Novel Technologies for Food Processing and Shelf Life Extension

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Lecture – 48
Rapid & Non-destructive Methods for Quality Analysis

Hello everybody. Today, in this class, we shall study Rapid and Non-destructive Methods for Quality Analysis. You know the chemical methods which are used generally for determination of quality in food materials, they are many a times tedious and they are time consuming. Also, the methods of preparation of the sample takes adopt time, many a times the sample gets, destroyed, it is gets changed. So, to avoid all these problems, the rapid determination or non-destructive methods for the analysis of food materials have significant importance, particularly these rapid methods. Once they are developed and perfected, they can be used continuously for routine analysis of the food materials and so on.

So, in this half an hour also, I will discuss some of the important methods which are rapid and non-destructive methods. And although these methods can be used in wide range of food materials for analysis of various quality attributes, but I will concentrate to mostly on the analysis of these grains, analysis of grains using rapid and non-destructive methods. But the similar principle similar way these can be applied to other foods as well.

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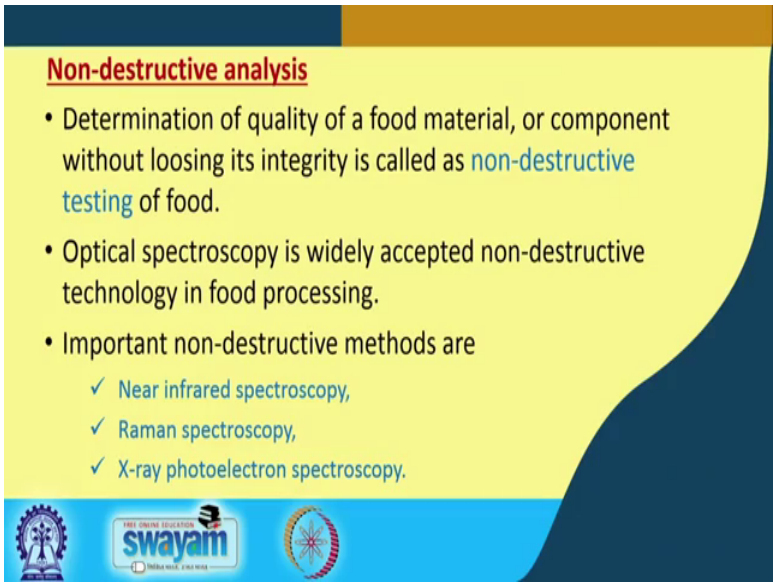

First let us see what a non-destructive test means that is a determination of quality of a food material or component without losing the integrity of the sample that is the sample is analyzed as it is without it does not need any a specific preparation of the sample to make it good for analysis. So, that is what is the non-destructive testing means. Then only optical a spectroscopy is used or it is widely accepted non-destructive technology for food processing or for food quality testing.

Important non-destructive methods for analyzing food quality attributes include Near infrared spectroscopy, Raman spectroscopy, and X-ray photoelectron spectroscopy. In our lecture, we will mainly concentrate on near infrared spectroscopy.

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So, the NIR Near Infrared Spectroscopy, there are basically two processes which are used either Fourier Transform Infrared Spectroscopy that is FTNIR or FTIR.

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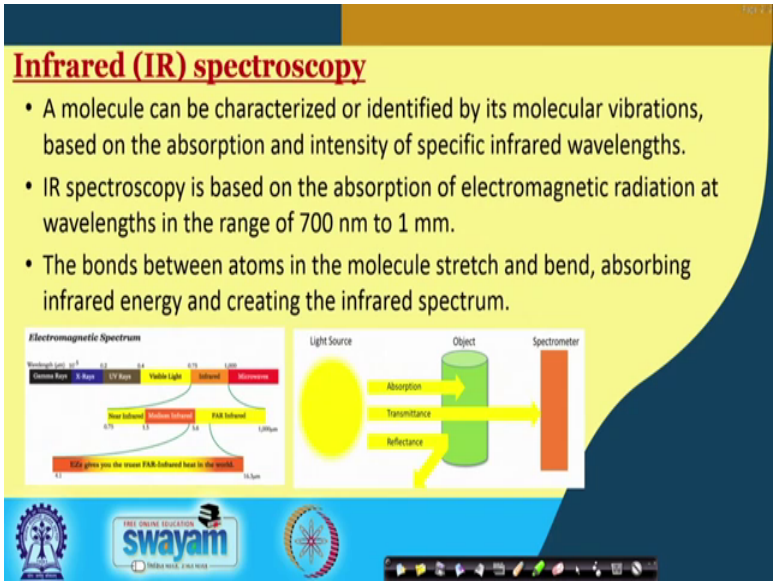

So, infrared spectroscopy, what does it mean that is in this method a molecule can be characterized or identified by its molecular vibrations based on the absorption and intensity of a specific infrared wavelengths. IR spectroscopy is based on the absorption of electromagnetic radiation at wavelengths in the range of 700 nanometer to 1 millimeter. The bonds between the atoms in the molecule is stretch and bend, absorbing

infrared energy and creating the infrared spectrum. So, this is the whole in brief the principle of this method.

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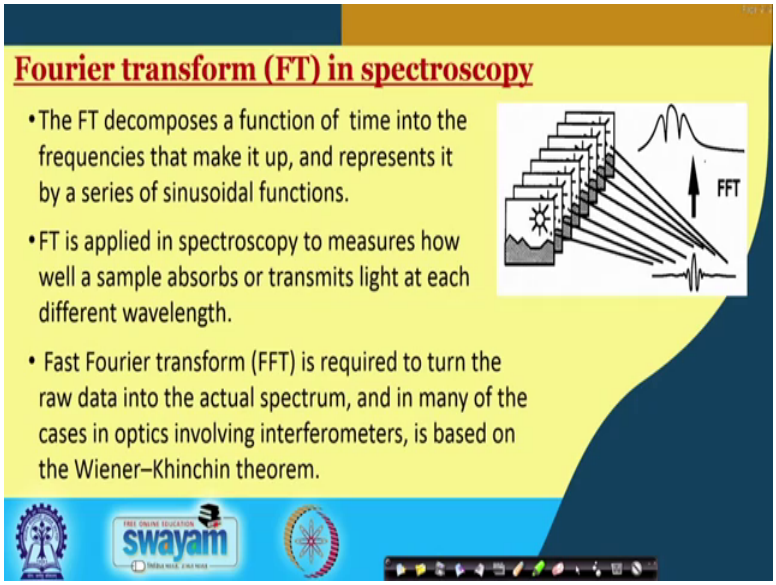

Regarding Fourier transform in spectroscopy, the Fourier transform decomposes a function of time into the frequencies that make it up, and represents it by a series of sinusoidal functions. Fourier transform is applied in a spectroscopy to measures how well a sample absorbs or transmits the light at each different wavelength. The fast Fourier transform is required to turn the raw data into the actual spectrum, and in many of the cases in optics involving interferometers; that is the fast Fourier transform is based on the Wiener-Khinchin theorem.

