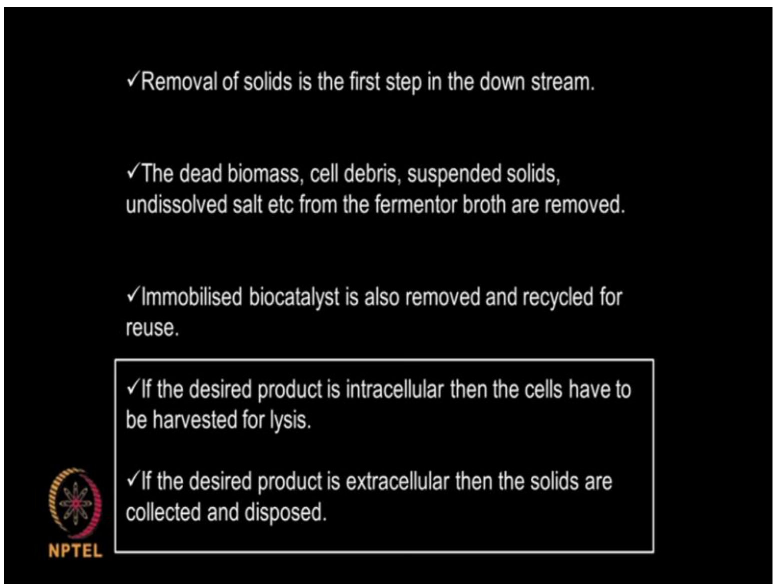
Principles of downstream techniques in Bioprocess – a short course
Prof. Mukesh Doble
Department of Biotechnology
Indian Institute of Technology, Madras
Lecture – 5
Solid Liquid Separation

In this class we are going to look at solid liquid separation. As I had mentioned before, immediately after a fermenter you have a solid liquid separator. There are different types of solid liquid separator and solid liquid separation is performed in order to remove the cell debris, the dead biomass, and precipitated media components and so on.

(Refer Slide Time : 0:41)

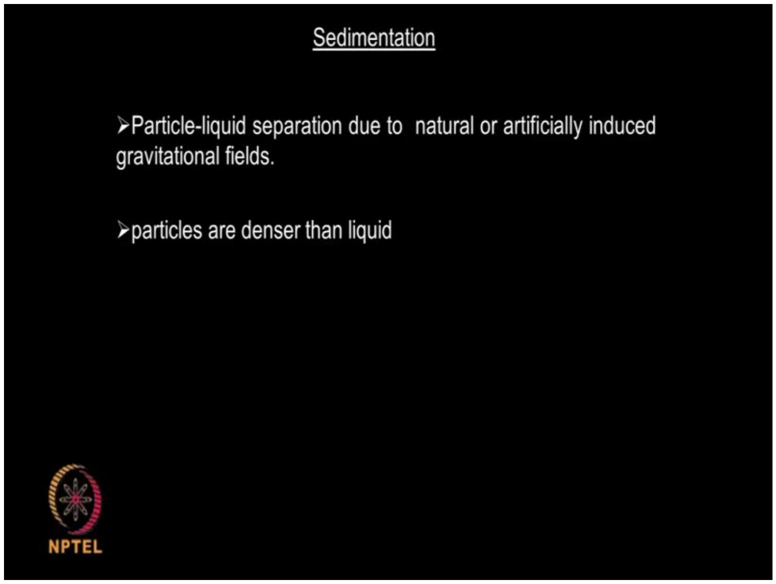


So we are going to look at these various types of solid-liquid separating processes actually. So if you look at removal of solids, as I said this is the first step in the downstream. It is going to involve dead biomass, cell debris, suspended solids, undissolved salt. All these have to be removed first before you actually recover your desired product and purify your desired product. So if it is a bio catalyst like we are looking at enzyme catalyst, biotransformation, you like to recover the bio catalyst, recycle it back into the bioreactor.

So again you need some sort of a filter or a membrane and so on actually and there are two different situations when you are looking at the solid liquid separation. So if the desired product is intracellular, then we have to harvest the cells, that means your product is inside the cell, So you have to harvest the cells, lyse the cells to get your product out. If the desired is extracellular, you are not bothered about the solids. So what do you do?

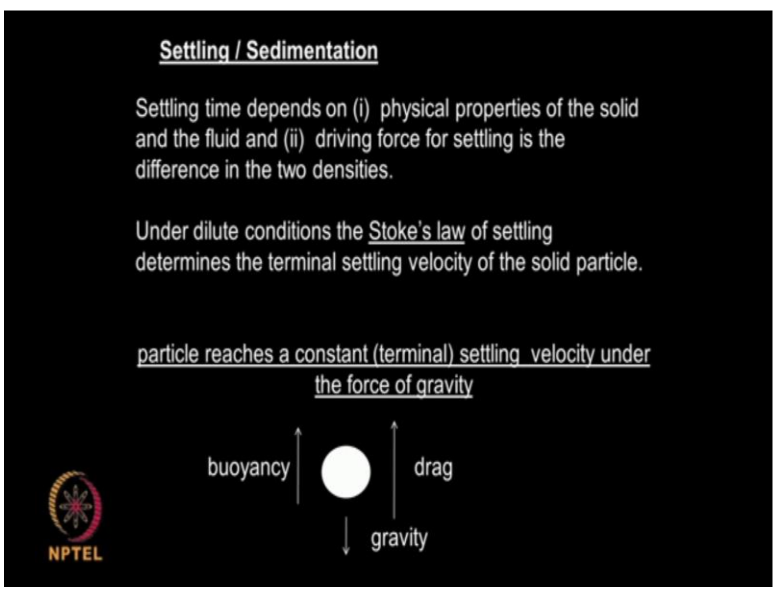
You actually filter the solid and then dispose the solid because the liquid is what your desired product is. So you collect the liquid, then you start recovering your product which is inside the liquid whereas in the other case if it is intracellular, there desired product is inside the cells.

(Refer Slide Time : 1:59)



Okay so one of the easiest solid liquid separation is called sedimentation. Okay. So why does it happen? The solids settle down due to natural or artificially induced gravitational fields. It could be a natural gravitational field or it could be because of a centrifugal force, the solids could be separated actually.

(Refer Slide Time : 2:17)

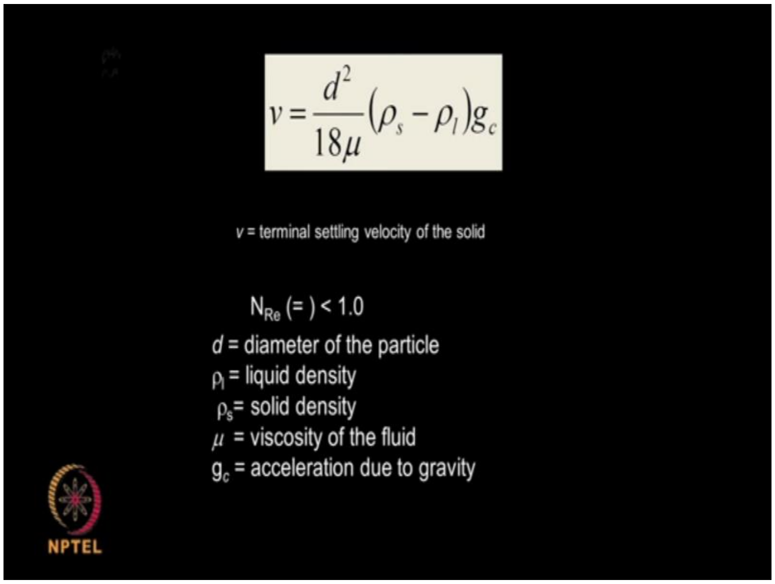


So particles have to be denser than the liquid. That is very very important and because of these density difference that gives you a driving force, so that settling time of the solid in this liquid, depends on the physical properties of the solid and the fluid, like density, viscosity of the liquid and so on and the driving force is the difference between the two densities. So what is the law which governs the solid-liquid separation in sedimentation or it is also called settling.

