

Suitability of solvent extraction for jatropha curcas

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Introduction

The research underlying this document was performed to judge the applicability of chemical extraction instead of cold pressing to gain oil from jatropha curcas seeds. Current practice for FACT members is to use cold pressing because of its simplicity and affordable investment cost at all scales. Extraction of oil from the seeds with a solvent could prove useful to increase the amount of oil per ton of seed.

History

Oilseeds are extracted in two primary ways, by mechanical pressing, or squeezing, and with petroleum solvents. Prior to the 1940's, mechanical pressing was the primary method used. Mechanical extraction, however, had its limits in terms of oil recovery. Because pressing generates heat and high temperatures damage both the oil and meal, an oil content of the presscake below 5-6% was difficult to achieve. Solvent extraction was developed because it allows a more complete extraction at lower temperatures. It begins to be economically attractive where large quantities of seed can be processed (at least 200 tons per day for continuous-feed processes); where storage, transportation, power, water, and solvent supply are adequate; and where occupational safety and training standards can be enforced. There are solvent extraction plants with capacities of up to 4,000 tons per day. In solvent extraction, the solvent drains through a deep bed of flakes and one of the critical requirements is to prepare a bed that will allow the solvent to drain easily.

Mechanical extraction

Figure 1 shows a block diagram for the mechanical pressing of cottonseed or sunflower seed meats whether they are being full pressed or pre-pressed prior to solvent extraction. The meats collect in a metering bin and then are fed at a regulated rate into the process. They pass first through a flaking mill and then a conditioner to complete their preparation before entering the screw press. In the press the flakes pass between a specially designed screw and a cage which is lined with steel screen bars spaced a fraction of an inch apart. An adjustable choke mechanism at the discharge end of the press creates the back pressure to "squeeze" the oil from the seed. The cage retains the extracted material or presscake and the oil passes thorough the openings into the screening tank. This oil contains some solids called fots. In the screening tank the larger fots settle to the bottom and are conveyed back to the inlet of the screw press. The oil is pumped into the unfiltered oil tank and then through a filter to remove any fots that may still be present. The oil, now free of fots, collects in the filtered oil tank and is then pumped to storage. Periodically, the flow of oil to the filter is stopped and oil from the press collects in the unfiltered oil tank. The filter is opened and cleaned. The filter cake, wet with oil, drops into a collection tank. It is then conveyed back to the inlet of the screw press. Part of the oil in the screening tank is cooled in a heat

exchanger and pumped over the cage to minimize the heat build-up generated by the pressing operation. This build-up must be controlled not only to prevent damage to the oil and presscake but also to prevent fusing of the cake into a solid mass that will not discharge easily from the press. The screw may also have a water cooled shaft to provide additional cooling.

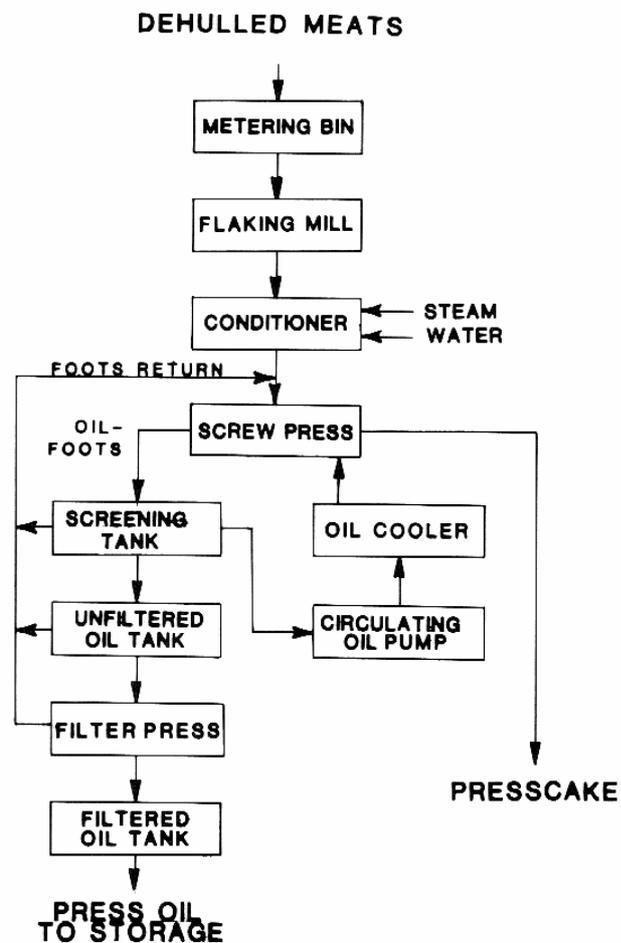


Figure 1 – mechanical extraction [1]

As shown in figure 2, the presscake from a full press operation usually is passed through a cake grinder to create a more uniform sized product for sale. Some processors also use a two-stage full press operation to obtain better oil yields. In this two stage operation the first press acts as a pre-press in that it is operated to recover only part of the oil; the presscake is then passed through a cake sizer to reduce the size of the larger pieces and then a flaking mill before entering a second press which extracts the remainder of the oil. The oil from each stage mixes in the screening tank; the second stage presscake then goes to a cake grinder.

