INDIAN INSTITUTE OF TECHNOLOGY ROORKEE

NPTEL

NPTEL ONLINE CERTIFICATION COURSE

Biomedical Nanotechnology

Lec-03
Synthesis of Nanomaterials by Physical and Chemical Methods

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Hello everyone today we are going to see the third lecture of this course, the third lecture is on synthesis of nanomaterials by physical and chemical methods.

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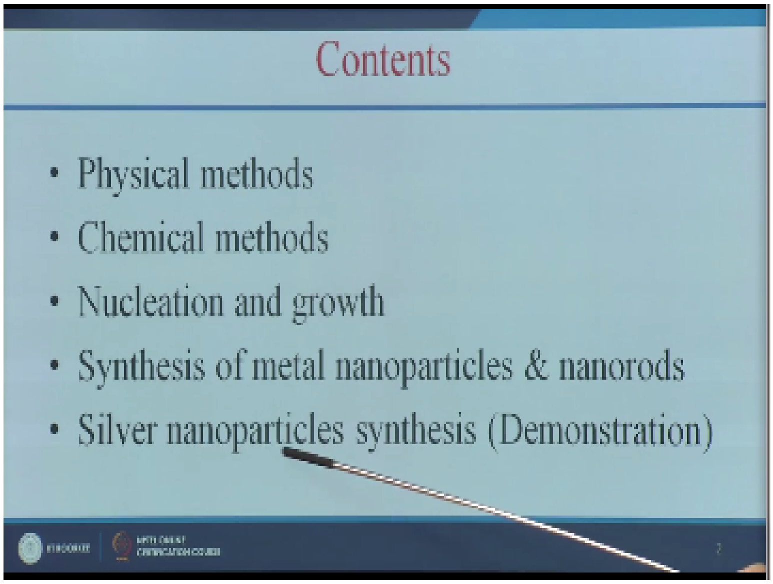

So in this lecture we are going to synthesize nanomaterials by physical methods and chemical methods. And we are also going to learn what is nucleation and growth, and how to synthesis metal nanoparticles and metal nanorods. And we are also going to see a small simple experiment to understand how we can synthesis silver nanoparticles in the lab with the available chemicals.

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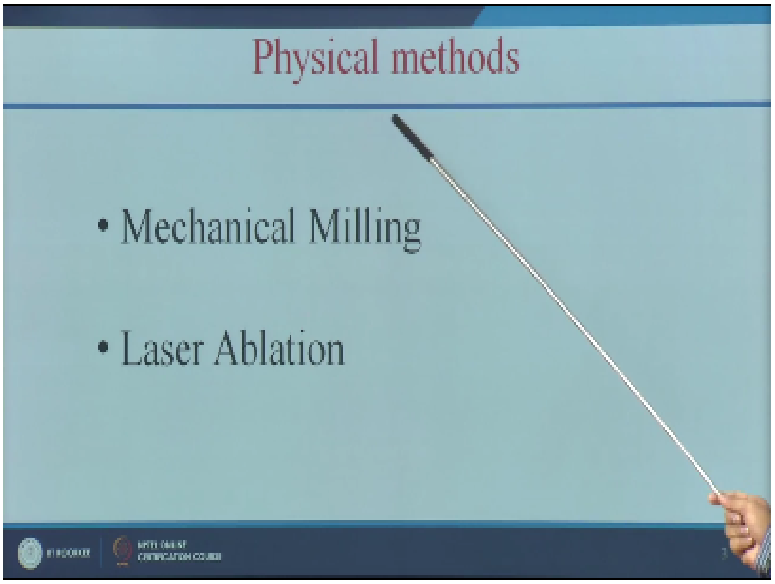

So let us see physical methods, under the physical methods there are several types are available, but in this lecture we are going to mainly focus on mechanical milling and laser ablation.

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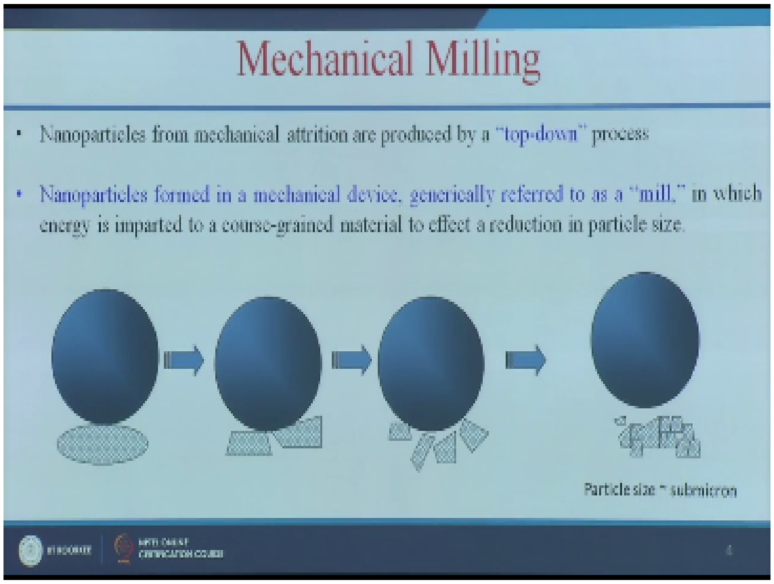

So we will first see what is mechanical milling. So it is a top-down process you know what is top-down process it means you are making from bulk to nanomaterials and here the nanoparticles formed in a mechanical device is generally referred to as a mill. So here we are going to use the metallic ball, so that is going to break your bulk material into nano-size particle.

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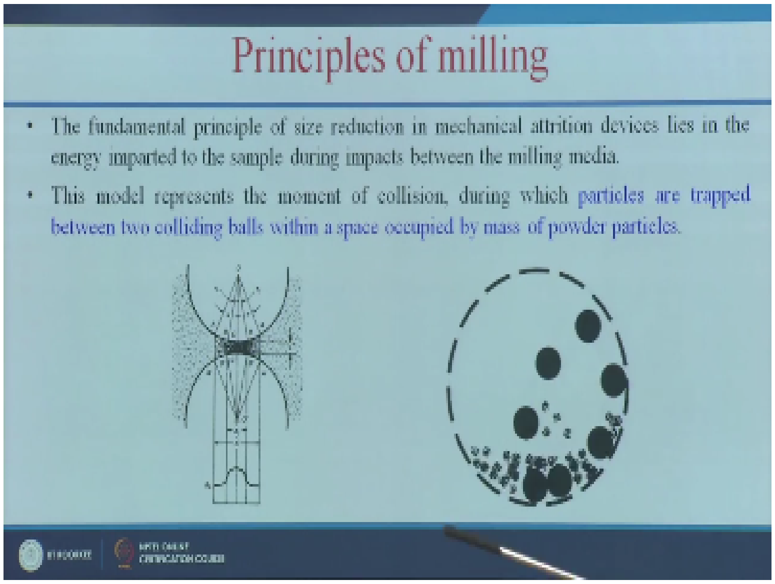

Let us see the principle of milling, so the fundamental principle of size reduction in a mechanical device is the energy imparted to the sample doing impacts between the milling media. So this model represents the moment of collision, here you can see here the particles are trapped between the two colliding balls, within that space occupied by the mass of powder particles. So this animation will tell you how the mechanical milling works.

Now here you can see here the metallic balls are rotating, so the metallic drum is rotating and the metallic balls are falling on the powder, and it is going to break the micro sized powder into nano size particles.

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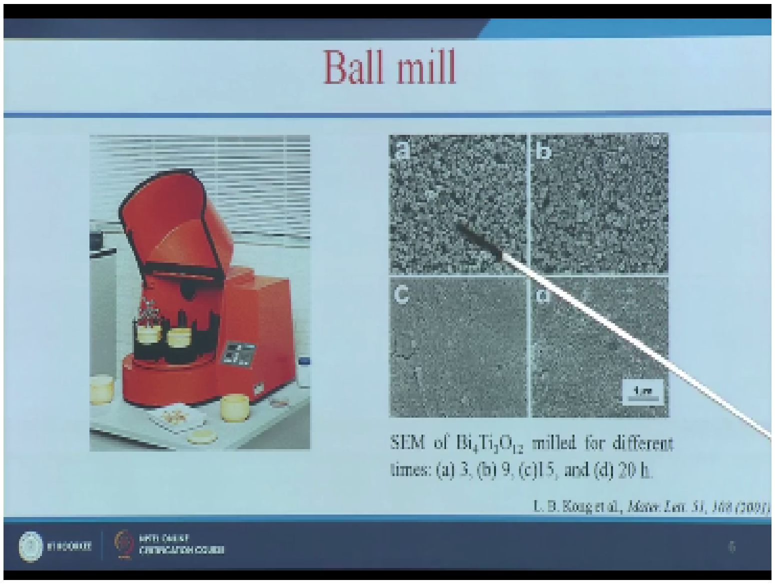

So this is the mechanical milling or ball milling equipments, we can also call it like a ball milling, because we are using metallic balls. And this is a electron microscope picture, so as I told you earlier for seeing these nanoparticles you need a electron microscope. So this is the scanning electron microscope picture, you can see here figure A is after 3 hour of milling, and figure B is after 9 hour of milling, and C is 15 hour and D is 20 hour.

So we can see here with respect to time the particles size is going down, and whenever you take the picture you have to insert the scale bag, so the scale bag is very important which will be useful for calculating the size of the particles.

