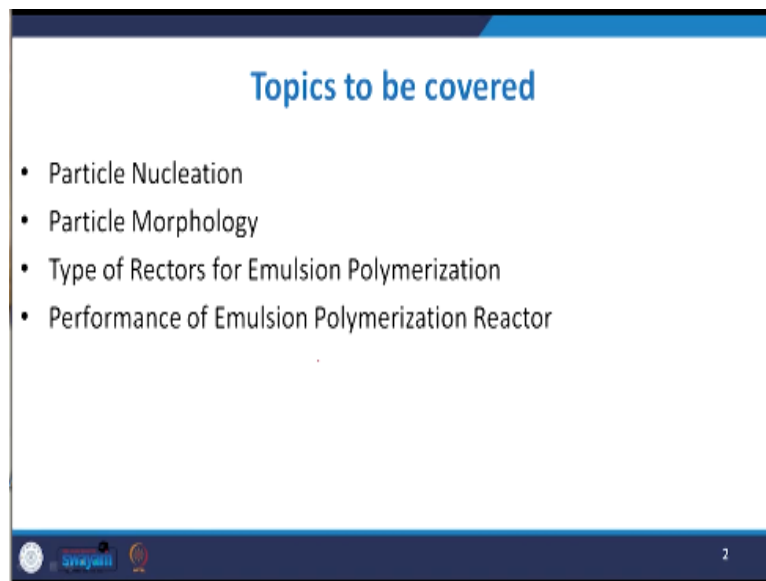


Polymer Reaction Engineering
Prof. Shishir Sinha
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Indian Institute of Technology – Roorkee

Lecture - 52
Nucleation, Morphology and Reactor Types - II

Welcome to the second part of the nucleation, morphology and reactor type chapter under the head of emulsion polymerization. We discussed in the particle nucleation and we started the particle morphology in the previous lecture. In this lecture, we will continue this particle morphology concept.

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Apart from this, we will discuss about the various type of reactors involved for the emulsion polymerization and we will perform the performance study of emulsion polymerization reactor.

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Particle morphology

- After this separation, the polymerization reaction can be found to occur at both regions i.e. at the cluster as well as in the polymer matrix.
- Due to the involvement of large polymer-polymer interfacial area from the developing polymers, thermodynamic imbalance will develop which leads to increase in the free energy.
- Hence, polymer migration towards the equilibrium morphology will start happening to reduce the free energy of the system.



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just a brief overlook that what we discussed in the previous lecture that we were at the phase of particle morphology. when there was a separation in the polymerization process, this can be found in different regions like cluster as well as in the polymer matrix. And we talked about the thermodynamic imbalance, which intend to develop during the course of this polymerization and subsequently it may increase the free energy.

Therefore, there may be a chances of polymer migration towards the equilibrium morphology and this may create several issues. this polymer migration, this results to increase in the size of the cluster. This may be due to the polymerization within the cluster, diffusion of polymer into the cluster, coagulation with other clusters.

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Particle morphology

- polymer migration results to the increase in the size of the cluster due to:
 - polymerization in the cluster
 - diffusion of polymer into the cluster
 - coagulation with other clusters



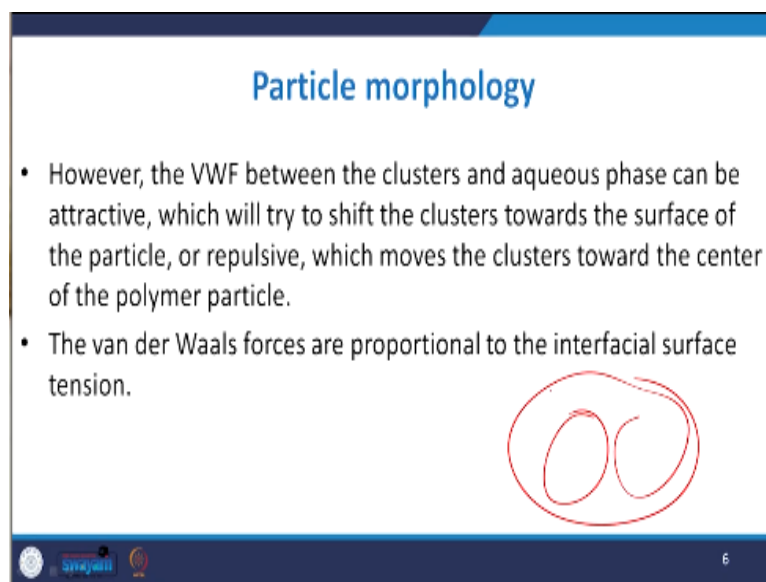
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let me give you a brief outline about this, because if you see that we have formed one hollow network where the things are attached with respect and then there was a starting of polymerization. And this cluster become more and more bigger in size and then the polymer migration took place. So, these developed polymers sites over the cluster may intend to form the further polymerization within this cluster.

Then there may be chance that the polymer particle may get diffused into the cluster. And then they may be formed that there may be chances that they may form the coagulation with the other clusters. So, these are the several issues related to this particle morphology. So, whenever these 3, any of these 3 situation occurs, then there may be a change in the morphological segments of these polymer particles.

usually when we talk about the migration then question arises that who tutored this migration. So, this migration is basically tutored by 2 interfacial forces. One is that is the van der Waals forces of attraction and repulsion is usually a very weak forces. Then second is the resistance to flow that arises from the viscous drag. these van der Waals forces between the clusters always try to attract each other. So, that is one of the reason why this migration is tutored.

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Particle morphology

- However, the VWF between the clusters and aqueous phase can be attractive, which will try to shift the clusters towards the surface of the particle, or repulsive, which moves the clusters toward the center of the polymer particle.
- The van der Waals forces are proportional to the interfacial surface tension.

