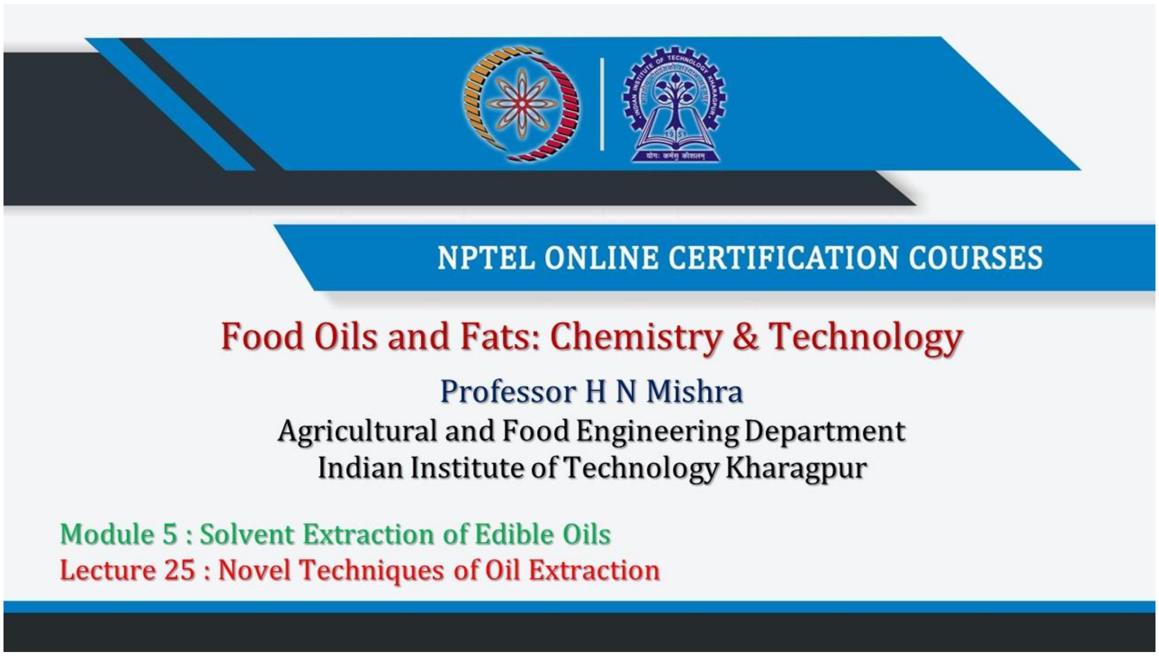
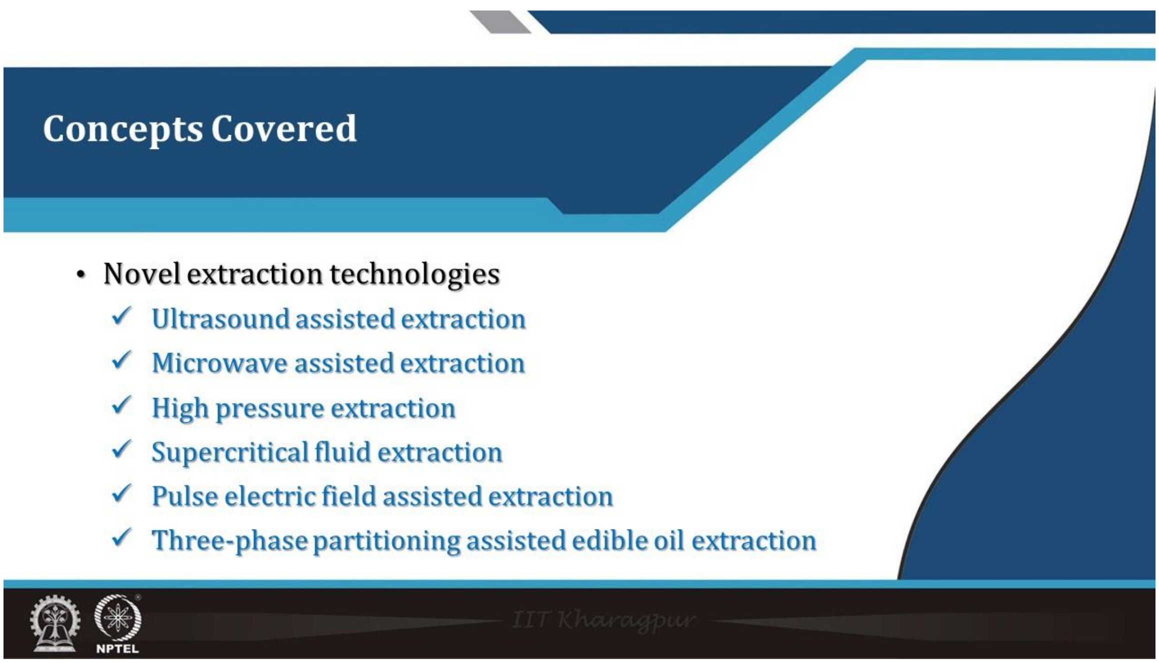
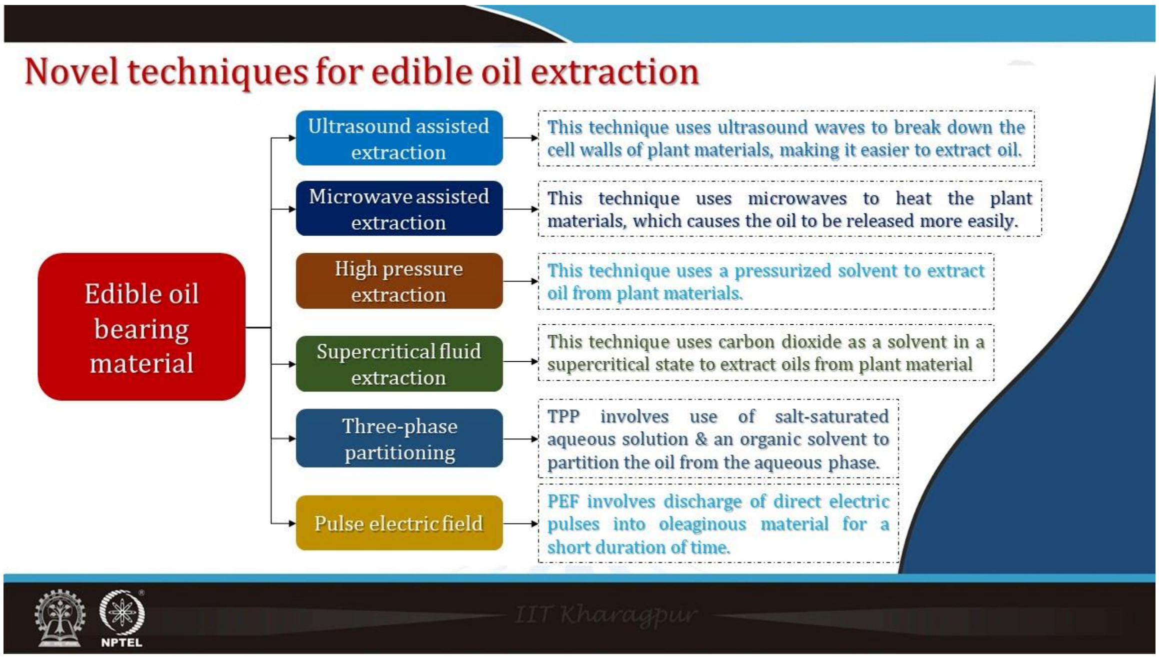
Food Oils and Fats: Chemistry & Technology
Professor H N Mishra
Agricultural and Food Engineering Department
Indian Institute of Technology Kharagpur
Module 5 : Solvent Extraction of Edible Oils
Lecture 25 : Novel Techniques of Oil Extraction

