

hand, if one were to imagine the conveying of bone dry dirt to a screw press, he would expect that this material would either immediately plug the press or would be conveyed on through with very little work done. It is apparent therefore that any material to be handled by a mechanical screw press must be in a specific physical condition to permit efficient operation of the press. It is true that a material like flaxseed may be handled by a screw press with no preparation other than cleaning. In fact, in the early days of screw press operations on flaxseed, cold press linseed oil was made in this fashion. However if the greatest yield of oil is to be obtained with a minimum of solids (foots) escaping through the barrel bars, the seed must be rolled and cooked to solidify the protein and phosphatide constituents of the seed.

The cake produced on a mechanical screw press is usually ground and sold as meal. The oil that flows from the presses is first settled, as in hydraulic operation, and then filtered, ready for sale as crude oil.

It is apparent from the foregoing that a mechanical screw press with a specific worm arrangement and bar spacing cannot handle, with equal efficiency, a high-fiber seed and a low-fiber seed, or a cooked material and a raw material, or a thoroughly cleaned seed at one instance and a high chaff seed at another instance. There are, in fact, more than 50 worm shaft and bar spacing arrangements in use for the many different materials being processed by mechanical screw presses. To obtain the maximum efficiency from a mechanical screw press therefore seed preparation methods must be controlled, seed cleaning procedures must be effective, and then proper worm

arrangements with maximum effective bar spacings must be utilized.

In a subsequent paper F. P. Parkin will discuss the use of mechanical screw presses in combination with solvent extraction equipment for the extraction of oil from high oil content vegetable seeds. It should be indicated however that the generalities of the preceding discussion on expression of oil with the mechanical screw press are adaptable to the use of the mechanical screw press as a prepress machine. In order that this adaptation may be economical however the machines must be designed for high capacity. The residual oil content of the prepressed cake is, of course much higher than the 3.5 to 4% goal of single press operations.

The advances that have been made in the past years in mechanical screw press design and operation and the work that is presently being undertaken in the improvement of these machines indicate that the mechanical screw press will always have a utility in the processing of vegetable seeds. It appears that the mechanical screw press and solvent extraction and a combination of the two will go hand in hand to provide even more efficient production of vegetable oils for those engaged in this field.

Acknowledgment

The author wishes to thank The French Oil Mill Machinery Company and the National Cottonseed Products Association for certain pictures used in this manuscript.

REFERENCES

1. Ennis, W. D., "Linseed Oil and Other Seed Oils," D. Van Nostrand Co., New York (1909).
2. Bailey, A. E., "Cottonseed and Cottonseed Products," Interscience Publishers, Inc., New York (1948).

The Solvent Extraction of Drying Oils

F. P. PARKIN,* Minnesota Linseed Oil Company, Minneapolis, Minnesota

THE solvent extraction of drying oils is the application of modern science and engineering to the winning of oils from seeds and nuts. These oils through past history have found many uses in the preservation of documents and paintings. The first oils were very crudely obtained in wedge presses or



F. P. Parkin

by steeping the seeds and collecting the oil that floats on top. From this humble beginning in which only a small portion of the total oil in the seed was obtained, we have progressed to our present-day operations in which less than 1% extractable fat is left in the meal or residue from the seeds or nuts. The need for solvent extraction of drying oils has been brought about mainly by the economics imposed on the industry in general. That is to say, the percentage of total oil recoverable

by solvent extraction as compared to mechanical pressing of the seeds is much smaller in the higher oil content drying oil seeds than will be found in the case of soybeans and other oil bearing materials that have a low oil or fat content.

