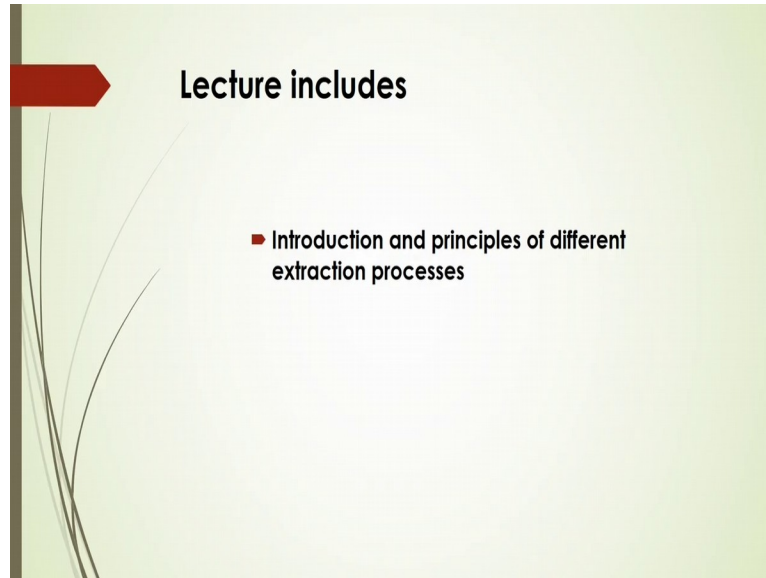


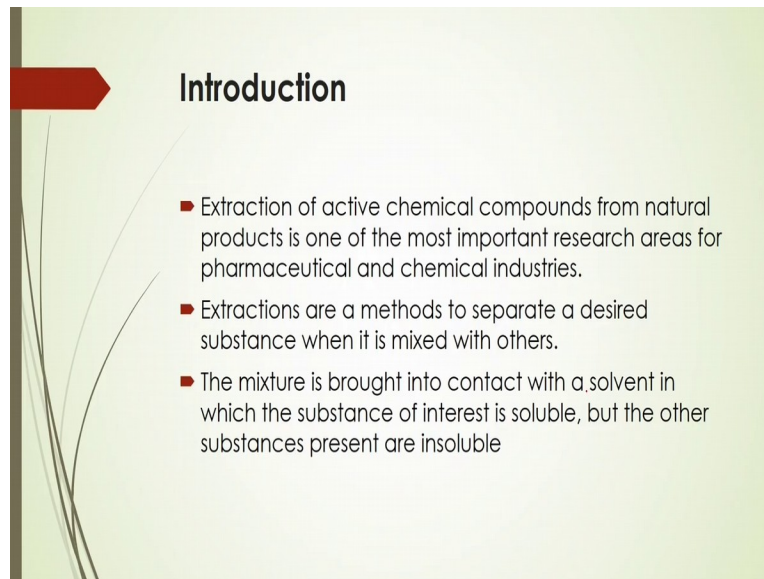
Chemical Process Intensification
Professor Subrata K. Majumder
Department of Chemical Engineering
Indian Institute of Technology Guwahati
Module 10 – Process Intensification in extraction
Lecture 10.1 - Introduction and Principles

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Welcome to massive open online course on Chemical Process Intensification, so in this module process intensification in extraction, we will discuss about its introduction and principles under this module and in this lecture of course we will discuss the different extraction processes and their principles and how that extractions will be helpful in different applied systems and also in you know process intensification aspect how it can be actually improved that will be discussed in this lecture.

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Introduction

- Extraction of active chemical compounds from natural products is one of the most important research areas for pharmaceutical and chemical industries.
- Extractions are a methods to separate a desired substance when it is mixed with others.
- The mixture is brought into contact with a solvent in which the substance of interest is soluble, but the other substances present are insoluble

So you know that what is extraction? That extraction actually just that separating of that some active chemical compounds from natural products and this is actually one of the most important research areas for pharmaceutical and chemical industries because different that medicinal compounds should be extracted from the plants and that depends on this extraction principles and in that case this method is basically the separation to a desired substance when it is mixed with others and the mixture is generally brought into contact with a solvent in which the substance of interest is soluble but the other substances present are insoluble.

So you will see that this extraction process is very important aspects in our daily life because all the products whatever we are actually using that are coming by certain process and in that case extraction is of the, of course the heart of that process because you to extract that process and then by utilising it in different way because those products whatever you are actually getting that is of course natural sources.

So from that natural sources you have to extract it anyway and after using that maybe it will be again actually converting into another products and then again there will be mixture and then from that mixture again you have to separate it and then extraction one of the process by which you can extract that unwanted materials all those things. So extraction is a process in the chemical engineering discipline where some useful or active chemical compounds to be extracted from the natural products and there are of course several that equipment and even techniques to improve that extraction process. Now we will try to actually discuss different mechanism and techniques based on which that intensification of this extraction process is done.

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Extractions use two immiscible phases (these are phases that do not mix, like oil and water) to separate the substance from one phase into the other.

Example In case of Tea-making you boil tea leaves in water to extract the tannins, theobromine, polyphenols, and caffeine out of the solid tea leaves and into the liquid water.

The diagram illustrates two types of extraction. On the left, 'Liquid-liquid extraction' is shown in a cycle: a 'Solvent (light phase)' (e.g., Paraffin) is mixed with a 'Feed solution (heavy phase) with mixture of solutes' (e.g., water + caproic acid). This creates a 'Dispersed phase' (paraffin droplets in water) and a 'Continuous phase' (water). After 'PHASE SEPARATION', the 'Extract' (paraffin with caproic acid) is separated from the 'Raffinate (with non-extracted solute)'. On the right, 'Solid-liquid extraction' is shown with two images: 'Tea leaf extract' (a dark liquid) and 'Olive leaf extract' (a green liquid).

And in that case first of all you have to know that what is the basic fundamental of that, that extraction, for extraction generally **two** immiscible phases are being used, these are generally you know that immiscible liquid that is called solvent extraction if you are performing. That means those phases will not mix to each other like oil and water and in that case to separate that substance from one phase into the other, then you have to use this mechanism of you know that transferring of one molecule from one immiscible phase to the other immiscible phase. And you will see that as an example like suppose here it is given in the slide that if there is a solution like heavy phase like water you can say with some mixture of solutes like if in a water suppose caproic acid is there is mixed there.

So if you want to extract that caproic acid from this water then you have to use this extraction process where you have to use some immiscible liquid to extract those caproic acid compounds from water. So in that case if you use that paraffin it will be a light phase, so if you mix this paraffin in a you know that mixture or if you can mix it in different way then this paraffin since it is lighter phase compared to that heavy phase here in water then this lighter phase of this paraffin of course will be dispersed in the water as a dispersed phase of droplets.

Whenever these droplets will be forming here in the water, it will be paraffin droplet. Since it is the lighter phase then droplet will be formed and during that formation of the droplet you will see there will be a transfer of this caproic acid molecule to the water through the interface of the droplet.

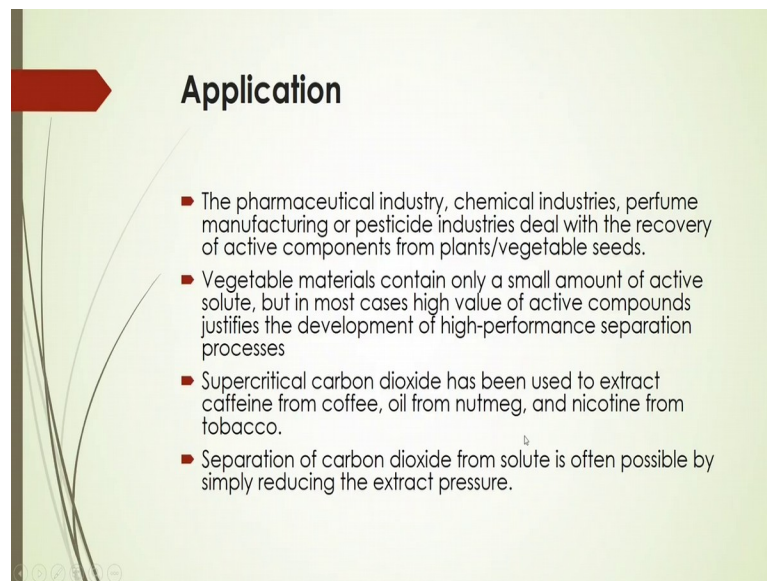
So this is the mechanism by which you can say that you can extract, you can take it from that water **to the** paraffin. Now the transferring of that caproic acid is basically from the water to the paraffin through a certain barrier, what is that barrier it is called that is surface interface. Now this interface there will be a certain you know that the assistance, so because of that some interfacial resistance there will be some mechanism to be followed to get that thin layer of the interfaces through which that solute will be transferred from that water to the paraffin.

So basically there will be an interface through which there will be a transfer of the molecules of this caproic acid to the **paraffin liquid that** is from the heavier phase to the lighter phase and after mixing and getting that transfer of this molecule it is called that mass transfer or basically it is called that extraction. You will see if you allow this mixer again for a certain time to settle, then you will see that again this paraffin liquid will be that settle it over that liquid there but at this moment you will see this paraffin will be reached to that caproic acid.

So in this way you can say that this caproic acid is being separated or extracted from this water to this paraffin. After that if you boil that mixture of this paraffin and caproic acid then you will see that paraffin will be more volatile than that caproic acid, in that way that paraffin will be separated from that caproic acid. So in this way you can extract or separate this caproic acid from the water and this is called that liquid-liquid extraction. Now there are other techniques, you can extract some you know that useful compounds from the solid material that will be called as liquid-solid extraction or solid-liquid extraction.

Like you know that if you think about that tea making, there you boil tea leaves in water to extract the tannins, theobromine, even polyphenols and caffeine out of the solid tea leaves and into the liquid water, so here that is called this solid-liquid extraction.

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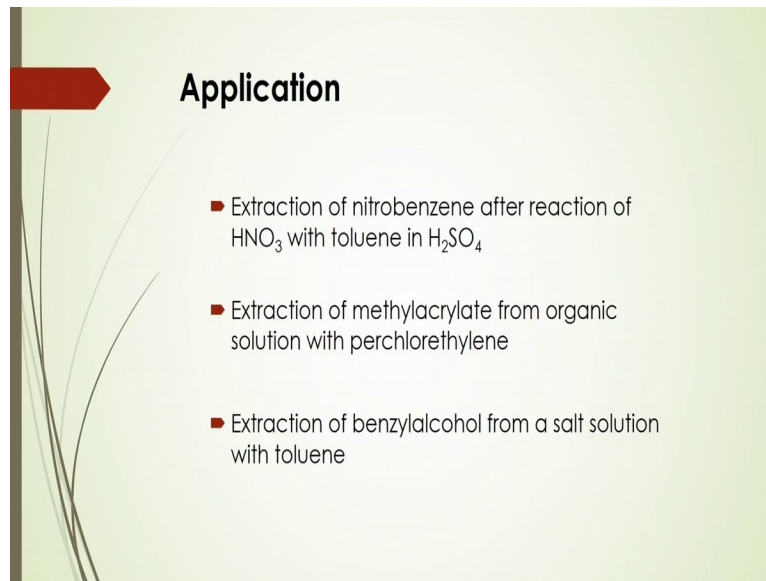
The slide features a light green background with a dark green vertical bar on the left side. A red arrow points to the right from the top of this bar. The title 'Application' is written in bold black text. Below the title, there are four bullet points, each starting with a red square. The text is as follows:

- The pharmaceutical industry, chemical industries, perfume manufacturing or pesticide industries deal with the recovery of active components from plants/vegetable seeds.
- Vegetable materials contain only a small amount of active solute, but in most cases high value of active compounds justifies the development of high-performance separation processes
- Supercritical carbon dioxide has been used to extract caffeine from coffee, oil from nutmeg, and nicotine from tobacco.
- Separation of carbon dioxide from solute is often possible by simply reducing the extract pressure.

Now there are several applications of this extraction processes; the pharmaceutical industry, the chemical industry, perfume manufacturing or you know that pesticides industries, they deal with the recovery of active components from plants or vegetable seeds and in that case you will see that vegetable materials contain only a small amount of active **solute** but in most cases high value of active compounds justifies the development of high-performance separation processes.

Another important you know that extraction process it is called supercritical extraction, in that case carbon dioxide gas is being used as a solvent at its supercritical condition to extract caffeine from the coffee and oil from nutmeg and nicotine form tobacco. This type of examples where supercritical condition is utilised to extract this compound by carbon dioxide gas. Now separation of carbon dioxide from solute is often possible by simply reducing the extract pressure, so in that case it will be sometimes more feasible to extract those material by this supercritical condition of carbon dioxide as a solvent.

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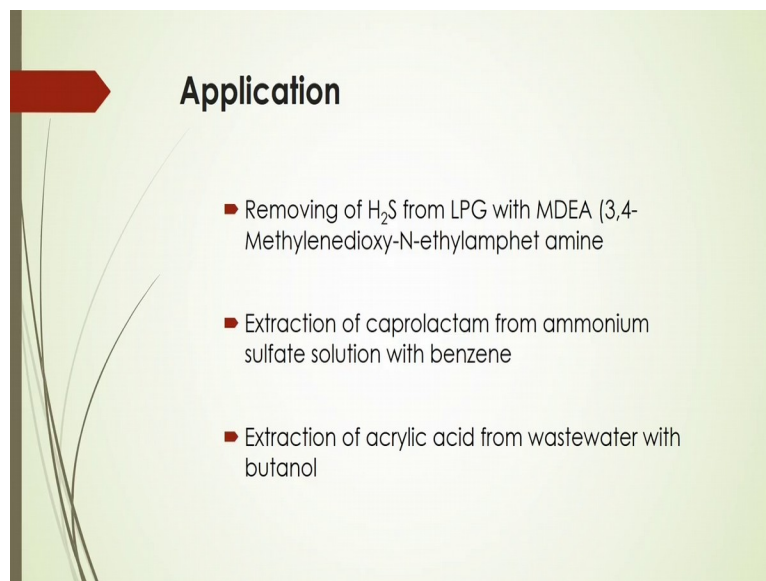


Application

- Extraction of nitrobenzene after reaction of HNO_3 with toluene in H_2SO_4
- Extraction of methylacrylate from organic solution with perchlorethylene
- Extraction of benzylalcohol from a salt solution with toluene

Other examples of applications like extraction of nitrobenzene after reaction of nitric acid with toluene in sulphuric acid. Extraction of methylacrylate from organic solution with ethylene, even you know that extraction of benzylalcohol from a salt solution with toluene.

