Food Oils and Fats: Chemistry & Technology

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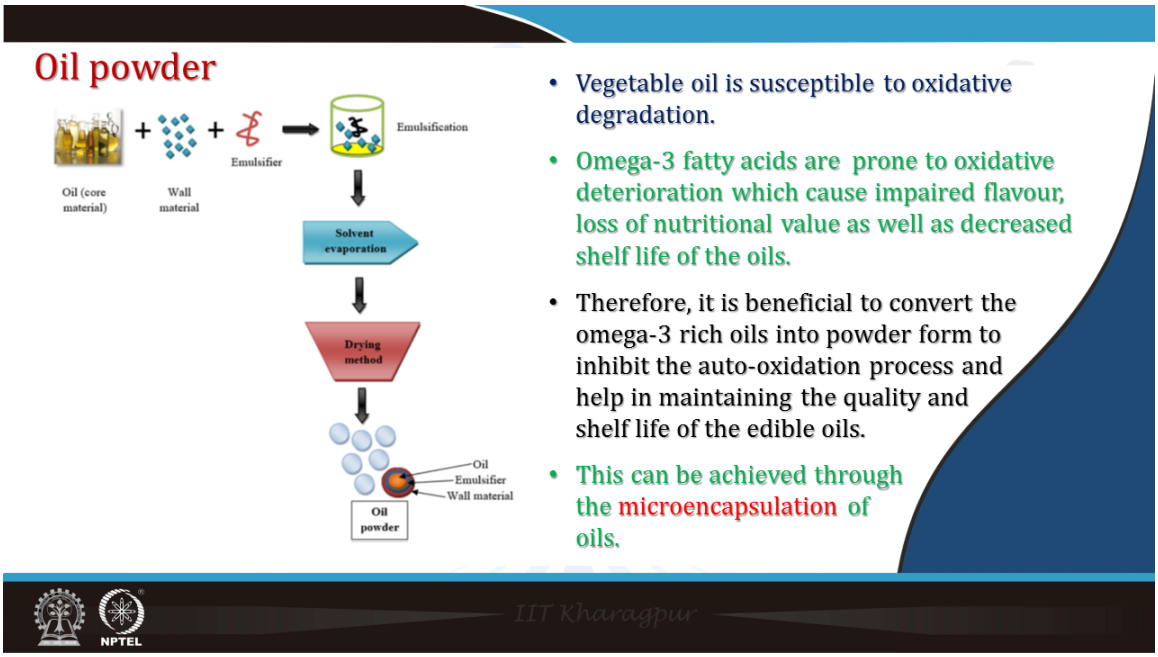
Module 10: Specialty Oils and Fats Products

Lecture 50 : Oil Powder and Liposomes

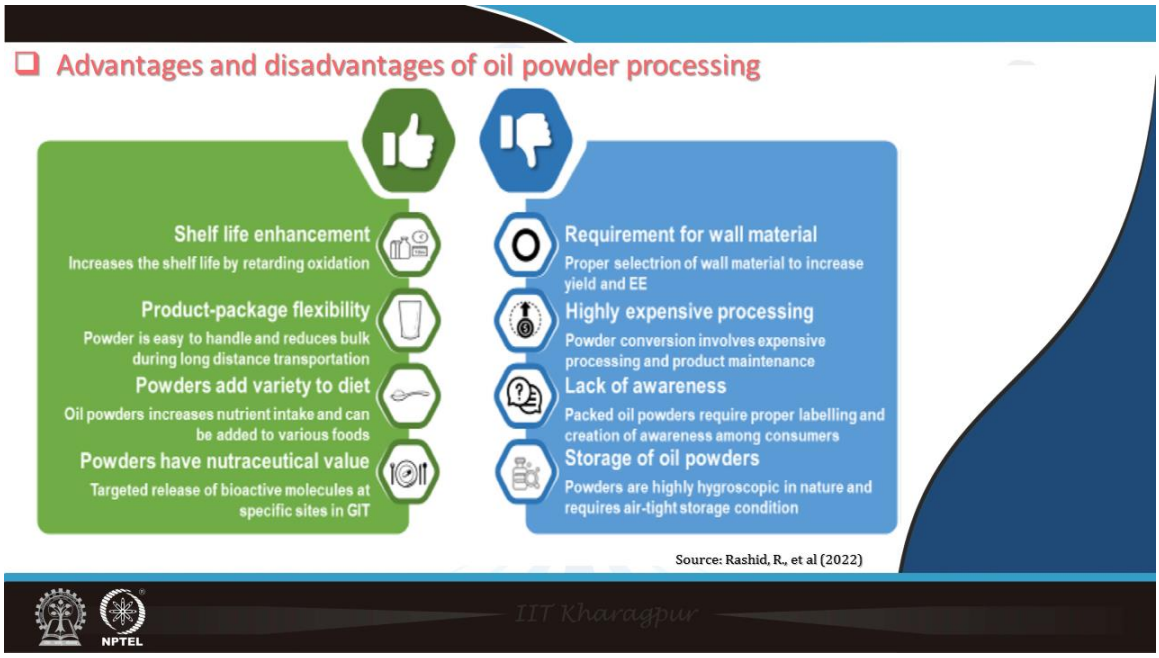
Hello everybody. Namaskar. We are now in the last lecture of module 10. In this lecture 50th, we will talk about, in the next half an hour or so, about oil powder and liposomes.

We will discuss: what is an oil powder, how oil powder can be made, what are the various techniques including encapsulated techniques, then characterization and application of oil powder. And then we also will talk about liposomes, its classification and method of preparation.

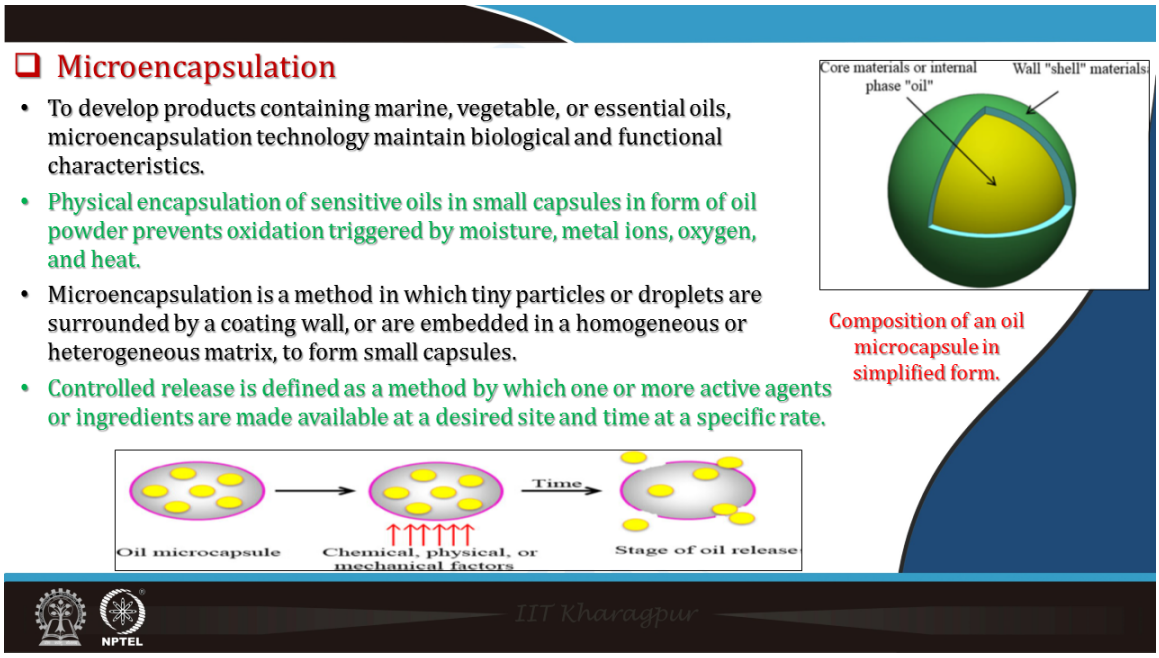
And then finally, we have in our lab work done and we have a patented technology on heart healthy blended oil powder and liposomes. So, we will discuss a brief of that as well.



So, the oil powder from the name itself indicates, that is, you know that vegetable oil is susceptible to oxidative degradation. Omega 3 fatty acids are prone to oxidative deterioration which cause impaired flavor. It may result into loss of nutritional value as well as decreased shelf life of the oil. Therefore, it is beneficial to convert these highly unstable omega 3 rich oils into more stable powder form. And this inhibits the auto oxidation process and helps in maintaining the quality as well as shelf life of the edible oils. And this can be done by a technique called microencapsulation of oil. We microencapsulate like the oil which is taken a consider as a core material and this is some wall material is used and from emulsifiers are there, then emulsion is prepared and then this emulsion is finally dried using suitable technology and it gives a some sort of coating is applied appeared on the top.



