Surface Engineering Nanomaterials
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Lecture - 31
Microencapsulation processes

Hello. Today we are going to start our new lecture on the Microencapsulation Processes. So, before going to details about the microencapsulation first we have to know that what is the microencapsulation means actually.

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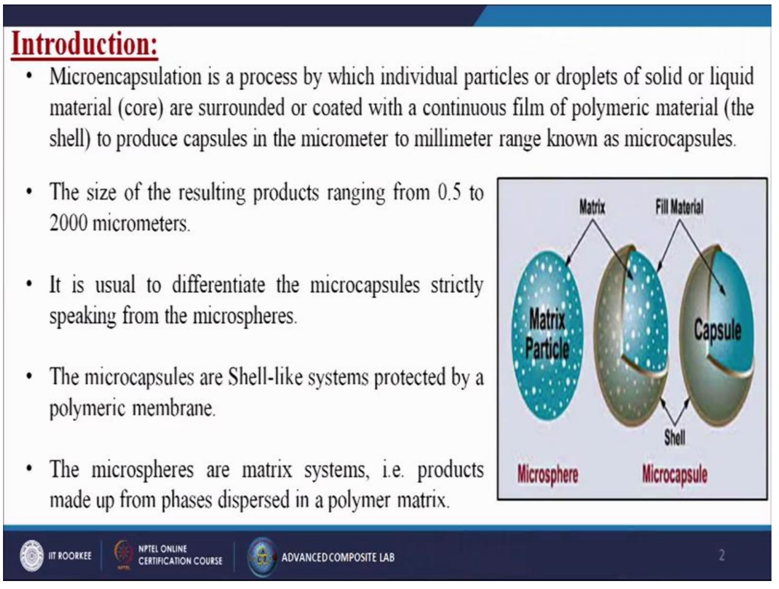

So, microencapsulation is nothing but it is a one kind of coating processes. So we can coat some kind of solid material or maybe we can coat some kind of liquid materials, just giving the coating of any kind of polymer or maybe any kind of ceramic material onto that, so that it can be surrounded by another material onto the substrate. So, in another word we can say that this is also the one kind of formation of a core shell structure.

So, microencapsulation is a processes by which individual particles or droplets as I already told in this particles or droplets either may be in the solid from or in the may liquid form or solid or liquid material core are surrounded or coated with the continues films of polymeric material, the shell to produce the capsules in the micrometer to millimeter range known as Microcapsules. So, from this particular figure we can understand that we are having some kind of matrices then on top of that we are making a shell on another material by which we are covering our base material or may be the substrate. So, this is known as the Microcapsulation or may be the Microcapsules.

The size of the resulting product ranging from 0.5 to 200 micrometers, so it is controllable; we can easily control the thickness of that particular coating. It is usual to differentiate the micro capsules strictly speaking from the microspheres. The microcapsules are shell like system protected by a polymeric membrane. So, as I told already we are giving a coating of some kind of polymeric materials onto the substrate or may be onto the materials which we are going to coat. The microspheres are matrix system that is product made of from phases dispersed in a polymer matrix.

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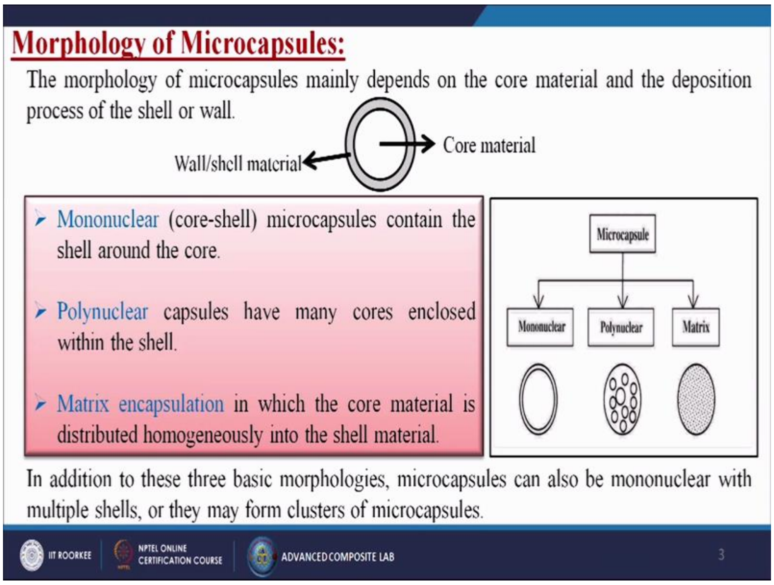

Now, when we are talking about the microencapsulation fist we have to know that; what is the science behind it? So, how we are making that system, how we are making the materials? So, the morphology of microcapsules mainly depends on the core material and the deposition process of the shell or wall. So here, you can see from this particular figure that inside generally we are calling it as substrate or maybe it is our material which is known as the core material on top of that we are adding or maybe we are providing a layer into that which is known as the wall or maybe the shell material. So, here wall or shell material is the coating material and core material is that on which we are doing the coating.

So, by a method we can divided this parts into 3 different joules rather; one is called the mononuclear- core shell microcapsule contain the shell around the core, it is like this. So, here inside is the core material outside is the layer, so that is why it is known as the mononuclear. Then polynuclear capsules have many cores enclosed within the shell. So here, this is the shell in which so many cores are enclosed; and the matrix encapsulation, in which the core material is distributed homogeneously into the shell material. So, depends upon how we are putting the filler inside. So, if it is a single material we are doing the coating, if it is a mixing of different core material we are doing a coating, and if it is some matrix material means like a composites and then we are making a coating; so based on that we are having mononuclear, polynuclear and the matrix.

In addition to these three basic morphologies microcapsules can also be mononuclear with multiple shells or they may form clusters of microcapsules. So, this is like a something a different verity, so depending upon our requirement, depending upon our application we can make different types of microencapsulation.

