Polymer Reaction Engineering
Prof. Shishir Sinha
Department of Chemical Engineering
Indian Institute of Technology – Roorkee

Lecture - 45
Heterogeneous Polymerization Introduction - I

Welcome to the new chapter of heterogeneous polymerization. Now, usually polymerization process is carried out in 2 different phases: heterogeneous and homogeneous. And heterogeneous phase is again very important, especially with respect to the industrial application. So, in this introductory lecture, we are going to give you some insights of the heterogeneous polymerization process.

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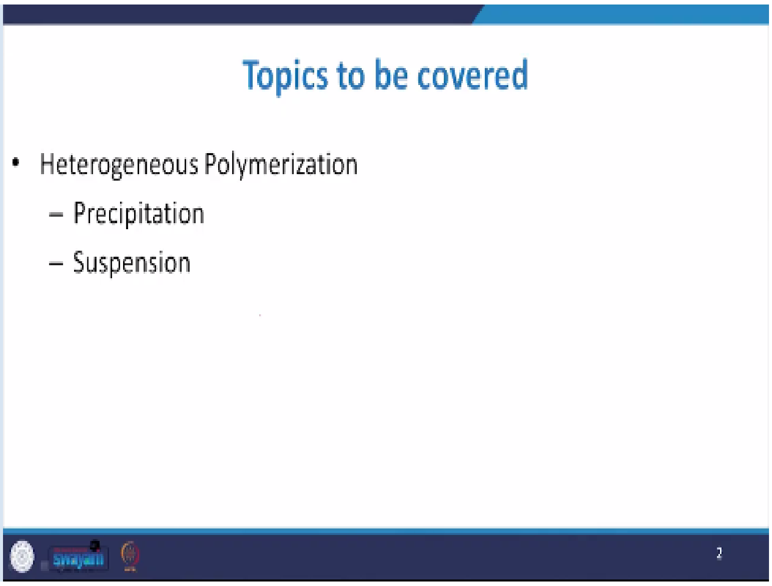

Topics to be covered

- Heterogeneous Polymerization
  - Precipitation
  - Suspension

In this particular chapter, we will discuss the 2 broad spectrum of this heterogeneous polymerization process. One is precipitation and another one is suspension. So, let us have a deep insights of this heterogeneous polymerization. It is commonly used to control the thermal and viscosity issue, if you see that both the issues are extremely important in polymerization process.

And especially, when we talk about the viscosity, it plays a very crucial role in determining the further process-ability of whatever polymer being developed in due course of time. So, when we talk about these heterogeneous polymerization process, so, there are 3 different type of heterogeneous polymerization; one is precipitation, second one is the suspension and emulsion one.

So, all 3 processes, all 3 types are having very good industrial usability with respect to the development of various products for day to day of your life.

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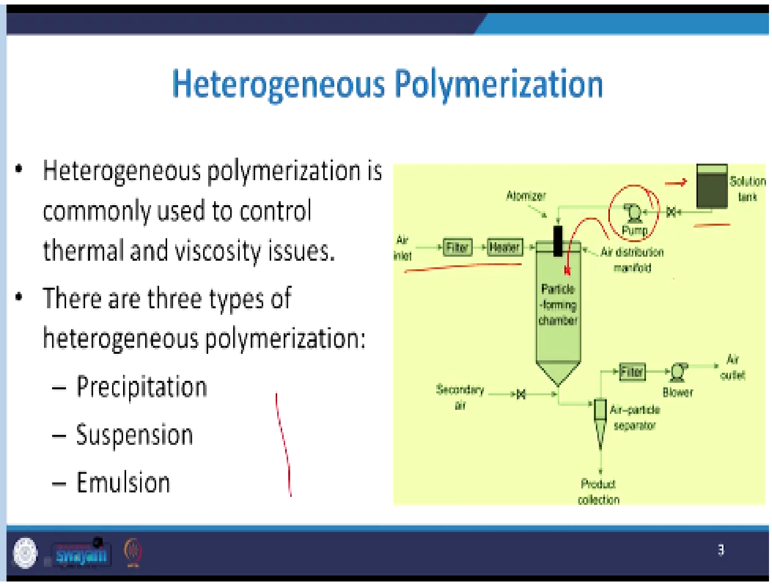

Heterogeneous Polymerization

- Heterogeneous polymerization is commonly used to control thermal and viscosity issues.
- There are three types of heterogeneous polymerization:
  - Precipitation
  - Suspension
  - Emulsion

Now, this is the broad sketch of or rough diagram of the process of heterogeneous polymerization. Here, you have the solution tank in which you are forming the solution with the help of a pump, it carries out to the particle forming chamber. See, initially you may have the substrate, maybe have in the form of a homogeneous system, but once it introduced to the particle forming chamber and with the help of air inlet or some other supporting materials etcetera.

So, it may govern to the small tiny particles, now, this decides, this particular thing decides by this precipitation, suspension or emulsion one. Now, here with the help of this optimizers, it creates a small droplets and it is suspended to this particular chamber. Here, you are having a heated pre-heated air is circulated or subjected to this particle chamber and a secondary air under the counter current direction is subjected to this one.

So, now, over the period of time, when it forms the particle, then it is subjected to the separator. Here, you can have the air particle separation, now, the particles are being formed during the course of the passage of all throughout this particle forming chamber and then with the help of this separator, the exhaust air is usually going out and you can collect the product as per your desired levels.

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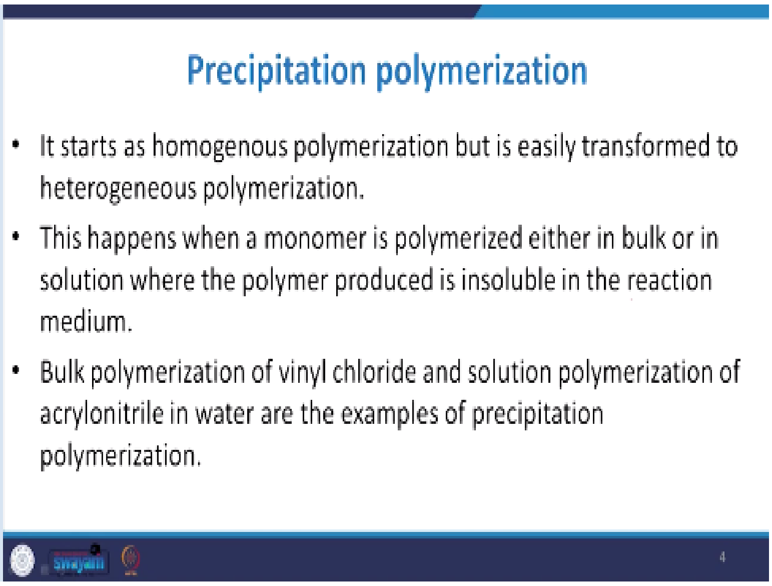

Precipitation polymerization

- It starts as homogenous polymerization but is easily transformed to heterogeneous polymerization.
- This happens when a monomer is polymerized either in bulk or in solution where the polymer produced is insoluble in the reaction medium.
- Bulk polymerization of vinyl chloride and solution polymerization of acrylonitrile in water are the examples of precipitation polymerization.