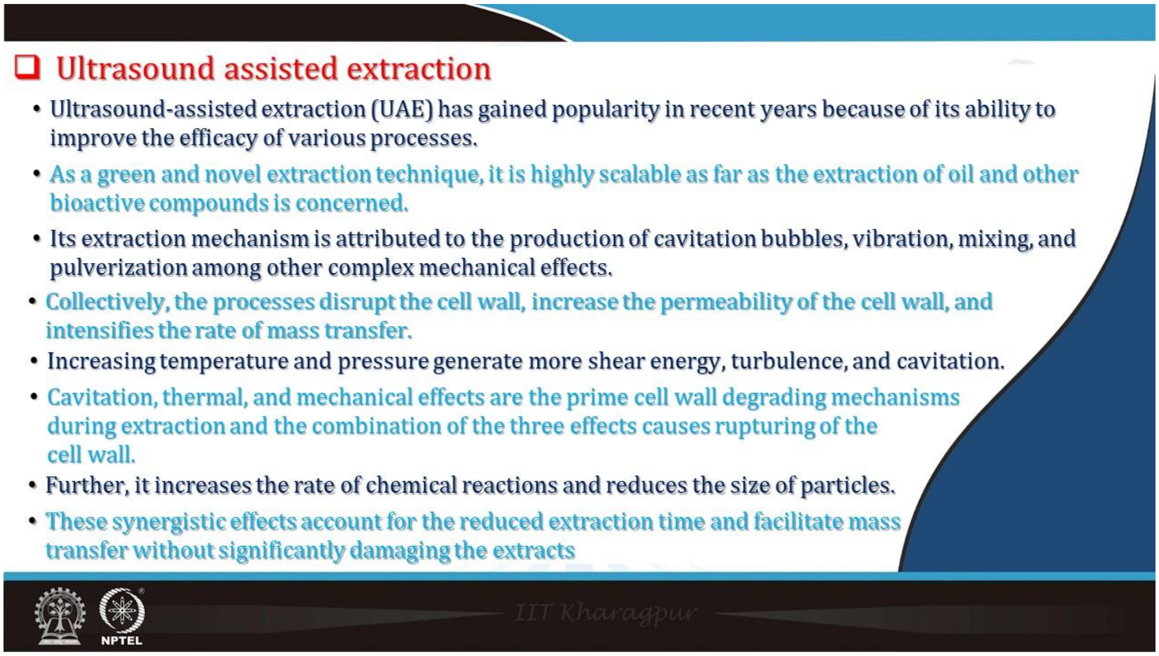




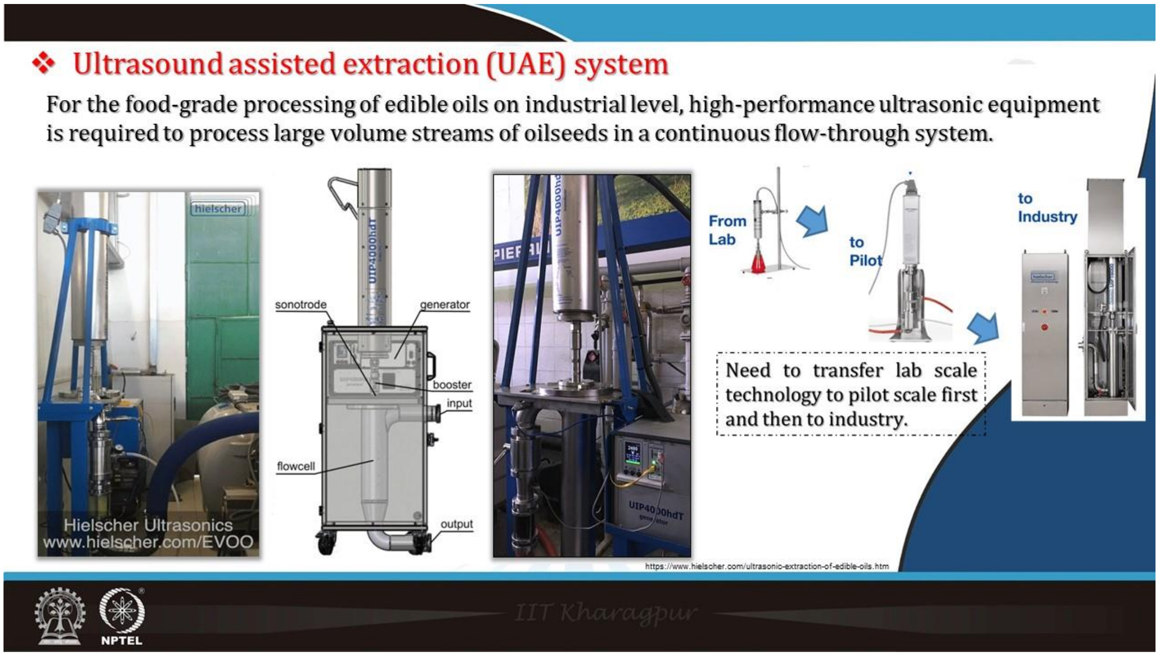
Hello everybody. Namaskar. Now, in this lecture 25, we will discuss Novel Techniques of Oil Extraction. The traditional technologies that are used by the industry have certain problems that is sometimes if the process parameters etcetera are not properly controlled, they may lead to degradation in the quality and also they are more energy-intensive and costly processes. The setup which is for the traditional oil extraction units etcetera, requires more capital. They are capital and energy-intensive processes. So, there is a need for modernization of these units and with this, I will just discuss some of the novel and emerging technologies that can be used for extraction of the oil from oil-bearing materials. These technologies include ultrasound-assisted extraction, microwave-assisted extraction, high-pressure extraction, supercritical fluid extraction, pulse electric field-assisted extraction, and three-phase partitioning-assisted edible oil extraction.



In ultrasound-assisted extraction, we use ultrasound waves to break down the cell wall of the plant materials, therefore, making it easier to extract the oil. In the case of microwave-assisted extraction, the microwaves are used to heat the plant materials which causes the oil to be released more easily. High-pressure extraction techniques use a pressurized solvent to extract the oil from the plant materials. The supercritical fluid extraction process uses carbon dioxide as a solvent in a supercritical state to extract the oil from the plant materials. The three-phase partitioning method involves the use of a salt-saturated aqueous solution and an organic solvent to partition the oil from the aqueous phase. And finally, the pulse electric field involves the discharge of direct electric pulses into oleaginous materials for a short duration of time. All these processes result in efficiency and a process that gives both oil and meal of good quality. Good quality because most of these techniques involve non-thermal processes or alternate thermal processes and here the oil yield also is higher as compared to the traditional processes. So, let us see briefly one by one.



Ultrasound-assisted extraction (UAE) has gained popularity in recent years because of its ability to improve the efficacy of various processes. As a green and novel extraction technique, it is highly scalable as far as the extraction of oil and other bioactive compounds is concerned. Its extraction mechanism is attributed to the production of cavitation bubbles, vibration, mixing, and pulverization among other complex mechanical effects. Collectively, the processes disrupt the cell wall, increase the permeability of the cell wall, and intensify the rate of mass transfer. Increasing temperature and pressure generate more shear energy, turbulence, and cavitation. Cavitation, thermal, and mechanical effects are the prime cell wall degrading mechanisms during extraction and the combination of the three effects causes rupturing of the cell wall. Further, it increases the rate of chemical reactions and reduces the size of particles. These synergistic effects account for the reduced extraction time and facilitate mass transfer without significantly damaging the extracts



So, it is you can see here the ultrasonic system. For food-grade processing of edible oils on an industrial level performance ultrasonic equipment is required to process large volumes of streams of oilseeds in a continuous flow-through system. So, from laboratory equipment one needs to convert it into a pilot plant and finally, into industry. There is a significant need and various different systems are available. One such system and its operation mechanism you can see how basically the various high frequency ultrasound waves are used to break the cell wall and it increases the flowability of the oil and gives better yield.

❖ Factors affecting UAE

- The type of solvent, its concentration, temperature, time, and frequency of the ultrasound waves determine the effectiveness and efficiency of the extraction process.
- The thermal effect produced by high extraction temperature favours solvent diffusion rates. On the other side, lower temperature enhances cavitation and consequently the yield.
- Higher extraction time favors oil yield, but induces undesirable nutritional & sensory changes in extracted product.

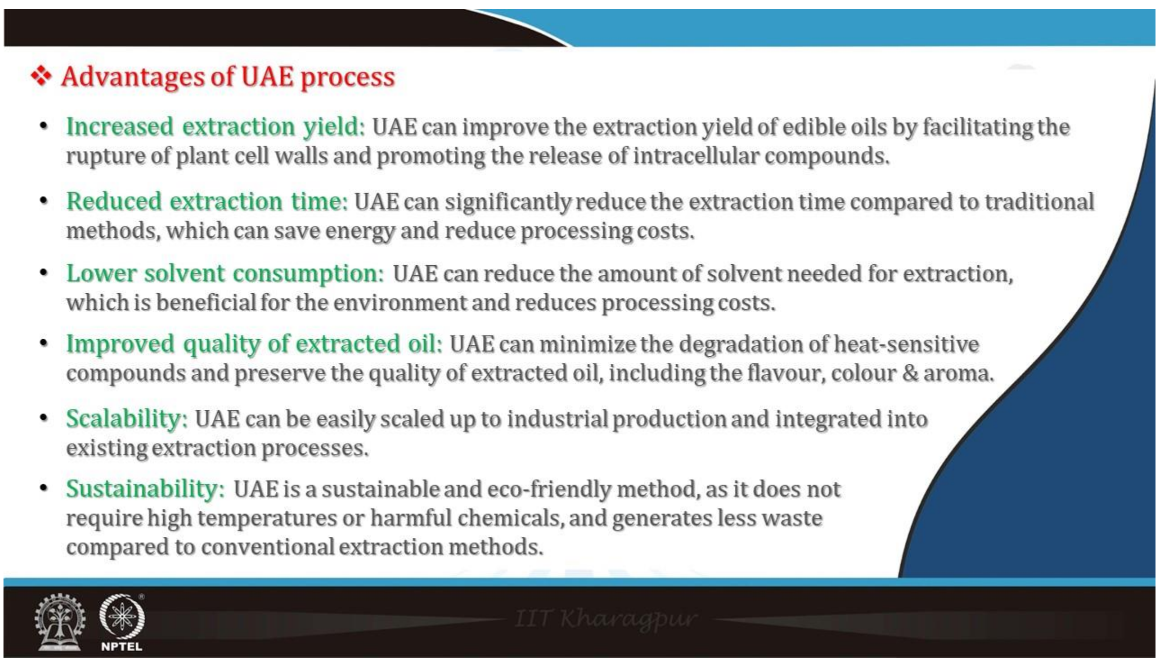
Source	Extraction technique	Experimental conditions	Yield
Pomegranate seeds	Ultrasound-assisted enzyme extraction	Enzymes used: cellulase and Peclyve®; Temp: 55 °C; extraction time: 2 hr	15.80%
Flaxseed	Ultrasound-assisted enzyme extraction	Enzymes used: immobilized cellulase, pectinase, and hemicellulase; ultrasound frequency: 20 kHz; ultrasound power: 250 W; Temp: 45 °C; extraction time: 30 min	62.50%
Perilla seeds	Ultrasound-assisted enzyme extraction	Enzymes used: cellulase, Viscozyme L®, Alcalase 2.4 L®, Protex 6 L®, and Protex 7 L®; ultrasound power: 250 W; Temp: 50 °C; extraction time: 30 min	50.20%
Moringa oleifera	Ultrasound-assisted extraction	Ultrasound power: 200 W; Temp: 30 °C; extraction time: 15 min	91.35%