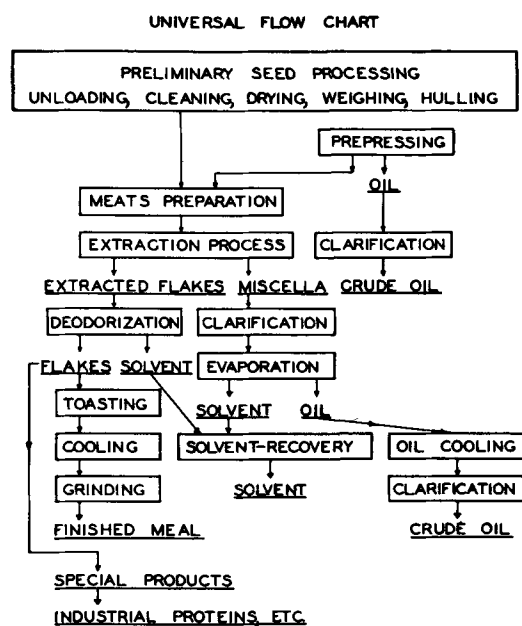
For purposes of comparison as to why solvent extraction has not been applied to the higher oil content seeds previously we may take the case of low oil content material, such as soybeans, and increase the yield of oil approximately 20% by solvent extraction over mechanically pressing the beans. In the case of flaxseed, which is considered to be a medium oil content seed, the yield of oil is increased by about 6% while in the case of the higher ranges of oil contents the oil yield is increased only about 3% by solvent extraction. Thus economics dictated that there must be high oil prices to warrant the additional cost of processing by solvent extraction.

Although our commercial experience to date has been mainly the solvent extraction of flaxseed to obtain linseed oil, the employment of solvent extraction for obtaining castor oil for treating to be a drying oil, has been used for years. Therefore, we should include oils that have been used, are now being used,

* Present address: 4815 Nokomis, Minneapolis, Minn.

and those that have good potential uses as drying oils. Of these we will classify them as those being processed without dehulling, such as flaxseed, perilla, and chiasseed; and those requiring some form of dehulling before processing, such as tung (1, 2), oiticica, safflower, walnut, stillingia (3), and wild gourd (4, 5).

The economic picture of the past five years has made it necessary to solvent-extract the drying oils in order to obtain greater yields of oil. This naturally has brought about a rather thorough study and application of available equipment to the processing of drying oil seeds. In general, all of the drying oil seeds or beans contain more than 25% oil in the whole seed or kernel fraction, thus necessitating a pre-pressing of the material to remove a sufficient quantity of oil so that it may be properly conditioned for solvent extraction.



In Figure 1 a flow chart shows the path or paths that will be taken by the various materials. In general, the material to be processed will follow the usual steps necessary for the particular materials. In many cases the material may be withdrawn at various stages of processing for the manufacture of special products. The main steps carried out in the processing are: the proper conditioning of the material for bulk storage, preparation of the material for pre-pressing or extraction, pre-pressing and extraction, desolventizing of miscella, and the desolventizing of extracted solids. The following types of extractors are either being used or have been used successfully or have been proposed for use in the solvent extraction of drying oils:

Basket type or Bollman extractor as manufactured by the French Oil Mill Machinery Company and the Blaw-Knox Company.

Bonotto and modifications as manufactured by Allis-Chalmers, the V. D. Anderson Company, and Struthers-Wells. Conveyor type, such as the Hildebrand, which is in the form of a "U" tube.

Detrex horizontal or inclined conveyor.

Crown Iron Works, extractor as developed at the University of Iowa (6), in which a Redler conveyor is used.

Kennedy (7) extractor, which uses curved paddles to move solid materials from one section to another.

Centrifugal and Rotoceel methods of Blaw-Knox.

Horizontal basket of the French Oil Mill Machinery Company.

SOME of this equipment has been discussed in previous articles (10, 11) and I shall endeavor to explain its application to obtaining linseed oil from flaxseed. Although flaxseed may be totally extracted, it is more feasible to pre-press and avoid many mechanical difficulties that arise from a total extraction of flaxseed. One point might be made, that it would be very desirable to have the seeds decorticated before pre-pressing as this would only mean handling about 60 to 70% as much material, thus eliminating the hulls which normally contain only about 1% extractable fat. Of course, this also would apply to many other seeds.