So, usually people may ask that what is the protocol behind this polymerization scheme. So, let us begin with the precipitation polymerization. Now, the source of this precipitation polymerization start as homogeneous polymerization process, but is easily transformed to the heterogeneous polymerization step or heterogeneous polymers particles like this during the course of this particular thing, it polymerizes over the period of time and then we can discharge like this.

Now, this happens when a monomer is polymerized either in bulk or in solution, where the polymer produced is insoluble in the reaction media. So, the selection of a reaction media is very much important. Now, the bulk polymerization of vinyl chloride and the solution polymerization of acrylonitrile in water are some of the best example of this precipitation polymerization.

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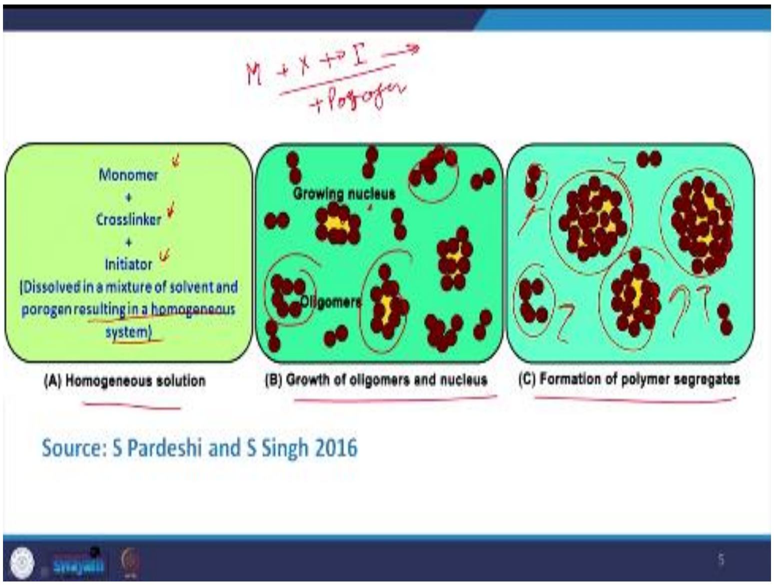

$M + X + I \longrightarrow$
$+ \text{Porogen}$

Monomer
+
Crosslinker
+
Initiator
(Dissolved in a mixture of solvent and porogen resulting in a homogeneous system)

(A) Homogeneous solution (B) Growth of oligomers and nucleus (C) Formation of polymer segregates

Source: S Pardeshi and S Singh 2016

Now, this is, again gives you a very small outlook about how to carry out this precipitation polymerization. Now, here let us talk about say one monomer, let us say vinyl chloride monomer with one cross linking, my intention is to produce the cross link polymerized polymer. Now, here monomer and say cross linker X plus, you may have some initiator, this can produce the things.

Now, here several things need to be noticed or several thing need to be carried out or they require some more attention. Now, since we talk about that initially we may have the solution in the form of a homogeneous aspect. So, in that case, you are having one monomer; you are having one cross linker; you are having the initiator. Now, they should form the homogeneous phase.

So, usually they are dissolved in a mixture of solvent and origins sometimes you may need to create the porosity within the particles, those who are generated in due course of time. So, all another 4 factor that is called the porogen. So, they are resulting in the homogeneous system. So, the affinity among all factors like monomer, cross linker, initiator and porogen system is essential in this part, therefore, you require and affiliating solvent in this structure that things related to the oligomers.

And nuclear growth comes into the picture. Now, here you see that you are having certain growing nucleus over here and they subsequently forms these oligomers over the period of time. So, if you carry on this particular thing in a reactor, then they accumulate the agglomerate because all other things all favourable things are present over here monomer, cross linker, initiator, porogen etcetera.

Then there is a need to form that polymer aggregates or segregates. So, you form the polymer over the period of time, you give some retention time to this particular reaction mass and then you form this segregates and these segregates are thus whatever, segregate, you can remove, so, this is the basic protocol for development of these polymers, development of things under the edges of precipitation polymerization.

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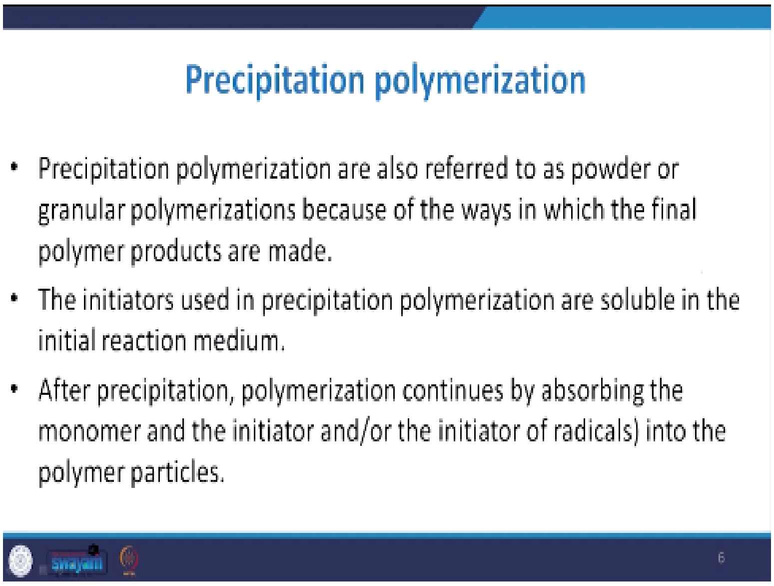

Precipitation polymerization

- Precipitation polymerization are also referred to as powder or granular polymerizations because of the ways in which the final polymer products are made.
- The initiators used in precipitation polymerization are soluble in the initial reaction medium.
- After precipitation, polymerization continues by absorbing the monomer and the initiator and/or the initiator of radicals) into the polymer particles.

Now, this precipitation polymerization are also referred to as powder or a granular polymerization, because the way in which the final polymer products are made. Now, if you see this diagram, here, you see that these kind of polymer particles because it depends on the level of optimization, excessive optimization, you will produce the fine particles and so on. Now, the initiator used in the precipitation polymerization are usually soluble in that initial reaction media.

Now, if you see that previously, we discussed about the concept of initiators and different type of initiators under the head of radical polymerization. So, the initiator role are very much important in this thing, so, that it may get dissolved in the solvent, so, it may have an affinity with the monomer etcetera. Now, after precipitation, the polymerization continues by absorbing the monomer and the initiator or initiate a radical set of whatever in the picture into the polymer particles.

So, thereby, it tends to have an opportunity to grow among themselves. Next is the suspension polymerization.