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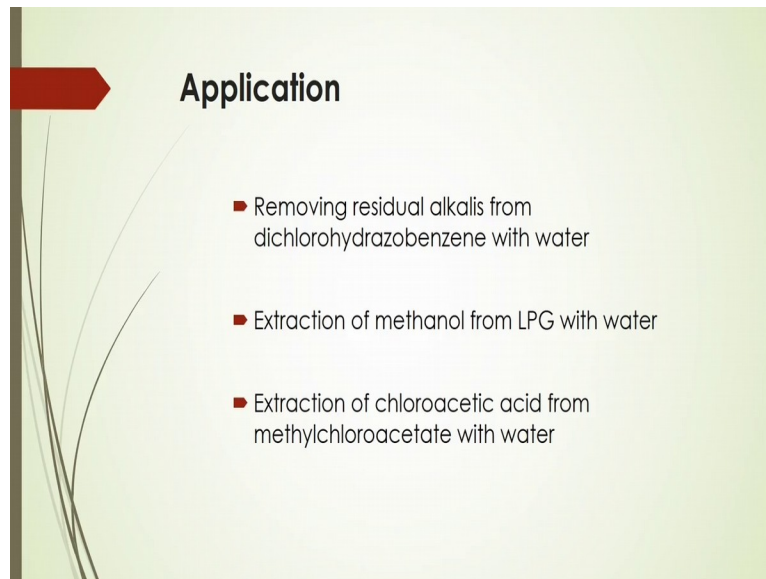


Application

- Removing of H_2S from LPG with MDEA (3,4-Methylenedioxy-N-ethylamphet amine)
- Extraction of caprolactam from ammonium sulfate solution with benzene
- Extraction of acrylic acid from wastewater with butanol

In that case you have to use this extractive mechanism of remove all those compounds. Even other applications like that removing of hydrogen sulphide from LPG with MDEA and also extraction of caprolactam from ammonium sulphate solution with benzene, extraction of acrylic acid from wastewater with butanol.

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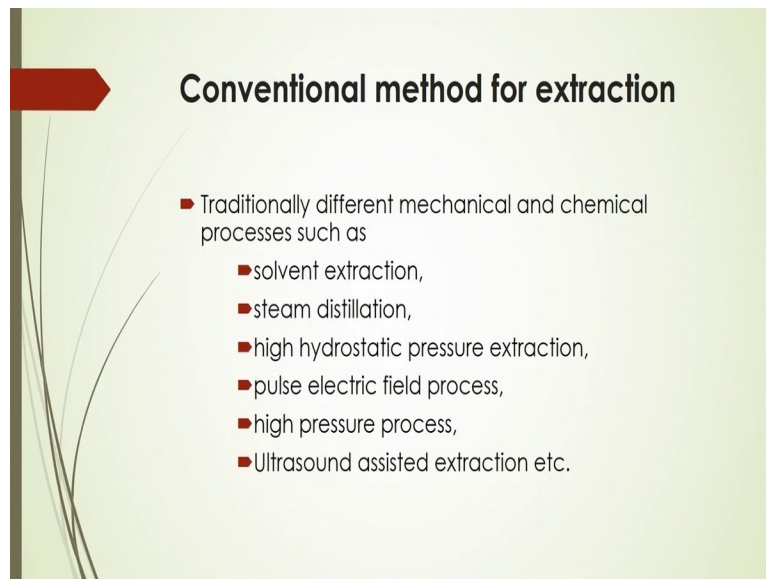


Application

- Removing residual alkalis from dichlorohydrazobenzene with water
- Extraction of methanol from LPG with water
- Extraction of chloroacetic acid from methylchloroacetate with water

Removing the residual alkalis from dichlorohydrazobenzene with water, extraction of methanol from LPG with water, extraction of chloroacetic acid from methylchloroacetate with water, this type of applications are there for the extraction process.

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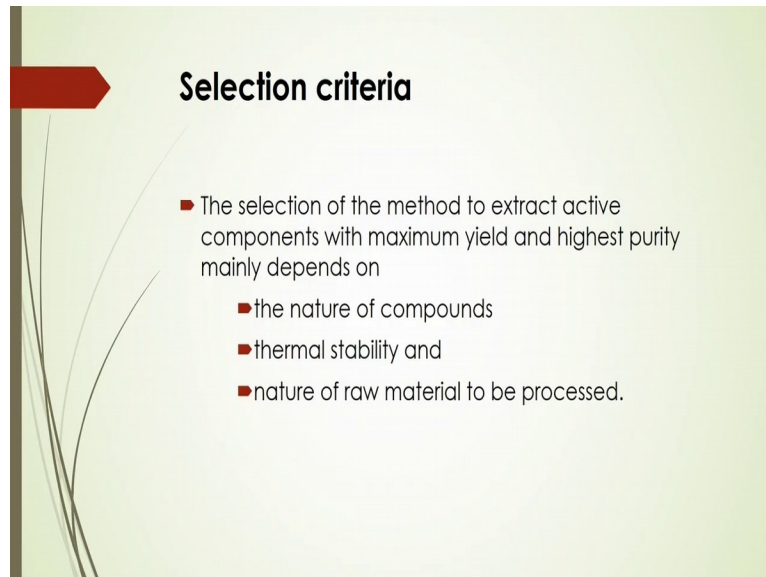
Conventional method for extraction

- Traditionally different mechanical and chemical processes such as
 - solvent extraction,
 - steam distillation,
 - high hydrostatic pressure extraction,
 - pulse electric field process,
 - high pressure process,
 - Ultrasound assisted extraction etc.

Now what are the different conventional methods for extraction? Traditionally different mechanical and chemical processes are followed to extract these compounds, valuable compounds from the plants, vegetables or other solid materials. Now in that case solvent extraction is one of the important part of that category of this liquid-liquid extraction. Steam distillation is also used to you know that separate those compounds from the sources. High hydrostatic pressure extraction, pulse electric field processes, high-pressure processes,

sometimes ultrasound assisted extraction they are, so these are the different methods by which you can extract the valuable compounds from the natural sources.

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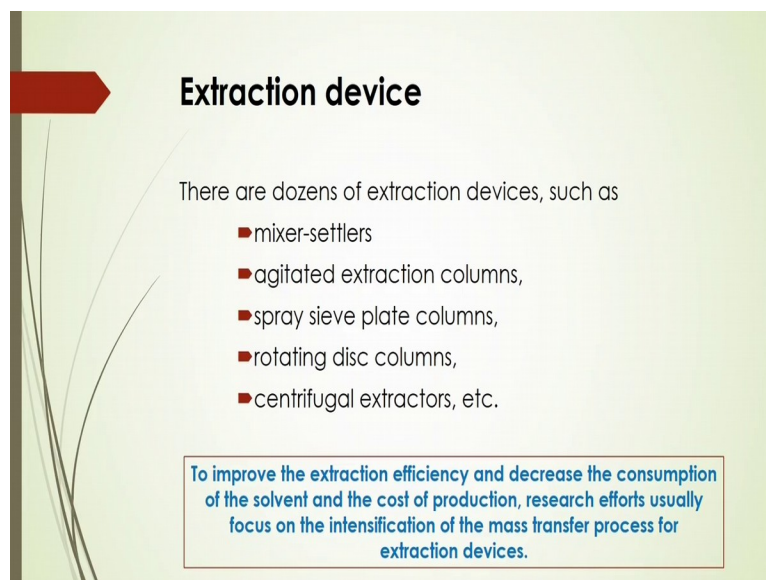


Selection criteria

- The selection of the method to extract active components with maximum yield and highest purity mainly depends on
 - the nature of compounds
 - thermal stability and
 - nature of raw material to be processed.

Now there should be certain criteria to use those methods, now the selection of the method to extract active components with maximum yield and highest purity that mainly depends on the nature of compounds, thermal stability and nature of raw materials which is actually being to be processed.

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Extraction device

There are dozens of extraction devices, such as

- mixer-settlers
- agitated extraction columns,
- spray sieve plate columns,
- rotating disc columns,
- centrifugal extractors, etc.

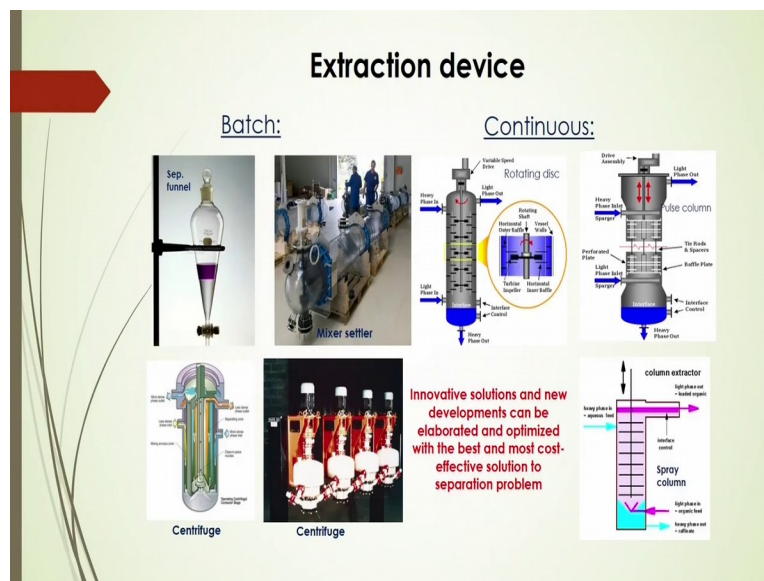
To improve the extraction efficiency and decrease the consumption of the solvent and the cost of production, research efforts usually focus on the intensification of the mass transfer process for extraction devices.

Now what are the extraction devices generally being used? There are dozens of extraction devices such as mixer settlers, agitated extraction columns, spray sieve plate columns,

rotating disc columns, centrifugal extractors et cetera and to improve the extraction efficiency and decrease the consumption of the solvent and the cost of production, research efforts usually focus on the intensification of the mass transfer process for these extraction devices. That is why the intensification of the chemical process are one of the very attractive field where you can have more idea about how to actually intensify the process based on their mass transfer, based on their techniques, based on their different mechanisms, based on the hydrodynamics, based on the equipment size, based on the other mechanism like integrating the systems or consuming the energy, so these are the you know way to intensify these processes.

Thus similarly that extraction also one of the important processes where you have to intensify the process, that conventional system are not giving that much of you know that attractive, even that 100 percent efficiency of that. So to improve that extraction efficiency and also parallely consumption of the solvent and cost of the production taking into account, research should be done on the intensification of the mass transfer process for extraction devices.

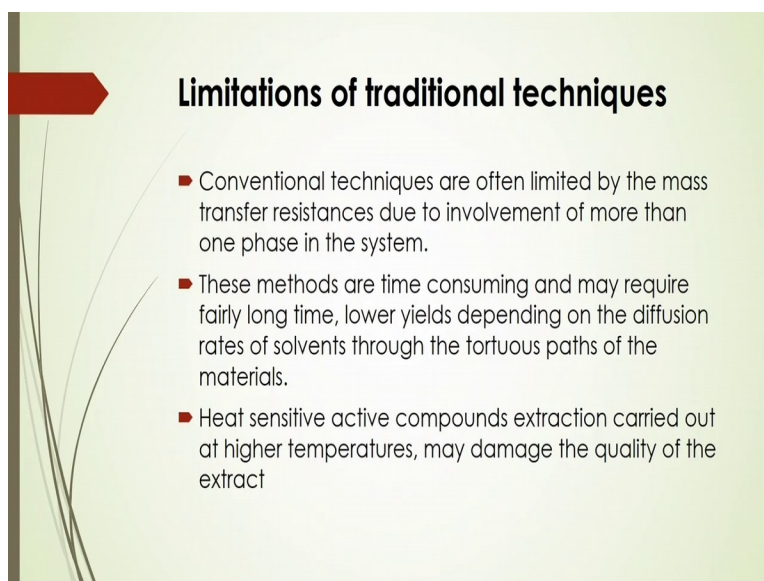
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Now see here several extraction devices are actually being used for this extraction process, traditionally like you know that in batch process separating funnel earlier stage they have used. Even the mixture settler, centrifuge and centrifuge are different types of centrifuge are being used for extraction in batch systems. For continuous system you know that rotating disc column even that column extractor when it is called the pulse extraction columns, spray extraction column they are different mechanism. So these are the different intensification of this process from the traditional extraction process.

So how to get this continuous operation for the extraction process, that you can do it in a rotating disk, even in spray column systems and pulse column system. Every system has its own advantage but it should have some disadvantage also. So you have to optimise those processes where you can get that more intensified way of mass transfer for this extraction process. So innovative solutions and new developments can be elaborated and optimised with the best and most cost-effective solution to separation problem.

