Soil Science and Technology
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Lecture - 54
VisNIR - DRS Applications for Soil

Welcome friends to this lecture 4 of week 11 of Soil Science and Technology.

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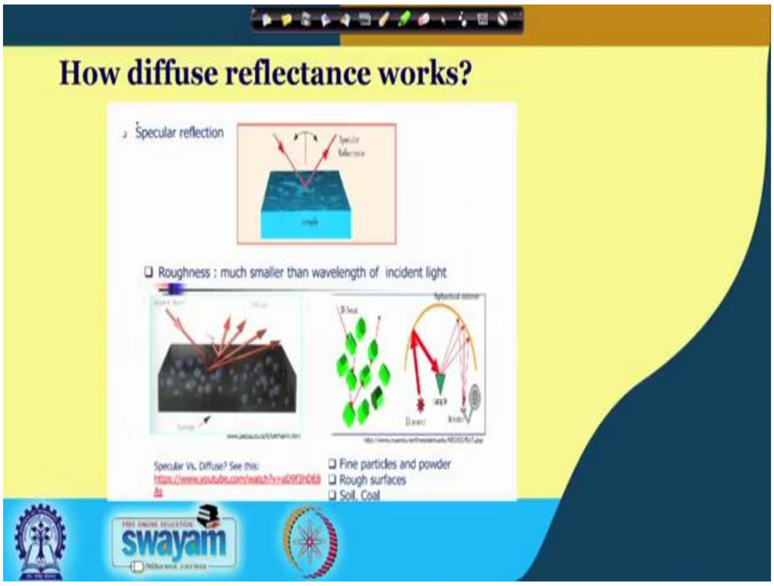

How diffuse reflectance works?

- Specular reflection

☐ Roughness : much smaller than wavelength of incident light

Specular Vs. Diffuse? See this:

☐ Fine particles and powder
☐ Rough surfaces
☐ Soil, Coal

And we will be starting today's you know we will be starting this lecture by describing the basics of diffuse reflectance spectroscopy. In the last lecture was stopped here we talked about the difference between specular reflection as well as the you know diffused reflection.

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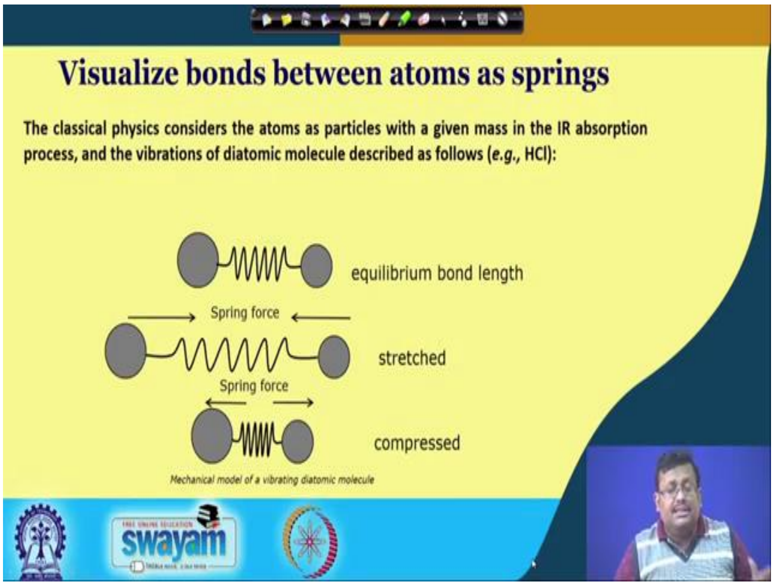

Visualize bonds between atoms as springs

The classical physics considers the atoms as particles with a given mass in the IR absorption process, and the vibrations of diatomic molecule described as follows (*e.g.*, HCl):

Mechanical model of a vibrating diatomic molecule

And let us talk about let us go ahead and see the basics of this near infrared spectroscopy.

