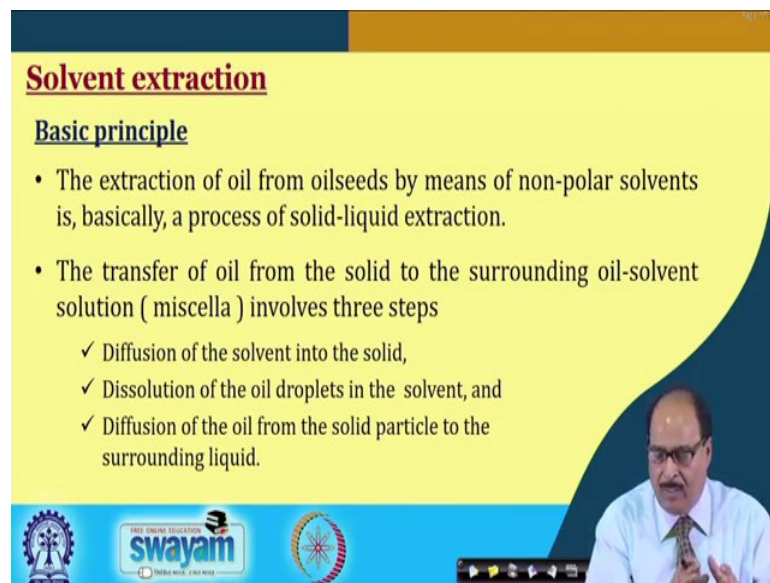


Novel Technologies for Food Processing and Shelf Life Extension
Prof. Hari Niwas Mishra
Department of Agricultural and Food Engineering
Indian Institute of Technology, Kharagpur

Lecture – 31
Extraction of Oil (Part 2 – Solvent Extraction)

In the second part of the lecture on Extraction of Oil from oil seeds, solvent extraction method will be studied.



Solvent extraction

Basic principle

- The extraction of oil from oilseeds by means of non-polar solvents is, basically, a process of solid-liquid extraction.
- The transfer of oil from the solid to the surrounding oil-solvent solution (miscella) involves three steps
 - ✓ Diffusion of the solvent into the solid,
 - ✓ Dissolution of the oil droplets in the solvent, and
 - ✓ Diffusion of the oil from the solid particle to the surrounding liquid.

The slide also features logos for IIT Kharagpur, Swayam, and a small video inset of Prof. Hari Niwas Mishra in the bottom right corner.

Solvent extraction

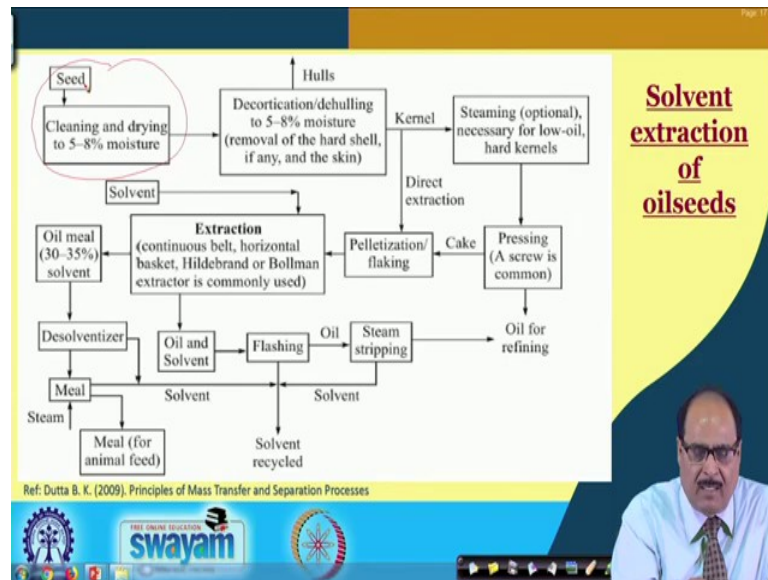
Basic principle

- The extraction of oil from oilseeds by means of non-polar solvents is, basically, a process of solid-liquid extraction.

