Physics of Renewable Energy Systems

Professor Amreesh Chandra
Department of Physics
Indian Institute of Technology Kharagpur

Lecture 47

Fourier Transform Infrared Spectroscopy

So, let us continue with our module 11, where we are talking about the different types of characterization techniques to understand the properties of materials that are useful to the various devices or systems, which have been discussed in this course.

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The next technique which we will be talking about is the FTIR technique that is the Fourier Transform Infrared Spectroscopy technique. In today's lecture after giving you the basics of FTIR, please note, I will be mostly mentioning FTIR in place of Fourier Transform Infrared Spectroscopy, I will be only using the term FTIR.

So, in today's lecture after going through the basics of FTIR, we will again give you the way sample is prepared for investigation in a spectrometer, which is using the IR radiation. What are the advantages and limitations of this Fourier Transform Infrared Spectroscopy, will also be presented at the end.

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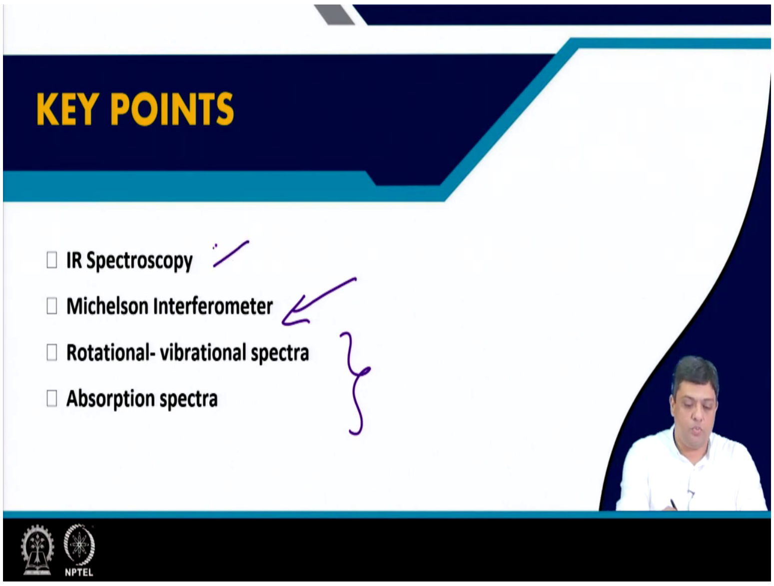

And you will understand once again, that there is lot of physics involved in this technique. You will see the usefulness of Michelson’s interferometer which you may have studied in your first-year course, how that is used to make such a high-end characterization tool, how do you use the data to extract information about rotational spectra, vibrational spectra or absorption spectrums and that can all be done using this spectroscopic technique.

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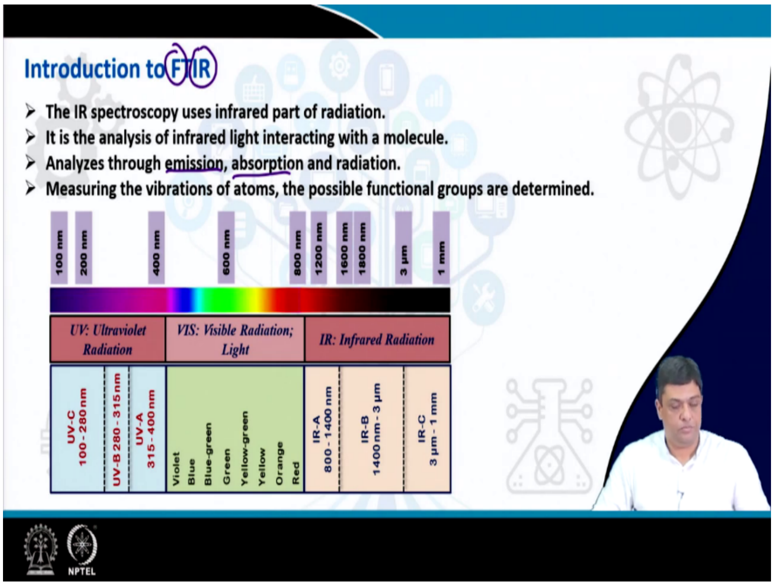

So, very quick revision on the topic of what is FTIR, as you can see, we are talking about two terms the first two stands for Fourier Transform FT, IR stands for Infrared. Very clearly, if I ask you what will be this technique, then you can tell me the IR spectroscopy is the one which uses infrared part of the radiation.

So, we are going to have the infrared part of the radiation as the incident and what all features can be extracted after this radiation interacts with the sample is the details of this technique. So, in this incident, infrared light will interact with the sample and give us some signal, if we can analyse that signal using some laws, then maybe useful information will come out.

This is what you will tell me and that is exactly what FTIR is all about. This technique is able to give you information by analysing the emission, absorption of the incident radiation and basically, it measures the vibrations of the atoms and the possible functional groups, which are associated to a molecule can also be determined. So, this is what the whole technique is all about.

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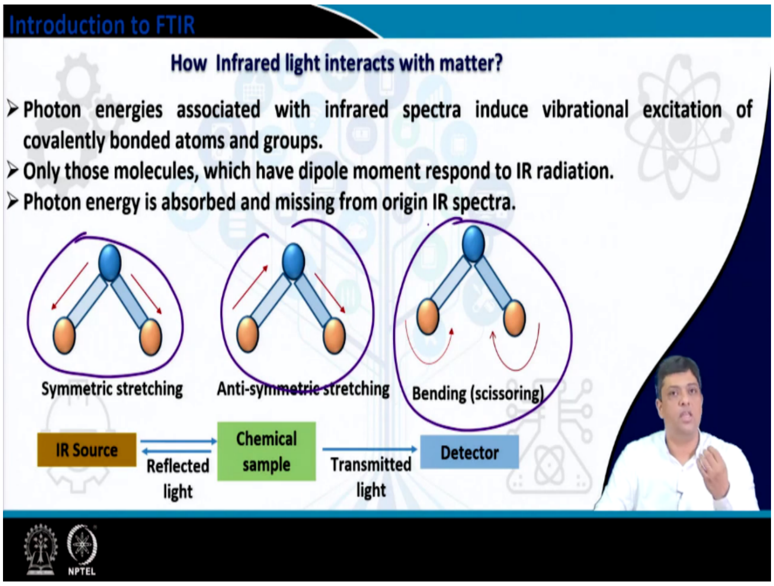

Now, let us say we have an incident IR, what will happen? The question I am asking is how would this infrared light interact with the matter? This is explained in this slide, what happens, the photon energies associated with the IR spectra induce vibrational excitation of the covalently bonded atoms and groups. So, if you have atoms which are bonded then the photon energy will be absorbed by these atoms and that will induce the excitation in the vibration.

Now, if there is excitation, then what type of vibration are we talking about? There are three types of terms which we use in IR spectroscopy. You have symmetric stretching, anti-symmetric stretching or the bending modes and the nature of vibrations are indicated by the arrows in these three types of excitations.

So, you can clearly see what is happening in the bending case, you have the vibrations such that the atoms are coming near to each other. Whereas, in anti-symmetric the vibrations are opposite, while in the case of symmetric stretching you have the symmetric stretching of the bonds. You can understand that only those molecules which have dipole moment they respond to IR radiation.