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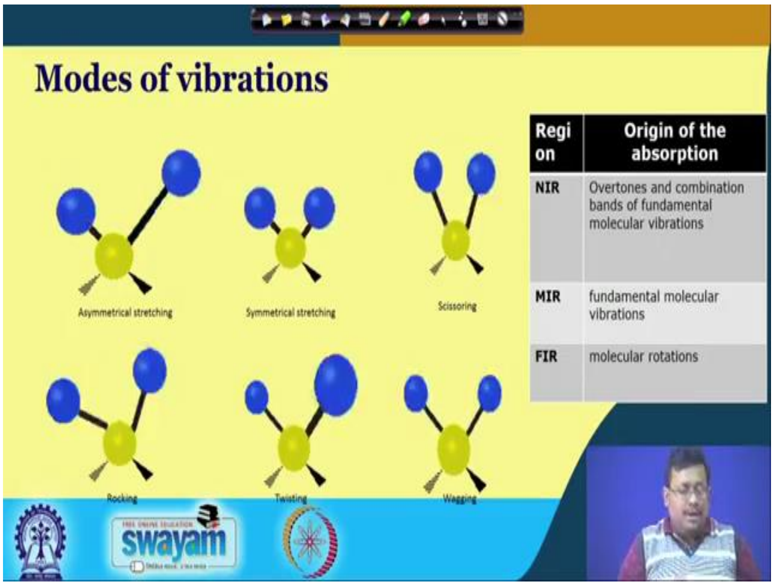

Modes of vibrations

Region	Origin of the absorption
NIR	Overtones and combination bands of fundamental molecular vibrations
MIR	fundamental molecular vibrations
FIR	molecular rotations

So, you know about now right now what is the difference between the diffuse reflection and specular reflection. We talked about the visible near infrared diffuse reflectance spectroscopy. Now, let us see what is the basics of near infrared spectroscopy. So, in classical physics considers the atom as particles with a given mass in the infrared absorption process and the vibration of diatomic molecules described as follows. For example, here it is diatomic molecules HCl and you can see the there are 3 types of you know forces you can see one is stretch and is compress it is a just like a spring.

So, it basically shows the mechanical model of a vibrating atomic molecule. So, when a molecule like this is absorbs some infrared radiation it shows 6 different types of stretching and bending. So, this 6 are these are the 6 different types of stretching and bending as you can see here asymmetrical stretching, symmetrical stretching, scissoring, rocking, twisting, wagging. So, as a result of that and you know as a result of that there is some special absorption and some spectral signatures. So, you know there are different region as you can see near infrared region, middle infrared region and far infrared region and the origin of the absorption in all these regions varies.

For example, in case of mid infrared region you will see that fundamental of molecular vibrations will you can see, in case of far infrared region you will see that molecular rotation it is responsible for molecular rotation. Or in other words in the far infrared region absorption will occur due to molecular rotation in the mid infrared region absorption will occur due to the fundamental molecular vibration. And in the near infrared region you will see over tones and combination bands of fundamental molecular vibrations. So, that is why you see these different types of spectral signatures.

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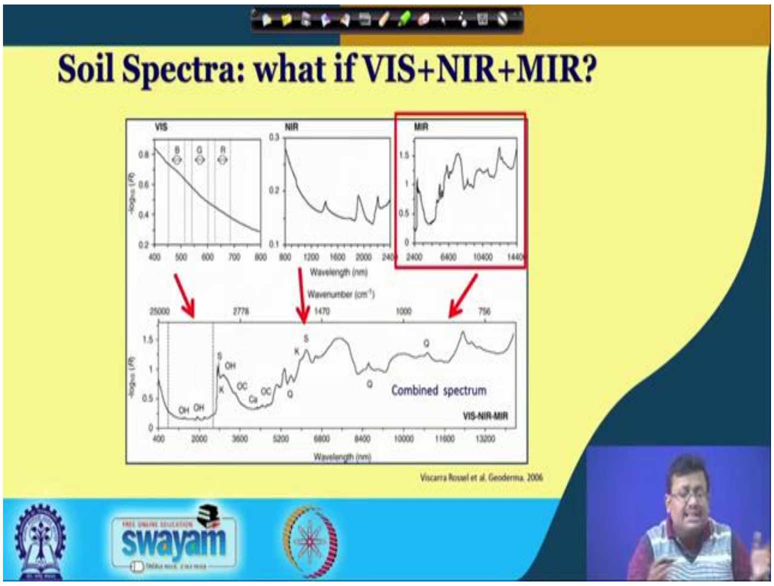