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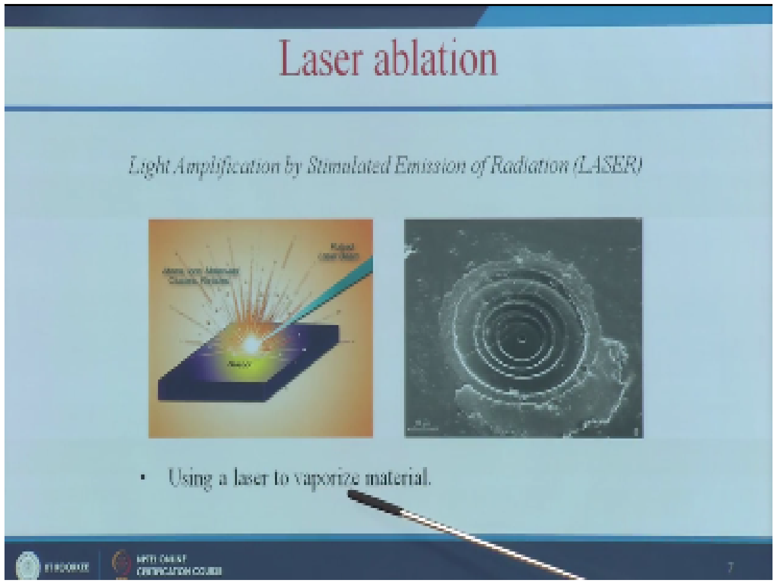

So let us see the next method that is, laser ablation. Here you will be using the laser light to vaporize your material.

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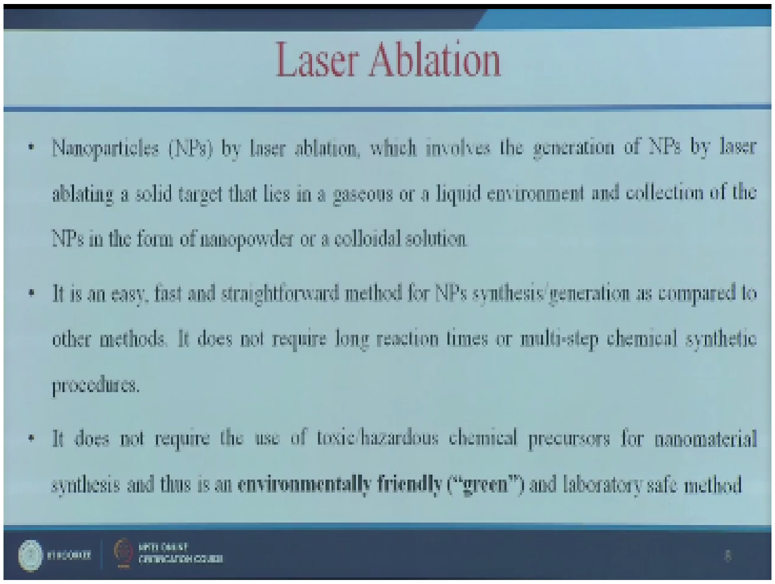

And the nanoparticles by laser ablation which involves the generation of nanoparticles by a laser ablating of a solid target okay. So we can get the nanoparticles in the gaseous or it can be obtained from the liquid environment also. So it depends on the environment, it can get the particles in the nano powdered form, or it can be in a colloidal solution form. And it is one of the easiest and fast method, and here when you compare to the other methods it do not have any long reaction times are it do not have any multiple steps.

And it does not require any toxic chemicals for its synthesis so it is also called as environmentally friendly or green method.

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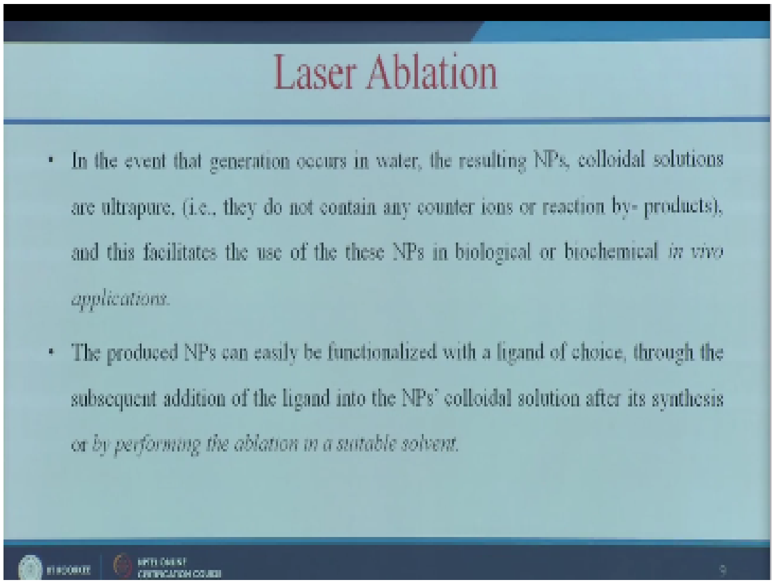

So in this method the resulting nanoparticles which we are obtaining in the colloidal solutions are ultra pure. So these particles do not contain any counter ions or any reaction by products, so we can use that particle as such for the biological applications. And again we can also add ligand to these particles, we can add the ligand after the synthesis process or we can add the ligand in the suitable solvent itself, so it can be coated on the surface of the nanoparticles.

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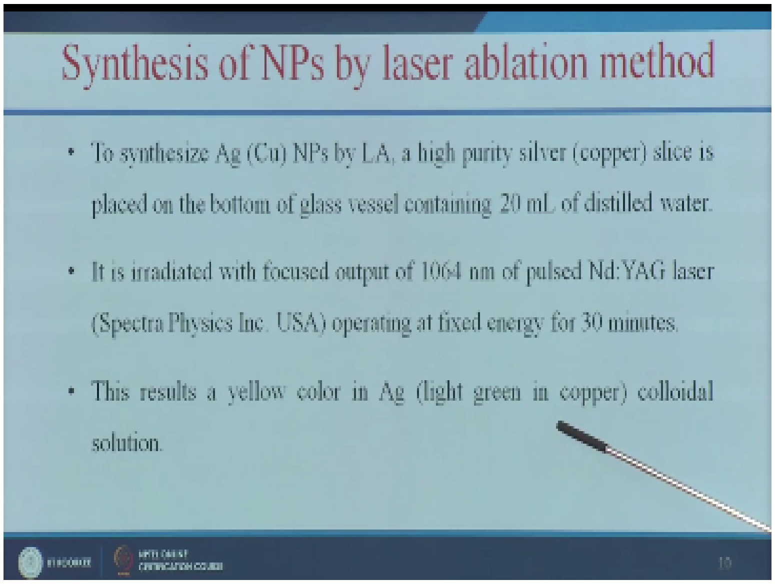

How to synthesis this nanoparticles by laser ablation, if you want to synthesis silver nanoparticles by laser ablation you need a high purity silver slice. And if you want to make the copper nanoparticles you need a high purity copper slice at the bottom of the glass vessel containing 20ml of water. And if you apply the laser light like 1064 nanometer pulsed Nd:YAG laser and operating at fixed energy of 30 minutes.

So this will result in yellow color in case of solver nanoparticles and it will appear light green in the case of copper nanoparticles.

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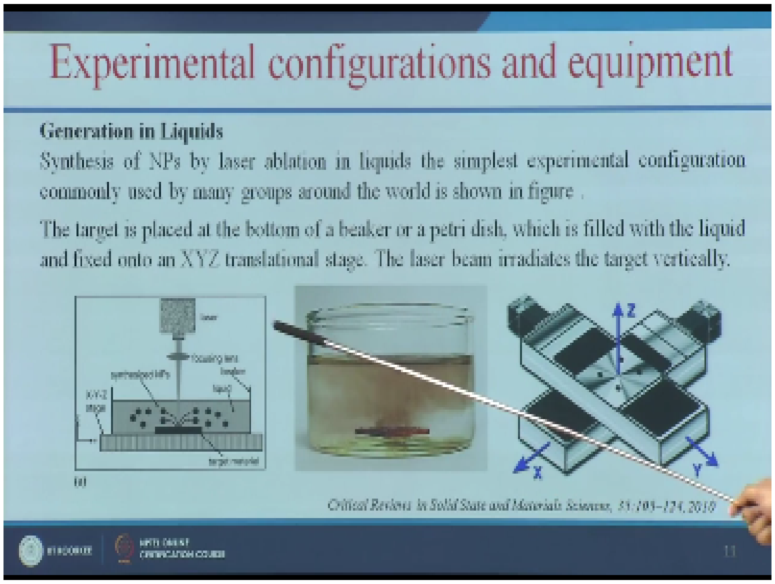

So this is the experimental setup of laser ablation you can see here so the synthesis of nanoparticles by laser ablation liquids it is one of the simplest experimental configuration. And here we are going to keep your metal slice, if you make the silver nanoparticles we are going to give the silver slice, and if you want to make the copper you can keep the copper slice in that here. And when you apply the laser light it will vaporize the material and the nanoparticles can be obtained in the colloidal solution form and here we can use that X Y and Z stage to move the target and you can unitize the complete material for making the nano practices.

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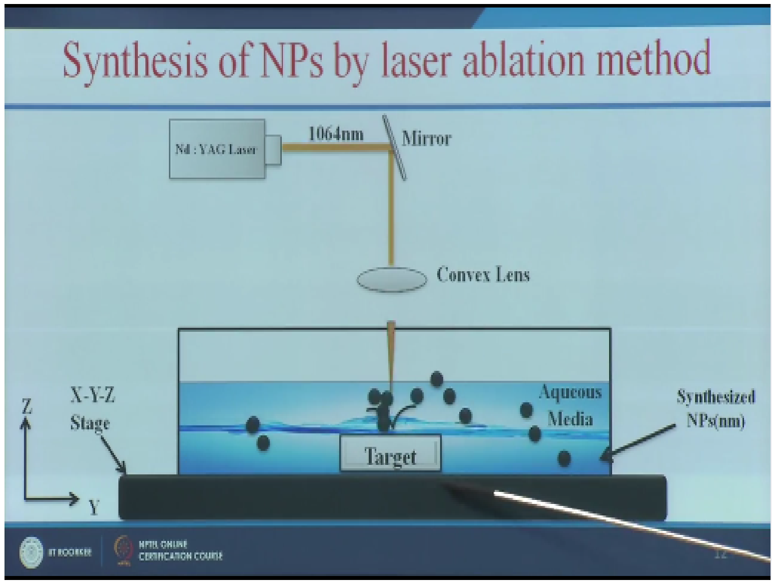

So this summation will give a clear idea how the laser ablation works can see here this YAG laser is applied to the metal target for example if you want to make the silver nano particle you will be keeping this sliver metal here and if you want to make the copper nano particle you will be keeping the copper metal hers when you apply this laser light it will vaporize the metal and this nano particle be collected in the Aqueous media and using this X, Y stage we can move this target to the X, Y and Z direction.