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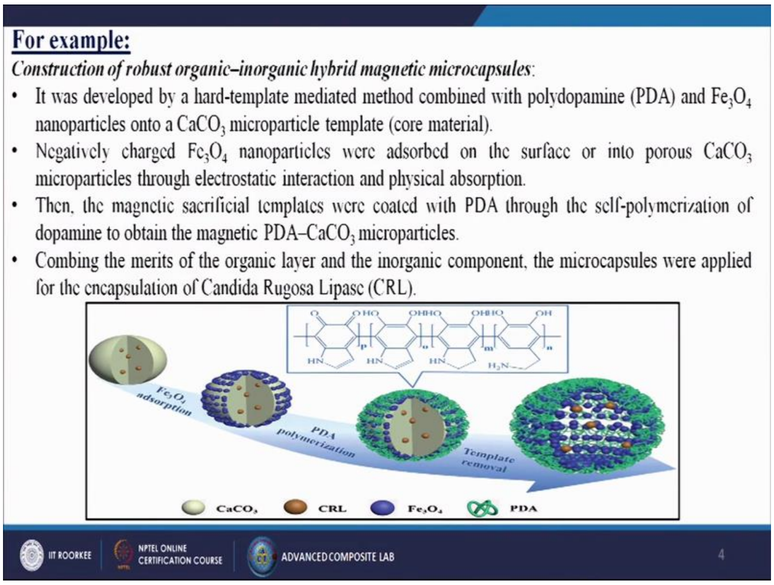

So, it is a one example just I am going to give so that we can better understand how we are doing the microencapsulation. So, it is example of a construction of robust organic inorganic hybrid magnetic micro capsules. So here, generally we are taking the polymer which is a nothing but the polydopamine and Fe 3 O 4 nanoparticle onto calcium carbonate microparticle templates which is known as the core material. Now, negatively charged Fe 3 O 4 nanoparticles were absorbed on the surface or into porous calcium carbonate microparticle through electrostatic interaction and physical abortion.

So, in that calcium carbonate we are incorporating the Fe 3 O 4 inside, then the magnetic sacrificial templates were coated with the PDA through the self polymerization of the dopamine to obtain magnetic PDA calcium carbonate microparticles. Combing the merits of the organic layer and the inorganic components and microcapsule were applied for the encapsulation of Candida Rugosa Lipacs. So, this is a one kind of composite structure generally we are making by this coding coring technology.

So, inside we are having that material which is known as the calcium carbonate in which we are inserting the Fe 3 O 4 and then the whole thing we are making the coating by polydopomine or may be the PDA solutions. So like this way we can do this kind of microencapsulation for some biomedical application or may be some kind of magnetic microcapsules application.

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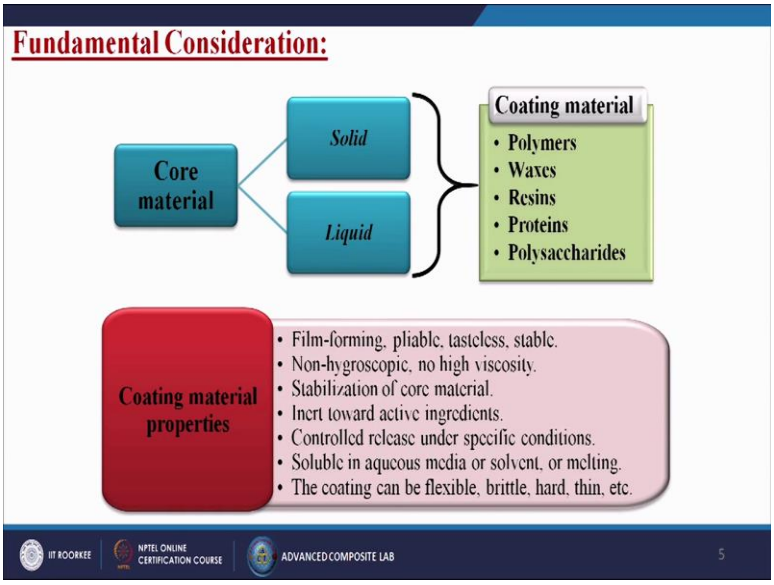

Now the fundamental consideration; so as I told already our core material can be anything means in the form of solid or may be in the form of liquid. So now, this coating material is divided into polymers, waxes, resins, protein and polysaccharides. As I told already this kind of microencapsulation the mainly application is the biomedical purpose or may be that medical application or may be that drug delivery applications.

So, generally we are using this kind of materials as a coating materiel, like polymers, waxes, resins, protein, polysaccharides; then coating material properties these properties should have means while choosing that coating materials we are taking care all this things into our minds. So, fist one is called the film forming pliable tasteless and stable; then non-hygroscopic, no high viscosity; stabilization of the core material sp that it cannot change with the environmental condition; inert toward active ingredients, when we are inserting that materials inside our body may be some acid may be some base so it should not react with acid or base and it should do the proper justices inside our body.

Controlled release under specific condition, so when the exact involvement it will get then only it will start acting or maybe it will be activated. Soluble in aqueous media or solvent or melting; the coating can be flexible, brittle, hard, and thin. So, per our requirement we have to choose the coating material best on these properties.

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Examples of some core materials:

Core material	Characteristic property	Purpose of Encapsulation	Final product form
Acetaminophen	Solid	Taste masking	Tablet
Aspirin	Solid	Taste masking; sustained release; reduced gastric irritation	Tablet or Capsule
Vitamin-A palmitate	Nonvolatile liquid	Stabilization to oxidation	Dry powder
Urease	Water soluble enzyme	Permeselectivity of enzyme, Substrate and reaction	Dispersion
Progesterone	Slightly water soluble solid	Sustained release	Varied
Isosorbide dinitrate	Water-soluble solid	Sustained release	Capsule
KCl	Highly water soluble solid	Reduced gastric irritation	Capsule
Activated charcoal	Adsorbent	Selective sorption	Dry powder
Liquid crystals	Liquid	Conversion of liquid to solid	Flexible film for thermal mapping of anatomy

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So here, we are giving some examples of that core material; what are those? Fist is called the acetaminophen. So generally this one is all the medical terminology or may be that medicine names. So, simple the thing is that inside is our medicine and outside we are giving the coating so that we are taking that medicine since our inside our body.

The best example is our capsule. So, if you see the capsule the outside is made by the polymers or may be some kind of protein or may be some kind of materials which can easily digest in our body, not only that can be melted in our stomach, and inside actually we are putting certain kind of medicines. If we do not do like this, so we cannot take the medicine directly in our stomach, maybe the medicine is too bitter or may be it cannot be tolerable. So, that is why we are doing the microencapsulation by some kind of polymeric materials and we are solving that medicine into our body.

So, here the core metical may be acetaminophen or may be aspirin or may be vitamin-A palmitate, urease, progesterone, some kind of harmon, then isosorbide dinitrate potassium chlorite activated charcoal liquid crystals. So, here characteristics properties acetaminophens generally solid, aspirin are also one kind of solid materials, vitamin-A palmitate is non volatile liquid, some kind of potassium chlorite is highly water soluble solid. So, here we can see that either the characteristics of the core material may be the solid or may be that liquid or may be some kind of viscous materials.

Now, purpose of a encapsulation why we were doing, generally we are using it for the taste making purpose, taste making sustained release reduced gastric irritation, stabilization to oxidization may be some kind of sustained release, then gastric irritation. So, this all are the purpose for which we are doing the microencapsulation solution. And what is the final product form generally we are making the tablet with the help of acetaminophens or may be aspirin generally we are taking it as a tablet or in the form of capsules, some kind of some cases we are using some kind of dry powder; so flexible powder, flexible films of thermal mapping of anatomy. So, these all are the application for which we are doing the microencapsulation.

