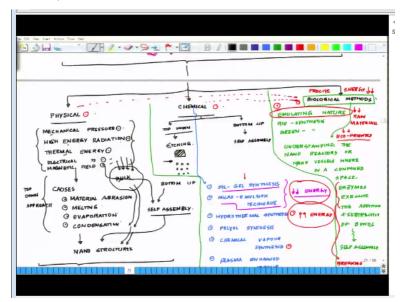
#### Nanotechnology in Agriculture Prof. Mainak Das Biological Sciences and Bioengineering and Design Programme Indian Institute of Technology–Kanpur

## Lecture-13 Detailed Chemistry Techniques

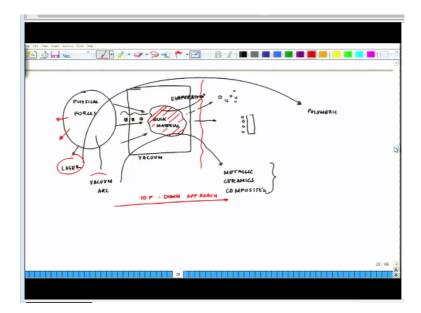
Welcome back to the lecture series and application of a nanotechnology in agriculture. So at this stage of the course we are talking about the basics of nanotechnology and in that context we have talked about the classification of nanomaterial based on their size, geometry, shape and based on their origin or chemical origin, post that we have talked about the different methods of synthesis broadly the outline of physical methods, chemical methods and the biological methods. And within that we have talked little bit in depth about the physical methods.

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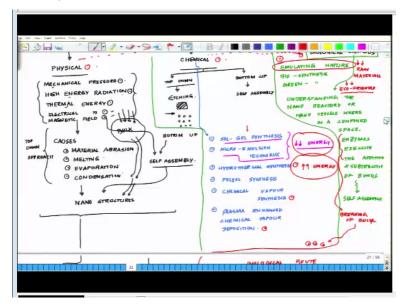
So, if you recollect this is what we have already talked about the physical, chemical and biological methods. And within that we talked about the 5 different techniques high energy ball milling, pulse vapor deposition, laser pyrolysis, flash spray pyrolysis, electro-spraying, melt mixing and polymer composite development. And basic idea what we have discussed in the physical methods of synthesis is that here you having a bulk material which I am now shading in red.

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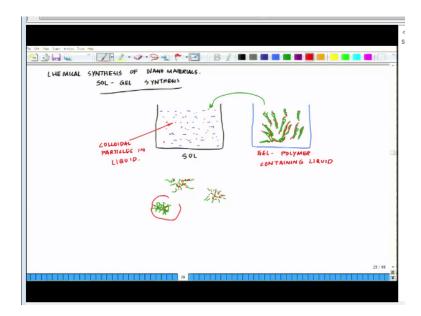
This bulk material is exposed to different physical forces like lasers, mechanical forces, heat, light and all sorts of different forces very high temperature. And inside a vacuum chamber the material gets abraded, the abrasion takes place and that leads to either the deposition or formation of a smaller size particles. And this is essentially a top down approach, so now our goal will be to explore the second level of synthesis which is the chemical level of synthesis.

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So, here we will be discussing about SOL-gel, micro emulsion technique, hydrothermal synthesis, polyol synthesis, chemical vapor synthesis and plasma enhanced chemical vapor synthesis.

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So, let us start with a SOL-gel synthesis which is pretty straight forward technique SOL gel synthesis under the umbrella of chemical synthesis of nanomaterials. So, in the SOL-gel synthesis what we do there are 2 types of components a SOL which is a colloidal suspension of solid particle in a liquid. So basically you have a SOL here which is you are having this colloidal particle all over the place in a liquid suspension okay.

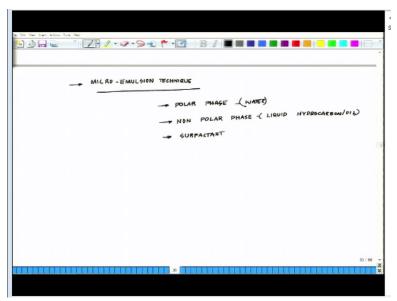
And what you are having is a gel which is a polymer containing liquid. So you have, so this is your gel which is polymer containing liquid. And you are having colloidal particles in liquid, so thus this process includes the creation of salts in a liquid that led to the formation of network of discrete particles or network polymer by the connection of SOL particles. So essentially what you are doing is that actually I pick up a different color out here, let me kind of modify this colors that will these are the polymer particles shown in green.