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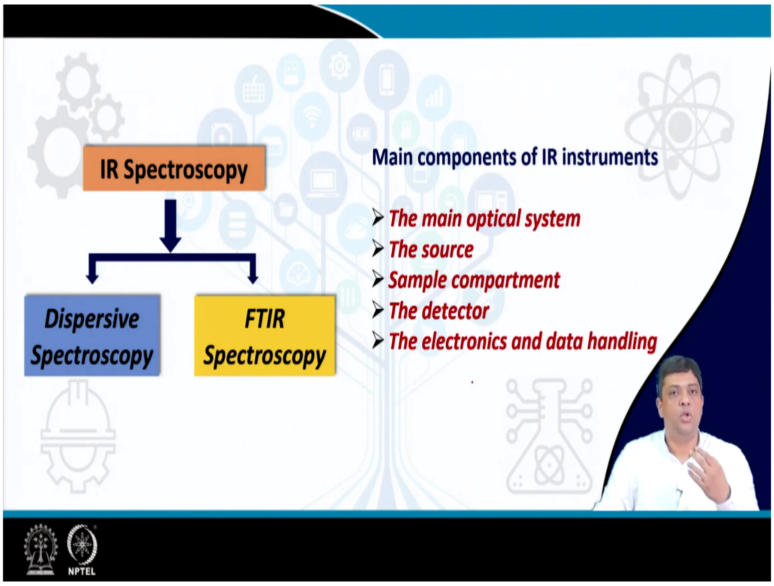

So, IR spectroscopy can be divided into sub classifications, where you will talk either about dispersive spectroscopy or FTIR spectroscopy. And in both these kinds of instruments there are the components which are used, there are the main optical system.

Obviously there will be source, then you will have a place where you will put the sample after the incident radiation has interacted with the sample, it will lead to generation of certain kind of signal, that signal has to be collected, that is collected using the detector and the detector transfers this information to your electronic or data collection center, where you analyse the data. So, this is the main component of IR instruments.

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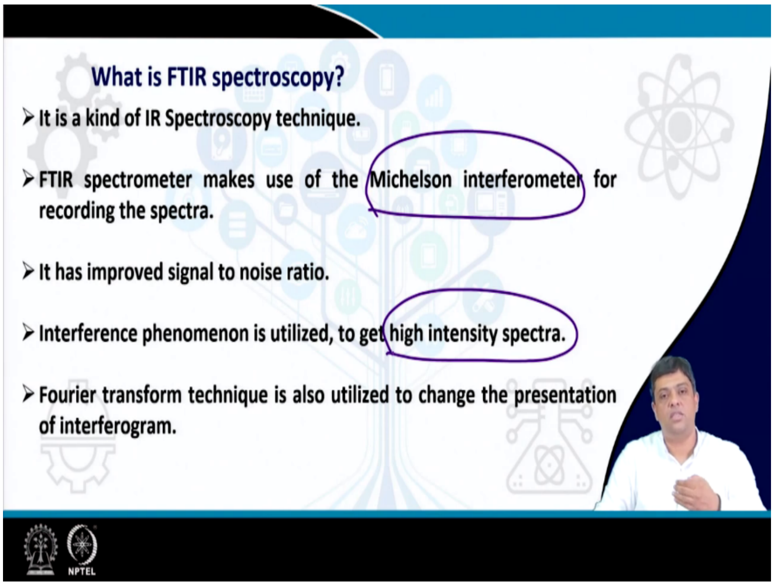

As I said FTIR spectroscopy uses the principle of Michelson's interferometer for recording the spectra. Because of this you have an improved signal to noise ratio. So, signal is predominant in the overall data and the noise levels are much lower, this is what you mean by the sentence it has improved signal to noise ratio. Interference phenomena is utilized and if interference phenomena is utilized, you can claim that you will be analysing high intensity spectras.

And then Fourier Transform technique is utilized to change the presentation of the data and analyse even slight changes which may be lost in the initial or the original data if you take the Fourier Transform of the data then slightest of change also manifests itself in the data. So, you take the data and then take the Fourier Transform and so that you can analyse even the slightest of change in the pattern.

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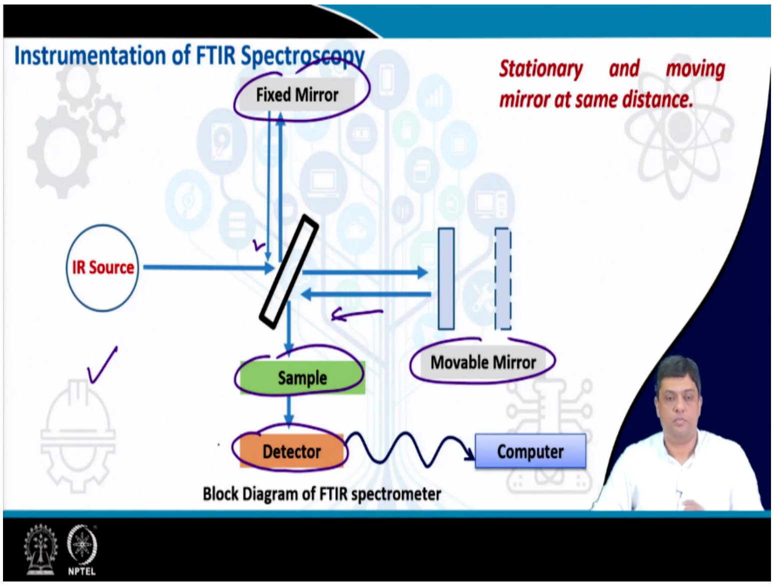

This is the block diagram of FTIR spectrophotometer and you have seen this earlier and this is the typical Michelson's interferometer. So, what you have, you have the source, you have the let us say the mirror, you have the movable mirror, the fixed mirror, you have the two rays which then after crossing the mirror falls on the sample, the sample leads to the appearance of the signal and that is collected by the detector. So, this is what you actually have in the FTIR spectroscopic data collection instrument and the principle is that of the Michelson's interferometer.

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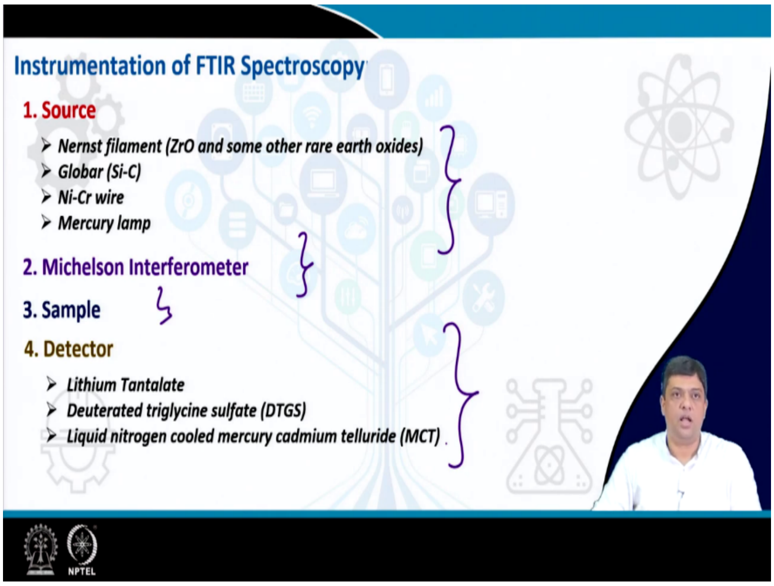

So, what we have? we have source which can be of different type, you have the interferometer the sample and the detector and different types of detectors are used.