https://doi.org/10.1111/1541-4337.12507

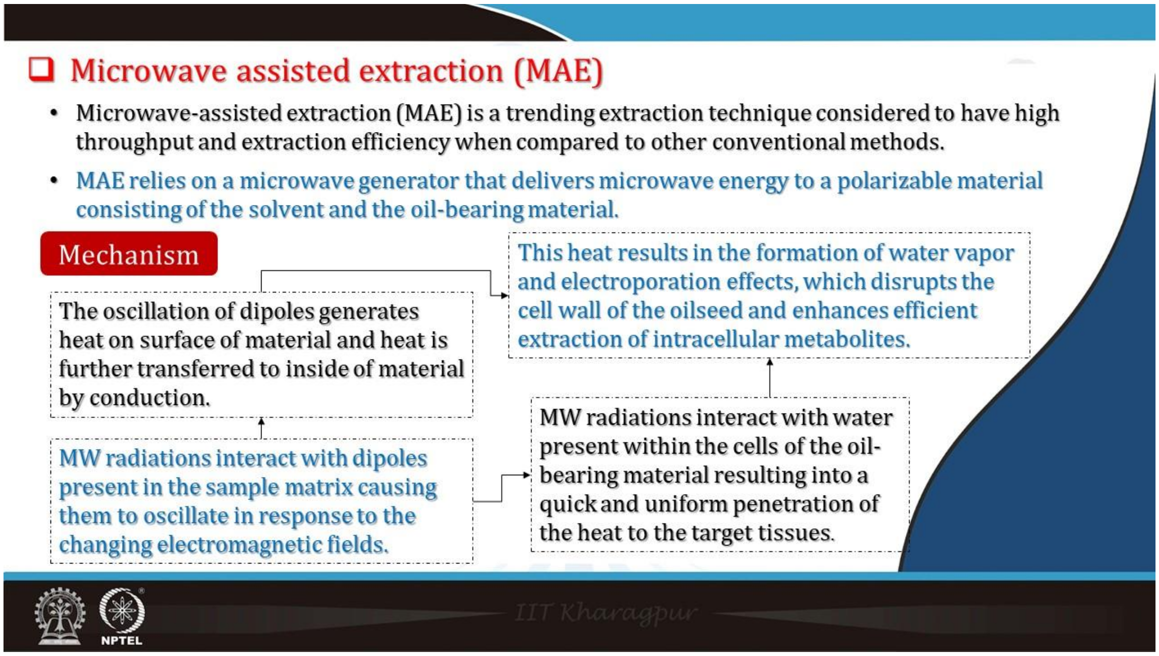
NPTEL

IIT Kharagpur

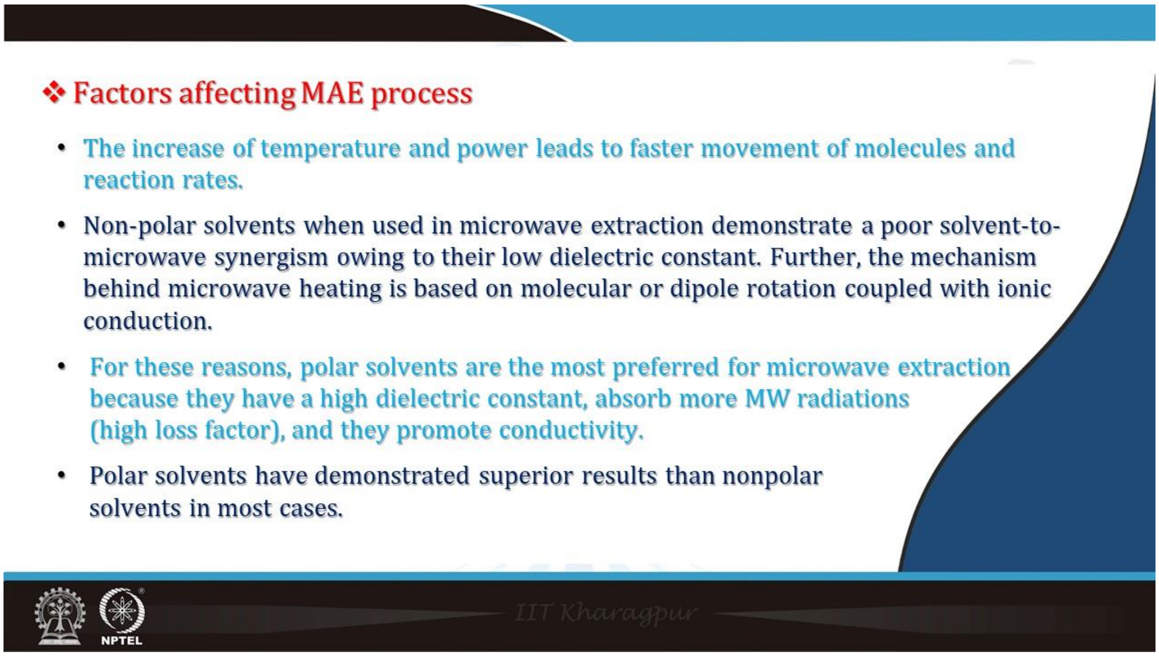
Factors that influence the UEA extraction process are type of the solvents, their concentration, temperature, and time and frequency of the ultrasound waves, etc, that determine the effectiveness and efficiency of the extraction process. The thermal effect produced by high extraction temperature favors solvent diffusion rates. On the other side, low temperature enhances cavitation and consequently, it increases the yield. The higher extraction time favors oil yield but induces undesirable nutritional and sensory changes in the extracted product. So, here in this, I have taken from the literature that the ultrasound-assisted enzyme extraction process and yield when reported from about 15.8 percent in pomegranate seed to as high as 91.35 percent in *moringo oleifera* seeds. In perilla seeds, the yield was reported using ultrasound-assisted extraction of 50 percent and in flax seeds, it is 62 percent. The enzymes used normally are cellulase or immobilized cellulase, pectinase, hemicellulase or acetylase, and other such enzymes and the different conditions used like temperature generally may vary from 45 degrees Celsius to 55 degrees Celsius. The general time of extraction in the case of moringa was 15 minutes, perilla seeds 30 minutes, flax seeds 30 minutes, and in the case of pomegranate seeds 2 hours. So, these are sufficient to show how various factors like enzyme type, temperature, time, and other things influence the yield of oil from various materials.



So, the advantages of the ultrasound-assisted extraction process include increased extraction yield: UAE can improve the extraction yield of edible oils by facilitating the rupture of plant cell walls and promoting the release of intracellular compounds, reduced extraction time: UAE can significantly reduce the extraction time compared to traditional methods, which can save energy and reduce processing costs, lower solvent consumption: UAE can reduce the amount of solvent needed for extraction, which is beneficial for the environment and reduces processing costs, improved quality of extracted oil: UAE can minimize the degradation of heat-sensitive compounds and preserve the quality of extracted oil, including the flavour, colour & aroma, Scalability: UAE can be easily scaled up to industrial production and integrated into existing extraction processes, and Sustainability: UAE is a sustainable and eco-friendly method, as it does not require high temperatures or harmful chemicals, and generates less waste compared to conventional extraction methods.



Then microwave-assisted extraction is another novel technology. It is a trending extraction technique considered to have high throughput and extraction efficiency when compared to other conventional methods of oil extraction. Microwave-assisted extraction relies on a microwave generator that delivers microwave energy to a polarizable material consisting of the solvent and the oil-bearing material. Its mechanism is that this microwave radiation interacts with the dipole present in the sample matrix causing them to oscillate in response to the changing electromagnetic fields. The oscillation of dipoles generates heat on the surface of the material and heat is further transferred to the inside of the material by conduction. Also, this microwave radiation interacts with the water present within the shell of the oil-bearing material resulting in a quick and uniform penetration of the heat to the target tissues, and both these ok. The oscillation or direct interaction that is this heat generated by these results in the formation of water vapor and the electroporation effect which disrupts the cell wall of the oil seed enhances efficient extraction of the intracellular metabolites.



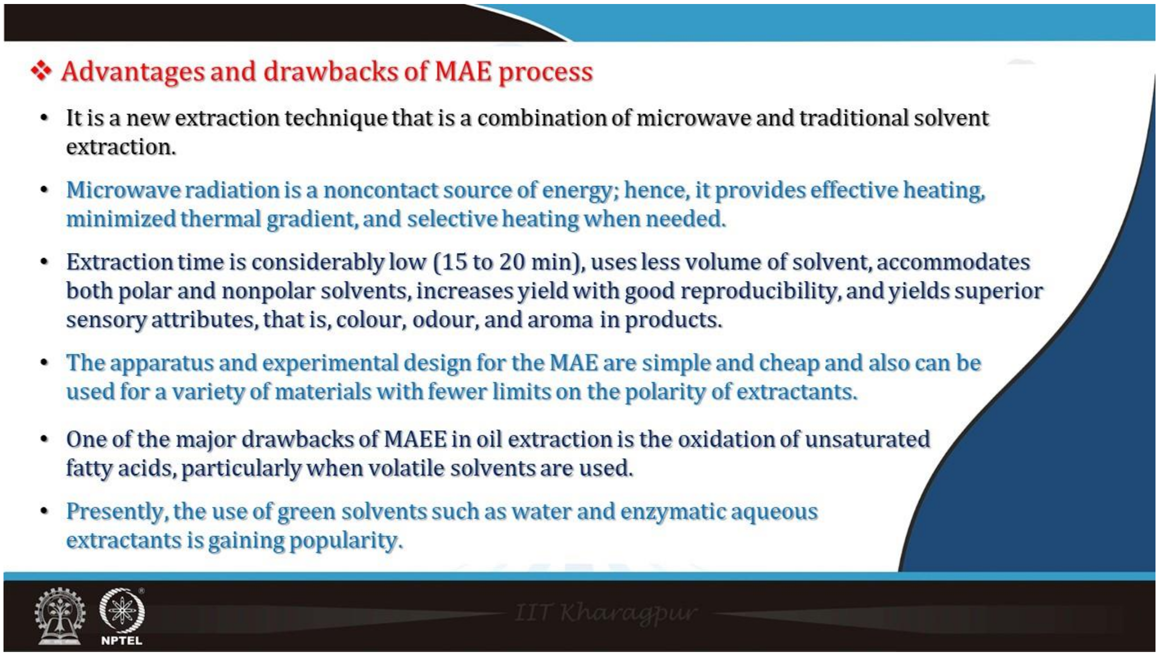
The factors that affect the microwave-assisted extraction processes include an increase in the temperature and power leads to the faster movement of the molecule and reaction rates. Non-polar solvents when used in microwave extraction demonstrate a poor solvent-to-microwave synergism owing to their low dielectric constant. Further, the mechanism behind microwave heating is based on molecular or dipole rotation coupled with ionic conduction. For these reasons, polar solvents are the most preferred for microwave extraction because they have a high dielectric constant, absorb more MW radiations (high loss factor), and they promote conductivity. Polar solvents have demonstrated superior results than nonpolar solvents in most cases.