That is called the Stokes' law. It is very very simple but it is quite powerful in doing certain calculations. We look at some problems as we go along and so when a solid, freely suspended solid is settling, so there are many things that are happening actually. You have the buoyancy, you have the drag force and then you have the gravitational force. All these three are taking place actually okay so the buoyancy, the gravity and the drag force.

So if you try to balance all these together you end up with an equation, which determines the velocity at which the solid will move down. This is called the terminal settling velocity. Okay. That is called the terminal settling velocity which is just because of the gravitational force.

(Refer Slide Time : 3:43)



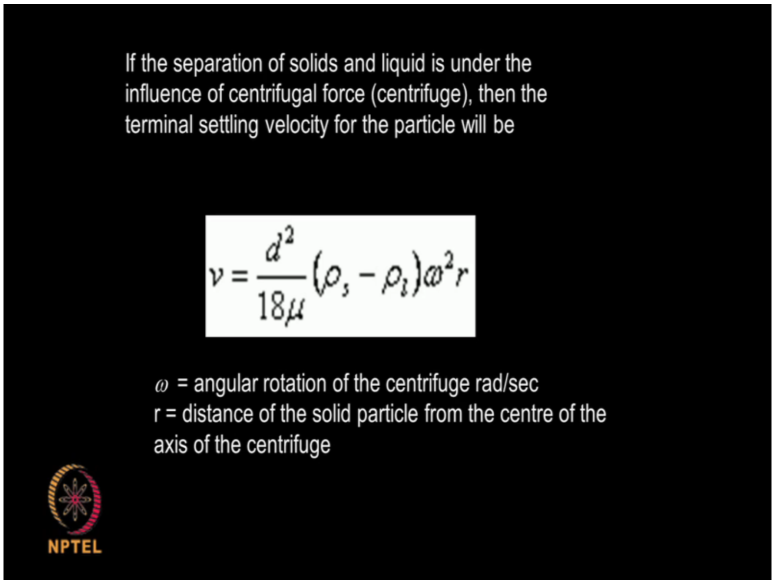
So you have to balance all these forces and you end up with the new equation like this. The terminal settling velocity = d square. That is the diameter... d is the diameter of the solid, Mu is the viscosity of the solid, row s is the density of the liquid, solid sorry density of the solid and row l is the density of the liquid and you have the g the gravitational force. Okay so the velocity, that is the terminal settling velocity depends upon the solid.

So larger the diameter, faster it will come down, inversely proportional to viscosity, if the viscosity of the medium is high, the settling velocity becomes low and the driving force here is row s - row l. So if I have a large difference, then I will have a large settling velocity. If a small difference I will have a small settling velocity. So the most important point here to consider is the solids should be moving at a very low velocity that means at a very low Reynolds number. That is very very important. Generally Reynolds number should be in the order of less than 1.

Okay how do you calculate Reynolds number? The equation for Reynolds number, you all may be knowing okay = d, that is the diameter of the particle okay, then u that is the velocity at which the particle is moving, row, row is the density okay that is the liquid density, Mu is the viscosity of the liquid okay.

So d u row by Mu is what is Reynolds number and that should be generally less than 1. So you need to see whether your Reynolds number is less than 1, otherwise this particular equation was not... is not reliable to use. Reynolds number has to be less than 1 okay that is very very important.

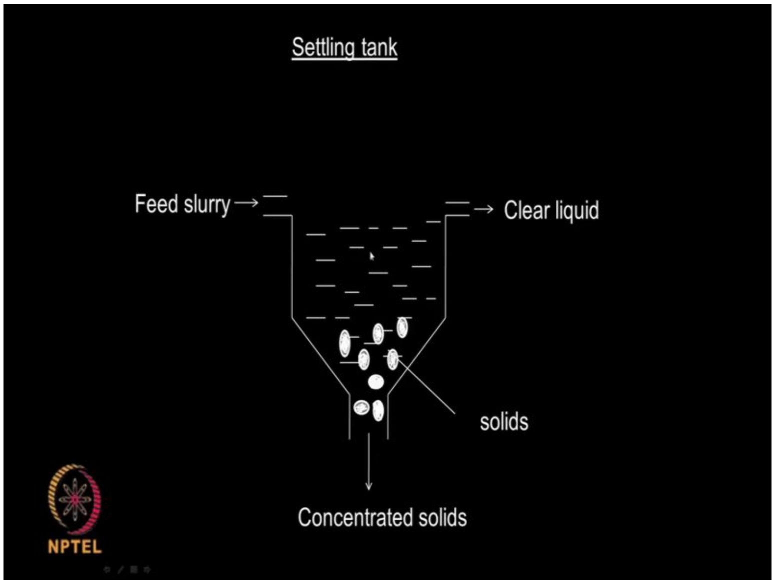
(Refer Slide Time : 05:29)



When you have settling taking place in a centrifuge, then instead of g which is the gravitational force, you will have omega square r. Okay what is the omega square r? Omega is your angular rotation of the centrifuge, that is radiance per second, R is the distance of the solid particle from the center of the axis okay. Further the particle away from the center going to have high driving force. That is why you have r here and larger is your rpm, larger will be your settling velocity.

So you use this particular equation when you are using a centrifuge for settling a solids and you use the previous equation if you are using a normal gravitational force for settling of solids. So instead of omega square r you are going to have g. Otherwise the equation remains the same so as you can see g is always fixed, whereas if I am doing a settling in a centrifuge, I can keep increasing my omega, that means I can keep increasing my rpm so that I achieve very high settling values. Okay whereas in the normal gravitational type of settling, I will not be able to achieve such very high velocities.

(Refer Slide Time : 06:45)

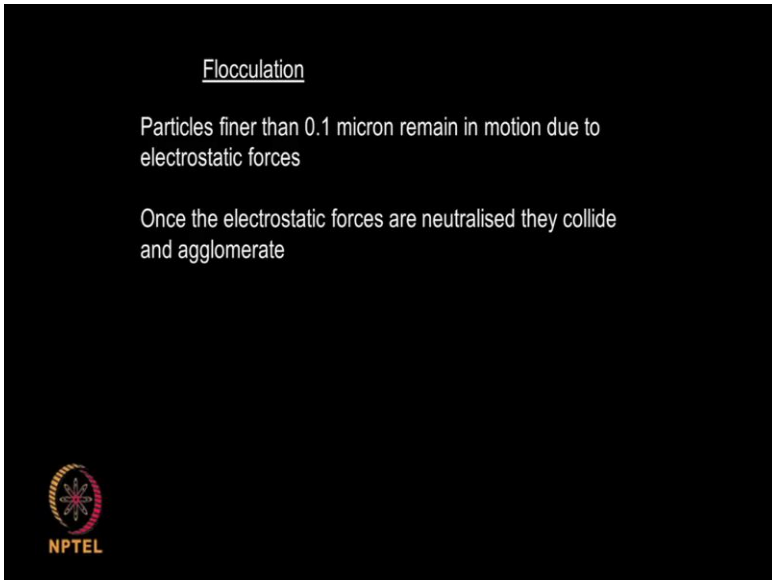


So this is how a settling tank looks like. The settling tank is the huge tank, there feed comes in.. the slurry, the solid, you give enough time inside so that the solids will settle down and then the bottoms will contain concentrated solids whereas the top will contain clear liquid. This is used very common in effluent treatment plant to remove the initially solids that are present in the effluent. This is a very simple setup. You do not need any energy for this, you do not require any agitation or aeration and so on

And it is very cheap only thing is, you need to give sufficient time inside your tank so that the solids get settled. So if you know the solid property like density and diameter if you know the density and viscosity of the liquid, we are using generally in effluent treatment, you will have water as the liquid. So you know the density and viscosity of the water, we can calculate the velocity and we can... once I know the height I will know the time for which need to allow the slurry to stay in the tank, so that the solids will settle down very effectively.

