

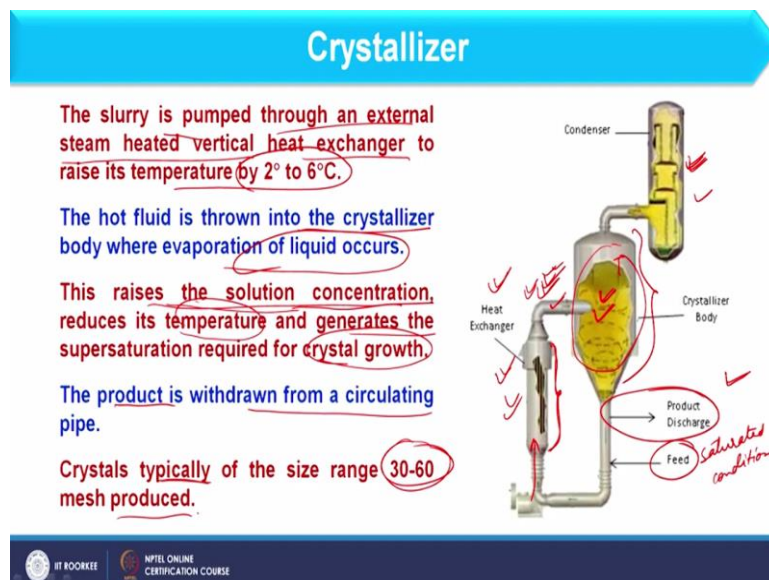
**Process Equipment Design**  
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**Lecture –40**  
**Design of Crystallizer-2**

Hello everyone. This is the 40th lecture of the course Process Equipment Design and I welcome you all in this lecture and here we are going to discuss design of crystallizer. So, this topic we have started from the last lecture where we have introduced the crystallization it means we have defined the crystallization, we have seen different form of the crystal, we have seen some basic mechanism of that, that how the crystallizer works that how the crystallization occurs and what are the solubility curve.

And what is solid liquid phase diagram. So, all that point we have discussed in the last lecture and here in this lecture we will first focus on that how crystallizer works and then we will focus on some points under design consideration and finally we will see the steps for designing of crystallizer.

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So, let us start with the working of crystallizer. So, as far as crystallizer is concerned it looks like this. So here we have some basic unit which are involved in crystallizer such as heat exchanger and here we have the condenser to condense the vapour and so it generates vacuum in the system. So, as far as working of crystallizer is concerned whatever solution is there that solution we consider as a feed and this is basically saturated condition.

So, feed is usually available at saturation condition. So, when the feed enters it is first entering through this pump to this heat exchanger and in this particular heat exchanger temperature of this feed is increased. And that temperature is usually not much it is usually 2 to 6 degree increment. So, when you see this the slurry is pumped through an external steam heated vertical heat exchanger which is nothing, but this to raise its temperature by 2 degree Celsius to 6 degree Celsius.

So, when we consider this solution which is heated up it is basically at saturation condition over here. And after this it enters into this vessel and when it is entering into this vessel it is basically entering tangentially not perpendicularly and whatever solution is there it revolves into this and when we consider the temperature over here. Here temperature is less than whatever temperature available over here and here I am having the super saturation condition.

You see here when the temperature the hot fluid which is available after the heat exchanger it is thrown into the crystallizer body where evaporation of liquid occurs because what I have told you here temperature is lesser than whatever temperature is available over here. So, this feed basically come across at temperature lesser than the feed temperature in this vessel and because of this reduction in temperature the evaporation takes place.

So, the solution which is available in this vessel reaches to super saturation condition. So, when it is at super saturation condition the solution concentration increases and as temperature is reduced and therefore generation of crystal occurs here. So, when I am having the heat exchanger the temperature of the feed is increased and so it acquires a saturated condition and then it enters into the vessels where temperature is less.

So, some evaporation takes place from the solution and so the solvent is removed and so the solute available in the slurry increases and that condition we consider as super saturated condition. And when this condition occurs the crystal formation starts and once I am having the crystal the product can be withdrawn from the circulating pipe. So, you can see from here we can get the product out.

So, this complete assembly is basically crystallizer body, here I am having the heat exchanger and here we have the condenser. So, the purpose of condenser is whenever evaporation takes

place in the crystallizer body vapour is generated and that vapour is condensed into the condenser. And when the vapour is condensed at the same time vacuum is also generated and so the temperature is reduced.

So, when I consider the crystallizer the crystals typically of size 30 to 60 mesh is produced. So, I hope you are understanding the meshing that is important section for screen operation. So, as the mesh number increases the size of the particles decreases the opening in the screen reduces. So, in that case we consider that 30 to 60 mesh so it has a particular opening. So, that is available in standards of the screen.