Oil source	Extraction technique	Experimental conditions	Yield
Pumpkin seeds	Microwave-assisted enzyme extraction	Enzymes used: cellulase, hemicellulase, pectinase, β-glucosidase, and neutral proteinase; microwave power: 419 W; Temp: 44 °C; pH 5; solute/solvent: 1:10; extraction time: 66 min	65%
Yellow horn seed	Microwave-assisted enzyme extraction	Enzymes used: cellulase, hemicellulase, and pectinase; Solid–liquid ratio: 5 mL/g; microwave power: 500 W; Temp: 60 °C; extraction time: 30 min	60.50%
Isatis indigotica seeds	Microwave-assisted enzyme extraction	Enzymes used: 1.82% (1:1:1) cellulose, proteinase, and pectinase; microwave power: 375 W; Temp:43 °C; extraction time: 83 min	59.27%
Sandbox seed	Microwave-assisted extraction	Liquid–solid ratio: 40:1 (ethyl acetate); microwave power: 180 W; extraction time: 5 min	72.75%
Sandbox seed	Microwave-assisted extraction	Liquid–solid ratio: 40:1 (*n*-hexane); microwave power: 180 W; extraction time: 5 min	56.25%

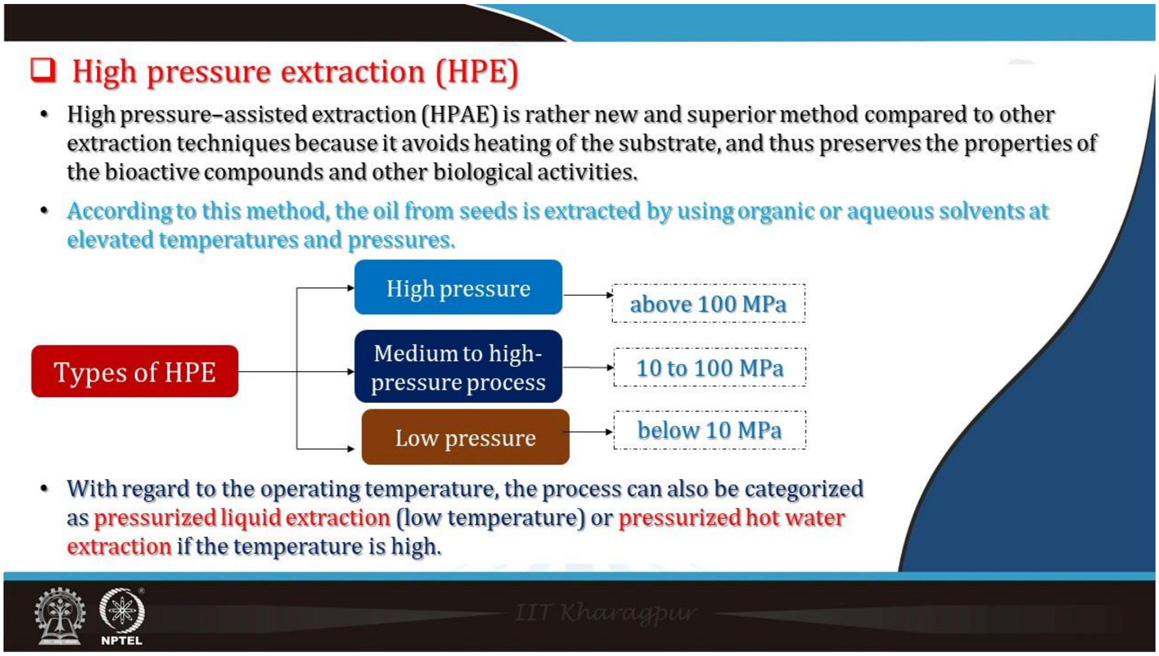
Note. Solvent used in all the studies in this table is hexane unless otherwise stated.

IIT Kharagpur

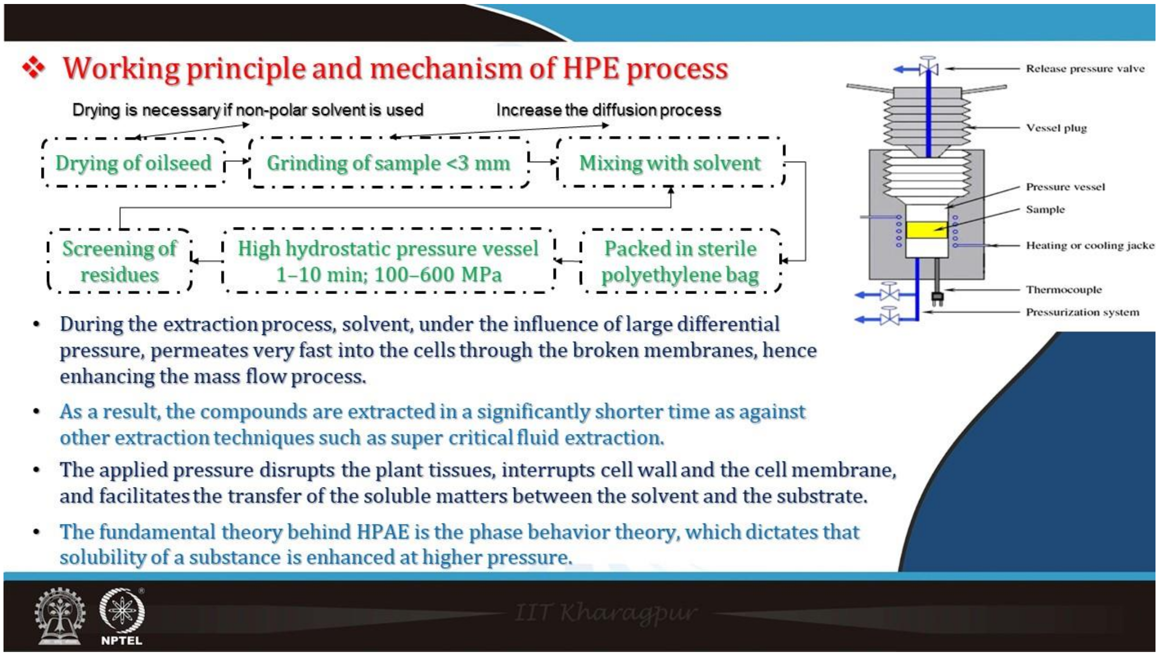
So, again from the literature I have taken this data that this microwave-assisted enzyme extraction technology was used to extract the oil from various seeds like example pumpkin seeds, yellow horn seeds, *Isatis indigotica* seeds, sandbox seeds, and so on. In the case of pumpkin seed 65 percent oil yield was reported, the enzymes used were cellulose, hemicellulose, proteinase, beta-glucosidase, and neutral proteinases. Microwave power used was 419 watts, temperature was 44 degrees Celsius, pH was 5 solute to solvent ratio was 1:10, and extraction time was 66 minutes. So, similar to the sandbox seed you can see that the range when the extraction time was 5 minutes, solid liquid ratio of 40:1, and microwave power was about 180 watts, the extraction was obtained from 56 to even 72 percent in the different varieties. In the yellow horn seed, the extraction yield was around 60 percent.



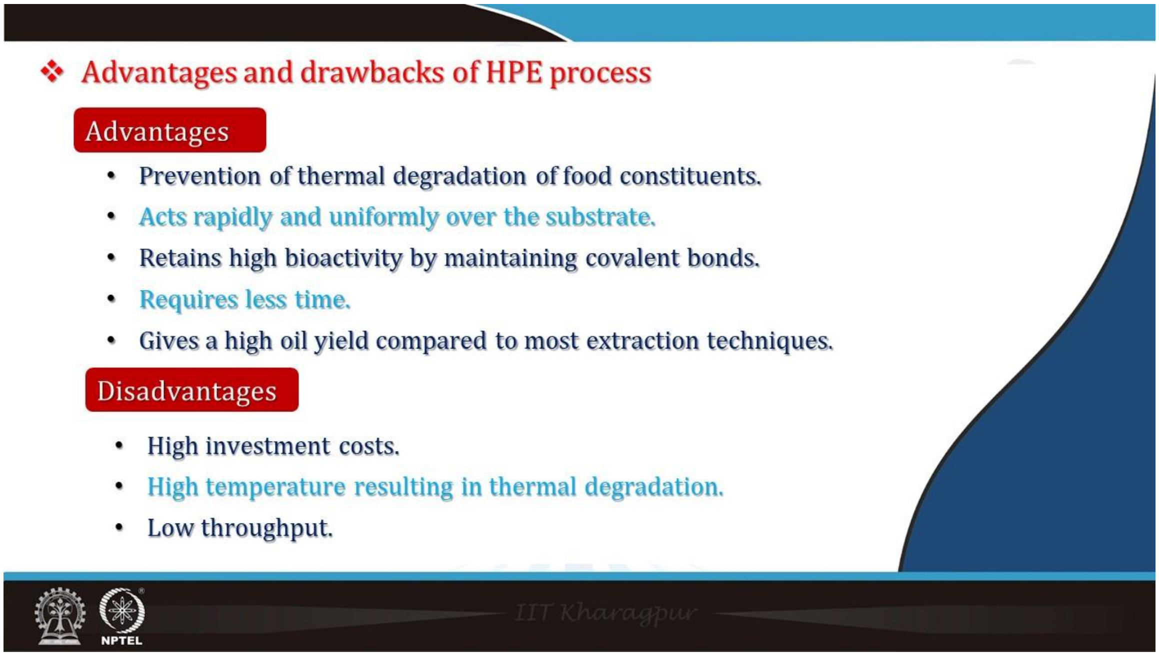
So, the advantages and disadvantages of the microwave-assisted extraction processes include It is a new extraction technique that is a combination of microwave and traditional solvent extraction. Microwave radiation is a noncontact source of energy; hence, it provides effective heating, minimized thermal gradient, and selective heating when needed. Extraction time is considerably low (15 to 20 min), uses less volume of solvent, accommodates both polar and nonpolar solvents, increases yield with good reproducibility, and yields superior sensory attributes, that is, colour, odour, and aroma in products. The apparatus and experimental design for the MAE are simple and cheap and also can be used for a variety of materials with fewer limits on the polarity of extractants. One of the major drawbacks of MAEE in oil extraction is the oxidation of unsaturated fatty acids, particularly when volatile solvents are used. Presently, the use of green solvents such as water and enzymatic aqueous extractants is gaining popularity.