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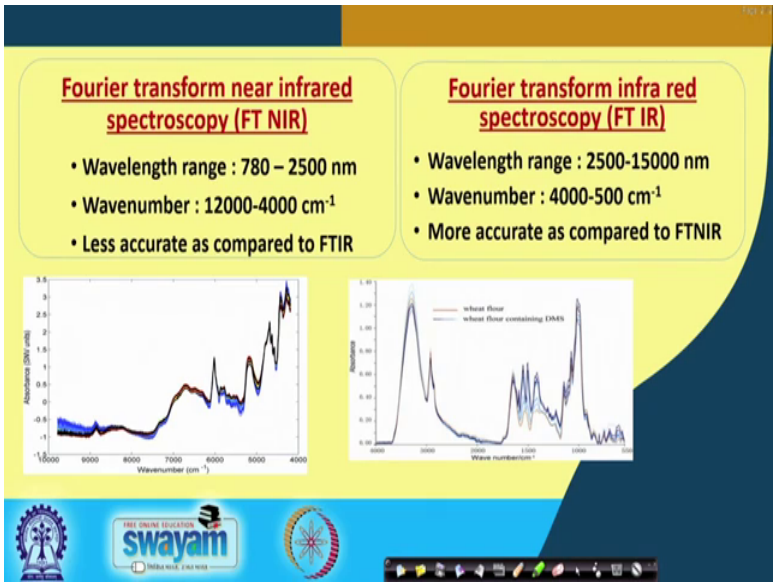

So, the FT NIR and FTIR, there is two most commonly used methods are there which are used for analyzing various food components. So, both of them are generally the same principle of operation is same; the only difference or measured difference is the wavelength range or wave numbers which is used like for example as you can see here that in the case of FT NIR, the wavelength range is generally 780 to 2500 nanometers or wave number up to 12000 to 4000 per centimeter.

In the case of Fourier transform infrared spectroscopy FTIR, the wavelength range used is 2500 to 15000 nanometers; and wave numbers may be 4000 to 500 per centimeter. The accordingly because of these differences in the wavelength and wave numbers, the FT NIR analysis is considered comparatively less accurate than that of the FTNIR.

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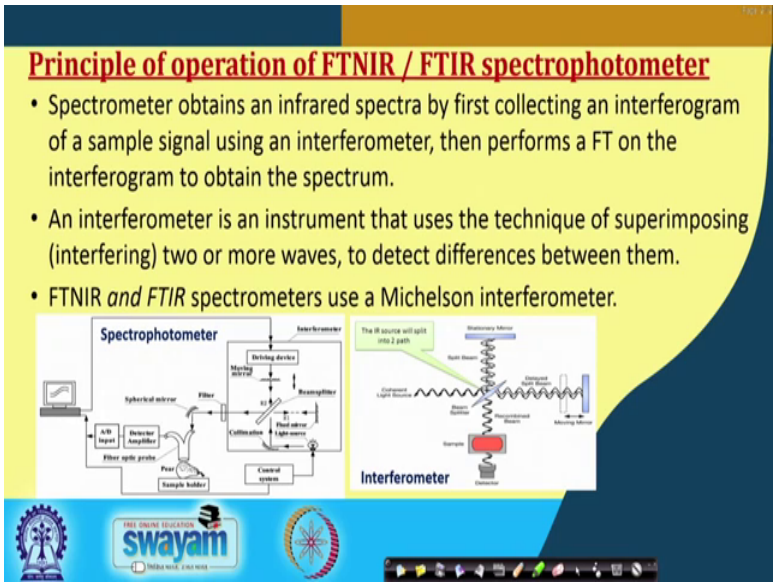

Principle of operation of FTNIR or FTIR is spectrophotometer that is as you can see in this picture. It is there are spectrophotometer are provided by some interferometers. So, a spectrophotometer obtains an infrared spectra by first collecting an interferogram of a sample signal using an interferometer, then it performs Fourier transform on the interferogram to obtain the spectrum.

An interferogram is an instrument that uses the technique of superimposing or interfering two or more waves, to detect differences between them. So, FTNIR and FTIR spectrophotometers use a Michelson interferometers that is here shown in the figure.

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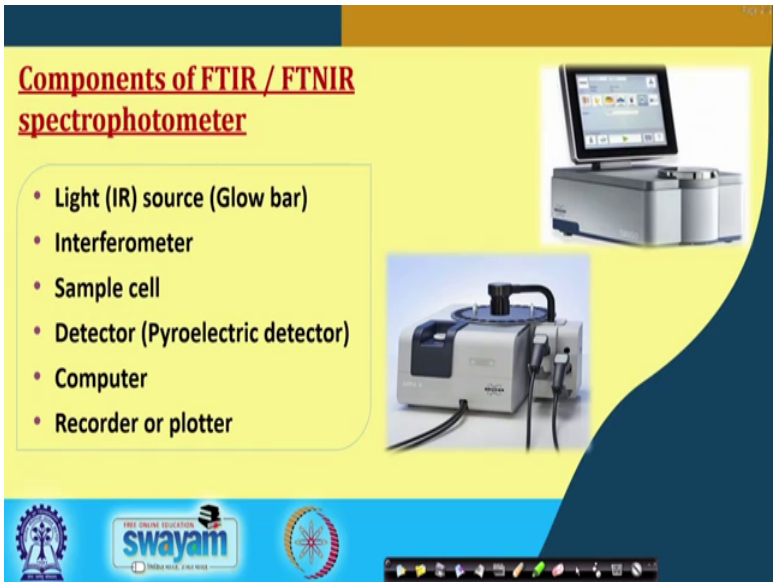

So, accordingly the components of an FTIR or FTNIR spectrophotometer includes that is a light source glow bar, an interferometer, a sample cell, then a detector, computer, and recorder or plotter. So, this is one FTNIR system which we have in our laboratory or FTIR system etcetera that is the simple it is very simple and handy instrument which can table top instrument, and it is compact system that is computer. In this case, the computer is connected separately.

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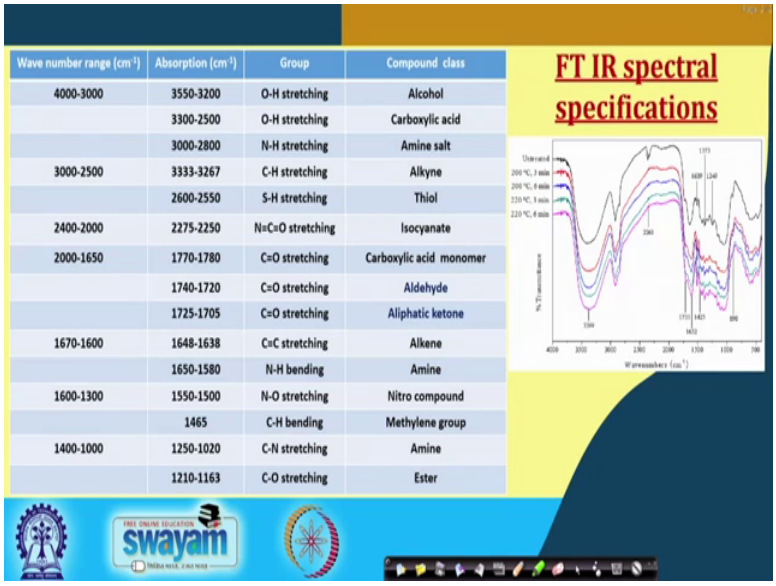

Wave number range (cm^{-1})	Absorption (cm^{-1})	Group	Compound class
4000-3000	3550-3200	O-H stretching	Alcohol
	3300-2500	O-H stretching	Carboxylic acid
	3000-2800	N-H stretching	Amine salt
3000-2500	3333-3267	C-H stretching	Alkyne
	2600-2550	S-H stretching	Thiol
2400-2000	2275-2250	N=C=O stretching	Isocyanate
2000-1650	1770-1780	C=O stretching	Carboxylic acid monomer
	1740-1720	C=O stretching	Aldehyde
	1725-1705	C=O stretching	Aliphatic ketone
1670-1600	1648-1638	C=C stretching	Alkene
	1650-1580	N-H bending	Amine
1600-1300	1550-1500	N-O stretching	Nitro compound
	1465	C-H bending	Methylene group
1400-1000	1250-1020	C-N stretching	Amine
	1210-1163	C-O stretching	Ester

So, the different components present in the food materials. They give different a spectra under different wave number ranges or different wavelength at different wavelengths. And accordingly these groups of a spectra are obtained as you can see here that is FTIR a spectral is specifications. Like for example, the group O-H stretching in the compound alcohol this is a spectra is generated in the wave number ranges of 4000 to 3000 centimeter are absorption spectra pattern of 3550 to 3200.