Soil Spectra: what if VIS+NIR+MIR?

Viscarra Rossel et al. Geoderma 2006

So, what are these spectral signatures? You see here the you know we have combined the soil absorption you know soil spectra of this three ranges. For example, this is the visible range, this is the near infrared range and this is the mid infrared range. Now, in the mid infrared range these are basically the fundamental absorption features which basically occurs from absorbing the near infrared rays by different organic molecules groups. And they produce different types of further you know also some overtones and combination bands.

And these are basically seen in this near infrared range. So, it is basically combined visible near infrared and mid infrared spectrum of soil. And you can see these absorption features and reflectance features and very well very very much you know we can see that. And these absorption basically shows you know basically reflects you know this absorption features basically or reflects the chemical composition of the soil itself. Because, this absorption actually happens from different I would say from different you know chemicals or organic chemicals which are present inside over at the in the soil.

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VisNIR-DRS equipment

- AgriSpec DRS (Analytical Spectral Devices, Inc.)
- PSM-3500 DRS (Spectral Evolutions)

So, let us see some equipments for VisNIR - DRS spectro radiometer. So, you can see there are two equipments: one is called AgriSpec DRS by analytical spectral devices of USA and this is spectral evolution 3500 DRS. So, it is actually PSR 3500. So, please correct it is PSR 3500 or so, these are two important instruments nowadays available for VisNIR - DRS measurement. So, let us go ahead and see what is the internal config of these two instruments.

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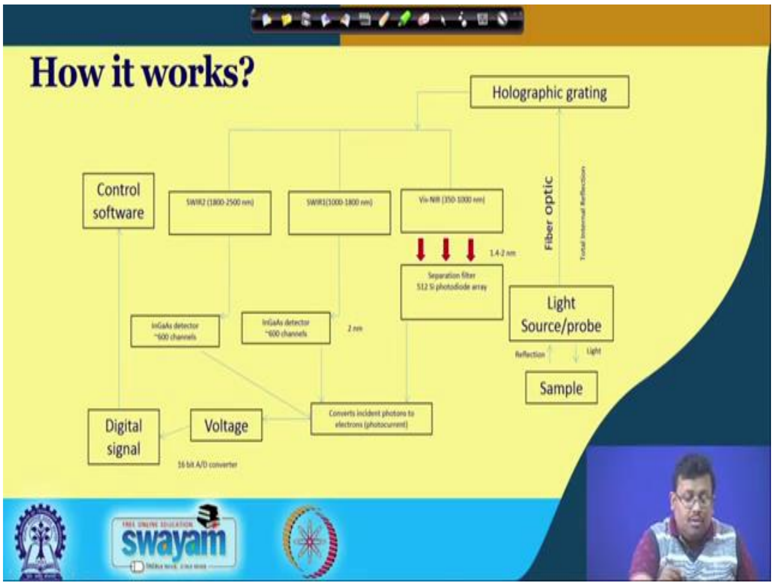

How it works?

So, let us see how it works. So, if you see the internal config of these instruments; obviously, this is sample this is starting point and obviously, it is an you know hand held probe which has a source. So, basically it is shinning halogen white light source and it is some amount of light getting reflected back from this surface, due to diffused reflection. And these reflected radiation goes to this fibre optic cable to a holographic grating which basically further divides these into 3 different regions.