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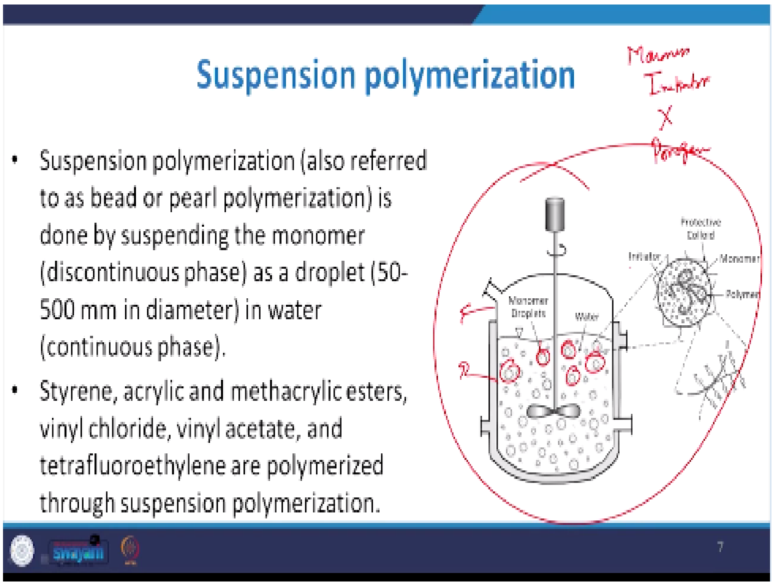

Suspension polymerization

Monomer
Initiator
X
Porogen

- Suspension polymerization (also referred to as bead or pearl polymerization) is done by suspending the monomer (discontinuous phase) as a droplet (50-500 mm in diameter) in water (continuous phase).
- Styrene, acrylic and methacrylic esters, vinyl chloride, vinyl acetate, and tetrafluoroethylene are polymerized through suspension polymerization.

It is a very useful thing to produce the beads or pearl type of particles and these beads are very much popular or very much being useful for the as a support for various capitalist functional groups, as well as they are being used as a support for the various ion exchange protocols. So, they are also referred because it is small size beads and the size of these beads are diameter or spherical size always depends on the end use in which you are going to use all those beads.

So, because of this particular factor, they are also referred as bead or pearl polymerization. Usually, this is performed by suspending the monomer that is continuous phase as a droplet may be 50 to 500 millimetre in diameter in water in continuous phase. So, you see here, again you are requiring one say, monomer, then obviously you require some initiator and if you are performing any kind of cross linking polymerization, then some cross linker.

And if you are looking for some enhanced a surface area, then you may require certain porogen. This is the basic reactor. Now, here you may see that the monomer droplets, they are subjected to in the water which is termed as a continuous phase and this is the discontinuous phase and you may have certain initiators and you see that they may form the small tiny particles.

Now, several issues are attributed to this suspension polymerization like sometimes to develop your small tiny droplets, you require to agitate the things vigorously and the size of those particles are sometimes depend on the RPM of this particular agitator, if you are having high

RPM, in that case, you may experience the fine particles, very small fine particles and sometimes it may not solve your purpose. So, it all depends on the end use which you are going to start.

Now, styrene, acrylic and methacrylic esters, vinyl chloride, vinyl acetate, tetrafluoroethylene, they are polymerized to through the suspension polymerization. Now, usually, waters to monomer weight ratio ranges from 1:1 to 4:1 in most polymerization Again, it all depends on the end use and the size of the particle you require forever your purpose.

So, if you require some different size, then you can vary this ratio. Now, the monomer droplets which are then converted into the polymer particles, now, see, this is again a very crucial part. Now, usually these are stopped from coalescing by friction and the presence of suspension stabilisers, sometimes referred as a surfactant or dispersant. Because, see, these monomer droplets, they are having a tendency to agglomerate or to coalesce each other.

So, that if you are not having this kind of dispersant or surfactant in the reaction mass, then in that case, they may form a big lump and entire irrespective of your agitation, irrespective of your rotating speed, they may form the lumps and sometimes, it may not be feasible to use those lumps for subsequent processes, because you are very much requiring the size of those particles being generated in the suspension polymerization.

So, this particular thing is again important. Now, another thing is that once you have these droplets being generated or the polymer particles being generated, then obviously, you require certain mechanical strength to those polymer particles. Now, you can achieve these mechanical strength by changing the various parameters, maybe the rotating speed, maybe you may enhance that temperature.

So, that these polymer particles may have some mechanical strength. Now, we were talking about the dispersant and surfactant. So, there are 2 type of stabilisers are used or dispersant are used. One, they are having the water soluble polymers often in the presence of certain electrolytes or buffers and some others are like water insoluble inorganic compounds. So, they may form a protective coating across the generated polymer particles so, that they this inhibits the joining of those degenerated polymer particles in due course of time.

Now, the water soluble type, they may include certain the polyvinyl alcohol, sometimes hydroxypropyl cellulose, sodium polystyrene sulfonate, sodium salt of acrylic acid, acrylic esters, co-polymers etcetera. The list of water insoluble type includes teta-hydroxy compounds the barium sulphate purely in magnesium carbonate, hydroxide, calcium phosphate, aluminium hydroxides etcetera and they do not participate in the regular mode of polymerization.

So, you can get rid of all these things which are present in the excess by simply washing and drying. Now, certain initiators because we are discussing about the importance of initiation. So, certain initiators are also used in the suspension polymerization for the ease of the reaction. Now, these are usually soluble in a monomer droplets. So, these initiators are also referred as oil soluble initiator.

Now, each monomer droplet in as a suspension polymerization is considered to be the miniature bulk polymerization system. So, whatever monomial droplets being formed in due course of time, they are considered as a small or miniature bulk polymerization system. So, the kinetics of a polymerization of within each droplet, it is the same as for the subsequent bulk polymerization.

So, you can say, it is a small replica of your bulk polymerization process. Now, sometimes, you may have certain inverse suspension polymerization. Now, it involves an organic solvent as the continuous phase with the droplet of a water soluble monomer sometimes, like example of acrylamide, they are either neat or dissolved in water. There are certain micro suspension polymerization.

Now, these are suspension polymerization in which the size of monomer droplet is about 1 millimetre. So, these 2 are again very important type of a suspension polymerization thing. Now, let us talk about the micro suspension polymerization.