Similarly, the compound like carboxylic acid, monomer etcetera were C-O a stretching group, they give the spectra absorption spectra at 1770 to 1780 per centimeter are in the wave number ranges of that 2000 to 1650 per centimeter. So, means when this spectra is generated, then there are some techniques I will tell you little later using those techniques these spectra are processed like, and finally, the compounds is done that is both quantification as well as that detecting that is both qualitative and quantitative measurements can be done using this.

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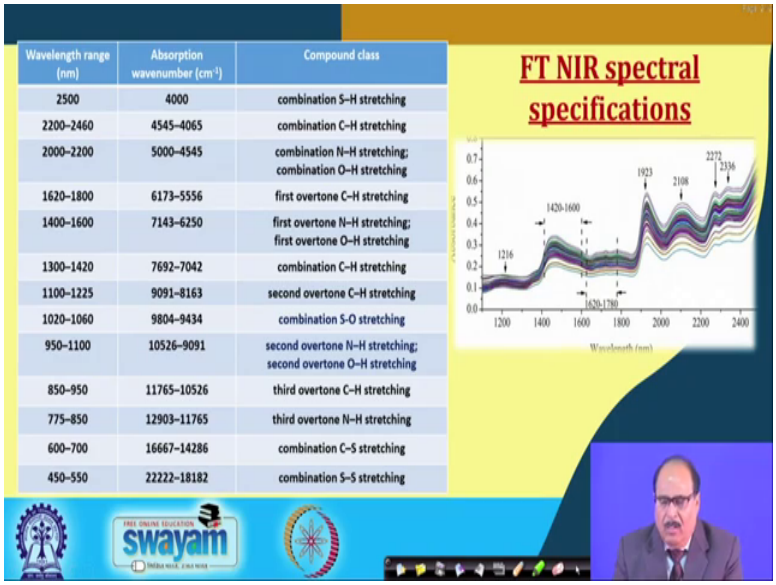

Similarly, this slide gives you this table gives you that FTNIR spectral specifications that is in this spectra, this is the spectra where the wavelength versus absorbance y-axis absorbance is there, and x-axis wavelength. So, you can see that is at the different wavelength and how different types are these spectra are obtained. And these spectra like for example wavelength 2500, absorption spectra that is absorption wave number 4000 per centimeter that is that is compound class may be, combination S-S stretching that at

in the 1300 to 1420, absorption wave numbers six 7692 to 7042, it may be a C-H stretching combination.

So, depends the spectra is obtained. These a specifications which are a spectra which are specific to these specific wavelength or wave numbers. They are identified and used in the analysis and even finally, for that identification of the compounds and its amount etcetera.

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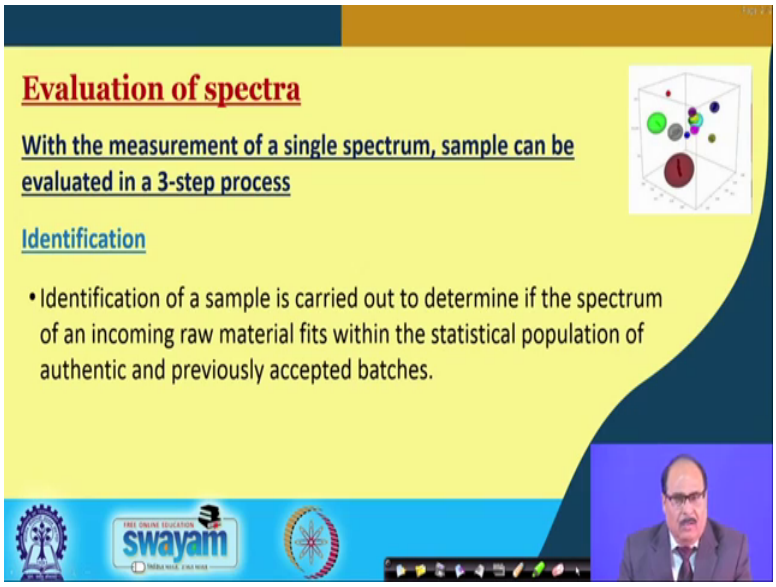

So, the spectra is created. So, as I told you next step is the evaluation of the a spectra that is with the measurement of a single spectra even sample can be evaluated in a 3-step process that is the first process is the identification. That is a identification of a sample is carried out to determine if the a spectrum of an incoming raw material fits within the a statistical population of authentic and previously accepted batches. In fact, what is done in this case in both in FTNIR, FTIR etcetera that is a important aspect in the library creation. The first the material is given and large number of spectra is generated, and then library are created. I will come little later to this aspect. That is after the identification the next becomes the conformity.

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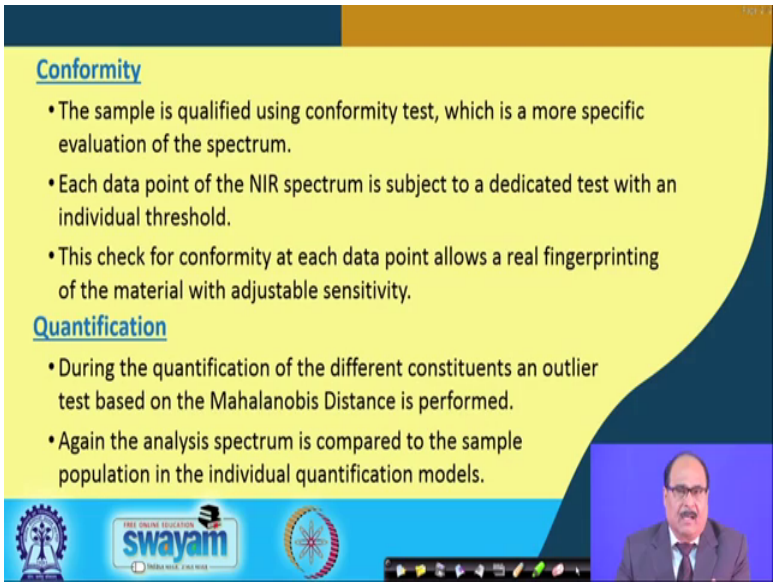

The conformity in conformity what is done, the sample is qualified using conformity test which is a more a specific evaluation of the spectrum, that is the spectrum which is generated by the interferometer, then it is evaluated are and the depend as I told you that wave number and wavelength whether this is spectra which is obtained it is for C-H, or it is for C-O, it is for which functional group and accordingly for then each data point of the NIR spectrum is subject to a dedicated test with an individual threshold. And this check for conformity at each data point allows a real finger printing of the material with adjustable sensitivity.

So, after the conformity quantification, the last step of the evaluation, that is during the quantification of the different constituents and outlier test based on the based on the MD is performed that is Mahalanobis distance test is performed. Again the analysis spectrum is compare to the sample population in the individual quantification model and the that is are qualified, depending upon the absorption spectra.

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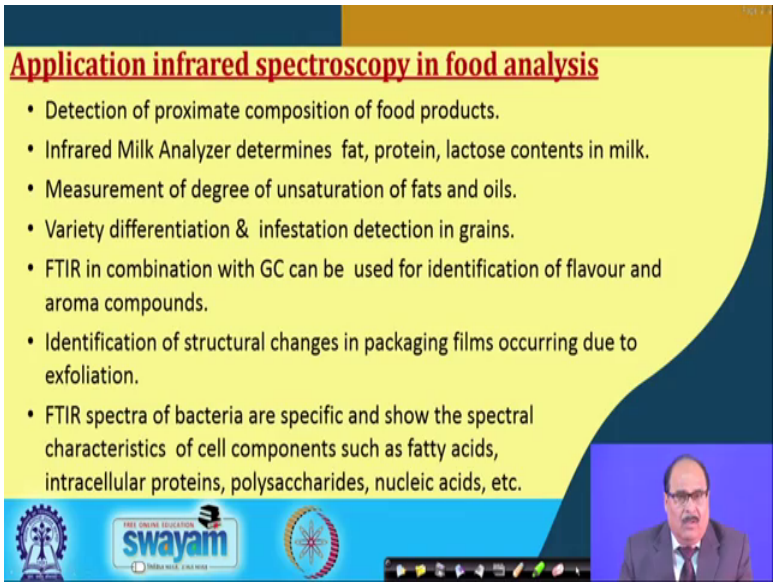

So, these were the basic principles in which this n NIR or FTIR systems are used. And as I told you in the beginning of the lecture that infrared spectroscopy has a wide ranging application input analysis. It can be used for detection of proximate composition of food products quick and rapid detection of proximate composition of food products without any even the sensors are touch proof sensors etcetera, and IR sensor IR sensor are there where even the it can be used to detect the quality and quantity of the food value and nutrients present inside food which is packed. So, even touch proof sensors can be used, so completely non-destructive method.