Okay so it is quite simple, it is a quite powerful equation although the equation is very simple, we can use it for calculating the time for which the slurry has to stay inside the settling tank okay.

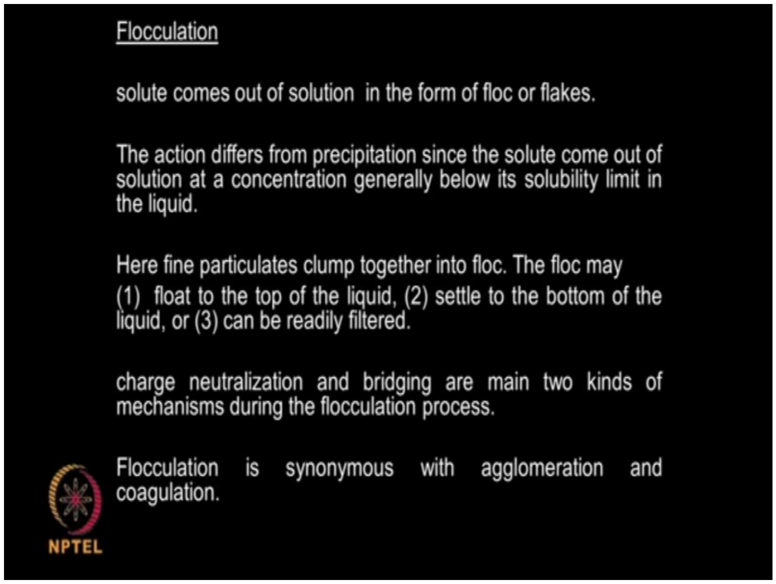
(Refer Slide Time : 08:14)



Flocculation, what is flocculation? Sometimes very fine particles, they do not settle down at all. That is because there are lot of electrostatic forces like particles with 0.1 micron, they always remain away from each other because of this electrostatic forces. So they do not agglomerate at all. So you want to separate them. Filtration may be a difficult option because the particle sizes are very small, so you resort to something called flocculation or agglomeration.

So basically here you have to make the electrostatic forces, neutralize the electrostatic forces so that the particles come together and form larger flocs, then it may be possible for it to settle down inside a settling tank. So basically what you do here is, you add something called flocculating agent so that it breaks electrostatic forces and allows the particles to come together and form the larger particles which settle down. That is what is done in flocculation actually okay.

(Refer Slide Time : 09:25)



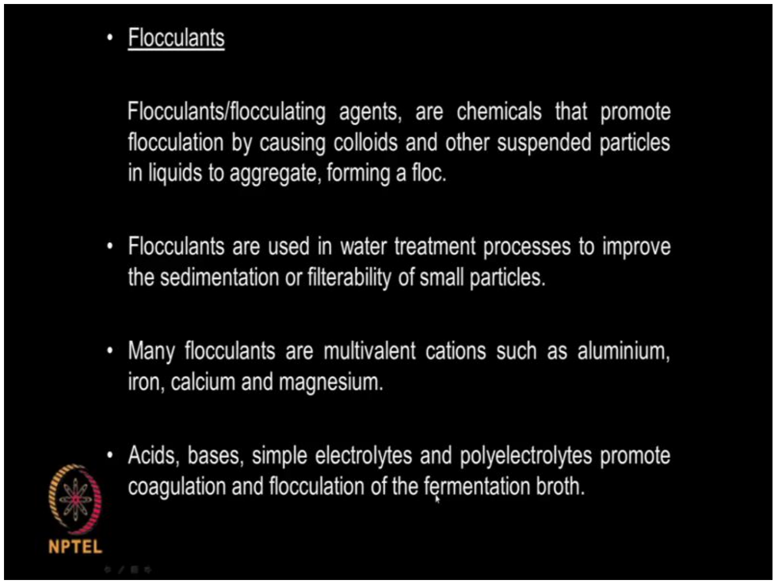
So you can remove it as a flake or it can be like a flock, like a precipitate basically but there is a difference between precipitation and flocculation. In precipitation, the concentration is larger than the solubility of the solid in the liquid. That is why it precipitates whereas in flocculation, we have finely divided solid present inside the liquid, they are not coming together because of the electrostatic operations.

So you had a flocculating agent allow them to come together, collide and then form larger particles which can settle down. that is what happens here in flocculation okay. So they will clump together or they will also floc together. So sometimes they can float at the top of the liquid in the density of the flocs or the bulk density of the flocs are less than the liquid density or they can settle to the bottom of the liquid.

If the density of the flocs are much larger than the density of the liquid or they can be easily filtered because when the particles are very fine, it may be very difficult to filter and through a filtration unit of course you can go into some nano filtration but that is very expensive. So you can make into larger flocs, then it becomes easy to use very normal filtration unit to filter it out, that is what is done in flocculation okay. Basically you are doing a charge neutralization and bridging.

These are the things that happen mechanism by which flocculation takes place. Basically the flocculant neutralizes the charge on the particle so hence there is no repletion between particle and particle or they form a bridge between two charged particles so the particle becomes larger or flocs okay. So instead of flocculation, we can also called agglomeration, we can also called coagulation, so you had a coagulating agent or you had a flocculating agent, you had a agglomerating agent, so they are all synonymous to each other actually okay.

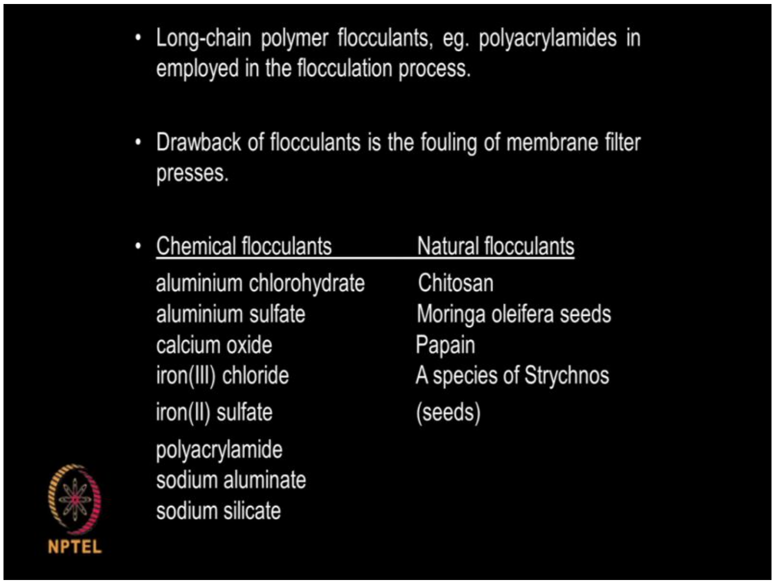
(Refer Slide Time : 11:26)



So there are flocculants that are used synthetic chemicals, natural chemicals and so on that are used to break this type of charges on that actually. They are generally used quite a lot in water treatment plant, they are used in environmental plant, when you are remediating the effluent that is getting discharged and so on actually. So what are these flocculants? They are generally multivalent cations like aluminum.

