

# Solvent Recovery

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## ABSTRACT

A brief overview is presented of the economic significance of solvent recovery in the oilseed extraction industry in view of the steep price advance of hexane since 1974. The present solvent recovery practices in oilseed plants are outlined with addition of operating techniques since 1976. Also, industry trends are noted in terms of improved equipment. Information is presented on the expected solvent losses in various recovery steps in the normal solvent plant operation. Techniques for measuring solvent losses in vent gas effluent, waste water effluent and extracted oil production stream are outlined. The areas of greatest solvent loss are the desolventizer-toaster (DT) meal outlet stream and the various mechanical leaks in the process machinery. Some discussion is presented on the lack of ability to better define the solvent loss associated with meal discharge stream from the DT unit. Various deficiencies are pointed out in testing procedures and a suggestion is made for increased work in the field to substantiate a better value for solvent loss determination in DT meal.

Solvent recovery in the USA extraction plants is receiving much emphasis due to the high price of solvent. The price of hexane has risen from 20 cents/US gal to about \$1.50/US gal in the last 10 years. At a standard usage of 3/4 US gal per US short ton of seed, the solvent cost of milling one bushel of seed has risen from ca. 1/2 cent/bushel to about 3.5 cents/bushel and promises to go even higher in the future. For a 1000 ton plant the daily solvent cost is ca. \$1200 U.S. Consequently, much attention is being given to increasing recovery efficiencies of hexane in the ca. 100 extraction plants in the USA.

During the 1960s, most solvent plants in the USA were retrofitted with mineral oil systems. Today the standard plant has cooling tower condensers at 27 C (summer conditions) plus a mineral oil system. Some plants are now using supplementary condensers ahead of the mineral oil system using either mechanically chilled water at 5 C or well water at 12 C where ground water is available. This practice reduces the load on the mineral oil system by some 30% and increases the efficiency of the system. Some plants use carbon adsorbers as a back-up for the mineral oil system where the vent gas from the mineral oil system is passed through a carbon bed before discharge to the atmosphere. Although the carbon system is very effective and automatic recycling units are quite efficient, this practice is not widespread because it is generally more advantageous to install an oversize mineral oil system. Mineral oil absorption is the mainstay of solvent recovery practice in the USA and increased attention is being given to installing larger, more efficient units.

Recent papers should be of interest to operators of mineral oil recovery systems (1-4). There are other sources of solvent losses in extraction plants, however, apart from just the vent gas system, i.e., several exit streams and mechanical leaks.

Effluent stream	Solvent recovery system
Meal	Desolventizer-toaster
Oil	Oil stripper
Waste water	Waste water evaporator
Vent gas	Mineral oil system
Mechanical losses (leaks, drips, etc.)	Better plant design

Solvent loss in extraction plants is greatly affected by

the continuous running time of the plant. Plants with good maintenance schedules that can run for 30 days without a major breakdown are going to be much more efficient for solvent recovery than a plant that is up and down to repair breakdowns or plug-ups of material that release solvent vapors to the atmosphere. A plant manager will have to decide if his maintenance plan is contributing to unusual solvent losses by looking at frequency of downtime. The figures presented in this report are based on well maintained plants.

*Waste water evaporator.* Solvent loss from a waste water evaporator is negligible if exit water is boiled for 5-10 min and exited to the sewer at 92 C minimum. This waste water must be monitored periodically because an upset condition in the plant could force excess hexane from the water separation tank and overload the evaporating capacity of the waste water evaporator.

*Oil stripper.* Recent data published by the USDA (4) related flash point in the crude oil from the final oil stripper to hexane content in parts per million (ppm). The hexane content of crude oil is 1000 ppm at a flash point of 120 C, which is the trading standard in the USA. At a flash point of 160 C, the hexane content is 550 ppm. When graphing these results the hexane content of soy oil approaches an asymptote of 500 ppm, which means that any soy oil, regardless of the flash point, will have a minimum of 500 ppm of hexane chemically bound. These results of 500 and 1000 ppm of hexane as shown in Table I and are, respectively, 0.035 and 0.070 gal/ton solvent loss.

*Vent gas effluent.* One of the most effective ways to control hexane losses through the vent gas system is to smell the vent gas effluent. The threshold smell level of the human nose is ca. 500 ppm of hexane, and an effective final unit on a vent gas stream such as an adequate mineral oil system should produce vent gas streams of under 500 ppm. Therefore the vent gas stream when sniffed by a human nose should not indicate the presence of hexane and will be below 500 ppm. At 200 ft<sup>3</sup>/min vent gas flow with 200 ppm of hexane, the solvent loss for a 1000 ton plant should be below 1 gal/day. At 1000 ppm hexane, the daily loss would be 5 gal/day or 1% of the total solvent loss of a well run plant.

Thus in a well run 1000 ton/day soybean plant averaging .5 gal/ton of solvent loss, we can only identify 7% of the solvent loss in the oil and 1% of the solvent loss in the vent gas. We must therefore look for 92% of the solvent loss in other loss points.