And there are several types of FTIR system or FTNIR systems are available, which are now many food processing industries they use these systems for analysis. Even this infrared moisture meters are available infrared milk analyzers are available which determines fat, protein, lactose content in milk. This can be used for measurement of degree of unsaturation of fats and oils. Even this technology can be used for finding the varietal differentiation to differentiate one variety of grain from the other variety are to detect the infestation in the cereal grains or in food grains.

FTIR in combination with GC can be used for the identification of flavor and aroma compounds in different food materials. It can be used for identification of structural changes in packaging films which might occur due to its a exfoliation. FTIR spectra bacteria are specific very specific, and they show the spectral characteristics of cell

components of the bacteria. So, the bacterial cell components like fatty acid, intracellular proteins, polysaccharides, nucleic acid etcetera can be evaluated or analyzed or qualified using spectroscopic techniques.

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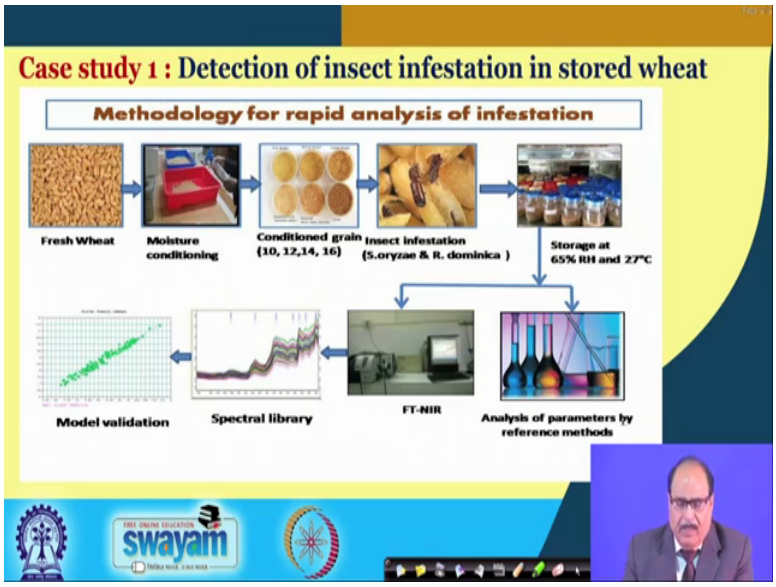

So, now I will give you a case study which is a depend based on the a study worked in my laboratory. We have used this FTNIR method. And even a standardized developed process for the evaluation at detection of insect infestation in stored wheat grain. So, the as far as the methodology is concerned actually that is in the earlier class also discussed little bit in this aspect, where we are studying hyper spectral imaging. So, in the similar manners, the infested grain samples are prepared that is the fresh wheat is taken; it is conditioned to different moisture content, because the moisture content is an important variable which influences the grain spoilage during storage or infestation etcetera.

So, the different moisture ranges were taken from 10 to 16 percent, 12 to 13 or 13 percent normally is considered a safe storage moisture, safe moisture content for a the storage of the grain. So, this little this side and other side the so 10 to 16 percent; it was conditioned. And then two molds S. orzyan and R. dominica where use to get the infested grain, they were required number or counted numbers of these insect of different life stages. They were infested or inoculated with the grain. And then this inoculated grain inoculated with these fungi or bolts was stored under specific conditions at of in incubators at 27 degree Celsius and 65 percent relative humidity.

So, means that is using this procedures, samples were prepared that is a grain samples with known infestation because we were in the process of standardizing. So, in the online detection, these are you can take directly that is the sample from the field or from the factory or even from the storage godowns etcetera where the unknown because here it is the as I told you earlier that is the library creation is a important task. So, in this manner we normally that is the and the sample which we are taking it is by using a standard analytical techniques may be chemical method or microbiological method in case the microbiological.

In this case the microbiological methods, we analyze the sample for the creation of the library, and it is always better that is if you have a large number of sample, you have large number of sample this, it will give a better reproducibility of the data. So, the samples with known characteristics are taken, they are they are spectra generated, this spectra that is the spectral library is created means that is the large number spectra is processed, and the library is standardized. So, when you get given unknown sample like in this way, we have developed a spectral library for wheat infestation.

So, any grain, if it comes when it is given to FTNIR, it will generate a spectra of that particular grain even single spectra, and it will process and compare it with this its spectral library which were generated earlier. So, with the comparison, it can tell the system software can tell that a whether the grain is infested or not. So, this spectral library generation, and then finally, model validation.

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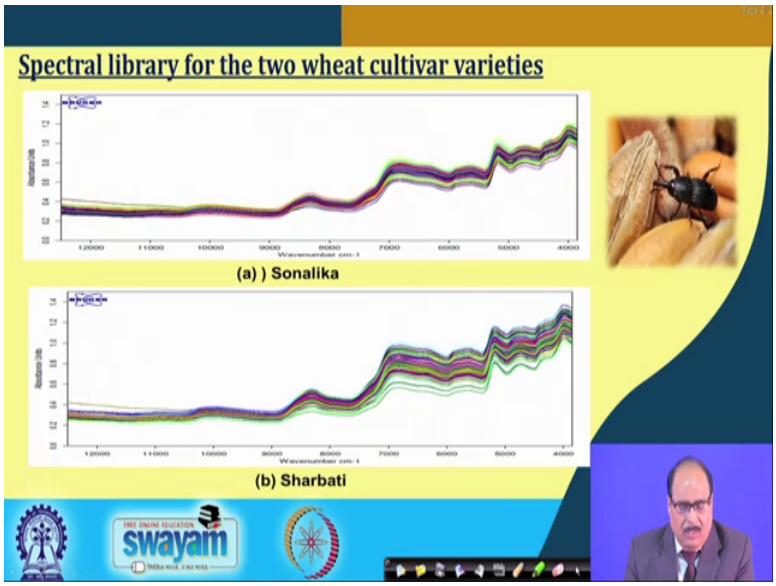

So, these are the a spectra, a spectral library which were generated in my laboratory you on Sonalika wheat variety and Sharbati wheat variety. You can see that there are differences can be seen in the nature of the spectra and some there wheat and peaks etcetera which are obtained.