Aluminum has got aluminum chloride, iron, calcium, these are all multivalent mg2, fe3, ca2 are sort of salts okay. That sort of inorganic salts, that are used so they will generally neutralizes the charge that are present on the small sized particles okay or sometimes you can add acids, bases when simple electrolytes, poly electrolytes, polyacrylamide, all these are added and they are called flocculants okay.

(Refer Slide Time : 12:40)

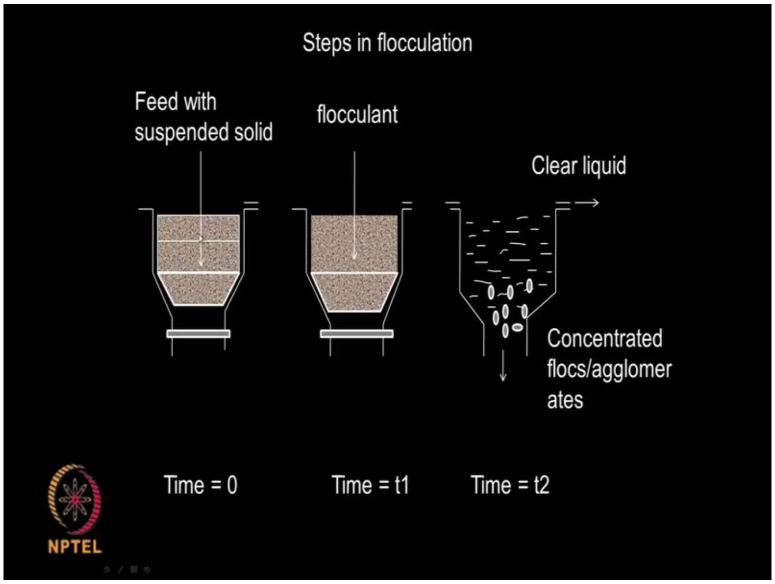


So it can be long chain polymer flocculants like polyacrylamides, so basically they do not have charges on them like acids or bases actually or like your inorganic salts. So the problem with these flocculants are the fouling. They can foul your membrane, number one. Number two they became impurity, if you are interested in the solid particles, then they are impurities that will be present in the solid particles, then it becomes very very difficult for you to separate them out.

If liquid is your desired product, then there is no problem actually flocculants can sort of foul your membrane. There are chemical flocculants and natural flocculants. Large number of flocculants are available in the market like aluminum chlorohydrate, aluminum sulfate, calcium oxide, iron chloride. In natural if you look chitosan, papain, so many things.. so many different types of material could be used as natural flocculants.

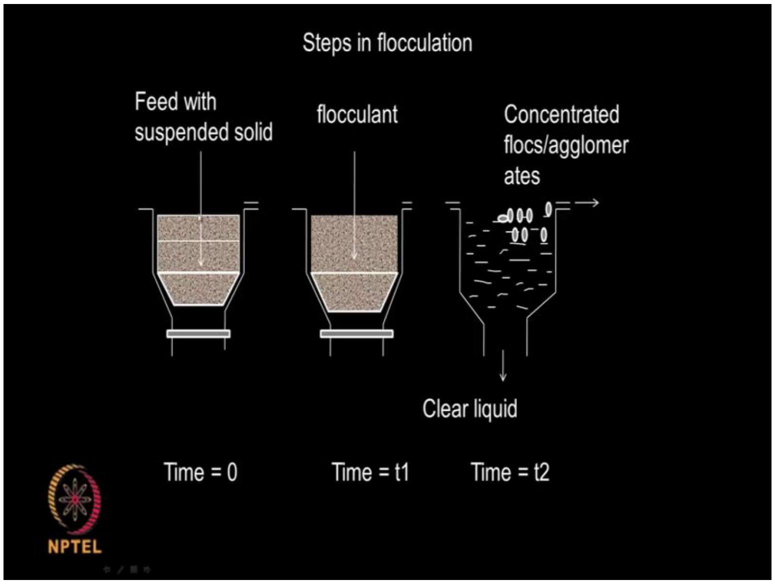
Polyacrylamide, sodium aluminate, sodium silicate, so large number of flocculants are available and of course they also start becoming an impurity. You need to think of removing them if they are very toxic or you discharge them into the open with the solids that you are throwing it out actually. So the solids will also contain this extra chemical which are adding to destroy the charge neutralization.

(Refer Slide Time : 14:13)



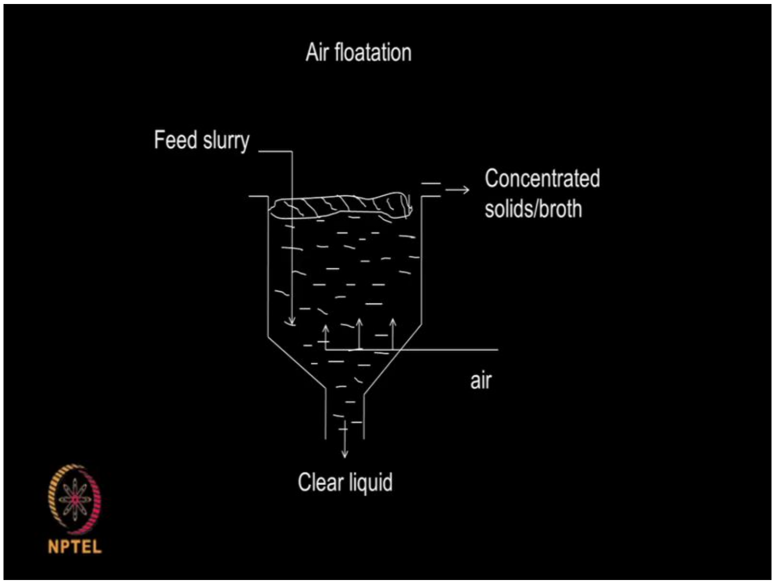
So what are the various steps in flocculation? So you may add the flocculants with the solid okay or you may add them separately suppose we have the feed with the suspended solid, you may add the flocculant. Then when you add the flocculant, the solids become very heavy the flocs become very heavy and they will all settle down so the liquid is removed. So it is a sort of a batch process you know this is at time 0 then you are adding flocculant at time t1 and at time t2, you can remove the liquid at the top and the concentrated flocs or agglomerates at the bottom.

(Refer Slide Time : 14:57)



Now analogous to that, if the flocs are lighter than the liquid, then it may float. So at time equal to 0 you take your feed, then you add your flocculant at time equal to t1 and then after sometime the solids which are floating or skimmed off or removed at the top and the liquid is at the bottom just clear so both the approaches are possible depending upon the flocs that are formed later or heavier or lighter than the liquid.

