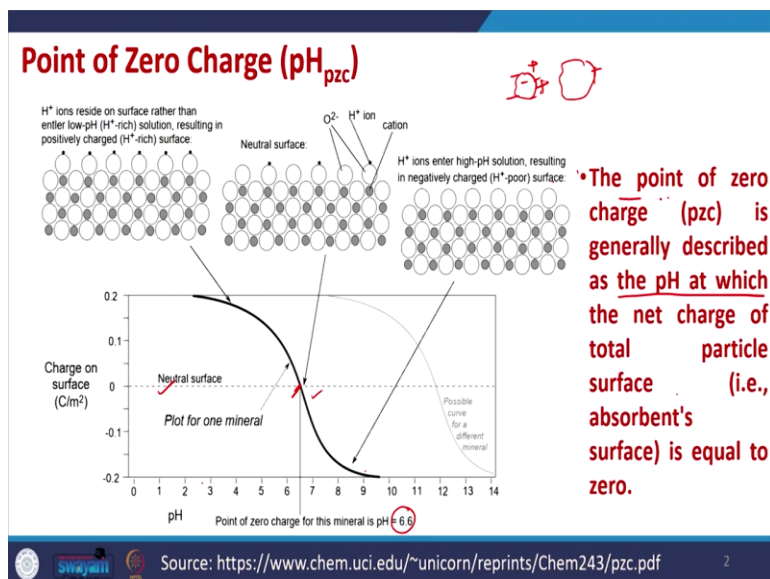


Physico-Chemical Processes for Wastewater Treatment
Professor V. C. Srivastava
Department of Chemical Engineering
Indian Institute of Technology, Roorkee
Lecture 19
Coagulation and Flocculation - II

So good day everyone and welcome to this lecture with respect to treatment, physico-chemical treatment of water and wastewater. And today we will be continuing with respect to the previous lecture. And that was in the previous lecture, we started with the coagulation and flocculation. We studied the basic fundamentals of the coagulation that how the coagulant work and what are the initial steps and then we started with the basic theory of colloids, we studied regarding the electric double layer, and within the electric double layer theory, we found that we have like, two three types of potential which are very important.

So, Nernst potential, Helmholtz potential and Zeta potential and out of that zeta potential is the one that we measure a lot and that gives the idea regarding the collide stability, so, if the zeta potential range can be reduced to plus-minus 20 millivolts or less, then it is very easy to use the coagulation-flocculation technique for removal of all those suspended particles, which cannot settle down easily or otherwise without using the chemical coagulants. So we will continue with the coagulation and flocculation today and one of the very important property and which is used very commonly in coagulation-flocculation in absorption and that term is called point of zero charge.

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- The suspensions were then manually shaken and allowed to equilibrate for 48 h with intermittent manual shaking and the pH value of the supernatant liquid was noted.
- The difference between the initial and final pH (pH_i) values ($\Delta\text{pH} = \text{pH}_0 - \text{pH}_i$) was plotted against the pH_0 .
- The point of intersection of the resulting curve at which $\Delta\text{pH} = 0$ gave the pH_{pzc} .
- This procedure was repeated for different concentrations of KNO_3 .

So, what is point of zero charge, so, it is described as the pH, at which the net charge of total particle surface, absorbent's particle or colloidal particle is surface is equal to zero. So, this is there and it is true for absorbent's it is true for colloids also. So, and in the previous lecture, we also studied that pH is very important. Below a certain pH they may have positive charge above a certain pH they may have negative charge. So, this is a possibility.

So, we always try to find the point or the pH at which the surface charge and the collide is neutral. So, this is there. So, there are different types of curves which are possible. So, for one of

the minerals, it may be there, in the same mineral are same collide, if suppose some other particles have got a job. So, it may move, move either ways left hand side or right hand side. So, this point of zero charge may change. So, for here it is 6.6. But this point of zero charge may change.

Now, why point of zero charge is important? First thing is this. So, this is important because above that any particle will be negatively charged and below that, any particle will be positively charged below the point of zero charge in terms of pH. Now, if we can manipulate this point zero charge sometimes by absorbing some amount of other materials.

So, we want to manipulate this zero charge, point of zero charge and also we want to operate in a range of pH where the zeta potential will change with pH. So that means the stability also will change with pH. So, this type of study with respect to point of zero charge, certainly the particles will settle easily our colloids will work better if our point of zero charge of the collide is in that range.

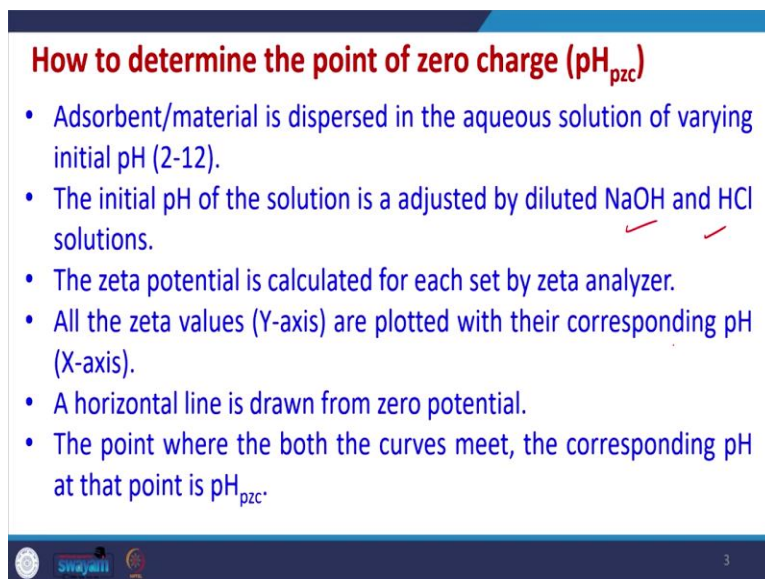
So sometimes you may have to adjust the pH. Sometimes we may have to select the chemical depending upon the pH or point of zero charge, the point of zero charge at whatever pH it is. So this, it is very, very necessary to determine the point of zero charge of the colloids, as well as of the Coagulant because the Coagulant's may have different negative charge. So, earlier we discussed that we want to settle down the negatively charged particles.