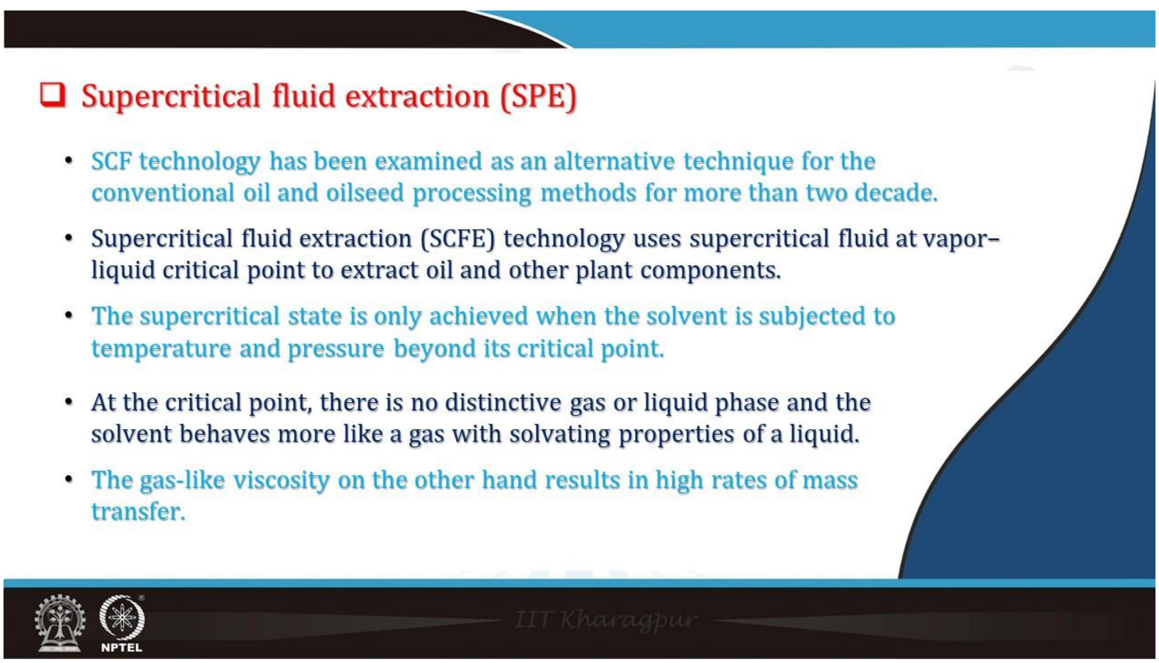
Then we come to the high-pressure assisted extraction. This HPAE is a rather new and superior method compared to the other extraction techniques because it avoids heating the substrate and thus preserves the properties of the bioactive compounds and the other biological activities. According to this method, the oil from seeds is extracted by using organic or aqueous solvents at elevated temperatures and pressures. Different types of HPE depending upon the pressure used can be categorized as high-pressure, medium to high-pressure process, or low-pressure process. If the pressure is above 100 mega Pascal, this is a high-pressure process. If the pressure is below 10 mega Pascal, it is a low-pressure process and between 10 and 100 mega Pascal, it is medium to high. With regard to the operating temperature, the process can also be categorized as pressurized liquid extraction which is low temperature, or pressurized hot water extraction if the temperature is high.



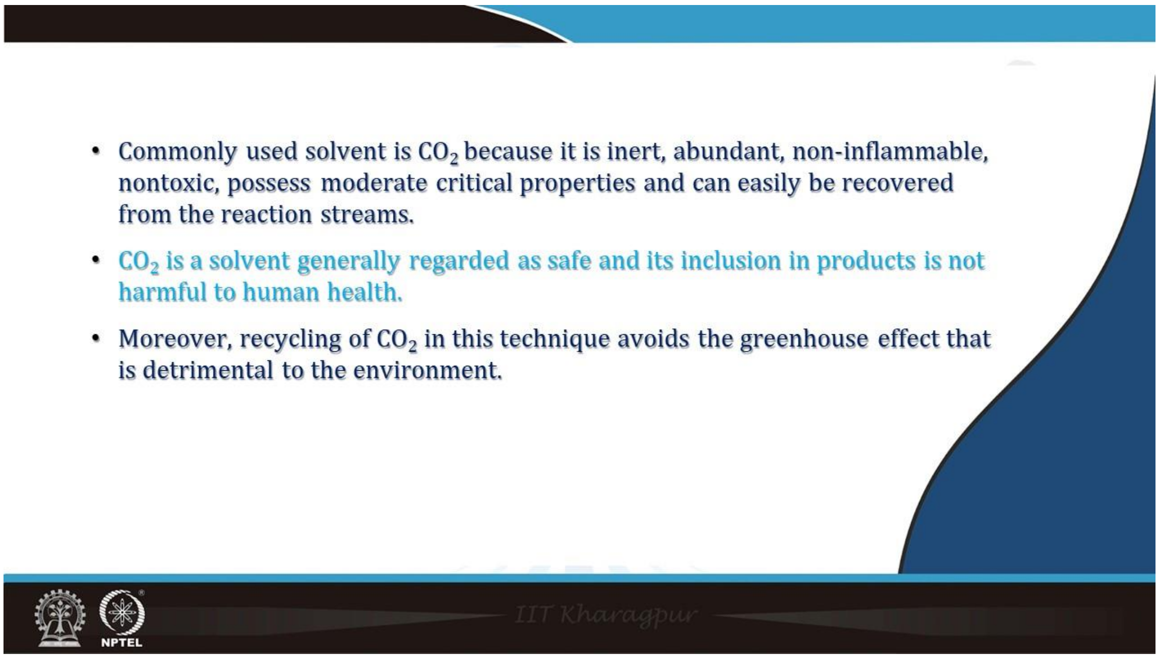
So, the working principle of and mechanism of high-pressure extraction you can see here. This is a high-pressure generating system here. It is a chamber where the material is kept. Drying of oilseed is necessary if the nonpolar solvent is being used. Then grinding of the sample to less than 3 mm just it is done to increase the diffusion process and mixed with solvent in a sterile polythene bag then this material is prepared and kept in a high hydrostatic pressure vessel where the pressure is applied maybe 100 to 600 mega Pascal for about 1 to 10 minutes and then screening of the residues and then this is further mixed with solvent and the process continue. During the extraction process, solvent, under the influence of large differential pressure, permeates very fast into the cells through the broken membranes, hence enhancing the mass flow process. As a result, the compounds are extracted in a significantly shorter time as against other extraction techniques such as supercritical fluid extraction. The applied pressure disrupts the plant tissues, interrupts the cell wall and the cell membrane, and facilitates the transfer of the soluble matter between the solvent and the substrate. The fundamental theory behind HPAE is the phase behavior theory, which dictates that the solubility of a substance is enhanced at higher pressure.



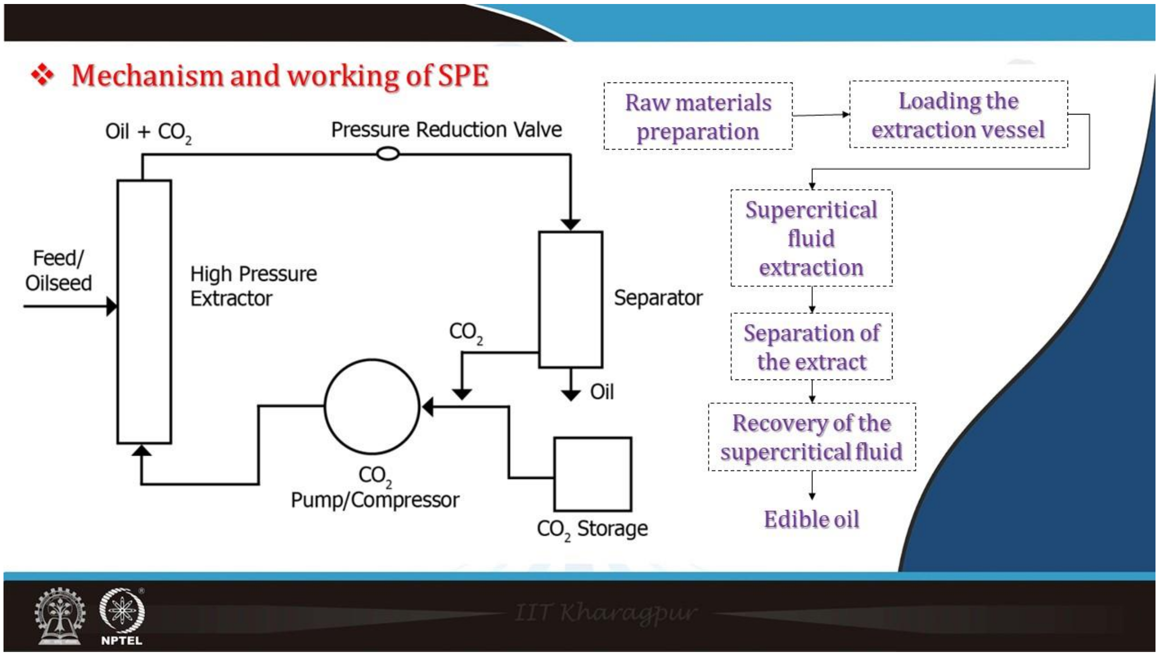
The high-pressure extraction process has the advantage of preventing the thermal degradation of the food constituents. It acts rapidly and uniformly over the substrate. It retains high bioactivity and maintains the covalent bonds, requires less time, and gives a high oil yield compared to most of the traditional extraction technologies or even some of the novel technologies. However, the disadvantage of this process is high investment cost, high temperature resulting in thermal degradation, and low throughput because when you are increasing the pressure of course, sometimes this effect of the temperature can be brought down by having a proper suitable arrangement in the setup.