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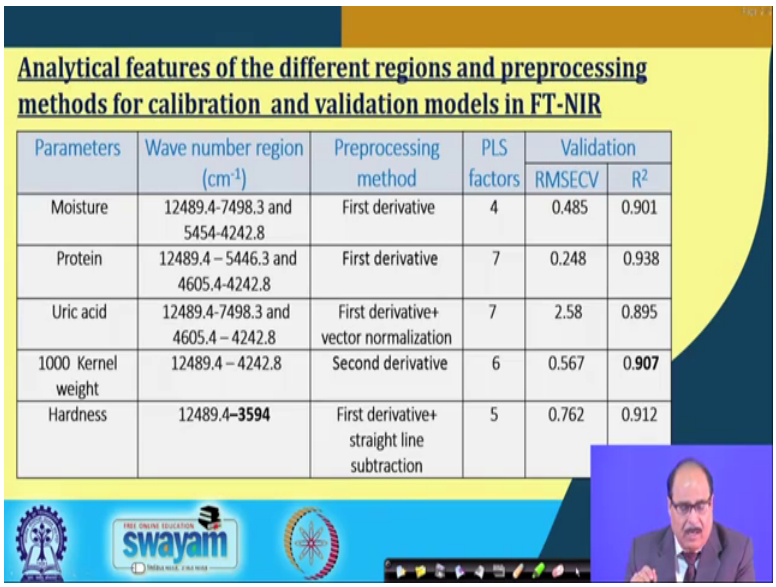

Parameters	Wave number region (cm^{-1})	Preprocessing method	PLS factors	Validation	
				RMSECV	R^2
Moisture	12489.4-7498.3 and 5454-4242.8	First derivative	4	0.485	0.901
Protein	12489.4 – 5446.3 and 4605.4-4242.8	First derivative	7	0.248	0.938
Uric acid	12489.4-7498.3 and 4605.4 – 4242.8	First derivative+ vector normalization	7	2.58	0.895
1000 Kernel weight	12489.4 – 4242.8	Second derivative	6	0.567	**0.907**
Hardness	12489.4**–3594**	First derivative+ straight line subtraction	5	0.762	0.912

Then these spectra, they are analyzed using different software are chemo metric methods analytical features of the different regions and preprocessing methods for calibration and validation of the model which were developed like moisture, protein, uric acid, 1000

kernel weight and hardness. These are some of the response parameter quality value on the basis of which the samples are compared. So, these are whether that is the all these values and the wave numbers respective wave numbers are given in this. Like for example, moisture it can be processed or occurred in the wave number range of 12489.4 to 7489.3, it and then maximum and minimum that is. So, in this ranges regions that is the moisture.

So, it is preprocessing method was used first derivative methods, PLS factors were 4; and the root mean square error for class validation was discussed 0.485. So, this in fact, RMSECV value should be lower; R square value should be higher, which indicates that the model is best speed and data is good, it has a good prediction.

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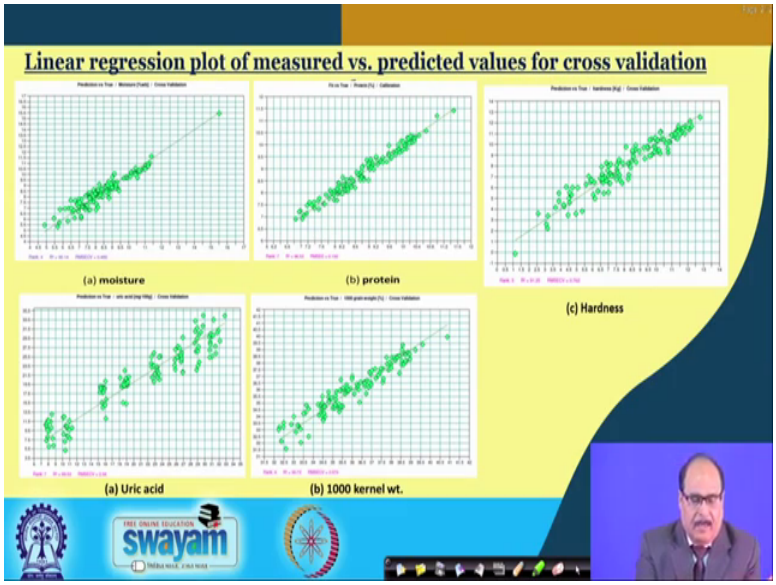

So, these are the linear regression plots which are obtained using the software or the measured verses predicted values for cross validation and the (Refer Time: 22:03) for different component like moisture, protein, hardness, uric acid, and 1000 kernel weight. So, we can see here that is if that this line if the all the predicted values as well as measured value, if they are closely the all the data fall on the line, it good gives a good prediction.

So, like in this case you can say that is moisture, protein, this production is much better, better production, the model which have been developed by this NIR system. They are predictive better rather here there is a scattered in the case of hardness, some scattering

in the initial level there is more or even in the uric acid, you can see the data are little more scattered. So, with this we compare that validate the results, and in our studies we have got good validation using standard techniques that with the predicted values even we feed with the or compare with the experimentally analyzed value and then determine ok.

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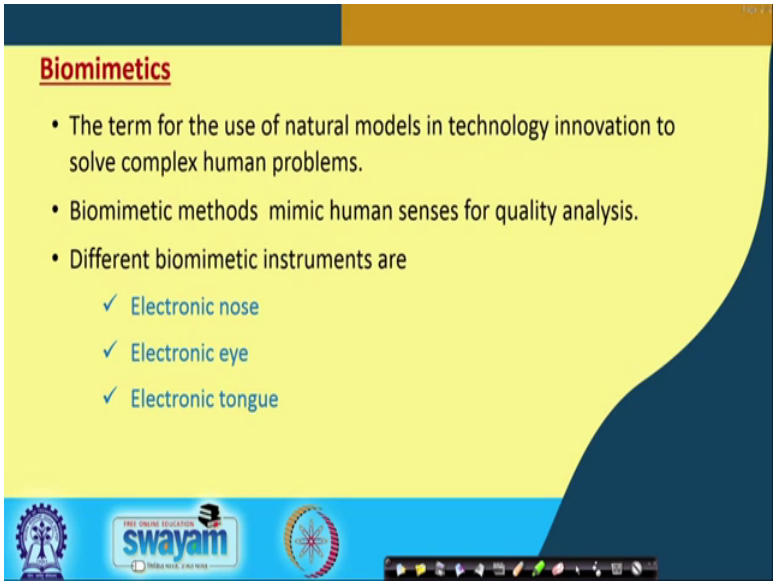

So, after this, the other method that is we studied that they have found out, the detail studied the details of the FTNIR or FTIR methods. Then another important methods, which can be used is in this regard for a quick testing a various food components which also worse on the similar principle, somewhat similar principle is the biomimetics that is this particularly is used for that is the attributes which are use human sense organs are used to analyze that an attributes of the food material.

So, those attributes they can also be used by the analyze or found out by using some instruments. So, biomimetics the term for the use of natural models in technology innovation to solve complex human problems biomimetics methods mimic human senses for quality analysis. So, different biomimetics instrument at different instruments which are available, they are like electronic nose, electronic eye or electronic tongue, because these three that is among the five sense organs. These three are the more commonly used sense organs that is nose, tongue and eye, these are the which evaluate the quality of the different food materials. So, accordingly these systems are available now.

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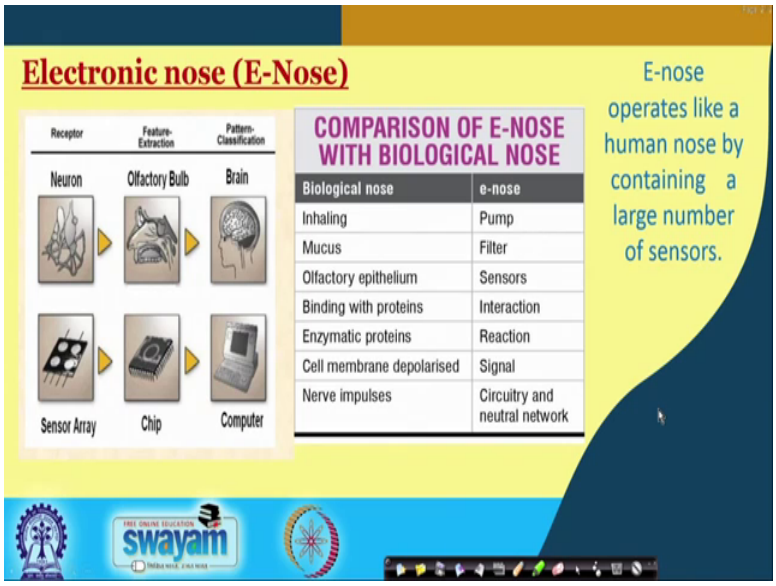

So, electronic nose, again that I tell which we have worked on something, so just this slide this gives a comparison between electronic nose. And this human sense organ that is like in the human organs, there are neurons, olfactory bulb, and the brain. So, in this e-nose system, the brain is the computer, olfactory bulb, these are the storage chips etcetera which are used in the system. And then these neurons, they are different sensor arrays which collect the data which take the information above. So, here further like inhaling for that purpose in the e-nose, the pump is given, this for olfactory epithelium the sensors are there; cell membrane, depolarized, so this signals are sent to the. So, it is in fact the

e-nose electronic nose, it works in the similar manner of course, it mimics our human sense organ system.