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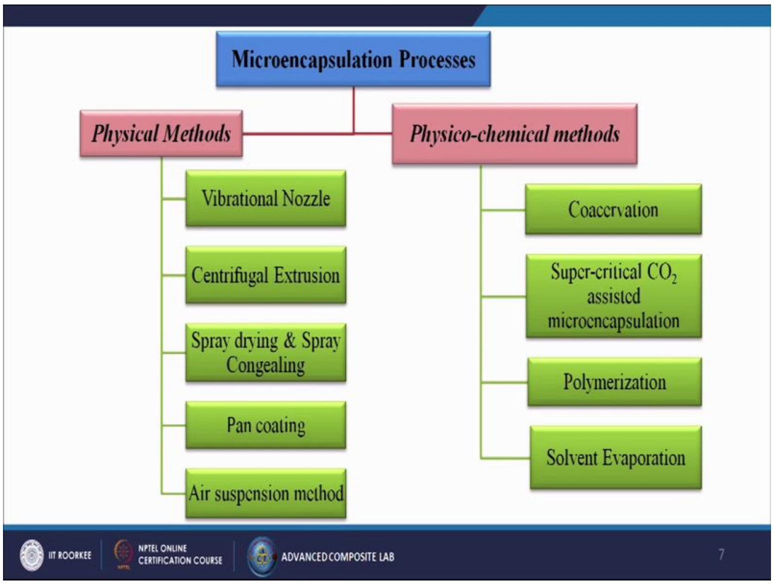

Now, we are going to discuses about the several processes which are related to the microencapsulation. So, that process is divided into two parts: one is called the physical methods another one is called the physicochemical methods. So, in the physical methods we are going to discuss about the vibrational nozzle, centrifugal extrusion, spray dying and spray congealing, then pan coating, and air suspension method. And physicochemical methods were we are talking about the coaccrvation, then super critical carbon dioxide assisted microencapsulation, polymerization and solvent evaporation. So, these all are the different process by which we can do the microencapsulation.

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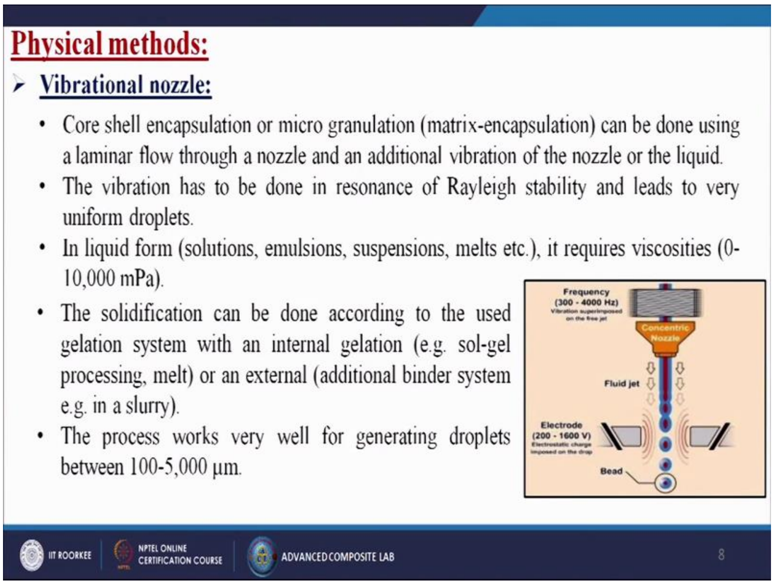

So, now fist we are going to do or maybe we are going to discuss only about the physical methods and fist one is the vibrational nozzle. So, in this particular you can see that we are having some kind of concentrating nozzle in which the high fluid is coming, then we are having some electrode and due to that frequency or may be that voltage potential difference that fluid is divided into small small parts or may be the small small bobbles or may be particular.

So, how it is working? Core shell encapsulation or micro granulation, matrix-encapsulation can be done using a laminar flow through nozzle and an additional vibration of a nozzle or the liquid. The vibration has to be done in resonance or Rayleigh stability and leads to very uniform droplets, so it will be divided into small small droplets. A liquid from solutions, emulsions, suspensions, melts etcetera it require viscosities generally 0 to 10000 mega Pascal. The solidification can be done according to the used gelation system with the internal gelation; sol-gel processing, melts or an external binder system in slurry. The process work very well for generating droplets between 100 to 5000 micrometer.

So, simply we are having that slurry in that particular slurry in middle also we are having another materials generally which is known as the core materials and it is surrounded by some kind of encapsulation materials. Then it is coming into a liquid from may be in viscosity is more higher, then we are just giving the vibration so that that liquid formation is can be dividing into small small particles or may be small small droplets.

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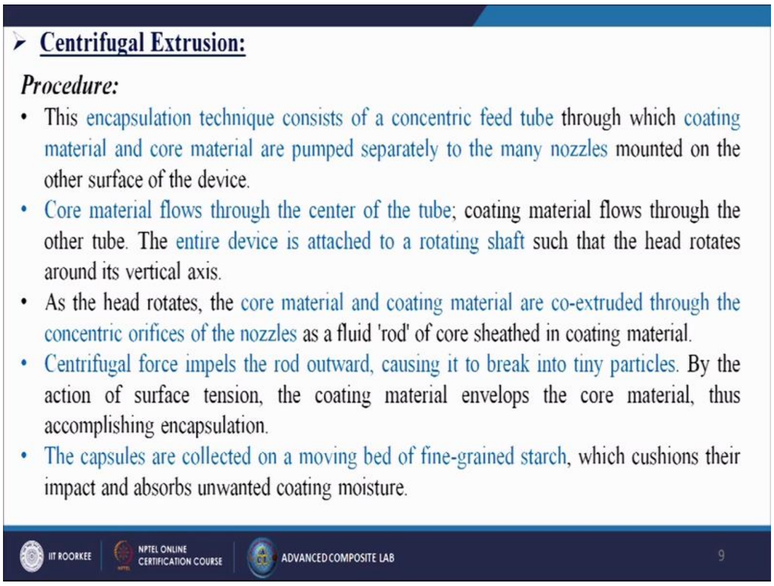

Next, we are going to discuss about the centrifugal extrusion. So, what is the procedure? The encapsulation technique conceits of a concentric feed tube through which coating material and the core material pumped separately to the many nozzle mounted on the other surface of the device. So, simply when I will come to the next slide I will show you that how we are going to do this kind of micro encapsulation. Simply we are using some kind of machine which is called the extruder in which we can use that some twins score extruder or may be the spring effect by in which we can put our core material and the our shell material together. Then core material flows through the center of the tube coating material flow through the other tube. The entire device is attached to a rotating shaft such that the heat head rotates around its vertical axis. As the head rotates the core material and coating material are co extruded through the concentrating orifices of the nozzles as a fluid rod of core sheathed in coating material.