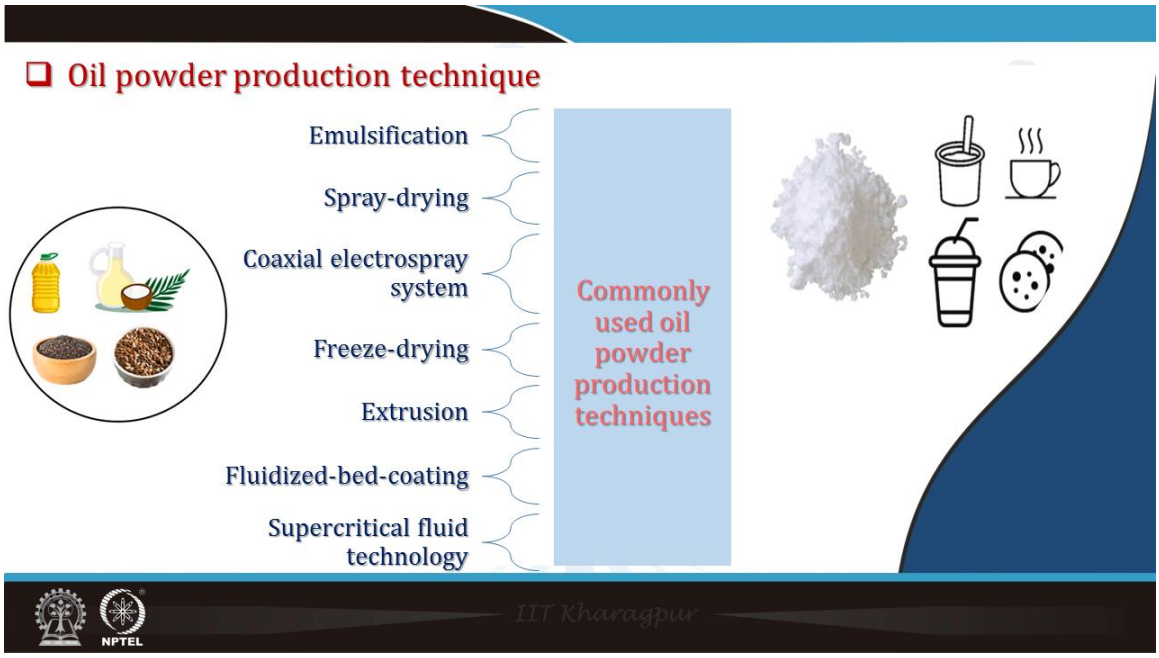
I will come little later again in this, but first advantages and disadvantages of the oil powder processing, that is, it is a, it gives a shelf life enhancement, it, that is the product package flexibility, it reduces the handling in energy or cost required for handling transportation etcetera ok. Powders add variety to diet, the powders have more nutraceutical values ok. They can be even targeted release a targeted delivery of the various bioactive etcetera can be used with this microencapsulation technology. However, there is the some of the disadvantages might include requirement of the specific wall material and this wall material may interfere with the general characteristic flavor etcetera of the compound. They are highly expensive processing required even not much people are aware about this technology or consumers etcetera. And then storage of oil powder, that is, because the powders are highly hygroscopic in nature. So, they require air tight specific storage material and storage conditions etcetera.



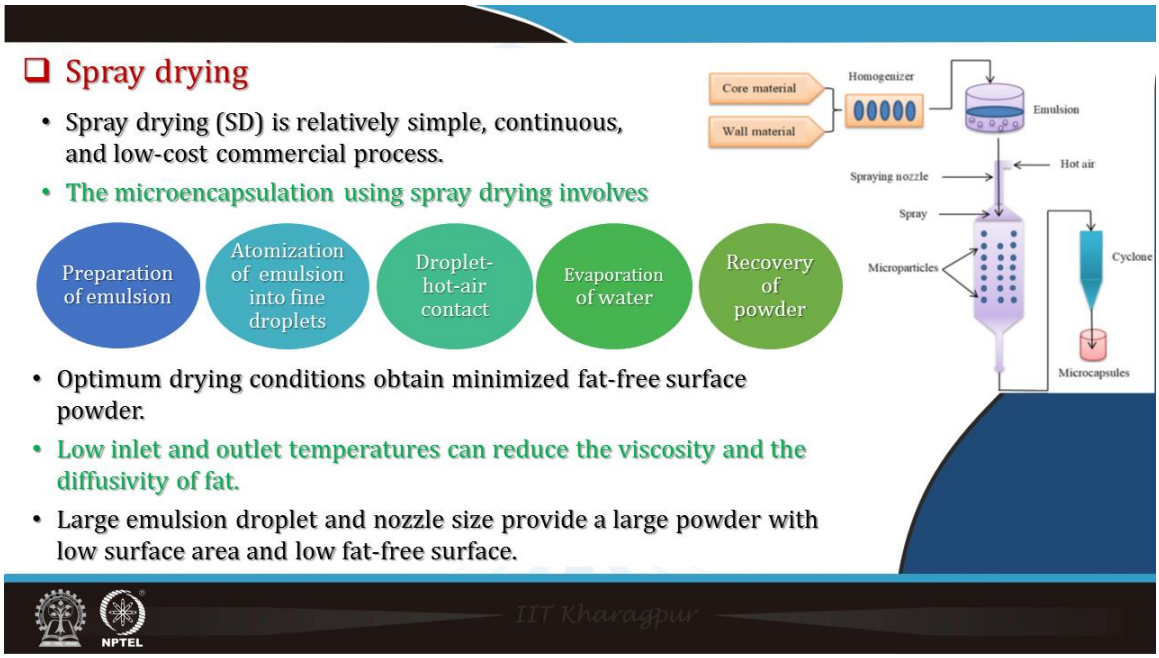
Now let us talk about microencapsulation technology. You can see here it is a micro capsulation that is where the active ingredient core material and it is include some site of the wall material or cell material that is this ok. And using a proper characteristics of these materials that is taken that is just a core ingredient is encapsulated is surrounded by thick wall material.

So, it is microencapsulation technology is used to develop products containing marine, vegetable or essential oils in microencapsulation technology. It maintains biological and functional characteristics. Physical encapsulation of sensitive oils in a small capsule in the form of oil powders prevents oxidation triggered by moisture, metal ions, oxygen, heat, etcetera. So, as I told you microencapsulation is method in which tiny particles or droplets are surrounded by a coating or they are embedded in a homogeneous or heterogeneous matrix to form a small capsules. And these the wall material is selected in such a way that even they can be engineered to degrade and therefore, targeted release of these bio-octaves can be achieved.

So, oil material here you see then chemical, physical or mechanical factors are required which will break this coating and then this it will take time and then the oil materials are released. And in fact, this microencapsulation technology is also used for various applications like targeted nutrition delivery and so many things ok.



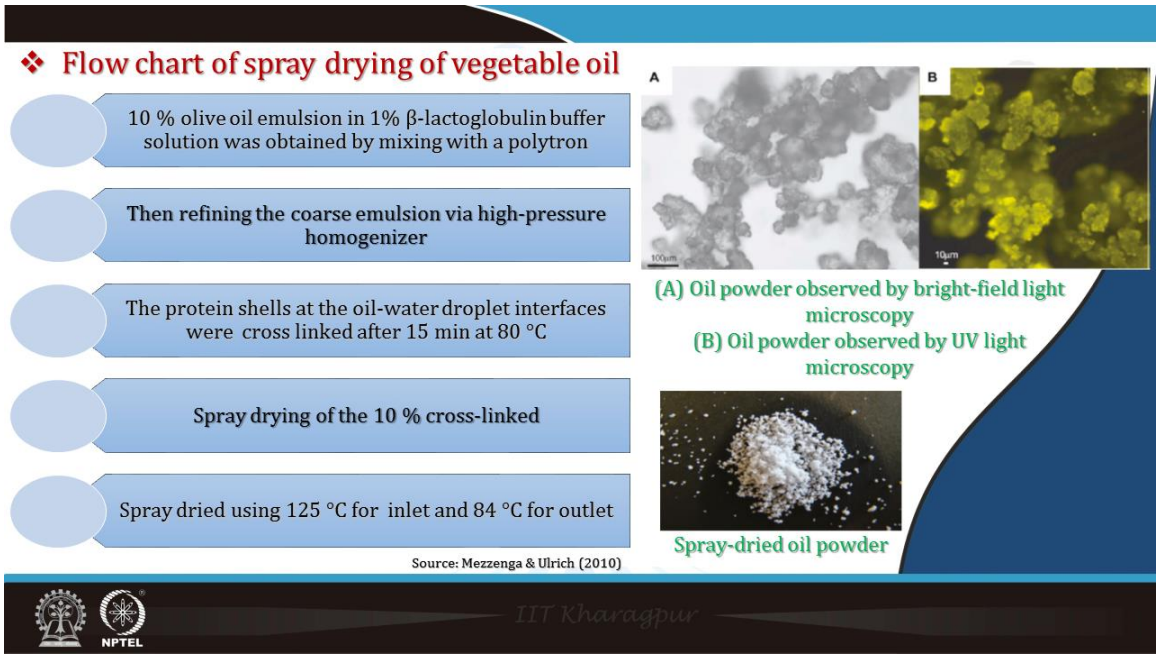
Then these oil powder production technologies; commonly used methods include emulsification, spray drying, coaxial electro spray system, freeze drying, extrusion, fluidized bed coating, and supercritical fluid technology ok.