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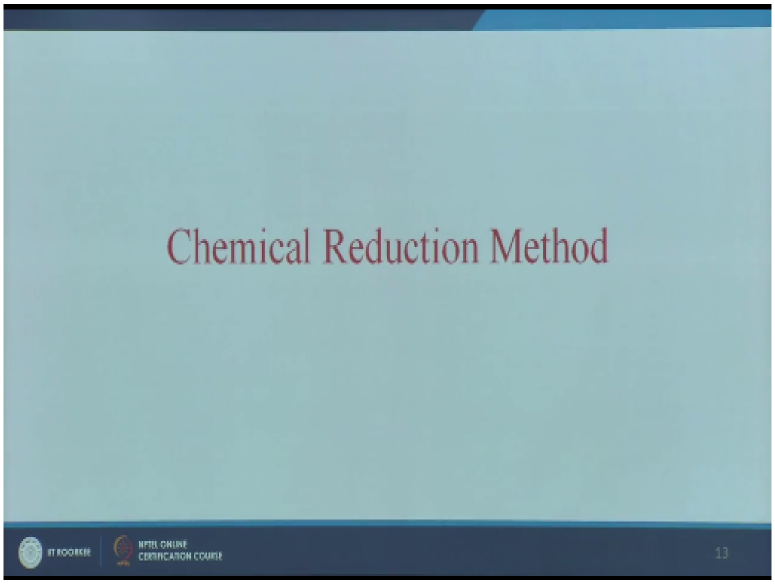

So you understood how we can make nano particle with physical methods using mechanical as well as laser ablation so no we will see how we can make nano particle by chemical method.

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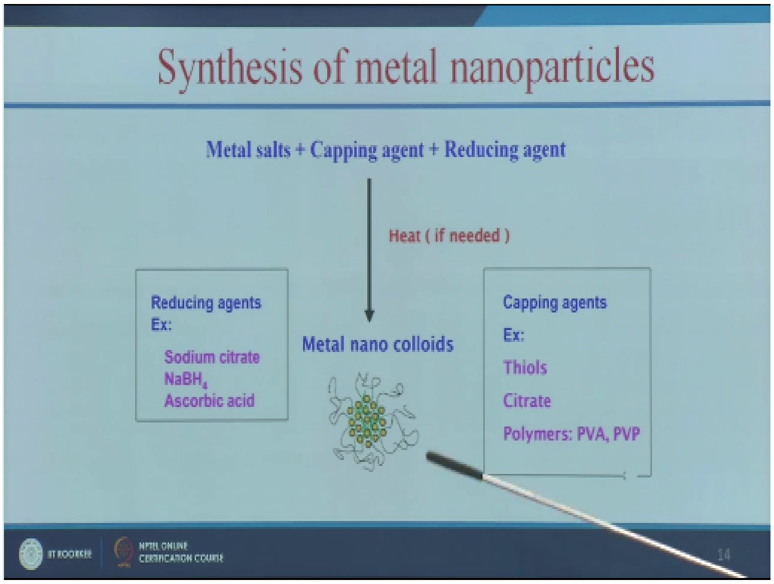

For synthesizing nano practical by chemical method we need 3 important components first one is metal slat and second one is reducing agent and third is capping agent so these are some of the examples for reducing agent sodium citrate sodium boro iodine and ascorbic acid and these are some of the examples of capping agents Thoils Citrate and Polymers like polyvinyl alcohol and PVP okay.

So what happens here is so this reducing agent will reduce your material salt into metal nano particle but this metal nano particles are highly unstable so it will try to join together and form the agglomeration or aggregation so to make the nano practice stable we have to add a capping agent or stabilizing agent that will prevent the aggregation or agglomeration.

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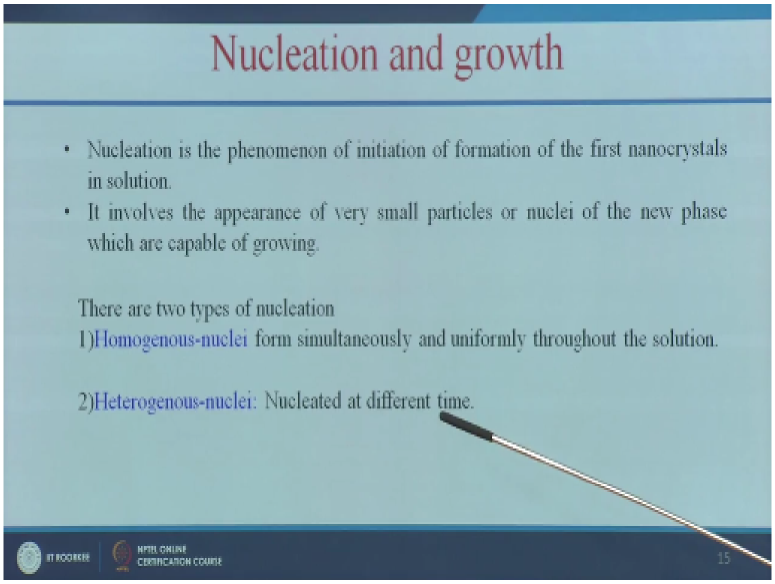

So synthesis of any nano materials involves two important step one is nucleation and other growth, so what is nucleation so nucleation is phenomena of imitation of formation first nano crystals in the solution. So it involves the appearance of very small particles or nuclei of the new phase which are capable of growing so for bio how nucleus is important similarly for nano particle this nuclei is very important there are two type of nucleation first one is homogenous nuclei it form simultaneously uniformly throughout the solution and next one is heterogeneous nuclei so it nucleated at different time point.

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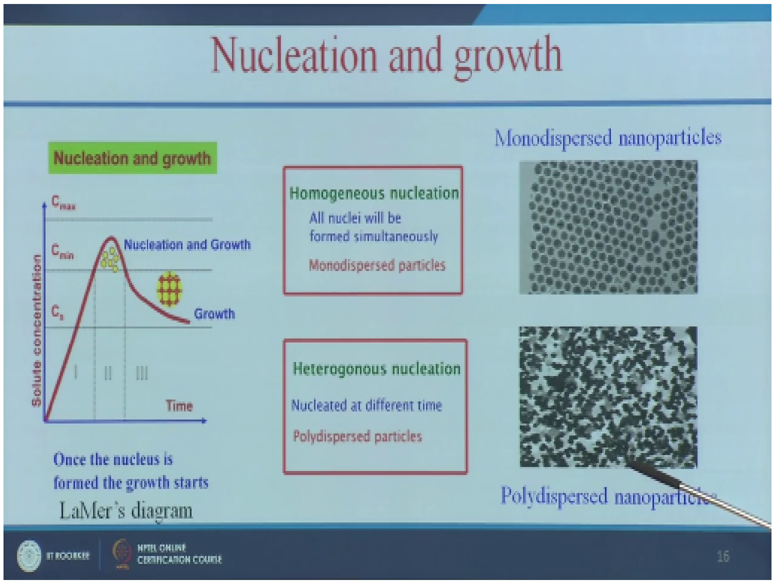

Let us see here this picture is LaMer's diagram adverse time verses solute concentration so once the nucleus is formed then the growth will start so if the nucleation is homogeneous nucleation that means all the nuclei forms simultaneously you will get mono dispersed particles mono dispersed means uniform size particle and if the nucleolus is hetergonous nucleated at different time that is means is poly dispersed practice that means it do not get the uniform size particles so when you see that nano particles under transmission electron microscope you can see here uniform size particles that is called as mono dispersed particles and it is un uniform size that is poly dispersed nano particles.

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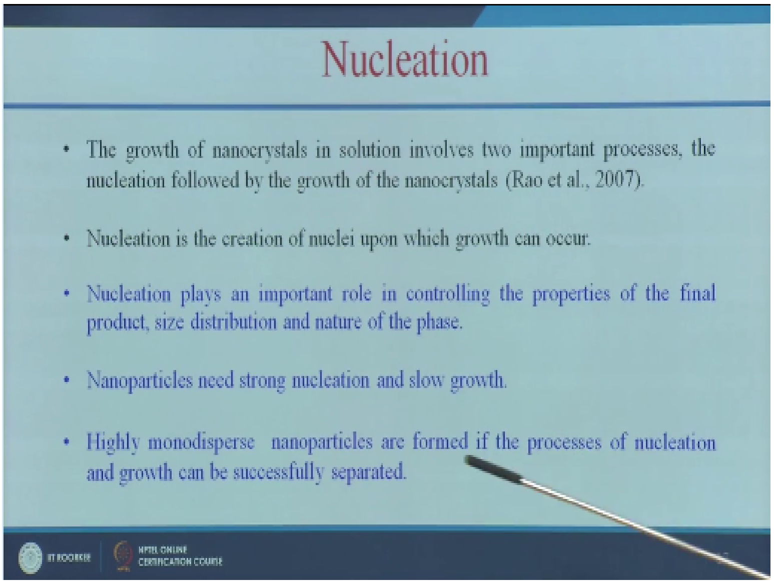

So this nucleation play a very important role in controlling the properties of final product like size distribution as well as the nature of the phase. For making as very small size nano particles you need a strong nucleation and slow growth, so you can get a highly uniformed size particles if the process of nucleation and growth can be successfully separated.