Centrifugal force impels the rod outward causing it to break into tiny particles. By the action of surface tension the coating material envelops the core material, thus accomplishing encapsulation. The capsules are collected on a moving bed of fine grained starch, which cushions their impact and absorbers unwanted coating moisture.

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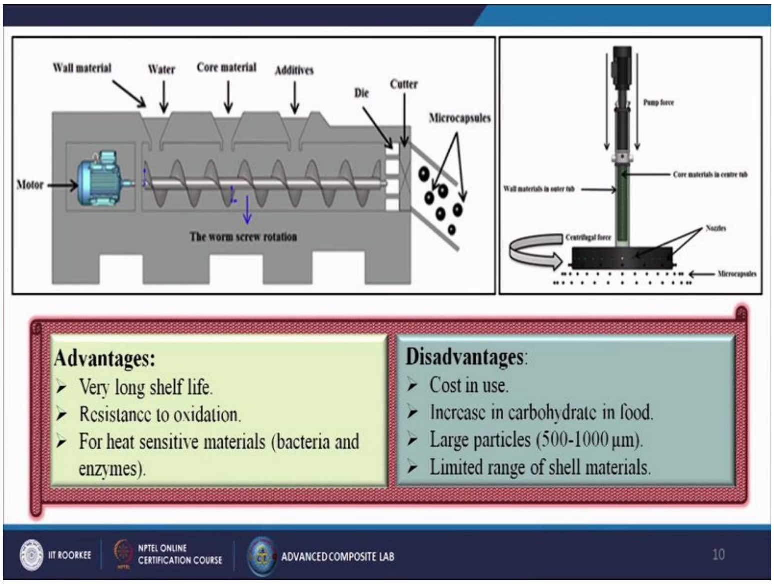

From this particular figure we can easily understand that we are having some motor, then we are have putting the water wall materials; that means the coating materials with water and the core materials, then we can add some kind of additive also, then we are rotating this one; so by centrifugal actions all the particles will come outside in a rod from and then due to that surface energy it is divided into the small small particles or maybe we are having some cutter by which we can cut that continues rod shapes into small small particles. And then we can get the materials in which the inside is a core material and outside will be you; sorry inside will be a substrate or may be that core materials yes and outside will be your shell material or may be the coating materials.

And also here is also another kind of technique by which we can do this kind of encapsulation. So, by centrifugal force by rotations we can make the small small micro particles, here we are having some nozzles. So, simply we are pumping that materials and then the inside will be your substitute or may be that core materials and outside is your coating materials, then the whole thing we are pumping into some device in which we are having small small holes. And then the disk is rotating in a very high speed so that through that holes the small small microparticles or may be small small microcapsules are coming, and we can collect it into some starch from so that whatever the moistures or may be that some kind of contaminants which can stick with that easily that can absorb by the starch materials. Not only that when it will come in a high velocity may be that can try to stick together. So, we are using some kind of starch so that it can easily that impacted can be easily absorbed and we can get the homogeneous structure of this micro encapsulations.

What are the advantages? Very long shell life, resistance to oxidation, for heat sensitive materials like bacterial and enzymes. There are certain disadvantages also; so cost in use, increase in carbohydrate in food, large particles generally 500 to 1000 micrometer, limited range of shell materials. So, these all are the disadvantage.

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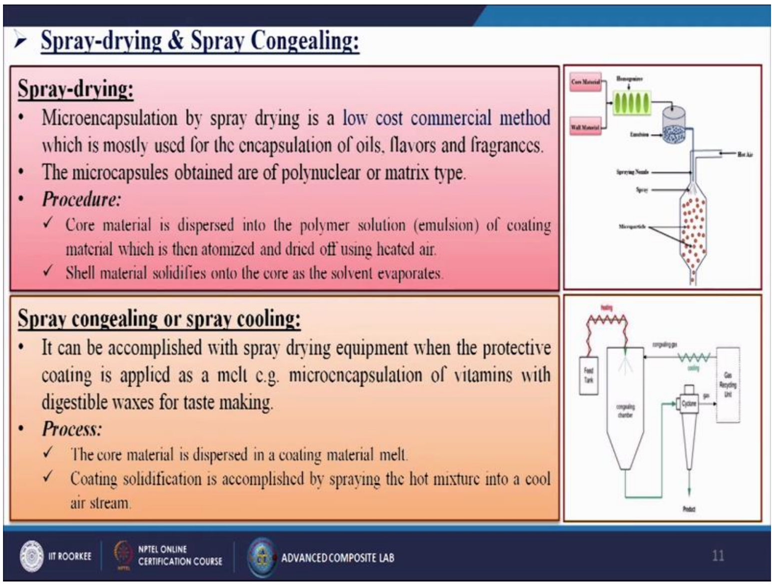

Now, next one we are going to discuses about the spray drying and spray congealing. So, in the spray dying process microencapsulation by spray drying is a low cost commercial method which is mostly used for the encapsulation of oils, flavors and fragrances. Generally we can use this one for our cosmetics products or maybe this one for our some food products or may be some other meals.

So, the micro capsules obtained are of polynuclear or matrix type; procedure core material is dispersed into the polymer solutions of coating material which is then atomized and dried off using heated air. Shell material solidifies onto the core as the solvent evaporates. So, simply we are having some polymer materials, we are putting that materials into some solvents, then we are applying a high air or maybe that high heated air or maybe that air pressure by which the shell material can dry easily and maybe it can evaporate and then it can became hardened and inside will be a core materials.

Next one is called the spray congealing or may the spray cooling methods. So, here it can be accomplished with high spray drying equipment when the protective coating is applied as melt microencapsulations of vitamins with digestible waxes for taste making; so generally these all are the applications. What is the process? The core material is dispersed in a coating material melt, coatings solidification is accomplished by spraying the hot mixer into a cool water systems. So here, the two methods is more or less same only the approach are different.