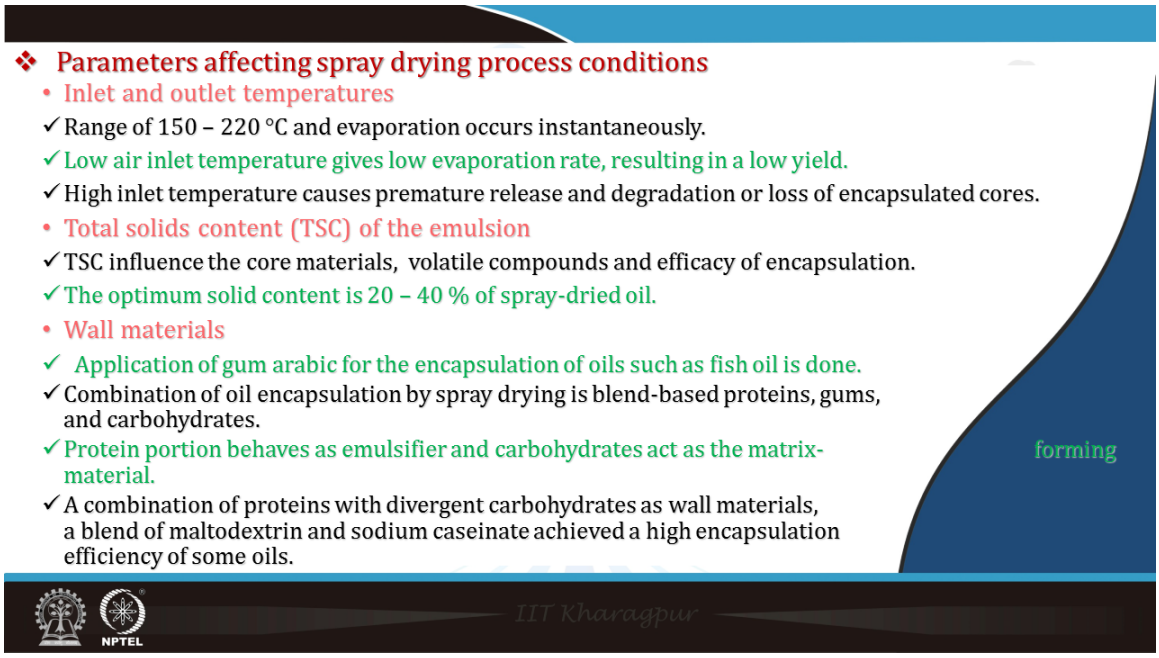
The spray drying, then the spray drying is relatively simple, it is continuous and low cost commercial process. The microencapsulation using spray drying involves first as I showed you in the earlier slide that is the preparation of suitable emulsion that is the

and wall material and core material are there, they are some homogenized, mixed properly and emulsion is formed.

And then this emulsion is a sprayed using fine spray nozzles etcetera into fine droplets, ok. There is a and it comes that is these emulsion comes in the fine droplets in the drying chamber where it comes in contact with hot air, ok. Then from this drying chamber, the water is evaporated and we get, that is, using cyclone, air is removed and micro capsule that is oil powder is recovered. So, optimum drying conditions to obtain the minimized fat free surface powder there and low inlet and outlet temperatures can reduce the viscosity and the diffusivity of fat. Large emulsion droplets and nozzle size provide a large powder with low surface area and low fat free surface, ok.



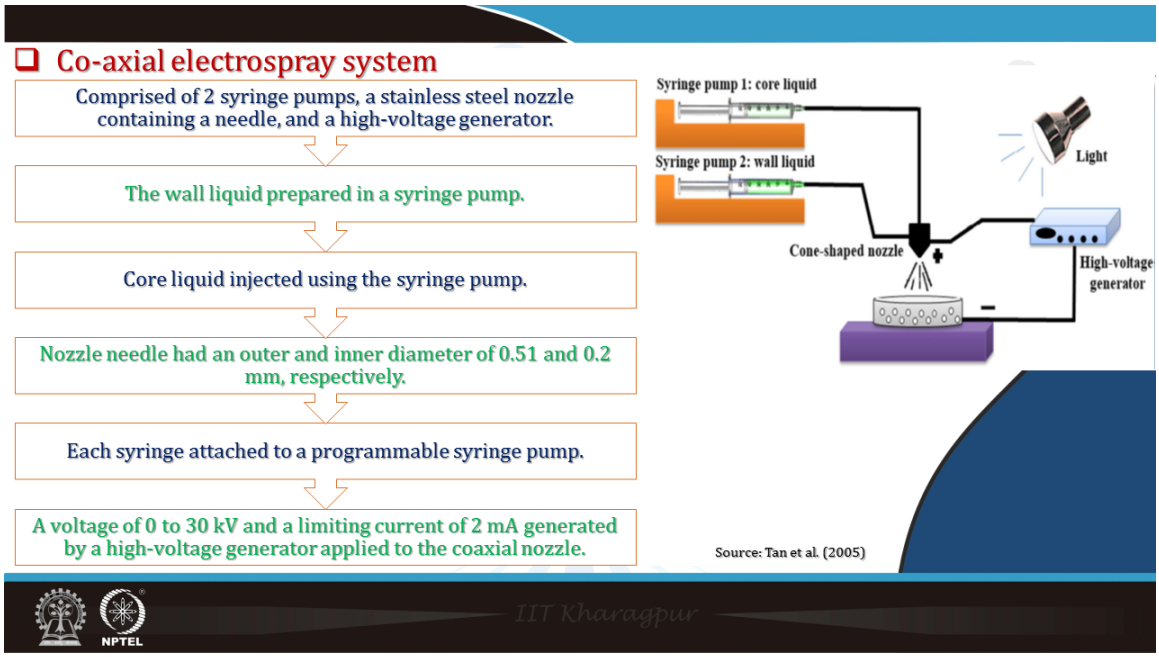
So, the process flow chart for the spray drying of vegetable powder particularly olive oil emulsion is taken 10 percent and 1 percent beta-lactoglobulin buffer solution is obtained by mixing with a polytron. And then the refining of the coarse emulsion by a high pressure homogenizer were done and the protein shells and the oil water droplets interfaces were cross linked for 15 minute at 80 degrees Celsius. And then finally, spray drying of the 10 percent cross linked material and it is spray drying conditions were 125 degree Celsius inlet temperature in the drying chamber and 84 degree Celsius in the outlet temperature.



And you can see that this is the oil powder which is as observed by bright field light microscopy and the other one is the oil powder observed by UV light microscopy and this is the picture of spray dried oil powder you can see ok. Emulsion parameters which influence the drying process conditions: number one: inlet and outlet temperatures in the spray drying chamber.

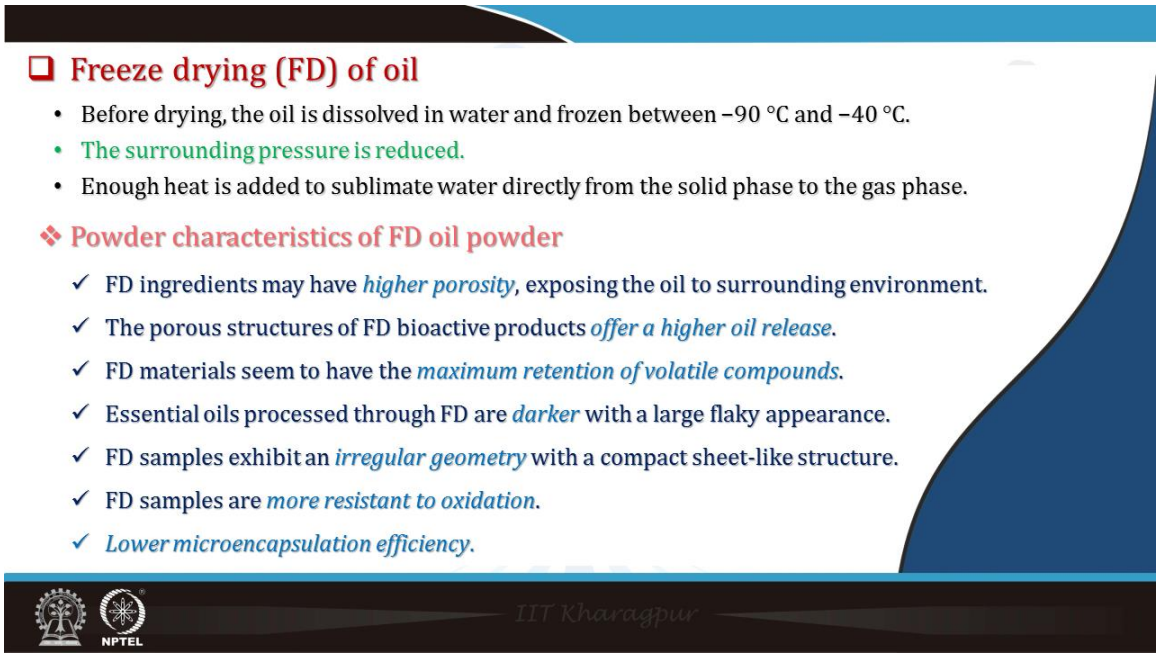
The ranges are 150 to 200 degree Celsius and evaporation occurs simultaneously. Lower inlet temperature gives low evaporation rate resulting in a obviously low yield. High air inlet temperature causes premature release and degradation or loss of encapsulated cores. Total solids content that TSC of the emulsion is, it is TSC, also influences the core material volatile compounds and efficacy of the microencapsulation. The optimum solid content is around 20 to 40 percent of the spray dried oil. The wall material type of the material that is used like application of gum Arabic for the encapsulation of oil is where has been popular ok particularly for the fish oil etcetera.

Then combination of encapsulation by spray drying is blend-based protein, gums and carbohydrates. Protein portion behaves as an emulsifier and carbohydrates acts as a matrix material. A combination of protein with different carbohydrate as well as wall materials, a blend of maltodextrin, sodium caseinates, etcetera are achieved a high encapsulation efficiency in some oils.