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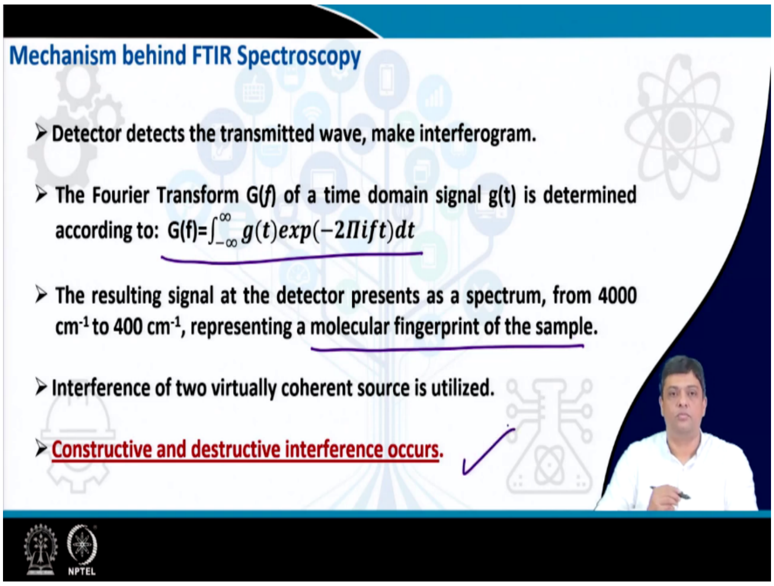

What happens? now I have ensured that there is an IR which is now falling on the sample and resulting to a generation of a signal. The detector detects the transmitted wave and gives rise to an interferogram.

The Fourier Transform in the time domain is determined by what? By the integration given here. And the resulting signal at the detector presents as a spectrum, for example, let us say from the in the range 4000 centimeters inverse to 400-centimetre inverse in the instrument which we are talking about. And this actually is linked to the molecular fingerprint of the sample.

What is happening? Just as in Michelson's interferometer, the interference is basically of the two virtually coherent source that is obtained and using this virtually coherent source, you can then lead to the condition of constructive or destructive interference and based on that you will have a spectrum. This is the basic concept of FTIR spectroscopy.

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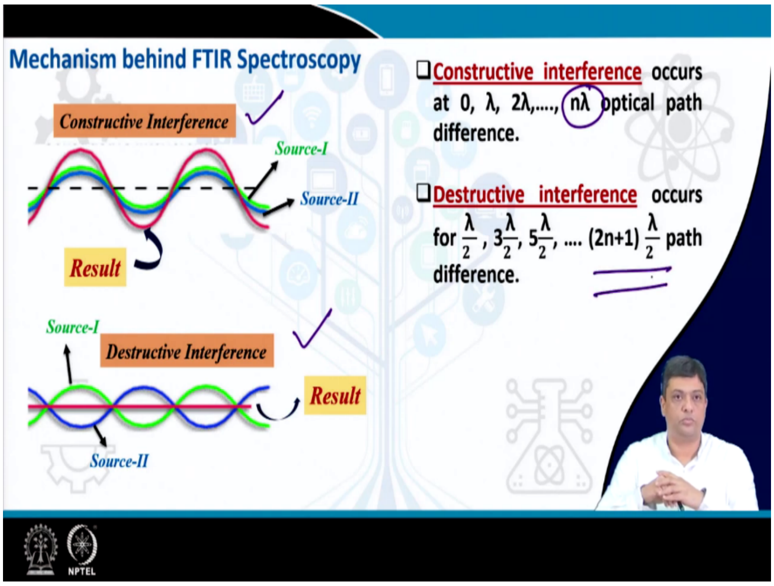

And the whole thing is explained in this curve. So, if you have constructive or destructive interference then what happens? The optical path length in constructive is equal to n lambda and in the case of destructive interference case the optical path length is 2 n plus 1 lambda by 2.

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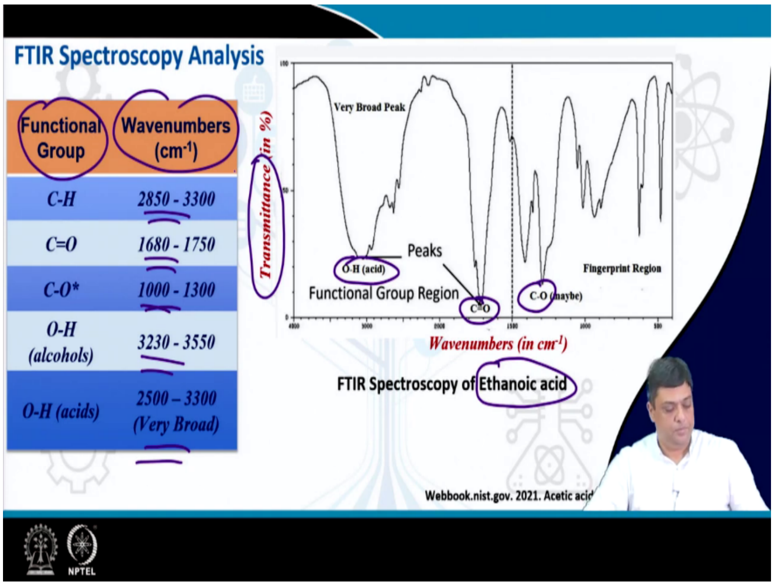

And this is what an FTIR typically looks like. And if you look about the spectrum given for acetic acid, you will find that you have functional groups and wave numbers. And this is the range given in which the signals appear in different samples and in this case the ethanoic acid. What do we mean about O-H? so we are talking about the O-H bonds, then there is C-O bond, C-O double bond.

So, these are the bonds, what will happen? They will absorb the energy which is coming in from the incident IR ray and then you will have the absorbance or you will plot the transmittance because you are taking the data after the ray has crossed the sample and so you are plotting the transmittance.

And you will find that absorbance or transmittance will depend upon the energy or molecular levels which are present that will lead to the absorbance of energy from the incidence source and that will lead to the signal.

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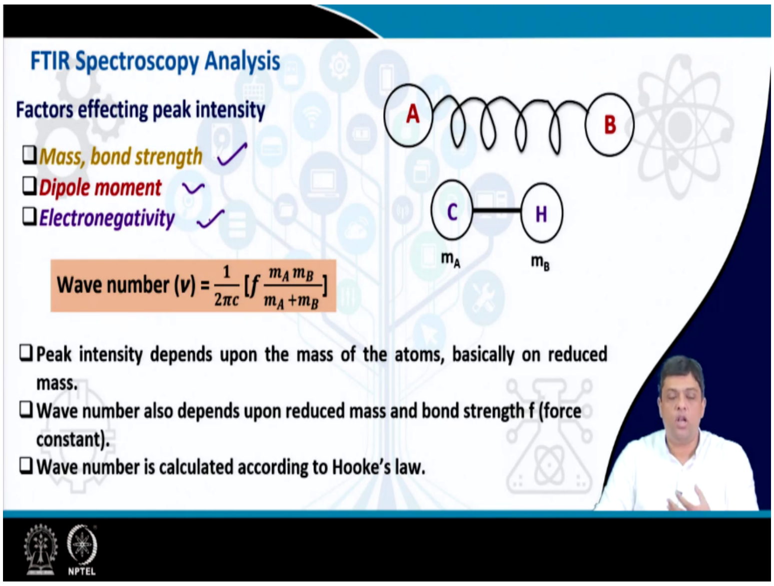

Obviously then what will happen the factors would be there that will affect the peak intensity. You will, if you have factors about the mass then bond strength, dipole moments, electronegativity. So, these are the typical factors which will affect the peak intensity which you see in an IR spectroscopic data. Because of these are the factors which will depend, which will control the amount of energy the sample will absorb from the incident radiation.