The basic principle of the process is the prepared oil seed is brought into intimate contact or direct contact with a suitable non-polar solvent.

- The transfer of oil from the solid to the surrounding oil-solvent solution (miscella) involves three steps:
 - ✓ Diffusion of the solvent into the solid
 - ✓ Dissolution of the oil droplets in the solvent
 - ✓ Diffusion of the oil from the solid particle to the surrounding liquid.

These 3 processes actually result into the final recovery of oil. And, depending upon how these processes are managed during the extraction process by properly selecting the appropriate parameters during the extraction and operation of the machines etc. that will govern these steps and the recovery of the oil.

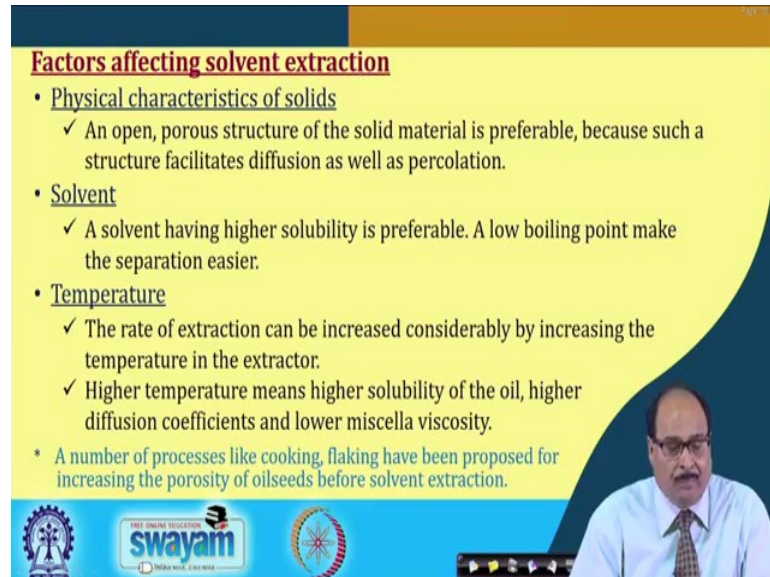


Here an overview of the extraction process in a factory there is the material flow and how the solvent extraction process is carried out is shown. The seed is cleaned and dried to suitable moisture content, then it is sent to the decorticating or dehulling where hard shells are removed, and then these dehulled materials are taken to the solvent extraction unit either directly or through the pelletizing/flaking units. The extraction unit may be a continuous belt extraction, horizontal box extraction or other different types of extraction which are commonly used in the industry.

Finally, oil is obtained as miscella which is a mixture of oil and solvent. Later the oil is recovered from miscella by flashing or stream stripping and using appropriate distillation method; the separated solvent is again recycled.

The meal also contains approximately 30 to 35 % of the solvent. This is passed to the desolventizer where again suitable heat treatment is given to the meal, and then solvent is recovered, recirculated to the extraction unit, and the desolventized meal is toasted or given some heat treatment.

In fact, at present in most of the cases the meal is used for animal feed, but it has a great potential for food applications. This can also be used for preparation of meat analogues or meat extender.



Factors affecting solvent extraction

- Physical characteristics of solids
 - ✓ An open, porous structure of the solid material is preferable, because such a structure facilitates diffusion as well as percolation.
- Solvent
 - ✓ A solvent having higher solubility is preferable. A low boiling point make the separation easier.
- Temperature
 - ✓ The rate of extraction can be increased considerably by increasing the temperature in the extractor.
 - ✓ Higher temperature means higher solubility of the oil, higher diffusion coefficients and lower miscella viscosity.

* A number of processes like cooking, flaking have been proposed for increasing the porosity of oilseeds before solvent extraction.

The slide also features logos for Swayam and other educational institutions, and a small video inset of a man in a white shirt and tie speaking.