Now, in this case what will happen? So, particles are negatively charged and we want to add some coagulants which we will be having positively charged. So, because they will neutralize this. So, at what pH condition they are going to whatever collide coagulants we are adding they have positive charge negative charge or otherwise.

So, all these things we can determine with by studying the characteristic with respect to pH and that gives a lot of idea regarding the type of charge which is there on the colloidal particles, that type of charge which is there in the coagulants which have been added. So, these because the coagulant itself after being added they react with the different species present in the water like hydroxyl ions as well as the H plus ions and depending upon pH they may form different types of negative or positive charge.

So, that all those things basics we can understand why missing the data potential and then determining the point at which they have virtually zero charge on their surface the colloids. So, how to determine the point of zero charge. So, any of the colloidal material, absorbent material, what we do is that we disperse them in the various like, for colloidal solution already so, the solution is there, we can change the pH a little bit by using the NaOH or HCl solution with dilute HCl solution or dilute a NaOH.

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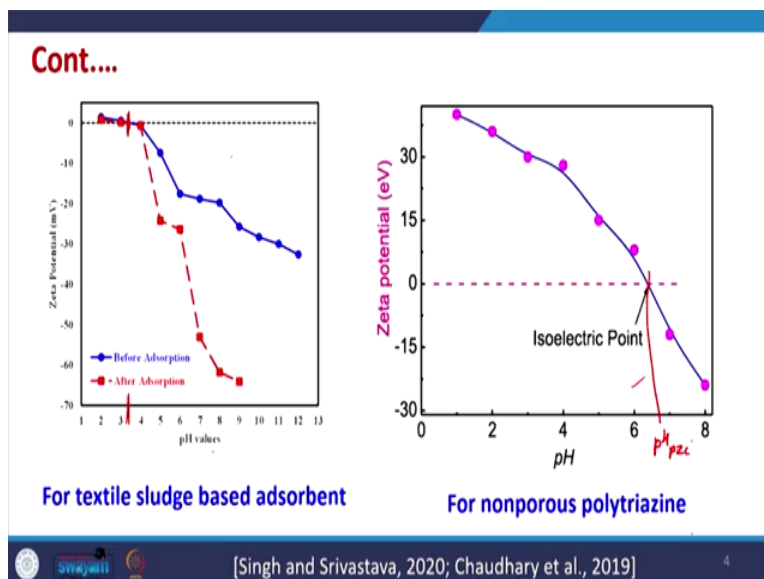
How to determine the point of zero charge (pH_{pzc})

- Adsorbent/material is dispersed in the aqueous solution of varying initial pH (2-12).
- The initial pH of the solution is adjusted by diluted NaOH and HCl solutions. ✓ ✓
- The zeta potential is calculated for each set by zeta analyzer.
- All the zeta values (Y-axis) are plotted with their corresponding pH (X-axis).
- A horizontal line is drawn from zero potential.
- The point where the both the curves meet, the corresponding pH at that point is pH_{pzc} .

So, we want to use the minimum amount of this. So, if we have to change the pH a lot then under that condition suppose the wastewater pH is initially 8 and we want to change it to 3. So, we it will be better to use a stronger acid because with one drop we can change it and otherwise if we use dilute very diluted HCl solutions we will add to add a lot of HCl. So, it will overall dilute the solution also. So, we can use the diluted or strong solution NaOH or HCl depending upon how much pH change we have to do.

Then the zeta potential is calculated for each set at each of the pH by the zeta analyzer and all the zeta values, y axis are plotted with their corresponding pH values like on the x axis and the horizontal line is drawn with respect to zeta potential and the point at which both the curves meet the corresponding pH is called the point of zero charge.

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So, here we can see the point of zero charge, we have for here we have for adsorbent it is shown also we can show for different types of other types of material. So, we can always for wastewater we can directly measure for adsorbent, we will have to dissolve in different types of materials etcetera. So, before adsorption after adsorption, we are always interested at this point and the pH corresponding pH here this will be around this. So, this gives the point of zero charge pzc and this can be represented differently in the literature.

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Another method to determine point of zero charge (pH_{pzc})

The solid addition method:

- In 100 ml conical flasks 45 ml of KNO_3 solution of known strength was taken.
- The pH_0 values of the solution were adjusted between 2-12 by adding either 0.1N HNO_3 or NaOH .
- The total volume of the solution in each flask was made exactly to 0.05 dm^3 by adding the KNO_3 solution of the same strength and the pH_0 of the solution was accurately noted.
- One gram of adsorbent ^{powder} was added to each flask, which were securely capped immediately.