Pre-pressing has been mentioned as a desirable feature in the processing of seeds with high oil content. This practice is not new, but methods of pre-pressing in this country differ from the European (8) practices which have been carried on for many years. Americans have taken the pre-pressing as a one-step operation, attempting to modify high pressure expeller and screw presses to give greater capacity with lower power consumption while the European practice has been to make successive pressings on the same material. As many as three pressings may be made on a material such as flaxseed to reduce the oil content to 15%. This has been done on both ground and unground, and heated and unheated material. The variation in the processing is undoubtedly done to favor further processing that would be carried out on the oils.

In discussing the European methods, it may be well to mention the batch system versus the continuous counter-current methods in regard to solvent extraction. Lower solvent losses and a lower solvent ratio are claimed for batch extraction over continuous counter-current methods. The efficiency of extraction is no doubt equal to or possibly even slightly better for the batch; however to weigh against this a large amount of labor is required for batch extraction. It is stated that it takes eight men in a batch system to produce the same amount of finished material that one man can do in a continuous operation. This immediately tells why this country uses the continuous method.

Another point that may be easily overlooked is that in a batch operation one is continuously opening up the equipment for discharging and charging, and this in effect simulates the condition in shutting down a plant, and perhaps all operators of solvent extraction plants will agree that one of the greatest dangers involved in a solvent extraction plant is not while the plant is operating but while starting up and shutting down.

Two methods of pre-pressing flaxseed may be followed, and commercially both methods are used: cold pre-pressing and hot pre-pressing. In the cold pre-pressing the whole seed is used and is fed through a screw press or expeller, in which case an oil is obtained which is comparable to the old cold pressed

linseed oil of commerce. In the hot pre-pressing the oil quality will be somewhere between this cold pressed oil and the oil obtained on total hot expeller operation, depending mainly upon the temperature and extent of the pre-pressing. It has been found that good operation can be obtained in a subsequent preparation and extraction operation if the pre-pressed cake contains 18% or less oil. The solid material coming from the pre-pressing operation is put through a breaker or cracking roll and into a conditioner where temperature and moisture can be adjusted so as to get proper flaking for the extractor. This will naturally vary with different plants, depending upon operations carried out on the various materials. The type of extractor used will determine the critical point in the preparation of the material for extraction.

In the case of the basket or Bollman type extractor, which is the closest to the perculator type extractor, it is necessary to have the proper flaking of material in order to obtain proper drainage and good extraction. In the other types of extractors, in which possibly all can be classified as immersion types, it is necessary only to condition the material so that proper penetration of the solvent is obtained without the excessive production of fines. The pre-pressing operation should be carried out so as not to obtain a glaze or impervious surface on the particles. Schmidt and Webber (9) have attempted to use this principle by controlling the rate of penetration of the solvent by the particle size of the material to be extracted.

To my knowledge the only continuous extraction operation to date in this country on drying oils is carried out on flaxseed. In the application of these various types of equipment success depends upon the treatment of material to be extracted before it is placed in the extractor.

For the basket type of extractor it is necessary to have a material that will give good drainage without channeling. This means a tough, thin flake that does not disintegrate to make fines as the oil is removed. The type of flake produced will depend largely upon the oil content, the amount of heat (cooking), and the moisture content.

With the high cost of tung oil it is my understanding that the cake from expeller operations has been extracted to obtain the last traces of oil. This would probably also enable the residue to be used for something other than fertilizer. The solvent extraction of tung oil is well covered by Holmes and Pack (1), who have included the work of McKinney (2). They have advocated two solvents, either normal hexane or trichlorethylene. There have been many reports of extracted tung oil readily solidifying by changing to the Beta form. It is thought that the best possible operation of a solvent extraction plant will overcome this difficulty. McKinney in his work with the Kennedy extractor has shown that tung nuts may be extracted, and the extraction of press cake is very easily accomplished.

The commercial extraction of safflower and other seeds and nuts will probably develop in accordance with the knowledge gained from processing flaxseed and tung nuts when the demand for more of these oils makes it possible to build economical plants.