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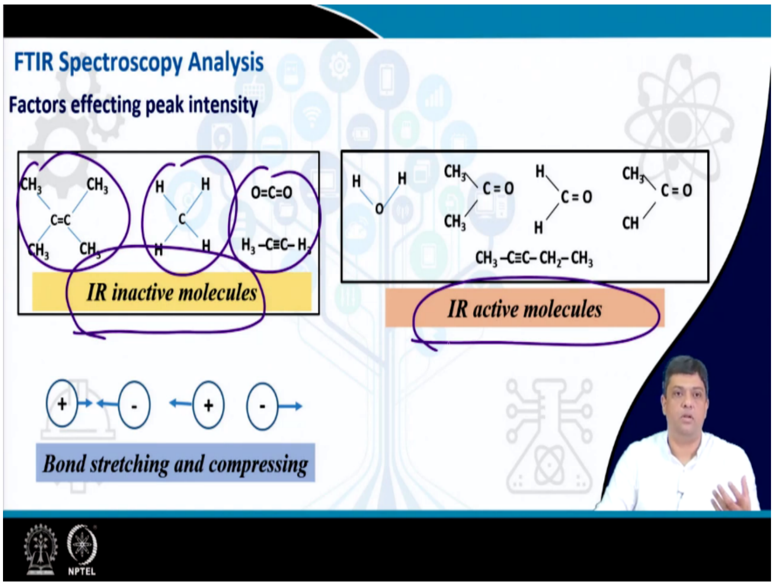

If you have systems like this, then they are IR inactive, whereas when you have samples which where you are seeing the dipole moments then you have the IR active molecules and you then have bonds either stretching bonds or compressing bonds. So, you also have IR active bonds or IR inactive bonds within the sample.

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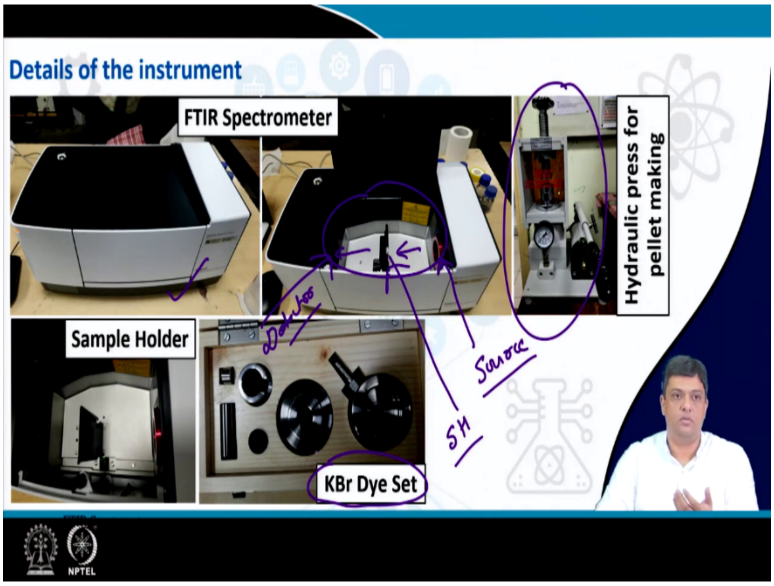

This is what a typical FTIR spectrophotometer looks like in picture. You have a photometer this is the compartment where you will have the source, you will keep the sample and then you have the detector. This is the load press machine where you are going to make the pellet which will be placed in the sample holder and then the source will generate the signal.

That signal will fall on the sample; the transmitted beam will be analysed by the detector. This is what is going to happen. So, this is typical KBr dye set because you are going to use KBr as the sample in which you will be putting your sample which has to be measured and KBr will be used as the standard.

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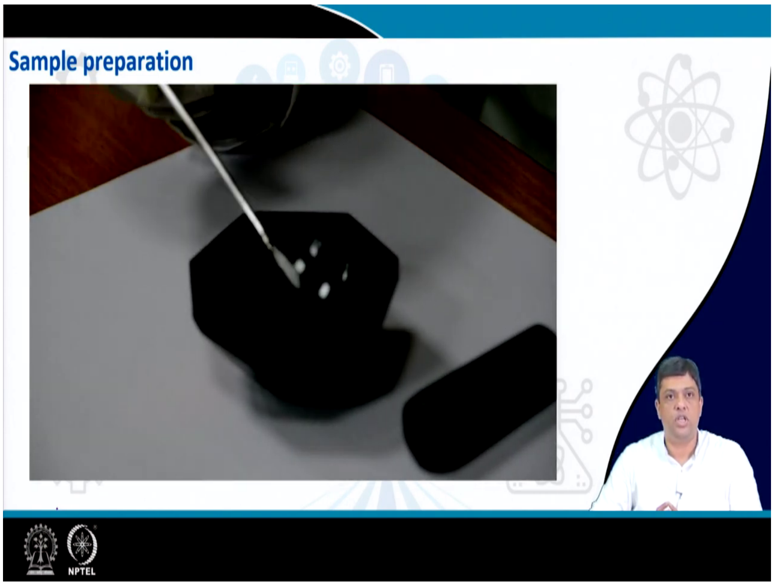

So, let us start from the beginning, that is sample preparation. So, what you will do you will take the powder in a mortar pestle, then you will crush the powder into fine and homogeneously dispersed form, so that you remove agglomerates then you have cuvettes.

And after the sample is placed, in these cuvettes, you will then have the solution or you will use the load press machine to prepare the pellet. In the case which we are discussing here, you will find that we are talking about the formation of a pellet in circular form. So, again you crush the powders using the movement of the pestle and in the form of 8, so that you do not induce any preferred orientation. And after the pellet is crushed and you obtain fine powders you will make the dyes.

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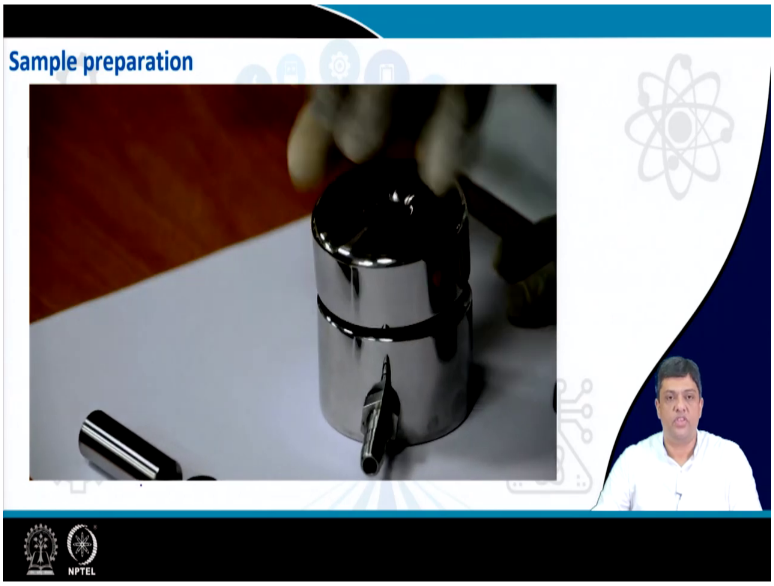