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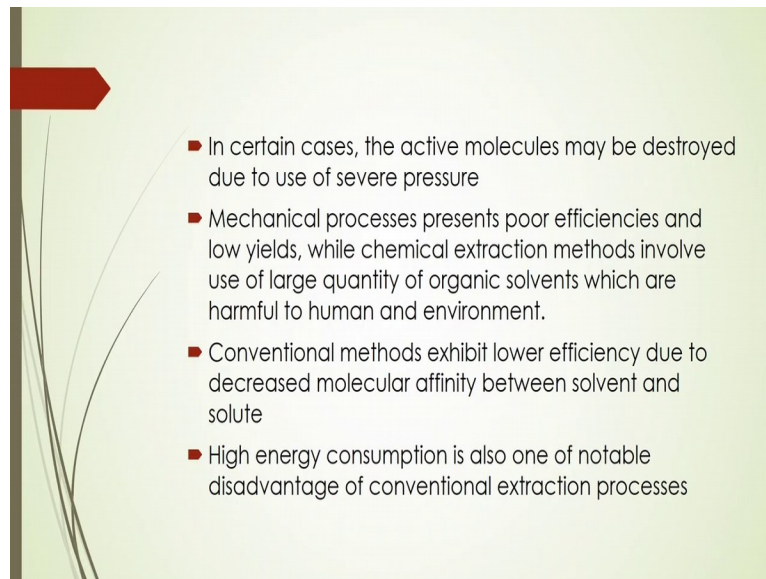


Limitations of traditional techniques

- Conventional techniques are often limited by the mass transfer resistances due to involvement of more than one phase in the system.
- These methods are time consuming and may require fairly long time, lower yields depending on the diffusion rates of solvents through the tortuous paths of the materials.
- Heat sensitive active compounds extraction carried out at higher temperatures, may damage the quality of the extract

Now conventional techniques are often limited by the mass transfer resistances due to the you know involvement of the more than one phase in the system and these methods are time-consuming and may require fairly long time, lower yields depending on the diffusion rates of the solvents through the zig zag paths or you can say tortuous parts of the materials. Sometimes heat sensitive active compounds extraction, that being carried out at higher temperatures and it may damage the quality of the extract there.

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- In certain cases, the active molecules may be destroyed due to use of severe pressure
- Mechanical processes presents poor efficiencies and low yields, while chemical extraction methods involve use of large quantity of organic solvents which are harmful to human and environment.
- Conventional methods exhibit lower efficiency due to decreased molecular affinity between solvent and solute
- High energy consumption is also one of notable disadvantage of conventional extraction processes

And also in certain cases you will see the active molecules may be destroyed due to use of severe pressure and also mechanical processes that presents poor efficiencies and low yields while chemical extraction methods involve use of large quantity of organic solvents which are harmful to human and environment. So that is why this is some you know the shortcomings of that using of extraction process by liquid-liquid mechanism or solid-liquid mechanism even in mechanical processes. So conventional methods exhibit lower efficiency due to the decreased molecular affinity between solvent and solute, this is also one another important shortcomings. High energy consumption is of course to be considered there, is one of the notable disadvantage of the conventional extraction process.

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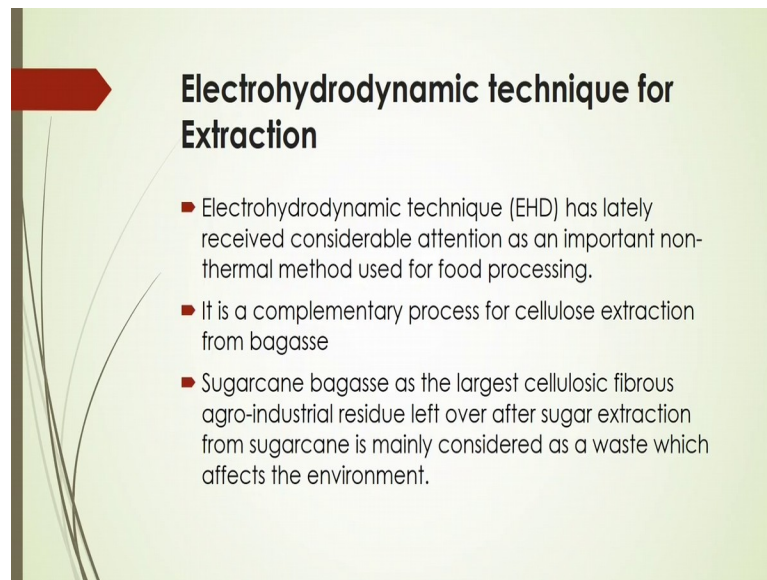
Why intensification required?

- Government, health and environmental regulatory requirements on the use of organic solvents have encouraged active research on clean extraction techniques
- The need for effective extraction technique active components from plants without any loss of activity and high purity.
- The confines of the conventional techniques make obstinate to search for the new techniques which are equally competent and at the same time economically viable.

Now that is why the intensification you have to think about, how to do that intensification and why that intensification required. Nowadays that government health and environmental regulatory requirements on the use of organic solvents that have encouraged active research on clean extraction techniques and for that it is required to have the effective extraction technique, effective way to separate that active components from the plants without any loss of activity and high purity.

And the confines of the conventional techniques make the obstinate to search for the new techniques which are equally competent and at the same time that is also economically viable, so that is why you have to think about that how to intensify the techniques to develop the process considering these shortcomings there.

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Electrohydrodynamic technique for Extraction

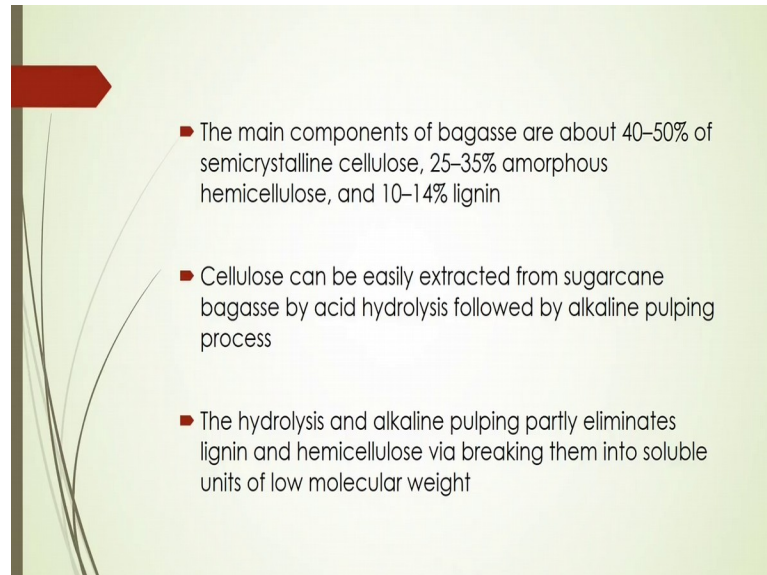
- Electrohydrodynamic technique (EHD) has lately received considerable attention as an important non-thermal method used for food processing.
- It is a complementary process for cellulose extraction from bagasse
- Sugarcane bagasse as the largest cellulosic fibrous agro-industrial residue left over after sugar extraction from sugarcane is mainly considered as a waste which affects the environment.

Now based on that thinking there are several techniques are coming to intensify that extraction process, in that case electro-hydrodynamic techniques is one of the important the extraction techniques for its intensification. In this case that electro-hydrodynamic techniques has lately received considerable attention as an important non-thermal method used for food processing, in that case you can save some energy where that conventional techniques, that thermal energy is being used for that extraction of available products from the solid materials. In that case if you use that electro-hydrodynamic process you can save some thermal energy.

It is a complementary process for cellulose extraction from the bagasse and also sugarcane bagasse as the largest cellulose fibrous agro-industrial residue left over the sugar extraction from sugarcane, that is mainly considered as a waste which affects the environment. So in

that you have to think about that how to use effective techniques for getting rid of this utilisation of that sugarcane bagasse and also utilisation of that sugarcane bagasse waste for your useful beneficiation.

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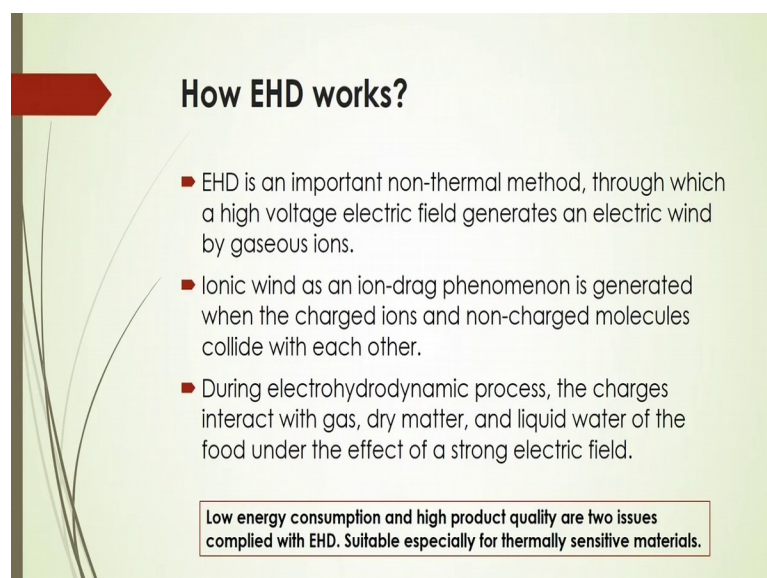


- The main components of bagasse are about 40–50% of semicrystalline cellulose, 25–35% amorphous hemicellulose, and 10–14% lignin
- Cellulose can be easily extracted from sugarcane bagasse by acid hydrolysis followed by alkaline pulping process
- The hydrolysis and alkaline pulping partly eliminates lignin and hemicellulose via breaking them into soluble units of low molecular weight

Low energy consumption and high product quality are two issues complied with EHD. Suitable especially for thermally sensitive materials.

And the main components of bagasse are about 40 to 50 percent of semicrystalline cellulose, 25 to 35 percent that will be amorphous hemicellulose and also 10 to 14 percent lignin. So in that way that cellulose can be easily extracted from sugarcane bagasse by acid hydrolysis that followed by alkaline pulping process and hydrolysis and alkaline pulping partly eliminates that lignin and hemicellulose via breaking them into soluble units of low molecular weight there.

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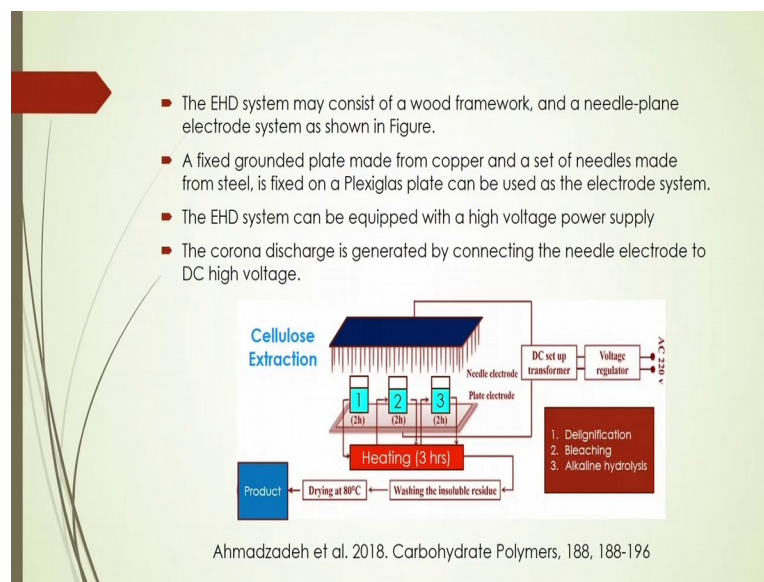
How EHD works?

- EHD is an important non-thermal method, through which a high voltage electric field generates an electric wind by gaseous ions.
- Ionic wind as an ion-drag phenomenon is generated when the charged ions and non-charged molecules collide with each other.
- During electrohydrodynamic process, the charges interact with gas, dry matter, and liquid water of the food under the effect of a strong electric field.

Low energy consumption and high product quality are two issues complied with EHD. Suitable especially for thermally sensitive materials.

Now how this actually electro-hydrodynamic process of extraction works there? So that EHD is an important non-thermal method you know that in this case a high voltage electric field actually is being used for this extraction process. In that case a high-voltage electric field generates an electric wind by gaseous ions, that ionic wind as an ion drag phenomenon is generated when the charged ions and non-charged molecules collide with each other. During that EHD process the charges interact with the gas, dry matter, and liquid water of the food under the effect of strong electric field. So in this case low energy consumption and high product quality are 2 issues that compile with EHD, in this case suitable you can say that it is a suitable process especially for the thermally sensitive material.

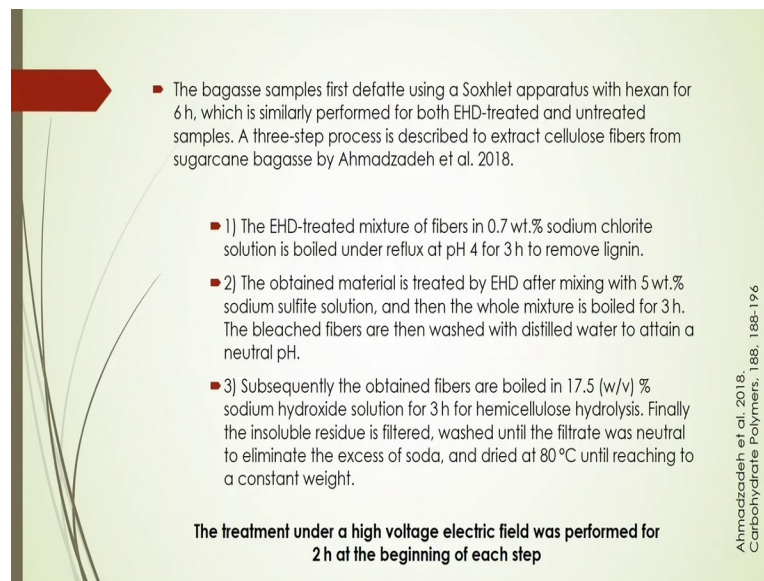
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And there is actually system that it is shown in the picture that this EHD system consist of a wood framework and a needle-plane electrode system as shown in the figure and in this case a fixed grounded plate made from copper and a set of needles that made from steel is fixed on a Plexiglas plate and it is actually used as the electrode system there and this also can be procured with a high-voltage power supply and in that case this mechanism to generate this ionic wind by that corona discharge method.