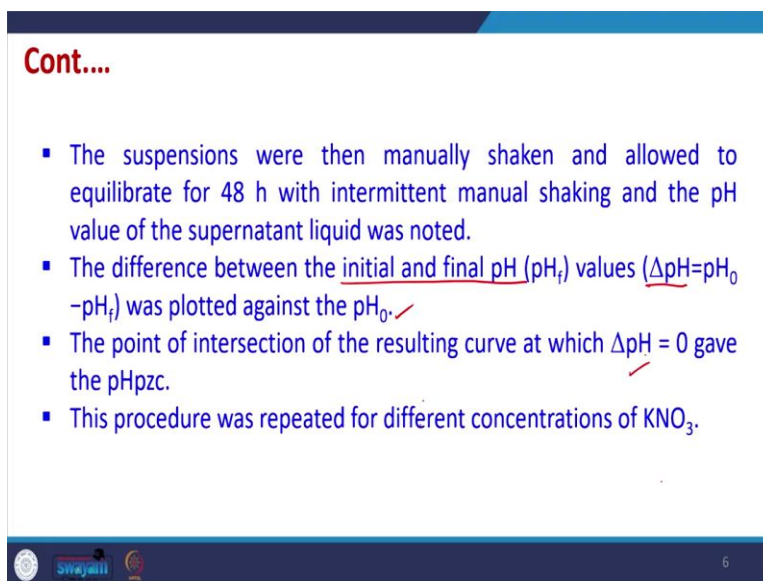
There is another method to determine the point of zero charge of absorbents and other types of solid materials and this is called titration method or solid addition, it is called solid addition method. So, in different conical flask, we prepare different types of potassium nitrate solution of known strength. So, strength may be different and then what is done is that the pH value of these solutions is adjusted between 2 to 10 by adding some different amounts of again acid and bases.

And then the total volume of the solution is, in the each flask is kept around 50 ML. We can change it depending upon our experimental conditions and then what we do is that we add the absorbent or any other material into each of the flask or coagulant we can add the coagulant also to determine and each of the flask and then they are kept immediately.

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Cont....

- The suspensions were then manually shaken and allowed to equilibrate for 48 h with intermittent manual shaking and the pH value of the supernatant liquid was noted.
- The difference between the initial and final pH (pH_i) values ($\Delta pH = pH_0 - pH_i$) was plotted against the pH_0 . ✓
- The point of intersection of the resulting curve at which $\Delta pH = 0$ gave the pH_{pzc} . ✓
- This procedure was repeated for different concentrations of KNO_3 .

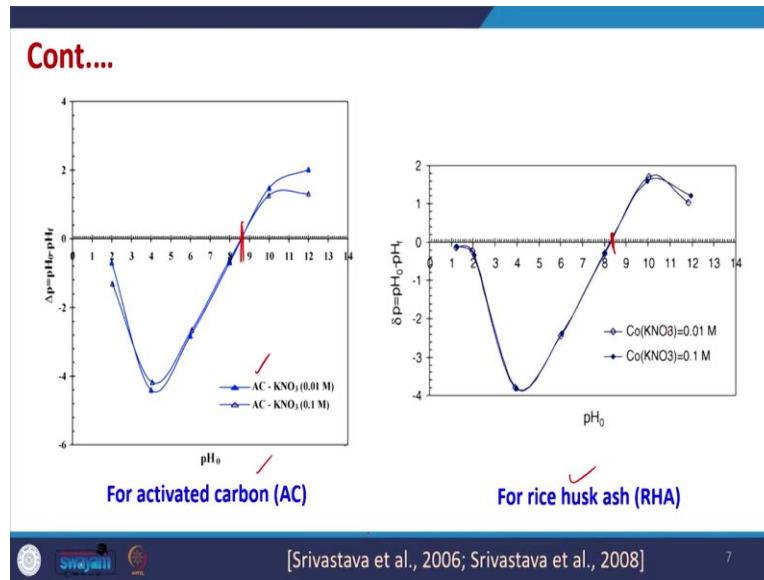


And after that the suspension is first manually shaken very quickly or otherwise it may be a shaken very quickly in between. Also and with intermittent manual shaking we continue equilibrate the solution for 48 hours or more, and the pH of the supernatant liquid is noted. So, any other coagulants if it is dissolved, so, after some time we measured it our for absorbent if it is dissolved, we measure it and the difference between the initial and final pH.

A Delta pH we determine So, we have initial pH we have final pH and this is plotted against pH zero. So, this is done and the point of their section of the resulting curve at which the delta pH is zero gives the point of zero charge. So, this can be repeated with different concentration of

KNO₃.

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There you can see here, this is a point of zero charge for one of the activated carbon. Similarly, with some other material it is shown here for rice husk ash. So, we can have different types of point of zero charge for different absorbents, we can use it for finding out for different other minerals etcetera also, so this point of zero charge is very important and we can determine using zeta potential or the solid addition method.

Now, when the coagulation is done, when we use coagulants. So, there are different types of mechanism by which removal happens or by which the coagulation happens. So, in the next stage, now, we are going to study these four mechanisms by which generally the removal happens during the coagulation process.