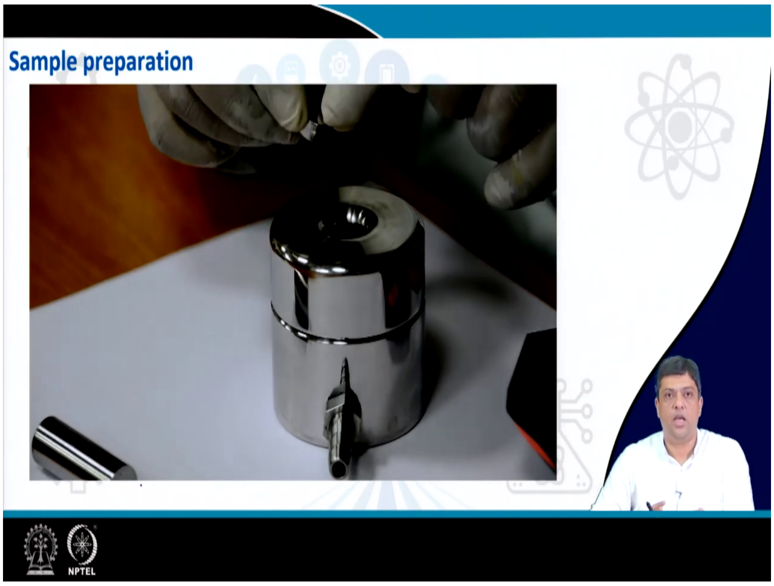
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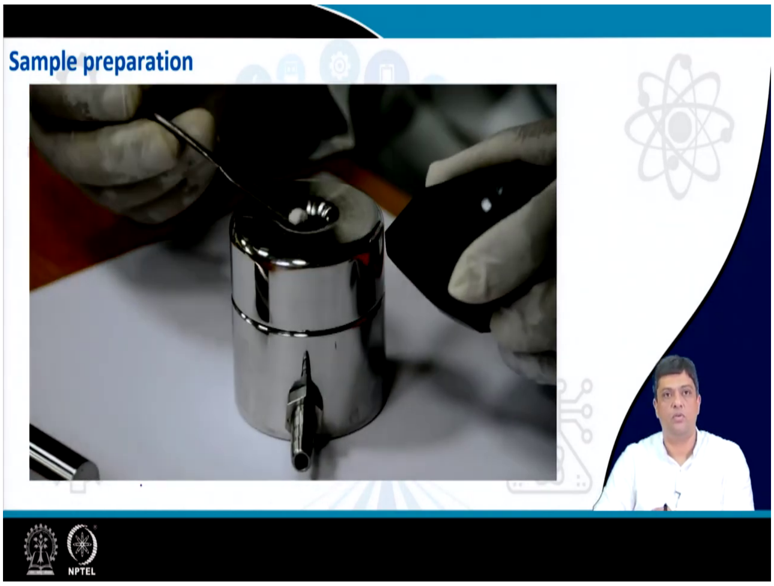
Sample preparation
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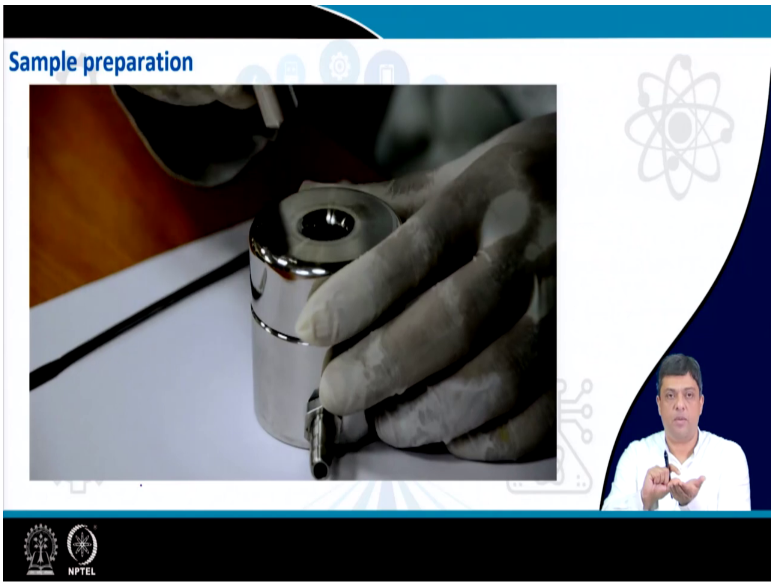
Sample preparation
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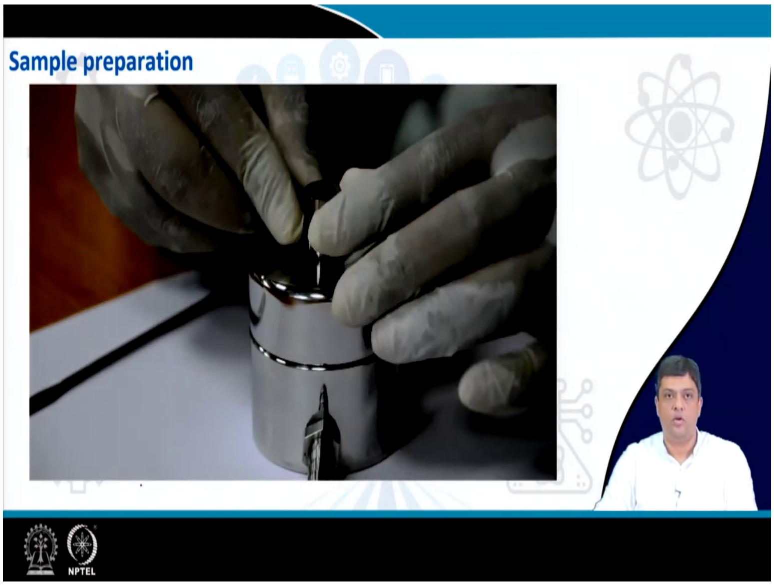

This is the dye in which you will first press a small tablet at the bottom, then you will put the powder on top of it. Why you put a small tablet before you put the powder? because it has to be formed on top of this tablet so you can remove it from the time. Then you have the plunger you put the plunger, put the O-ring so that you do not have any force acting outwards, when the plunger is put under high load.