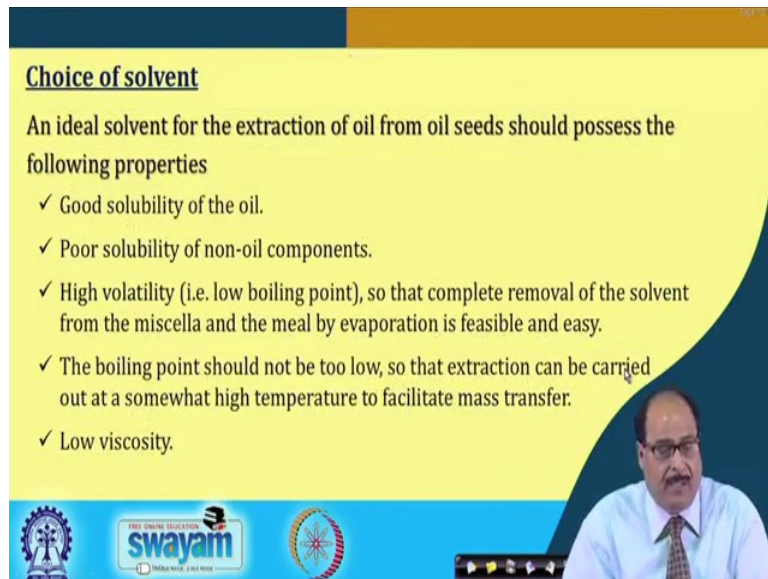
Factors affecting solvent extraction

- Physical characteristics of solids
 - ✓ An open, porous structure of the solid material is preferable, because such a structure facilitates diffusion of the solvent into the bed solid bed, and it also results in better percolation and ultimately the better yield.
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Choice of solvent

An ideal solvent for the extraction of oil from oil seeds should possess the following properties

- ✓ Good solubility of the oil.
- ✓ Poor solubility of non-oil components.
- ✓ High volatility (i.e. low boiling point), so that complete removal of the solvent from the miscella and the meal by evaporation is feasible and easy.
- ✓ The boiling point should not be too low, so that extraction can be carried out at a somewhat high temperature to facilitate mass transfer.
- ✓ Low viscosity.




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An ideal solvent for the extraction of oil from oil seeds should possess the following properties

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- ✓ Poor solubility of non-oil components. As far as possible only oil should be extracted other materials should not dissolve in the solvent
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- ✓ Low viscosity.

Choice of solvent ...contd.

- ✓ Low latent heat of evaporation, so that less energy is needed for solvent recovery.
- ✓ Low specific heat, so that less energy is needed for keeping the solvent and the miscella warm.
- ✓ Chemically inert to oil and other components of the seed flakes.
- ✓ Absolute absence of toxicity and carcinogenicity, for the solvent and its residues.
- ✓ Non-inflammable, non-explosive, non-corrosive.
- ✓ Commercial availability in large quantities and low cost.



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Advantages and disadvantages of the solvent extraction process

Advantages

- High oil yield as compared to mechanical extraction.
- Spends lower production cost and requires less time.

Disadvantages

- The expeller pressed oil is cleaner more pure oil, higher in natural colors and flavors compared to the solvent extracted oil.
- Requirement of large volume of solvent, pressure requirements, etc.
- High construction cost of extraction and refining workshop.
- High maintenance cost.
- Residual oil content in meal.

Advantages and disadvantages of the solvent extraction process

Advantages

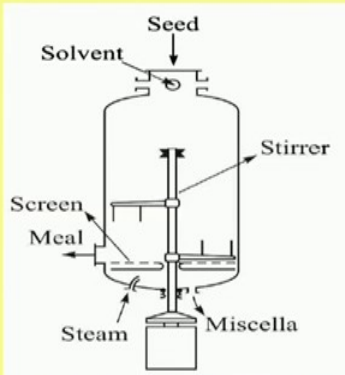
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So, that becomes very important factor if the solvent can be completely removed without any reduction in the quality of the meal. So, the quality meal can be utilized for food purposes.