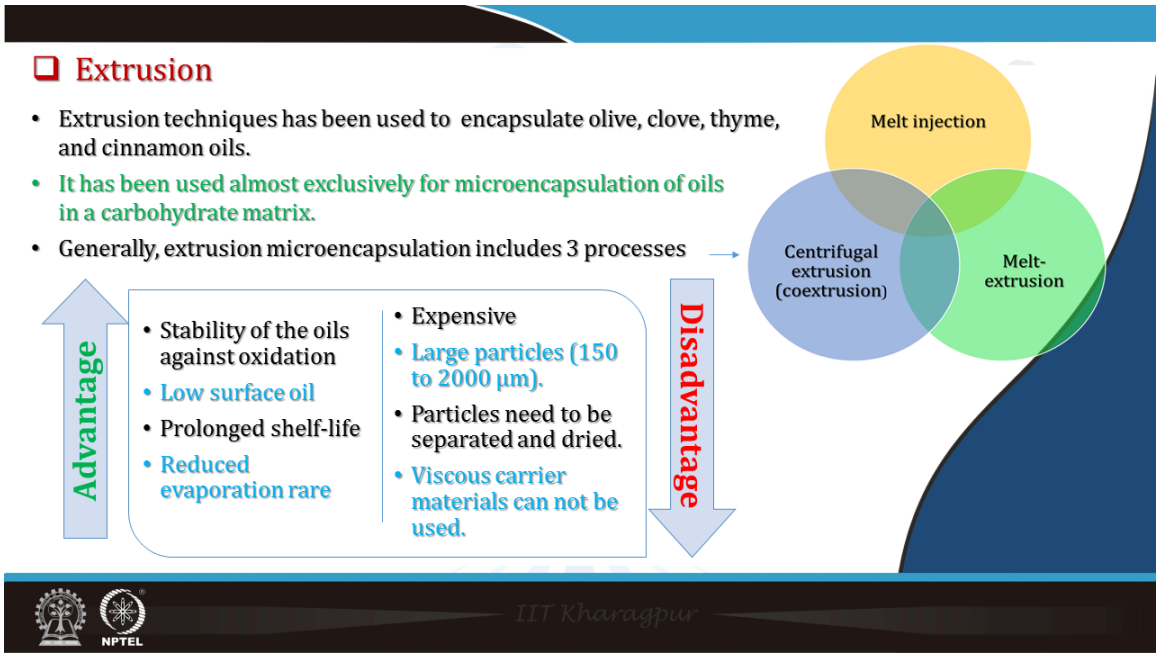
Then another method is the coaxial electrospray system. In this, there are this system consists of two syringe pumps, one is the syringe pump; one for the core liquid and syringe pump two for the wall liquid.

And the wall liquid prepared in a syringe pump; and core liquid is injected using a, this syringe pump. Then nozzle needle, that is nozzle needle, had an outlet and inner diameter of 0.51 to 0.02 mm, respectively; and each syringe attached to a programmable syringe pump. And then with the help of this; these are the voltage of around 0 to 30 kiloVolt and a limiting current of 2 mA generated by a high voltage generator which is applied to a coaxial nozzle and here these materials are put and then sprayed and then the capsule micro capsules are obtained, ok.



Then, freeze drying. The oil, before the drying, the oil is dissolved in water and it frozen at minus 90 degree to minus 40 degree Celsius, surrounding pressure is reduced and even heat is added to sublimate water directly from the solid phase to the gas phase. So, powder characteristics of freeze-dried oil powder include that the freeze dried ingredient may have higher porosity exposing the oil to surrounding environment. The porous structure of freeze dried bioactive products it offers a high oil release. Freeze-dried materials seem to have the maximum retention of volatile compounds. Their essential oils processed through freeze drying are darker with a larger flaky appearance.

Freeze dried samples exhibit an irregular geometry with a compact sheet like structure. Freeze dried samples are more resistant to oxidation and they have lower microencapsulation efficiency.

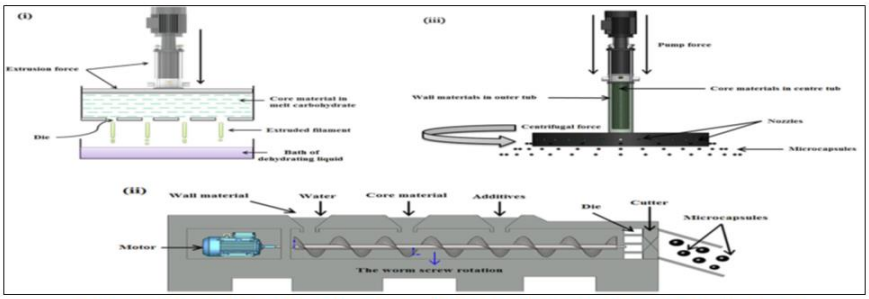


Then extrusion is the, it is a, extrusion technique has been used to encapsulate olive, clove, thyme and cinnamon oils, etcetera. It has been used to almost exclusively for microencapsulation of oils in a carbohydrate matrix. Generally, extrusion microencapsulation involves three processes those are melt injection, centrifugal extraction, that is coextrusion and melt extrusion, these are the three processes, ok.

So, advantages of the extrusion process in microencapsulation include stability of the oil against oxidation, low surface oil, prolonged shelf life and reduced evaporation rate. However, the disadvantages include, that is, the process is sometime expensive. There are large particles: may be 150 to 2000 micron are formed; particles need to be separated and dried and viscous carrier material cannot be used in this case.

❖ Melt extrusion

- The melt-extrusion process is similar to that of melt-injection.
- Adding core material to plasticized carrier matrix at a later stage of extrusion process
- Protects sensitive bioactive (PUFAs) from harsh extrusion conditions.

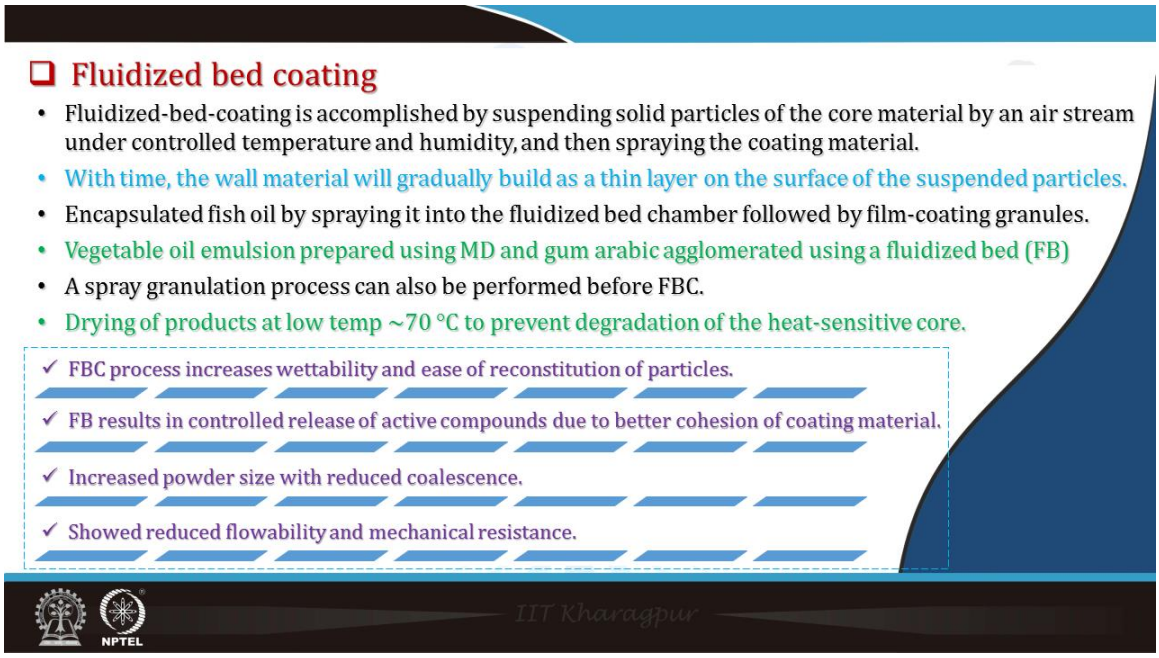


(i) Melt injection, (ii) Melt extrusion, and (iii) Centrifugal (coextrusion) process

Source: Bakry et al. (2016)

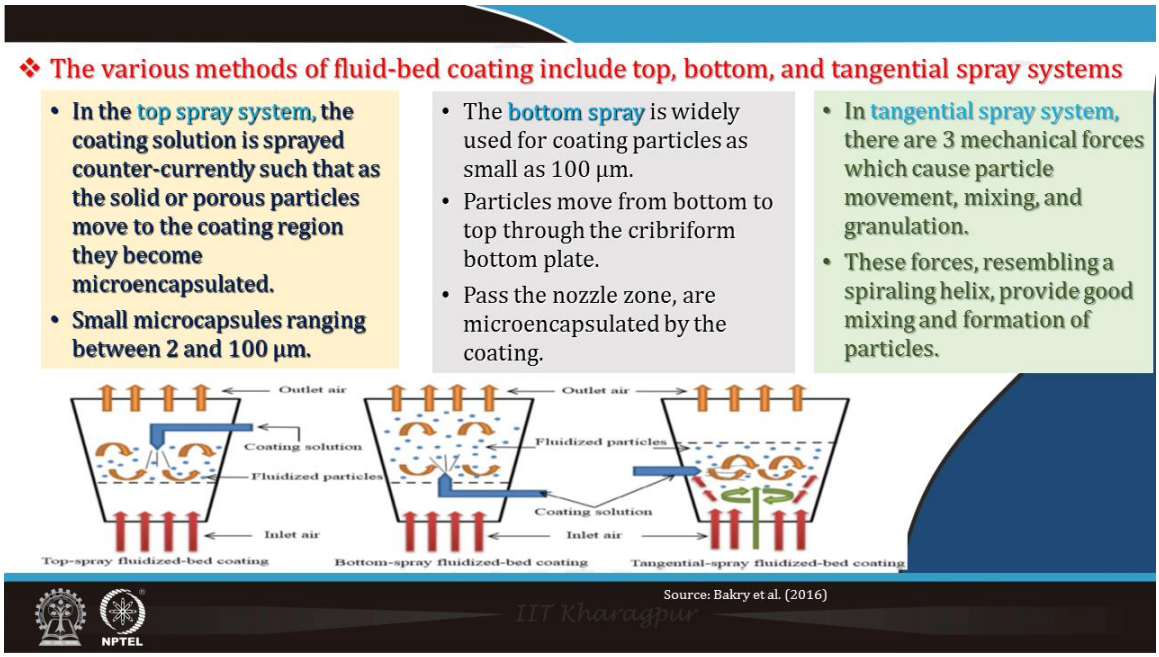
So, the melt extrusion process. As you can see here in the figure 1, that is the, in the melt extrusion process, the core material is dispersed in molten carbohydrate, then pressed through a dye into a bath of isopropanol and liquid nitrogen. The wall material solidifies with liquid forming an encapsulating matrix to interrupt the cored material.