So, in that way you can find the total range of the crystals which are formed in crystallizer. So, in this way we consider the working of a crystallizer and now we will focus on design considerations. So, as far as design considerations are concerned we have some points let us discuss these functions.

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**Design Considerations**

- ✓ The function of a crystallizer is to produce crystals of a given size specification from a feed at a specified rate.
- ✓ A suitable and adequate supersaturation is created by cooling the feed or by partial evaporation of the solvent.
- ✓ Second method is more common in industrial practice.
- ✓ Narrow particle size distribution of the product is desired to maintain a good product quality.
- ✓ Besides the correct supersaturation and environment (i.e. agitation, pumping rate, etc.), techniques like fine redissolution or classified product removal are helpful to achieve a better product quality.
- ✓ Batch crystallizers required seeding (i.e. addition of fine crystals that act as nuclei).
- ✓ Secondary nucleation occurs continuously in a continuous crystallize and seeding is not generally necessary.

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So, the function of crystallizer is to produce crystals of given size specification from a feed at a specified rate. So, usually we consider that feed enters into the crystallizer at a particular rate and production of crystals are also with a rate. So, that all we have to specified when we are going to start design of crystallizer. Further, a soluble and adequate super saturation is created by cooling the feed or by partial evaporation of the coolant.

So, that also we have to ensure and further second method is more common in industrial practice. Second method means the partial evaporation as also we have discussed in the last

slide. So, when we consider narrow particle size distribution of the product it is desired to maintain good product quality. So, how we check the quality of the product that particle size whatever available for the crystal should not be very wide in range.

So, almost same size of crystal should be form then we consider that as a good product. Besides the correct super saturation and environment that is basically agitation pumping rate etcetera, techniques like fine re-dissolution or classified product removal are helpful to achieve better quality product. So, all these points we should consider in design of crystallizer and further batch crystallizer when I am considering these requires seeding/

It means addition of fine crystal that act as a nuclei. So, that is the consideration when I am having batch crystallizer it means some foreign particles you have to insert which works as a nuclei and further crystal growth can occur over there. Secondary nucleation occurs continuously in a continuous crystallizer and seeding is not generally required. So, what is the point that when I am having the continuous crystallizer because feed is usually coming into this continuously and crystal is removed continuously.

So, some nuclei will remain there so we should not suppose to add nuclei from outside as we usually do in batch crystallization. So, in industry when I am dealing with continuous crystallization secondary nucleation starts or secondary nucleation takes place.

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**Design Considerations**

**Important parameters and quantities involved in the design**

The feed rate and state (concentration, temperature, pressure, etc.).  
The desired crystal size distribution (CSD) and yield. The percentage theoretical yield is defined as,

$$\text{Yield (\%)} = \frac{100(Q_i C_{in} - Q_o C_s)}{Q_i C_{in}}$$

$Q_i$  = feed rate,  $Q_o$  = rate of outflow of mother liquor,  $C_{in}$  = feed concentration, and  $C_s$  = solubility of the solid at the exit temperature.  
If the temperature in the crystallizer is fixed,  $C_s$  can be obtained from the solubility data. From the specified yield (%),  $Q_o$  is calculated using above equation. The required rate of evaporation is determined by a solvent balance.

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So, these are some important factors about design consideration now we will consider a few important parameters and quantity which should be involved in design of crystallizer. So,

first is the feed rate and state means concentration temperature and pressure. So, that is very important parameter and secondly desire crystal size distribution and yield what should be the crystal size because we do not have a particular size of the crystal we usually have the range.

So, we consider that as crystal size distribution and also we should focus on the yield. So, how we consider that yield it can be defined as yield percent that is basically  $Q_i C_{in} - Q_o C_s$  divided by  $Q_i C_{in}$  into 100. So,  $Q_i$  is basically the feed rate  $Q_o$  is the rate of outflow of the mother liquor it means what is the flow rate of mother liquor when crystals are removed from it.

And then  $C_{in}$  is the feed concentration and  $C_s$  is the solubility of solid at exit temperature. So, based on these parameters we can find out yield in crystallizer and further we should consider that if temperature in a crystallizer is fixed.  $C_s$  we can obtain from the solubility data because solubility curve basically draws concentration with respect to temperature. So, when we fix the temperature  $C_s$  value can be obtain from that graph.