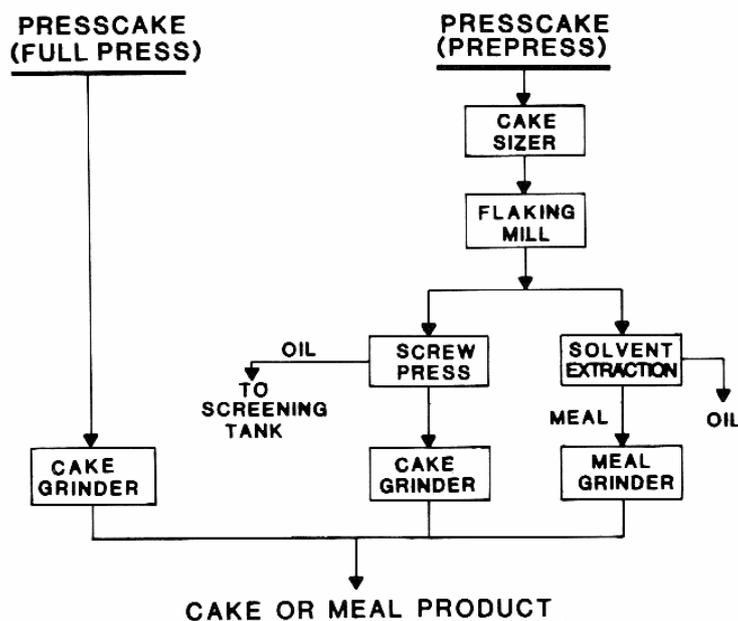


Figure 2 – processing of presscake [1]

Solvent extraction

Even with a two-stage operation, the oil content of the final presscake will be about 5 – 6%. Extracting the presscake with a solvent offers a way to reduce the oil content to less than 1%. In a pre-press solvent extraction the press is operated to give a presscake with 15 – 18% oil and the balance is recovered during solvent extraction. The presscake is first passed through a cake sizer and flaking mill before being conveyed to the solvent extractor. The meal is ground prior to sale as animal feed.

Many solvents, both petroleum and non-petroleum types have been used in the laboratory to extract oil from oilseeds. The most important examples are [2]:

- Hydrocarbon solvents (hexane, heptane, pentane): Usually hexane is used. Extraction-grade hexane has an *n*-hexane content between 48 – 98% and a narrow distillation range. It is free of nitrogen and sulphur and unsaturated compounds and sufficiently stable to be used indefinitely. Commercial heptane might be preferred for the extraction of castor oil, which is not freely miscible with hydrocarbons except at elevated temperature.
- Halogenated solvents (trichloroethylene, dichloromethane): because of environmental concerns the commercial use of halogenated hydrocarbons in solvent extraction has not occurred.
- Water (with and without enzymes): Water only finds application as a solvent in the extraction of oil from palm, olive and coconuts. Its drawbacks are more than 10% residual oil in cake and energy-intensive separation and drying.

Enzymes can be used to digest the cell wall to set the contents free. A 1:2 hexane-water mixture was reported useful. Although enzymatic assisted extraction currently is economical only in olive oil processing (mild conditions, high oil quality), it has potential for future use.

- Acetone: only works well on cottonseed oil, causes off-flavour in the meal.
- Alcohols (ethanol and isopropylalcohol): these seem promising as biorenewable solvents.
- Supercritical solvents (mainly CO₂). At the Dutch Twente University research is done on the use of supercritical CO₂ as a means of rupturing the cell walls to set the oil free.

From these hexane is the only solvent used commercially on a large scale. Hexane is a highly flammable, colourless liquid that readily forms explosive mixtures with air. Long term exposure to this toxic air pollutant can cause permanent nerve damage in humans. Hence care must be taken to prevent emission of hexane from the closed system and to prevent sources of ignition near the extraction system.