So the idea is for mixing them what you are getting is these are the polymer particles. So, polymer particles are getting partly aggregated and you have different techniques to separate them out by sonication or other techniques and on top of that the colloidal salts are getting adhered something like this. These are the polymeric matrix and within the polymeric matrix you are having the nanoparticles which are shown in red okay.

So, in other word this process include creation of SOL in the liquid that lead to the formation of network of discrete particles. So this is the network of discrete particles or network polymer by the connection of SOL particle, hydrolysis and condensation at typical steps of SOL-gel process in which the formal users water to disintegrate the bonds of the precursor that is also the first step in the formation of the gel phase.

This process is then followed by condensation that leads to the formation of nanomaterials after which excess water is removed to determine the final structure of the material. So basically condensation evaporation basic techniques hydrolysis and condensation and evaporation. These are the typical processes which are being used in the SOL-gel synthesis. So this is the basic SOLgel synthesis what you needed to understand.

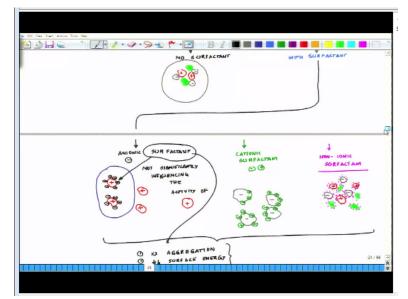
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So, the next technique from here we will be moving is and this section is micro-emulsion technique okay. So, micro-emulsion can be defined as thermally stable macroscopically homogeneous optically transparent and isotopic dispersions constituting minimum of 3 components okay. So what are the 3 component micro-emulsion techniques have, so it has a polar phase which is generally water, polar phase is mostly water.

And even non-polar phase generally hydrocarbon liquid or oil you have a non-polar phase which is mostly liquid hydrocarbon or oil okay. And you have a surfactants, the third thing is a surfactants and we have already discussed about surfactants in the physical synthesis if you just recollect where we talked about surfactants.

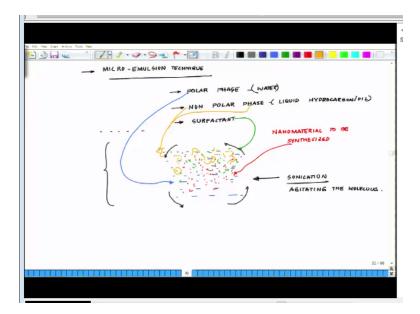
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Here we talked about with surfactants, so these are the surfactants it could be an anionic surfactants, it could be a cationic surfactants, it could be a non-ionic surfactants which are kind of helping in not promoting aggregation reducing the surface energy and reducing the size. There are 3 things what a surfactants does and here also the same principles of surfactants will apply the surfactant which will ensure that.

So say for example you have no you have to understand micro emulsion is something like this say for example you have this polar phase which is water.

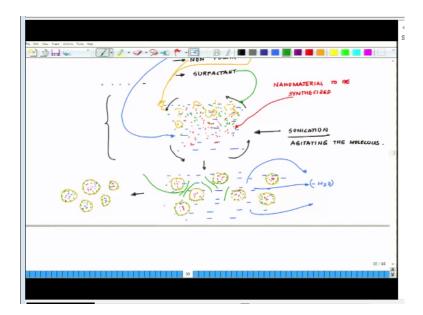
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And in that you have oil phase which I am showing in which is immiscible in water. This is the non-polar phase, this is the polar phase which is water. Then you have a surfactants which I am representing with say green color which is this. And now you wanted to synthesis a particle say for example some particular particle you wanted to synthesis. And that material is in colloidal suspension like out here which is red.

So, you wanted to create a nanostructure of these ones which is nanomaterial to be synthesized okay. Now say for example you take this whole mix and you sonicated or you give some kind of forces by which they are it will create an agitation out here okay, some form of an agitation which is happening either by you know say sonication or shaking or somewhere other, you creating an agitating the molecules. So, what will happen is these non-polar phase these ones, they will try to self assemble, so something like this will rise out of it.

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These non-polar moieties within the liquid, so this is the polar phase which is water within water these non-polar moieties will form some kind of a spherical in order to reduce their surface energy. And within that you have this surfactants which will ensure they remain separated from each other and since during agitation randomly some of the particles will get trap inside. So in other word each one of this is sphere what you are observing out here is acting as a nano reactant nano vessel.