Types of extractors




Ref: Dutta B. K. (2009). Principles of Mass Transfer and Separation Processes

Solvent extractors are of two types

Batch type

- ✓ In batch processes, a certain quantity of flakes is contacted with a certain volume of fresh solvent.
- ✓ The miscella is drained off, distilled and the solvent is recirculated through the extractor until the residual oil content in the batch of flakes is reduced to the desired level.



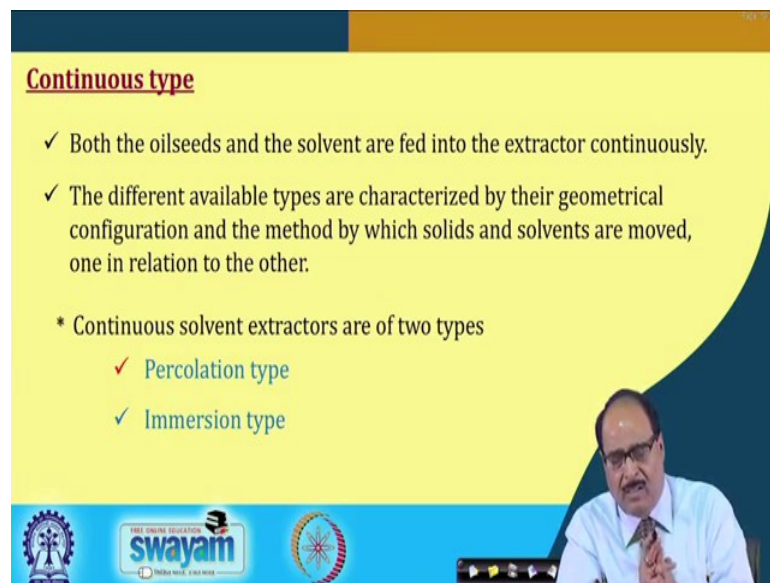
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The porous mass is fed to the solvent extractor. The proper conditions of temperature, pressure etc. are maintained and there is an intimate contact of the prepared mass with the solvent. Solvent flows and penetrates into the mass; the oil dissolves into it and then it comes out along with the solvent.

Solvent extractors are of two types

Batch type

- ✓ In batch processes, a certain quantity of flakes is contacted with a certain volume of fresh solvent.
- ✓ The miscella is finally drained off, distilled and solvent is recirculated through the extractor until the residual oil content in the batch of flakes is reduced to the desired level.



Continuous type

- ✓ Both the oilseeds and the solvent are fed into the extractor continuously.
- ✓ The different available types are characterized by their geometrical configuration and the method by which solids and solvents are moved, one in relation to the other.

* Continuous solvent extractors are of two types

- ✓ Percolation type
- ✓ Immersion type

The slide also features a video inset of a man in a white shirt and glasses speaking, and logos for 'swayam' and 'THE ONLINE EDUCATION' at the bottom.

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However, modern extractors are also available which are of the combination type i.e. both percolation as well as immersion set up or assembly is provided in the same extractor.

Percolation method

- The solvent trickles through a thick bed of flakes without filling the void space completely.
- A film of solvent flows rather rapidly over the surface of the solid particles and efficiently removes the oil which has diffused from the inside to the surface.
- This mode of contact is preferable whenever the resistance to diffusion inside the flake is relatively low (thin flakes with large surface area, open tissue structure).

Immersion method

- The solid particles are totally immersed in a slowly moving, continuous phase of solvent.
- Immersion works better with materials offering a greater internal resistance to oil transfer (thick particles, dense tissue structure).