The slide includes a diagram of two overlapping circles drawn in red, representing clusters or particles. At the bottom left, there are logos for 'swayam' and 'swayam' with a '6' in the bottom right corner.

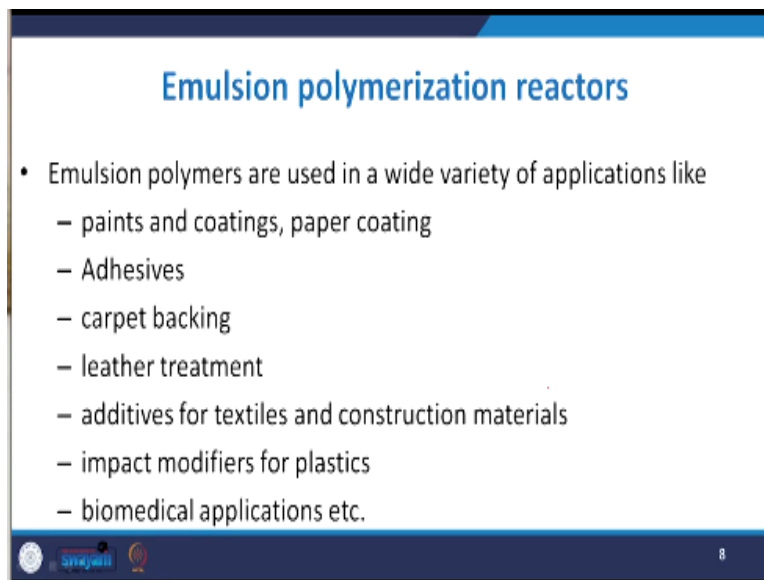
However, these van der Waals forces between the cluster and aqueous phase, this is the cluster and the aqueous phase. This can be attractive, which will try to shift the cluster towards the surface of the particle and sometimes to be repulsive. So, this moves the cluster towards the centre of the polymer particle. these van der Waals forces, they are proportional to the

interfacial tension. And this interfacial tension is again a very important role in the migration tendency.

if the internal viscosity of the particles are high, then meta-stable morphology will be obtained. this low interfacial tension will promote the grafting reaction. So, if you are intended to perform this grafting aspect, then it is a very useful platform. on the other hand for the case of particles with the low internal viscosity, equilibrium morphology can be achieved.

So, the polymer obtained this could be the highly unstable due to the large interfacial surface tension which may lead to increase the van der Waals forces. So, the process time forces system will also be very long, because of this involvement of weak van der Waals forces. let us have a look about the emulsion polymerization reactor that is the next segment of our chapter.

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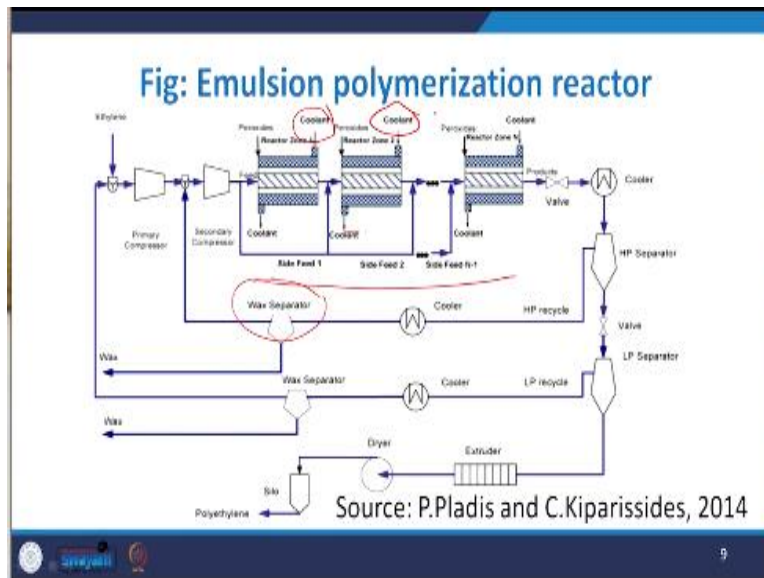


Emulsion polymerization reactors

- Emulsion polymers are used in a wide variety of applications like
 - paints and coatings, paper coating
 - Adhesives
 - carpet backing
 - leather treatment
 - additives for textiles and construction materials
 - impact modifiers for plastics
 - biomedical applications etc.

these emulsion polymerization are used in a wide variety of applications like paint, coating, paper coating, adhesive, carpet backing, leather treatment, additives for textile and the construction material, impact modifiers for plastics, biomedical applications etcetera. So, they are having very wide spectrum of uses.

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here you see that we have represented emulsion polymerization reactor, this is for the production of polyethylene. Here, the ethylene is subjected to this primary compression and secondary compressor. And then there are 2 stage side feeding where the peroxide, these are the initiators and peroxides are added to these different reactor zones. there is a subsequent addition of the coolant because, you know that these reactions are some sort of exothermic in nature.

And if when we do not add these coolant, then there may be a chance of a thermodynamic imbalance which may create a further issues. So, these are the 3 zones, I am not going to detail about these zones related to the things. But, the thing is that this is a typical type of polymerization process reactor. Here, you may have a several coolers. This may tend to reduce the temperature.

Then high pressure separators; then here you are having certain wax separators etcetera. So, these are the some certain side products and by-products.

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Emulsion polymerization reactors

- Each of them requiring a particular microstructure and a different production rate.
- Secondly, emulsion polymers are **products-by-process** whose microstructure is determined in the reactor.