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Physics/Mechanisms of Coagulation

There are four mechanisms employed to destabilize natural water suspensions:

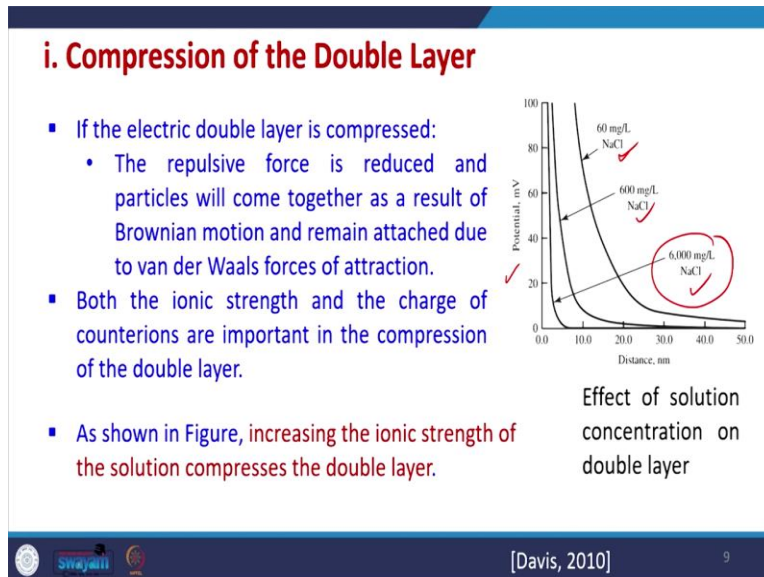
- Compression of the electric double layer ✓
- Adsorption and charge neutralization ✓
- Adsorption and inter-particle bridging ✓
- Enmeshment in a precipitate ✓

[Davis, 2010]

So, we are going to study in detail and these mechanisms are called as the compression of electric double layer, this is one thing, some idea was we discuss in the previous lecture that we always want to compress this electric double layer and if we can do so. So, the potential which is there will be reduced and we can overcome that potential easily. So, this is there. Second mechanism is called adsorption and charge neutralization.

So, through this we neutralize the charge by adsorption of opposite charge. So, this is there, then adsorption and interparticle bridging, this is one of the mechanism and then there is a sometimes a coagulants actually form precipitate depending upon the pH and that precipitate settles down and during settling we have, enmeshment of the colloids, that means the colloids get trapped inside the precipitate while the precipitate is settling down. So, through that mechanism also the removal may happen.

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Now, compression of the double layer. So, in this case if that electric double layer is compressed by any means, by using different amount of salts etc then the repulsive force is reduced and the particles can come together as a result of Brownian motion also and they can remain attached due to the van der Waals force of attraction. So, if we can compress the double layer and that can be done by additional addition of opposite charges. So, it is possible to do so. So, here we can see when the amount of NaCl is less the electric double layer will be more.

So, potential in millivolt is higher, we can see here we are, this is how the variation with respect to distance is shown, but when we are using very high amount of NaCl this electric double layer is reduced very drastically. So, ionic strength and the charge of counter ions are important in the compression of the double layer and increasing the ionic strength of the solution compresses the double layer. So, this is one of the basic fundamentals that can be used during coagulation as well.

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Cont....

- The van der Waals forces extend out into solution about 1 nm.
- If the double layer can be reduced to less than this, a rapidly flocculating suspension is formed.
- Increasing the ionic strength of the solution compresses the double layer.
- The ionic strength is much greater than would be acceptable for potable water. ✓

Ionic strength is calculated as $I = 1/2 \sum C_i Z_i^2$ ✓

where, I=ionic strength, mole/L; C_i =concentration of species i, mole/L;
 Z_i =number of replaceable hydrogen atoms or their equivalent
(for oxidation-reduction, Z_i is equal to the change in valence)

[Davis, 2010] 10

Ionic strength is calculated as

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where, I=ionic strength, mole/L; C_i =concentration of species i, mole/L;

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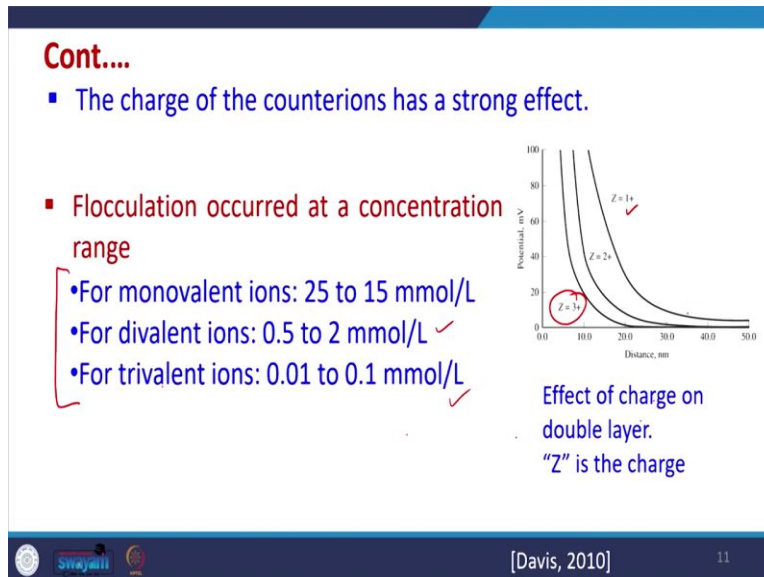
(for oxidation-reduction, Z_i is equal to the change in valence)

The van der waals forces which extend out only up to around one nanometer and if we can compress the near around this. So, van der waal forces of attraction will become dominant. So, this is what is desirable. So, if the double layer can be reduced to less than this, a rapidly flocculating suspension is formed and that will increase the size and later on in the suspended particles can be settled down. So, this is all the suspended particles solids etc can be settled down.