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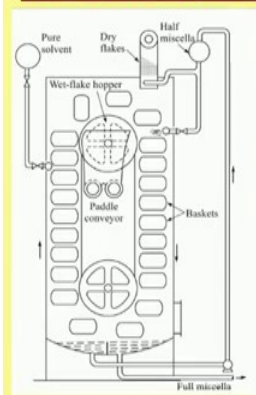
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The vertical basket or the Bollman extractor



The diagram illustrates the vertical basket extractor. It shows a central vertical shaft with a series of baskets attached to a paddle conveyor. The process starts with 'Pure solvent' being sprayed on the top bucket in the ascending leg. 'Dry flakes' are fed from a 'Wet-flake hopper' into the basket at the top of the descending leg. 'Half miscella' is sprayed on the solids in the descending leg. At the bottom, 'Full miscella' is collected in two chambers separated by a partition plate. A 'Paddle conveyor' is shown at the bottom of the shaft.

- The extractor consists of baskets fixed to an endless chain having descending and ascending leg enclosed in vapor tight chamber.
- Each basket has wire-mesh bottom.
- Two chambers at bottom of vessel separated by partition plate hold the extract stream.
- Liquids percolating along the two leg flows down to these chambers.
- The solids fed from a vapor tight hopper into the basket at the top of descending leg and a partially enriched solvent (half miscella) is sprayed on the solids.
- Fresh solvent is sprayed on the top bucket in the ascending leg; percolates and collects in the bottom chamber as half miscella.

Ref: Dutta B. K. (2009). Principles of Mass Transfer and Separation Processes

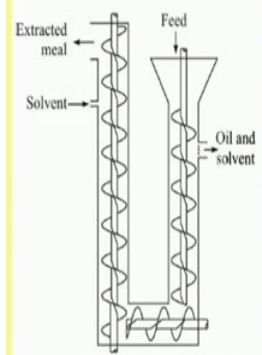
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So, this is how this vertical basket type extractor works.

Hildebrand extractor (Immersion type)



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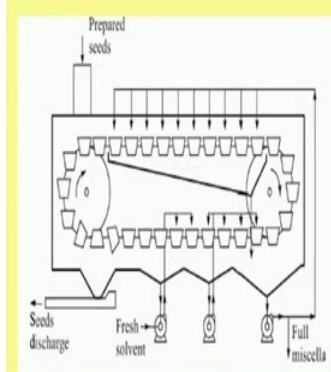
- This is vertical, U shaped total immersion extractor consist of screw conveyors with three screws – one each in vertical legs and one in horizontal section of barrel.
- The helical profile of the screw are perforated to allow flow of the solvent.
- The solid is fed at the top of the one leg and solvent is fed at the top of another leg and flows counter-current to solids through perforation.
- The rich miscella is withdrawn through a strainer for further treatment and solvent separation.



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Horizontal basket extractor



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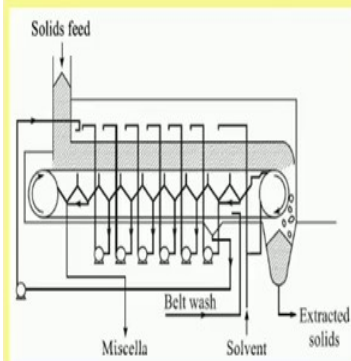
- It consists of a train of horizontal baskets fitted to a drive chain enclosed in a big chamber.
- Each basket has a wire mesh or the perforated bottom.
- There are arrangement for feeding the solids and spraying the solvent on the baskets.
- As the loaded basket advances, it is sprayed with full miscella from bottom compartment
- Before making complete turn, the nearly exhausted solids sprayed with fresh solvent.
- The full miscella is taken out for further processing (Solvent separation and oil recovery).



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Continuous belt extractor




The diagram illustrates a continuous belt extractor. It shows a horizontal endless moving belt with a perforated bottom. Solids are fed at one end, and fresh solvent is fed at the opposite end. The liquid drains into a catch basin and is pumped to the top of the adjacent section of the bed. The belt is washed with solvent, and the extracted solids are collected. The diagram is labeled with 'Solids feed', 'Miscella', 'Solvent', and 'Extracted solids'.