The granules are then recovered by filtration or centrifugation. The residual solvent is removed by air drying or vacuum drying. Melt extrusion process; it is almost similar to that of the melt injection; only thing here, adding the core material to plasticized carrier matrix at a later stage; you can see here, that is, the plasticized carrier matrix and melt core material is added at a later stage in the middle of the extrude almost, ok. The in the later stage in the extrusion process, the process, it protects the sensitive bioactive, etcetera from harsh extrusion conditions, ok. In the co-extrusion process, you can see here it is a concentric feed tube through which wall material and core material are pumped separately to the many nozzle mounted on the outer surface. Core material flows through the center tube, ok. Wall material flows through the outer tube and encapsulation of olive oil has been using alginate microspheres is done using this co extrusion techniques, ok.



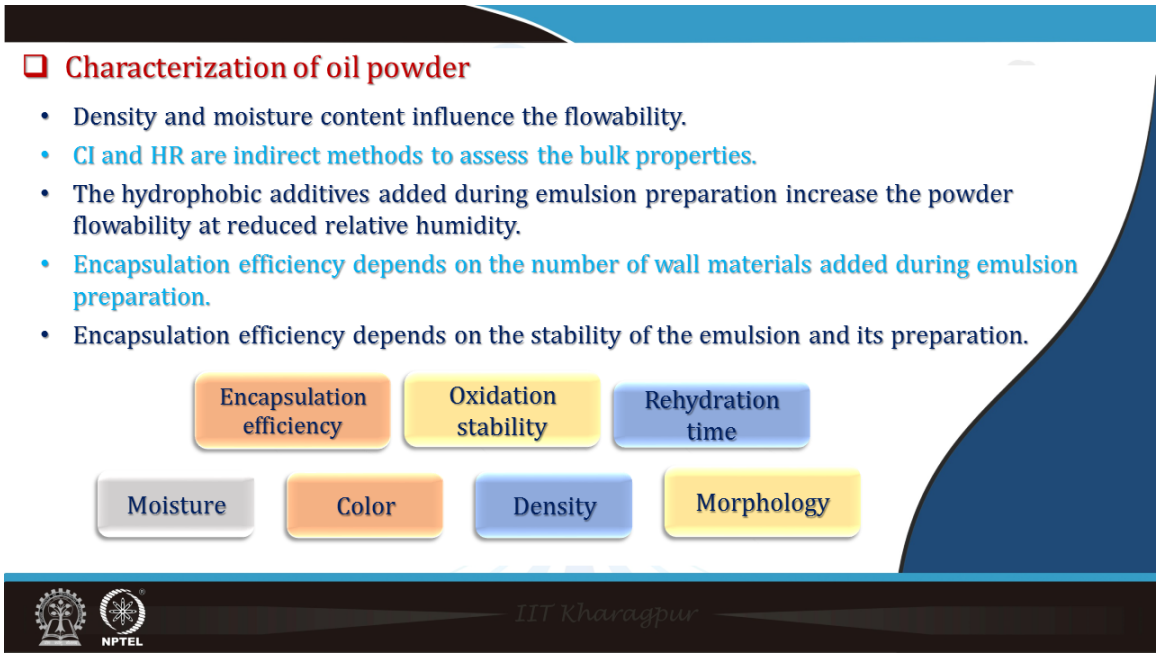
Then fluidized bed coating. It is accomplished by suspending solid particles of the core material by an air stream under controlled temperature and humidity and then spraying the coating material. With time, the wall material will gradually build as a thin layer on the surface of the suspended particles. Encapsulated fish oil by spraying it into the fluidized bed chamber followed by film coating granules are done.

Vegetable oil emulsion prepared by using maltodextrin and gum Arabic agglomerated using fluidized bed drying is done. A spray granulation process can also be performed before fluidized bed coating and drying of the products at a temperature of around 70 degrees Celsius is done to prevent degradation of the heat sensitive core. So, the fluidized bed coating process increases wettability and ease of reconstitution properties. Fluidized bed process results in controlled release of bioactive compounds due to better cohesion of the coating material. It is increased powder size with reduced coalescence is obtained here. It shows reduced flowability and mechanical resistance.



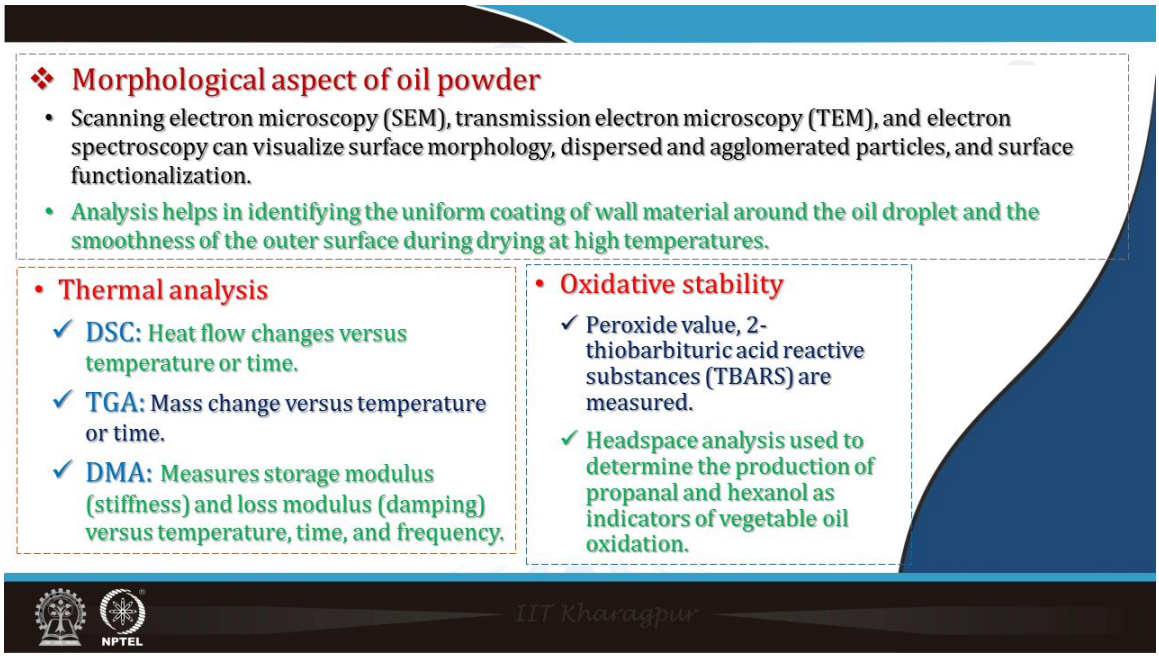
Now, this the various methods of fluid board coating which included were top spray, bottom spray, and tangential spray system. In the top spray system, as you can see in this figure 1, that is the coating solution is sprayed counter currently such that, as the solid or porous particles move to the coating region, they become micro encapsulated. So, a small micro capsule ranging between 2 to 100 micrometer can be formed using this top system. In the bottom spray system, just a reverse of the top system, it is widely used for coating particles as small as 100 micron.

Proximity particles move from bottom to top through the cribriform bottom plate and they pass the nozzle zone and these are micro encapsulated by the coating. In the tangential spray system as you can see in the third figure, there are three mechanical forces which cause particle movement, mixing and granulation. And these forces resembling a spiraling helix provide good mixing and the formation of particles.



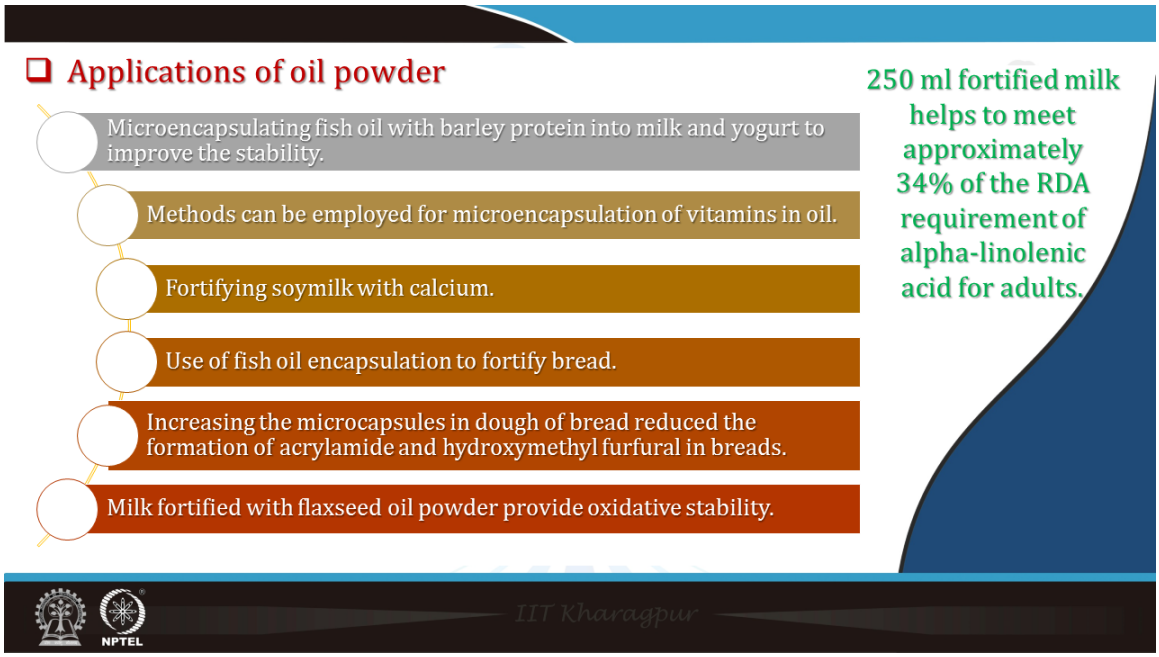
Now the characteristics of the powder. Obviously, the density and moisture content influence the flowability of the characters. So, these become important parameter. Then Carr index and Hansen ratio are indirect method to assess the bulk powder properties particularly free flow, etcetera. The hydrophobic additives added during emulsion preparation increase the powder flowability at reduced relative humidity. Encapsulation efficiency depends on number of wall materials added during emulsion preparation. Encapsulation efficiency depends on the stability of the emulsion and its preparation. The various methods to use to characterize the oil powder include the encapsulation efficiency, oxidation stability, even rehydration time, release time, moisture content, colour, density and its morphology.