And further from a specified yield percent  $Q_o$  is calculated using above equation. So, what should be the flow rate of mother liquor if we know the yield we can find that through this equation. So, the required rate of evaporation is determined by the solvent balance. So, in this way we consider the feed rate, desire crystal size distribution yield and other parameters.

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**Design Considerations**

**Important parameters and quantities involved in the design**

**Solvent evaporation rate and the heat transfer area required**

These are to be calculated. The evaporation rate is calculated from material balance. A heat balance over the crystallizer gives the required rate of heat input. Heat of crystallization should be included in the heat balance. The steam or the heating fluid rate and the heat transfer area are then calculated.

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So, another parameter for design of crystallizer is the solvent evaporation rate and heat transfer area. So, solvent evaporation rate means it is occurring in the crystallizer and heat

transfer area which is the area of heat exchanger. So, these are to be calculated the evaporation rate is calculated from the material balance because how much feed is entering, how much crystal is formed and how much vapour is generated all that we can calculate by material and energy balance.

And this point we will also consider in detail when we discuss the design of crystallizer. So, a heat balance over the crystallizer gives the required rate of heat input. So, evaporation rate as well as heat requirement can be calculated by material energy balance and further we have the heat of crystallization and this should be included in the heat balance because when crystal is formed it takes some heat from the solution.

So that is basically the heat of crystallization. The steam or the heating fluid which is used in a heat exchanger and accordingly the heat transfer area can be calculated from energy balance. So, in this way solvent evaporation rate as well as heat transfer area can be calculated.

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**Design Considerations**

**Important parameters and quantities involved in the design**

**Crystallizer volume** ✓

This is to be calculated. For this purpose, experimental data on nucleation ( $B^0$ ) and the growth rate ( $G$ ) are required. These data can be obtained from a laboratory crystallizer. Data collected from a pilot plant crystallizer of volume around 50 litres or more can be more reliably used.

From the known values of  $G$  and  $L$  (characteristic size), the holdup time and volume can be calculated. The diameter of a crystallizer is frequently determined on the basis of possible entrainment of liquid with the vapor generated.

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And further we have very important parameter that how to decide the crystallizer volume. So, as far as calculation of crystallizer volume is concerned we need the experimental data. Experimental data for what? Experimental data for nucleation and the crystal growth rate. So, nucleation that is  $B^0$  and crystal growth rate that is basically  $G$ . So, in this way depending upon the solution we can find these data experimentally.

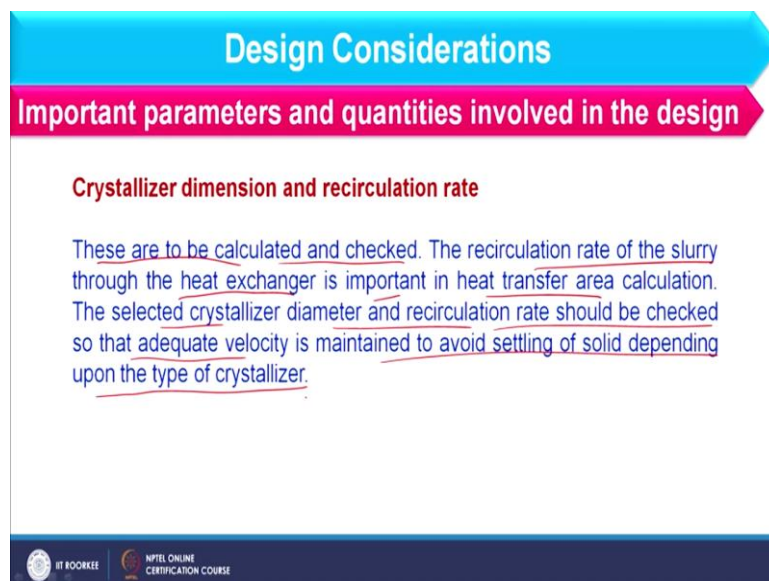


And these data can be obtained from laboratory crystallizer. So, usually we obtain these data from crystallizer which is available in labs. However, when we consider large size crystallizer it means which is available at least at pilot level and as far as volume is concerned that should be at least 40 liters or so. So, the data obtained from that, that is data of  $B_0$  as well as  $G$  are more reliable.

So, if that is available we should use that data also. So, from the known values of  $G$  and  $L$  and  $L$  is basically the characteristic size or we can consider the size of the crystal the holdup time and volume can be calculated and further once I am having the volume diameter of a crystallizer is determined on the basis of possible entrainment of liquid with vapour generated.

So, when we decide the crystallizer volume it is not the volume of the solution only, it is the volume of the space where vapour will be generated through evaporation. So, considering all these parameters we will decide the volume of the crystallizer and then we will decide the diameter of the crystallizer. How we will consider that? Step by step that will be discussed in design of crystallizer. And further we have important parameters like crystallizer dimension and recirculation rate.