So, increasing the ionic strength of the solution compresses the double layer already we have discussed. So, if the ionic strength is much greater than that, that would be acceptable for potable

water. So, this is also the thing that we have to take care and ionic strength can be calculated using this. So, but we will have to see with respect to last point that the Ionic strength that we have to see that how much ionic strength will be acceptable in the potable water because it is related to TDS. So, that thing we have to see.

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Now, the charge on the counter ion also has a strong effect, if we see if the counter ion is only a mono charge, then still the potential has the effect up to a large distance. So, electric double layer is large. Now, if the charges plus three it is reduced drastically. So, we can very easily see the counter charge on the counter ion also has a very strong effect. Now, flocculation occurs in this zone for different types of ion.

For mono valent ions, it is in the range of 25 to 15 milli mol per litre or 15 to 25 milli mole per litre we can see for divalent ions. So, here the range is much reduced and for trivalent ions it is further reduced. So, the flocculation occurs at different concentration range depending upon the counter ions which are there. So, counter ions and ionic strength both are important fundamental for electric double layer compression.

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- Coagulants are not "indifferent," they will undergo many interactions in addition to electrostatic attraction and repulsion.
- For example, phosphate is present, substantially more trivalent coagulant will be required because the coagulant will react with the phosphate.
- If multivalent ions comprise the fixed layer next to the negatively charged particle:
 - The double layer will be reduced significantly and the critical coagulation concentration will be much lower than predicted by the Schultz Hardy rule.



[Davis, 2010] 12

Now, with coagulants also they may undergo interactions in addition to electrostatic attraction or repulsion. For example, if phosphate is present and more trivalent coagulants will be required because the coagulants will react with the phosphate under that condition. So, depending upon the types of ions which are present inside the water, different types of coagulants and coagulants with different ionic size will be required.

If multivalent ions are there, then they comprise in the fixed layer next to the negative charge layer, that double layer is reduced very significantly in place of single. So, we have a negative charge layer and there is a positive which is charged layer just adjacent to that in the previous lecture we had seen the photograph, so, we have a positive charge layer. Now, if this positive charge layer has ions, which are multi ions, then the overall double layer will also get reduced a lot. So, this has been predicted under Schultz Hardy rule, so this is there.

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ii. Adsorption and Charge Neutralization

- Hydrolyzed metal salts, prehydrolyzed metal salts, and cationic polymers have a positive charge.
- They destabilize particles through charge neutralization

The diagram shows a red circle containing two '+' signs, representing a positive charge. Below the circle, there are some faint, illegible markings.

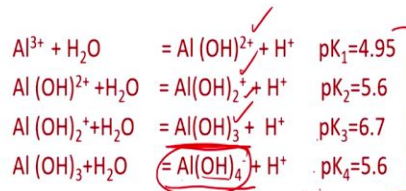
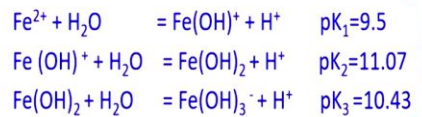
[Davis, 2010] 13

The second mechanism by which the removal happens is using the hydrolyzed metal salts. So, generally different types of coagulants and these coagulants are generally metallic salts which are there. So, these salts are added like aluminium sulphate alum may be added. Now, these get hydrolyzed as soon as they are added. So, similarly cationic polymers have a positive charge metal salts get hydrolyzed and get positive charge. So, they de-stabilize the particles because they have positive charge. So, generally they will be having positive charge and whether this is mono or di or tri that will depend upon the pH.

So, this we are going to study and when as soon as these charges are added, they neutralize the negative charge. So, overall zeta potential gets reduced and then also the particles can come together because we are providing very quick mixing and as soon as they are coming together the van der Waal force of attractions come into picture and they are able to attract different colloids together and they form a bigger colloids. So, this is there.

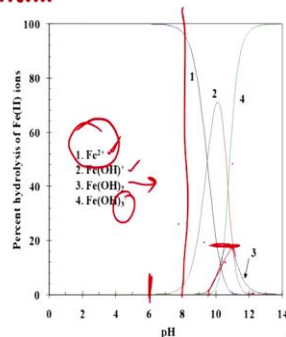
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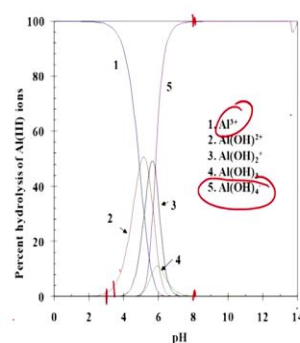


The speciation diagram of Fe^{2+} and Al^{3+} as drawn using above stability constants is presented as following:

Cont....