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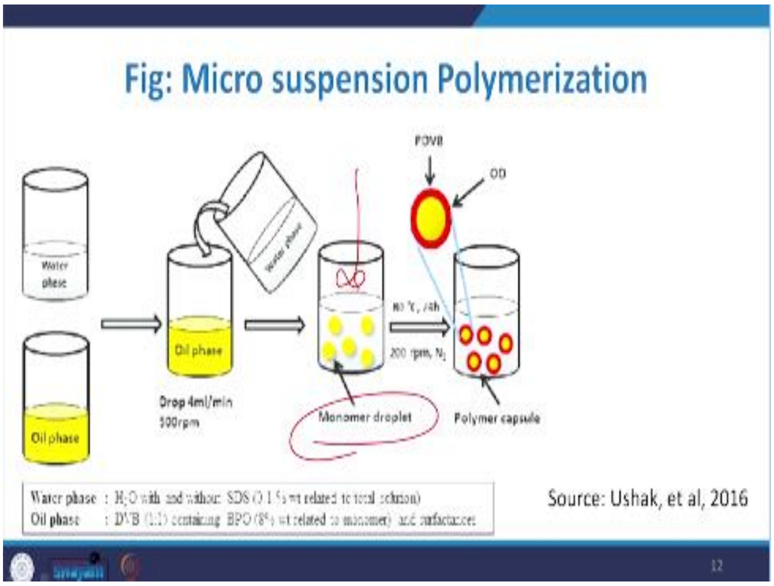

Fig: Micro suspension Polymerization

Water phase : H_2O with and without SDS (0.1 % wt related to total solution)
Oil phase : DVB (1:1) containing BPO (8% wt related to monomer) and surfactants

Source: Ushak, et al, 2016

You see, the protocol for this micro suspension polymerization or suspension polymerization is a very important that usually we may have 2 different phases; sometimes, you may refer these phases as inorganic phase; sometimes maybe inorganic and organic phase; sometimes, like water phase and oil phase. So, let us give the example of say water phase and the oil phase.

Now, here, the water phase and oil phase and then the water phase is mixed with the oil phase. Now, water phase may consist of water and with or without with the surface agent and the oil phase maybe the diagonal benze, cross linking agents and other components. So, you give sufficient time and sometimes, you may introduce the agitator. So, the monomer droplets may get formed and over the period of time if you add, then you may experience the formation of polymer capsules or a polymer in bulk.

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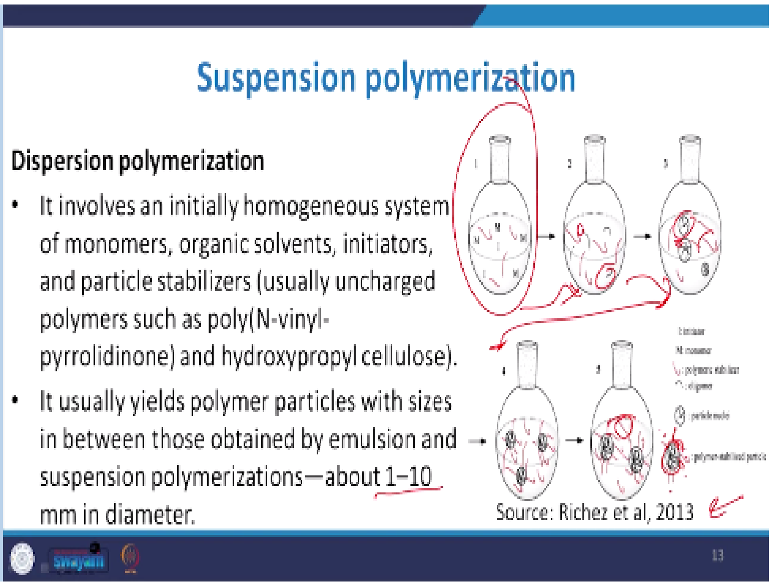

Suspension polymerization

Dispersion polymerization

- It involves an initially homogeneous system of monomers, organic solvents, initiators, and particle stabilizers (usually uncharged polymers such as poly(N-vinyl-pyrrolidinone) and hydroxypropyl cellulose).
- It usually yields polymer particles with sizes in between those obtained by emulsion and suspension polymerizations—about 1–10 mm in diameter.

Source: Richez et al, 2013

Now, let us have a look about the dispersion polymerization. So, it involves and initially homogeneous system of monomer, organic solvent, initiator and particularly stabilisers. Usually, this is uncharged polymers like poly-vinyl pyrolidinone and hydroxypropyl cellulose. So, these are the some particle stabilisers. Now, it usually yields polymer particles with the size in between those obtained by the emulsion and suspension polymerization and say about 1 to 10 millimetre in diameter.

So, this is the various steps. We have taken it from this particular source. So, initially, you are having monomer and initiator within the reaction mass and then they carry out, thye form certain chains and over the period of time, it tends to agglomerate. Now, these are the certain you see that there are certain stabilisers, then these are the oligomers, you see over here and this red colour things are polymer stabilisers. So, you see that here, this is the oligomer and it is duly covered up by the polymeric stabilisers.

So, that is why it prevents the agglomeration of these polymer particles. So, that you may avoid or you may prevent the formation of a large chunk or large molecule. Now, let us talk about the free radical suspension polymerization. Now, it is commonly employed for producing a wide variety of commercially available important polymers like poly-vinyl chloride, PVC, polystyrene, sometimes expandable polystyrene, high impact polystyrene, various styrene co-polymers with acrylonitrile and acrylonitrile polybutadiene.

So, these are the having these polymers, they are having vast industrial as well as the domestic application in our day to day affair. So, in general, this suspension polymerization, we can distinguish into 2 different type. One is the bead type of suspension polymerization. Another one is the powder type of suspension polymerization. So, if we talk about the bead suspension polymerization, the polymer is soluble in its monomer and a smooth spherical part particles are formed like this one.

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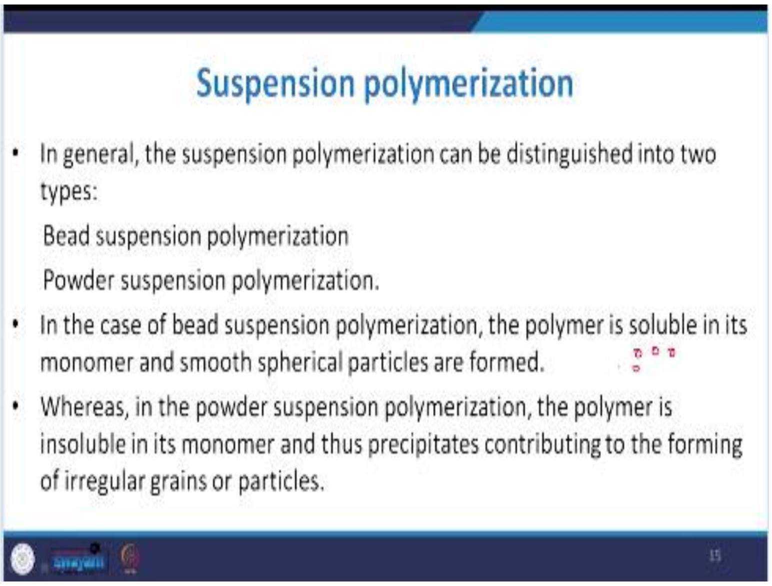

Suspension polymerization

- In general, the suspension polymerization can be distinguished into two types:
  Bead suspension polymerization
  Powder suspension polymerization.
- In the case of bead suspension polymerization, the polymer is soluble in its monomer and smooth spherical particles are formed.
- Whereas, in the powder suspension polymerization, the polymer is insoluble in its monomer and thus precipitates contributing to the forming of irregular grains or particles.