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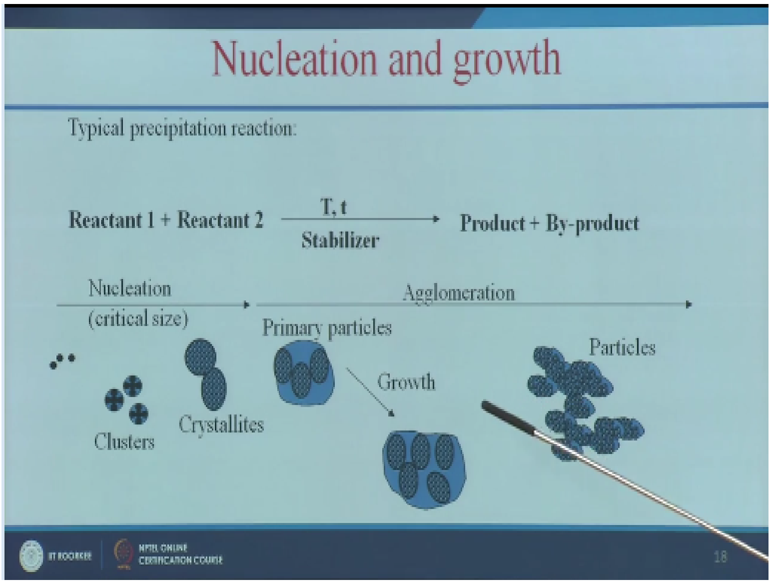

So this is the typical precipitation reaction so here we are going to use the metal slat and reducing agent so this reducing is going to reduce the metal slat into metal atoms this metal atoms companies and form the clusters this clusters come and form the crystal size so this crystallite will compare and form the primary particles this is the nucleation step and this is the growth step so this primary particles combine and start growing so we have to stop the growth otherwise what happen this all the particles combine and grow like a very big size particle that is agglomerated practice which is not useful we had a small particles for our applications.

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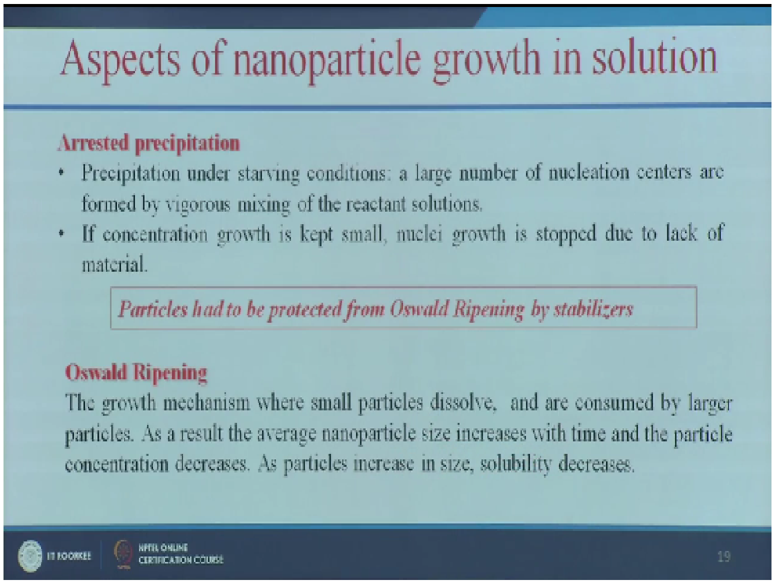

So how we can achieve that by arrested precipitation method what is arrested precipitation here a large number of nucleation centers are formed by vigorous mixing of the reactant solution. So when you make your nano particle we have to mixed vigorously when you mix it vigorously you will have more number of nucleation so this I will show in the last with the a simple experiment how to make this and how to mix it vigorously.

And here if you keep the concentration growth kept small the nuclei growth will be stopped due to lake of material now this called as arrested precipitation and due to stope the particles from the Oswald ripening so how we can stop the Oswald repining by using the stabilizer what is Oswald ripening the growth mechanism where small particles dissolve and are consider by the larger particles what happens is at the result of this average nano particle size get increased with time so and the particle concentration get decreases so if the particle size is increased it decreases at the solubility so this is called Oswald ripening that means it the small particle dissolve and consumed by the larger particles by using this stabilizers agent we can protect it from the also like we can protect the particle from the Oswald ripening.

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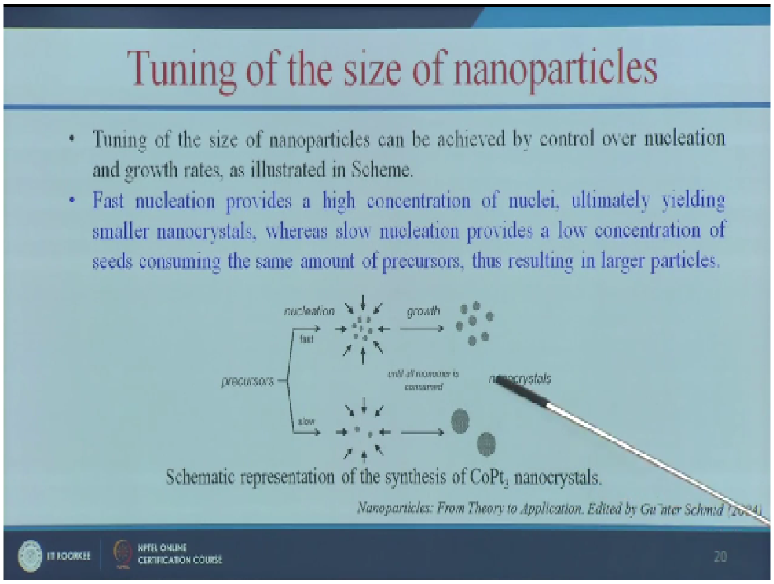

So how we can tune the size of particle the tuning of the size of the particles can be achieved by controlling the nucleation as well as the growth rate for example fast nucleation will provide high concentration of nuclei, and it will yield at smaller nano crystals but when you use this slow nucleation it provides the low concentration of seeds and again the same concentration of precursors so that results in the larger particles the simple example is we have 5% and we have 50 rupees each will get 10 rupees.

But I and if we have that 2 persons each can share like 24 rupees each, so similarly here you can here if we have the more number of nuclei and the precursors amount is same so what happen this will get a small size particle and doing a slow nucleation you are having only two nucleus but again the precursors is same so everything is consumed and the crystals is size is bigger it is going bigger size.

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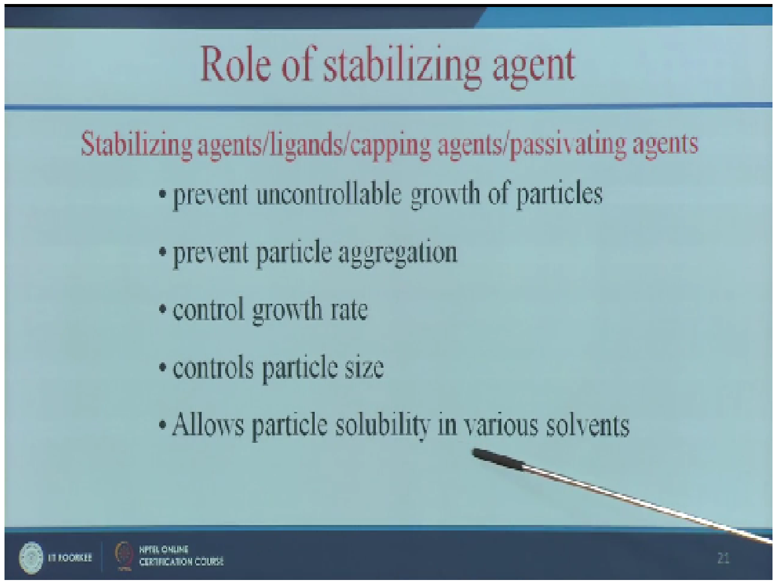

And what is the role of stabilizing agent it is preventing the uncontrollable growth of particles and it will prevent the particle aggregation and it will control the growth rate and also will control the particle size and it will allow the particles solubility in various solvents also.

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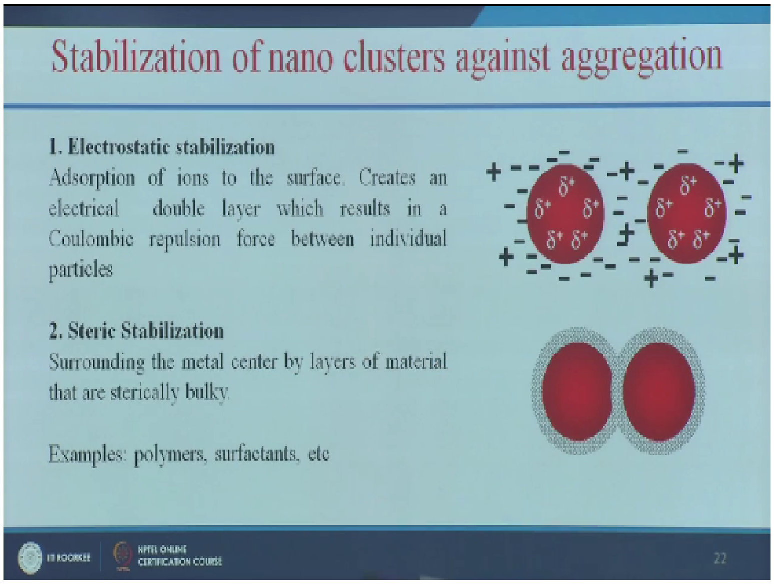

Let us see how the stabilization agent works there are two types of stabilization one is electrostatic stabilization the other one is Steric stabilization so what is electrostatic stabilization it means absorption of irons to the surface so it create an electrical double layer so which results in a Coulombic repulsion force between the individual particles so the uniform particles start it will repulse each other so it will give a electrostatic stabilization the next one is Streic stabilization that means the metal is covered by a layer of material.

That was sterically bulky okay so for example polymers and surfactants to be used so that is example for the Steric stabilization.