Now at this is stage if you evaporate the water out or lyophilize or do something to get rid of the polar phase. So what you are left with is this, so your water is gone, water removed, what you are left with is something like this. And you can control the size by what kind of force, what kind of temperature you are allowing this reaction to occur and some of the polar moieties may get trap or may not get trap depending on what level of evaporation or lyophilization you are doing.

So, this is the second technique which is a fairly low temperature technique mostly and pretty commonly used and pretty popular among the people who work in the area. So, surfactant molecule thus creates the interfacial layer separating the aqueous and the organic phases, reducing the interfacial tension. So this is what the surfactants is doing, so out here it is reducing the interfacial tension okay creates the interfacial layer separating the aqueous and the organic phase phase reduce the interfacial tension between the micro-emulsions.

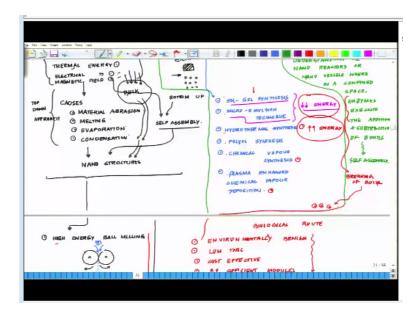
And the excess phase act as a steric barrier preventing the collisions of the droplets. So, it also prevents this droplets you know to collides together, micro-emulsion system consist of monodisperse is spherical droplets of the diameter ranging from 800 to 8000 nanometer of oil in water or oil or water in oil depending on the surfactant use. And with water in oil reverse micellar system act as an excellent reaction type for nanomaterials synthesis.

And reverse micellar is water in oil micro-emulsion where polar head groups of the surfactants creating the aqueous score and resides towards inside where as the organic tail of the surfactant molecule directed outside okay. So, you could have a reverse micell, you could have normal depending on a whether it is it water in oil or oil in water okay. In general there are 2 micro-emulsion roots to synthesis nano material synthesis.

One is micro-emulsion method and two is basically water in oil and or oil in water okay as I have mention earlier. These are some of the very I should say non-energy intensive processes if you compare a micro-emulsion with respect to any of the physical methods what we have already discuss earlier. So, you can understand that these kind of techniques does not require heavy machinery or high energy.

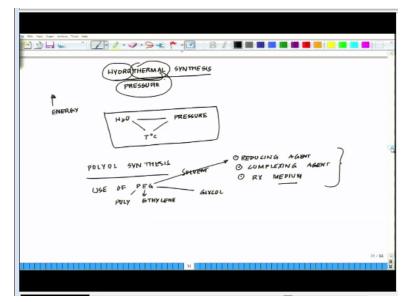
And yet they are very user friendly techniques for most of the labs who work on nanomaterials okay, from here we move onto the next synthesis which is our we are moving onto the hydrothermal synthesis.

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So, hydrothermal synthesis if we look at the methods used for the fabricate of nanomaterial metal oxides mostly it is used for okay.

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Now we are into hydrothermal synthesis, so hydrothermal synthesis used fabricate nanomaterial metal oxide, iron oxide or lithium iron phosphate keeping control over the characteristics of the particle by varying the properties of near or super critical water by using different pressure and temperature condition as its says. So they are is this parameter pressure, so depending on what kind of pressure you are giving an what kind of temperature thermal and pressure.

These 2 parameters in the presence of how much water you are keeping into the system near or super critical water by using different pressure and temperature. So 3 parameters water, pressure and temperature, this is the interaction of these 3 which dictates the hydrothermal synthesis. But this is again a high energy system, hydrothermal synthesis requires pretty high energy okay. So, it can be perform in 2 types of system.

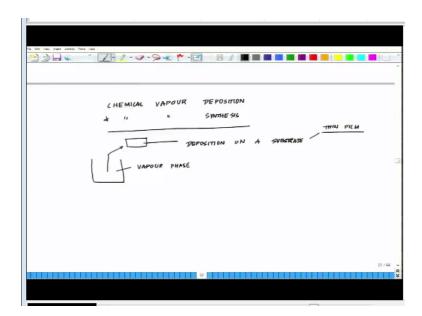
A batch hydrothermal or continuous hydrothermal process the formal is able to carry out a system with desired ratio phases while the latter allows a higher rate of reaction to be achieved at a shorter period of time. So this is another technique which is pretty commonly used in different polymers and different metal oxides okay. Next we will move on to polyol synthesis. So what is polyol synthesis, polyol synthesis is basically the synthesis of metal containing compounds using poly ethylene glycol PEG, use of poly ethylene glycol.