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each of these reactor or each of these zones, they require a particular micro-structure analysis and a different production rate and that is why you see there are different side feed zones. secondly, the emulsion polymers they are product by process, whose micro-structure is determined in the reactor. there are certain prerequisites or certain requisite aspect for these emulsion polymerization reactor.

these are that they should allow the fine tuning of polymer micro-structure that is quite obvious. They should be useful to prepare many different grades in the same reactor and they should be adoptable to a wide range of production rates. So, these are the certain requisite aspect for these emulsion polymerization reactors. these reactors either can be batch type semi-continuous or CSTR.

semi-continuous stirred tank reactor is the most widely used reactor for this emulsion polymerization process. for obtaining very large capacities, a small number of grades, batteries or CSTR are also being used. But due to certain limitations of the process or process requirement, that batch reactors are less frequently or rarely used. tubular reactor are also less popular and only continuous loop reactors are usually used in the commercial approaches.

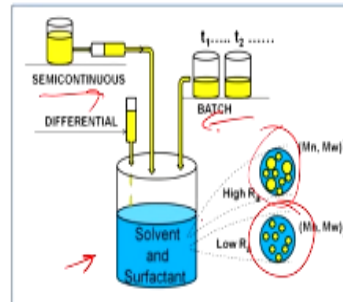
let us have a look about the semi-continuous stirred tank reactor. This process, we have already discussed in the previous lecture.

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Emulsion polymerization reactors

Semicontinuous stirred-tank reactor

- The process is already discussed previously.
- The reason behind the wide acceptability of these reactor is their great flexibility towards the reaction parameters.



Source: Jorge E. Puig and Martín Rabelero, 2016



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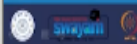
this is you see that this is a solvent and surfactant then these are the semi-continuous batches and you may find different polymers having the different number average and weight average molecular weight. the reason behind the wide acceptability of these reactor is the great flexibility towards the reaction parameters. You know that we have already discussed the various reaction parameters, those who are involved in the emulsion polymerization process.

So, this type of a configuration, semi-continuous type of a configuration offer a great flexibility in with respect to all those parameter and that is why because of this particular governing factor, these reactors are formed themselves. They had a very good candidate for performing this emulsion polymerization process.

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Emulsion polymerization reactors

- The reaction temperature and the polymer composition in the feed can be adjusted depending upon:
 - the composition and the volume of the initial charge
 - the composition and flowrate of the feed
- The major limitation in this operation mode is its comparatively low productivity.



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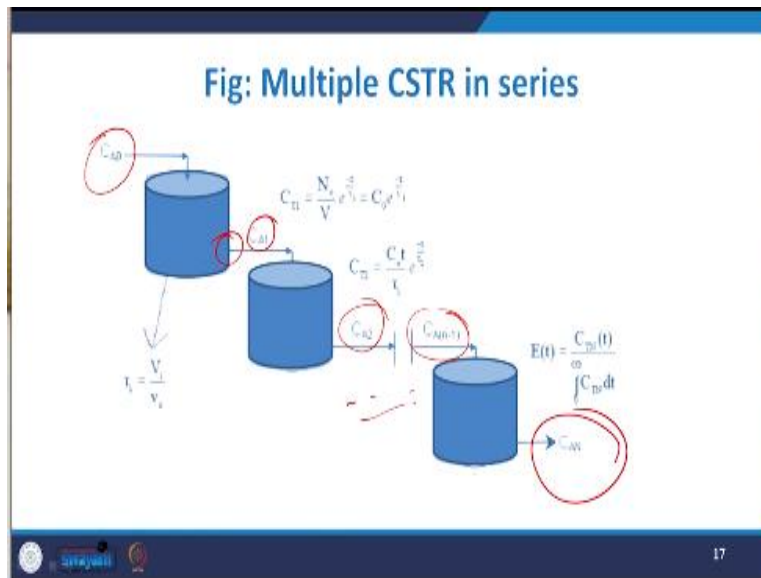
The reaction temperature and the polymer composition in the feed can be adjusted depending upon the composition and the volume of the initial charge. And second aspect in this category is the composition and flow rate of the feed. The major limitation in this operation mode is the comparatively low productivity. So, that is you can say that this one is slight disadvantage of this particular process.

various commercial need polymers, they are manageable through these reactors. As they allow the modification of various unique polymer properties such as sometimes, you may require the copolymer composition to be addressed as per your need, you may require molecular weight distribution approach; you may require different polymer architecture; you may require different particle morphology and a particle size distribution. So, based on your requirement, this particular approach offer a better candidate see.

next is your CSTR that is the continuous stirred tank reactor. this CSTRs, they always grab attention due to the capabilities related to providing the product in a large scale. They have a broad residence time distribution and sometimes in the reaction engineering term referred as RTD. And that is why they are obtaining a narrow particle size distribution in single CSTR that is the quite tough.

incorporating the multiple CSTR in series, this can help to overcome that particular issue with the particle size distribution, because this helps in effective heat removal and narrow residence time distribution inside the reactor. This may, this will lead to obtain narrow particle size distribution.

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you can see this is the multiple CSTR in the series. Here, you are having this initial concentration of your reactant, then the output of this reactor is the input of a second reactor. And then the output of your second reactor is the input of your subsequent reactor. So, you can have n number of reactors in series to get the final product in this concentration.

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Emulsion polymerization reactors

- In fact, commercially it is not feasible to incorporate a large CSTR because of various complexities involved related to the grade transition. Hence, the use of multiple CSTRs in series is quite beneficial in practical sense.
- During polymerization in CSTRs, it is very likely that intermittent nucleation will occur which results to the formation of multimodal PSD.
- To overcome this issue, tubular reactors are usually employed before CSTR, which helps in smoothening the particle formation.

In fact, commercially, it is not at all feasible to incorporate a larger CSTR because the various complexity may involve in due course of time and that is attributed to the great transition etcetera. Therefore, the use of multiple CSTR as reflected in this figure is quite beneficial in the practical approaches. During the polymerization in CSTR, it is very likely that intermittent nucleation may occur, which results to the formation of multimodal particle size distribution.