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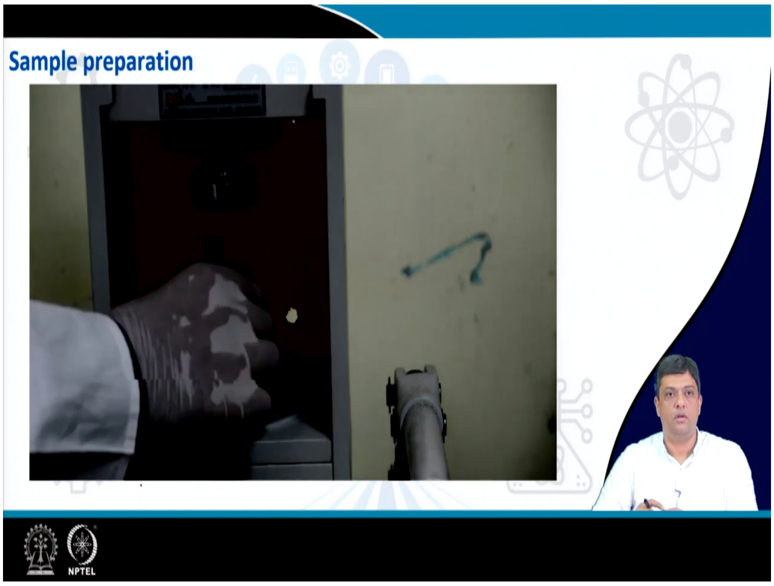

Sample preparation

Sample preparation

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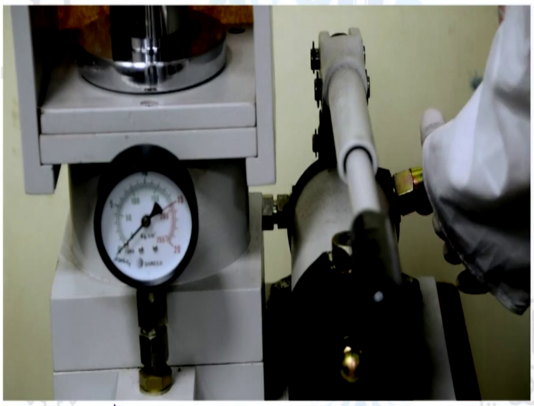

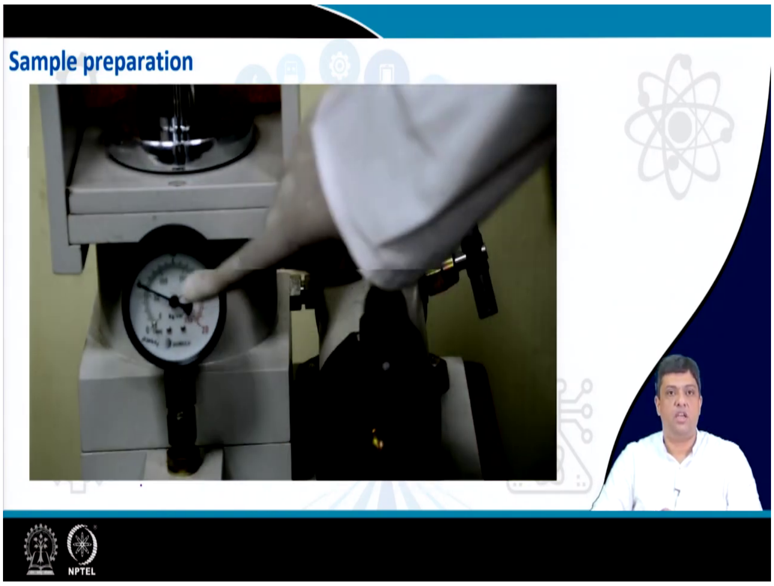

After you have obtained this combination this is taken to a load press machine. Please remember that it should be so placed that the plunger is vertically downwards when you are putting the load from the top if it is misaligned that the plunger may break. Then you apply let us say 5 ton.

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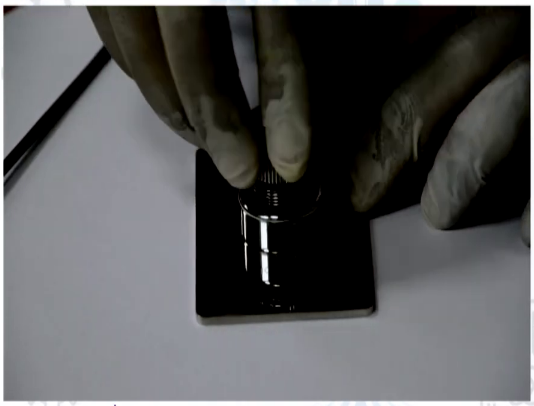
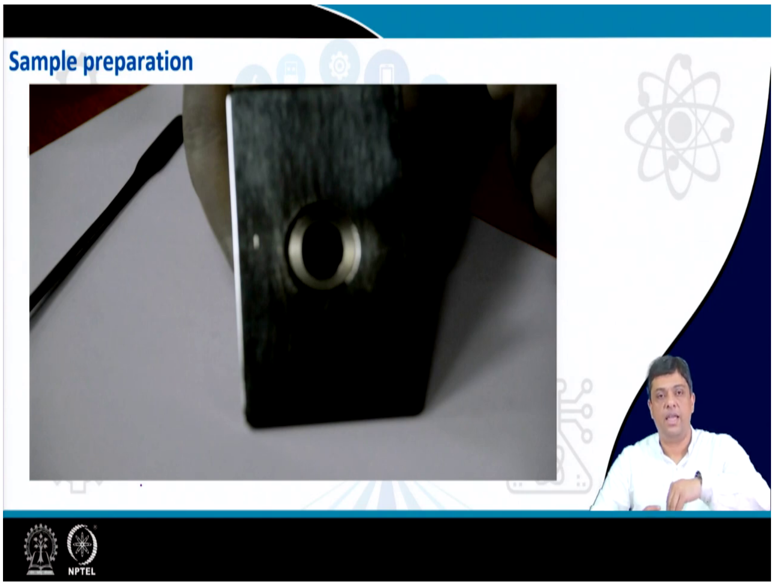
Sample preparation

Sample preparation

Sample preparation





And after you have applied 5 ton, you can leave it for some time then you slowly release the pressure, once you release the pressure then you will open the dye and you will get the brown pellets, those pellets are picked up and then they are taken and placed in the sample holder.

And this sample holder will be placed in the path of the IR light and the signal which is obtained behind that is the transmitted signal, is then analysed and you extract information regarding the various modes which are present in the sample. And as the nature of the sample changes the nature of vibration changes and you can get lot of information about the material.

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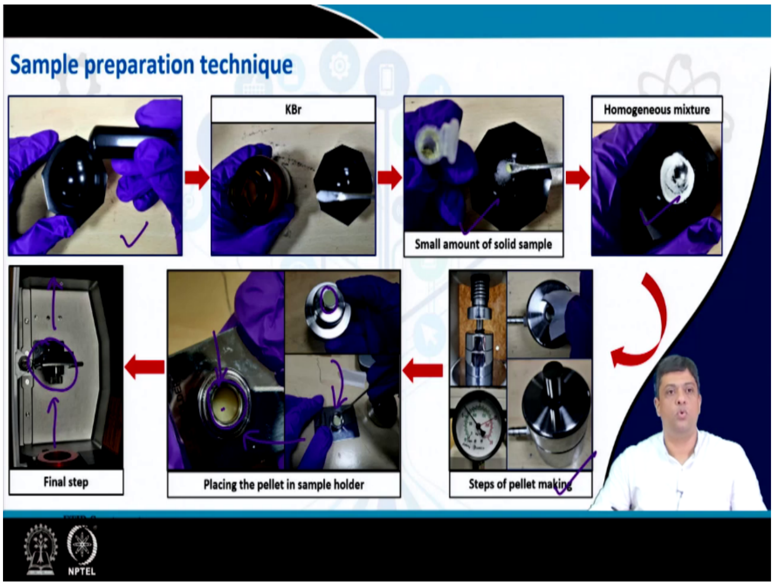

So, once again very quick revision in just pictures sample preparation crushing of powders, homogenization of the powders, then preparation of pellets KBr pellets and picking up of this pellet, placing it in the sample holder and then using the screw to tighten the sample inside the holder while ensuring that the sample is actually having an opening through which the IR from the source will enter and hit the sample and so source sample holder detector, this is what you must ensure and these are the steps involved in data collection.