In that case this corona discharge is generated by connecting the needle electrode to DC high voltage there. So cellulose extraction can be done by these electro-hydrodynamic techniques. It is actually suggested by this Ahmadzadeh et al. in 2018 and it is published in carbohydrate polymers in 2018. So this is one of the important direction of the research to get more intensification of the valuable compounds by non-thermal processes.

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- The bagasse samples first defatted using a Soxhlet apparatus with hexan for 6 h, which is similarly performed for both EHD-treated and untreated samples. A three-step process is described to extract cellulose fibers from sugarcane bagasse by Ahmadzadeh et al. 2018.
- 1) The EHD-treated mixture of fibers in 0.7 wt.% sodium chlorite solution is boiled under reflux at pH 4 for 3 h to remove lignin.
- 2) The obtained material is treated by EHD after mixing with 5 wt.% sodium sulfite solution, and then the whole mixture is boiled for 3 h. The bleached fibers are then washed with distilled water to attain a neutral pH.
- 3) Subsequently the obtained fibers are boiled in 17.5 (w/v) % sodium hydroxide solution for 3 h for hemicellulose hydrolysis. Finally the insoluble residue is filtered, washed until the filtrate was neutral to eliminate the excess of soda, and dried at 80 °C until reaching to a constant weight.

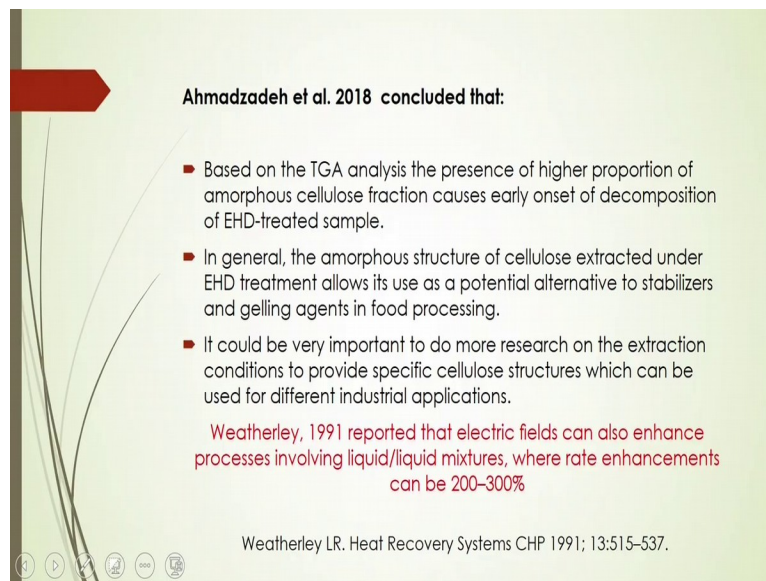
The treatment under a high voltage electric field was performed for 2 h at the beginning of each step

Ahmadzadeh et al., 2018, Carbohydrate Polymers, 188, 188-196

And in this case you will see this Ahmadzadeh et al. in 2018 they actually given this process of you know for extraction of cellulose fibres from the sugarcane bagasse here. They have actually given this three-steps of this EHD treated extraction of the cellulose fibres, in that case they suggested that this EHD treated mixture of fibres in 0.5 weight percent sodium chloride solution is to be boiled and reflux at pH 4 for 3 hours to remove that lignin and this can be obtained by treating after mixing with you know that 5 weight percent sodium sulphide solution and then the whole mixture to be boiled for 3 hours and then you know that the bleached fibres are then washed with distilled water to obtain a neutral pH.

So this is the way by which you can extract these cellulose fibres according to this report of this Ahmadzadeh et al. And in this case subsequently be obtained fibres are boiled in 17.5 weight per volume percent sodium hydroxide solution for three hours for hemicellulose hydrolysis and finally this insoluble residue is to be filtered, watched until the filtrate was neutral to eliminate the excess of soda and dried at 80 degree centigrade until it reaches to a constant weight. So this is the treatment under a high-voltage electric field and it is generally being done for two hours at the beginning of each step. So this method can be used to extract these cellulose fibres from the sugarcane as suggested by these authors.

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Ahmadzadeh et al. 2018 concluded that:

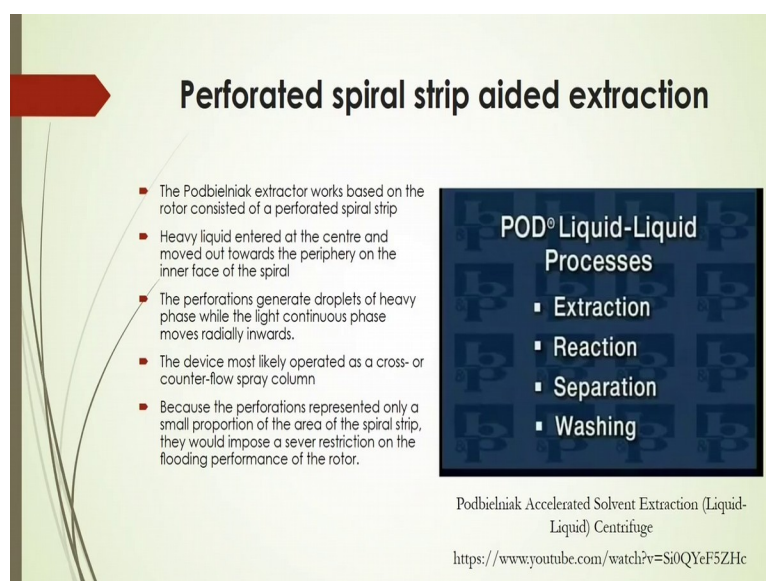
- Based on the TGA analysis the presence of higher proportion of amorphous cellulose fraction causes early onset of decomposition of EHD-treated sample.
- In general, the amorphous structure of cellulose extracted under EHD treatment allows its use as a potential alternative to stabilizers and gelling agents in food processing.
- It could be very important to do more research on the extraction conditions to provide specific cellulose structures which can be used for different industrial applications.

Weatherley, 1991 reported that electric fields can also enhance processes involving liquid/liquid mixtures, where rate enhancements can be 200-300%

Weatherley LR. Heat Recovery Systems CHP 1991; 13:515-537.

And as per their conclusion that based on the TGA analysis the presence of higher proportion of the amorphous cellulose fraction causes early onset of decomposition of EHD treated sample and also it could be very important to do more research on the extraction conditions to improve specific cellulose structures which can be used for different industrial applications. Also Weatherley in 1991 reported the electric fields can also enhance the process that involves liquid-liquid mixture where rate enhancements can be 200 to 300 percent, so this is one of the important research field. By this principle you can intensify the extraction process where this thermally you know that viable materials are to be extracted there.

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Perforated spiral strip aided extraction

- The Podbielniak extractor works based on the rotor consisted of a perforated spiral strip
- Heavy liquid entered at the centre and moved out towards the periphery on the inner face of the spiral
- The perforations generate droplets of heavy phase while the light continuous phase moves radially inwards.
- The device most likely operated as a cross- or counter-flow spray column
- Because the perforations represented only a small proportion of the area of the spiral strip, they would impose a severe restriction on the flooding performance of the rotor.

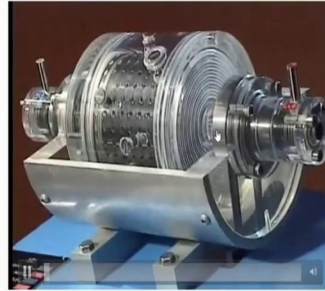
POD[®] Liquid-Liquid Processes

- Extraction
- Reaction
- Separation
- Washing

Podbielniak Accelerated Solvent Extraction (Liquid-Liquid) Centrifuge
<https://www.youtube.com/watch?v=Si0QYeF5ZHc>

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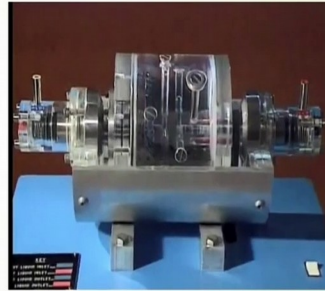


Podbielniak Accelerated Solvent Extraction (Liquid-Liquid) Centrifuge

<https://www.youtube.com/watch?v=Si0QYeF5ZHc>

Perforated spiral strip aided extraction

- The Podbielniak extractor works based on the rotor consisted of a perforated spiral strip
- Heavy liquid entered at the centre and moved out towards the periphery on the inner face of the spiral
- The perforations generate droplets of heavy phase while the light continuous phase moves radially inwards.
- The device most likely operated as a cross- or counter-flow spray column
- Because the perforations represented only a small proportion of the area of the spiral strip, they would impose a severe restriction on the flooding performance of the rotor.

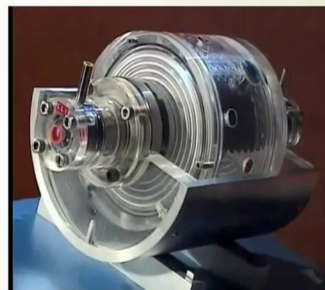


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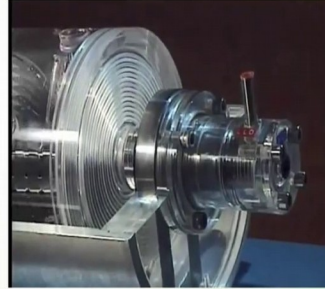


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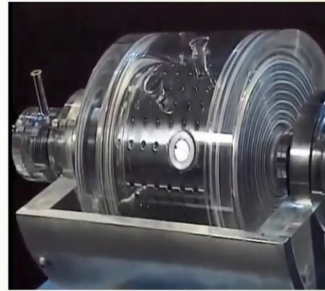


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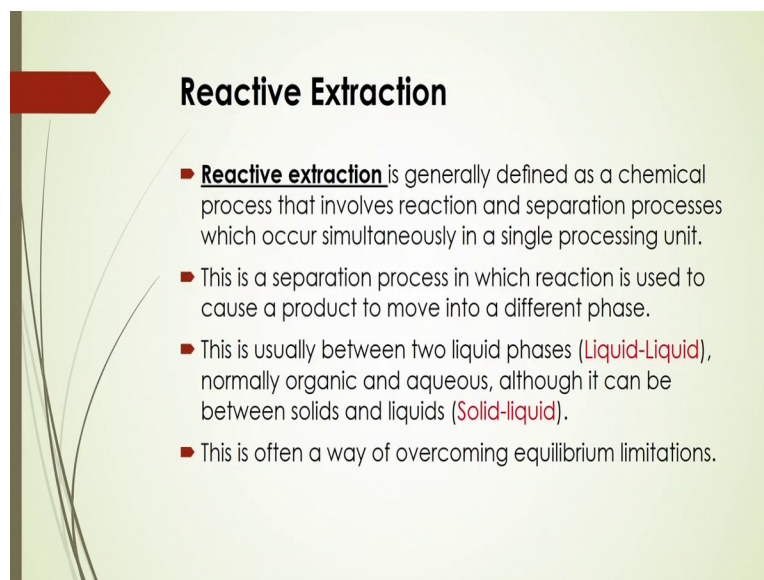
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Another important you know that extraction process that is called a spiral strip aided extraction and it is named as special you know that consideration of some extraction process based on the name that scientist Podbielniak extraction process and that extractor works based on the rotor that will be consisted of a perforated spiral. So in this case see how this you know perforated spiral strip aided extraction works. In this case heavy liquid enters at the centre and moved out towards the periphery on the inner face of the spiral and the perforation generates droplets of heavy phase while the light continuous phase moves radially inwards there. And the device most likely operate as a you know that cross or counter flow spray column.

As shown here you will see that the slides in the video and in this case the because the perforations that represented only a small proportion of the area of spiral strip, this extractor you know that would impose a severe restriction on the flooding performance of the rotor. So this is one of the important process intensification on the extraction process based on which you can produce that interface by supplying that lighter feed to the you know in this extractor where you can produce more droplet and interfacial area where mass transfer will be intensified and more extraction will be there.

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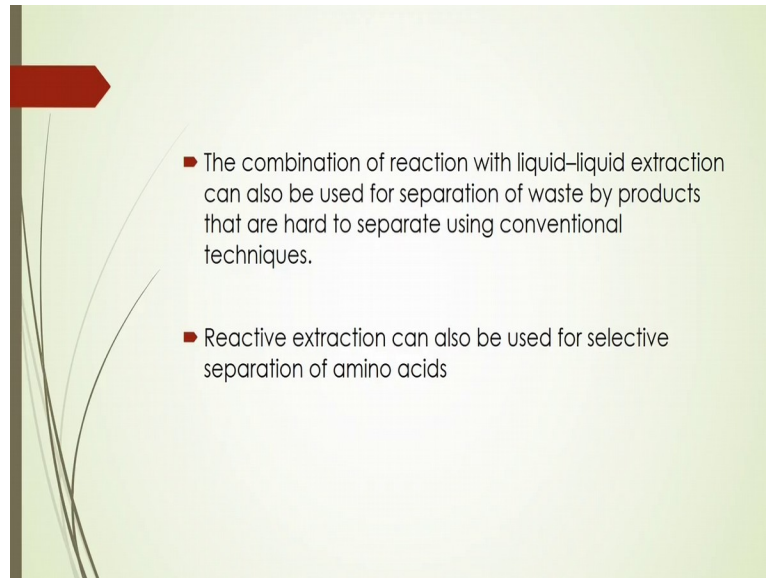
Reactive Extraction

- **Reactive extraction** is generally defined as a chemical process that involves reaction and separation processes which occur simultaneously in a single processing unit.
- This is a separation process in which reaction is used to cause a product to move into a different phase.
- This is usually between two liquid phases (**Liquid-Liquid**), normally organic and aqueous, although it can be between solids and liquids (**Solid-liquid**).
- This is often a way of overcoming equilibrium limitations.