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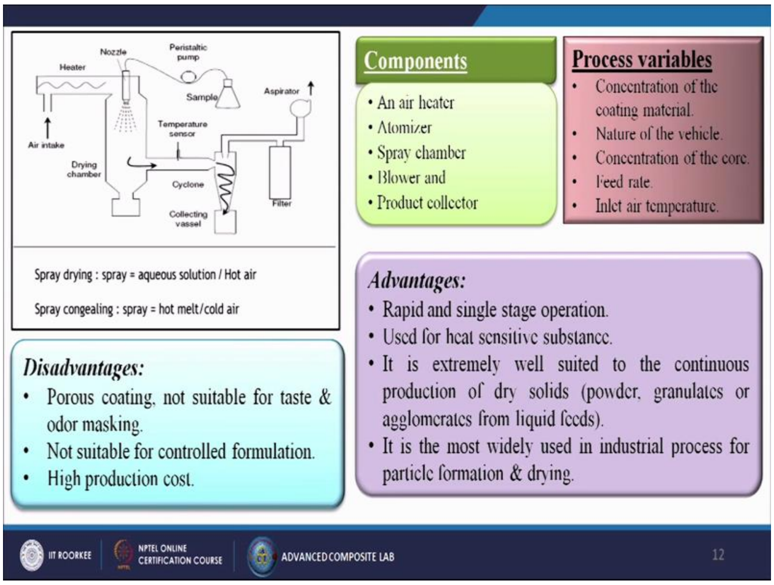

So, this is called the spray drying in which spray is equals to aqueous solutions or maybe the hot air. Spray congealing; spray is equal to hot melt oblique cold air. So here, only this is the difference; one case we are using the aqueous solutions or maybe the hot air another case we are using the hot melt oblique cold air. So, here the component is that air heater, atomizers, spray chamber, blower and product collector. So, process variables generally we can do these concentrations of the coating materials we can vary, nature of the vehicle we can vary, concentration of the core material we can vary, feed rate we can vary and inlet air temperature we can vary.

So, there is certain kind advantages also present: rapid and single stage operations, used for heat sensitive substance, it is extremely well suited to the continues production of dry solids, like powder, granulates or agglomerates from liquid feeds, it is the most widely used in industrial process for particle formations and drying. Now there are certain disadvantages also: first one is called the porous coating not suitable for taste or odor masking, not suitable for controlled formulations, high production cost. So, these all are the disadvantage of these particular techniques.

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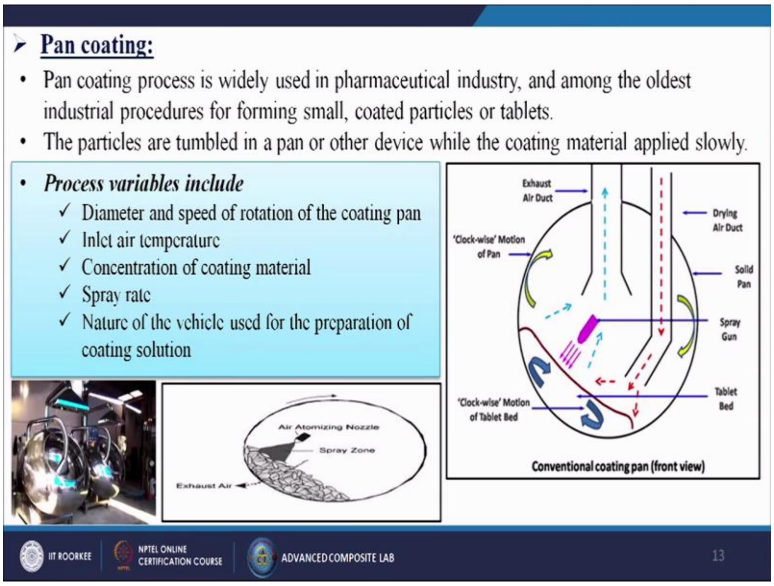

Next we are going to discuss about the pan coating. So, pan coating is nothing but the this is the best example is that when for the structural purpose we are going to use or we are trying to make the slurry what we are doing, we are having a big chamber in which we are putting the sands, in which we are putting the concrete, in which we are putting the stones and then we are rotating all the materials into some rpm by which the proper mixing can be done and we are getting the slurry which we are using for our concrete purpose or maybe some building purpose.

So, the process is same is here also. So here, the pan coating process is widely used in pharmaceutical industry and among the oldest industrial procedures for forming small coated particles or tablets. The particles are tumbled in a pan and other device while the coating material applied slowly. The simple thing we are putting the core material inside the tumbled or maybe that pan and then form top of that is slowly slowly we are releasing our coating materials and then we are rotating that tumbled in rpm so that all the core materials can get the coating materials onto it so that the proper coating can be done.

So, process variables include: diameter and speed of rotations of the coating pan, inlet air temperature, concentration of coating materials, spray rate, nature of the vehicle used for the preparation of coating solutions. So, left hand side corner here you can see that here is a big tumb or maybe that big a vessel are there we in which we can put our core materials and there is one nozzles by which we can put our coating materials. So, schematic diagram is looks like this. So here, we are having some kind of nozzles by which air recognizing nozzles by which we are putting the core materials onto the core materials and these whole things is rotating in a high rpm. So, by which we can do the homogeneous coatings. So, this is known as the Pan Coating Microencapsulations.

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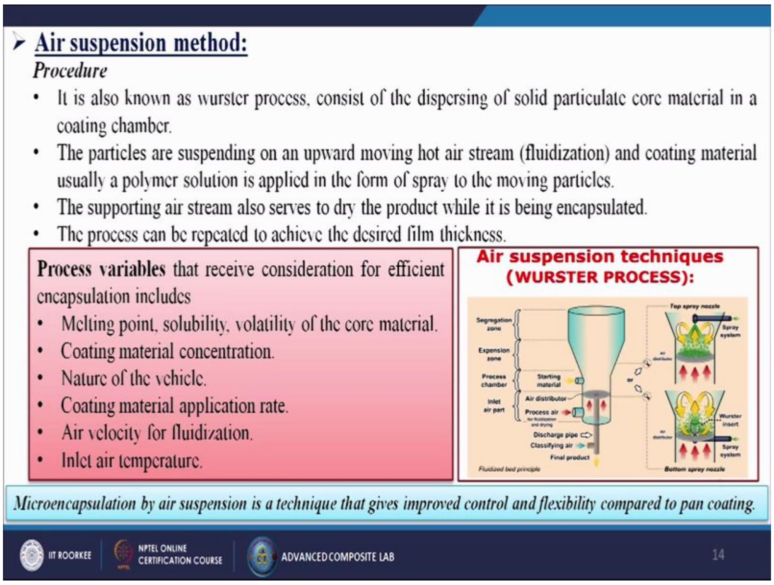

Next we can discuss about the air suspension methods. So, this is also one procedure by which we can use the high air pressure. So here, it is also known as Wurster processes consists of the dispersing of solid particulate core materials in a coating chamber. The particles are suspending on an upward moving hot air stream fluidizations and coating material usually a polymer solution is applied in the form of spray to the moving particles. The supporting air stream also serves to dry the product while it is being encapsulated. The process can be repeated to achieve the desired film thickness. So, simply we are having that core materials and from the bellow we are applying high air pressure onto that core materials. So, it is like that something we are making for using the popcorn.

So, when this core materials is getting a high pressure of air from the bottom so just it is trying to fly, and the form side wise we are trying to give the core materials spray so that the core material can be deposit onto the core materials itself. So, by this way we can do, so that is why this method is known as the air suspension method. So, process variables that receive consideration for efficient encapsulation includes melting points solubility, melting points solubility volatility of the core materials; coating material concentrations; nature of the vehicles; coating material application rate; air velocity for fluidization; inlet air temperature.