Now, in powder suspension polymerization, the polymer is insoluble in its monomer therefore, the precipitates contributing to the forming of irregular grains or particles. So, you do not have any control over the grain size or the particle size, but here in the beads suspension polymerization, you may have a proper sized spherical part particles. Now, let us talk about the advantages and disadvantages of this suspension polymerization.

Now, the major factor or major benefit of suspension polymerization relative to the bulk phase, they are easier to regulate of the temperature and the reaction due to the availability of dispersant media. So, if you recall that we talked about dispersant media may be water in that case, so, you may have a larger size of the dispersant media so, they have enough opportunity to grow within that dispersant media.

On the other side, the major drawback of a suspension polymerization process, this is attributed to the poor efficiency of the reactor due to existence of dispersant media sometimes, it may be bigger up to say 50% volume by volume and unnecessary post-treatment of a dispersant medium to eliminate any unnecessary impurities like suspending agent.

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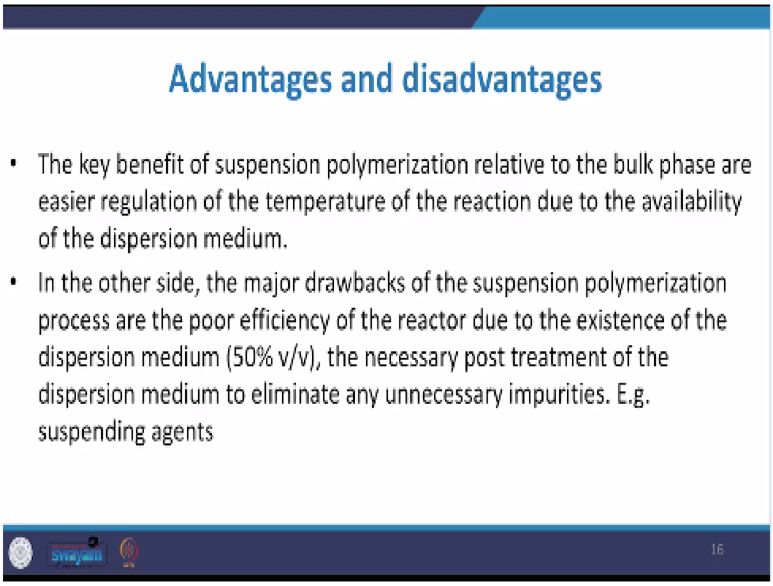

Advantages and disadvantages

- The key benefit of suspension polymerization relative to the bulk phase are easier regulation of the temperature of the reaction due to the availability of the dispersion medium.
- In the other side, the major drawbacks of the suspension polymerization process are the poor efficiency of the reactor due to the existence of the dispersion medium (50% v/v), the necessary post treatment of the dispersion medium to eliminate any unnecessary impurities. E.g. suspending agents

So, if you recall that when we talk about the stabiliser the concept of a stabiliser, then sometimes, you may not be in a position to control the stabilisers, in that case, you need to remove and you need to get rid off all those stabilisers over the period of time because they may form a coat over the surface of the particles being generated in due course of time. So, you need to carry out the elimination process for those suspending agents.

Sometimes, certain un-reacted because you have a component because you are having dispersant, sizable quantity of dispersant medium that is 50% V/V. So, you need to get rid of all those un-reacted things. So, these are the some, this is one of the major drawback attributed to the suspension polymerization. Now, let us have a look about the surface active agents, the concept of surface active agent.

Now, the surface active agents, they play a very important role in stabilisation of liquid-liquid dispersion. They can be water soluble co-polymers like poly-vinyl alcohol or they may be have a cellulose esters or they may have the collider inorganic powders like Pickering dispersants like tri-calcium phosphate, barium sulfate, calcium carbonate etcetera. So, you are having very wide spectrum of these surface active agent.

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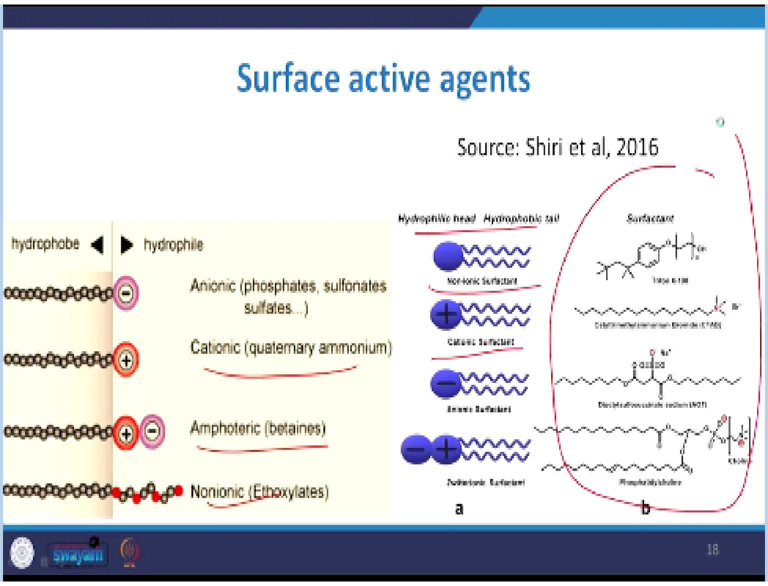

Surface active agents

Source: Shiri et al, 2016

Now, some of the surface active agents, we have enlisted like they are having the hydrophile, hydrophillic nature like an ionic phosphates, sulfonates etcetera, cationic, then amphoteric, non-ionic. So, if you see that these because surface active agents, when they are dispersed in the media, they may have a head and tail type of concept. So, these are certain hydrophilic head and the hydrophobic tail like non-ionic surfactant, cationic surfactant and ionic surfactant and these are the some of the commercially available surfactant being used in the polymerization process.

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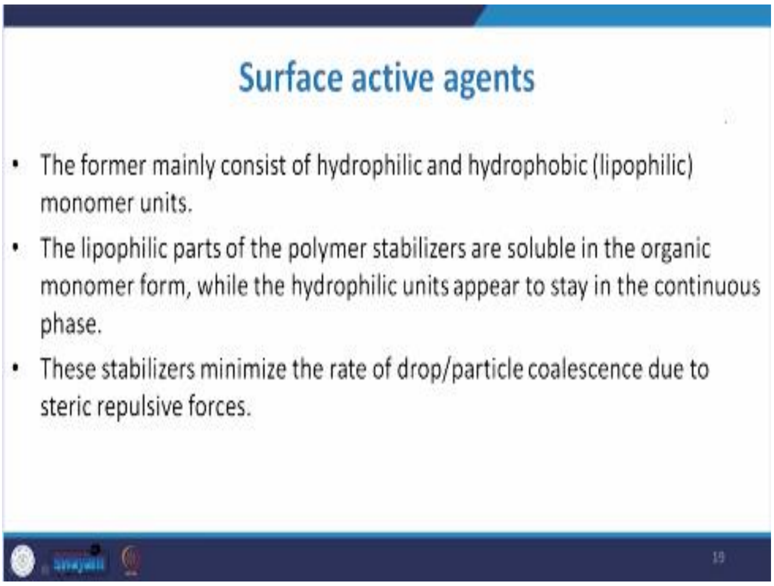

Surface active agents

- The former mainly consist of hydrophilic and hydrophobic (lipophilic) monomer units.
- The lipophilic parts of the polymer stabilizers are soluble in the organic monomer form, while the hydrophilic units appear to stay in the continuous phase.
- These stabilizers minimize the rate of drop/particle coalescence due to steric repulsive forces.

