings in dollars/year plotted against vent vapor temperature leaving the vent condenser, all for a 1000 TPD plant. Other plant capacities show proportional savings. The warmer the cooling water and the vent temperature, the greater the saving, in fact doubling as the vent temperature goes from 62 F to 86 F, with savings of from \$40,000/year to \$80,000/year.

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