So use of PEG as a reaction medium that play a role of solvent reducing agent complexing agent at the same time. So the role of PEG is as reducing agent, complexing agent, at the same time it is acting as a reaction medium okay and plays as a basic solvent. So it has multiple role to play in the polyol synthesis. This chemical process was used to synthesis wide range of metal based nanomaterials of like silver, platinum, palladium, copper several metal oxide like zinc oxide indium tin oxide, gadolinium oxide different magnetic nanomaterials so and so forth.

So, as we will move through you realize poly ethylene glycol is used for several delivery systems apart from being used for different kind of metal or metal oxides metal oxide system. So, we will later into that one through will be using, so here you should have an idea that what we synthesis are this terminologies really mean to you. But when we will come to the real synthesis or real application we will talk about for these particles.

We can use these kind of synthesis for this particular particle or this kind of a nanomaterial, we can use this kind of synthesis okay. So now from polyol synthesis we move onto the next one which is chemical vapor deposition or chemical vapor synthesis which is again on the same line as physical synthesis.

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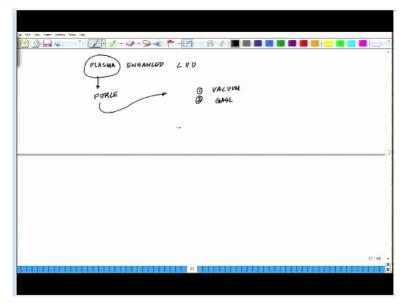
So, chemical vapor deposition CVD and chemical vapor synthesis okay CVD or CVS is a process which is often use for deposition of solid firm from the vapor phase. So you have something in the vapor phase as we mention in the physical synthesis something in the vapor phase that you are depositing it on a substrate. It is kind of used for all sorts of thin film deposition on a substrate most likely we use for thin films synthesis okay.

So thin films produce by CVD processes at certain conditions also contain ultra fine particle. Hence the synthesis of nanomaterial can also be possible by this method. If CVD system is modified, say for example you have high temperature in auto reactor, high super saturation or low residence time on a small substrate. If you have all these parameters and you will really wanted to use very small amount to synthesize nanomaterial on a substrate.

Then the best idea is to follows CVD, but the problem with CVD is the very moment you move to CVD, CVD demands a lot of a intensive instrumentation and high pressure requirements, vacuum requirements and several other complex technical knowhow is essential for chemical vapor deposition okay. So, from CVD, so we are into chemical vapor deposition and plasma enhanced chemical vapor deposition which is a last one.

Plasma enhanced chemical vapor deposition is pretty much further modification of this which is plasma enhanced chemical vapor deposition.

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So, basically plasma enhanced chemical vapor deposition also called plasma assisted chemical vapor deposition is a popular CVD process which is chemical vapor deposition process which is widely used for the deposition of thin films. And plasma enhanced chemical vapor deposition process can also be used for synthesis nanomaterial, as the name suggest the plasma enhances the chemical reactions.

So, you are using plasma as that force just we are talking about the physical forces which is enhancing basically plasma enhancing the nanomaterial giving sufficient energy enhances the chemical reaction for the synthesis of thin films and nanomaterial. So when we talk about such processes as I was mentioning earlier you will be needing a vacuum processing unit. So, these are those requirements or vacuum unit you will be needing.

Then you will be needing a very high power supply, you need a gaseous precursor, so in other word okay you need pretty decently high power supply. And you need pretty specialized apparatus you have this plasma enhanced technical vapor deposition to take place. So you realize that these processes which are as of now we have discuss the chemical and the physical process is. They are the one which help in initiating the field, but now slowly we are realizing much of these techniques require extremely high energy.

And needs very very specialized training in the contrary if you try to emulate nature the way biology has done things. We may be able to make a very different industrial development or very different kind of industry for nanoparticle or nanomaterial synthesis by using biological rule. So, that will be our next target while we will be winding up the basic nanotechnology section.

And from the biological routes we will move onto the application part which will be much more easy to you know slowly bringing down the energy of synthesis and then moving into the agriculture domain. So I will close in here, so our next target will be understanding the biological route of nanomaterial synthesis which in the current context of sustainability, sustainable development is much more relevant to us in terms of delivery of agro chemicals, release of agro chemicals in the agriculture sector.

And as well as boosting the economy of small scale industries which can achieve these things who do not have such sophisticated instrumentation as well as very high infrastructure okay. So, with this I will conclude and in our next class we will move onto the biological synthesis of nanomaterials the future, the sustainable future thank you.