And also another important aspects of process intensification in the extraction process, it is called that reactive extraction, in this case chemical process of this extraction here it will involve the reaction as well as separation process that is called integrating system of this reaction and extraction and both are actually simultaneously occurred in a single processing unit. So that is why this sizing effect when reducing the equipment number and also that

other energy consumption based on which that process intensification is being done and in this case the separation process in which the reaction is used to cause a product to move into a different face there.

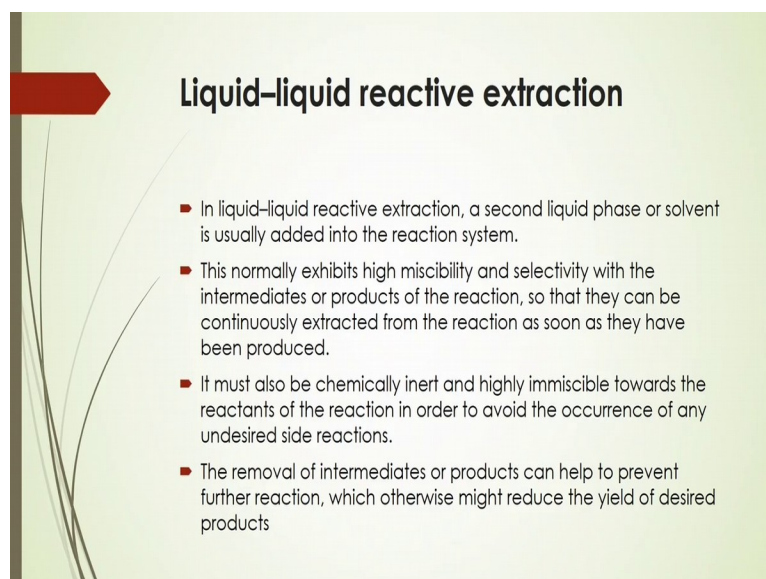
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- The combination of reaction with liquid-liquid extraction can also be used for separation of waste by products that are hard to separate using conventional techniques.
- Reactive extraction can also be used for selective separation of amino acids

And the combination of this reaction with liquid-liquid extraction can also be used for separation of waste by products that are you know hard to separate using conventional techniques and reactive extraction can also be used for you know selective separation of amino acids.

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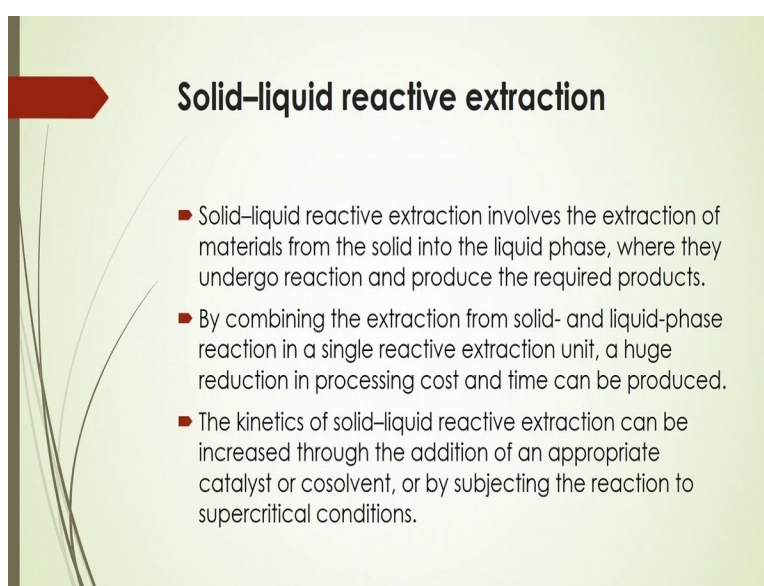


Liquid-liquid reactive extraction

- In liquid-liquid reactive extraction, a second liquid phase or solvent is usually added into the reaction system.
- This normally exhibits high miscibility and selectivity with the intermediates or products of the reaction, so that they can be continuously extracted from the reaction as soon as they have been produced.
- It must also be chemically inert and highly immiscible towards the reactants of the reaction in order to avoid the occurrence of any undesired side reactions.
- The removal of intermediates or products can help to prevent further reaction, which otherwise might reduce the yield of desired products

And also in this case you will see that this normally exhibits high miscibility and selectivity with the intermediates or products of the reaction so that they can continuously be extracted from the reaction as soon as they have been produced and in this case that a second phase or solvent is usually added into the reaction system. And in this case we have to remember that this you know that it must be chemically inert and highly immiscible towards the reactants of reaction in order to avoid the occurrence of any undesired side reactions there and the removal of intermediates or products can help to prevent further reaction which otherwise might reduce the yield of desired products there.

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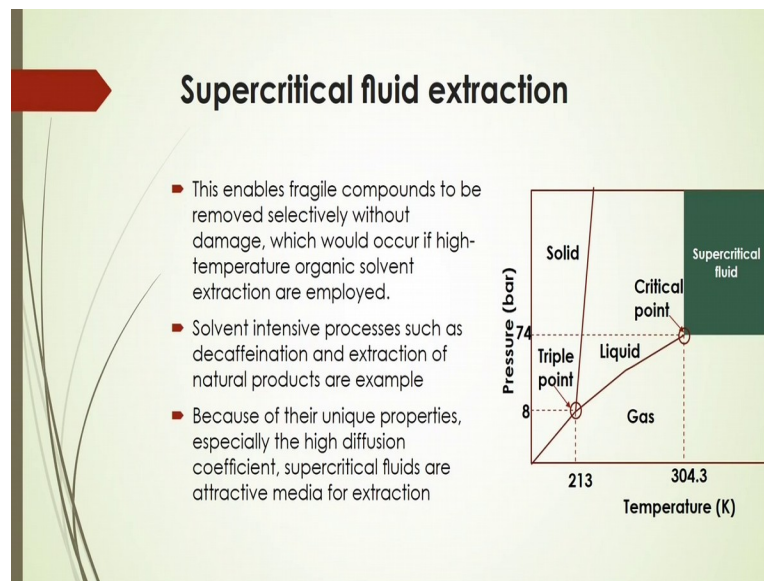


Solid-liquid reactive extraction

- Solid-liquid reactive extraction involves the extraction of materials from the solid into the liquid phase, where they undergo reaction and produce the required products.
- By combining the extraction from solid- and liquid-phase reaction in a single reactive extraction unit, a huge reduction in processing cost and time can be produced.
- The kinetics of solid-liquid reactive extraction can be increased through the addition of an appropriate catalyst or cosolvent, or by subjecting the reaction to supercritical conditions.

Another you know that aspects of that solid-liquid reactive extraction based on this process intensification of integrating system of this reaction as well as extraction. In that case solid-liquid reactive extraction in this process intensification system involves the extraction of materials from the solid into the liquid phase where they undergo reaction and produce the required products. By combining the extraction from the solid and liquid phase reaction in a single reactive extraction unit, a huge reduction in processing cost and time can be produced. In this case the kinetics of that reaction that is basically solid and liquid extraction can be increased through the addition of an appropriate catalyst or you can use some co-solvent or by subjecting the reaction to the some other operating condition like supercritical conditions there.

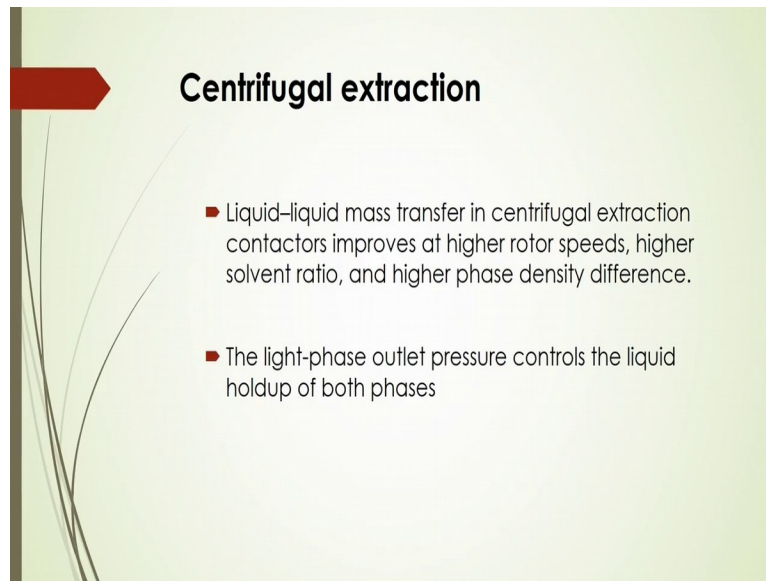
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And the supercritical fluid extraction systems is also one of the important process intensification of the extraction where you can get fragile compounds without damage which should occur at high temperature organic solvent and in that case the solvent which are being used at this supercritical condition, you know the solvent intensive process such as decaffeination and extraction of the natural products are simple example for this supercritical fluid extraction. And in this case because of their unique properties, especially at this you know above this critical point of the fluid, the high diffusion coefficient, even supercritical fluids exhibits that some other physical properties and because of which you can get that more mass transfer and more extraction based on this supercritical condition.

So here in this figure it is shown that some supercritical conditions of this gas liquid-solid system here, so how **at this, what** is the triplet point of that and then what is a critical point that 304.3 and pressure at 74, above which that the water will be converted into a supercritical fluid. Even for different gases also if you use that high pressure even high-temperature, you will see there will be some supercritical conditions of that carbon dioxide gas and that carbon dioxide gas would be used as a solvent for extraction there for liquid-liquid extraction and the supercritical condition.

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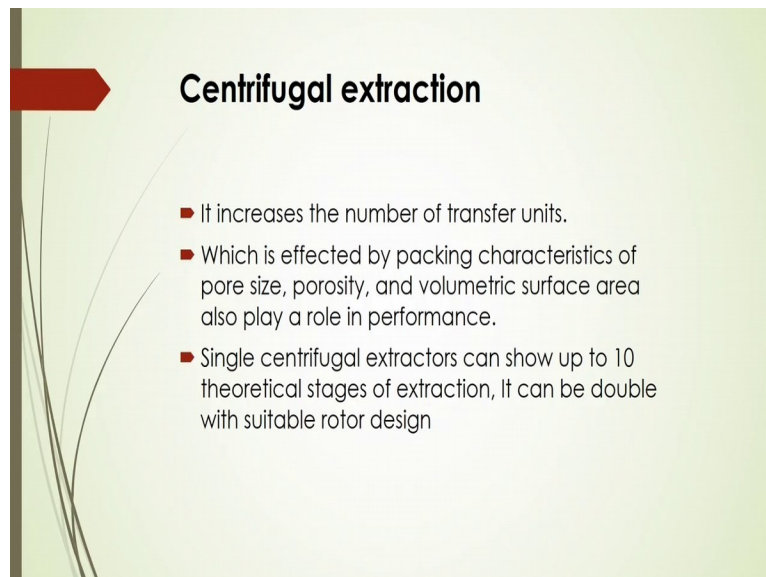


Centrifugal extraction

- Liquid-liquid mass transfer in centrifugal extraction contactors improves at higher rotor speeds, higher solvent ratio, and higher phase density difference.
- The light-phase outlet pressure controls the liquid holdup of both phases

Another important aspect of process intensification of extraction it is called centrifugal extraction. In that case liquid-liquid mass transfer in centrifugal extraction contactors improves at higher rotor speeds, higher solvent ratio and higher phase density difference. The light-phase outlet pressure controls the liquid hold-up of both phases there.

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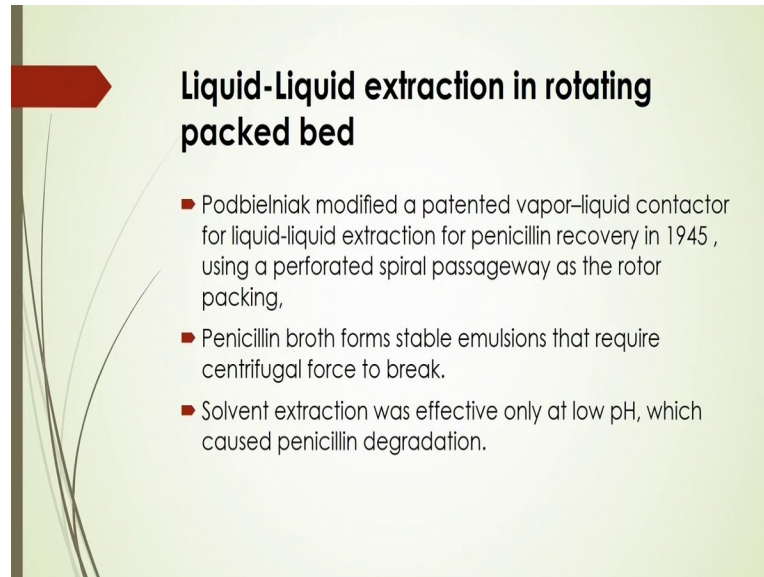
Centrifugal extraction

- It increases the number of transfer units.
- Which is effected by packing characteristics of pore size, porosity, and volumetric surface area also play a role in performance.
- Single centrifugal extractors can show up to 10 theoretical stages of extraction, It can be double with suitable rotor design

And it increases the number of transfer units, also it is actually effected by packing characteristics of pore size, porosity and volumetric surface area and also the single centrifugal extractors can show up to 10 theoretical stages of extraction and it may be that double with suitable rotor design. So if you suitably design the rotor, you can increase the extraction efficiency and also that efficiency of that extraction in the centrifugal extraction

system can be increased also by suitably design your packing characteristics like pore size, porosity and also if you can increase that volumetric surface area that may also effect on extraction performance.