So, this is the experiment methods what I am talking about. So, simply we are applying high air pressure from the bottom so that the coating material is flying inside the chamber and either from the top or maybe from the side we can inject the coating material suspensions or maybe the droplets so that it can easily deposit onto your core materials. So this is the techniques; microencapsulation by air suspension is a technique that gives improved control and flexibility compare to the pan coating. This is the added advantage over the pan coating method. And this process is known as the Wurster Process.

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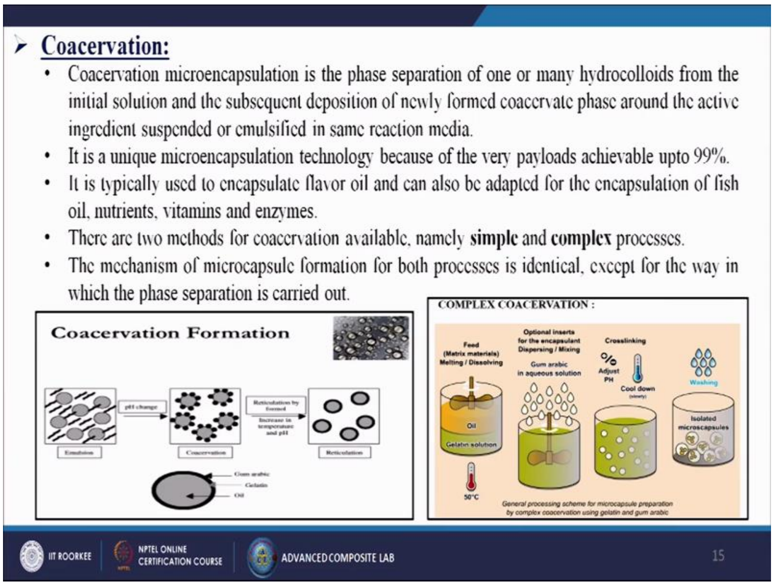

Next we are talking about the coacervations. So, this is also a one kind of new technology generally we are using for making the microencapsulations. So, these technology tells us like that, that it is a phase separations of one or many hydrocolloids from the initial solutions and the subsequent depositions of newly formed (Refer Time: 24:06) phase around the active ingredient suspended or emulsified in same reaction media. It is a unique microencapsulation technology because of the vary payloads achievable up to 99 percent. It is typically used to encapsulate flavor oil and can also be adapted for the encapsulation of fish oil nutrients vitamins and en enzymes.

Generally, we can use this kind of techniques for preparations of the medicines. There are two methods of coacervations available namely: simple methods and another one is called the complex processes. The mechanism of microcapsule formation for both processes is identical except for the way in which the phase separation is carried out. So, in this particular case we are doing the emulsions first, and then we are changing the ph of that, so that the coating material can be deposited onto the drop drop wise then we are using the temperature and the ph so that the reticulations can occur and by this it can form a gelatin on top of that.

So, either we can core by this technology we can core the oil or maybe some kind of liquid particles or maybe some kind of vitamins or maybe some kind of medicines by this kind of techniques. So here, this is the complex coacervations method for in which first we are having the gelatin solutions and we are having the oil. So, first matrix materials melting and dissolving, then next case optional insert for the encapsulated dispersing or mixing we are doing. We are putting some kind of gum Arabic in aqueous solutions we are adjusting the ph we are cooling it down. So, that that Arabic gum can be coated by some kind of cross linking materials and then simple we are washing and we are isolating these microcapsules.

So, general processing scheme for microcapsule preparations by complex coacervations using gelatin and the gum Arabic; so this is the simple technique of these particular methods

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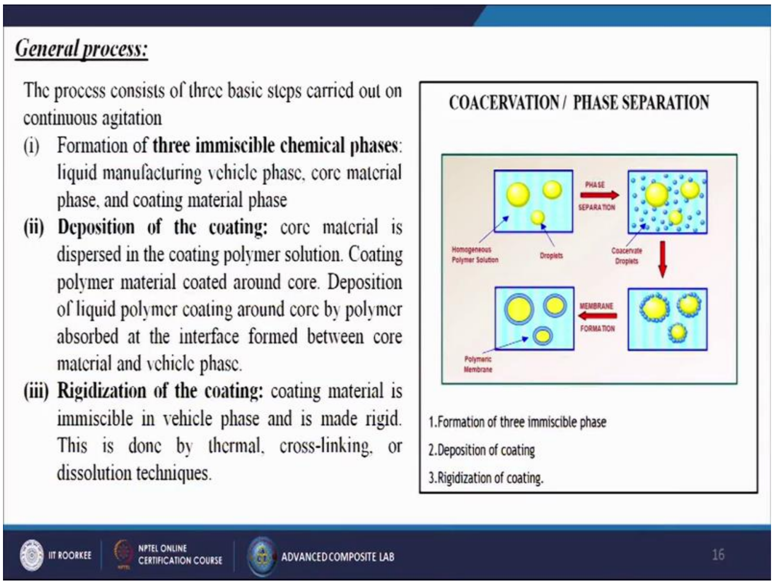

Next we are going to discuss some general process about these techniques. So, the process consist of three basic steps carried out on continuous agitation: first one is called the formation of three immiscible chemical phases, liquid manufacturing vehicle phase, core material phase and coating material phase. Next one is called the deposition of the coating; a core material is dispersed in the coating polymer solutions. Coating polymer material coated around core deposition of liquid polymer absorbed at the interface formed between core material and vehicle phase. And then the last one is called the rigidizations of the coating. Coating materials is immiscible in vehicle phase and is made rigid; this is done by thermal cross linking or dissolution techniques. So, these all are the figures by which we can do this kind of process.

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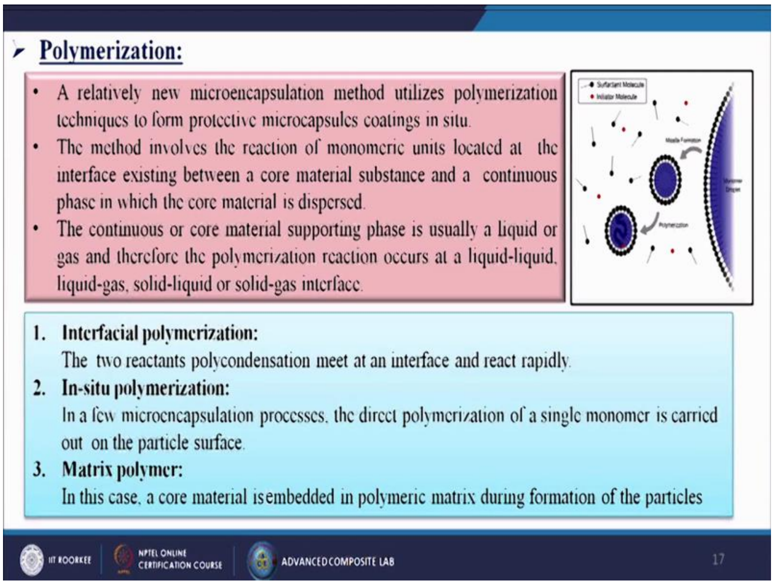

Next is called the simple polymerizations methods. It is also a randomly and most easily used method by which we can do the coating of our nanofillers or maybe any kind of core materials. So, what this process tells actually? A relatively new microencapsulation method utilizes polymerizations techniques to form protectives microcapsules coating in situ.