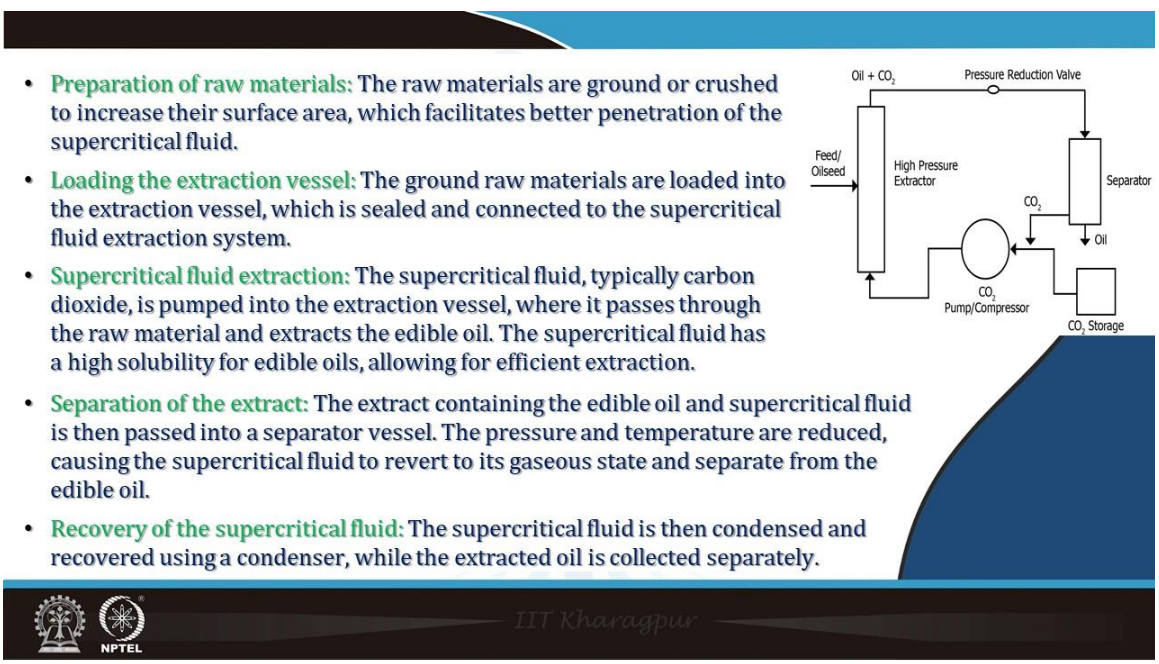
Then we come to the supercritical fluid extraction technology. SCF technology has been examined as an alternative technique for conventional oil and oilseed processing methods for more than two decades. Supercritical fluid extraction (SCFE) technology uses supercritical fluid at vapor–liquid critical points to extract oil and other plant components. The supercritical state is only achieved when the solvent is subjected to temperature and pressure beyond its critical point. At the critical point, there is no distinctive gas or liquid phase and the solvent behaves more like a gas with solvating properties of a liquid. The gas-like viscosity on the other hand results in high rates of mass transfer.



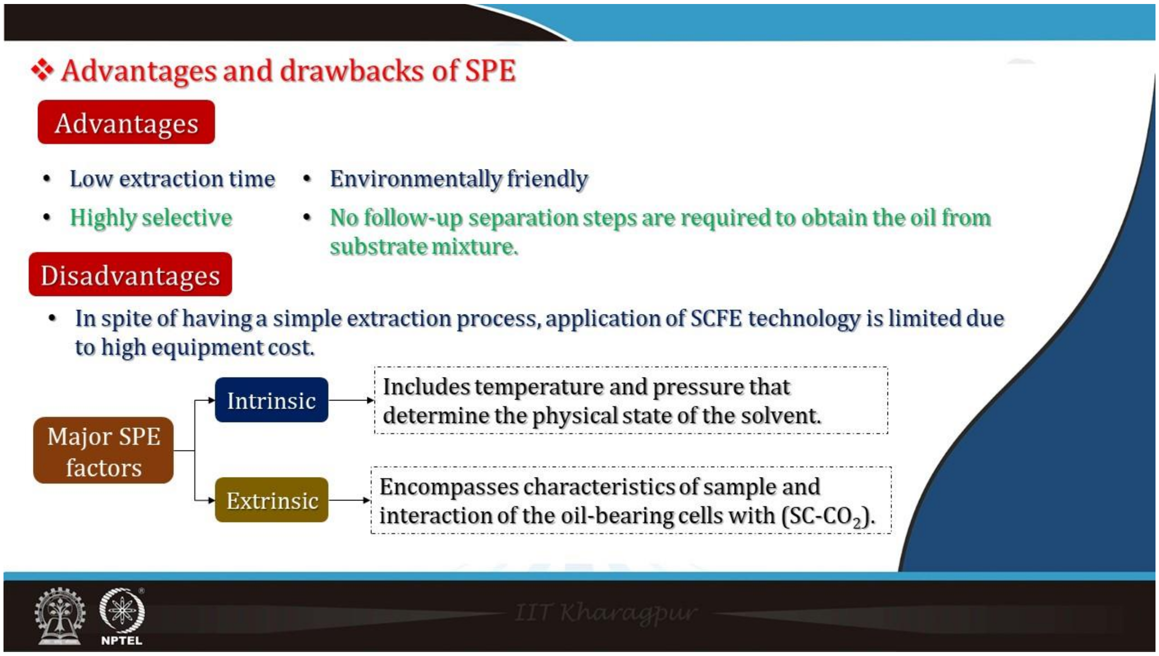
A commonly used solvent is CO_2 because it is inert, abundant, non-inflammable, nontoxic, possesses moderate critical properties, and can easily be recovered from the reaction streams. CO_2 is a solvent generally regarded as safe and its inclusion in products is not harmful to human health. Moreover, recycling CO_2 in this technique avoids the greenhouse effect that is detrimental to the environment.



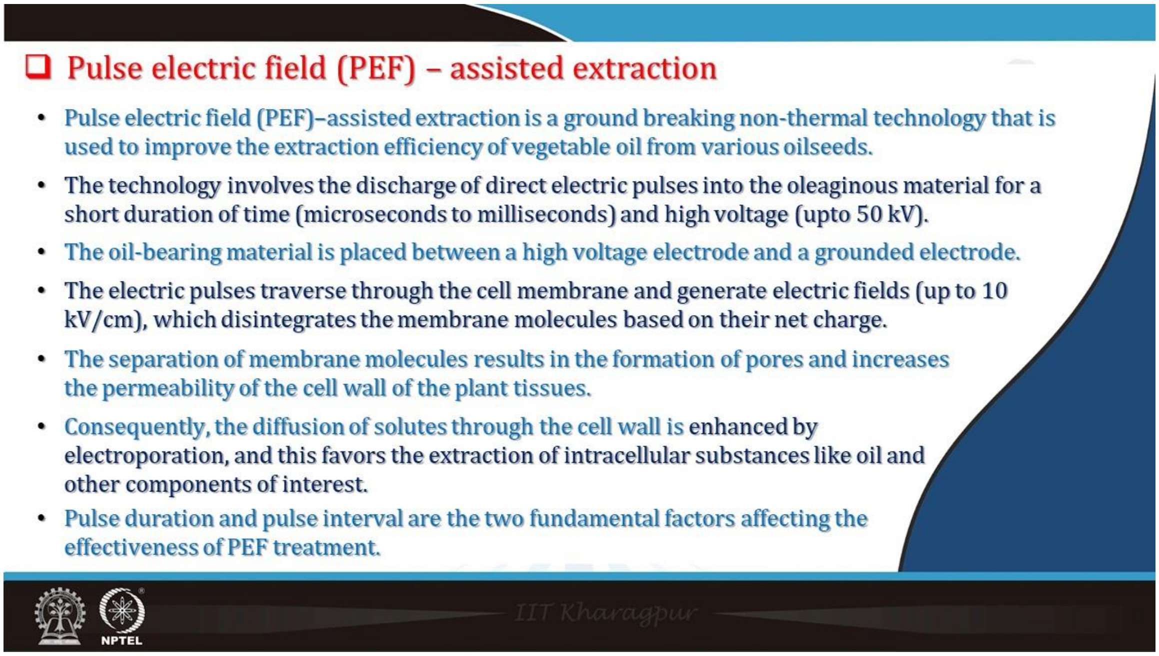
This is a setup that is the mechanism of working the supercritical fluid extraction process. Here is the extractant which is high pressure extractor where the feed or oil seed material is used and in there this carbon dioxide storage or carbon dioxide cylinders are there, circulated with the help of a pump or compressor. The carbon dioxide as a high-pressure supercritical carbon dioxide is passed through this extractor where it comes into contact and dissolves the oil. The oil and carbon dioxide come to the pressure reduction bulb where it is oil and carbon dioxide are separated. The separation of the extract and finally, the carbon supercritical fluid is recovered and you get edible oil.