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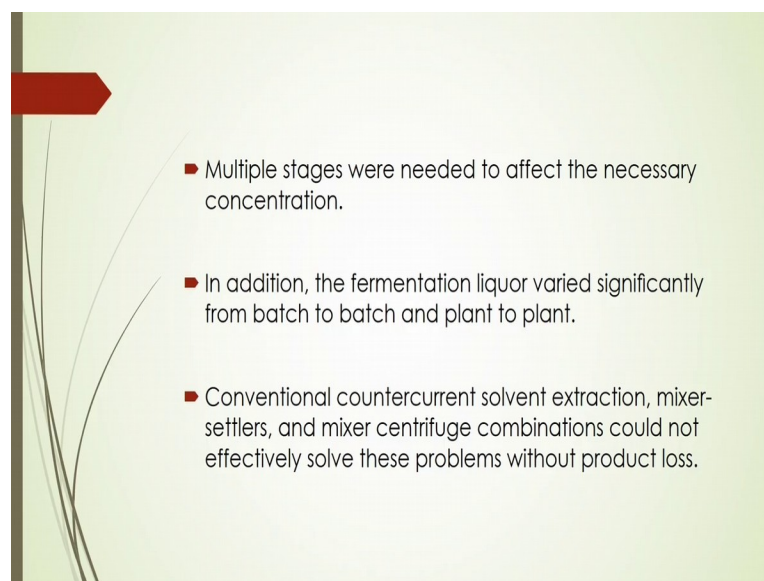


Liquid-Liquid extraction in rotating packed bed

- Podbielniak modified a patented vapor-liquid contactor for liquid-liquid extraction for penicillin recovery in 1945 , using a perforated spiral passageway as the rotor packing,
- Penicillin broth forms stable emulsions that require centrifugal force to break.
- Solvent extraction was effective only at low pH, which caused penicillin degradation.

And rotating packed bed is also another important you know that extraction process for intensification that is continuous processes, in that case Podbielniak you know that modified a patented vapour-liquid contactor for liquid-liquid extraction for penicillin recovery in 1945 using a perforated spiral passageway as the rotor packing. In that case solvent extraction was effective only at low pH, which cost penicillin degradation.

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- Multiple stages were needed to affect the necessary concentration.
- In addition, the fermentation liquor varied significantly from batch to batch and plant to plant.
- Conventional countercurrent solvent extraction, mixer-settlers, and mixer centrifuge combinations could not effectively solve these problems without product loss.

And multiple stages were needed to affect that necessary concentration and in addition, the fermentation liquor varied significantly from batch to batch and also plant to plant. So conventional countercurrent solvent extraction, mixer settlers, mixer centrifuge combinations could not effectively solve these problems without product loss.

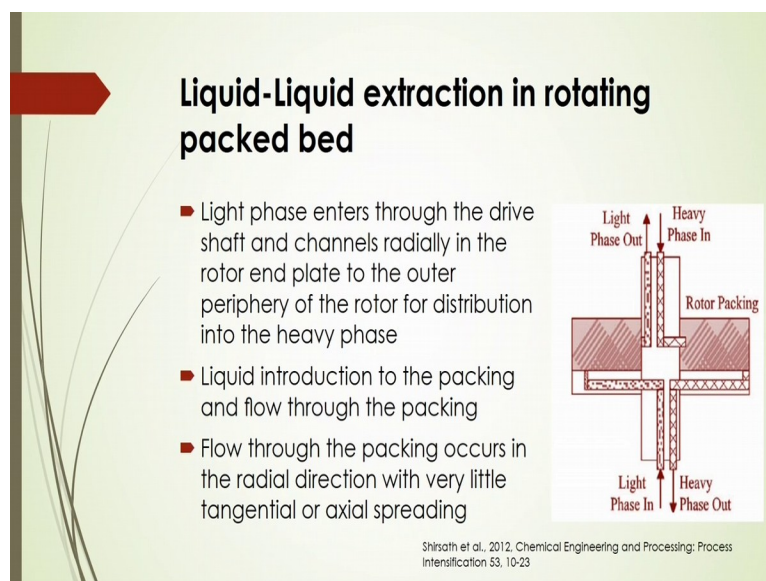
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- The centrifugal solvent extractor achieved 98% product recovery by taking advantage of its low liquid holdup, short residence time, high centrifugal force, and multistage countercurrent contacting.
- The use of centrifugal fields for liquid-liquid extraction was perhaps the first commercially successful application of rotating packed beds.

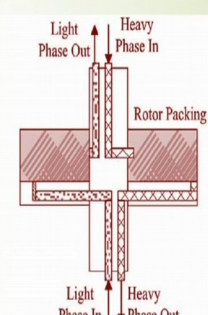
That is why that intensification of this extraction process are actually considered for the research and in that case some idea of that centrifugal fields to use for liquid-liquid extraction and in that case for the continuous operation the application of rotating packed beds were suggested that way.

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Liquid-Liquid extraction in rotating packed bed

- Light phase enters through the drive shaft and channels radially in the rotor end plate to the outer periphery of the rotor for distribution into the heavy phase
- Liquid introduction to the packing and flow through the packing
- Flow through the packing occurs in the radial direction with very little tangential or axial spreading

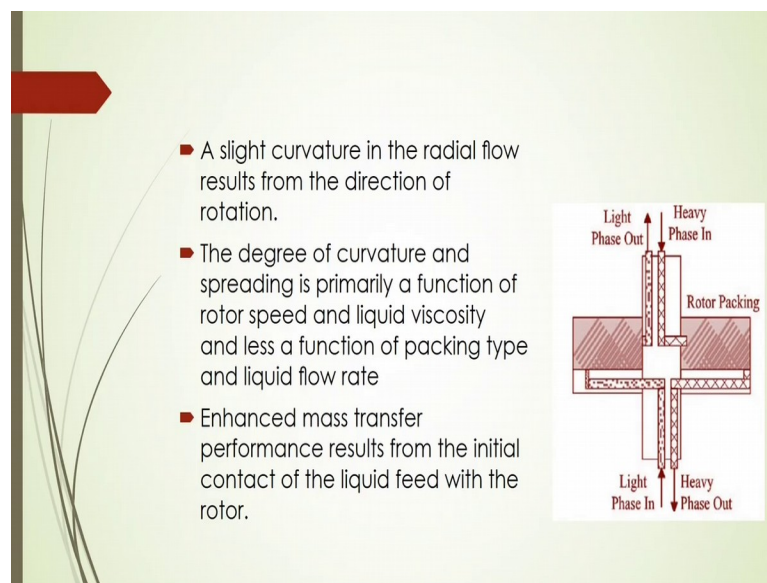


The diagram illustrates the internal structure of a rotating packed bed. It shows a central drive shaft with a rotor end plate. The rotor is filled with packing material. The flow paths are labeled: 'Light Phase In' enters from the bottom, 'Heavy Phase In' enters from the top, 'Light Phase Out' exits from the top, and 'Heavy Phase Out' exits from the bottom. The rotor packing is shown as a central column with radial channels.

Shirsath et al., 2012, Chemical Engineering and Processing: Process Intensification 53, 10-23

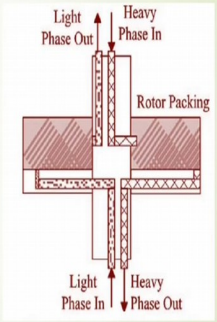
And in that case you know see this figure some rotating packed bed for that liquid-liquid extraction, in this case you will see that as per figure shown here according to Shirsath et al. 2012 that light phase enters through the drive shaft and channels radially in the rotor and you know that plate to the outer periphery of the rotor for distribution into the heavy phase. In this case liquid is being introduced to the packing and flow through the packing there. Flow through the packing occurs in the radial direction with very little tangential or axial spreading. So based on this you know that you know centrifugal action that extraction is being intensified.

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Also the degree of you know that mass transfer depends on that initial contract of the liquid field with the rotor. Also the properties of the fluid is one important aspects, in that case you know that liquid viscosity and also that liquid flow rate actually play an important role for that increase the efficiency of the centrifugal based extraction in the rotary packed column.

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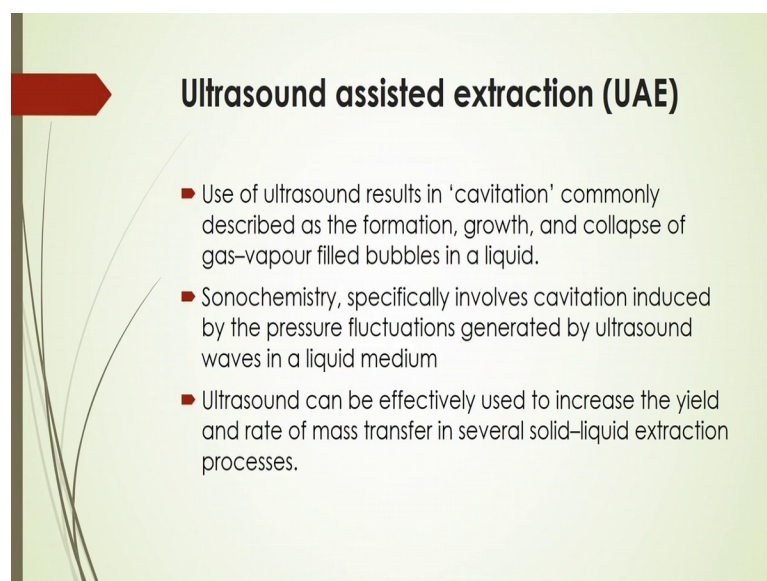


The diagram illustrates a rotor-packing extraction column. It shows a central vertical shaft with a rotor inside. The rotor is surrounded by packing material. The column is divided into two sections. The top section is labeled 'Light Phase Out' and 'Heavy Phase In'. The bottom section is labeled 'Light Phase In' and 'Heavy Phase Out'. The rotor is labeled 'Rotor Packing'.

- A breakup of the liquid feed into smaller droplets that filled the void spaces of the packing.
- This effectively increases the interfacial surface area of the liquid beyond that of the surface area of the packing.
- The degree of mass transfer enhancement at the inlet is a function of the type of packing (porosity, shape of packing structure, etc.), rotor speed, method of liquid distribution, and liquid properties

And whenever centrifugal force is acting in the packing system during its rotation, you will see there will be a breakup of the liquid feed into the smaller droplets that filled the void spaces of the packing. And this effectively increases the interfacial surface area of the liquid beyond that of the surface area of the packing and the degree of mass transfer enhanced at that inlet is a function of the type of packing. In that case porosity, shape of packing structure, rotor speed, method of liquid distribution and liquid properties to be considered to get that degree of mass transfer one step ahead.

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The diagram illustrates the process of ultrasound assisted extraction (UAE). It shows a liquid medium being subjected to ultrasound waves, which cause cavitation. The cavitation results in the formation, growth, and collapse of gas-vapour filled bubbles in the liquid. This process is used to increase the yield and rate of mass transfer in solid-liquid extraction processes.

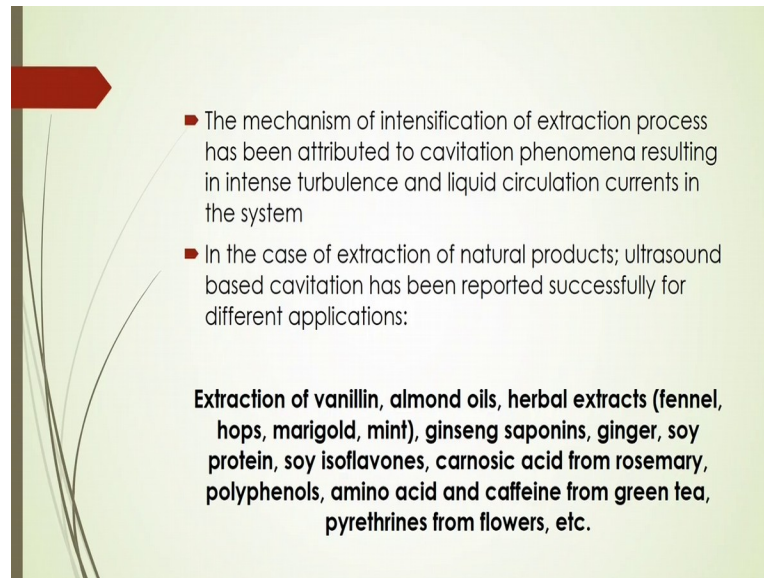
Ultrasound assisted extraction (UAE)

- Use of ultrasound results in 'cavitation' commonly described as the formation, growth, and collapse of gas-vapour filled bubbles in a liquid.
- Sonochemistry, specifically involves cavitation induced by the pressure fluctuations generated by ultrasound waves in a liquid medium
- Ultrasound can be effectively used to increase the yield and rate of mass transfer in several solid-liquid extraction processes.

Another important aspect of process intensification for the extraction is ultrasound-based extraction and use of ultrasound results in generally cavitation which is described earlier also

that cavitation how actually that interfacial area can be produced just by forming and collapsing of that two-faced systems in terms of bubbles or liquid. In that case this ultrasound involves cavitation induced by the pressure fluctuations that is generated by ultrasound waves in a liquid medium and ultrasound can be effectively used to increase that yield and rate of mass transfer in several solid-liquid extraction processes.