Table I indicates typical solvent loss quantities assuming total usage is .5 gal/ton for a better than average plant and .7 gal/ton for an average plant on soy; or 500 or 700 gal/day for a 1000 ton/day soy plant.

Literature values of desolventizer-toaster (DT) exit soy meal show results between 100 ppm of hexane and up to 3000 ppm. But if we assume DT exit meal values of 500 and 1000 ppm it shows that the DT solvent loss can vary between 142 and 284 gal/day. By difference, we arrive at a mechanical loss of 322 gal/day for a better than average plant and 341 gal/day for an average plant. This is assuming a continuous running plant with waste water evaporator discharge in excess of 92 C and with a good effective mineral

TABLE I  
Solvent Loss Quantities

	Better than average plant		Average plant	
	gal/ton <sup>a</sup>	gal/day	gal/ton	gal/day
DT meal	.142 (500 ppm)	142	.284 (1000 ppm)	284
Oil	.035 (500 ppm)	35	.070 (1000 ppm)	70
Vent gas <sup>b</sup>	.001 (200 ppm)	1	.005 (1000 ppm)	5
Mechanical	.322	322	.341	341
	.5 gal/ton	500 gal/day	.7 gal/ton	700 gal/day

Continuous plant operation.  
Waste water evaporator at 92 C or better.  
With mineral oil system.  
1000 ton/day soybean plant.

<sup>a</sup>US gal/short ton.

<sup>b</sup>200 ft<sup>3</sup>/min air.

oil system.

Table I shows that the most solvent is lost through mechanical losses, i.e., seals, packing glands, gaskets, etc. To aid in determining solvent losses to the atmosphere, Foxboro is now producing a portable organic vapor analyzer which can be carried into a solvent plant and hexane readings can be obtained instantaneously on air samples taken at potential leak sources. This analyzer uses the gas chromatographic (GC) technique and is certified for use in Class 1 areas. The unit costs ca. US \$6000.

There is growing doubt in the industry that current analytical and sampling methods are adequately reporting true solvent contents of DT meal in the bottom tray of the desolventizer. Problems with these methods are as follows.

*Flashing of the sample.* The sample for analysis is taken after the meal leaves the bottom tray of the DT and is captured in a container. This flashing is very rapid, with 105 C meal flashing to 60 C in a matter of seconds. This flashing must release solvent to the atmosphere during sampling.

*Inadequacy of sample size.* The method of Du Puy (5) is probably the most widely used analytical method. The test calls for .1 to .5 g of meal sample. This sample size is far too small to be representative of DT meal with its thin flake, thick flake, small balls and large balls. The sample size should be more in the order of 25 or 50 g to be representative.

*Time lapse between sampling and analysis.* Chessen (6) reported that hexane in DT meal appeared to become modified or bound as the sample aged. It appears that the analysis should be performed immediately after sampling.

*Destructive degradation of sample before analysis.* Todd (7) recognizes the value of large samples (5-25 g) that are chemically treated for 30 min prior to analysis with excess water, antifoam, TIDE and sodium sulfate to free bound hexane from the matrix of the meal. This degradation of the sample takes on significance when one recognizes the severity of plant desolventizing treatment that is done in the presence of superheated steam at 105 C for 15 min in a modern DT generally 5-7 trays high; and there is still much discussion about how much residual solvent is left.

Table I shows that 30-40% of the total plant loss is in DT meal using the conservative analytical results reported. Chessen (6) says that 15-50% of the total reported loss of hexane is in the DT meal, and these results he acknowledges as probably low because of storage and sampling problems. With improved sampling and analytical techniques from the DT, it seems entirely possible that 70-80% of the total reported solvent loss could be in the DT meal. It is the thrust of this paper that the principal loss source of hexane in oil

extraction is from the DT when all sampling and analytical factors are taken into account.

In the absence of improved sampling and analytical techniques, it is going to be difficult to convince plant owners and operators that a new DT costing probably half a million dollars is necessary to reduce solvent losses. But it is felt that for the industry at large to achieve solvent losses of 25-30% of present loss levels the performance of existing DT units must be critically evaluated.

It would seem that the magnitude of the problem, in view of the significant amount of solvent savings possible and the large capital expense necessary to the industry as a whole, should warrant the formation of an industry task force composed of public and private laboratories, technicians and engineers studying on plant sites to determine the actual solvent level of meal emitted from the DT. A specially designed sampling system could be designed and set up, withdrawing measured quantities of meal from the bottom tray of the DT, cooling, conveying and preparing the sample of meal and effluent air for modified GC analysis on the site.

It is interesting to note that there are about half a dozen soybean extraction plants in the USA, out of a total of some 100 mills, that are privately admitting to .2 to .3 gal/ton solvent loss. Most of these mills were built or remodelled in the last 5 years. Hence the opportunity based on much future work is offered to the remaining 90 odd mills to achieve similar results.

The first area that must be concentrated on for better solvent recovery in the next decade is improvement of DT performance. More studies are needed on DT design and operation based on improved sampling and analytical techniques suggested above.

The other area needed for improved solvent recovery is better design of equipment with regard to seals, gaskets, flanges, etc. The equipment design must be improved over those of the present, which were designed in the times of low cost solvent.

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