And there are set procedures protocols for measuring and finding out these characteristics and so that the powder can be characterized using and then importantly that is these should be characterized to have a more efficient release or target release system whenever it is required.



So, morphological aspect of the oil powder. Scanning electron microscopy, transmission electron microscopy, electron spectroscopy, etcetera can visualize surface morphology, disperse and agglomerated particles and surface functionalization. Even analysis helps in identifying the uniform coating of wall material around the oil droplet and the smoothness of the outer surface during drying at high temperatures. Thermal analysis like DSC, TGA and DMA can be used. DSC uses heat flow changes versus temperature or time. In TGA, it takes a mass change versus temperature or time. And DMA measures the storage modulus like stiffness and loss modules (damping), the oil, versus temperature, time and frequency.

Oxidizing stability like peroxide value, 2 thiobarbituric acid reactive substances, etcetera are measured using standard protocols. Headspace gas analysis is done to determine the production of propanol and hexanol, etcetera which are the indicator of the vegetable oil oxidation. So, these are the set protocol by which the oil powder can be characterized.

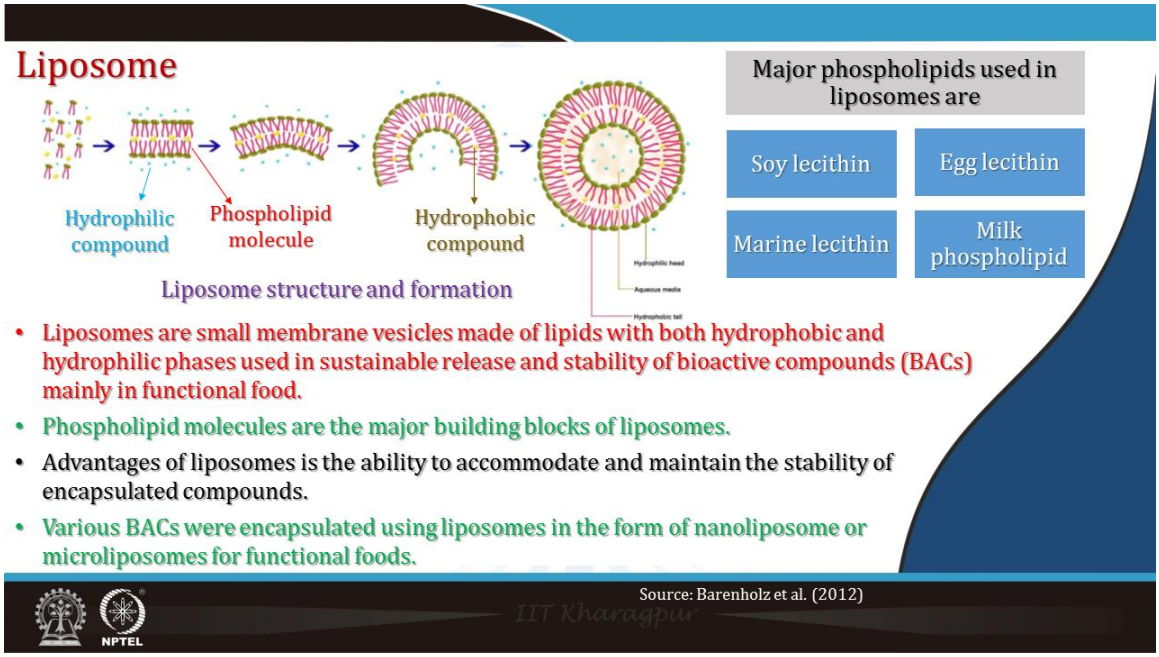


As far as the application of oil powder is concerned, that is, microencapsulating fish oil with a barley protein into milk and yogurt to improve the stability has been used. Methods can be employed for microencapsulation of vitamins in the oil, fortified soya milk with calcium, use of fish oil for encapsulation to fortified bread or even these encapsulation technology has been used to mask the fishy flavor of the fish oil and then these encapsulated fish oil containing high content of the omega fatty acids, they are put into the milk and then omega fatty acid rich milk is using encapsulation technology which is available in the market, even some dairies are doing using this technology. Then increasing the microcapsule in dough of the bread reduce the formation of acrylamide and hydroxymethylfurfural in the breads.

Milk fortified with flax oil powder provide oxidative stability to the system. Even 250 mL fortified milk helps to meet approximately 34 percent of the RDA requirement of alpha linolenic acids for adults using this fortified and encapsulated alpha linolenic acid, encapsulated linseed oil, fish oil etcetera. So, that becomes a very energy to mask flavor or even the core material that is wall material is used in such a way that the bioengineered wall material or nano materials are used, that is, which can be even seen they can get this released as a target site particular site because of the in our digestion system the

conditions is it another things are different. So, the material can be selected in such a way.

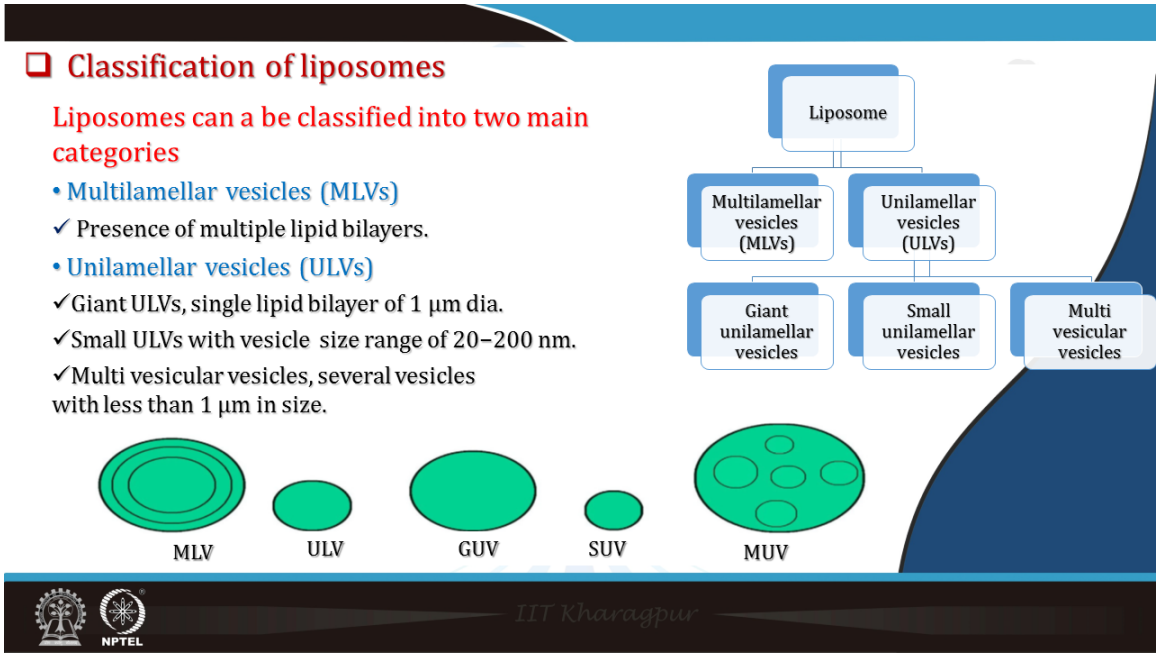
So, that they these micro encapsules at the one time that they provide stability to this material and at the same time they get released they get degraded or disintegrated at a proper location in our system and then we can get targeted release of the bioactives are micronutrient in the desired location.



Then comes the liposomes. These are the liposomes, you know that they are hydrophilic compounds and phospholipids molecule. So, using this is standard control techniques micro capsular these hydrophobic compounds are formed and the layer is hydrophobic both hydrophilic head and aqueous media and hydrophobic tail these are contained inside that is contain liposomes. So, the liposome structure and formation how it is formed that they contain all these hydrophobic hydrophilic and the aqueous media. So, liposomes are a small membrane vessels made of lipids with both hydrophobic and hydrophilic phases used in sustainable release and stability of bioactive compounds mainly in the case of functional foods, etcetera.