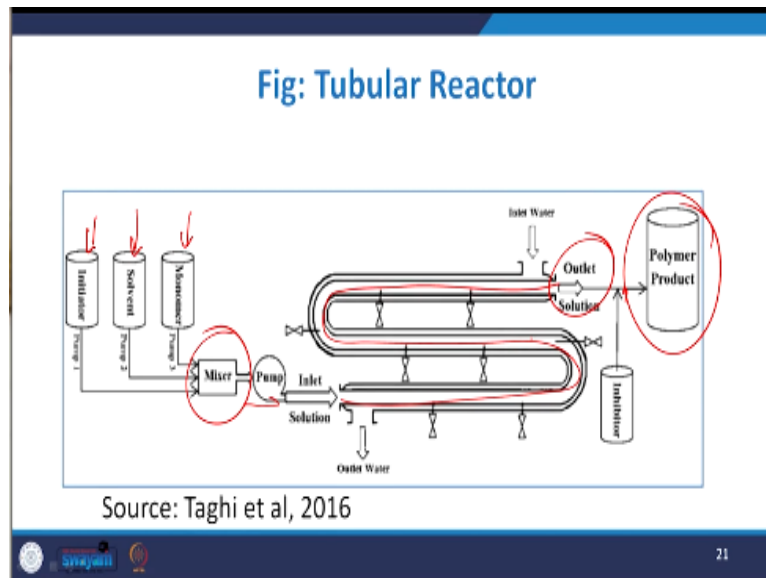
sometimes, we need to overcome such type of issue. The tubular reactors usually are employed before the CSTR in this polymerization process. These reactors, they help in the smoothening the particle formulation. So, you may have a combination of different type of reactor to achieve the desired goal. due to the uniform mixing inside the reactor, the copolymer composition sometimes, remain constant throughout and you may need to perform certain manipulation in this approach.

So, the manipulating the intermediate feed to the monomer, this can also help to control the copolymer composition and molecular weight because both the things are extremely important to carry out the desired product formation. Incorporating the multiple CSTRs, they provide the another advantage of distribution of average residence time among each CSTR attached in the series. So, this will increase the flexibility of the reaction.

So, therefore, based on this the production of variety of speciality emulsion polymerization product in a single setup can be possible. So, they offer more and more flexibility maybe you can use the multiple reactor in combination, you can have a different reactor in series. So, by these permutation and combination, you can achieve your desired product parameters. let us have a brief look about that tubular reactor.

the tubular reactor they provide a very large surface to volume ratio as compared to the other reactors. due to this very high conversion can be achieved in a small volume. Therefore, the size of reactor, the installation space, these can be significantly reduced and economic of your process can be can be improvised. Secondly, these reactor have quite simple mechanical design and they can perform the efficient heat removal. So, due to their compact design sometimes, they found to be quite economical.

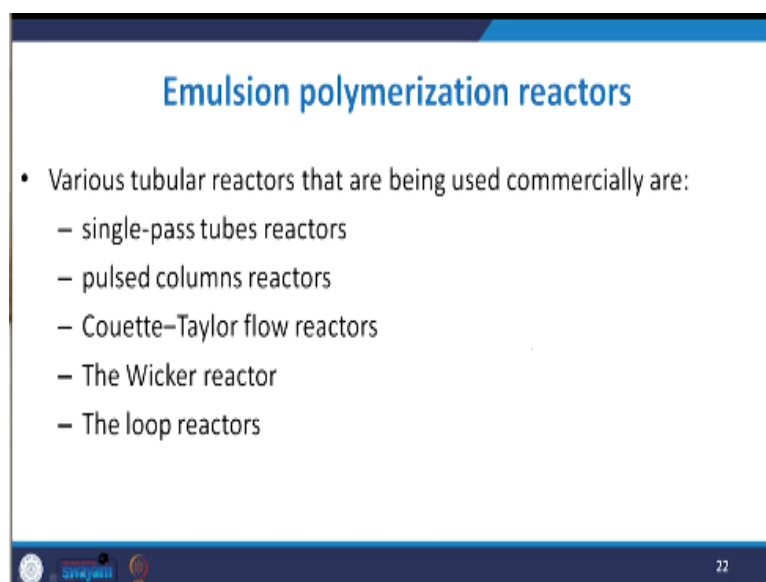
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this is the one of the example of the tubular reactor. Here you see that we are feeding the monomer, we are having the initiator, we are having the solvent, they all together mixed in a mixture. And subsequently, they are pumped to this tubular reactor, where they are performing the former reaction to get the polymeric product in the form of a solution. here at this stage, you can introduce the inhibitor.

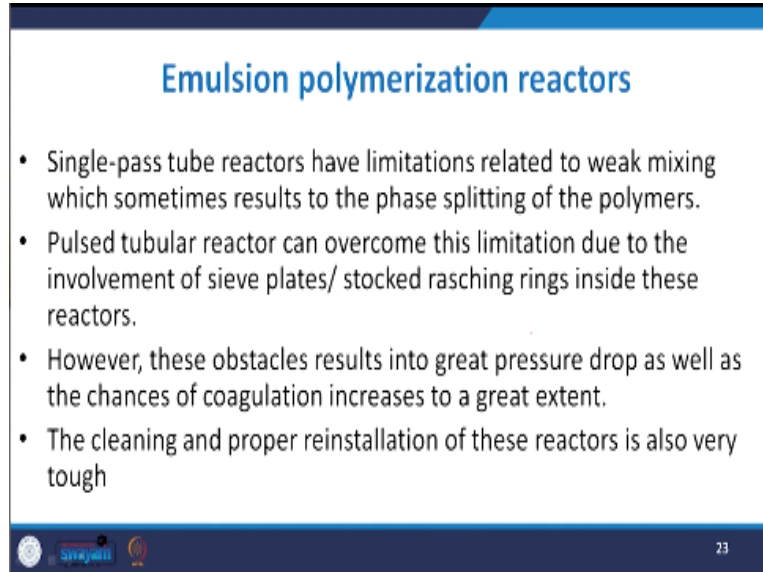
So, that you can truncate the further polymerization reactions. So, that you can control the PSD - particle size distribution, you can maintain the appropriate molecular weight distribution and by this way, you can collect your polymer. So, this is a typical design or typical layout of tubular reactor.