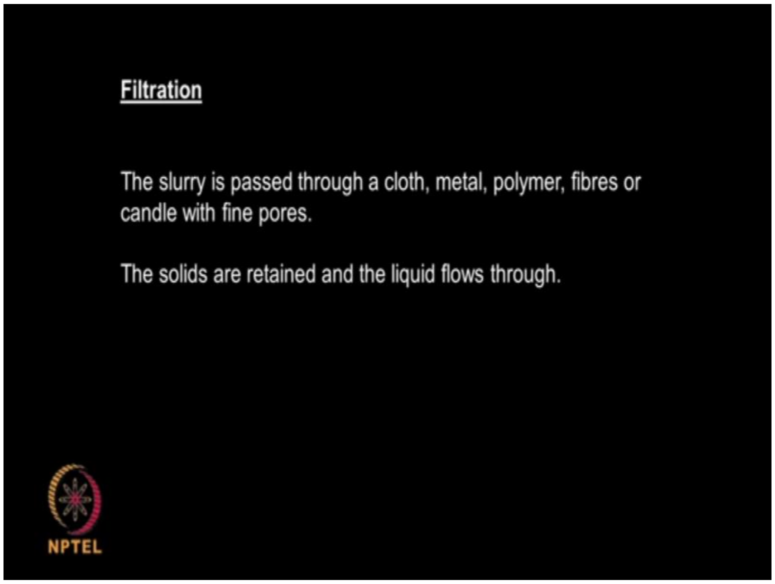
(Refer Slide Time : 15:24)



Now there is another method that is called air flotation. So what you do is, again the particles, suspended solids are very light, they do not agglomerate because of the charge. So you bubble air, so the air acts as some sort of trapping the solids and you end up having broth which will have lot of concentrated solids on the top. Because air is there generally the density will be lower so on the top you have broth and all the solids trapped which you will be which can be skimmed here and the clear liquid can be collected on the bottom so here you require air here.

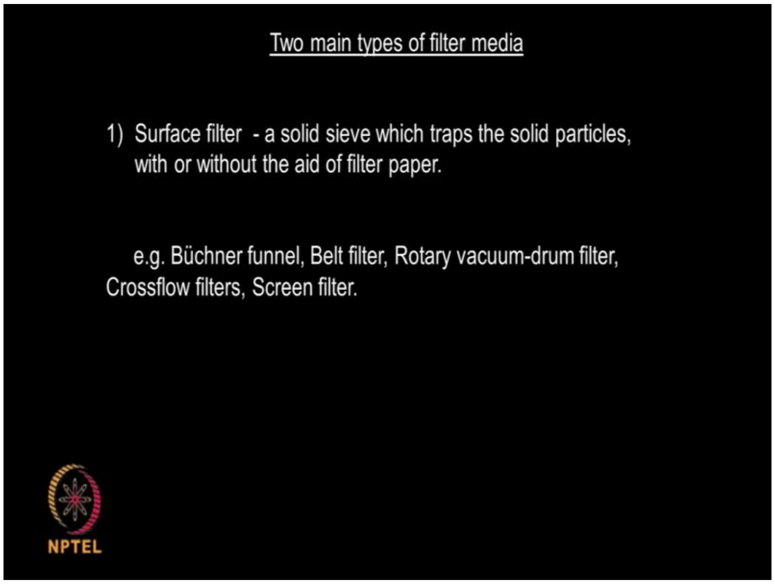
The advantage of this method is you are not adding extra chemical and contaminating the setup the setup you are just bubbling air into it so that the solids are the fine particles are trapped in the air which can be skimmed out. So this is much more advantageous than adding a flocculant or adding a coagulant but of course in a very large systems that may become a problem. So it is much easier to add a coagulant and in a water treatment especially, we generally use a coagulating agent.

(Refer Slide Time : 16: 44)



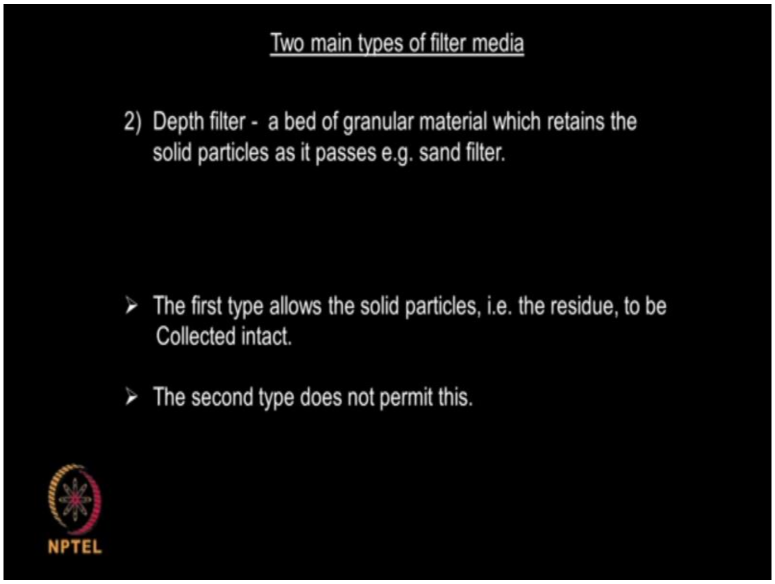
Okay now we saw sedimentation, we saw flocculation and so on now the next one is filtration. Okay you must have all seen filtration from very early ages, so the slurry is passed through a cloth, metal, polymer fibers or candles with fine pores solids get retained and liquid flows through (())(17:02)

(Refer Slide Time : 17:05)



There are two types of filtrating system, one is called the surface filter. When you have a solid sieve or a solid piece of cloth or metal or ceramic like your Buchner funnel belt filter or rotary vacuum-drum filter all these come under surface filter. So the solids are retained on the surface, the liquid flows through, that is why the name is called solid surface filter.

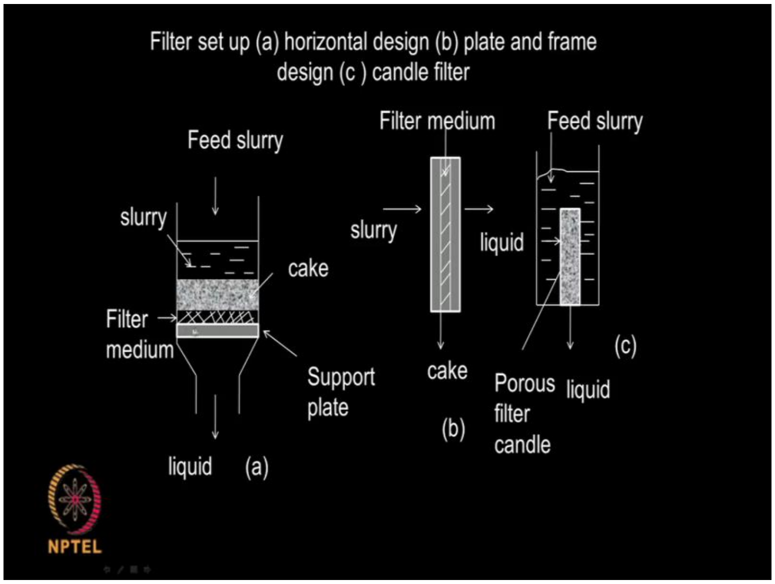
(Refer Slide Time: 17:32)



So similarly we have some death filter here, you can have a long tube which may contain solid particles or sand or gravel, so when your slurry flows through the solids hit these various solid particles and they get trapped and the liquid flows through, so the typical example is sand filter. Here we cannot collect the solids whereas in the surface filter we can collect the solids and if you are interested in the solids, then surface filter is always better when I ended up in the depth filter you cannot recover the solids okay.

So the first type okay, the residues can be collected intact, the second type does not permit. So the sand filter like you have a bed of sand where liquid flows through with the solids, the solids get trapped by the sand especially they are used in the water treatment okay so this is very simple setup this is very cheap setup. Rain water harvesting, they use a sand filter so the sand just traps the solids that are flowing through and generally this is used the concentration of the solids are very very low whereas in this first method they used the concentration of the solids are much higher.