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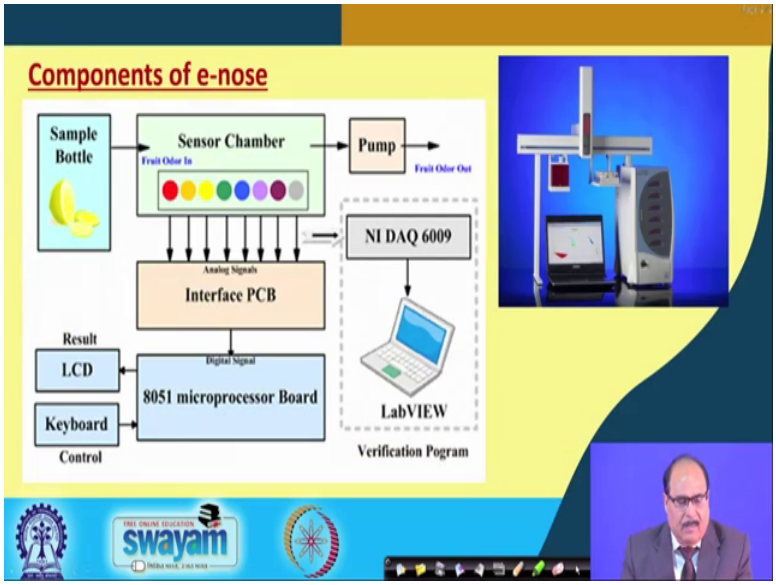

There is the components of e-nose system this is the picture you can see of the e-nose system which we have in our laboratory. And this basically it contains that is a sensor chamber where different sensors are there. Then the some sample preparation assembly that is the sample holder. So, different samples are prepared that is a and this sample there are some auto samplers which takes the volatile sample in the volatile forms and puts are into the that is the sample (Refer Time: 26:30) this sensors. They sense sample holder sample sensor and the finally these data which is recorded by this sensor is sent to the computer or which is processed and it is quantified or it is identified.

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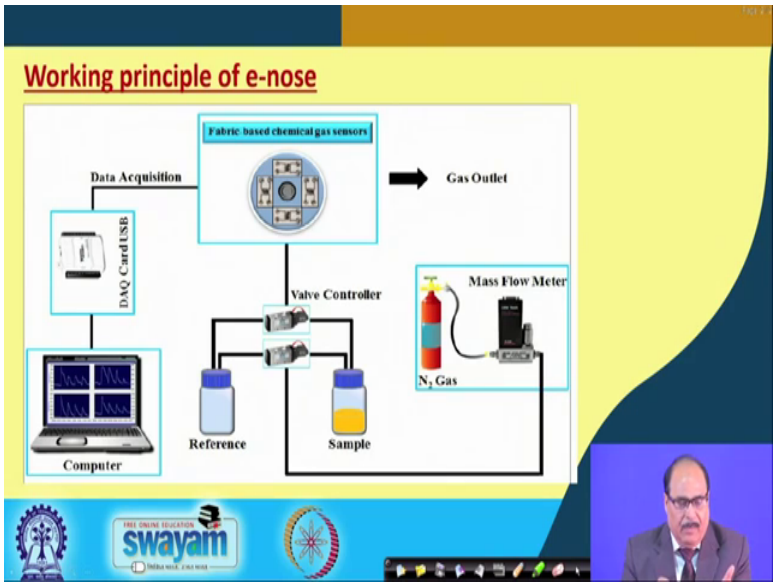

So, accordingly the working principle in the same manner as I told you that is the there is a some nitrogen or some other gas, whether the samples broken or evaporated, then the samples components in the form of vapors are taken. And these vapors are sensed by that volatiles, they are sensed by the different sensors and the data is sent to the computer.

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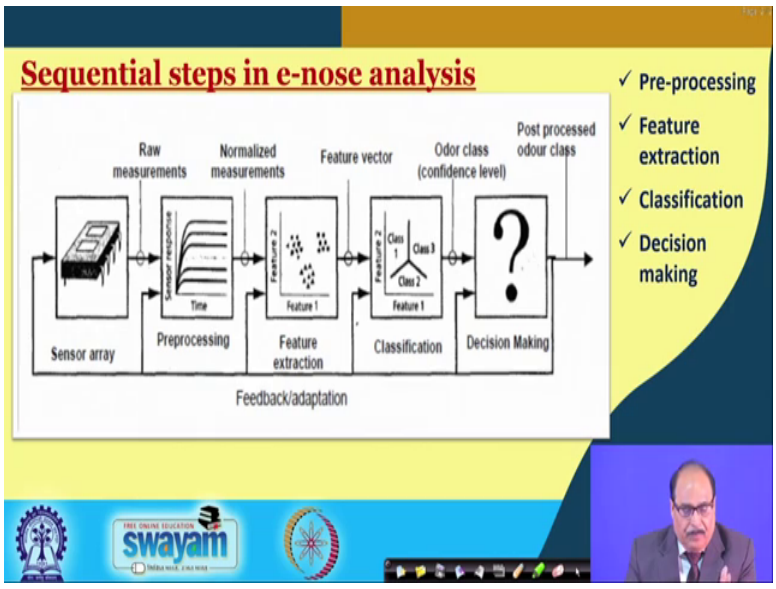

So, these sequential steps in the e-nose data analysis are the pre-processing that is first is the sensor array which collects data, then it is pre-processed or normalized, then feature

extraction, finally classification and decision making. So, these are the four major steps in the e-nose analysis.

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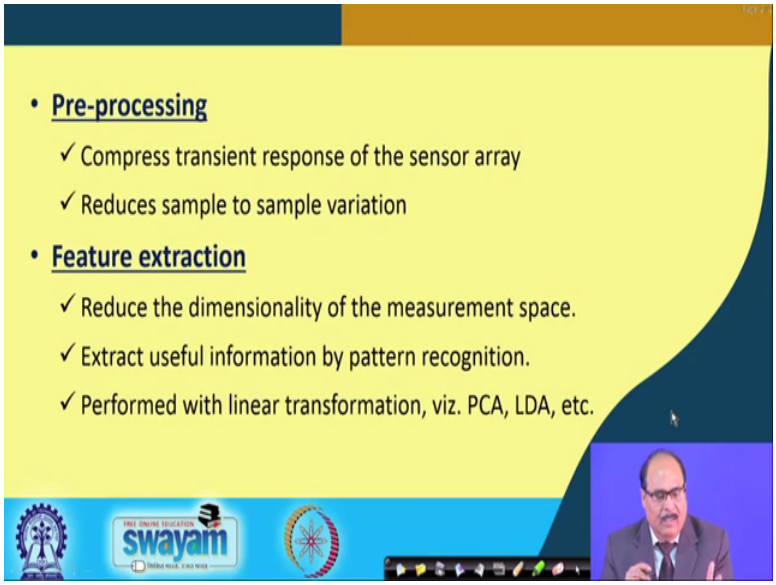

In the pre-processing as you have seen the earlier case it compress transient response of the sensor array; reduces the sample to sample variation. Feature extraction, reduce the dimensionality of the measurement space; extract useful information by pattern recognition; it is performed with the linear transformation, so which PCA, LDA etcetera.