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various tubular reactors are being used commercially, they are quite common. the some of them, we have already listed like a single pass tubular reactor, pulsed column reactor, Couette-Taylor flow reactors, the wicker type of reactor, the loop reactor.

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Emulsion polymerization reactors

- Single-pass tube reactors have limitations related to weak mixing which sometimes results to the phase splitting of the polymers.
- Pulsed tubular reactor can overcome this limitation due to the involvement of sieve plates/ stocked rasching rings inside these reactors.
- However, these obstacles results into great pressure drop as well as the chances of coagulation increases to a great extent.
- The cleaning and proper reinstallation of these reactors is also very tough

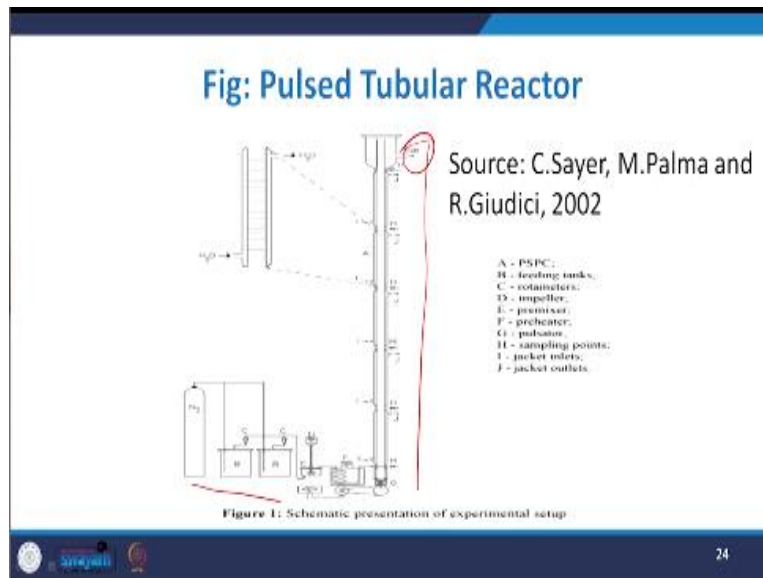
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single pass tubular reactor, just have a brief look at what are the advantages and disadvantages associated with all these kinds of reactors. The single pass tube reactor, they have limitations related to weak mixing which sometimes, result to the phase splitting of the polymer. And sometimes, it may create a problem in the final product or morphologic. The pulsed tubular reactor can overcome this particular limitation which is associated with the single pass tube reactor.

this is attributed due to the involvement of sieve plates, stocked rasching rings inside these reactors So, that they can remove all those splitted particles in due course of time. these obstacles sometimes, result into the great pressure drop as well as the chances of a coagulation. This coagulation sometimes increases to a great extent. So, this is one of the disadvantages associated with this pulsed tubular reactor.

And because of this coagulation approach, the cleaning and proper reinstallation of these reactor are extremely difficult or sometimes, it is very tough and comparison in this approach.

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this is a typical figure of pulsed tubular reactor. You can see this is tubular reactor. Here, you are feeding the different ingredients to this reactor and you are having the outlet of this one. You may have a feeding tank rotameter, impeller, pre-mixers, pre-heaters etcetera.

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Emulsion polymerization reactors

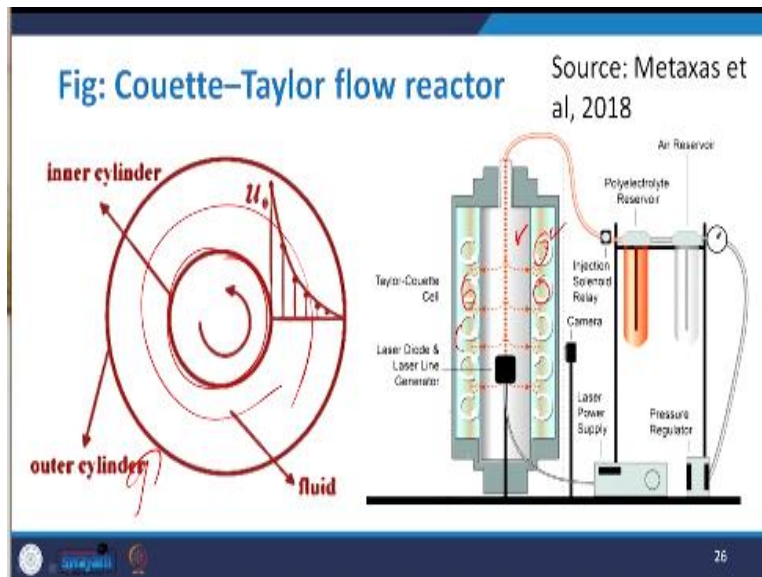
- The **Couette–Taylor flow reactor** is another category in this series which and it consists of two concentric cylinders.
- The immovable outer cylinder is fixed and jacketed for efficient heat transfer, while the inner cylinder rotates to its axis.
- The sample is places between the cylinders and under particular operating condition, counter-rotating toroidal vortices are formed.
- The mechanism of Couette–Taylor flow is similar to that of multiple CSTRs in series and this makes the RTD in these reactor similar.

the Couette-Taylor type of a flow reactor: this is another category in this series which consists of 2 concentric cylinders. The immovable outer cylinder is usually fixed and jacketed for efficient heat transfer, while the inner cylinder rotates to its axis. Sometimes, the samples is placed between the cylinders and under particular operating condition, the counter rotating toroidal vortices are formed.