Both enzyme-assisted aqueous extraction, ethanol and isopropylalcohol seem promising for application on the shorter term in developing countries. Supercritical CO₂ is interesting because of its non-toxic nature. More research will have to be done before these alternative solvents can be used commercially.

Batch-wise operation

Like pressing, solvent extraction can be done with equipment that processes the oilseed in batches, or with equipment that processes it continuously. A continuous extractor is not considered economically practical unless it processes at least 200 tons per day. Batch solvent extraction is likely to be the appropriate method if you plan to process less than 200 tons of seed per day, but enough to yield oil in commercial quantities.

Very few batch plants are in use today. A batch solvent extraction plant can be as simple as an enclosed steel tank with a false bottom made of screen or metal slats. The flakes are dropped into the tank, where they lie on the false bottom. The tank inlet is closed, and solvent is pumped in to flood the bed of flaked oilseed. The solvent is allowed to contact the seed for 10 to 20 minutes; then the drain valve at the bottom (under the false bottom) is opened to complete the extraction. After the final extract has been fully drained, steam is introduced into the bottom of the extractor. This evaporates the solvent out of the flakes. This combination of steam and solvent is piped as vapour into a condenser that contains water-cooled tubes. The solvent is lighter than water, so it is readily freed of water by standing in a tank from which water is decanted, or overflowed. The flakes now are nearly solvent free, but are wet from the steam treatment. They are conveyed out of the extractor to a steam-heated dryer to reduce the moisture to about 12% for best storage quality. Most of the washes, or miscellas, are saved and reused on a later batch. However, fresh, oil-free solvent must be used for the final wash of a batch. And the first, oiliest miscella is pumped to a steam-heated, tubular evaporator, which boils most of the solvent out of the mixture, recovering solvent for reuse. The oil then goes to a vacuum stripper, where it is heated to about 100 °C and steamed as it passes down through a series of steel baffles or a column of stoneware rings or saddles. The purpose is to expose every portion of the oil to steam, which is needed to remove the last 5 to 10 percent of the solvent from the oil.

Continuous operation

The heart of any solvent extraction process is the extractor which must convey the flakes and provide the required residence time in contact with the hexane. The percolation extractor is the most used type today. The hexane is pumped onto the flake bed before the discharge of the spent, extracted flakes; the flakes are then allowed to drain before dropping into a discharge hopper. The hexane flows countercurrent to the flow of flakes increasing in oil concentration each time it is removed from one of the drainage areas of the extractor. The hexane-oil mixtures are called miscella, and the most concentrated miscella leaves the reactor after draining through the feed flakes. Each time a miscella is returned to the extractor it is spread over the bed of flakes, percolates down through the bed, and leaves the bottom through a perforated plate, mesh screen or wedge wire screen. There are several types of percolation extractors on the market; they differ basically in the mechanical design utilized to move the flakes through the extractor. One carries the flakes in individual cells; another drags the flakes across a wedge-wire screen and a third uses a moving, perforated screen.

The balance of the extraction system consists of equipment to separate and recover the hexane from the miscella and extracted flakes as shown in figure 3.

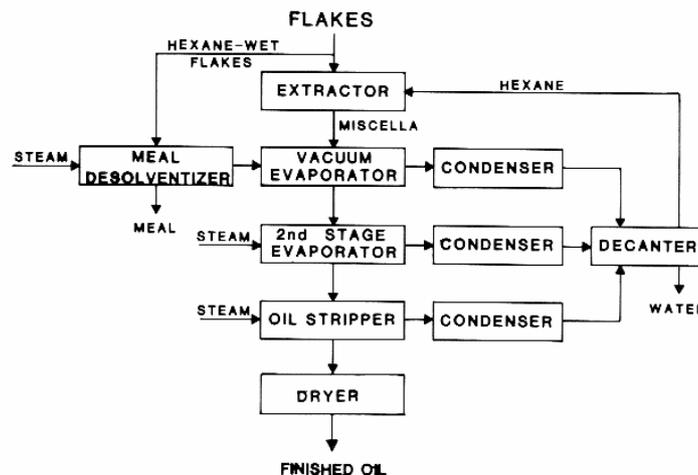


Figure 3 – steps in solvent extraction [1]

Hexane is recovered from the miscella in a three-stage evaporation system. The first stage is under vacuum and uses vapour from the meal desolventizer as the source of heat. A second evaporation step using steam is followed by steam stripping of the oil under vacuum. The oil is then dried to remove any traces of water and pumped into storage.