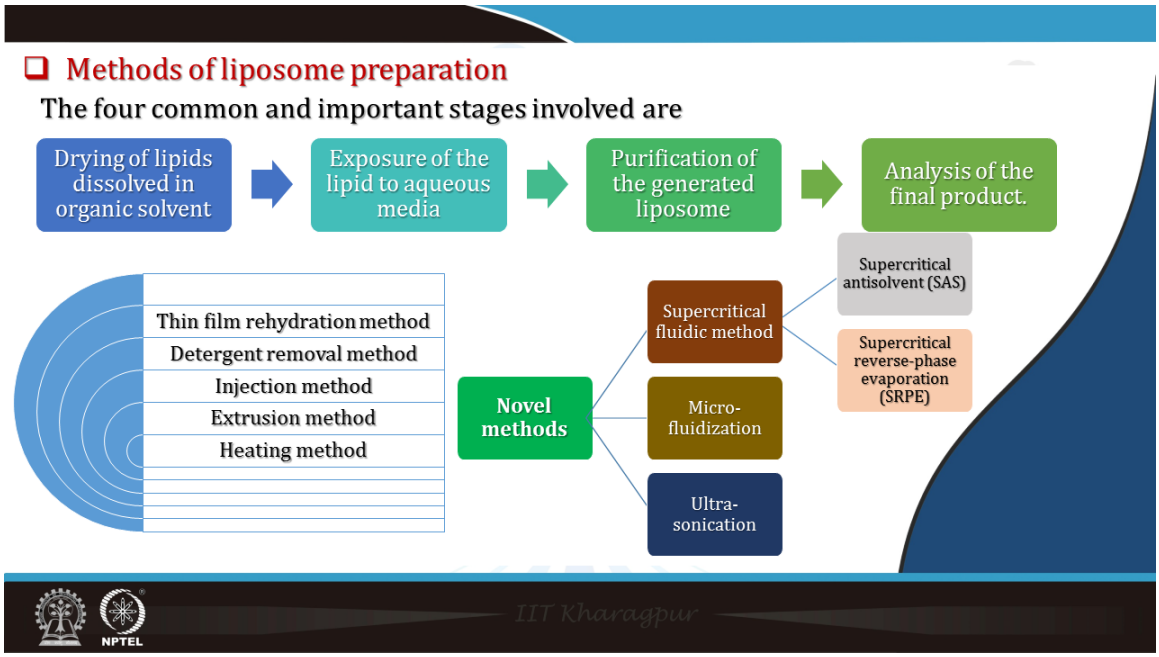
Phospholipid molecule are the major building blocks of liposomes. Advantages of liposomes is the ability to accommodate and maintain the stability of encapsulated bio

compounds. Various bioactive compounds were encapsulated using liposomes in the form of nanoliposomes or micro liposomes or functional foods. So, major phospholipids which are used in the synthesis of or preparation of liposomes, include soy lecithin, egg lecithin, marine lecithin and milk phospholipids ok.



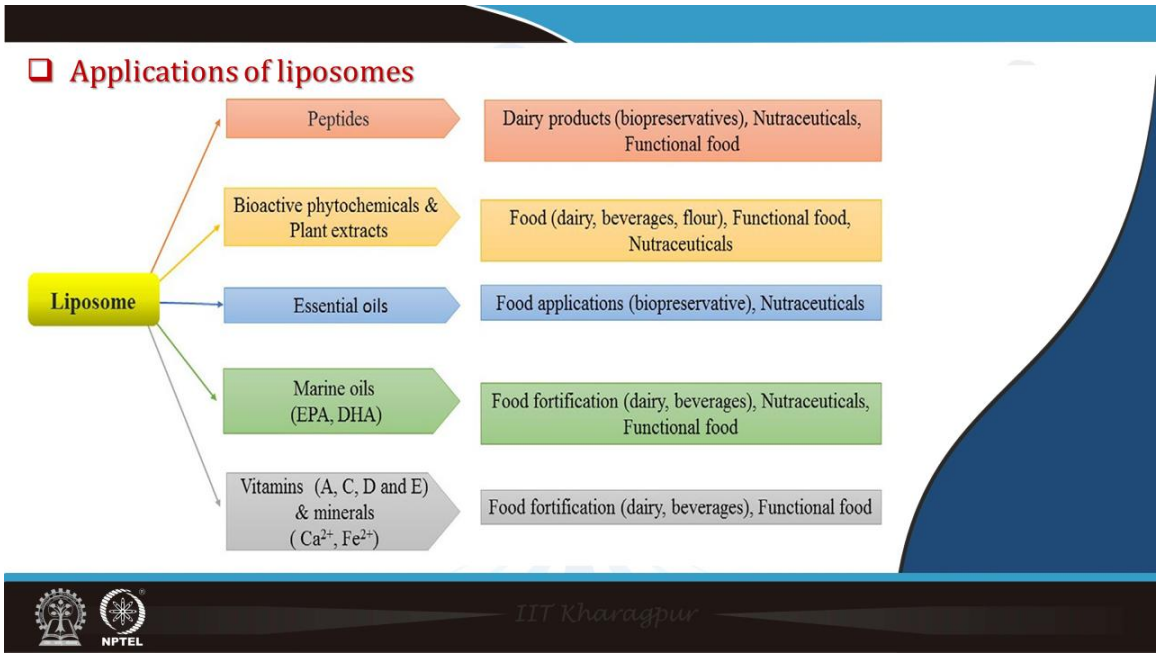
So, liposomes can be classified in various ways that is two main categories of liposomes include multilamellar vesicles, popularly known as MLVs or unilamelllar vesicles that is ULVs.

The multilamellar vesicles are the presence of multiple lipid bilayer as you could see here in this figure, ok. Whereas, the unilamelllar vesicles that is they may be joint, grant, ULV that is single lipid bilayer of 1 micron dia, there may be a small ULV that is with the vessel size range of 20 to 200 nanometer that is SUV. Then there may be multi vesicular vesicle of several vesicles with less than 1 micron in size that is MUV. So, it may be joint unilayer vessel GUV, SUV or MUV all those things ok.

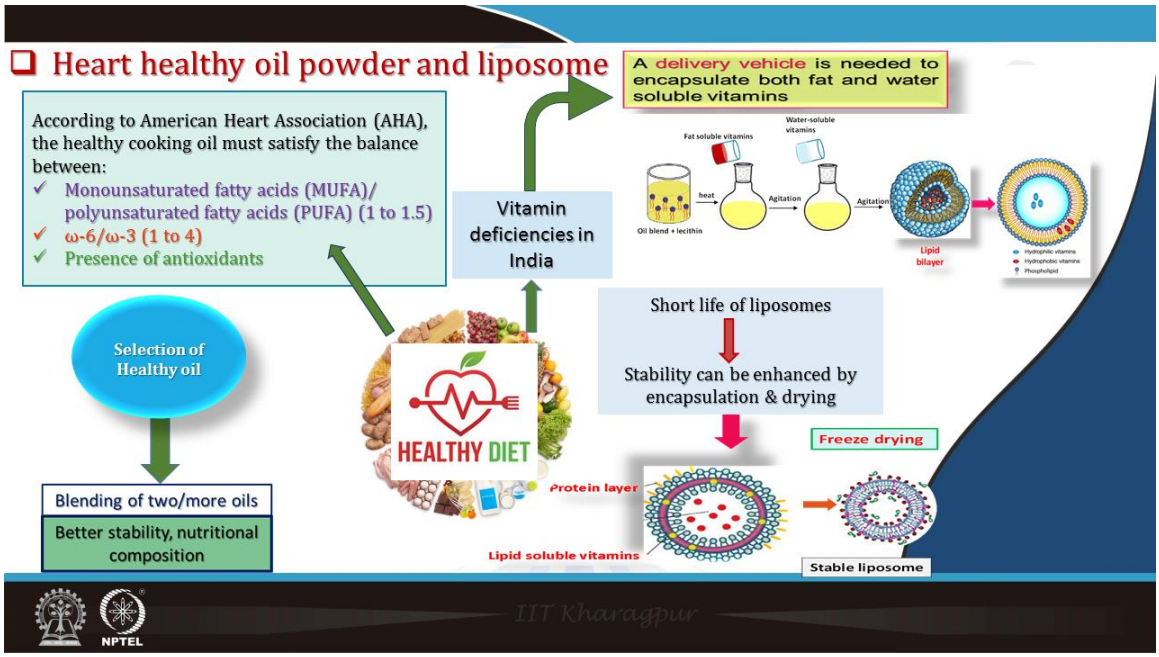


Then, method for the liposome preparation. There are four common and important stages involved here are: first is the drying of lipid dissolved in organic solvents, then exposure of the lipid to aqueous media, purification of the generated liposomes and then finally, analysis of the final products.

As this can be used as a thin layer, thin film rehydration method, detergent removal method, injection method or extrusion method or heating method can be. But novel method include supercritical fluid method like supercritical antisolvent (SAS) or supercritical reverse phase evaporation method, ok. Then, microfluidization or ultrasonication, these are the other ways which can be methods which can be used for preparing the liposomes.



Now liposomes can be applied like peptides, dairy products, bio preservatives, nutraceutical or as functional food. They are used as bioactive phytochemicals and plant extracts particularly in the dairy beverages, flours, etcetera or functional food and nutraceuticals. Then the essential oils, that is, are liposomes in the form of liposomes are used in the bio preservatives in nutraceuticals, then in marine oil like EPA, DHA, etcetera. They are used in food fortification in dairy, in beverages, in nutraceutical, and functional foods and vitamin. So, like A, C, D and E and minerals like calcium and ferrous they are used in food fortification using liposomes, dairy beverages as usual in functional foods ok.