So, the mechanism of Couette-Taylor flow is similar to the multiple CSTR in series and this makes the residence time distribution in these reactors similar. So, this is one of the added

benefit. here you see, this is the typical diagram of Couette-Taylor flow reactor. You see that there are 2 cylinders. One is this is the inner cylinder and this one is the outer cylinder and the fluid is just rotating like this. Similarly, here larger spectrum, you see that this is the Taylor-Couette cell.

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Now here, this is the inner cylinder and this is the outer one and you see, they are different vertices being formed in this particular cell to facilitate the polymerization reaction.

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Emulsion polymerization reactors

- During polymerization, the Taylor number is prone to change inside the reactor due to the change in viscosity of the solution in linear direction.
- As the viscosity change follows a particular trend, the Taylor number can be managed by modifying the cylindrical outer cylinder with the conical one. This can counteract the viscosity increase factor.

during the polymerization that Taylor number is prone to change inside the reactor due to the change in the viscosity of the solution in a linear direction. So, it is quite evident from this particular figure. as the viscosity change follows a particular trend, the Taylor number can be managed by modifying the cylinder, cylindrical, outer cylinder with the conical one.

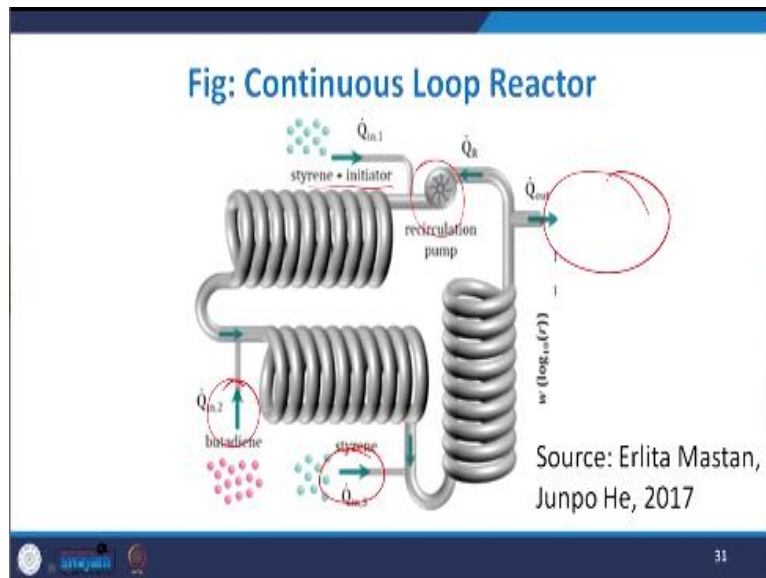
So, this can counteract the viscosity increase factor. Because again viscosity plays a vital role. Sometime, if it increases drastically, then the process may get hampered in due course of time. So, therefore, this is one of the major way through which you can counteract the viscosity increase factor. One factor that induce the coagulation is that gravity induced phase separation that is the creaming sometimes, referred as a creaming.

So, the wicker reactor configuration is found to be quite useful to overcome such type of issue. the tube menders between the fixed cylinder supports, this is the integral part of the wicker reactor. the tubes moves in such a manner that it develops the huge number of curves in the parameter of the cylindrical supports, while maintaining the curvature. these tubes are quiet from the bottom to the upper part of support.

These type of reactors are newly modelled configuration that is found to be an effective solution of coagulation related issues in the emulsion polymerization. However, because of the complexity in nature, there is no practical application commercially has been made as on date for these type of reactor. Another form of reactor is the continuous loop reactor. These can be said as the modified and improved version of CSTR in the emulsion polymerization.

it consists of a tubular loop that connects the inlet and outlet of the recycle pump. these reactors have high surface to volume ratio as compared to the conventional CSTR and that allows very high conversion and shorter residence time. So, these reactor provide a greater flexibility and many minimum losses during the polymerization. Therefore, it offers a good economy related to the other available configurations.

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here you see that this is the continuous loop reactor, you see different loops are there and this styrene and initiator, they are pumped with this recirculation pump and you may have the other zone for the styrene and this one is butadiene. So, you may have the styrene butadiene rubber at this outlet.

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Emulsion polymerization reactors

- However, these reactors are also bound with some limitations:
 - The core-shell particles cannot be produced inside these reactors
 - It is difficult to adjust the PSD and to obtain smaller particles, large amount of surfactant is required.
 - The feed should be mechanically stable to prevent shear induced coagulation

these reactors are bound with several limitations although they offer a very good flexibility, but they are having certain limitations. The core shell particles cannot be produced inside these reactors. And it is difficult to adjust the particle size distribution and obtain the smaller particle large amount of surfactant is usually required. And the separation of those surfactants sometimes very tedious job from the developed or produced a polymer mass.

And second aspect is that the feed should be mechanically stable to prevent the shear induced coagulation. And sometimes, it is quite difficult to maintain this stability issue in the reaction aspect. Sometimes, another disadvantage is that the intermittent nucleation may occur that may promote the further or the parallel polymerization. This process is new and very limited information is available regarding these reactors. And sometimes the more technical information is available to various research labs.

So, therefore, if the application is restricted to the manufacturing of homo-polymer and copolymer of vinyl acetate. Let us have a look about the performance of emulsion polymerization reaction reactors. We can conclude that only CSTR and loop reactors have found attention for commercial applications. These reactors are designed to provide the efficient mixing. The modelling, this includes the macroscopic balance by considering the reactor as a whole.