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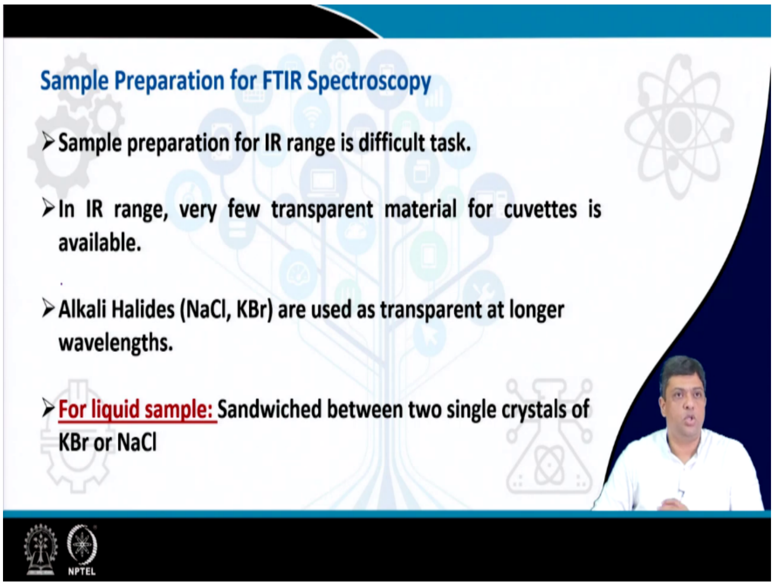

Sample preparation in IR spectroscopy is sometimes difficult. And you must ensure that you have to use materials which are transparent, materials for cuvettes if they have to be used in IR. And alkali halides are used as transparent at holders or reference backgrounds at longer wavelengths, but for liquid samples, the measurement is mostly in the sandwich geometry, where the sample is sandwiched between two single crystals of KBr and NaCl. So, this is the process of handling the sample in FTIR spectroscopy.

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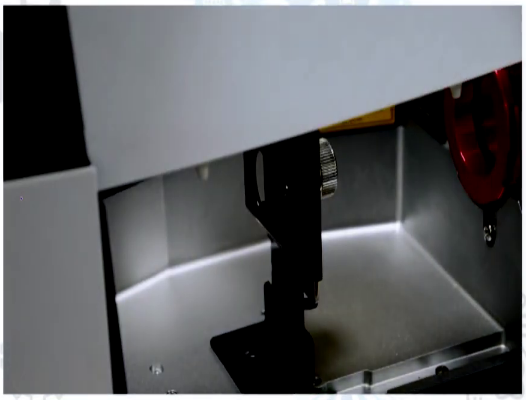
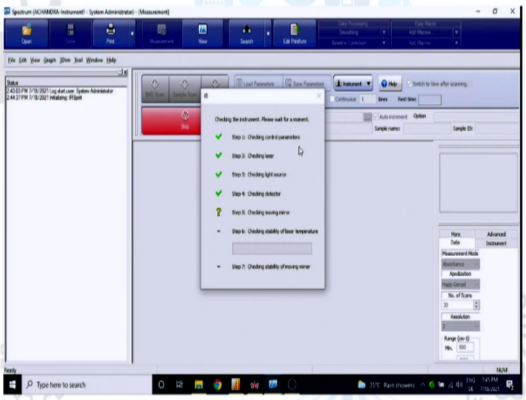
Data collection

Data collection



Data collection



Again, this same concept after you have placed the sample in the chamber and you are having the source sending out signal, you will close the chamber so that there is no light or extrinsic factors appearing, you will set the measurement conditions, and once that is done you can start the measurement.

For example, you will have the sample which will check the control parameters you will, the instrument will check the detector, it will detect the mirror because you are using the Michelson's interferometer, you have mirror 1 which is movable and after all these processes are performed, you can start your measurement.

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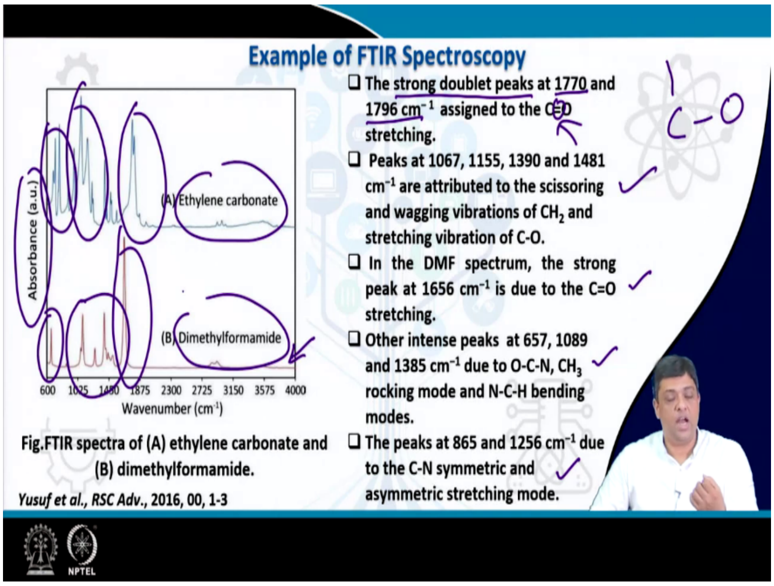

And the typical data which you obtain let us say for ethylene carbonate or any other sample DMF which is shown in the bottom, you will have the spectral lines and you see the spectrum is giving us peak profiles and they are all originating because of the stretching or any other kind of bending or rocking modes between the different atoms which are forming this molecule.

For example, the strong doublet peak at approximately 1770 and 1796 centimeters inverse is mostly assigned to the stretching mode of the C-O double bond. And once you start obtaining the data you will be able to analyse each of these materials.

Now, for example, suppose I tell you that this double bond is not present but you have single bond, then if that happens what is happening? There is a change in the material or the characteristic nature of the molecule. If that is happening what will happen?

What will be the thing that you expect, that means the way this molecule will absorb energy would be different. And if that is different the signal which you will get will also be different and so you will see appearance or disappearance of certain peak profiles as a function of changing nature of the molecules.

So, when you go from one molecule to the other then you have different characteristic IR spectrum associated with those materials. You can clearly see the spectrum for ethylene carbonate is not similar to that of dimethylformamide, why? Because the nature of bonding is very different.

What happens, what is the consequence? I can understand different materials using this technique and slightest of change in these materials will be indicated by variation in the spectra. As this is an optical base technique it is very sensitive to change so you can get quite rigorous information about the materials.