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**Design Considerations**

**Important parameters and quantities involved in the design**

**Crystallizer dimension and recirculation rate**

These are to be calculated and checked. The recirculation rate of the slurry through the heat exchanger is important in heat transfer area calculation. The selected crystallizer diameter and recirculation rate should be checked so that adequate velocity is maintained to avoid settling of solid depending upon the type of crystallizer.

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So, as far as crystallizer dimension is concerned it is basically the shell diameter and the height of the crystallizer. Height of the crystallizer means height of the shell of the crystallizer along with the tapered section because bottom section of crystallizer is usually tapered that also you have observed when we were discussing the working of crystallizer. So,

along with the shell the tapered section is also important and dimension of that should also be considered.

So, these parameters are to be calculated and checked the recirculation rate of the slurry through the heat exchanger is important in heat transfer area calculation because when we consider the heat transfer area we should also consider that how much feed is going to be heated up in heat exchanger and depending upon that we consider the steam flow rate. So, it will depend on the feed as well as recirculation ratio because both feed as well as the fluid which is coming from recirculation should be considered in heat exchanger for further heating.

Further, the selected crystallizer diameter and recirculation rate should be checked so that adequate velocity is maintained to avoid settling of solid depending upon the type of crystallizer. So, all these points we should consider in design of crystallizer.

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**Design of Crystallizer**

**1. Material balance to know total crystal formed, water evaporated and volume of magma leaving the crystallizer.**

- Solute in to crystallizer ✓
- Solvent (water) in to crystallizer ✓
- Crystal produced in crystallizer ✓
- Solute leaving the crystallizer ✓
- Water leaving the crystallizer ✓
- Volume of mother liquor leaving per hour ✓
- Volume of the slurry leaving per hour ✓

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So, let us focus on design of crystallizer. So, as far as design of crystallizer are concerned we first consider on material balance and why material balance is required to know how much crystals are formed, how much water is evaporated and how much volume of magma as well as mother liquor which are leaving the crystallizer. So, let us see the steps involved in this. First of all, we should include the solute in the crystallizer that how much solute is entering into the crystallizer.



How we will obtain all these steps that we will consider with the help of example, but right now let us focus on the steps only. So, initially you have to consider that how much solute you are going to handle in crystallizer so that will be solute in. Solvent that how much solvent is included that is basically how much water is included because usually solvent in crystallizer is water.

Other solvents may be there, but usually we consider water as solvent and after that we should calculate that how much crystals are produced in crystallizer and how much solute is leaving the crystallizer. So, from the total solute how much crystals are formed and what is the concentration of solute remaining in the solution in the crystallizer which will leave the crystallizer because further crystallization will not occur when the solution is not at super saturation condition.

And after that we should also focus on that how much water is leaving, so how this water will leave. When feed enters into the crystallizer body you understand that first of all evaporation takes place and then the solution will enter into the super saturation region. So, once evaporation will take place then the solvent which is available in the solution it is converted into the vapour form and that vapour form basically leaves the crystallizer body from the top which is further entering into the condenser.

So, water leaving means in the form of vapour. So, how much evaporation takes place that also we should consider to find out that how much water should be removed from the crystallizer. Once we have that, we can calculate total volume of the mother liquor which is leaving per hour. So, in this way you should consider that how much crystals are formed, how much vapour is produced, how much mother liquor is available.

And how much solute which is remaining in the mother liquor so all that we should consider in designing. And then we should also focus on volume of the slurry leaving per hour. So, what is the meaning of this volume of the slurry it means volume of the mother liquor and volume of the crystal.

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## Design of Crystallizer

### 2. Computation of crystallizer volume

- Residence time,  $L_c/3G$
- Volume of the slurry in the crystallizer
- Total volume of crystallizer considering 60% more volume to account for vapor bubble and froth
- Assume diameter of vessel
- Volume of the conical section considering cone angle as  $45^\circ$
- Volume of the cylindrical section
- Height of cylinder

So, in this way we should carry out material balance to know different parameters as we have just discussed and then we should find out the crystallizer volume. So, as you know that crystallizer volume does not depend only on the solution it depends on the space which is required for vapour generation. So, to calculate the crystallizer volume first of all we should focus on the residence time which is available in crystallizer because that residence time will depend on that how much crystals are formed.