Meal desolventizing

The flakes leaving the extractor will carry 25 – 40% by weight of solvent, which is recovered by evaporation with steam in equipment designed for indirect heating, direct steam addition or a combination

of the two. In addition some oilseed meals, soybean being one of them, require cooking and toasting to a light brown color to produce a final product that has the required nutritional characteristics for use in animal feed formulations. This cooking and toasting is done most effectively with moist heat. Thus, the main function of a conventional meal desolventizer is to produce a desolventized, cooked and toasted meal for animal feed.

Hexane recovery

All the hexane and water which is condensed in the various system condensers collects in a decanter where, because of their difference in density, the hexane and water separate; the water is discharged to the sewer after heating to insure it is free of hexane; the hexane is returned to the extractor. The extractor, the discharge of the condensers and other process tanks all vent into a common header. Not only does this provide measure of safety in operating the plant by confining all hazardous vapours in a single system, but it also provides a way to recover any hexane in the vent vapour. In early extraction plants recovery of the hexane in the vapour, if any, was limited to passing the vapour through a condenser. Even before the rapid increase in petroleum prices, the amount of hexane lost represented a large portion of the operating cost. Absorption of the hexane on activated carbon was then tried and although this improved recovery considerably, it proved a difficult system to operate and maintain. The system that was then developed and is used today is absorption of the hexane in a mineral oil.

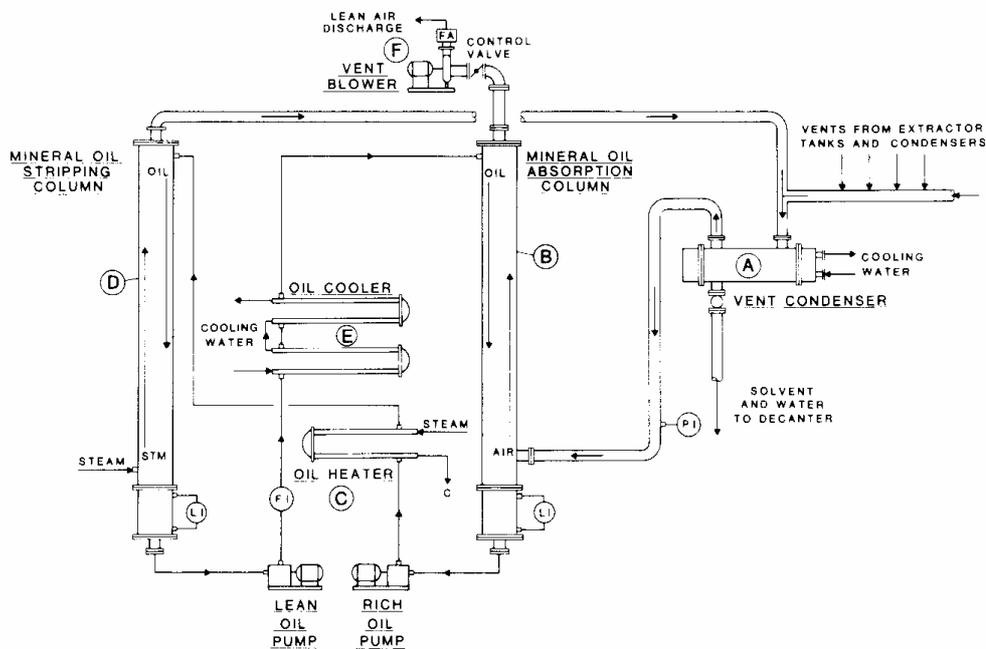


Figure 4 – Oil absorption system for hexane recovery [1]

As shown in figure 4 a vent blower (F) maintains a slight negative pressure on the entire extraction system. If there is a leak somewhere air will be pulled into the system instead of hexane leaking out. The vent vapour first goes through a surface type vent condenser (A) and then through the mineral oil absorption column (B). In the column the vapour flows countercurrent to a flow of cooled mineral oil. The vapour, now free of hexane exits the top of the column and is discharged to the atmosphere. The mineral oil absorbs the hexane, collects in the bottom of the column, and is pumped through a heater (C) to the mineral oil stripping column (D). In this column the heated mineral oil-hexane mixture flows countercurrent to steam which effectively strips the hexane from the mineral oil. The hexane vapour and steam from the top of the column are condensed in the vent condenser; the hot mineral oil, now free of hexane, is pumped through an oil cooler (E) and back to the absorption column. Newer systems may also include a heat recovery exchanger where the mineral oil-hexane mixture from the bottom of the absorption column is partially heated by the hot mineral oil from the stripper. Despite all measures to reclaim the hexane used, current solvent extraction plants consume some 1 – 4 kg of hexane per ton of oilseed.