The method involves the reaction of monomeric units, units located at the interface existing between a core materials substance and a continuous phase in which the core material is dispersed. The continuous or core material supporting phase is usually a liquid or gas, and therefore the polymerization reaction occurs at a liquid liquid, liquid gas, solid liquid or maybe that solid gas interface. So, any phase by using the core materials or maybe by the coating materials we can do this kind of microencapsulations.

So, what is that? First is called the interfacial polymerizations. The two reactants poly condensations meet at an interface and react rapidly. In-situ polymerizations in a few microencapsulation processes the direct polymerization of a single monomer is carried out on the particle surface. Matrix polymer in this case a core material is embedded in polymeric matrix during formations of the particles. So, these all are the three different methods by which we can do this polymerization process.

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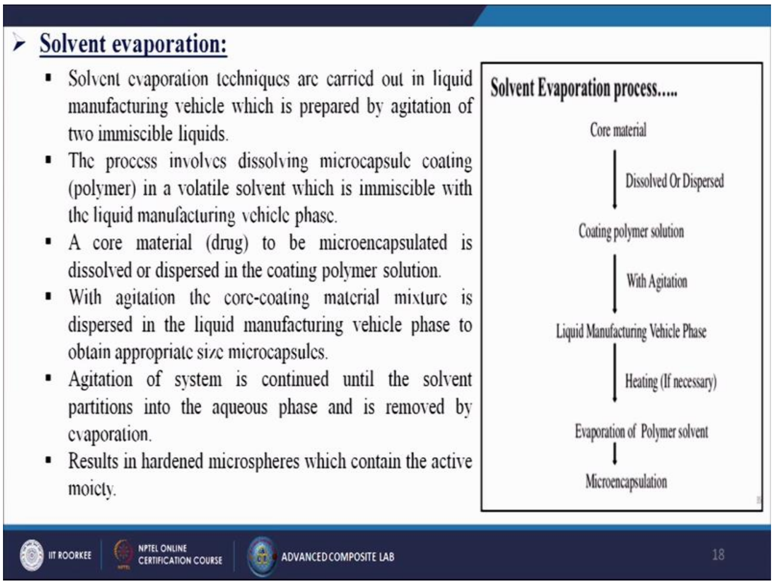

Next one is called the solvent evaporation methods. It is also a one kind of microencapsulation techniques. What this a techniques telling us? It is the techniques which are carried out in liquid manufacturing vehicle which is prepared by agitations of two immiscible liquids. So, simple two liquids which we are not mixing properly we can take for this particular purpose.

The process involves dissolving microcapsule coating polymer in a volatile solvent which is immiscible with the liquid manufacturing vehicle phase. A core material generally drug to be microencapsulated is dissolved or dispersed in the coating polymer solutions. With agitations the core coating material mixture is dispersed in the liquid manufacturing vehicle phase to obtain appropriate size microcapsules. Agitation of system is continued until the solvent portion partitions into the aqueous phase and is removed by evaporations. Results hardened microspheres which contain the active moiety.

So here, the solvent evaporation process first we are taking the core materials, then we are dissolve the thing that core materials into or maybe a dispersing into some medium, then we are making the coating polymer solutions, and then we are doing some kind of agitation process so that liquid manufacturing vehicle phase is forming, then we doing the heating and then we are evaporations of the polymer solvent. And by these methods we are doing the microencapsulations.

So, this is the whole diagram that how we are following the solvent evaporations microencapsulations technique.

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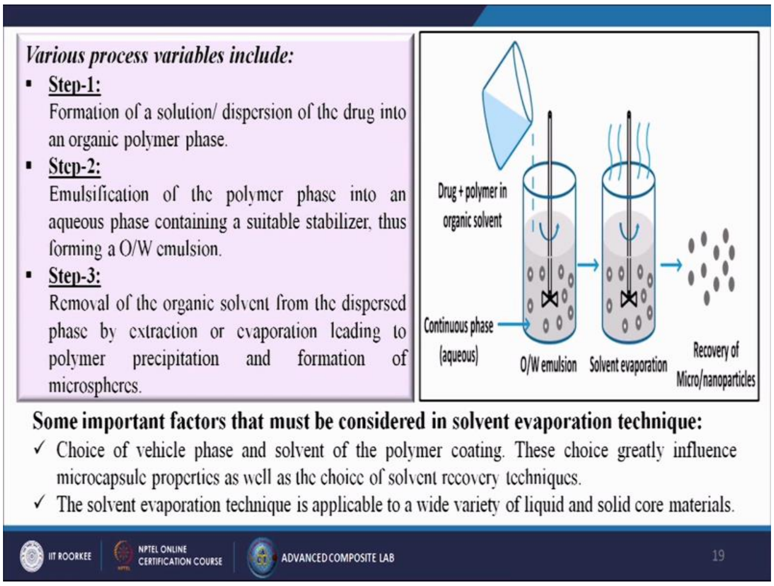

So here, also the various steps are available. So first is called the formation of a solution or dispersion of the drug into an organic polymer phase; this is the step one which we are doing. Next step emulsifications of the polymer phase into an aqueous phase containing a suitable stabilizer thus forming oil water emulsions. So here, oil and water two immiscible liquids which we are taking for this particular purpose as already I have discussed; next, removal of the organic solvent from the dispersed phase by extractions or evaporation leading to polymer precipitation and formation of the microspheres.

So, in this particular case we can see that we are adding that drug and polymer in organic solvent, then we are putting into some vessels, then we are doing the continuous cheering of oil water emulsions, then we are doing the solvent evaporations and then after that we are getting the recovery of the micro oblique nanoparticles. So, some important factors that must be considered in solvent evaporation techniques are: choice of vehicle phase and solvent of the polymer coating. These choices greatly influence microcapsule properties as well as the choice of the solvent recovery techniques. The solvent evaporation technique is applicable to a wide variety of liquid and solid core materials.