This hydrophobic, they are consist of hydrophilic and hydrophobic system like lipophilic or monomer units. Now, the lipophilic part of the polymer is stabilisers, they are soluble in organic form, while the hydrophilic unit appear to stay in the continuous phase. Now, these stabilisers usually minimise the rate of drop particle coalescence due to the steric repulsive forces. So, what they used to do?

They used to deposit over the surface and then they prevent the further agglomeration of those polymeric chain to the surface of the polymer particles being formed in due course of suspension polymerization process. Now, one of the most widely used stabilizer in the suspension polymerization is poly-vinyl acetate and which has been partly hydrolyzed to poly-vinyl alcohol.

So, by varying the acetate content that is sometimes referred as hydrolysis, we can alter the hydrophobicity of poly-vinyl alcohol and therefore, the conformation and surface activity of polymer chain at the monomer water interface. So, you are having better opportunity to alter this thing so, that you can have the desired result under the edges of suspension polymerization.

Now, the solubility of poly-vinyl alcohol in water, it depends on overall degree of polymerization, sometimes attributed to the molecular weight. So, the sequence chain length distribution of this vinyl alcohol and vinyl acetate units in co-polymer and the degree of hydrolysis and temperature. So, as I described that agitation rate plays a very vital role. So, depending on the agitation rate, the concentration and the type of surface active agent, the average droplet size can exhibit, maybe in the U shape variation with respect to the agitation speed.

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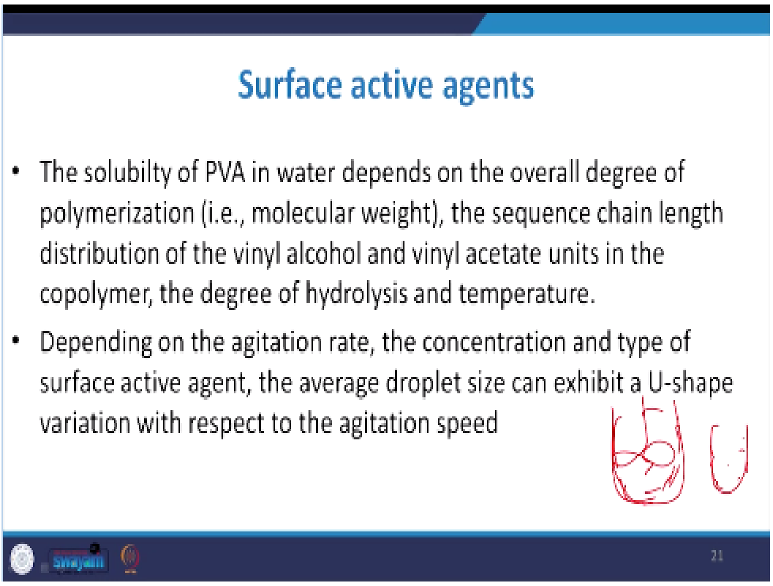

Surface active agents

- The solubilty of PVA in water depends on the overall degree of polymerization (i.e., molecular weight), the sequence chain length distribution of the vinyl alcohol and vinyl acetate units in the copolymer, the degree of hydrolysis and temperature.
- Depending on the agitation rate, the concentration and type of surface active agent, the average droplet size can exhibit a U-shape variation with respect to the agitation speed

So, what is this U shape means, suppose, you are having agitation and all the reaction mass agglomerate, so, you may form this type of a structure and sometimes, it may get deposited over the wall of your reactor. So, this is an very important thing you need to be addressed during the course of this suspension polymerization.

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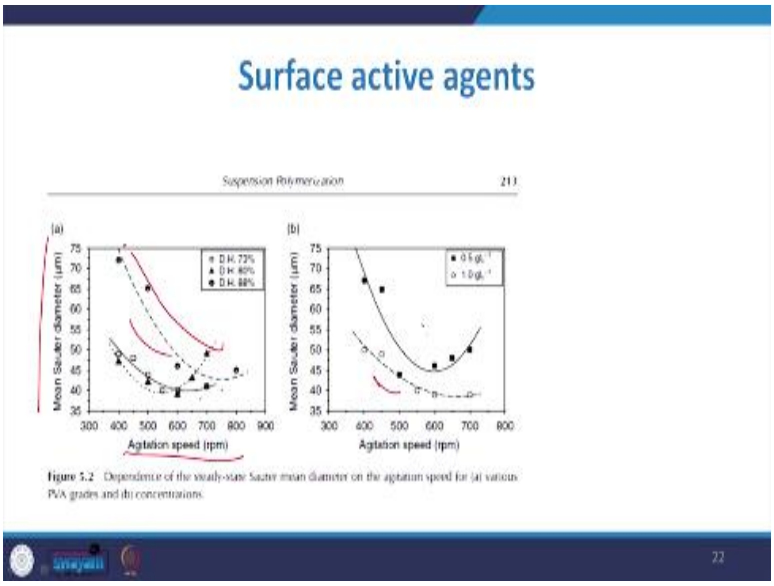

Surface active agents

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Figure 5.2 Dependence of the steady-state Sauter mean diameter on the agitation speed for (a) various PVA grades and (b) concentrations.

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You can see that here the rule of suspension polymerization, the agitation speed and sometimes, means saturation diameter. So, you can see that different components are dependency of various steady state mean diameter. So, there is a variation across the time when you change the agitation speed. So, that is why agitation is speed plays a very, very vital role. Now, let us have a look about the mixing phenomena.

Now, it is a very important thing and it has long been known that some of the key factors influencing the particle size density and sometimes, referred as PSD in the suspension polymerization reactor, they are the geometry that is purely based on the geometry of the reactor, the form, the size and the bottom clearance of the impeller. Now, sometimes people may get confused over the, what is the bottom clearance of the impeller.