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- The mechanism of intensification of extraction process has been attributed to cavitation phenomena resulting in intense turbulence and liquid circulation currents in the system
- In the case of extraction of natural products; ultrasound based cavitation has been reported successfully for different applications:

Extraction of vanillin, almond oils, herbal extracts (fennel, hops, marigold, mint), ginseng saponins, ginger, soy protein, soy isoflavones, carnosic acid from rosemary, polyphenols, amino acid and caffeine from green tea, pyrethrines from flowers, etc.

And the mechanism of intensification of extraction process, in this case basically attributed to cavitation phenomena that results in intense turbulence and liquid circulation currents in the system based on which you can intensify the distribution of the energy to produce more finer droplet through the packing material as well as you know that other systems also you can apply but based on this cavitation process by ultrasound you can use without packing I think more you know intensely because if you are using packing materials there may be some other fictional assistance will come because of which there may be some extra energy to be required to produce that droplet.

But without packing that by this ultrasound you can easily produce that cavitation which will give you that more interfacial area and also just by you know that changing that operating condition that you can produce more finer droplet for the liquid-liquid extractions.

In this case extraction of natural products like ultrasound-based cavitation has been reported successfully for different applications like extraction of vanillin, almond oils, herbal extracts, even you can show that ginseng saponins, ginger, soy protein you know, soy isoflavones and also you know that carnosic acid from rosemary, polyphenols, amino acid and caffeine from

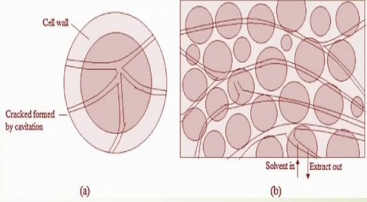
green tea even you can say that pyrethrines from you know flowers. So these are some examples of extraction of these compounds from the natural resources based on this ultrasound based extraction.

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Mechanism of UAE

- Intensification of extraction efficacy is due to the propagation of ultrasound pressure waves through the solvent and resulting cavitation phenomena.
- The controlling mechanism of ultrasound-assisted extraction is generally attributed to
 - mechanical,
 - cavitation, and
 - thermal effects

These can result in disruption of cell walls, particle size reduction, and enhanced mass transfer across cell membranes.

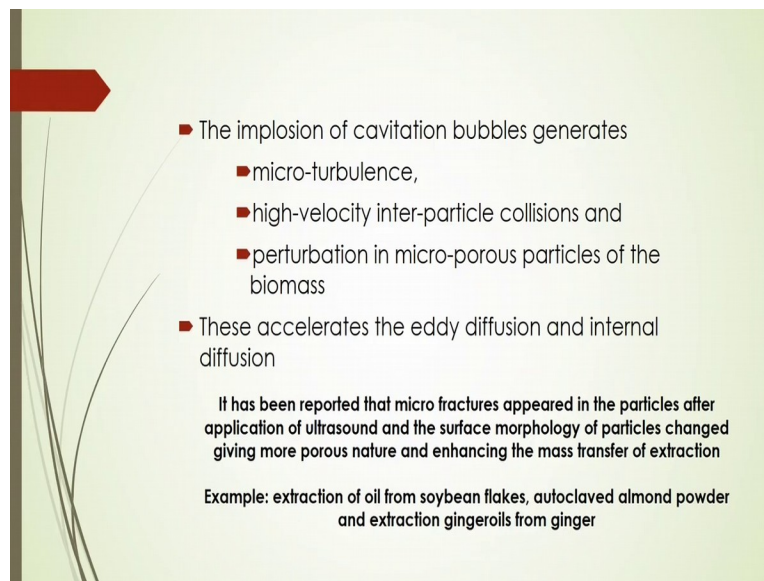


(a) breaking of cell wall due to cavitation. (b) diffusion of solvent into the cell structure.

Shirsath et al., 2012, Chemical Engineering and Processing: Process Intensification 53, 10-23

What is the mechanism of this ultrasound assisted extraction? The intensification of that extraction efficiency is due to the propagation of ultrasound pressure waves through the solvent and resulting that cavitation phenomena and the controlling mechanism of ultrasound assisted extraction is a generally attributed to mechanical, cavitation and also thermal effects there. So in this figure you will see that how this mechanical, cavitation and thermal effects can result in disruption of cell walls, particle size reduction and enhance the mass transfer across the cell membrane.

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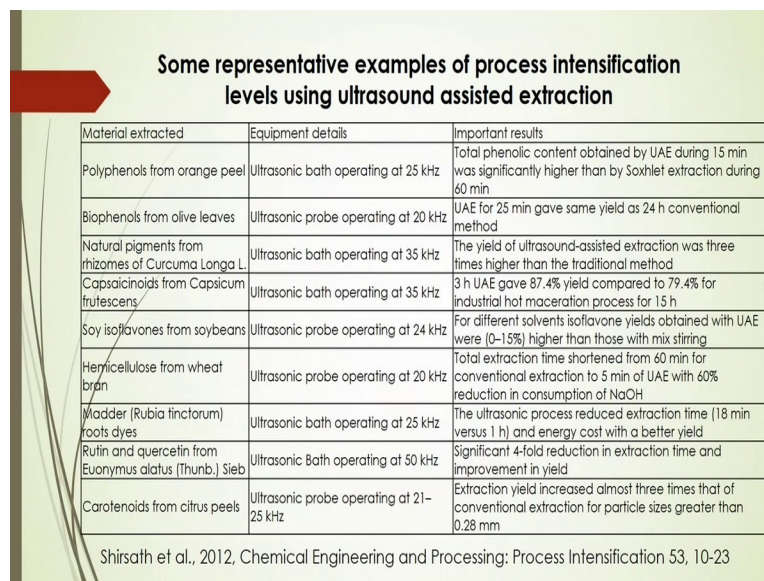
- The implosion of cavitation bubbles generates
 - micro-turbulence,
 - high-velocity inter-particle collisions and
 - perturbation in micro-porous particles of the biomass
- These accelerates the eddy diffusion and internal diffusion

It has been reported that micro fractures appeared in the particles after application of ultrasound and the surface morphology of particles changed giving more porous nature and enhancing the mass transfer of extraction

Example: extraction of oil from soybean flakes, autoclaved almond powder and extraction gingeroils from ginger

Also the implosion of cavitation bubbles generates micro-turbulence, high velocity inter-particle collisions and perturbation in micro-porous particle of the biomass. This accelerates the eddy diffusion and internal diffusion. Now it is reported that micro-fractures appeared in the particles after application of ultrasound and the surface morphology of particles that change giving more porous nature and enhancing the mass transfer for the extraction. Like extraction of oil from soybean flakes, autoclaved almond powder and extraction ginger oils from ginger.

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Some representative examples of process intensification levels using ultrasound assisted extraction

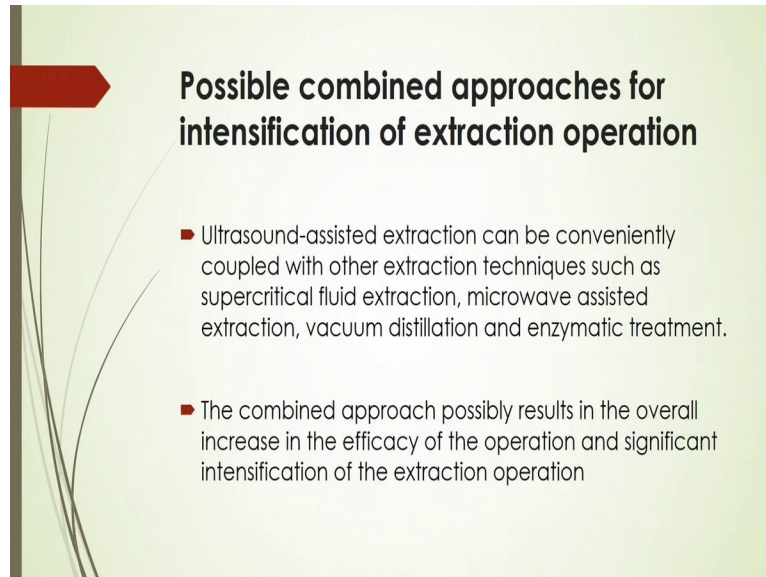
Material extracted	Equipment details	Important results
Polyphenols from orange peel	Ultrasonic bath operating at 25 kHz	Total phenolic content obtained by UAE during 15 min was significantly higher than by Soxhlet extraction during 60 min
Biophenols from olive leaves	Ultrasonic probe operating at 20 kHz	UAE for 25 min gave same yield as 24 h conventional method
Natural pigments from rhizomes of <i>Curcuma Longa</i> L.	Ultrasonic bath operating at 35 kHz	The yield of ultrasound-assisted extraction was three times higher than the traditional method
Capsaicinoids from <i>Capsicum frutescens</i>	Ultrasonic bath operating at 35 kHz	3 h UAE gave 87.4% yield compared to 79.4% for industrial hot maceration process for 15 h
Soy isoflavones from soybeans	Ultrasonic probe operating at 24 kHz	For different solvents isoflavone yields obtained with UAE were (0–15%) higher than those with mix stirring
Hemicellulose from wheat bran	Ultrasonic probe operating at 20 kHz	Total extraction time shortened from 60 min for conventional extraction to 5 min of UAE with 60% reduction in consumption of NaOH
Woader (<i>Rubia tinctorum</i>) roots dyes	Ultrasonic bath operating at 25 kHz	The ultrasonic process reduced extraction time (18 min versus 1 h) and energy cost with a better yield
Rutin and quercetin from <i>Euonymus alatus</i> (Thunb.) Sieb	Ultrasonic Bath operating at 50 kHz	Significant 4-fold reduction in extraction time and improvement in yield
Carotenoids from citrus peels	Ultrasonic probe operating at 21–25 kHz	Extraction yield increased almost three times that of conventional extraction for particle sizes greater than 0.28 mm

Shirsath et al., 2012, Chemical Engineering and Processing: Process Intensification 53, 10-23

Some representative example of process intensification levels using ultrasound assisted extraction given in this slides, please go through the slides, you can have that different

examples of that extraction based on this ultrasound assisted extraction process and also equipment details for that production of that ultrasonic web at a different you know that frequency and also what are the different important results that obtained, it is reported by Shirsath et al. in 2012.

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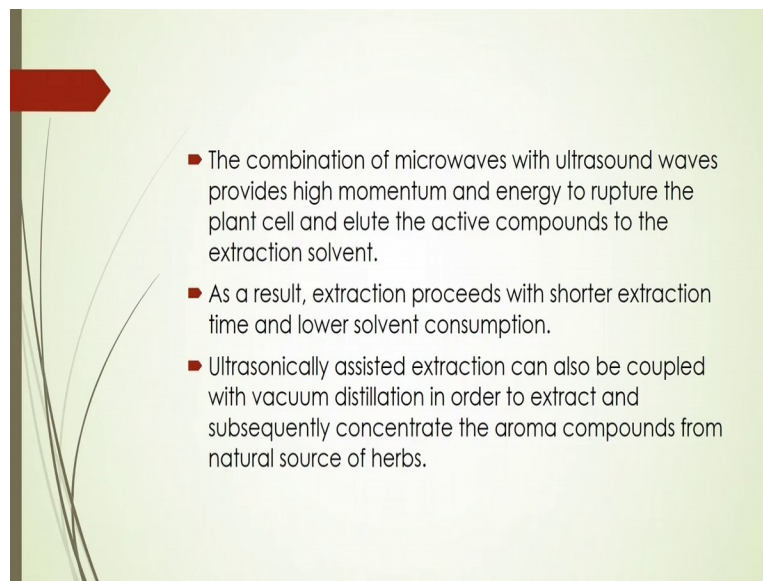


Possible combined approaches for intensification of extraction operation

- Ultrasound-assisted extraction can be conveniently coupled with other extraction techniques such as supercritical fluid extraction, microwave assisted extraction, vacuum distillation and enzymatic treatment.
- The combined approach possibly results in the overall increase in the efficacy of the operation and significant intensification of the extraction operation

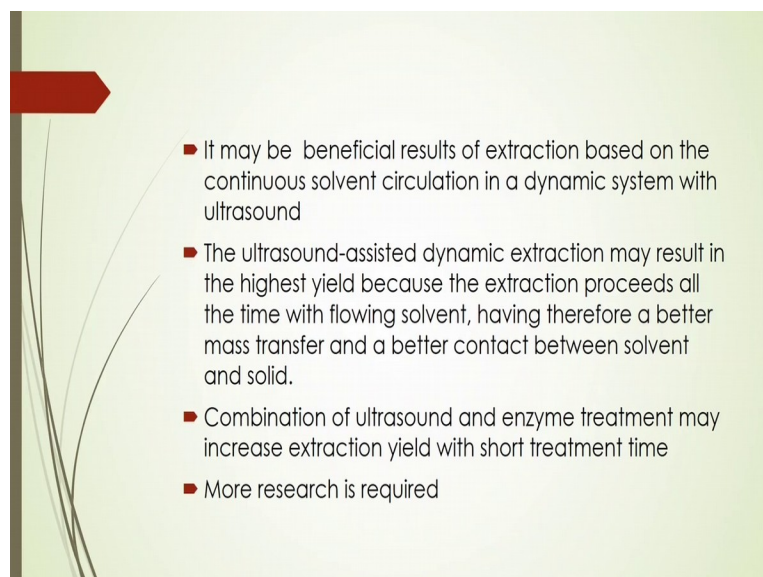
And possible combined approaches for the intensification of extraction operations are like ultrasound assisted extraction can be conveniently coupled with other extraction techniques such as supercritical fluid extraction, microwave assisted extraction, vacuum distillation and also enzymatic treatment. And the combined approach may results in the overall increase the efficacy of the operation and also you can say significant intensification of the extraction operation there.