So, these are the inlet and outlet streams, they are included in the balances for a generalisation of the model. For batch reactor, both the terms that is inlet and outlet extreme this can be neglected. For semi-batch reactor, outlet flow term can be neglected for designing aspects. Let us have a look about the performance related equations.

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Performance of Emulsion polymerization reactors

Mass balance

- The mass balance for any species is

$$\frac{dN_i}{dt} = F_{i \text{ in}} - F_{i \text{ out}} + (R_i)V$$
- Where, N_i is the amount of compound "i" in the reactor (mol);
 $F_{i \text{ in}}$ the inlet molar flow rate of component i (mol s^{-1});
 $F_{i \text{ out}}$ the outlet molar flow rate of i (mol s^{-1});

So, we can write the mass balance equation.

Mass balance

The mass balance for any species is

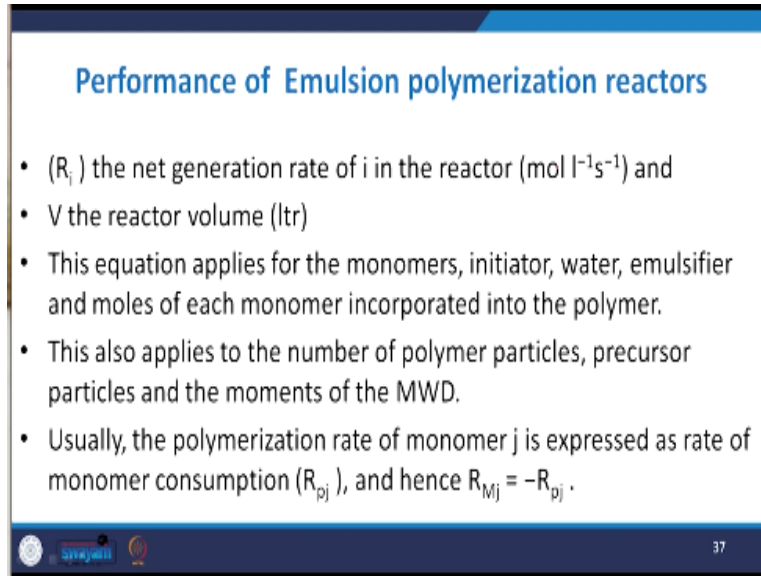
$$\frac{dN_i}{dt} = F_{i\text{ in}} - F_{i\text{ out}} + (R_i)V$$

Where, N_i is the amount of compound “i” in the reactor (mol);

$F_{i\text{ in}}$ the inlet molar flow rate of component i (mol s⁻¹);

$F_{i\text{ out}}$ the outlet molar flow rate of i (mol s⁻¹);

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Performance of Emulsion polymerization reactors

- (R_i) the net generation rate of i in the reactor (mol l⁻¹s⁻¹) and
- V the reactor volume (ltr)
- This equation applies for the monomers, initiator, water, emulsifier and moles of each monomer incorporated into the polymer.
- This also applies to the number of polymer particles, precursor particles and the moments of the MWD.
- Usually, the polymerization rate of monomer j is expressed as rate of monomer consumption (R_{pj}), and hence $R_{Mj} = -R_{pj}$.

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Performance of Emulsion polymerization reactors

- For the continuous operation $F_{i\ out}$ can be calculated as follows:

$$F_{i\ out} = \frac{Q_{out} N_i}{V}$$

Here, the volume of the reactor, V , is constant in a continuous operation

Q_{out} is the volumetric outlet flowrate

- In emulsion polymerization, the density of the reaction medium does not change significantly.
- Therefore, the volumetric inlet and outlet flow rates can be considered to be the same

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In emulsion polymerization, the density of the reaction medium does not change significantly.

Therefore, the volumetric inlet and outlet flow rates can be considered to be the same.

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Performance of Emulsion polymerization reactors

Heat balance

- The energy balance is

$$\sum_i N_i w_i c_{pi} \frac{dT}{dt} = \sum_j R_{pj} (-\Delta H_r)_j V - \sum_i F_{iin} c_{piin} w_i (T - T_e) + Q_{removal} + Q_{loss} + Q_{stirring}$$

c_{pi} and c_{piin} ($\text{kJ kg}^{-1} \text{K}^{-1}$) are the heat capacity of compound i in the reactor and at the entry conditions, respectively,

R_{pj} is the polymerization rate of monomer j ($\text{mol l}^{-1} \text{s}^{-1}$)

Heat balance

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R_{pj} is the polymerization rate of monomer j ($\text{mol l}^{-1} \text{s}^{-1}$),

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Performance of Emulsion polymerization reactors

$(-\Delta H_r)_j$ is the polymerization heat of monomer j at the reactor conditions (kJ mol^{-1}),
 T is the reactor temperature (K),
 T_e the temperature of the feeds (K),
 Q_{loss} are the heat losses to the surroundings (e.g., through the reactor lid, kJ s^{-1}),
 Q_{stirring} the heat produced by the agitator (kJ s^{-1})

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Q_{stirring} the heat produced by the agitator (kJ s^{-1})

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Performance of Emulsion polymerization reactors

Q_{removal} the heat removed through the heat removal devices (cooling jacket, cooling baffles, external heat exchanger and reflux condenser, kJ s^{-1})

- For heat removal through the cooling jacket, Q_{removal} is given by:
$$Q_{\text{removal}} = -UA_w \Delta T_{ml}$$

U is the overall heat transfer coefficient ($\text{kJm}^{-2} \text{s}^{-1} \text{K}^{-1}$)

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Q_{removal} the heat removed through the heat removal devices (cooling jacket, cooling baffles, external heat exchanger and reflux condenser, kJ s^{-1})

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Performance of Emulsion polymerization reactors