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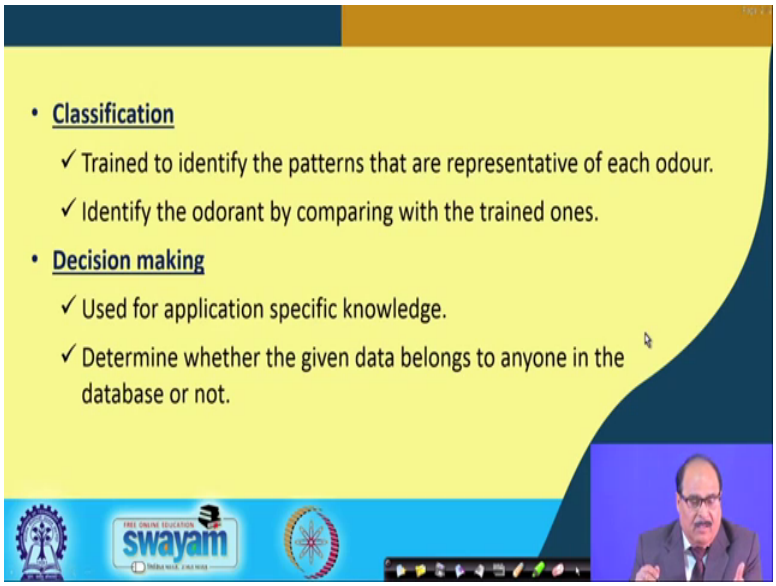

In the classification, it is done trained to identify the pattern that are representative of each odor that is an equipment is trained. And then these the odor is identified by comparing with the trained one. Finally, the decision making which used for application is specific knowledge, determined whether the given data belongs to anyone in the data base or not ok.

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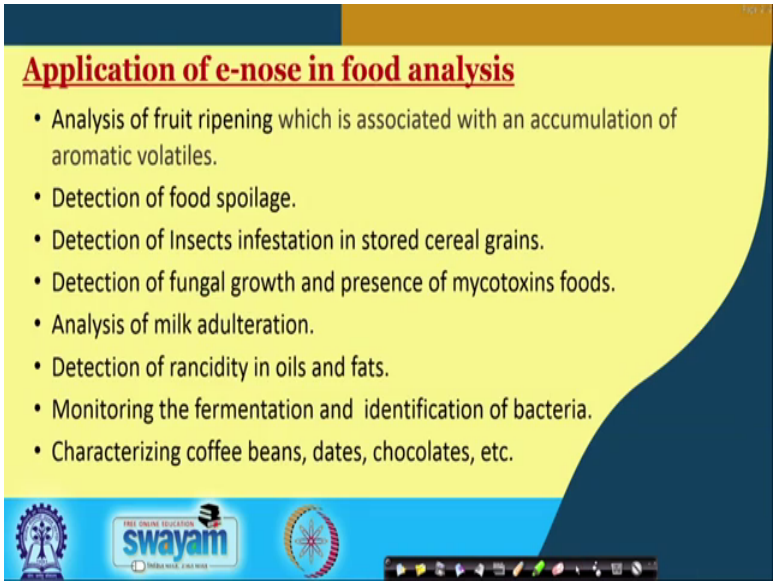

So, this also like FTIR, FTNIR this e-nose also has lot of application in all most detection of food spoilage, analysis of food ripening, fungal growth and presence of mycotoxin, analysis of milk adulteration, detection of rancidity in fats and oil, monitoring fermentation and identification of bacteria, or characterizing the different materials like on the basis of their flavor like coffee, beans, dates, chocolates, etcetera.

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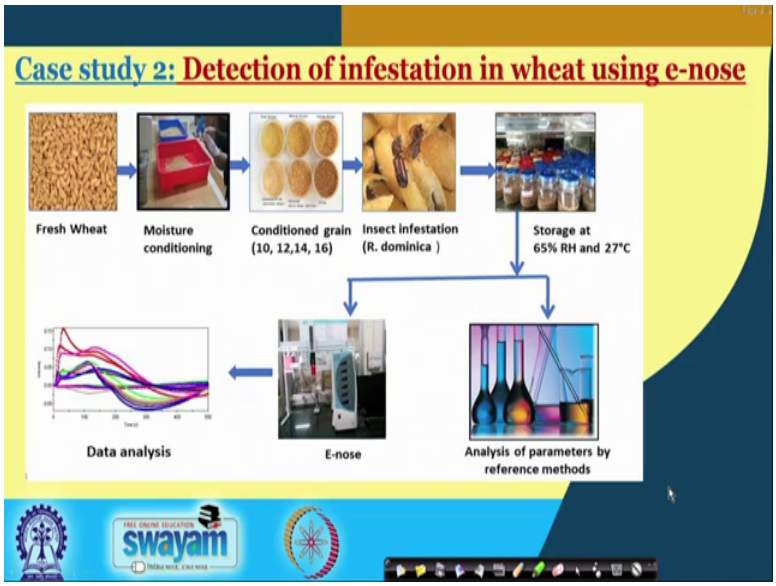

So, I will again case study 2, the detection of infestation in wheat using electronic nose. So, the sample is prepared in the similar manner. It is also so you have the sample with known characteristics. It is a given this samples that is the that is to sensors that sensors sense data, and sent it to the for that is a spectra which is generated. It is processed in the similar manner.

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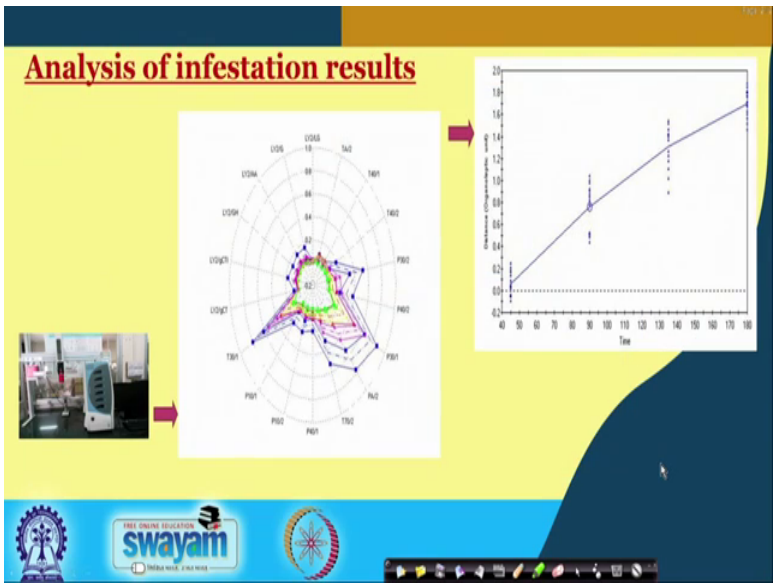

In this slide that it is the e-nose which we have the sensor data. Then it gives the radar charge there are e-nose which we have; it has 18 metal oxide sensors. So, these sensors

they sense the data and it is in the radar curve. And then finally, the third stage it is the data is processed.