One is called the visible to near infrared region which varies from 350 to 1000 nanometre. Then short wave infrared 1 region which varies from 1000 to 1800 nanometre region and then short wave infrared 2 that is 1800 to 2500 nanometre region. So, this VisNIR region basically then goes from this separation filter which contains 512 silicon photodiode array which has a you know which has 512 silicon photodiode array.

And this short wave infrared 1 is passed through this indium gallium arsenide detectors which has 600 channels. And this short wave infrared 2 also goes through this another detector that is in this detector with 600 channels. Ultimately, it converts incident photons to electrons or photo current and ultimately produce the voltages or digital signal and which is further measured by this control software. So, this is how this you know instrument basically works.

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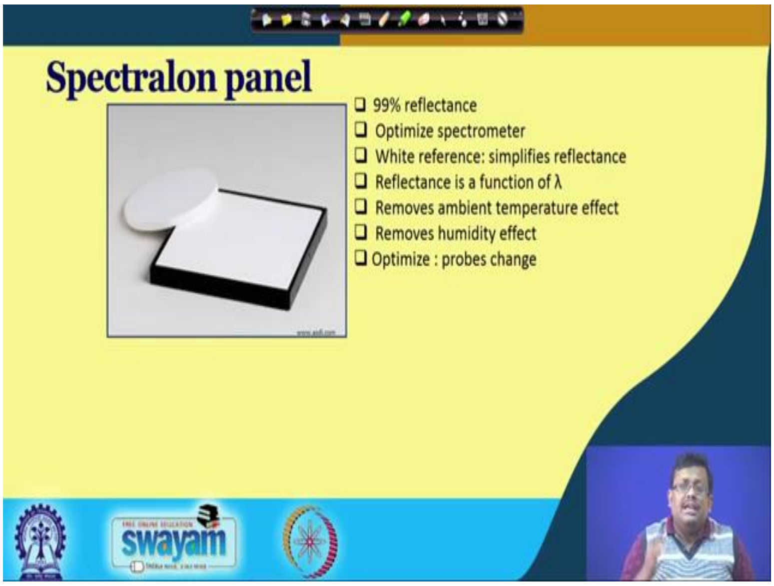

Spectralon panel

- ☐ 99% reflectance
- ☐ Optimize spectrometer
- ☐ White reference: simplifies reflectance
- ☐ Reflectance is a function of λ
- ☐ Removes ambient temperature effect
- ☐ Removes humidity effect
- ☐ Optimize : probes change

So, another important thing is that for each and every measurement we you know after couple of measurement we have to standardise this instrument. So, for standardizing this instrument we need a white reference panel and we call it spectralon panel and the spectralon panel we assume that it has got 99 percent reflectance. So, it basically requires for optimising the spectrometer and it requires for white referencing the spectrometer that is simplifies the reflectance. So, reflectance remember is a function of λ that is the wavelength and it removes the ambient temperature affect also.

And also removes the humidity effect and optimise when we change from one probe to another probe. So, there are different types of probe attachment, there is some handle probe, there is some mug light which you can which you can set over the desktop and then you can scan it from below scan a soil sample from below. So, that also can be done by this and when we change the probes you have to optimise the instrument by using this spectralon panel.

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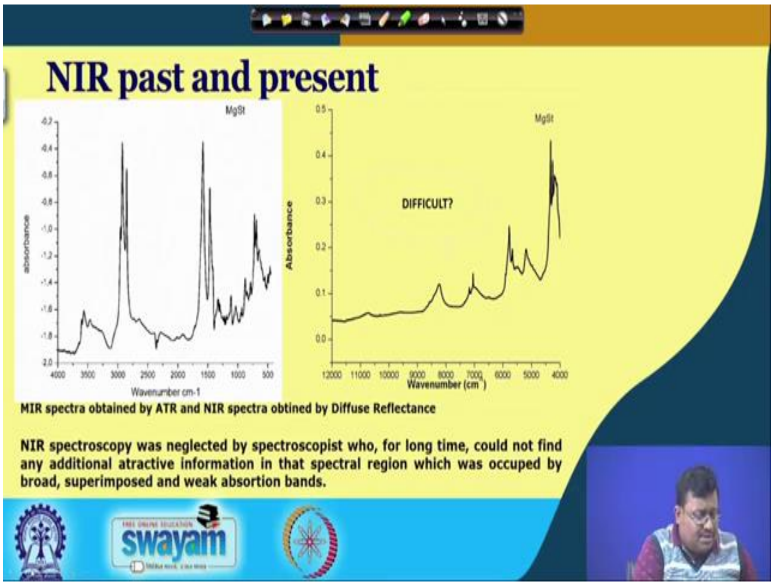