The references at the end of this paper indicate theory and practices of making solvent extracted oils which may be a repetition of material given during

the short courses on edible oils in the past two years (10, 11, 12).

UP to this point the discussion has been about obtaining the oil from the seeds. Other important steps in the successful operation of a plant comprise the removal of the solvent from the oil and from the flakes or extracted residue. In both it is necessary to remove all of the solvent so as to recover the solvent and to make the product salable as small traces of solvent will definitely have an adverse effect on the quality of the oil and the meal. In the case of the oil the solvent has to be removed without deleterious effect upon the oil as concerns its uses or further treatment. This will necessitate, in practically every case, a final removal of solvent under vacuum and sparging steam.

The quality of the product appears to be very much the same regardless of the type of distillation equipment used, whether it be pre-evaporators followed by film evaporators and bubble cap or packed column strippers. In the removal of solvents from the flakes or extracted residue the processing may be governed mainly by what future use the material is to be put, such as obtaining a high protein concentrate for glue or other such uses as protective colloids, etc. Two methods may be used in general, either vapor desolventizing or steam jacketed horizontal cookers, followed by a final deodorization which is accomplished by direct injection of low pressure superheated steam. The spent solids are then toasted, cooled, and ground to make finished meal. The flakes may also be taken from the deodorizer to equipment for special processing other than meal manufacture.

While the preparation of the material for solvent extraction is all-important in getting the oil from the seeds, the quality of the oil will depend in a large measure on the type of solvent chosen for the removal of the oil from the seed. Many factors are involved in determining the solvent to be used for the extraction of any given oil. The ideal solvent should extract an oil of good quality and high yield and leave a cake or residue of greater value than previously obtained. The cost of the solvent, being extremely important, is not necessarily determined by the price per pound or gallon of the solvent but upon its loss during operation and the quality of oil produced. That is to say, a solvent of high volatility, although it may be much cheaper per pound, would still be the more expensive solvent to use because of its high loss during operation, or a cheap solvent may not be the best to use because of the additional treatment necessary on the oil obtained.

Many solvents have been considered for solvent extraction as: straight chain narrow cut petroleum hydrocarbons, chlorinated hydrocarbons, aromatics, alcohols (13, 14, 15), esters, ketones, etc. To determine the solvent to use one must consider the available condenser water or other means of recovery of the solvent which will determine the boiling range of the desired solvent. If the specific gravity of a solvent is too high, separation of the solid material may prove slow or impossible in counter-current solvent extraction, particularly in the total immersion type extractors. Specific heat and heat of vaporization are important from the standpoint of cost of recovery of the solvents.

WITH these points in mind, first the straight chain hydrocarbons will be taken up. Propane, butane, and pentane possess such low boiling points as to require special pressure equipment to maintain them in the liquid state. Hexane has a boiling point of approximately 154°F. and can be condensed without too much difficulty so it is used in larger quantities for solvent extraction than are any of the other petroleum solvents. Although it is not the ideal solvent, it does have the distinct advantage of being cheap and plentiful in supply. Heptane has been found to be very useful where a higher temperature than hexane is needed to obtain miscibility with the oils being extracted. The extraction of castor oil (16) is an example of this.

Chlorinated solvents have been used for many extraction processes. These include chloroform, carbon tetrachloride, ethylene dichloride, trichlorethylene. Of these trichlorethylene (6) is the only one finding much favor in industrial application as far as oil extraction is concerned. It is non-hazardous as concerns fire and is a stable compound, which boils at a definite boiling point so that its removal from the extracted residue is relatively easy. There are some disadvantages to be weighed against these advantages: high specific gravity of these compounds in relation to the petroleum solvents, the high toxicity, the extraction in some instances of undesirable waxes and other constituents present in the seeds, and also the higher cost.

The aromatics have generally been eliminated because of their tendency to dissolve a much larger quantity of color constituents present in the seeds, thus yielding extremely dark oils.