So, if you see how we can calculate that residence time using this expression. So, here we have  $L_c$  divided by  $3G$ . So,  $L_c$  is basically the size of the crystal and this  $G$  is basically the growth rate. So, once I am having the residence time I know that how much volume of slurry is available in the crystallizer and based on that we can find the total volume of the solution depending upon this residence time.

And after that we should consider total volume of crystallizer considering 60% more volume to account for vapour bubble and froth. So, in this way you should consider volume of the slurry in a given residence time along with the space which we are providing for vapour formation and the froth formation. And after that when we decide the total volume we will assume one diameter value so that maybe a random value which we will check further.

So, next we have to assume the diameter of the vessel and then volume of the conical section considering cone angle as  $45^\circ$ . So, here you should consider that crystallizers are basically cylindrical in shape, but tapered section is provided at the bottom. And that tapered section ensures the smooth removal of the crystals which are formed. So, depending upon the

shape we should consider diameter of the vessel along with the volume which is available in conical section.

So, whatever volume we have discussed previously like volume of the slurry in crystallizer plus 60% extra that is basically the total volume of the crystallizer. And from that we will remove the volume of the conical section to find out the volume of the shell only. So, accordingly I can find the cylindrical section volume and once I fix the diameter I can simply calculate the height of the cylinder.

So, in this way dimension of the crystallizers are decided because we have assumed the diameter value, we will now verify that whether the assumed value are correct or not.

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**Design of Crystallizer**

**3. Checking for assumed diameter**

- Absolute pressure ✓
- Volumetric flow rate of vapor generation ✓
- Allowable velocity ✓

$$u_v = 0.04 \left( \frac{\rho_l - \rho_v}{\rho_v} \right)^{1/2} \quad \checkmark$$

- Area required for evaporation ✓
- Diameter of evaporation section ✓

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And to check that we will first consider the absolute pressure. What is this pressure? This pressure is corresponding to the pressure inside the crystallizer because usually the crystallizer is operated at vacuum pressure. Absolute pressure means atmospheric pressure minus the gauge pressure at which crystallizer is being operated. So, depending upon that we consider the absolute pressure.

And once I am having the absolute pressure we can find out the volumetric flow of vapour generation, how much vapour is generated its volumetric flow can be obtained. How we can obtain that volumetric flow all that we will discuss with an example. Once we decide the volumetric rate we can find out the allowable velocity through this expression and volumetric flow is given.

Velocity is given we can simply find out cross sectional area of the evaporation section and that is basically the area of crystallization section also. And so we can find out the diameter of evaporation section along with crystallization body because crystallization as well as evaporations both are taking place in a same unit. So, once we know the cross sectional area of evaporation and diameter these are basically for crystallizer body also.

So, whatever diameter we can obtain that we can compare with the assumed value. If it is close to that we can consider that diameter otherwise we can consider this diameter and carry out the whole calculation as per the previous slide. So, in that way we design the crystallizer and checked the assumed value.

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1	Dutta, B.K., "Principles Of Mass Transfer And Separation Processes", 2009, PHI Learning Private Limited, New Delhi.
2	Sinnott, R.K., "Coulson and Richardson's Chemical Engineering Series: Chemical Engineering Design", Vol. VI, 4 <sup>th</sup> Ed., 2005, Elsevier Butterworth-Heinemann.
3	Serth, R.W., "Process Heat Transfer: Principles and Applications" 2007, Elsevier Ltd.
4	McCabe, W.L., Smith, J.C. and Harriott, P., "Unit Operations Of Chemical Engineering" 5 <sup>th</sup> Ed., 1993, McGraw-Hill Inc., New York.


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And here I am having some references through which you can study about the topic crystallization.

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### Summary of the video

- ✓ Crystallization process is defined. ✓
- ✓ Process of production of crystals is discussed. ✓
- ✓ Solid-liquid phase equilibrium, supersaturation and solubility curve are described. ✓
- ✓ Nucleation and Crystal Growth are discussed.
- ✓ Important parameters and quantities involved in the design of crystallizer are discussed.
- ✓ Steps to design a crystallizer are detailed. ✓

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And now we are having the summary of the video and this summary is of the 4th and 5th lecture of this week that is 39th lecture and 40th lecture. So, summary of these videos are here crystallization process is defined, process of production of crystals is discussed and solid liquid phase equilibrium, super saturation and solubility curve are described, nucleation and crystal growth are discussed.

Then further we have discussed important parameters and quantities involved in design of crystallization and further we have discussed important parameters and quantities involved in design of crystallizer along with the working of crystallizer. And finally we have focused on steps to design a crystallizer and here I am stopping this lecture. In the next lecture, we will discuss the design of crystallizer with the help of example. So that is all for now. Thank you.