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And this combination of these microwaves with ultrasound waves sometimes provides high momentum and energy to rupture the plant cell and elute the active compounds to the extraction solvent and as a result that extraction proceeds with that shorter extraction times and also lower solvent consumption. That is why intensification are there, so ultrasonically assisted extraction can also be coupled with vacuum distillation in order to extract and subsequently concentrate the you know that aroma compounds from natural sources of herbs.

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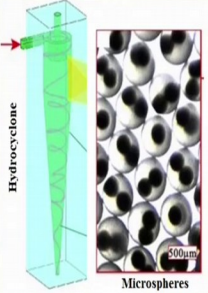


So you can combine that ultrasound assisted you know that process with other mechanism to get that more intensification of the extraction process.

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Intensification by micro-droplet rotation in a hydrocyclone

- A hydrocyclone as an extractor intensifies the mass transfer and largely reduces the consumption of extractant from 1800–2000 kg h⁻¹ to 30–90 kg h⁻¹. (Report by Huang et al., 2017)
- A high-speed rotation of dispersed micro-spheres caused by the anisotropic swirling shear flow
- Due to the conical structure of a hydrocyclone, the rotation speed maintains stability along the axial direction.



Huang et al., 2017, Scientific Reports, volume 7, Article number: 2678 (2017)

Now intensification can also be possible by micro-droplet rotation in a hydro-cyclone system, that hydro-cyclone as an extractor that intensify the mass transfer and largely reduces the consumption of extractant from 1800 to 2000 kg per hour to 30 to 90 kg per hour, that is reported by Huang et al. 2017. In this case a high-speed rotation of dispersed micro-spheres caused by anisotropic swirling shear flow and due to the conical structure of the hydrocyclone, the rotating speed maintains stability along the axial direction there.

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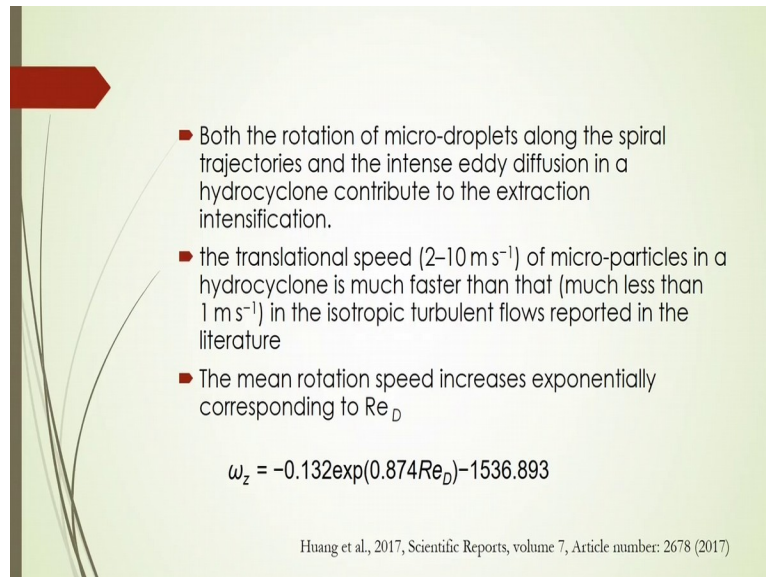
- The hydrocyclone has a slender structure that consists of a cylinder and a cone.
- The micro-spheres are injected tangentially into the swirling field and translate following the outer spiral before finally exiting from the underflow orifice.
- The rotation of spheres caused by the tangential velocity gradient.

Huang et al., 2017, Scientific Reports, volume 7, Article number: 2678 (2017)

And this method has you know that slender structure that consist of cylinder and a cone and these micro-spheres are injected tangentially into the swirling field and translate following

the outer spiral before finally exiting from the underflow orifice, so the rotation of that sphere caused by the tangential velocity gradient.

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- Both the rotation of micro-droplets along the spiral trajectories and the intense eddy diffusion in a hydrocyclone contribute to the extraction intensification.
- the translational speed ($2\text{--}10\text{ m s}^{-1}$) of micro-particles in a hydrocyclone is much faster than that (much less than 1 m s^{-1}) in the isotropic turbulent flows reported in the literature
- The mean rotation speed increases exponentially corresponding to Re_D

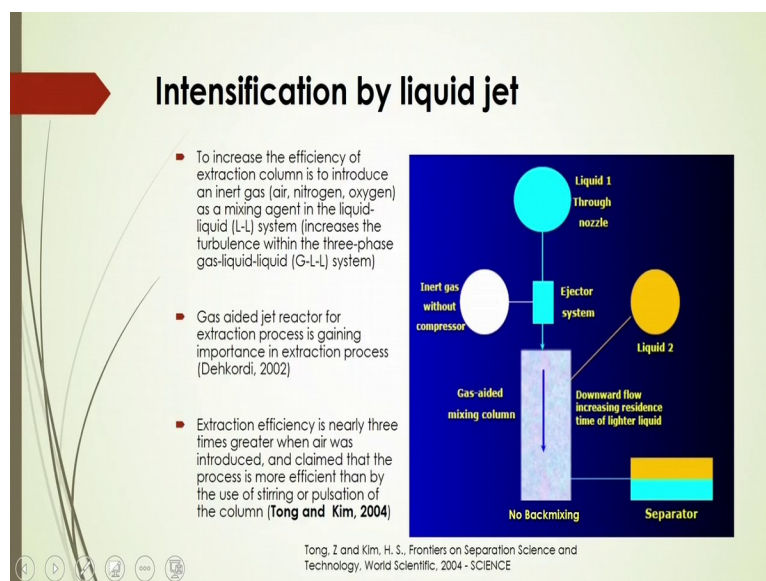
$$\omega_z = -0.132\exp(0.874Re_D) - 1536.893$$

Huang et al., 2017, Scientific Reports, volume 7, Article number: 2678 (2017)

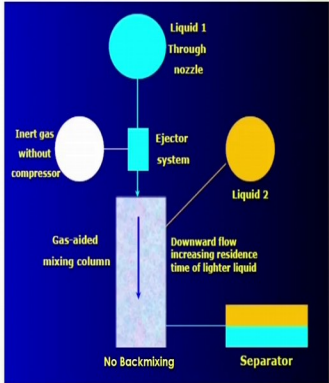
And because of which you can get that more you know micro-droplets along the spiral trajectories and based on which you can get that mass transfer there and the mean rotation speeds are basically corresponds to that you know that pattern of the fluids. In that case it is called you know identified by that Reynolds number. If you know that Reynolds number, then what should be the rotation speed to be followed that you can easily calculate from this correlation that is given in this slides and it is suggested by Huang et al., 2017.

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Intensification by liquid jet



- To increase the efficiency of extraction column is to introduce an inert gas (air, nitrogen, oxygen) as a mixing agent in the liquid-liquid (L-L) system (increases the turbulence within the three-phase gas-liquid-liquid (G-L-L) system)
- Gas aided jet reactor for extraction process is gaining importance in extraction process (Dehkordi, 2002)
- Extraction efficiency is nearly three times greater when air was introduced, and claimed that the process is more efficient than by the use of stirring or pulsation of the column (Tong and Kim, 2004)

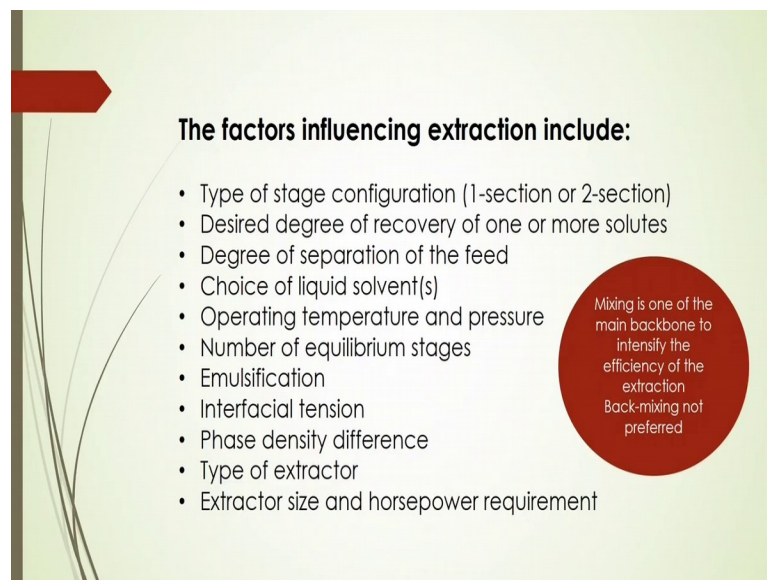


Tong, Z and Kim, H. S., Frontiers on Separation Science and Technology, World Scientific, 2004 - SCIENCE

Another important intensification of the extraction it is called that liquid jet based extraction process. In this case the liquid jet whenever you know plunging into the pool of the liquid, you will see that that too immiscible liquids whenever plunging into a pool of the liquid by the liquid jet, you can get more finer droplet and getting more you know that more finer droplet getting more interfacial area. Also sometimes gas aided liquid jet system, extraction system also are being nowadays developed for getting more mass transfer there. So it is called gas aided mixing column where gas liquid systems will be there where liquid jet along with the gas will be plunging into the other liquid pool.

So that final liquid droplet of immiscible lighter liquid will be formed and parallelly gas also will be supplied to get that more turbulence there and getting more mixing and based on which you can get that more interfacial area, more hold-up of the pages and get more mass transfer there. So this extraction efficiency is nearly in this case 3 times greater than air if you introduced it and that the process is more efficient than by the use of stirring or pulsation of the column there. So this is one of the important aspects of process intensification for the extraction.

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The factors influencing extraction include:

- Type of stage configuration (1-section or 2-section)
- Desired degree of recovery of one or more solutes
- Degree of separation of the feed
- Choice of liquid solvent(s)
- Operating temperature and pressure
- Number of equilibrium stages
- Emulsification
- Interfacial tension
- Phase density difference
- Type of extractor
- Extractor size and horsepower requirement

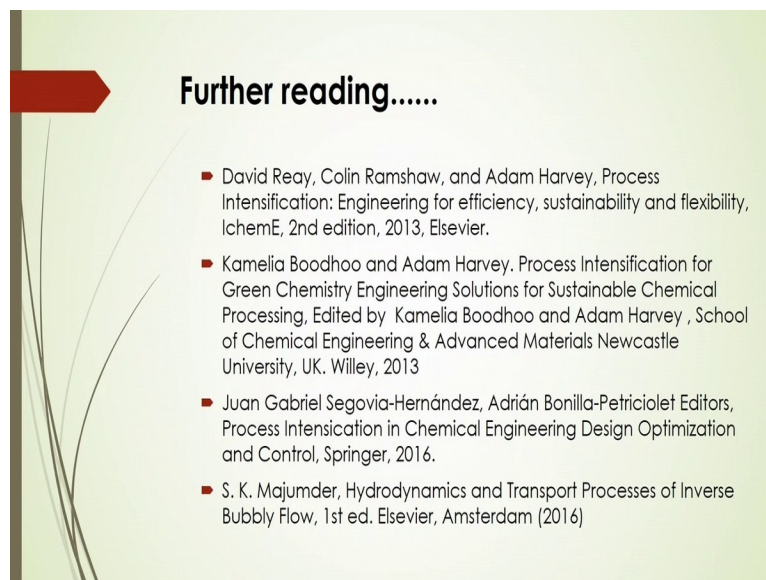
Mixing is one of the main backbone to intensify the efficiency of the extraction. Back-mixing not preferred

Now what are the factors that influence that extraction, maybe you know that different processes have different process conditions, the process conditions is one of the important factors that effect on the mass transfer and even some other factors like you know that if you are configured the system stage-wise then type of the stage configuration.

Even if you know that different parameters to be considered to get the desired degree of recovery of one or more solutes, in that case these are the influencing factor, degrees of separation of feed also another important factor. Choice of liquid solvents and also operating temperature and pressure. Sometimes you know that what type of extractor you will use that is also important for influencing that extraction process. You know extractor size and also power requirement that also is important factor and how many number of equilibrium stage to be actually procured for that extraction process.

Even what is the interfacial tension, whether you can increase the interfacial tension and based on that interfacial tension whether you can get that more mass transfer or not, that is also another aspect to consider for the influencing of extraction there. So mixing is one of the main backbone to intensify the efficiency of the extraction. Sometimes you know that backmixing also will not be preferred, so it will be better to you know that without having backmixing how liquid and liquid even gas liquid solid how they can get contact to get more retention time and also more mass transfer there. So these are the several influencing factors for the extraction processes.

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Further reading.....

- David Reay, Colin Ramshaw, and Adam Harvey, *Process Intensification: Engineering for efficiency, sustainability and flexibility*, IChemE, 2nd edition, 2013, Elsevier.
- Kamelia Boodhoo and Adam Harvey, *Process Intensification for Green Chemistry Engineering Solutions for Sustainable Chemical Processing*, Edited by Kamelia Boodhoo and Adam Harvey, School of Chemical Engineering & Advanced Materials Newcastle University, UK. Wiley, 2013
- Juan Gabriel Segovia-Hernández, Adrián Bonilla-Petriciolet Editors, *Process Intensification in Chemical Engineering Design Optimization and Control*, Springer, 2016.
- S. K. Majumder, *Hydrodynamics and Transport Processes of Inverse Bubbly Flow*, 1st ed. Elsevier, Amsterdam (2016)



So we have discussed a lot of things about the process intensification of extraction based on ultrasound assisted, based on jet systems, based on you know that other you know rotary packed bed systems, centrifugal system. Even electro-hydrodynamic systems, how those are being actually used for intensification of the extraction there and also supercritical extraction is one of the important aspects of that intensification of the extraction process. We will discuss more about that supercritical extraction process in the next lecture, so I would suggest you to follow these references to get more information about this intensification of the extraction. So thank you for your attention.