NIR past and present

MIR spectra obtained by ATR and NIR spectra obtined by Diffuse Reflectance

NIR spectroscopy was neglected by spectroscopist who, for long time, could not find any additional atractive information in that spectral region which was occuped by broad, superimposed and weak absorption bands.

So, let us see what is the difference between near infrared spectroscopy analysis in the past and now why it is getting importance. So, you see in the left it is basically the mid infrared spectra obtained by ATR and the right one is the near infrared spectra of obtained by diffuse reflectance. Thus, the difference which you can easily observe that the number of spectral features is more in case of mid infrared ray as compared to the near infrared region which contains some broad absorption features also.

So, NIR spectroscopy was neglected by spectroscopist who for long time could not find any additional attractive information. And in the spectral religion which was of you know which was occupied by broad superimposed and weak absorption bands. So, this is the reason that is why the NIR are you know based technologies were not so, popular previously.

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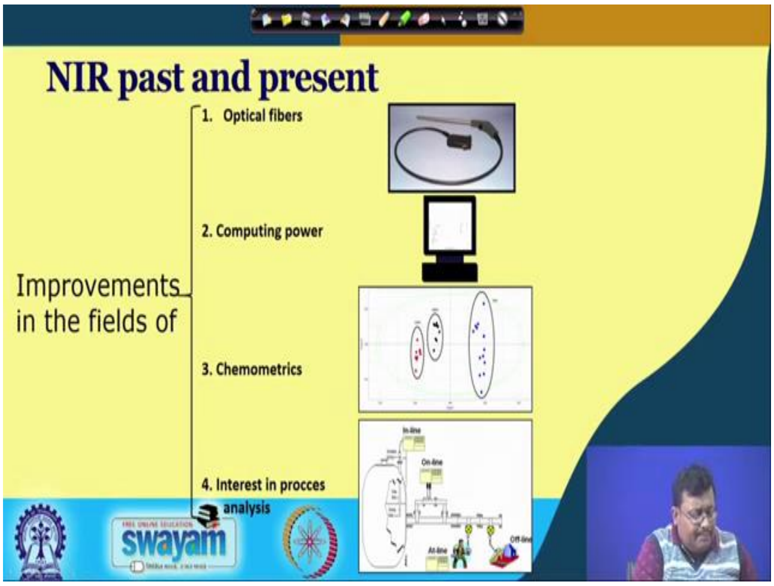

NIR past and present

Improvements in the fields of

1. Optical fibers
2. Computing power
3. Chemometrics
4. Interest in procces analysis

So, you can see it was difficult to interpret this broad absorption features. However, due to the absorb due to the advancement in these 3 different or 3 to 4 different forms you can see there is an advancement in the you know optical fibres. There is a advancement in computing power, there is an advancement in chemometrics, there is an advantage of interest in process analysis. So, as a result of these advancements now a days we can use this NIR spectrometer for better management and better prediction or better analysis of any particular soil property.

Now what is chemometric? Chemometric is a specialised data analysis where we measure the concentration of a particular element or chemical using some you know multivariate techniques or statistical means so, that is called chemometrics. So, there are several methods of chemometrics generally we use different types of machine learning algorithms for predicting as a chemometric tool like partial least square regression, then principle component regression and so on so forth.