- The solids are fed at one end of the perforated endless moving belt.
- Fresh solvent is fed at the solid discharge end.
- The liquid drains into a catch basin and is pumped to the top of the adjacent section of the bed.
- The throughput rate of the extractor can be adjusted by varying the speed of the belt.
- The height of bed can be adjusted according to expected rate of to the extraction.
- Difficult percolation is compensated by the bed height.

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Rotocel extractor (immersion type)

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- It consists of a number of cells (usually 15 to 18) fixed to a central rotor. Each cell has perforated or screen bottom for percolation of solvent.
- The solids enters at one end of the cell and the extracting liquid is sprayed on the top of the cell.
- Below the rotating cell is the circular tank divided into number of compartment to receive the miscella.
- The interstage miscella is pumped and sprayed on the next cell in the countercurrent direction.

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Miscella distillation

- Full miscella contains typically 30% oil. Thus, the excess solvent must be removed by distillation. Most manufacturers of solvent extractors also offer miscella distillation systems.
- The characteristics of a good miscella distillation system are
 - ✓ Good energy economy,
 - ✓ Minimal heat damage to the crude oil and its components,
 - ✓ Minimal solvent losses ,
 - ✓ Efficient removal of the last traces of solvent from the oil, and
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- The modes of solvent vaporization include flash evaporation, vacuum distillation and steam stripping.



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Miscella filtration

- ✓ To ensure the least possible fluid transport defects (clogging in pumps, pipes etc.) and heat transfer resistances, the miscella must be free of solid meal particles with special closed filter presses before proceeding to distillation.

The slide features a yellow background with a dark blue curved shape on the right. At the bottom, there is a blue banner with logos for 'swayam' and 'INDIA'S BEST EDUCATION'.

Apart from distillation, another important operation is the miscella filtration and it is done to ensure the last possible fluid transport defects, like clogging of the pumps, pipes etc. and heat transfer resistances. The miscella must be free of solid meal particles with special closed filter presses before proceeding to distillation.

Meal desolventizing

- ✓ The spent flakes carry with them about 35% solvent.
- ✓ The removal and recovery of this portion of the solvent is also one of the most critical operations in oil mill practice, since it determines, to a large extent, the quality of the meal and its derivatives.

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The spent flakes contain around 30 to 35 % solvent. So, it becomes very important operation that these solvents should be recovered from the meal which can be recycled and reused. The meal can be used for different food purposes. So, removal and recovery of the solvent is one of the most critical operation in oil mill.

And, a good meal desolventizing step will ensure a better recovery of the oil because the meal should have no oil residue, and at the same time the heat treatment given for the

meal desolventizing should not adversely affect the protein quality. It should not result into the excessive protein denaturation, it should not disturb its PDI or NSI values.

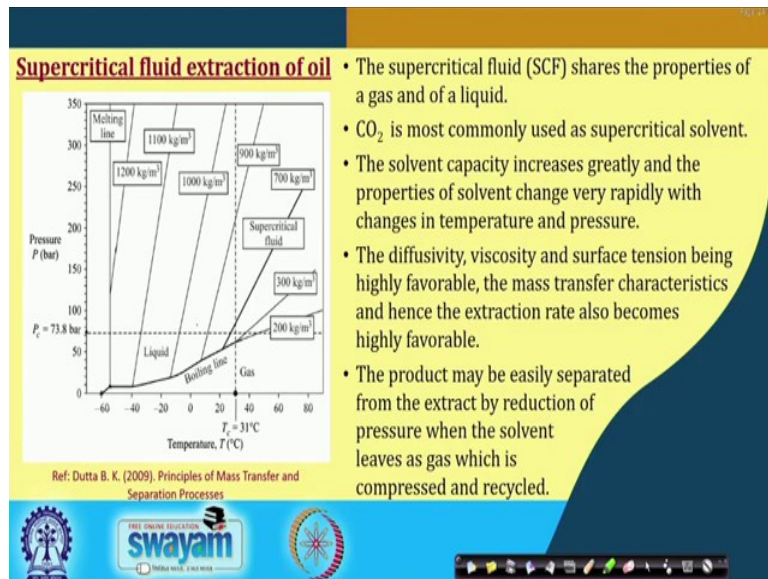
Desolventizer

*DTDC-Desolventizer-Toaster-Dryer-Cooler.
<http://www.crowniron.com/wpcontent/uploads/2018/01/DTDC.pdf>