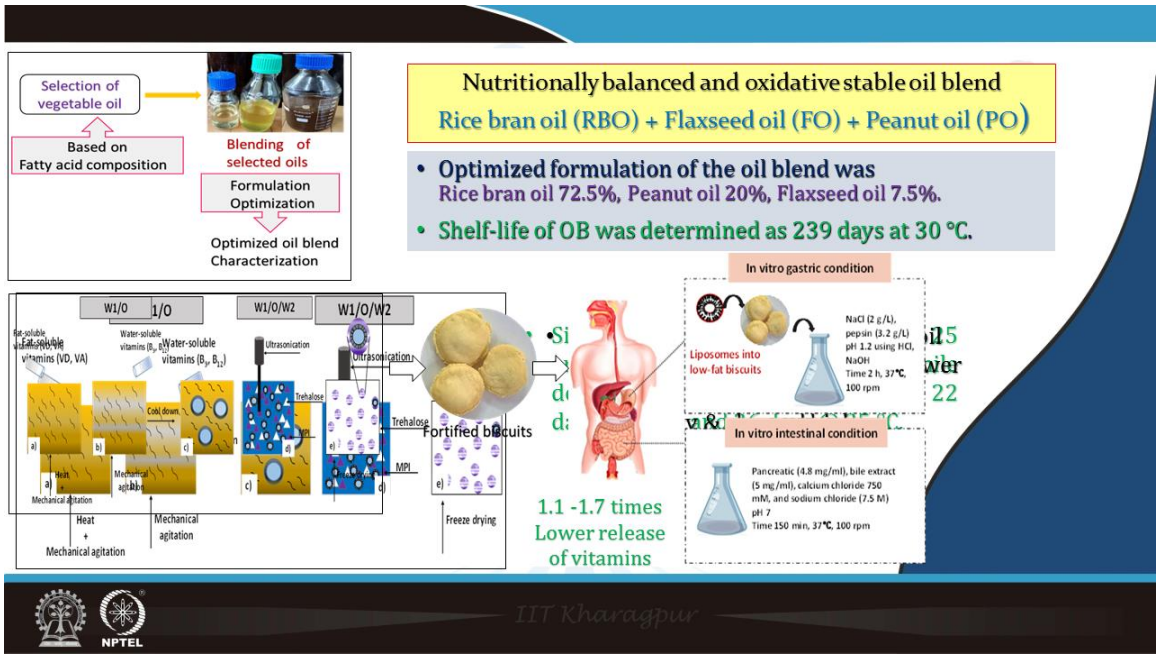


Now, let us talk about heart healthy oil powder and liposomes, ok. You know that for a healthy diet, heart healthy that the fat is very important, but at the same time, the first is fat is sometime bad fat if you use, that is, it will give lot of problems, obesity and many other problems will be there, ok. So, heart healthy fats include as per the American heart association (AHA), the heart healthy cooking oil must satisfy certain characteristics like there should be a balance in the MUFA and PUFA, monounsaturated fatty acids and polyunsaturated fatty acid should be in the range of 1 to 1.5 and omega 6 to omega 3 ratio should be 1 to 4 and there should be significant amount of antioxidant present in the system.

So, this oil which containing MUFA, PUFA in this ratio omega 6, omega 3 you know 1 to 4 and containing good amount of antioxidants in it natural antioxidants vitamin D, tocopherol, can be considered as a good heart healthy oil ok and this heart healthy oil can be converted into oil powder. So, first thing for making heart healthy oil powder the thing is that you can select the oil having these properties or formulate, that is, take the different blending of two or more oils to give to come into these properties to meet these requirements of heart healthy oil and also they should have better stability and nutritional composition or antioxidants are more that will give better stability, ok. Then even this heart healthy oil, vitamin deficiency in India you know, that is, there are it is well known.

So, it is a vehicle is needed to incorporate or encapsulate both fat and water soluble vitamins all right into this the oil powder etcetera, ok. So, using this liposome preparation technology or this powder etcetera you can take oil blend and lecithin, then heat it and fat soluble vitamins are added into properly agitated, then water soluble vitamins are added and then agitated and lipid bilayer is formed and this finally, liposomes and which contains almost all hydrophobic vitamins, hydrophilic vitamins, phospholipids, etcetera they are all enclosed in the one unit.

So, that is how beauty of this liposome preparations ok and, but the short shelf life of liposomes, that is, stability can be enhanced by encapsulation and drying, that is, the protein layer and the lipid soluble layer of vitamins, they can be freeze dried to give stable liposomes. So, that is in brief, you can say, that is, the proper selection of the oil, proper blending and then incorporating into it vitamin A and D and other various bioactive micronutrients can be used and then this can be stabilized and freeze dried and you get a material liposome, stable liposome, ok.

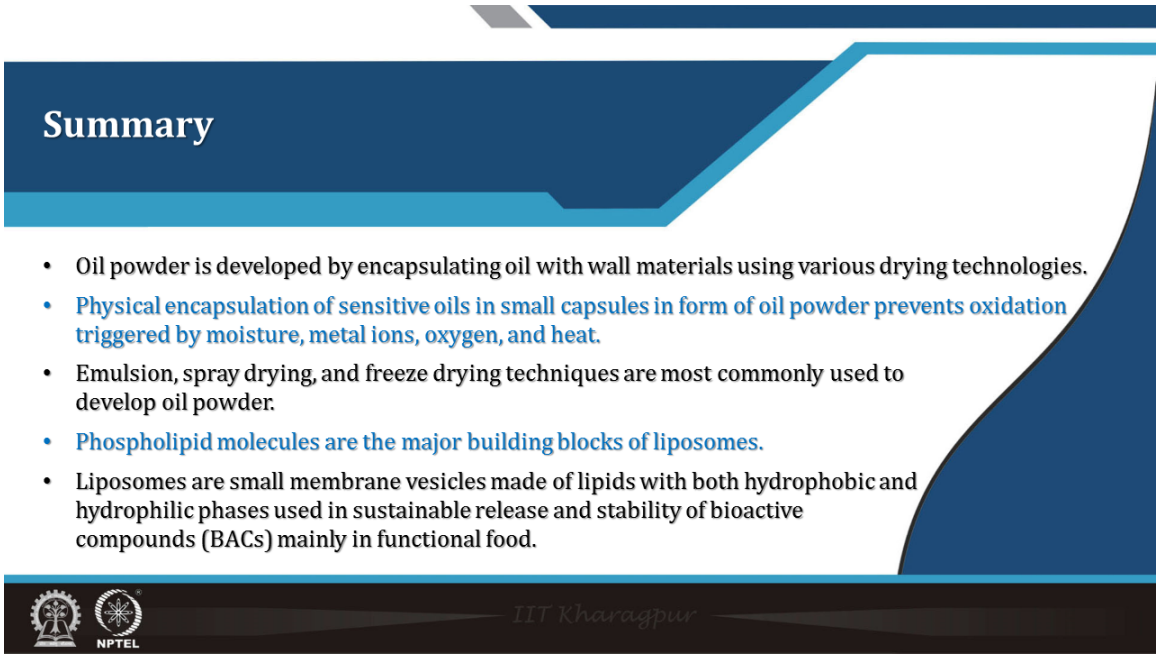


Now, what we in our work, what we have done that obviously, we selected that is the vegetable oil based on their fatty acid compositions and then blended these oils and we formulized the optimized formulation was optimized ok and the blend characterization the ratio etcetera. And, we in our study, we found that rice bran oil,

flaxseed oil and peanut oil in the proportion of 72.5 percent, 20 percent and that is 7.5 like rice bran oil 72.5 percent, peanut oil 20 percent and flaxseed oil 7.5 percent if it is blended in this it meets the requirement of AHA for the heart healthy oil, ok. And we could get the shelf life of this oil blend was found in our study as 239 days at 30 degree Celsius temperature, ok. So, these oils we have taken as I explained earlier also that is fat soluble vitamins and vitamin A and D were mixed into it by using that is heat and mechanical agitation and then water soluble, I mean, that is, the blends were emulsified by the various wall material and coating material, poly liposome formation were used then vitamin water soluble B9 and B12 were added into it then mechanical agitation form. Then, see it gives here that is water in oil and water. This emulsion is formed and using this is given ultrasonication techniques ok. And this gives finally, the freeze dried.

So, micro capsules are formed. So, single emulsion were unstable in our study we found that at 25 and 55 degree Celsius temperature while double emulsions were stable for about 22 days at 55 degree Celsius and 36 days at 25 degree Celsius ok. Then these micro encapsule liposomes, stable liposomes where we used in the various they can be obviously, used in various different products like frozen desserts, etcetera. Also, we used this in the preparation of fortified biscuits. So, biscuits that is the dough was incorporated with appropriate quantity of these freeze dried liposomes and biscuit made from the using standard methods standard and it was baked using and we were found, that is, that these baking temperature liposomes were stable up to that baking temperature, ok. And the biscuits were used with encapsulated oil provided a crisper texture with fewer surface cracks and irregular sticky morphology and showed higher antioxidant potential, ok. And this can also be used we also used in the frozen dessert, ok.

And then this is used, that is, biscuits. We studied that in vitro digestion in the gastric conditions. The liposomes into the low fat biscuits and sodium chloride (2 gram per liter), pepsin and pH and time these were in vitro gastric condition were simulated, ok. And in vitro in vitro intestinal conditions were simulated and we found, that is, they were stable and released properly in the gut as well as in the intestine. Their release kinetics were studied and we found it satisfactory, ok. And we found that 1.1 to 1.5 times lower release of vitamins is I have is over found here, ok.



So, with this, I come to the conclusion of this lecture, that is, oil powder is developed by encapsulating oil with wall materials using various drying techniques, physical encapsulation of sensitive oils in small capsules in the form of oil powder prevents oxidation triggered by moisture, metal ions, oxygen, and heat. Emulsion spray drying and freeze drying techniques are the most commonly used to develop oil powder. Phospholipid molecules are the major building blocks for the liposomes. Liposomes are small membrane vesicles made of lipids with both hydrophobic and hydrophilic phases which are used in sustainable release and stability of bioactive compounds mainly in the functional food development.

These were the references that used for this lecture.

Thank you very much for your patience here. Thank you.