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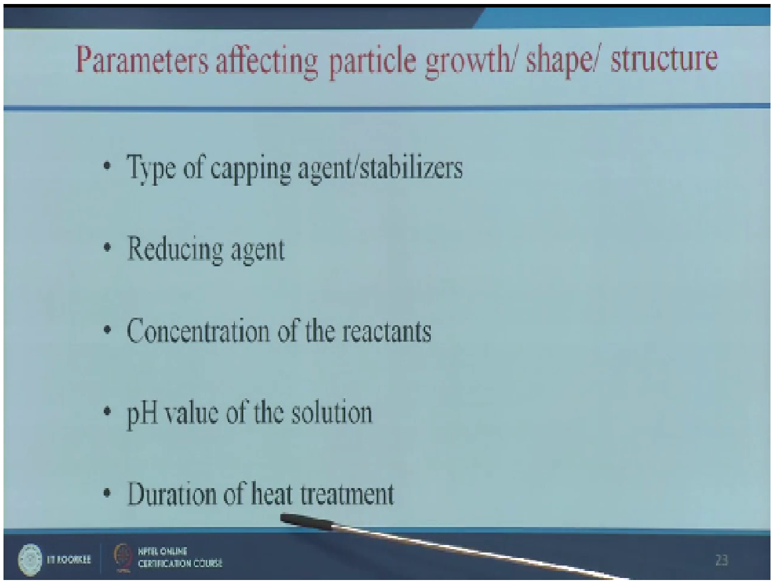

So these are the parameters which is going to affect the particle growth shape and structure so what can have capping agent you are going to use what is the reducing agent whether you going to reduce the strong reducing agent or weak reducing agent and what is the concentration of reactants and again the pH value of solution is important and duration of heat treatment in some cases the heat treatment is going to favor the fast nucleation.

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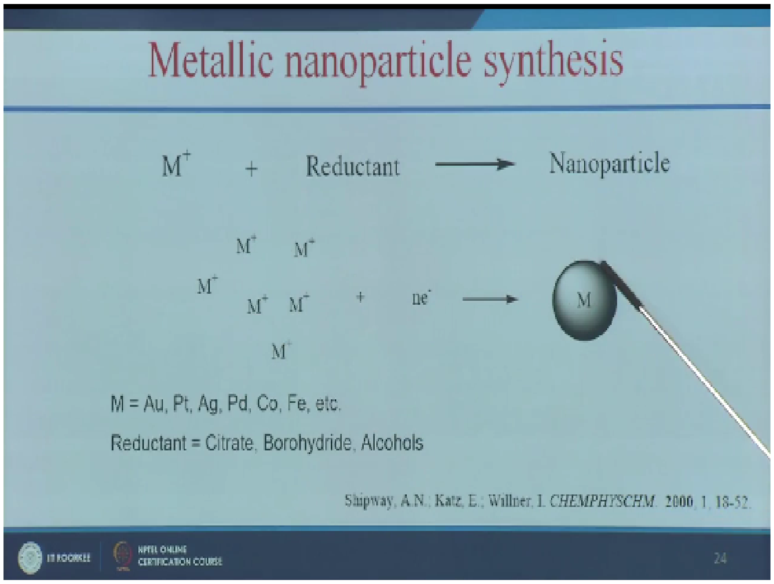

So this is a typical picture of your metal nanoparticle synthesis can see here if you want to make golden particle you can use the gold metal precursor and you going to reduce using reduce agent so that is going to reduce the metal salt into metal nanoparticle.

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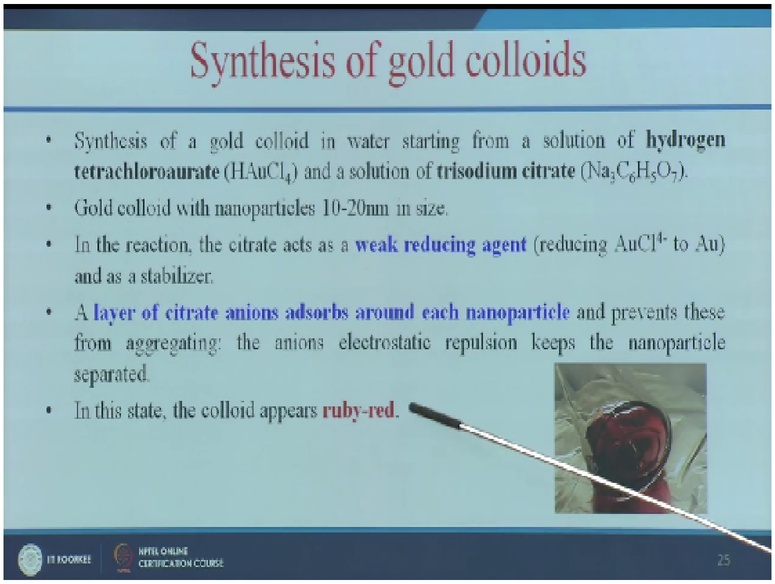

Let us see how we can synthesis gold colloids for synthesis gold colloid in water we have to use this metal precursor hydrogen, tetrachloroaurate and a solution of trisodium citrate it is your reducing agent so here the we can get a gold nanoparticles in the size of 10 to 20nm nanometer and again in this reaction the citrate act like a weak reducing agent and also it act like a stabilizer okay so a layer citrate anions adsorbs around each nanoparticle and it prevent from the aggregating because the citrate irons give the uniform charge to nanoparticles.

By electrostatic repulsion it will be separated out so here the citrate act like a reducing agent as well as it also act like a stabilizing agent so at the finally you will get a ruby-red color when you get the ruby red color you can conform you got the gold nanoparticle.

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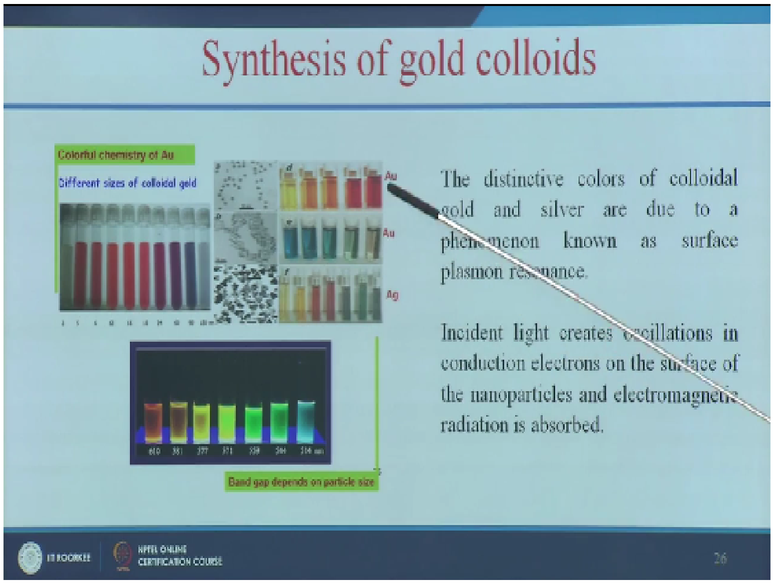

So why this metal nanoparticles showing different kind of colors you can see here different size and different shapes showing different kind of colors these are silver and it is why different kind of colors with respect to size and shape and again this is the goal spheres of different size and this gold nano rods and it is also showing different colors. So what is the reason for these colors the reason is the surface Plasmon resonance the metal nano particle will show the surface Plasmon that means the incident light creates oscillation in the conduction electrons on the surface of the nano particles and the electromagnetic radiation is absorbed.

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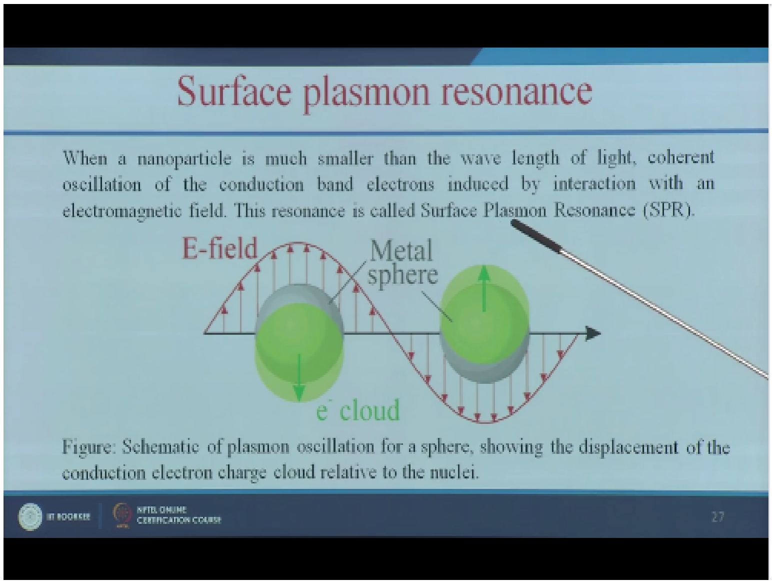

So let us see what is surface Plasmon resonance so when a nano particle is much smaller than the wave length of light, coherent oscillation of the conduction band induced by interaction with an electromagnetic field so this resonance is called surface plasmon resonance.