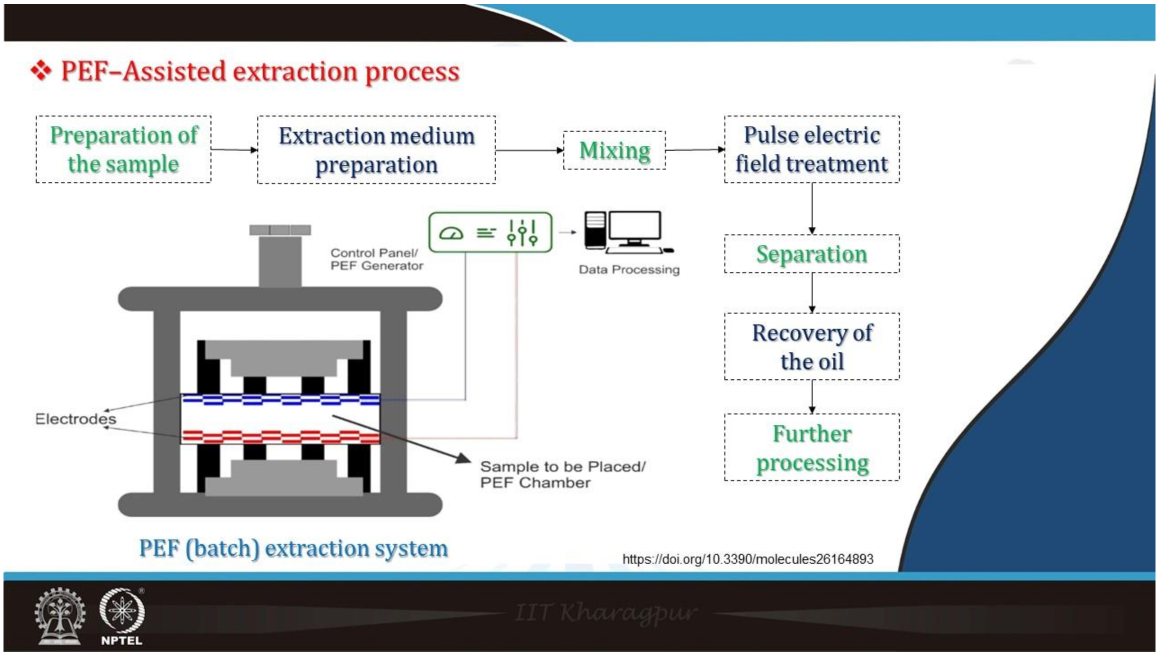
So, that is basically raw material preparation and loading it into the extraction vessel and in the extraction vessel there is intimate contact between the supercritical fluid, and desired conditions are maintained such as the air pressure, temperature, etc. to increase the extractability. In preparation for raw materials, the raw materials are ground or crushed to increase their surface area, which facilitates better penetration of the supercritical fluid. The loading into the extraction vessel. The ground raw materials are loaded into the extraction vessel, which is sealed and connected to the supercritical fluid extraction system. The supercritical fluid, typically carbon dioxide, is pumped into the extraction vessel, where it passes through the raw material and extracts the edible oil. The supercritical fluid has a high solubility for edible oils, allowing for efficient extraction. Separation of the extract is the extract containing the edible oil and supercritical fluid is then passed into a separator vessel. The pressure and temperature are reduced, causing the supercritical fluid to revert to its gaseous state and separate from the edible oil. The supercritical fluid is then condensed and recovered using a condenser, while the extracted oil is collected separately.



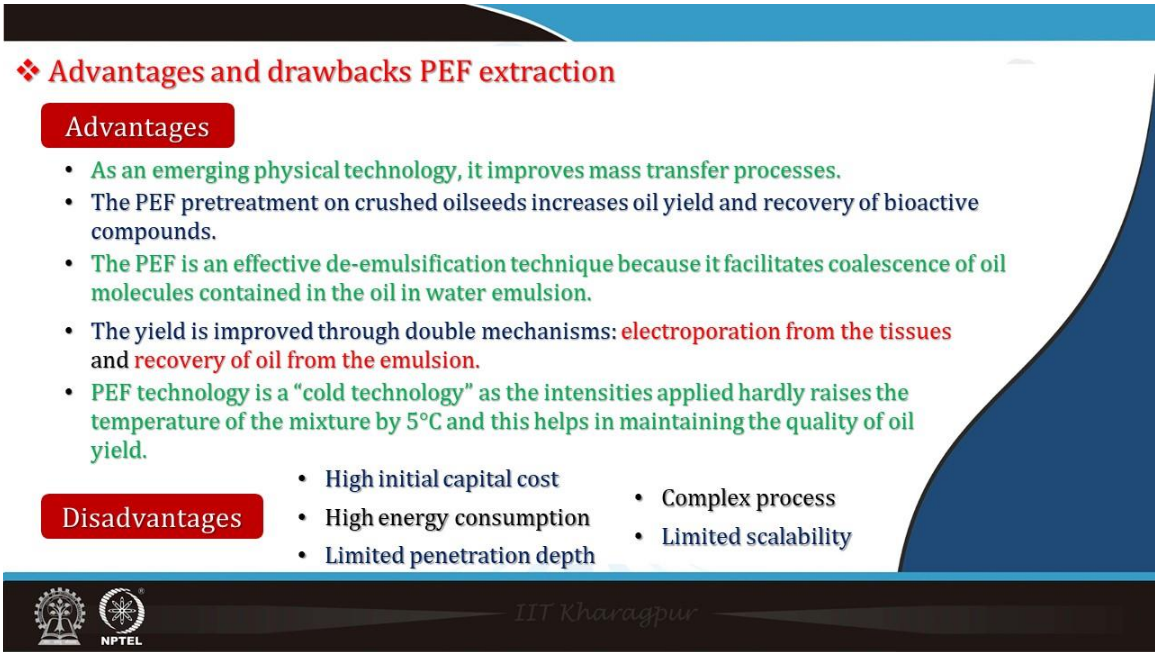
The advantages of the process are low extraction time, it is an environmentally friendly process, it is highly selective and no follow-up separation steps are required to obtain the oil from the substrate mixture. However, it has a disadvantage that is in spite of having a simple extraction process application of supercritical fluid extraction technology is limited due to high equipment cost, particularly for industrial scale and large scale process. Major SPE factors that are responsible like intrinsic factors include the temperature and pressure that determine the physical state of the solvent and extrinsic factors encompass characteristics of the sample and interaction of oil-bearing cells with the supercritical carbon dioxide and these influence the extraction process.



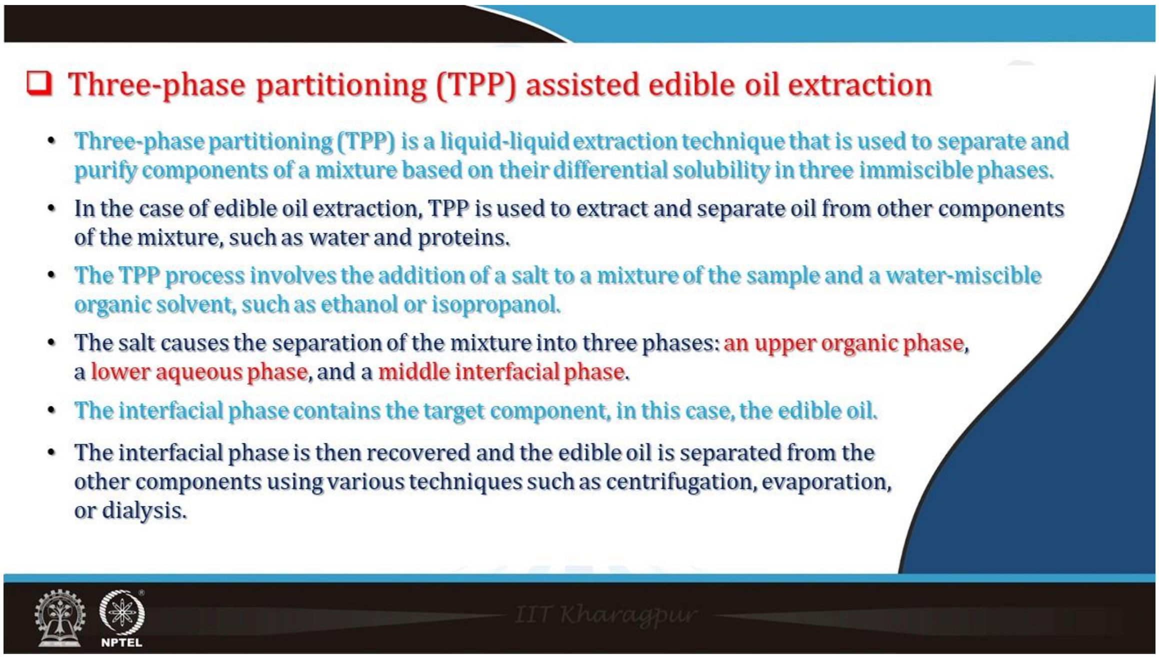
Pulse electric field (PEF) assisted extraction is a groundbreaking non-thermal technology that is used to improve the extraction efficiency of vegetable oil from various oilseeds. The technology involves the discharge of direct electric pulses into the oleaginous material for a short duration of time (microseconds to milliseconds) and high voltage (up to 50 kV). The oil-bearing material is placed between a high-voltage electrode and a grounded electrode. The electric pulses traverse through the cell membrane and generate electric fields (up to 10 kV/cm), which disintegrate the membrane molecules based on their net charge. The separation of membrane molecules results in the formation of pores and increases the permeability of the cell wall of the plant tissues. Consequently, the diffusion of solutes through the cell wall is enhanced by electroporation, and this favors the extraction of intracellular substances like oil and other components of interest. Pulse duration and pulse interval are the two fundamental factors affecting the effectiveness of PEF treatment.