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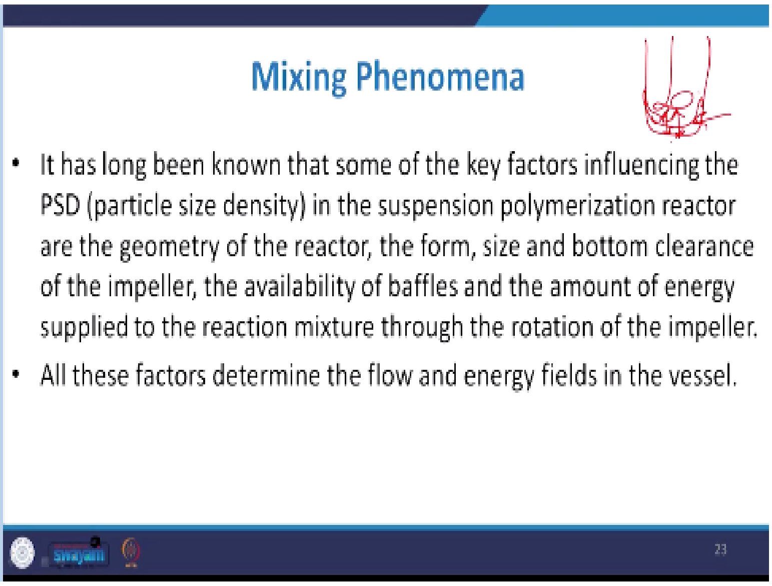

Mixing Phenomena

- It has long been known that some of the key factors influencing the PSD (particle size density) in the suspension polymerization reactor are the geometry of the reactor, the form, size and bottom clearance of the impeller, the availability of baffles and the amount of energy supplied to the reaction mixture through the rotation of the impeller.
- All these factors determine the flow and energy fields in the vessel.

It all depends on this particular distance that is how much it is having and so, sometimes whenever you need to look at the mixing rate or mixing speed, you need to look into that that there are certain formation of a stagnant zone within this range. So, this is the bottom clearance of impeller and sometimes availability of baffles, the amount of energy supplied to the reaction mixture through the rotation of the impeller.

So, all these factors determine the flow and energy field in the vessel. Now, to characterise the quality of mixing in a suspension polymerization reactor using a single parameter, the average rate of energy dissipation is again plays a very vital role sometimes referred to as epsilon.

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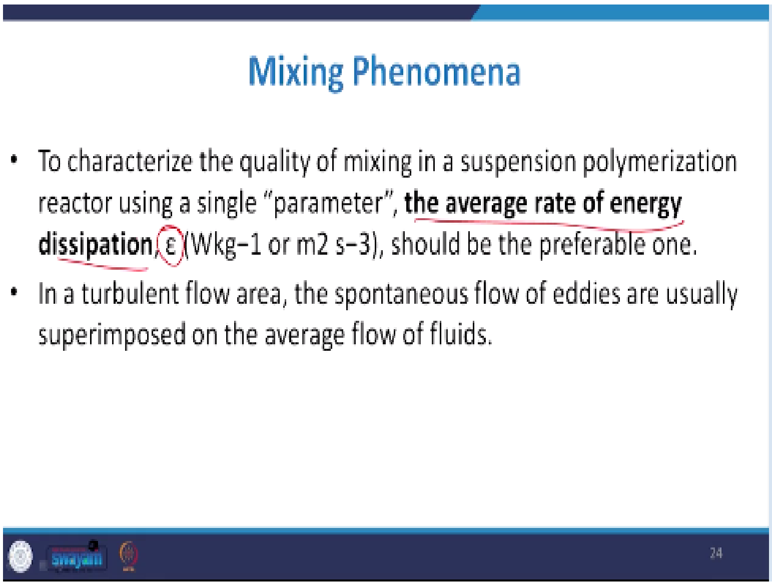

Mixing Phenomena

- To characterize the quality of mixing in a suspension polymerization reactor using a single "parameter", **the average rate of energy dissipation**, ε (Wkg−1 or m2 s−3), should be the preferable one.
- In a turbulent flow area, the spontaneous flow of eddies are usually superimposed on the average flow of fluids.

And it is having the unit of WKg^{-1} or m^2s^{-3}. This should be this particular thing that characterization is should be the preferable one. Now, in a turbulent flow area, the spontaneous flow of Eddies are usually superimposed over the average flow of fluids. So, sometimes, if we talk about that turbulent flow field in an agitated vessel, the average energy dissipation rate per unit mass maybe equal to the power input into the system P which is referred as here then per unit mass.

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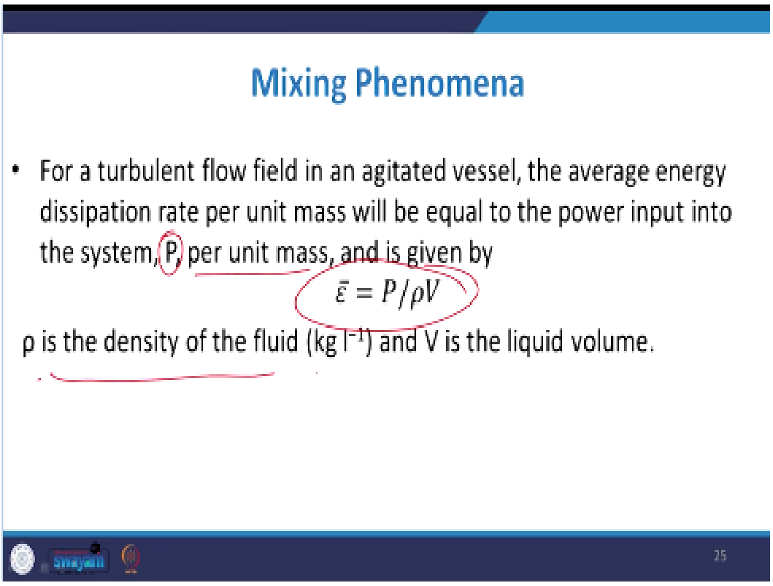

Mixing Phenomena

- For a turbulent flow field in an agitated vessel, the average energy dissipation rate per unit mass will be equal to the power input into the system, P, per unit mass, and is given by

$$\bar{\varepsilon} = P/\rho V$$

ρ is the density of the fluid (kg l^{-1}) and V is the liquid volume.

And it is usually given by

$$\bar{\varepsilon} = P/\rho V$$

Where, ρ is the density of the fluid (kg l^{-1}) and V is the liquid volume, whatever liquid volume being used in the suspension polymerization process.