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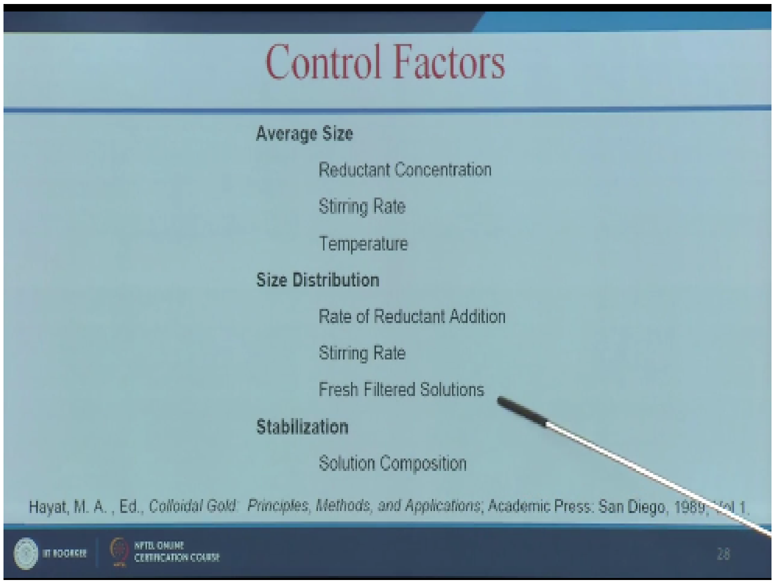

And these are the factors which are going to control the size and the shape of the nano particle. So you can see here the concentration of your reducing agent it is going to control the size of the particles and also the string rate and I again you have to use the fresh solution filter solution will always remained the particles again the stabilizing agent wanted of stabilizing agent you are going to use that is going to decide the size and shape of your Nano particles.

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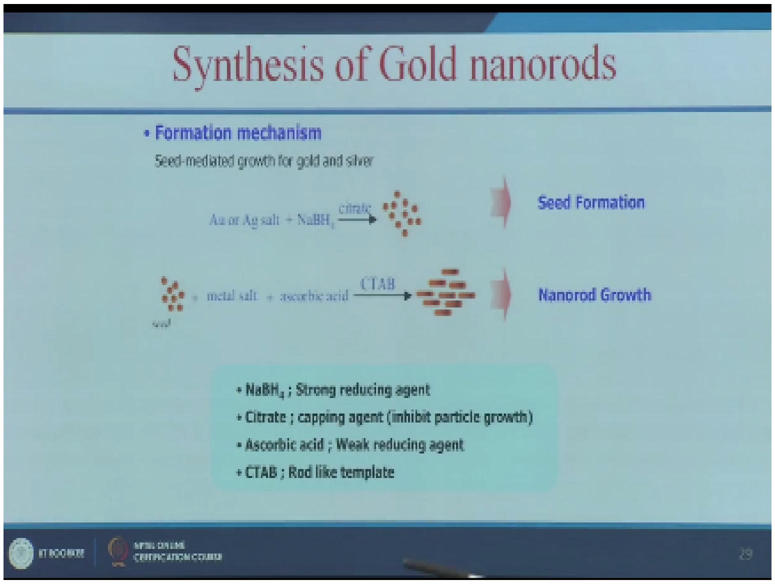

So let us see how to synthesize gold, Nano rods, okay. So it is a two step process like you can see here it is a Seed mediator grows for gold and silver here will be using this gold or silver salt if you want to make a silver Nano rods you have to use a silver salt and if you have to use a if you want to make gold Nano rods you have to use a gold as a metal salt and we are going to use the $NaBH_4$ that is sodium boro hydride.

It is a very good strong reducing agent what happens is, when you use that sodium boro hydride it will reduce its gold into very small size particle and it will get the seed formation okay and here citrate will act like a capping agent, stabilizing agent and it will inhibit the particle growth, so you will get the seed and in next step you will use the seed then you will add the gold or silver salt.

And you will have the weak reducing agent that is ascorbic acid, when you add the weak reducing agent what happens is, here it will favors the growth okay and here you are using a different kind of stabilizer that is CTAB so this CTAB stabilizer it will provide a lot like template so you will get a rod shaped Nano rods you will get in this step okay. So when you use that strong reducing agent you will get the seeds and when you use the weak reducing agent you get the Nano rods okay. And also we have to use the stabilizer this CTAB stabilizer place a major role in formation of Nano rods.

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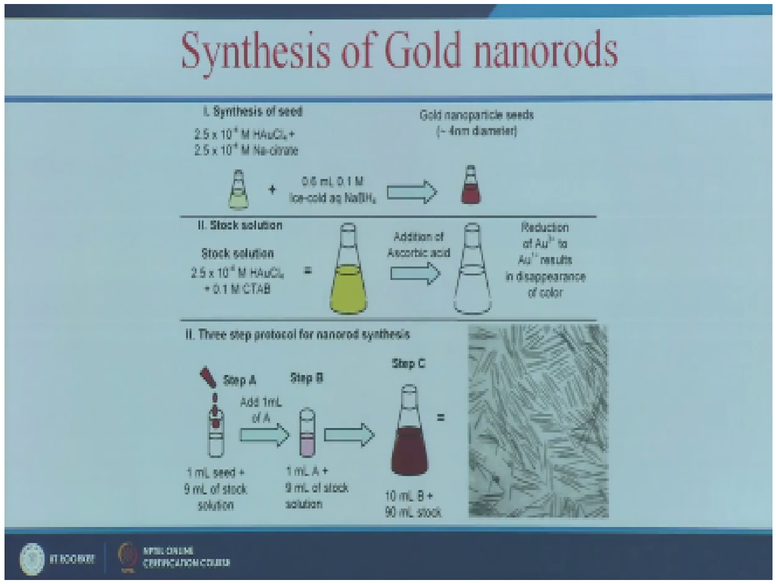

So this is the synthesis of gold Nano rods so here you can see here in the first step they are going to use that gold solution and sodium citrate and you are going to mix with the sodium boro hydride and you will get a Nano particle of four Nano meter, diameter so this is your seed and second step is stock solution here we are going to use the gold solution plus you are adding the CTAB stabilizer and you are going to add the ascorbic acid.

So when you add the ascorbic acid what happens is, it is reducing the gold Au_3^+ 2, Au 1^+ it results in the disappearance of color and it is a 3 step protocol for Nano sensors so first step is like you a 1 ml of seed and mix with 9 ml of your stock solution and from here you take 1 ml and mix 9ml of your stock solution and from here you take the 10ml + 90ml of this circulation finally you will get the Nano rods.

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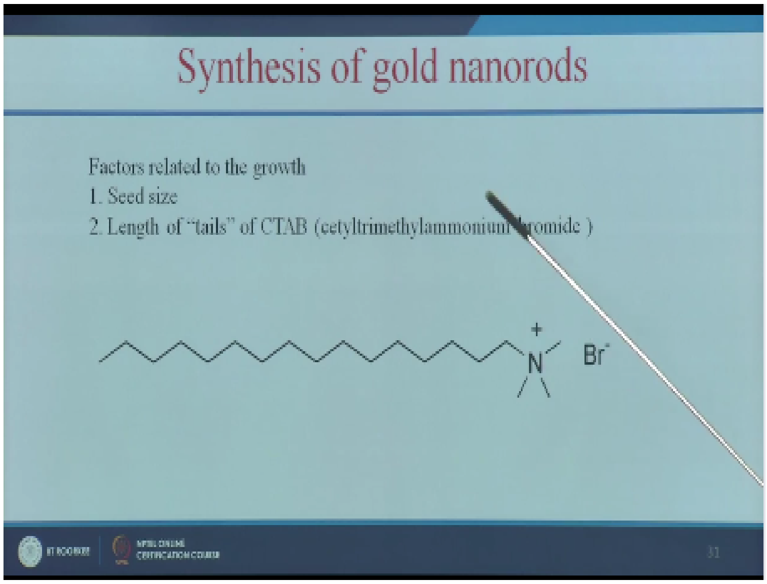

So this Nano rods size synthesis depends on the seed size as well as the length of tiles that is by the CTAB okay so we have the mode CTAB and other things it is going to play a major role in the Nano rod size and dimension.

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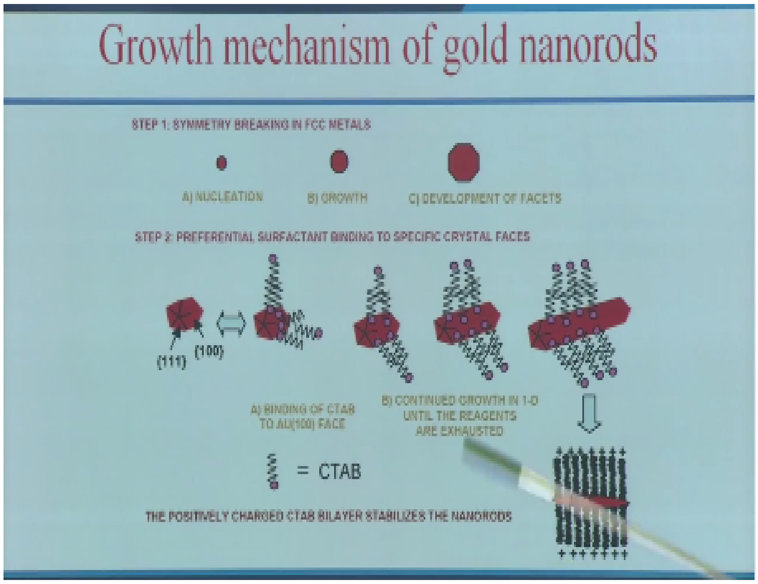

So what is a growth mechanism of gold Nano rods, the first step is nucleation followed by that you will have the growth and followed by that you have the development of phase, so once that faced is form what happens is, this CTAB stabilizer will go and bind to this facer and it will continue to grow in until all the reagents are exist, so this CTAB it is a partially charged by layer and it is a stabilizing Nano rods.