Factors to consider in planning an oil extraction system: energy and resources

Solvent extraction of vegetable oils should be seen as part of a technological and economic system that includes far more than the extraction plant itself. Factors affecting the operation of a solvent extraction plant include: potential markets; nature, timing, size, and reliability of seed and solvent supply; adequacy and reliability of power, water, and transportation, and of maintenance and storage facilities; and ability to find and train personnel and rigorously enforce safety standards. Table 1 gives information about some of these requirements.

Table 1 – Estimated requirements for solvent extraction of vegetable oils [5]

Required per ton of seed processed	Units	Batch processing	Continuous processing
Steam	kg	700	280
Power	kWh	45	55
Water	m ³	14	12
Solvent	kg	5	4
Labor	person hours	0,8	0,5

Size of operation

The size of the operation is the most important factor in determining which kind of process will be used. For intermediate-scale operations (operations that process up to 200 tons per day), the choice is between batch solvent extraction and expeller (pressure extraction) systems. Batch solvent extraction systems operate more slowly and less efficiently, are more labour intensive and dangerous, and use greater quantities of solvent than properly designed continuous systems do. Because of these drawbacks, expellers are usually preferred for installations too small for continuous solvent systems. However, there

are instances when expeller extraction is not suitable for a small operation; in those cases, batch solvent extraction may be the only practical way to proceed. Continuous solvent extraction should be considered only for systems that will treat 200 tons or more of seed per day.

Site and design

Solvent extraction plants are complex systems that must be carefully engineered for safety because of their special hazards. Because of the danger of explosion, solvent extraction plants need to be located a safe distance away from populous areas, and to be designed by experienced engineers. Installation of a plant without such engineering of details is a dangerous error.

Investment cost

The cost of solvent extraction plants is much higher than the cost of expeller extraction plants, usually about double. However, since a solvent plant recovers a greater proportion of the oil, it may still be the economically wiser choice. For example, solvent extraction should recover about 40 kilograms more oil per ton from dry soybeans than expeller extraction would.

Personnel and safety

It takes less labour but more sophistication to maintain and operate a solvent extraction plant than to maintain and operate an expeller plant. Two people per shift are required for the former, compared to three for the latter. The dangers of solvent explosion make tightly controlled procedures necessary. Workers must be trained to have a wholesome fear of exposure to the solvent and of solvent leakage.

Reliability of throughput

For continuous solvent installations especially, it is essential to be able to depend on a steady throughput. Unscheduled interruptions of production, or discontinuities because of the inability to transport the finished product, for example, mean that seed will pile up somewhere and possibly spoil, especially if storage arrangements are insufficient. Unanticipated interruptions of seed supply may cause buyers of oil and meal to turn to more reliable sources. Both batch solvent and expeller operations are less vulnerable to the effects of such interruptions than continuous solvent operations are.

Comparison of decentralised cold pressing to industrial extraction with solvents [4]

Decentralised cold pressing	Industrial extraction with solvents
Medium or small scale, co-operative or private enterprises	Large scale, mainly multinational concerns
Located near agricultural production	Located near central traffic points
Small capacity (typically up to 25 ton/day)	Large capacity (possibly over 500 ton/day)

Higher feed value and energy value of press cake (12 – 17% residual oil content)	Low feed and energy value of cake; oil content of extraction press cake <1%
Low investment cost	High investment cost
Low energy consumption (80 kWh/ton seed, no steam)	High energy consumption (470 kWh/ton seed, partially steam)
No use of chemical solvents or additives	Extraction with chemical solvent like hexane
No thermal conditioning of seed	Thermal preconditioning of seed
No waste water	Waste water from the refining (approx. 50 l/l oil)
Low security requirements	High security requirements
Few measures for environmental protection	Stringent measures for environmental protection

Conclusion

Because of several reasons the application of solvent extraction to *Jatropha curcas* seems too early at this moment. Main reason is that solvent extraction is only economical at a large scale (50 tons per day upwards), whereas none of the FACT partners currently has this capacity. Further reasons are of environmental nature (generation of waste water, higher specific energy consumption, emission of volatile organic compounds) and societal nature (working with a hazardous, flammable chemical). Finally research and ongoing experiences to date show that the quality of unrefined cold pressed *Jatropha* oil is satisfactory for both direct application (PPO) and for use as a biodiesel feedstock. Hence for the moment there is no technical need to develop chemical extraction, as would be the case with sunflower or soybean oil that are less suited for direct application after cold pressing.

A different solvent, like water with enzymes, supercritical CO₂ or bio-ethanol, would make a large difference in the objections against solvent extraction at this moment. For this reason the ongoing research into solvent technology is considered most desirable.

Literature used

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