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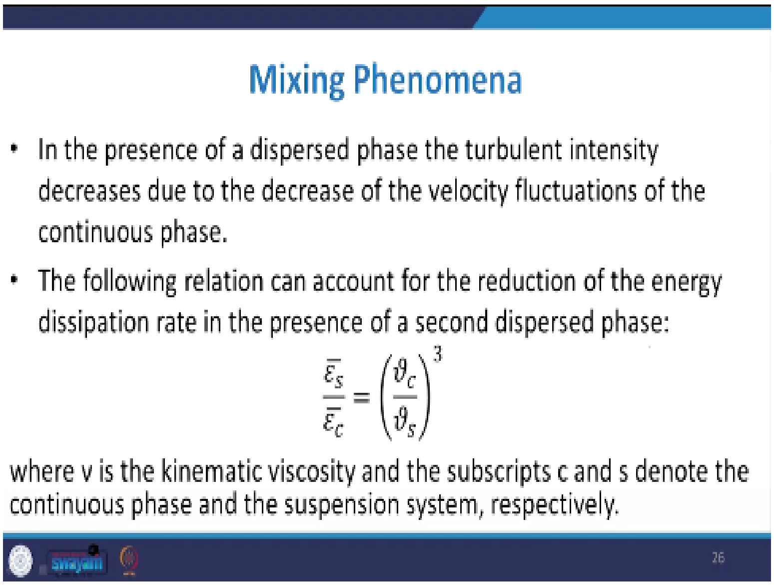

Mixing Phenomena

- In the presence of a dispersed phase the turbulent intensity decreases due to the decrease of the velocity fluctuations of the continuous phase.
- The following relation can account for the reduction of the energy dissipation rate in the presence of a second dispersed phase:

$$\frac{\bar{\varepsilon}_s}{\bar{\varepsilon}_c} = \left(\frac{\vartheta_c}{\vartheta_s}\right)^3$$

where v is the kinematic viscosity and the subscripts c and s denote the continuous phase and the suspension system, respectively.

So, in the presence of a dispersed phase in that turbulent intensity decreases, now, due to this decrease in the velocity fluctuations of the continuous phase. So, this particular relation can account for the reduction of the energy dissipation rate in the presence of second dispersed phase in suspension polymerization.

$$\frac{\overline{\varepsilon_s}}{\overline{\varepsilon_c}} = \left(\frac{\vartheta_c}{\vartheta_s}\right)^3$$

where ν is the kinematic viscosity and the subscripts c and s denote the continuous phase and the suspension system, respectively.

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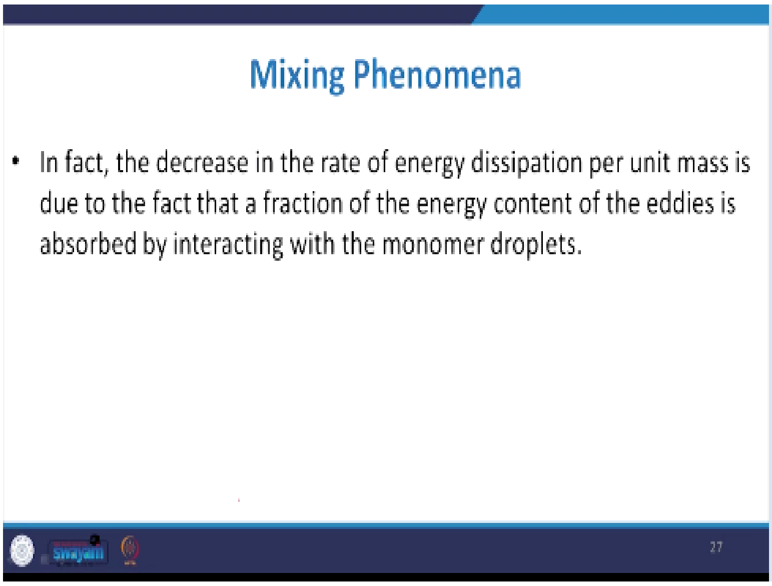

Mixing Phenomena

- In fact, the decrease in the rate of energy dissipation per unit mass is due to the fact that a fraction of the energy content of the eddies is absorbed by interacting with the monomer droplets.

Now, in fact, whenever we talk about the mixing phenomena, the decrease in the rate of energy dissipation per unit mass is attributed to the fact that fraction of the energy content of the Eddies is absorbed by interacting with the monomer droplets. So, whatever monomer droplets are there, then certain quantity of energy being absorbed by interacting those droplets.

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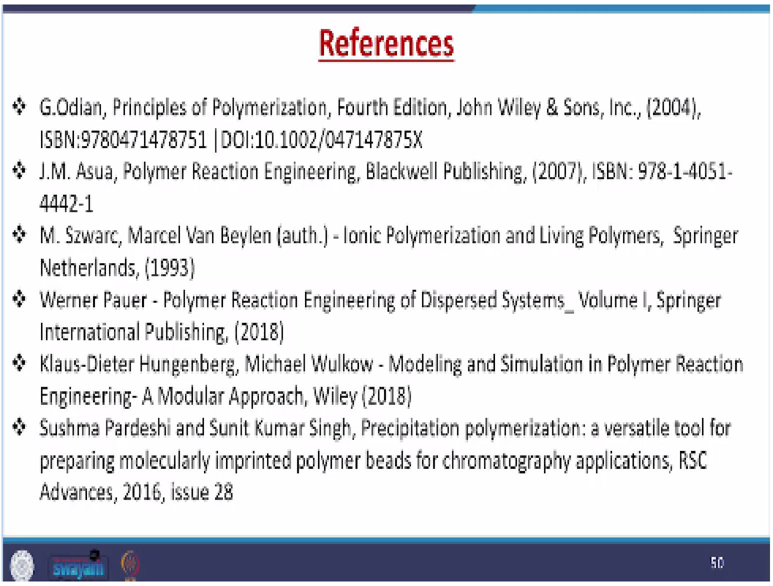

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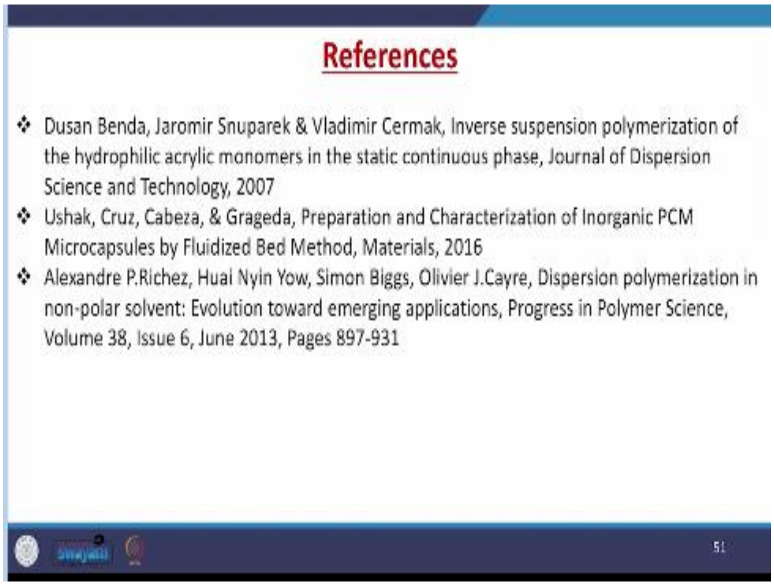

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So, in this particular lecture, we discussed the various aspects of suspension polymerization, different parameters associated with this suspension polymerization. We discussed about the mixing phenomena attributed to this suspension polymerization and what are the role of different ingredients in suspension polymerization protocol. We will continue this particular approach in the next lecture. Thank you very much.