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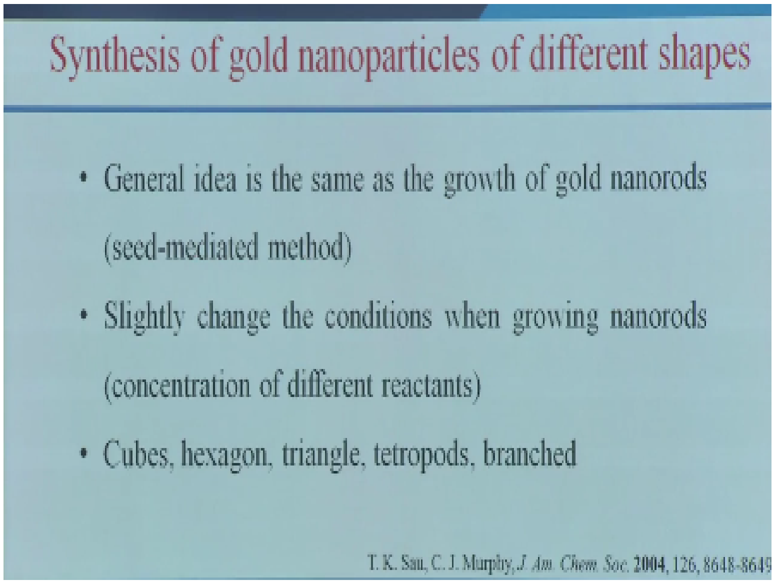

And based on this idea we can make a several size and shape of gold Nano particle only this is you are going to change the concentration of reducing agent and concentration of stabilizing agent by just playing around with this concentration you can get a different kind of shape like cubes, hexagon, triangle and you can have a different kind of shapes.

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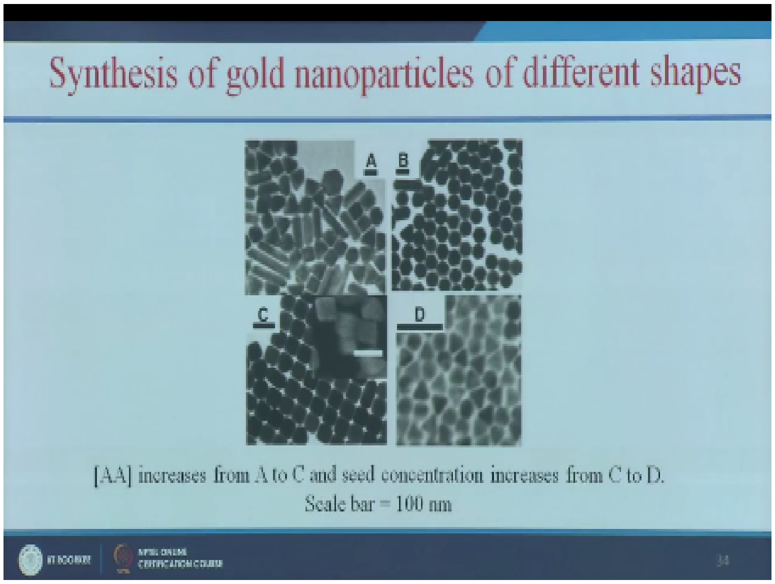

So here you can see a simple example, so we are going to use this ascorbic acid and we are going to increasing the concentration of ascorbic acid from A → C so when you increasing the concentration of A→C and we are increasing the seed concentration from C → D, so when you play around this with the ascorbic acid that is reducing agent as well as the seed concentration we will get a different kind of shapes and different kind of size.

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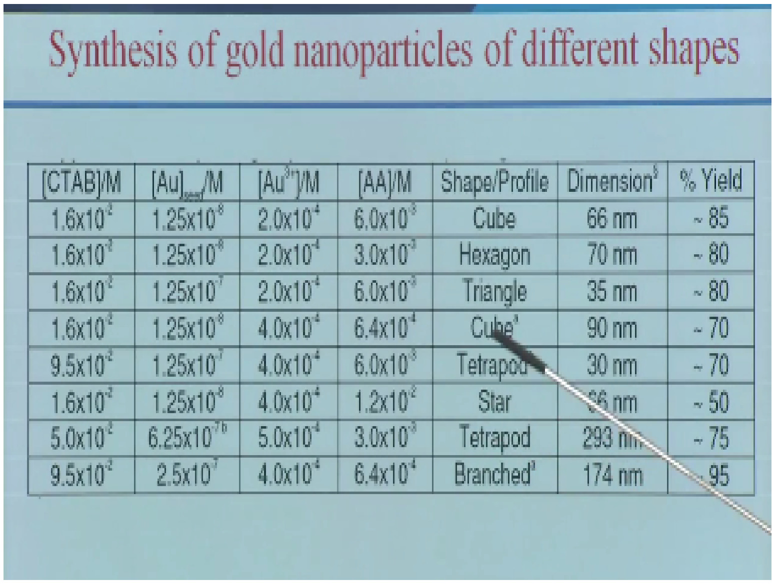

[CTAB]/M	$[Au]_{seed}$/M	$[Au^{3+}]$/M	[AA]/M	Shape/Profile	Dimension[$]	% Yield
1.6×10^{-2}	1.25×10^{-8}	2.0×10^{-4}	6.0×10^{-3}	Cube	66 nm	~ 85
1.6×10^{-2}	1.25×10^{-8}	2.0×10^{-4}	3.0×10^{-3}	Hexagon	70 nm	~ 80
1.6×10^{-2}	1.25×10^{-7}	2.0×10^{-4}	6.0×10^{-3}	Triangle	35 nm	~ 80
1.6×10^{-2}	1.25×10^{-8}	4.0×10^{-4}	6.4×10^{-4}	Cube[a]	90 nm	~ 70
9.5×10^{-2}	1.25×10^{-7}	4.0×10^{-4}	6.0×10^{-3}	Tetrapod	30 nm	~ 70
1.6×10^{-2}	1.25×10^{-8}	4.0×10^{-4}	1.2×10^{-2}	Star	66 nm	~ 50
5.0×10^{-2}	$6.25\times10^{-7 b}$	5.0×10^{-4}	3.0×10^{-3}	Tetrapod	293 nm	~ 75
9.5×10^{-2}	2.5×10^{-7}	4.0×10^{-4}	6.4×10^{-4}	Branched[a]	174 nm	~ 95

So this tabular column will give to a clear idea so if you use this concentration of CTAB and if you use this concentration of AU seed and this concentration of Au_3^+ and this concentration of ascorbic acid you will get this kind of different shape so we are going to use this four components and we are going to achieve different kinds of shapes and different size only by changing the concentration of this four components we are achieving different size and different shape.

So if I ask you to select a Nano particle based on size for a catalytic reaction which one you will select, so we will select yes you are right we will select the smallest size so if I ask you to select nano particle from this tabular column for catalytic activities based on the size we have to select the smallest size because the smallest nano particle have highest surface areas to volume issue it will have more catalytic activity.

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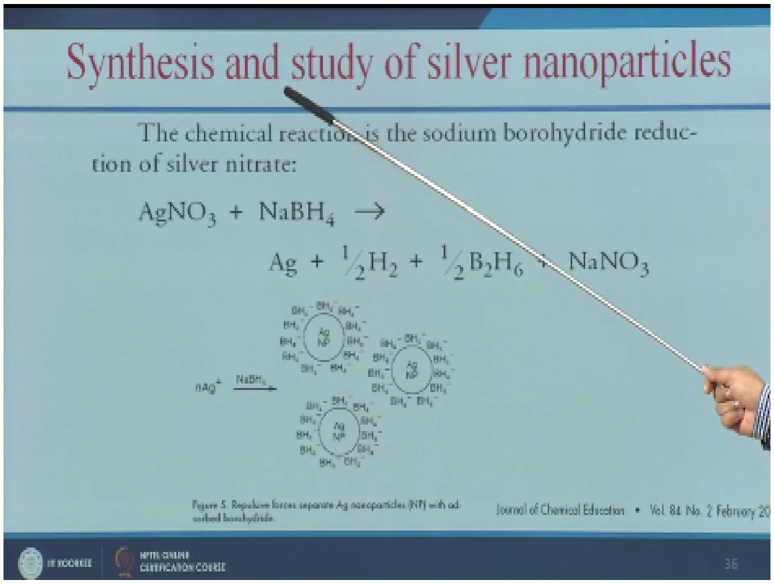

So let us see how to synthesis silver nano particles okay, so here we can use this silver nitrate $AgNO_3$ and your sodium borohydride this is your reducing agent and this reducing agent will act like a also stabilizer can you see here so from Ag^+ it became Ag0 and this borohydride protecting this nano particles from aggregation or agglomeration.

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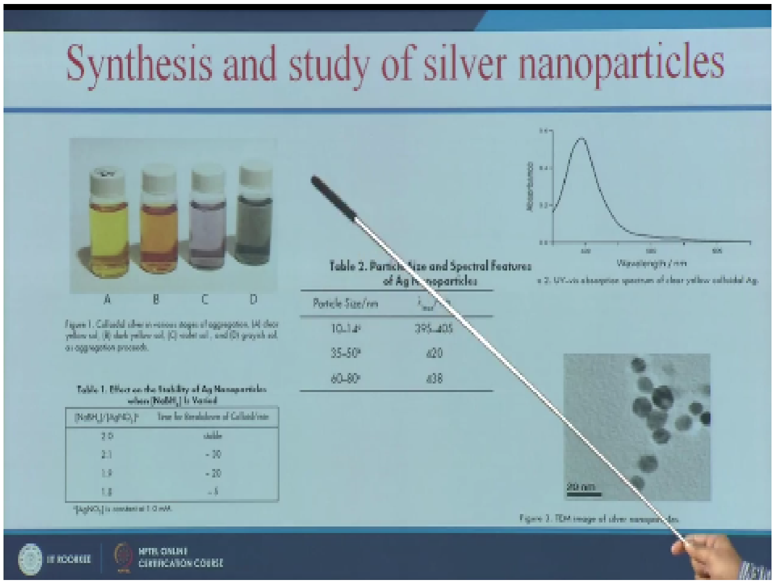

So in silver nano particle you will get yellow color we can see here these all silver nano particle but these are of different size okay, so you can see here the silver colloids of if you see this is a, if we have a yellow color pale yellow that is like we have a very small size particle and if we have this kind of color that means the particles are aggregated it is not useful for the further applications.