Speciation diagram of Fe^{2+}



Speciation diagram of Al^{3+}

[Ponselvan et al., 2009]

Now suppose we use some iron coagulant or aluminium coagulant. So, depending upon the pH and pK values, so, different types of pK values can be used together and they can be used to form a speciation diagram. So, a speciation diagram of Fe^{2+} plus. So, we are using any Fe^{2+} plus type of coagulant or Al^{3+} plus type of coagulant, the depending upon pH first the that alum will get hydrolyzed into Al^{3+} plus and sulphate, then Al^{3+} plus with react with water to form different types of ions.

So, we can see here, one is plus 2 charge, another is only single charge is there, only mono

atomic, then this is a neutral, this is a precipitate. And this is opposite charge in place of positive charge, they are forming negative charge, because this Al is combining with 4 hydroxyl radical.

So, this is opposite charge. So, the speciation diagram we can draw like the one this is shown in figure for Fe^{2+} plus and Al^{3+} plus. So, and these types of diagrams give a lot of idea that in which range different coagulants are going to act. Now for Fe^{2+} plus, we have this di chart. So, this is then mono chart and then there is opposite an ionic charge is there. So, if you see here up to 8 pH, if we use Fe^{2+} plus type of coagulant.

So, as soon as Fe^{2+} plus will be there up to 8 pH it will be in the form of Fe^{2+} plus only so that we this coagulant will work very well we within this. Now, if suppose the water the colloids which are present in the water are themselves positively charged in the pH range, because the water may have suppose the water is having a pH of 6. So under that 6 condition if we use the coolant, it will be Fe^{2+} plus.

Now if the colloids in the water at themselves positive charge, this Fe^{2+} plus will not work. So, this we have to see that at this condition, the colloids in the water may be negatively charged. Otherwise, we will have to see that what forms of these are present. So, we see at the these forms ferric chloride, this ferric this hydroxide of Fe^{2+} plus only in some particular range and only up to a certain value everything is not precipitate for this condition. If we use Fe^{3+} plus in fact, the amount of precipitate formation is higher and the pH range is also lower.

So, it will depend upon the pK value, the type of coagulant we are using and how they hydrolyze inside the water. So, this becomes very important for the coagulants that how they get hydrolyzed inside the water. For aluminium we can see here, so, it is only up to four or we can say three it is in the maximum cationic form. So, here the maximum charge is there, otherwise, the charge is getting reduced at the rate we are moving and suppose we are going to use this alum in 8 pH. So at the 8 pH it will be having negative charge because it is negatively charged. So it may not work.

So, this is very important the pH is very important, we should have a basic understanding of hydrolysis of metals how this happens, and this hydrolysis will vary with different types of other ions which are present inside the water, but that is also very, very important. And so, this

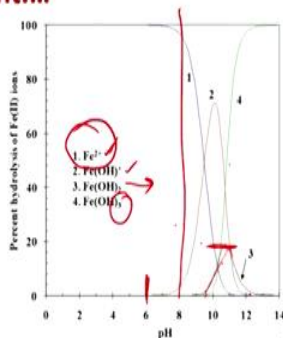
speciation diagram will change with the presence of other types of ions in the water. So, this is for pure water, but it may change with other.

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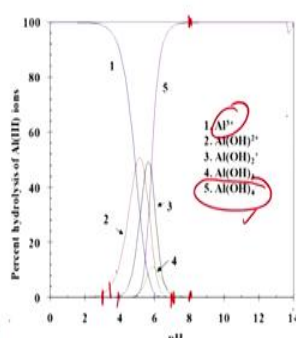
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- It can be seen from the speciation diagram of Fe(II) ions that the dominant soluble species are Fe^{2+} and $\text{Fe}(\text{OH})_3^-$ at low and high pH, respectively.
- The hydrolysis constants for aluminum cover a very narrower range, and all of the aluminum deprotonations are 'squeezed' into an interval of less than 2 units.
- Therefore, apart from a narrow pH region approximately 5.5–6.5, the dominant soluble species are Al^{3+} and $\text{Al}(\text{OH})_4^-$ at low and high pH, respectively

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Speciation diagram of Fe^{2+}



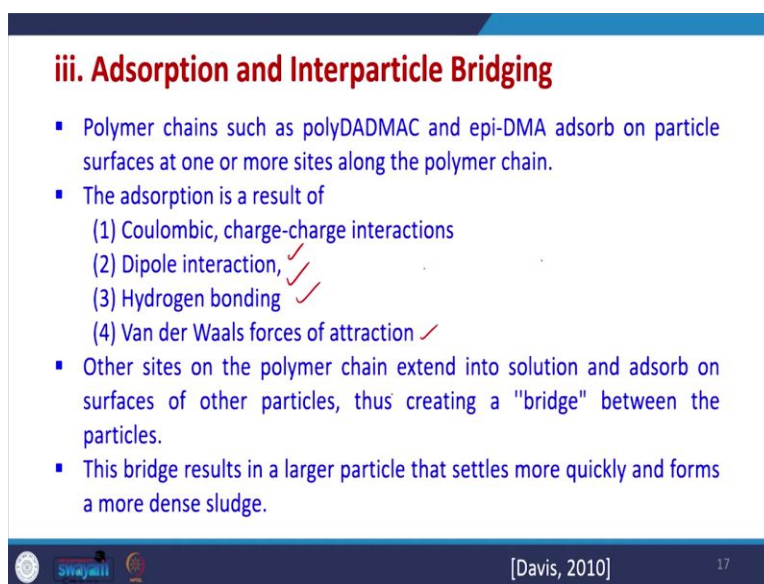
Speciation diagram of Al^{3+}

So, it can be seen from a speciation diagram of Fe2 plus and that the dominant solubility species are either Fe2 plus or $\text{Fe}(\text{OH})_3^-$ at low and high pH respectively. So, either it is Fe2 plus or this. And similarly for aluminium also. So, the hydrolysis constants of aluminium, they show that very narrow range they are there. So, the deprotonations are squeezed in the pH range of only two units we can see from around 4 to 6 or 7, 3 units, we have virtually whole entire change

happens from Al^{3+} to $\text{Al}(\text{OH})_4^-$.