The alcohols are coming into favor, being able not only to dissolve the oil from the seeds but also to perform a partial refining by preferential solution of fatty acids and minor constituents present in the oil. The disadvantage of these solvents however is low miscibility with oil, thus requiring higher temperatures for operations.

Of the ketones tried, acetone seems to offer the best possibility as it is extremely good as a solvent for oils and also allows for some refining to be done on the miscella prior to evaporation. The esters have been of little use to date as commercial solvents for oil extraction.

The last three groups, the alcohols, ketones, and esters, have a further complication in that they are either miscible with water or form constant boiling mixtures with water and therefore tolerate considerably more moisture than do the other groups of

solvents mentioned. The presence of small amounts of water in these solvents will change the solubility of the oil therein and thus render them rather difficult to use because of resulting poor plant control. The water also exerts some influence in dissolving sugars and other water soluble materials along with the oil and therefore there is the additional problem of separating these materials from the oil since they are usually found in the form of syrups.

In summary, it may be commented that the usual solvent extraction equipment has been or may be used for the extraction of drying oils when the seeds have been pre-pressed to remove the free flowing oil and reduce the oil content in the pre-pressed cake to about 18%. This allows the pre-pressed cake to be conditioned for proper flaking, or ground to a definite particle size to allow penetration to give successful operation of extractors, thus reducing the residual oil content of the finished meal below 1%. In the case of flaxseed the meal from solvent extraction gives as good results as the old processed meal when used as a high protein concentrate cattle food. Some of the other drying oils that may be solvent extracted may possibly be used for industrial proteins after practically all of the oil has been solvent extracted. At the present time it is estimated that 15 to 20% of the linseed oil produced is solvent extracted.

The author wishes to express his appreciation to the following for their assistance in providing descriptive literature and slides: the V. D. Anderson Company, Cleveland, O.; Blaw-Knox Company, Pittsburgh, Pa.; the French Oil Mill Machinery Company, Piqua, O.; and Vulcan Copper and Supply Company, Cincinnati, O.

REFERENCES

- Holmes, R. L., and Pack, F. C., *Bur. of Ag. and Ind. Chem. Bull.*, AIC 116, Sept. 1946.
- McKinney, R. S., *Ind. Eng. Chem.*, 36, 138-144 (1944).
- Bolley, D. S., and McCormack, R. H., *J. Am. Oil Chem. Soc.*, 27, 84 (1950).
- Ault, W. C., Swain, Margaret L., and Curtis, L. C., *J. Am. Oil Chem. Soc.*, 24, 289 (1947).
- Bolley, D. S., and McCormack, R. H., *Spring Meeting at Atlanta* (1950).
- Sweeney, O. R., Arnold, L. K., and Hollowell, E. G., *Bull.* 165, Iowa State College, Ames, Iowa.
- Lerman, F., Kennedy, A. B., and Laskin, J., *Ind. Eng. Chem.*, 40, 1753 (1948).
- Goss, W. H., "The German Oilseed Industry," Hobart Publishing Company, Washington, D. C. (1947).
- Schmidt, E. W., and Webber, W. F., U. S. Patent 2,430,535, November 11, 1947.
- Proceedings of the Short Course in Vegetable Oils, August 16-21 (1948), University of Illinois.
- Short Course of 1949, *J. Am. Oil Chem. Soc.*, 26, October (1949).
- Bailey, A. E., "Industrial Oil and Fat Products," Interscience Publications Inc., New York, p. 479 (1945).
- Singer, P. A., and Deobald, H. J., U. S. Patent 2,377,976.
- Beckel, A. C., Belter, P. A., and Smith, A. K., *J. Am. Oil Chem. Soc.*, 25, 10 (1948).
- Texas A. & M. Group, *J. Am. Oil Chem. Soc.*, 24, 370 (1947); 26, 719 (1949); 27, 273 (1950).
- Pascal, M. W., U. S. Patents 2,467,403 and 2,467,404.