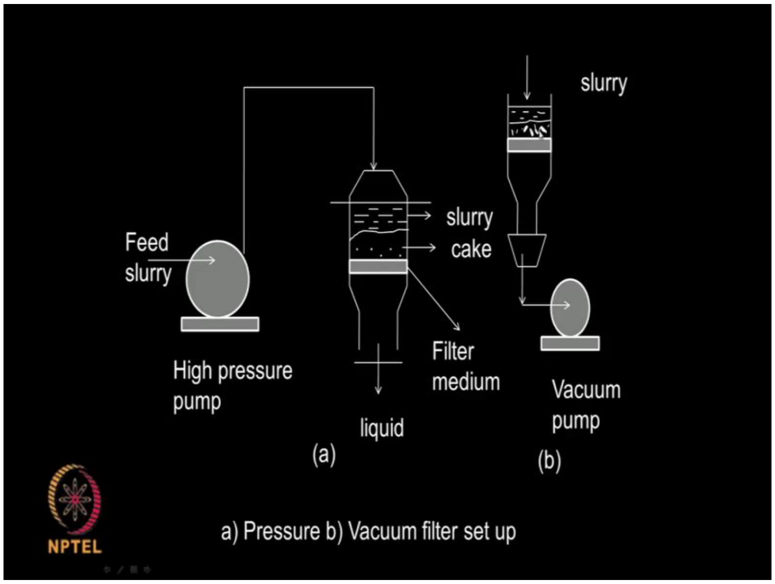
(Refer Slide Time : 18:58)



Okay so in a filter you can have this type of design, this is horizontal design or this is also called dead end filter, so the feed slurry comes there is filter cloth or filter medium here is called filter medium. It could be a metal ceramic and so your cake is formed here the liquid flows through liquid is collected here right. Or you can have plate and frame design so you have a frame you have a plate, porous plate through which your filter medium is placed slurry flows through cake is formed it is removed here

And then you collect the liquid, so this is a batch process because after sometime the cake accumulate so much that you need the flow of the liquid goes down and down so you need to stop it scrape out the cake and remove it. Also in this system also you need to stop the filtration remove the plate and frame again remove the cake that is formed again restart the whole process. This is a candle filter, so there is a candle which is it could be a ceramic or metal or porous holes, so the liquid slurry is outside. So the liquid flows through the solids are retained and the surface and the pure liquid comes down.

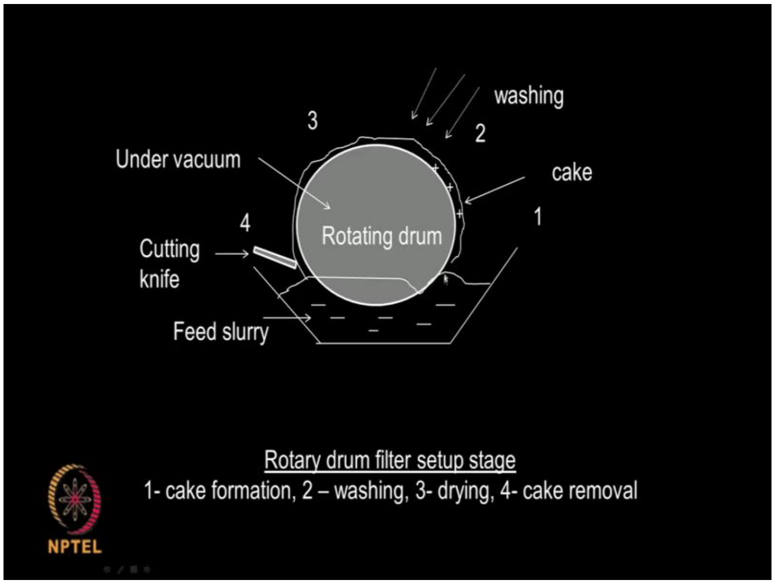
(Refer Slide Time : 20:30)



So this is generally used in water treatment even in households you may see. Okay so you can have pressure type of filtration, you can have vacuum type of filtration here. So pressure means this slurry is forced on top of the filter medium under pressure whereas in the vacuum, a vacuum is used at the bottom so that the slurry is sucked, the solids are retained and the liquid goes down and so both the techniques are commonly used the pressure type and vacuum type.

So if you are using the pressure type, we can achieve very large pressure radiance whereas vacuum you get only delta p of one bar so that is the main difference between pressure and vacuum-drum filtration.

(Refer Slide Time : 21:04)

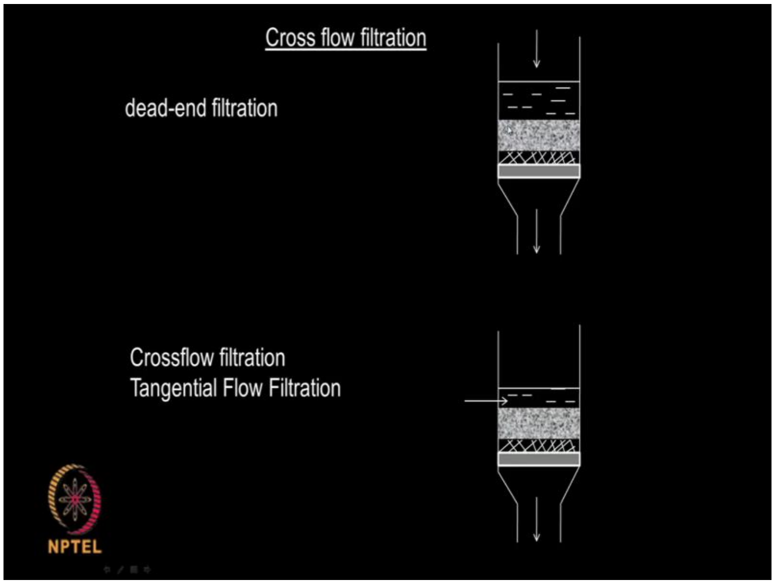


Okay you also have something called rotary drum filter. So there is a big drum it is porous and there is a vacuum inside, so this slurry is sucked, so the solids are coating on the drum liquids go inside this drum keeps rotating slowly, so the solids are settling and coating on the top of the drum and cake is formed here so we can wash the cake. In case there are some impurities which are washable, dissolved in water and that gets washed and sucked inside then we can dry it and finally the cake can be cut here.

So again the drum is empty or clean which dips into the slurry and again cake is formed. So this is a continuous process, the rotary drum filter is a very continuous process because you are forming the cake when the drum is dipped inside your feed slurry, so the cake that is formed can be washed here so that you can remove the impurities that are formed at the top and then you can dry and then you can finally you can cut it and recover all the wet solids.

And again the drum dips inside, so this is generally used in lot of pharmaceutical industries. So you are removing a solid from a slurry you are washing the solids partially drying it here because of the vacuum here it is partially dried it is cut and collected. Then later on you may go into further downstream like complete drying or some modifications and so on actually. So these rotary drying type of filter are generally continuous operations like the previous filters which we saw they are all batch operation okay.