So this gives idea that, the pH range in which these things may happen at them may work properly, we can determine from the speciation diagrams apart from narrow pH range, the this Al^{3+} plus OH^- that interchange takes place between very small pH range. So, the other types of dominant soluble species which are present are only in this range, so, it may work under certain conditions. So, depending upon other types of ions, this range may get broadened also. So, this is possible and we have to use the alkalinity has also a lot of importance during the usage of any coagulants or otherwise. So, that also we are going to discuss in the later section.

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iii. Adsorption and Interparticle Bridging

- Polymer chains such as polyDADMAC and epi-DMA adsorb on particle surfaces at one or more sites along the polymer chain.
- The adsorption is a result of
 - (1) Coulombic, charge-charge interactions
 - (2) Dipole interaction, ✓
 - (3) Hydrogen bonding ✓
 - (4) Van der Waals forces of attraction ✓
- Other sites on the polymer chain extend into solution and adsorb on surfaces of other particles, thus creating a "bridge" between the particles.
- This bridge results in a larger particle that settles more quickly and forms a more dense sludge.

[Davis, 2010] 17

But now, the third mechanism by which the coagulation works, it is called adsorption and inter particle bridging. In the previous case it was the adsorption of the ions on to the colloidal surface. So, that charge neutralization takes place. Here adsorption, these coagulants may work as adsorptions also and within coagulants, because many other coagulants are like polymeric in nature. So, there may be inter particle bridging also.

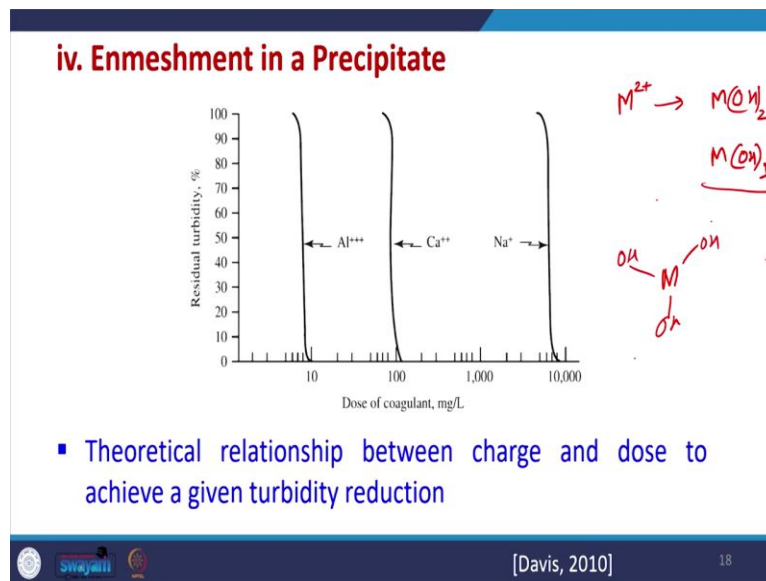
So, these coagulants in themselves may form a bigger size and they may in compass various types of other pollutants also. So, polymer polymeric chains such as polyDA, DADMAC or epi-DMA they adsorb on particle surfaces at one or more sites along the polymer chain and adsorption may occur because of various mechanisms like coulombic, Dipole interaction,

Hydrogen bonding, van der Waals forces of attraction.

So, all these are possible. So, adsorption of pollutants on these polymeric chains may happen in place of coagulation, the adsorption is occurring and during after adsorption because the colloids may interact with two different molecules itself. So, inter particle bridging is also possible. Other side on the polymer chain that extend into the solution can adsorb on the surfaces of other particles thus creating a bridge between the particles.

Now, this is possible and this bridge if formed, it will result in a very large particle that will settle very quickly and form a more denser sludge. So, this adsorption and inter particle bridging is very common in the adsorption in the coagulation process and it will be more common when we are using polymeric coagulants. So, this is there and sometimes we use Coagulants aid. So, these coagulants may add individually or as a Coagulants aid also so, that the removal is increased because they perform they have a lot of inter particle bridging which is possible.