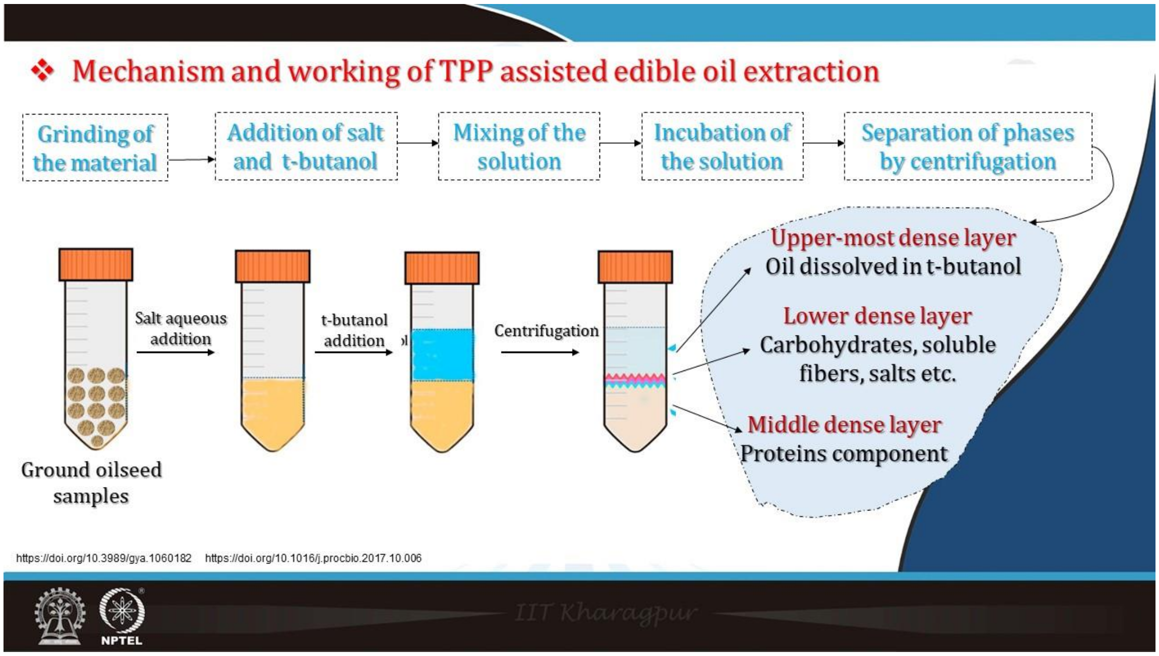
This is the PEF-assisted system where these are the electrodes and in between electrodes, the sample is placed. So, it needs first preparation of the sample, then extraction medium preparation, mixing, then pulse electric field treatment, separation, recovery of the oil, and then further processing. So, just after PEF treatment, it can be further subjected to the extraction process either by solvent extraction or by any other methods. So, PEF increases the pores.



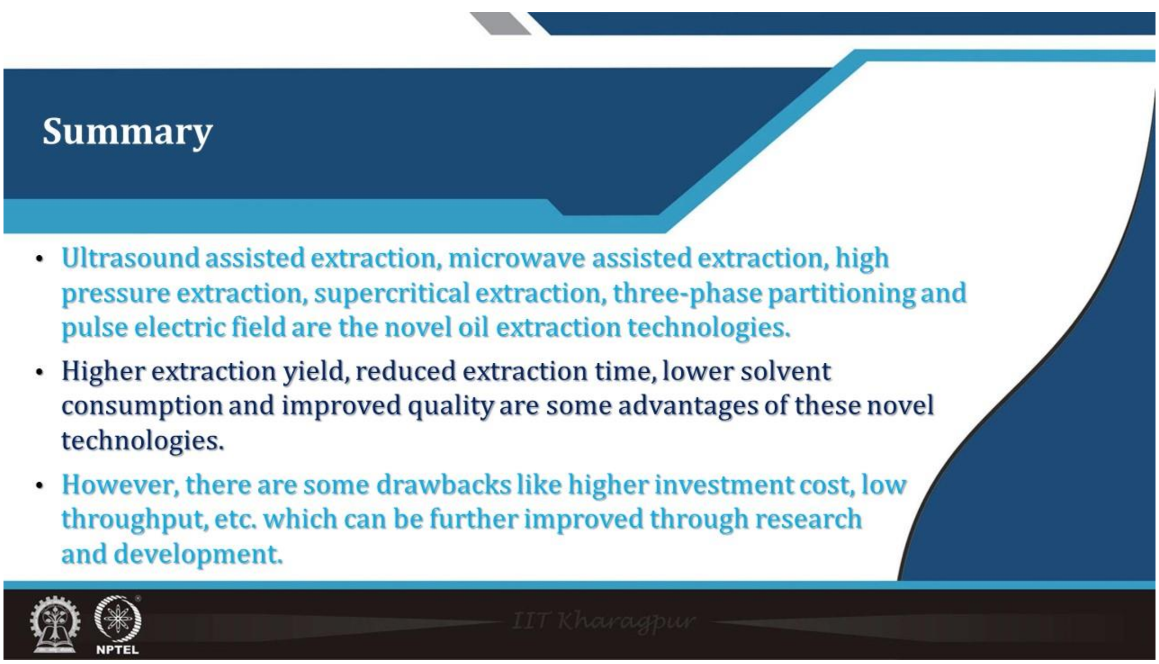
The advantages of the system PEF extraction are as an emerging physical technology it improves the mass transfer operation. The PEF pretreatment on crushed oil seed increases oil yield and recovery of bio-active compounds. The PEF is an effective de-emulsification technique because it facilitates the coalescence of oil molecules contained in the oil in water emulsion. The yield is improved through double mechanisms: electroporation from the tissues and recovery of oil from the emulsion. PEF technology is a “cold technology” as the intensities applied hardly raise the temperature of the mixture by 5°C and this helps in maintaining the quality of oil yield. However, the disadvantages of this technology include high initial capital cost, high energy consumption, limited penetration depth, complex process, and limited scalability.



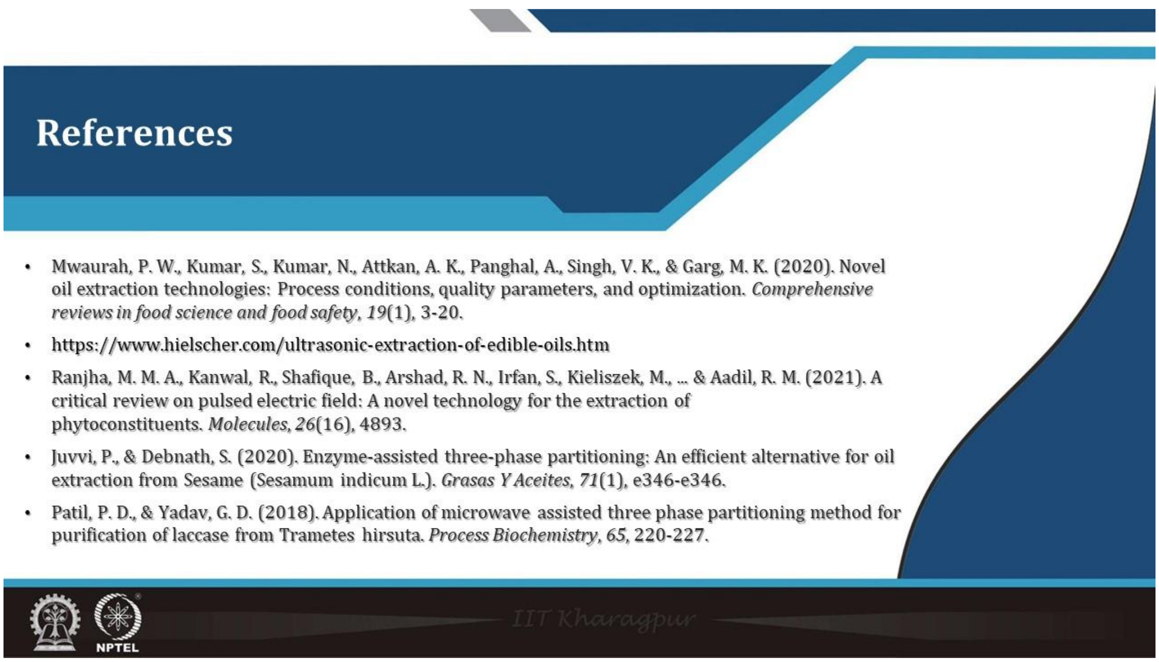
Three-phase partitioning (TPP) is a liquid-liquid extraction technique that is used to separate and purify components of a mixture based on their differential solubility in three immiscible phases. In the case of edible oil extraction, TPP is used to extract and separate oil from other components of the mixture, such as water and proteins. The TPP process involves the addition of salt to a mixture of the sample and a water-miscible organic solvent, such as ethanol or isopropanol. The salt causes the separation of the mixture into three phases: an upper organic phase, a lower aqueous phase, and a middle interfacial phase. The interfacial phase contains the target component, in this case, the edible oil. The interfacial phase is then recovered and the edible oil is separated from the other components using various techniques such as centrifugation, evaporation, or dialysis.



In this figure, I have shown the mechanism of working TPP-assisted edible oil extraction. First grinding of the oil material is taken from the system, then addition of salt and t butanol, and then these are properly mixed and incubation of the solution, given some time for the separation of the phases followed by the separation of phases by centrifugation. As you can see here that is these are separated into three layers, the uppermost layer, contains oil dissolved in t butanol, the lower dense layer includes carbohydrates, soluble fiber, salt etcetera, and the middle layer is the protein component. And then now these are separated by suitable techniques.



Now, I will summarize that Ultrasound-assisted extraction, microwave-assisted extraction, high-pressure extraction, supercritical extraction, three-phase partitioning, and pulsed electric field are novel oil extraction technologies. The detailed mechanism and working principle of the equipment and system of all these processes I have described in my other course the novel techniques for food processing preservation. But in this lecture, I just explained the application of these processes in oil extraction, how it is done, etc. So, higher extraction yield, reduced extraction time, lower solvent consumption, and improved quality are some advantages of these novel technologies. However, there are some drawbacks like higher investment costs, low throughput, etc. which can be further improved through research and development. There are many places where work is continued and indigenous equipment people are trying to develop systems and equipment. Once the equipment cost is reduced all these technologies can be versatile technology for getting more yield and even the drawback of the high capital investment can be compensated suitably by getting higher yield as well as good quality material both oil and meal.



So, these are the references used in this. Thank you very much for your patience. Thank you.