- ✓ Desolventizer is the newest innovation in removing the solvent (hexane) from the flakes and completing the toasting operation.
- ✓ It consists of a vertical cylindrical stack of compartments or trays.
- ✓ Each compartment is fitted with stirrers or racks attached to a central vertical shaft. Spent flakes are fed at the top of the desolventizer-toaster.
- ✓ The desired amount of heat is supplied to remove the bulk of the solvent from the meal.
- ✓ The pan floors are equipped with adjustable-speed rotating valve, to permit downward movement of the material, through the pans, at the desirable rate.

Desolventizer

- ✓ Desolventizer is the newest innovation in removing the solvent (hexane) from the flakes and completing the toasting operation. And then finally, towards the last stages of the desolventizer i.e. direct heat is given or super-heated stream is there, and the solvent is removed at the same time the meal is also toasted.
- ✓ It consists of a vertical cylindrical stack of compartments or trays.
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- The supercritical fluid (SCF) shares the properties of a gas and of a liquid.
- CO₂ is most commonly used as supercritical solvent.
- The solvent capacity increases greatly and the properties of solvent change very rapidly with changes in temperature and pressure.
- The diffusivity, viscosity and surface tension being highly favorable, the mass transfer characteristics and hence the extraction rate also becomes highly favorable.
- The product may be easily separated from the extract by reduction of pressure when the solvent leaves as gas which is compressed and recycled.

Supercritical fluid extraction of oil

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So, whatever little problems are there in the solvent extraction process relating to the quality of the oil and meal or the residual oil/solvent content in the meal etc. can be eliminated.

The supercritical fluid extraction can result into the lesser oil losses and better recovery of the oil with good quality. Therefore, it results in lower refining and other cost. So, this becomes a very good economically viable method.

Supercritical fluid extraction of oil:

The diagram illustrates the supercritical fluid extraction process. It starts with a 'Feed' entering an 'Extraction vessel'. The output from the vessel passes through a 'Cooler' and then to a 'Pressure reduction, Precipitation of the solute' chamber. In this chamber, the 'Solute' is separated. The remaining 'CO₂' gas is then sent to a 'CO₂ compressor'. The compressed CO₂ then passes through a 'Heat exchanger' and is recycled back into the 'Extraction vessel'.

- The supercritical fluid (SCF) unit consists of three units
 - ✓ Extractor
 - ✓ Solvent separator
 - ✓ Recycle pump
- The extraction in packed bed is common.
- The solute laden SCF from the extractor is cooled and goes to precipitation chamber where the pressure is reduced to separate out the solute.

- Batch extraction is more common since continuous feeding of solid and removal of the exhausted material is difficult.
- The gaseous CO₂ is compressed, cooled and recycled through heat exchanger in order to heat it to required temperature.

Dr. B. K. Dutta, IIT Bombay

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- The gaseous carbon dioxide is compressed, cooled and recycled through heat exchanger in order to heat it to required temperature.
- The gaseous CO₂ is compressed, cooled and recycled through heat exchanger in order to heat it to required temperature.

7/31/2016

Kinetics of soybean oil extraction using ethanol

- The fastest transfer of soluble components to the solvent at the beginning of the process, followed by a decrease in this transfer rate until it became constant, corresponding to the maximum yield of extraction.
- The values of soybean oil diffusivity coefficients increased with increasing temperature in both solvents and decreased with increases in the water level in the solvent for the same extraction temperature.
- The kinetics of extraction of the free fatty acids is the same as observed for the oil.
- The value of the diffusivity coefficient for soybean oil increased with increasing temperature and with decreasing hydration level in the solvent.