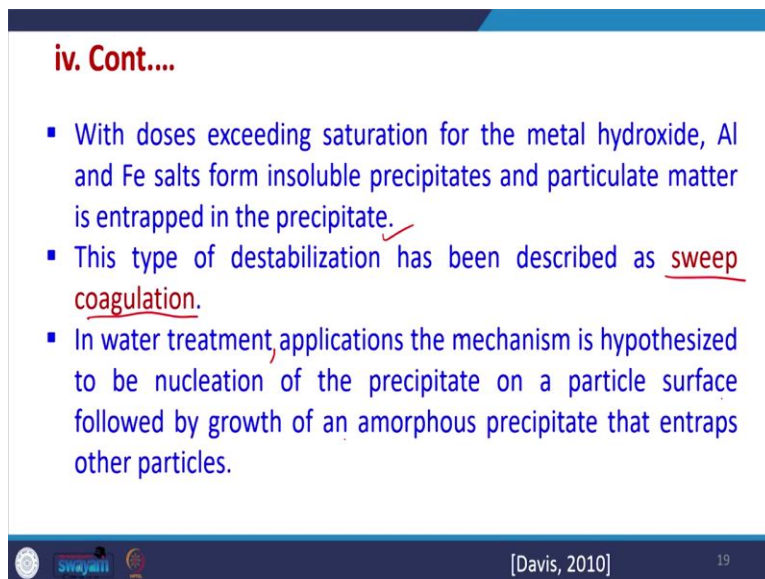
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Now, Enmeshment of precipitates. So, what does it mean here what we do is that whenever we are adding a Coagulants So, depending upon pH so, they may form respective hydroxide, so, if a metal is M^{2+} plus, so, it may form MOH_2 . Similarly, a MOH_3 et cetera is possible. So, this is there. Now, these metals they have bigger size because M is collected with 3 OH . Now, so, that means, they have much bigger size and also the molar mass is also higher.

So, they have they can settle down very easily. So, the while settling down they entrap other types of pollutants which are present inside the water and this because of this entrapment which happens those colloidal particles also get removed. So, we have lots of turbidity remover and other things happening during this process. Now, in this case or in the previous case, there is a very important thing that what should be the dose.

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iv. Cont....

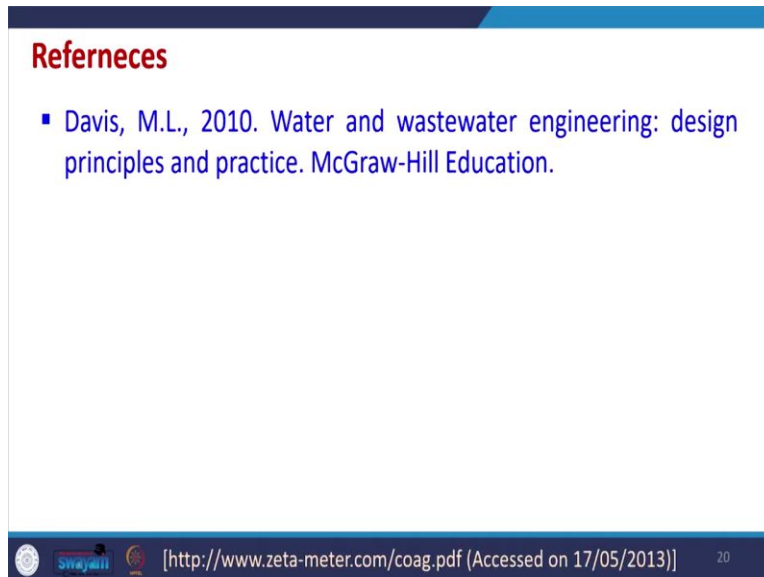
- With doses exceeding saturation for the metal hydroxide, Al and Fe salts form insoluble precipitates and particulate matter is entrapped in the precipitate.
- This type of destabilization has been described as sweep coagulation.
- In water treatment applications the mechanism is hypothesized to be nucleation of the precipitate on a particle surface followed by growth of an amorphous precipitate that entraps other particles.

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So, overall the entrapment process is also called as sweep coagulation also and sweep coagulation, because they are sweeping through all the other pollutants and colliding particles while it is settling down because of the formation of bigger size particle. So, this is there. So and that will happen with the precipitate formation more as compared to others. So precipitate with precipitate this deep coagulation is possible.

So, many times depending upon the pH chart neutralization may happen, sweep coagulation, may happen or the compression of double layer. It is also possible depending upon the what is the charge on the coagulant that we are adding and also the amount of coagulant or the ionic charge which is present in the solution. So, all any of these mechanisms may work during the treatment of water or wastewater by coagulation mechanism.

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So, in today's lecture we studied regarding the point of zero charge, it is important because point zero charge gives idea that whether anything will be positively charged or negatively charged. So, the pH at which it is neutral, it is called point of zero charge. So, below that pH below that pH generally it will be it will be positively charged, above the point of zero charge it will be negative charge. So, determining the point of zero charge helps in determining the pH range at which we should operate one thing.

Second thing where the coagulants will work more properly, because the first coagulants may require positive charge or negative charge depending upon the type of charge which is present on the colloids in the water. This point of zero charge is also very important for adsorption and adsorp. These colloids, these coagulants also work as a adsorption many times adsorbent.

So, that is why this is very important to understand point of zero charge. Now, during coagulation, there are four basic mechanisms by which removal may happen. So, those four we have discussed in detail. So electric double layer compression, then adsorption and charge neutralization, the second mechanism, then adsorption and inter particle bridging and then the fourth mechanism which is called a sweep coagulation. So, all the mechanism we have studied.

Now in the next lecture, we will try to know more about the different types of Coagulants, how they work. And then later on, we will perform some calculations or other things that how we can

calculate the dose etcetera of the Coagulants, how much dose will be required, and how much sludge gets generated because all these Coagulants, they settle the colloidal particles to the bottom. So we have a lot of sludge generation that takes place so we can perform lots of calculations with all those things. So we will continue the next. Thank you.