A_w the total heat transfer area (m^2) and
 ΔT_{ml} the logarithmic mean temperature difference between the cooling fluid and the reaction medium given by:

$$\Delta T_{ml} = (T - T_{jin}) - \frac{T - T_{jout}}{\ln((T - T_{jin})/(T - T_{jout}))}$$

T_{jin} and T_{jout} are the inlet and outlet temperatures of the cooling fluid in the jacket (K)

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A_w is the total heat transfer area (m^2) and

ΔT_{ml} is the logarithmic mean temperature difference between the cooling fluid and the reaction medium given by:

$$\Delta T_{ml} = (T - T_{jin}) - \frac{T - T_{jout}}{\ln((T - T_{jin})/(T - T_{jout}))}$$

T_{jin} and T_{jout} are the inlet and outlet temperatures of the cooling fluid in the jacket (K)

- So, we know that the several resistances play a vital role in defining the heat transfer coefficients such as the resistance due to the diffusion mass transfer chemical reaction. So, for this case, the internal resistance usually controls the heat rate of heat transfer.

The internal heat transfer coefficient is a function of:

- impeller type and dimension
- impeller speed
- reactor diameter
- physical properties of the reaction mixture

So, it can be calculated by means of empirical correlation based on the dimensionless groups.

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Performance of Emulsion polymerization reactors

- A practical method to determine the heat transfer through any cooling device is to measure the flow rate, and the inlet and outlet temperatures of the cooling fluid:

$$Q_{removal} = \dot{m}_w c_{pw} (T_{jin} - T_{jout})$$

- where \dot{m}_w is the mass flow rate (kg s^{-1}).
- These equations allows the estimation of the polymerization rate from temperature measurements.



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These equations allows the estimation of the polymerization rate from temperature measurements.

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Performance of Emulsion polymerization reactors

- Reaction calorimetry is the tool to determine the internal heat transfer.
- It is a influential non-invasive on-line monitoring technique.
- It measures the amount of energy released or absorbed by a chemical reaction.
- During on line monitoring, heat flux sensors are installed on the wall of the reactor vessel, that directly measures the heat across the reactor.



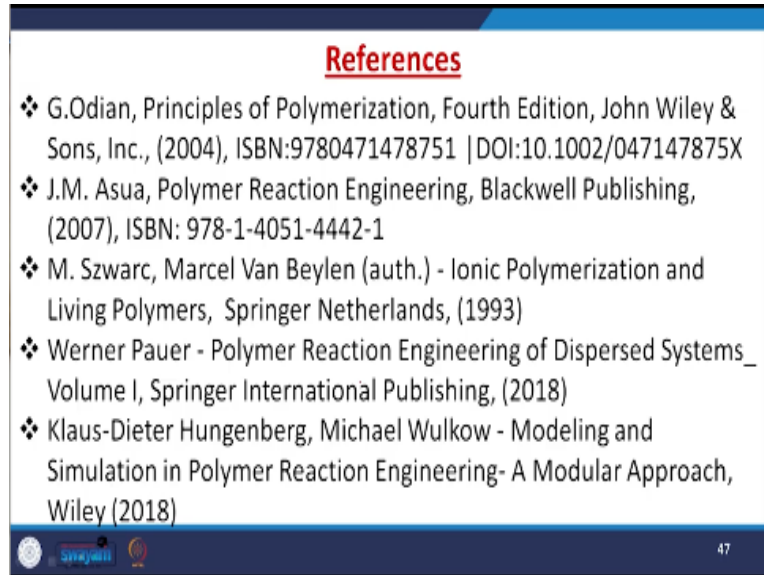
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reaction calorimetry is the tool to determine the internal heat transfer. It is an influential non-invasive online monitoring techniques. And measures the amount of energy released or absorbed by a chemical reaction. So, during online monitoring, heat flux sensors are installed on the wall of the reactor vessel that directly measures the heat across the reactor. So, in this

particular lecture, we discussed about the remaining aspect of emulsion polymerization reactor specially attributed to the morphology.

Then we discussed about the performance of emulsion polymerization reactors with the due emphasis to all kinds of applicable reactor as on date. We discussed the merits and demerits of those reactors and where we can apply all those reactors.

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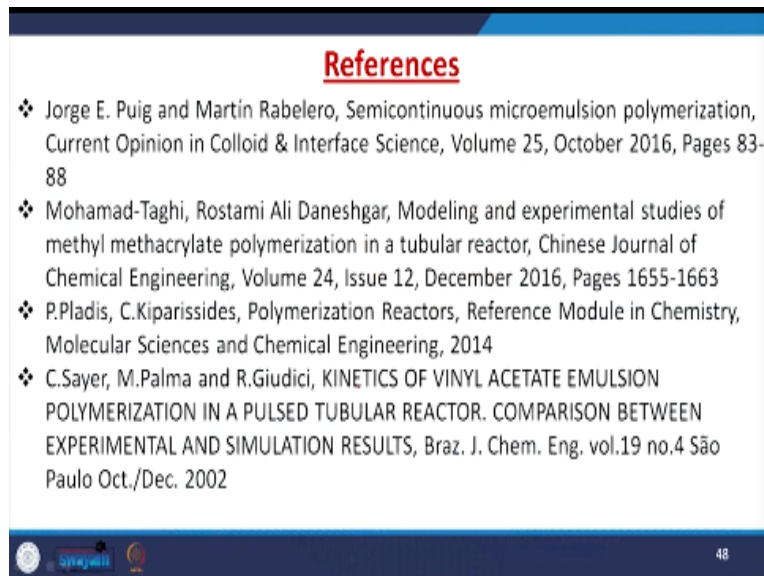


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If you wish to have further studies related to these reactors, we have listed several references for your convenience. You can have a look of all those references for further studies. Thank you very much.