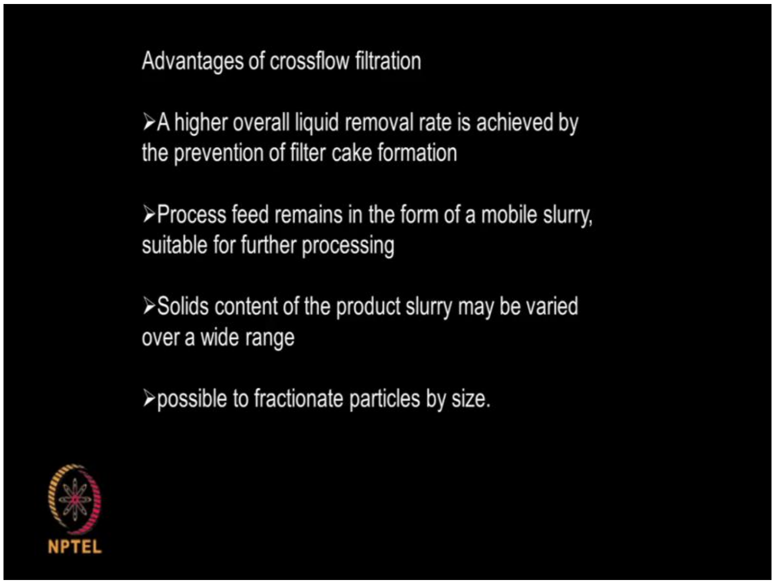
(Refer Slide Time : 22:44)



Now two types of filtration you need to know, the dead-end filter the liquid flows the slurry flows like this, so you have the cake that is formed at the top and the liquid is collected. So the problem in this type of filtration is, after sometime the cake thickness increases and increases and the flow of the liquid become negligible you need to stop it and you need to scrape off all the cake clear the top of the filter medium so that again the filtration can be started.

Whereas in the cross flow the liquid is the slurry is flowing in this direction okay cross. It is flowing tangential to the filter medium, so cake formation is very low. So this type of accumulation of cake is very less, so this is much more advantageous because the cake formation is very low well so the liquid and the filtration is always much more when compared to the dead-end type of filtration okay these are the two types one is the dead-end another is the cross flow filtration okay.

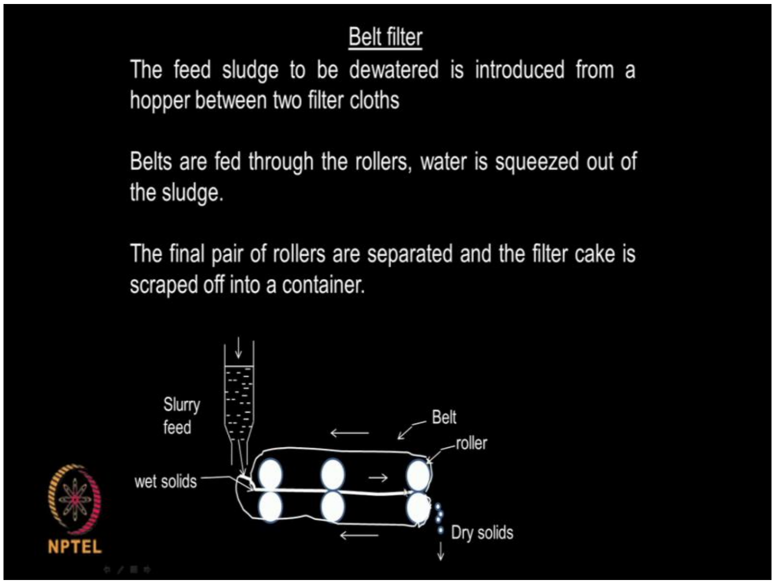
(Refer Slide Time : 23:50)



What are the advantages of cross flow? The liquid removal rate, a good liquid removal rate is achieved because you are not allowing it to form large thick cakes and in a dead-end thick cakes make the liquid flow less and less and after sometime the liquid flow can totally stop actually. And the feed always keeps moving on top, so we can take it and do further processing unlike thick cake thick and dry cake where we cannot do further processing unless you put it again in a slurry.

And the solid content of the product slurry may varies over a wide range and we can even use this for fractionating particles by size. So we can have initially filter a cross flow filter with very fine holes and then you can have bigger holes and so on actually so you can have a fractioned type of solid separation whereas we cannot do that in dead-end type of filtration.

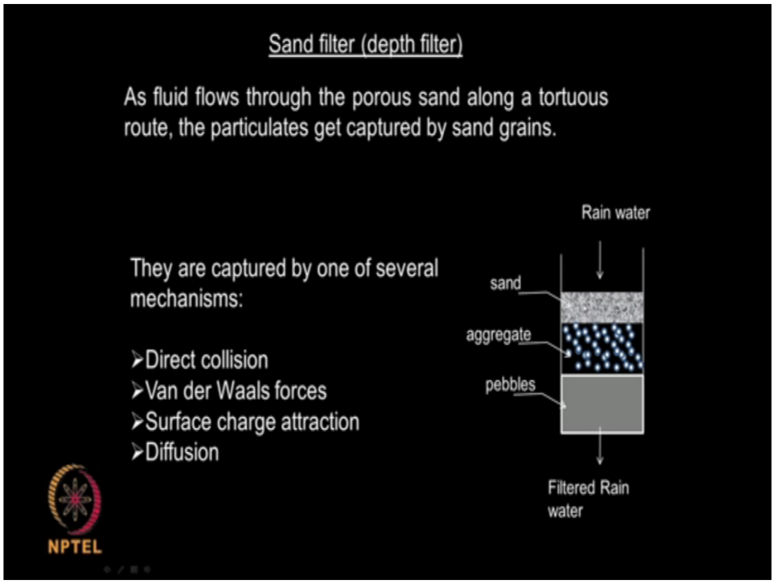
(Refer Slide Time : 24:51)



Then you have this belt filter, what happens is, this is a long belt this can be hundreds of feet long and the feed slurry is charged here okay, the feed slurry is charged here and then the it moves along between two rollers okay so the water is squeezed out as you go through these rollers actually. There are 2 different belts and the solid is between is these rulers and because of these rollers, the water gets squeezed out and then at the end of it the dry solids fall off.

So initially it is entering as a wet solid and it get squeezed and squeezed between 2 belts, so water gets removed and finally it falls out as dry and again the belt rolls so we can achieve continuously removing the liquid from the solid as well as to make the solids little bit dry. Later on we can go to dryer to completely remove the moisture. Okay. That is possible actually.

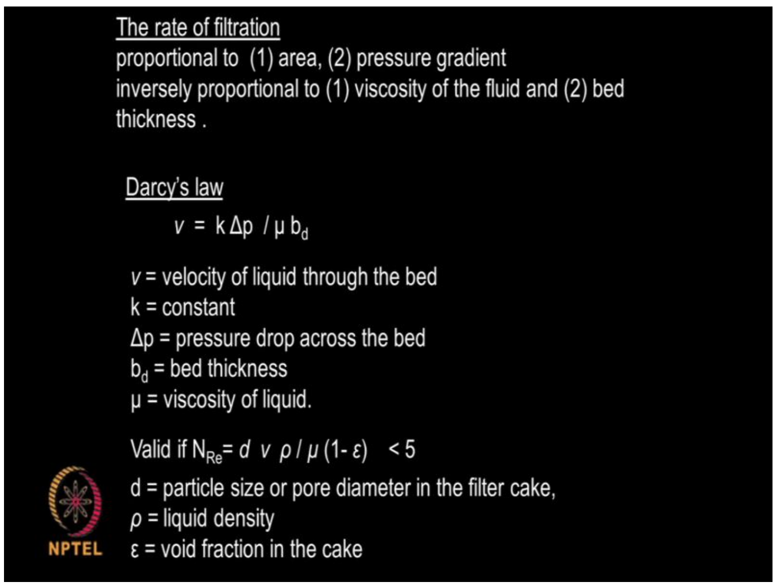
(Refer Slide Time: 25:56)



Depth filter that is called the sand filter or the depth filter so you have a bed pebbles which are of larger size then you have aggregate which are of smaller size then you have sand which are smaller much smaller size. So for example rain water can flow here and the particles that are present the impurities that are present gets captured be because it hits the solid material and gets captured and the liquid has to flow through a torturous part so the solids are removed and we get the filtered water.