Fig. 1 - Kinetics of extraction of soybean oil. Experimental data with absolute ethanol: □, 40.0 ± 0.1 °C; △, 50.0 ± 0.1 °C; ○, 60.0 ± 0.1 °C. Experimental data with ethanol with 5.98 mass% of water: ■, 40.0 ± 0.1 °C; ▲, 50.0 ± 0.1 °C; ●, 60.0 ± 0.1 °C. Calculated: —, model of So and Macdonald (1985); - - -, model of Perez et al. (2011).

Fig. 2 - Kinetics of extraction of free fatty acids. Experimental data with absolute ethanol: □, 40.0 ± 0.1 °C; △, 50.0 ± 0.1 °C; ○, 60.0 ± 0.1 °C. Experimental data with ethanol with 5.98 mass% of water: ■, 40.0 ± 0.1 °C; ▲, 50.0 ± 0.1 °C; ●, 60.0 ± 0.1 °C. Calculated: —, model of So and Macdonald (1985); - - -, model of Perez et al. (2011).

Source: Toda et al. (2016)

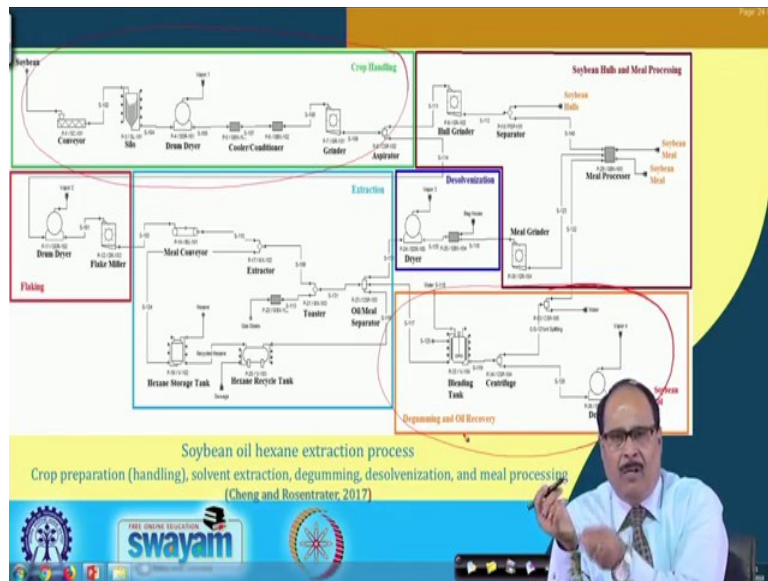
Expanded soybean mass (collets)
 Temperature - 40, 50, and 60 °C
 Ethanol (hydration level) - 0 and 5.98% mass of water

Kinetics of soybean oil extraction using ethanol

The kinetics of soybean oil extraction using ethanol has been explained by few researchers. Expanded soybean mass (colletes) temperatures are 40, 50 and 60 °C. The ethanol system (hydration level) was used at 0 and 5.98 % mass of water.

The results exhibiting the kinetics of the extraction of free fatty acids or kinetics of the extraction of whole soybean oil can be seen in Figure. It increases initially at a faster rate up to a certain level with increase in both the soybean oil as well as free fatty acids, then it becomes little bit slow. And then finally, it gets stabilized or becomes constant depending upon the material conditions, process conditions, temperature, pressures and so on.

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This figure commercial plant extraction facility is shown starting from the handling of the material (whole seed) until oil and meal are obtained. It is basically for soybean oil extraction and meal processing, but can be used for any other oilseeds or nuts.

In fact, in most of the solvent extraction plants, depending upon the process, along with oils certain phospholipids, gums etc. also get extracted. So, they are separated at the extraction plant itself using water degumming. So, most of the extraction plant are equipped with these facilities for removal of gums from the oil through water degumming process. The oil is then sent to refining plant where it is subjected to various other treatments like, acid degumming, chemical/physical refining, bleaching, deodorization, etc.