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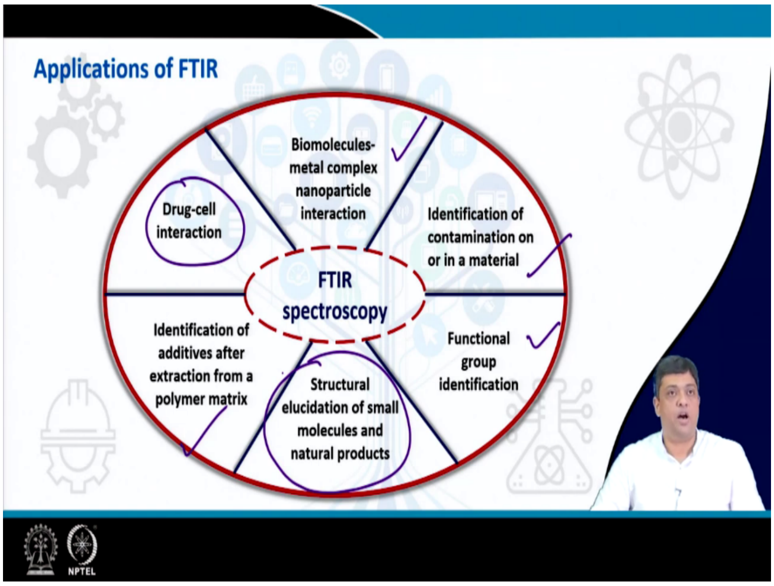

And the range of application of this spectroscopy is tremendous. You can get information about the functional groups, if there are contamination in the sample, you will get additional peaks or profile spectral lines you can determine using the FTIR spectrum. If you have metal complexes forming with biomolecule or any other nanoparticles that can be analysed.

You can talk about the drug and the interaction of this drug with the cell, because if there is interaction, that means the drug is actually interacting with the cell, then the nature of bonds will change and then as soon as soon as the nature of bonds will change, what you will see that the spectrum which you have obtained will change with different stretching or bending bonds.

You can talk about identification of additives in polymer matrix, structural changes in small molecules and natural products can also be investigated using this technique. You can talk about the occurrence of phase transition as a function of substituent, dopant or you can talk about structural phase transitions as a function of temperature. Because if there are phase transitions the atomic positions change in the unit cell.

And as the atomic positions change, the nature of bonds change, and as the nature of bonds will change the range in which the material will absorb the energy would also change and so if there are structural phase transitions that can also be investigated using this technique. So similar to x-ray diffraction FTIR is also a very powerful technique with a large number of applications.

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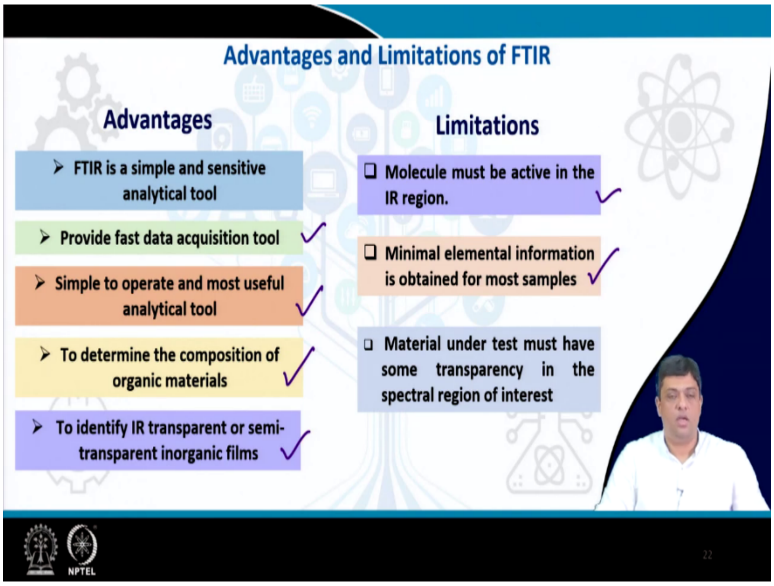

It is also associated with certain advantages and limitations. Because there is no technique which can give information about the material in such a way that no other technique is then required. So, there is always a limitation of a particular characterization technique. For example, you will see if you understand Michelson’s interferometer you can extend that technique to fabricate an FTIR instrument.

So, even the instrumentation is very simple and the whole analysis of the data is simple, it can be performed at a very rapid speeds, so the data collection is fast. This is simple to operate; you can have a large number of applications. This technique is also associated with certain limitations; it can only be used for molecules which have or which are active in the IR region. Very little information regarding the elements is obtained.

And material which is under the test must have certain amount of transparency in the spectral region of interest, otherwise you will not get any transmitted beam if the material is opaque in the range which we are having the instrument operated. And then no signal will come out and you have no information. So, you need to have transparency in the region where the instrument is obtained and materials should have these characteristic, then only they can be investigated using the FTIR.

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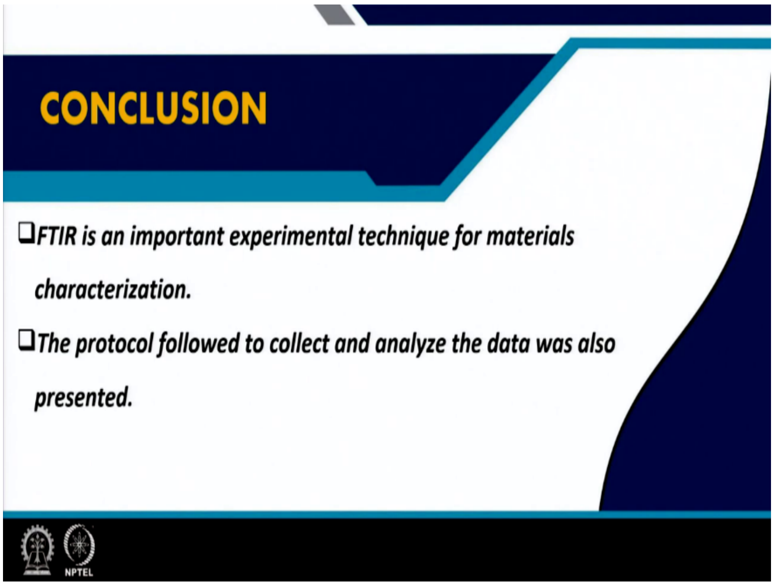

So, I hope it is clear to you that FTIR is an another important experimental technique for materials characterization. And the protocol followed to collect and analyse the data is simple and the details of all these things were also presented in today's lecture. So, I hope you will be able to perform the experiment by your own if you have an FTIR instrument in the lab.

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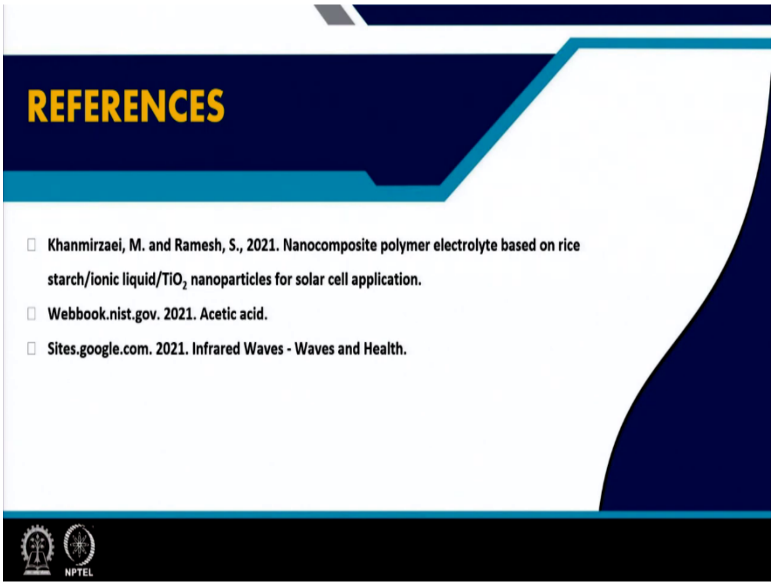

These are the references which were used for obtaining the data that were present in today's lecture. And I thank you for attending the lecture today, have a nice day!