So generally this is practiced in rain water and you do not need any mechanical moving parts actually so how do they remove this solids from the liquid in this sand filter direct collision Vander walls forces surface charge attraction diffusion so all these are present in a sand filter and that is how the solids are removed from the liquid. Generally sand filters are used if you have low concentration of solids whereas the other type of filter surface filters are used in the solid present is much much larger okay.

(Refer Slide Time : 27:08)



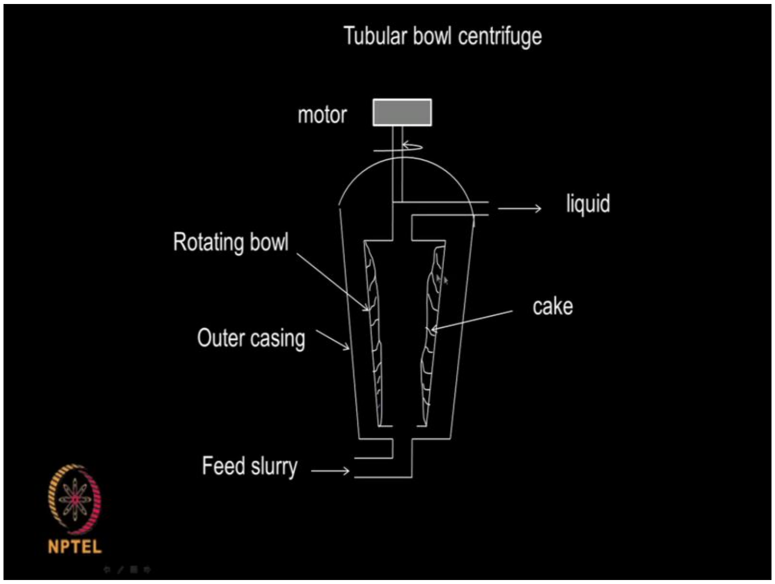
Now generally filtration is based on Darcy's law. We are going to spend some time on Darcy's law and this is what filtration is based on. Where the velocity of flow of the liquid through the bed is directly proportional to the pressure difference inversely proportional to the viscosity and v d is the bed thickness. So the bed thickness is higher and higher your velocity goes down if the bed thickness is lower and lower the velocity goes up.

And the driving force plays a very important role. K is a constant, it depends on the compress, so many factors, the compressibility and so on actually but this Darcy's law is valid only if the Reynolds number is less than 5 but it is a very useful equation we will look at some problems later actually okay.

(Refer Slide Time : 28:02)

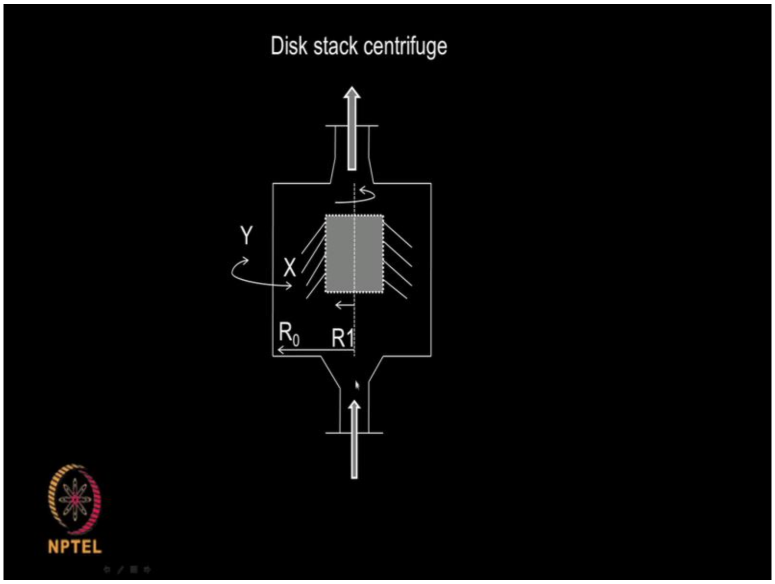
Commonly using filter medium, filter paper, woven material, cheese cloth, polymer fiber, woven glass, non woven material, sintered, perforated glass, perforated metal, ceramic, synthetic membrane, so almost anything can be used you can directly perform filtration at high temperature, room temperature and so on.

(Refer Slide Time : 28:20)



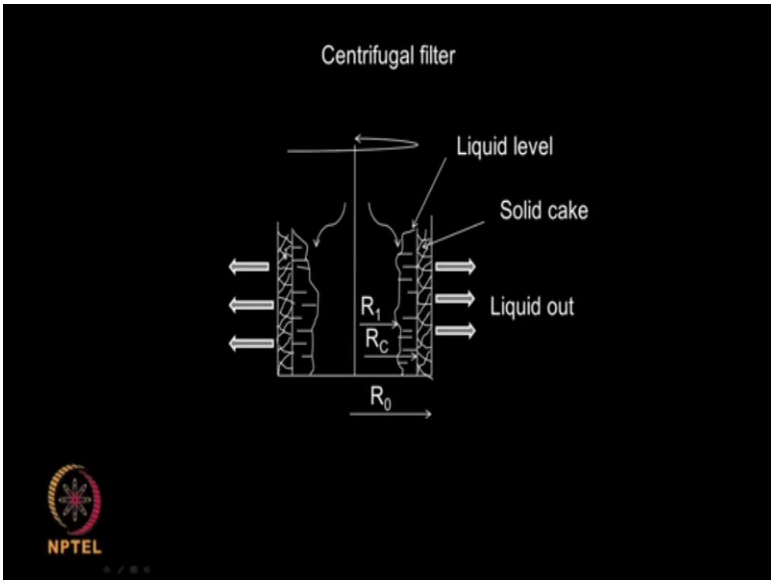
Okay after filtration, we have centrifuges. This is called a tubular bowl centrifuge. There is a ball here, so your feed slurry comes and it is pushed upwards because of the force, centrifugal forces, the solid hits the wall and it gets stuck to the wall the clear liquid flows through so after sometime, there will be buildup of solids on the surface of the bowl, so you need to stop the centrifugation and scrape of the solids it is called tubular bowl centrifuge because it is tubular in shape and there is a bowl.

(Refer Slide Time : 29:02)



You have disk stack centrifuge, you have lot of disk here, okay so the liquid is flow the slurry is flowing from the bottom and it hits this angular disk and the solids get thrown out and the liquid flows upwards so that because of this the solids gets thrown out and hits the wall and they are collected at the corners of this wall. So the solids are collected at the corners at the walls the liquid flows through, so that is called the disk stack centrifuge because we have disks.

(Refer Slide Time: 29:34)



We can also use centrifuge for filtration also in the outer walls of the centrifuge are porous, then when the slurry hits the wall, the liquid will come out through the hole, the solids will be trapped inside and it will form a coating and surely the solid will start building up. So the centrifuge can also be exactly looking like a filter so the liquid comes out the solids keep accumulating along the walls of the cylindrical centrifuge and they start building up.

So today we saw different types of solid liquid separating units like sedimentation, flotation, air flotation, filtration, two types of filtration, the depth filtration as well as the surface filtration and then we looked at the centrifugal systems like ball centrifuge, disk centrifuge, centrifugal filter and so on actually so we will do some problems in the next lecture.

Thank you very much!!!