And how do you control and how do you stabilize this nano particle you can see here the ratio between your metal salt and reducing agent is very, very important if the ratio is 2 it is highly stable, if the ratio is 2.1 it is stable for 30 minutes and if the ratio is 1.9 it is stable for only 20 minutes and if the ratio is 1.8 it is stable only for 5 minutes okay. so when you see optimize any synthesis protocol you have to think and you have to optimize, okay so this is very, very important first making a very good stable nano particles.

So when you made the nano particle the first step of your characterization is you will be using this UV visible spectroscopy, I guess most of them know what is UV visible spectroscopy and most of the lab you will be having this UV visible spectroscopy so what you have to do is, you have to transfer this 1ml of solution to a quart skewed okay, so you take it to that quart and you will do the scanning of this solution.

So when you start the scanning from 200 to 800 so you will get the λ maths at 400 wave length usually the silver nano particles gives the λ maths at 400 nano meter range okay, so if we get this kind of peak you are sure that you got silver nano particle so from the silver nano particle peak you can approximately calculate the size. For example, if you are getting the λ maths at 395 the particle says approximately 10 to 14 and if we get the peak at 420 the size of the particle is 35 to 50 and if we get the peak at 438 the size of the particle is 60 to 80.

And the next step is you have to take it for the transmission atom microscope you want to see the particle okay, so when you see under this electron microscope you can see the particles size and as I told you earlier when you take any microscope picture we have to include this scale bar. So here you can see this scale bar is 20 nano meter so assume this scale bar is approximately 1cm okay, and when you use this scale bar so when you use this scale bar and it move it to this particle so you can see here this is like 0.5cm.

Though if it is 0.5cm we can calculate it is like 10 nano meter particle, so based on this scale bar we get easily estimate and calculate the particle size. So let us see the nano rod formation so when you use this silver salt and if you make the nano rods what happens is like it will give two peaks.

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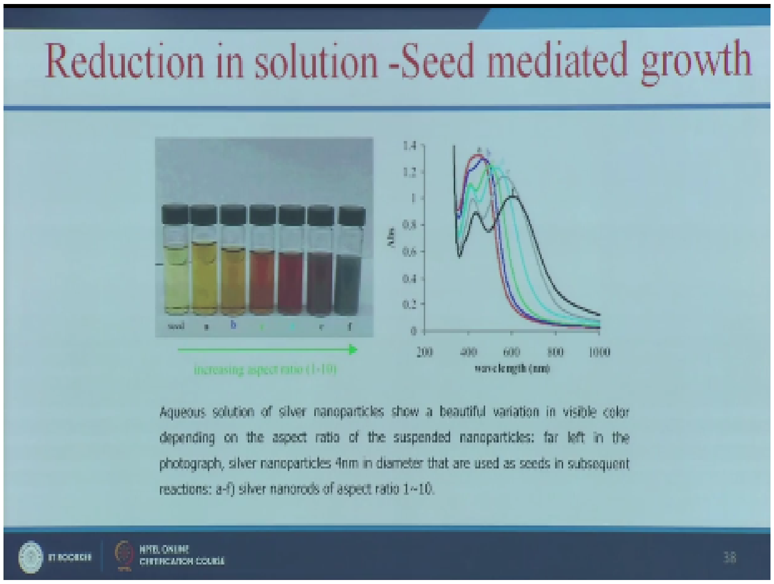

When you see the nano rods under UV visible spectroscopy it will give two peaks and if you see this is red color one is the seed okay, and remaining all are nano rods of different size nano rods so why it is giving two peaks what is the reason.

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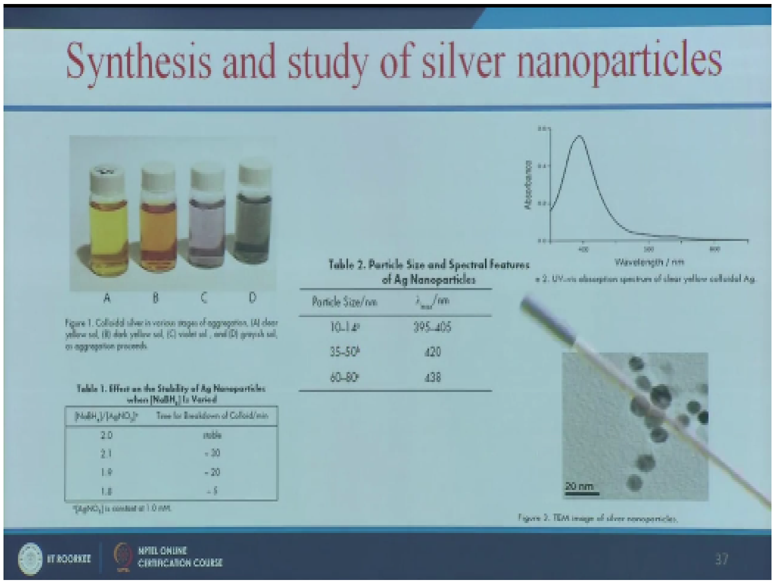

You can see here the nano particle is giving a single peat but the nano rod is given two picks so what is the reason the nano rod is giving two peaks because it has two dimension so it has dimension like this as well as like this like longitudinal and transfers so it will give a two picks transfers as well as a longitudinal peaks okay. So let us see a simple experiment I will demonstrate you how to make nano particles in the lab with the simple chemicals available in your lab oaky.

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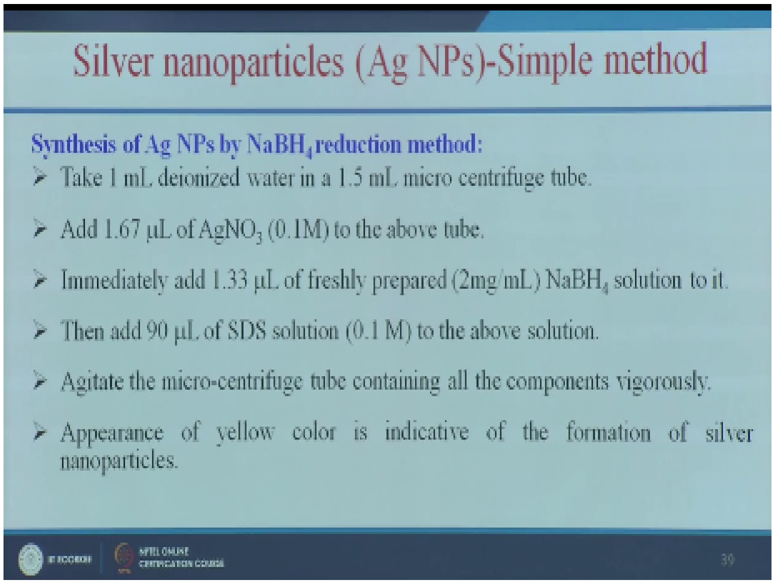

So for this we have to use 1ml of deionized water in a 1.5 ml micro centrifuge tube and we will be adding 1.67 micro drops silver nitro solution that is a 0.1m to the above tube and we sill immediately add 1.33 m drops for freshly prepared 3mg /ml $NaBH_4$ solution ton it okay. So the sodium boride should be freshly prepared solution and then you add 90m drop at this solution to this above solution it is the stabilizer it is going to stabilize your silver nano particle.

And you have to mix it vigorously then if you see the yellow color that means you are show that the silver nano particle is form. So let us see the experiment I will demonstrate you. So now we are going to see how to make silver nano particle by using a simple experiment with the available chemicals in the lab.

The first step is we have to take 1ml of deionized water in a 1.5 ml tube this is a 1.5mlmicro tube so here I am going to take 1mlof deionized water, so I am taking 1mlof deionized water in two tubes here to one tube I am going to add a reducing agent and a other tube I am not going to add reducing agent so that you will get a idea the tube which I add a reducing agent in to reduce a silver slat in to silver nano particle the other tube which will do not have the reducing agent it will be transparent.

It do not form any nano particles and from this I am going to remove 93 micro drops water because in this sub sequent step we are going to add 90 micro drops SDS and 1.67 of silver night rate solution and 1.33 of sodium boride solution, so the next step is I am going to add 1.67 micro drops silver night rate solution so this silver night rate solution is light sensitive so we have to use cover with aluminum soil and always were glues and also lab coat when you make silver nano particles.

So I am adding this silver night rate solution to both the tubes so now I am going to add 1ml of water to the sodium boride as I told you earlier the sodium boride should be prepared freshly so what you did is we already measured 2mg per ml that is we are measured 2mlin a 1.5 ml tube to this I am going to add 1ml of water and I am going to make sodium borohydride freshly.

Just mix it so that the sodium borohydride can dissolve completely and immediately you have to add the freshly pre pared sodium borohydride 1.33micro ton to this tube, so I am going to add 1.33 drops sodium boro hydride to only to one of the tube not to both the tubes. Now I am adding immediately the SDS 90ml drops SDS to both the tubes.

So I am going to mix it vigorously to both the tubes, so now you can observe the yellow color formation that means the nano particle is formed. So you can see here the tube which I added reducing agent it reduces the metal salt into sinner particle. You can see here it has become yellow color the other tube that I did not add the reducing agent it is transparent.

That means the sinner particle formed in this solution where I added the reducing agent the other tube that there is no reducing agent there is no formation of sinner particle. Hope you understood the experiments how we can make nano particles in the lab. The summary of this lecture so in this lecture we have learnt what mechanical milling is and what is laser absorption.

So these are two physical methods and these physical methods how to make nano particles and you learnt mechanical milling is the top on approach okay again the laser absorption is a green method and eco friendly method because they are not using the foxy chemicals and we also learnt how to make nano particle by chemical reaction method.

For chemical reaction method you need three important components and what are the three important components you need metal salt, reducing agent as well as canting agent or instant agent and we also learned nucleation and what is growth. So nucleation is formation of nano solution formed by the growth.

And we also learnt how to make nano particles and metal rods and what is the mechanism of nano dot formation and a simple experiment how to make nano particles in lab with the available chemicals okay. I will end my lecture here thank you all for listening I will see you in another interesting lecture.

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