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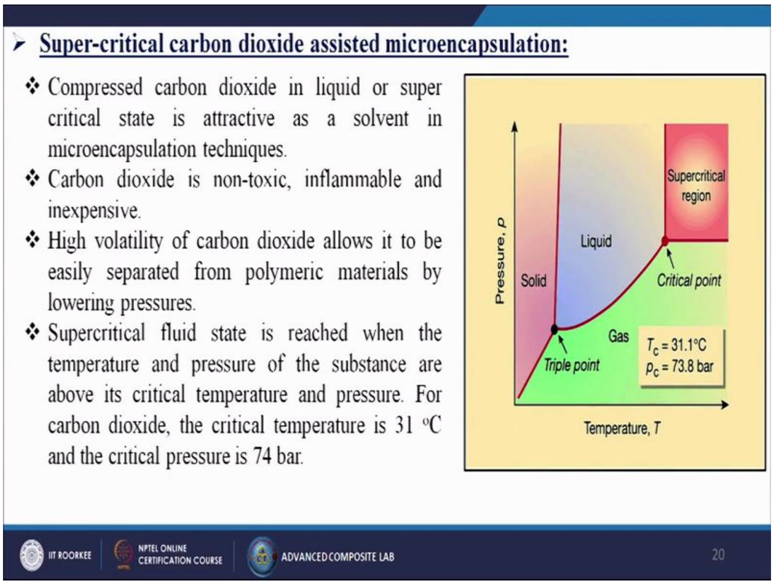

Next is that called the supercritical carbon dioxide assisted microencapsulations. It is also a one kind of latest technology by which we can do this kind of modifications. So, compressed carbon dioxide in liquid or supercritical state is attractive as a solvent in microencapsulation technique. Carbon dioxide is non toxic, inflammable and inexpensive; these all are the added advantage by which we are using the supercritical carbon dioxide. High volatility of carbon dioxide allows it to be easily separated from polymeric materials by lowering pressures. Supercritical fluid state is reached when the temperature and the pressure of the substance are above its critical temperature and pressure. For carbon dioxide the critical temperature is 31 degree centigrade and the critical pressure is 74 bar. So, supercritical is more than this temperature and more than this critical pressure.

So here, if we see the pressure temperature diagram, so we are having the solid phase, we are having the liquid phase, and we are having that gas phase and these points is calling the Supercritical region where we can get the pressure of around 73.8 bar and temperature is around 31.1 degree centigrade. So, beyond that the formation is known as the Supercritical formations.

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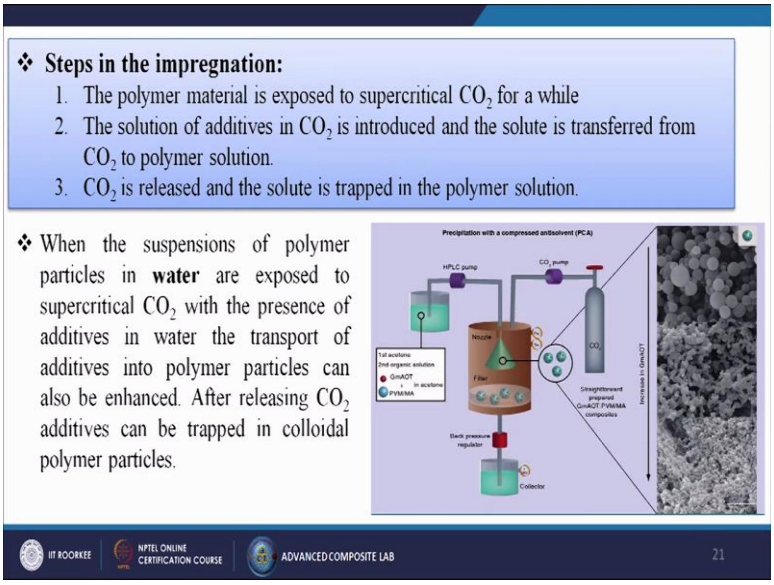

So, steps in the impregnations; how we are going to do this kind of techniques? The polymer material is exposed to supercritical carbon dioxide for a while. The solution of additives in carbon dioxide is introduced and the solute is transferred from carbon dioxide to polymer solutions. Carbon dioxide is released and the solute is trapped in the polymer solutions. So, this is the techniques.

So, here first material we are taking as a GMAOT plus acetone PVM or some kind of MA of polymeric solutions; so first is acetone and second is the organic solutions. Then we are putting it into the supercritical carbon dioxide chamber in which we are making these small small nanoparticles or maybe that microencapsulations matrix material. So here, we are having some kind of collecting materials. And from this particular chamber we are maintaining the supercritical carbon dioxide inside the chamber. And right hand side it is showing the FESA image that how we are getting the final view of that microencapsulation techniques.

When the suspension of polymer particles in water are exposed supercritical carbon dioxide with the presence of additives in water the transport of additives into polymer particles can also be enhanced. After releasing carbon dioxide additives can be trapped in colloidal polymer nanocomposites or maybe that nanoparticle.

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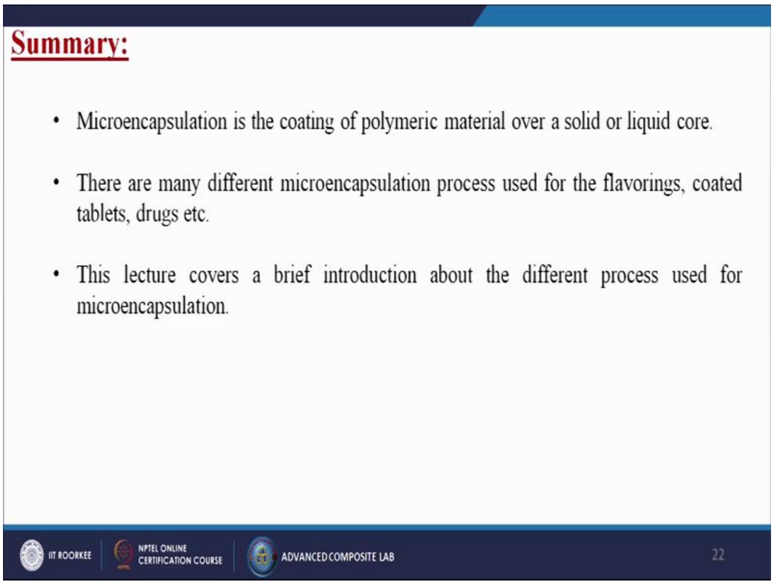

Next is the summary; so here briefly we can tell that what we have discussed this particular topic. So here, actually we have discussed that; what is the microencapsulation techniques, by which method we can do several kind of microencapsulations. So, based on our requirement, based of our materials, based of our core materials, based of our coating materials we can choose the based microencapsulation techniques. And also the lecture covers a brief introduction about the different processing used for the microencapsulations. So, this is the whole summary of the microencapsulation process, what we are doing for surface engineering methods.

Thank you.