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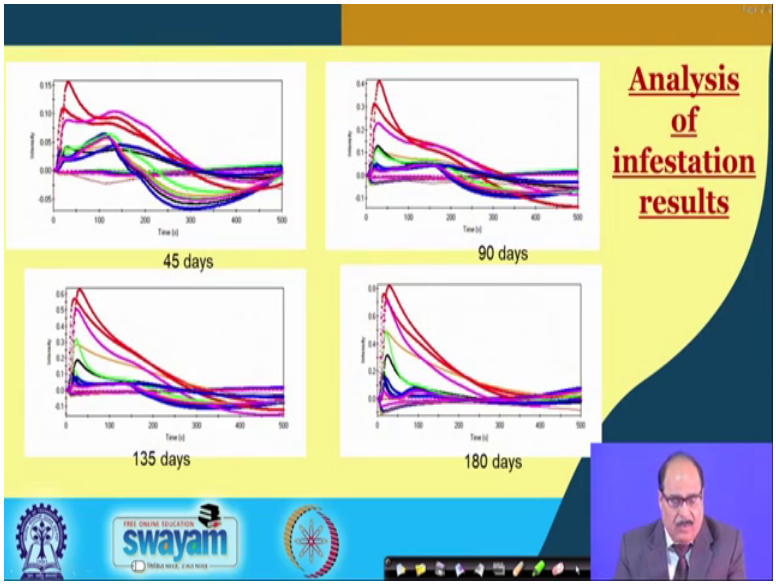

So, you can say the spectra here, different spectra finally, that is the of infestation results, infestation sample of 45 days, 90 days, 135 days, 180 days wheat rate and you can see there is a clear cut difference in the different spectra.

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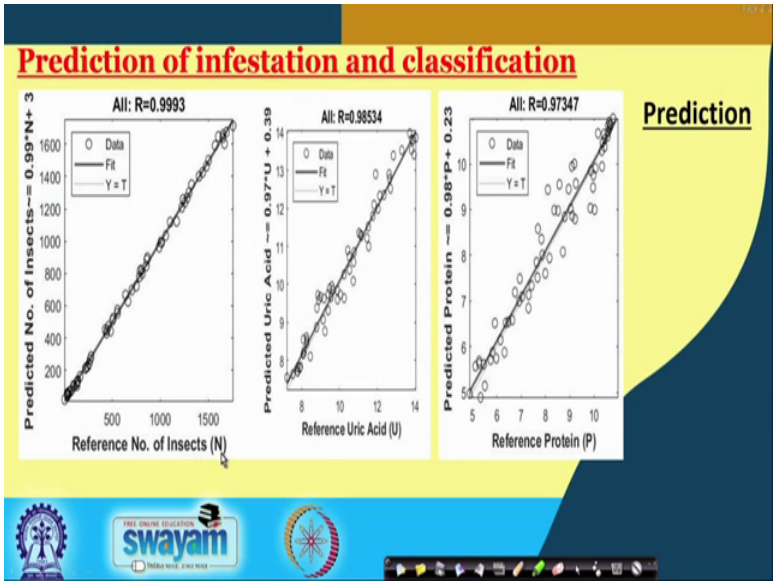

So, which is further quantified analyzed like the regression model or other prediction models by using in built software in the system, they are found out. And then here it is

the for the prediction of this reference number that is insect and then predicted number of insects. So, reference and prediction, the data like it is the for inspection and classification of the data which is used.

So, in the prediction again like in the real earlier case, here also you see if all the predicted values where, where as well as experimentally is determined value, if the almost form fall on this line, they are close. So, it shows that they are fit that is better fit the prediction is good. In this case, this the number of insects prediction is much better than the prediction in the case of proteins because in this case the data are points are scattered.

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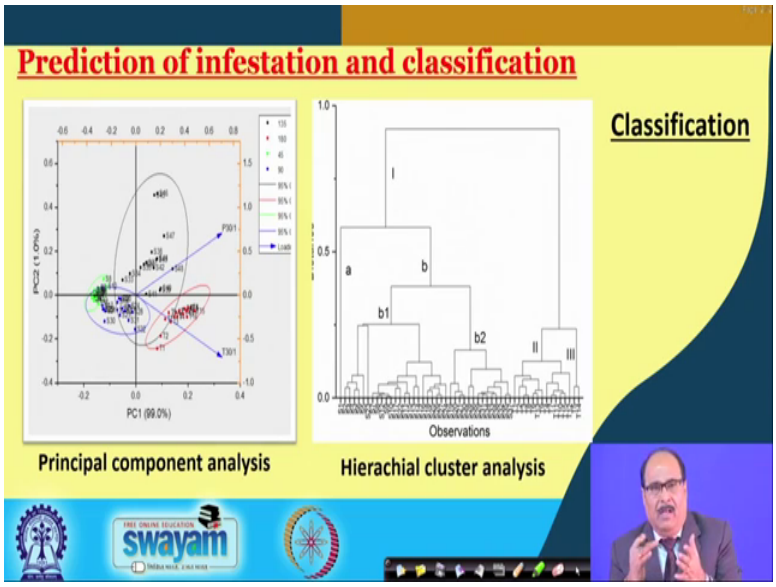

So, after the prediction, the next step is the classification of the data or the classification of the data or the classification of the data like software's are chemo metric like principle component analysis or high hierarchical cluster analysis can be used, either depending upon the sample depending upon the type of response one. Like for example, if you want to see that whether it is a spoiled fruit or a spoiled mango, so obviously, in which in this here that is the good mango or ripened mango, good quality mango can be first given to this e-nose to sense, the attributes or flavor of good mango. Then spoiled mango you give the sensors it analyzed and to the thereby the PCA the.

So, here it gives that is the bad samples are classified or they are. So, analysis can be done on the basis of any principle components, any components. So, samples are

accordingly made into cluster or made classified. So, this helps in the deciding that is to decide the what is the level of infestation, and to quantified like here in the hierarchical cluster analysis you can see that is the on the x-axis that infested grain, least infested samples, medium infestation sample, highly infested samples, they are clustered in different groups.

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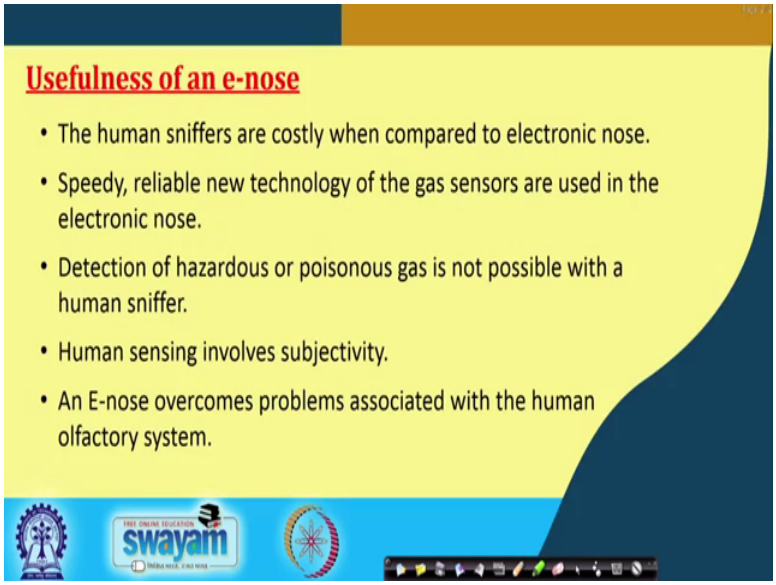

So, this is done. Obviously, the e-nose it is a very very good or useful technology, because the human sniffers are costly when compared to electronic nose. Many a times there is a human senses, they are the subject to, and the there might be several variations in the data found out by sensory analysis etcetera.

So, this the problems even sometimes the detection of hazardous or poisonous gas which sometime becomes toxic substances toxic flavors fumes becomes difficult which can be easily found out detected by this electronic instrument or electronic nose. So, the an e-nose overcomes the problems associated with the human olfactory systems. And it makes a it is a speedy reliable new technology all right where the gas sensors are used generally the metal oxide sensors or such other sensors are used to mimic the human sense organs or human nose, and give the reliable and good quality data.

So, these NIR technology is a near infrared or infrared spectroscopy or e-nose technology. They are good rapid and non-destructive methods or determination of food

quality. And apart from this, there are some other methods, but they work on the similar principles. So, one can work and do it this.

Thank you very much for your patience hearing.