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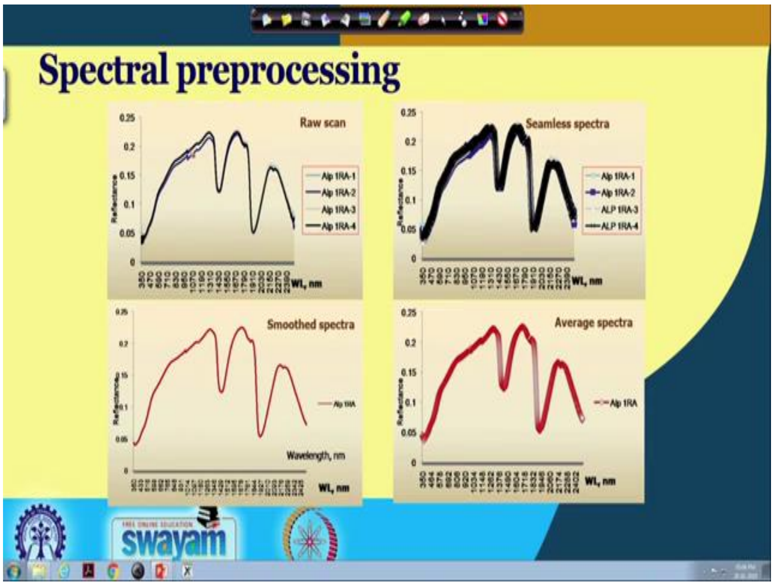

Spectral preprocessing

So, if we go ahead and see what this spectral features basically look like. So, you see here these are the spectral pre processing, this is actually the spectral signature of a raw soil sample. So, basically what we do we generally take 4 scan or 5 scan per sample by rotating the sample. Because, we want to take a homogenised measurement of the scan we or we want to take we want to nullify or we want to you know we want to offset the you know heterogeneity of the soil.

Because, you know soil is heterogeneous so, if we take several measurement and then make an average then it will produce us more homogeneous results or more average results. So, that is why you can see here we are taking 4 raw scan per a sample and after that we produce using some methods we produce different you know 1 average spectra.

And this is a typical reflectance spectra visible near infrared reflectance spectra of a soil which you can show source of some broad absorption features. Obviously, these broad absorption features are represented you know these absorption features are occur from the moisture or the water molecule which is present inside the soil.

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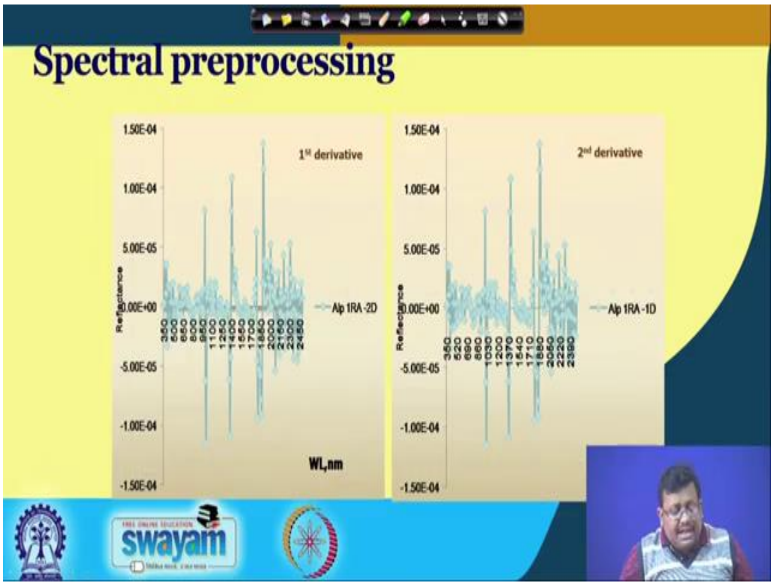

Spectral preprocessing

So, once we get this thing then we go for the spectral pre processing. Why? Because, you can see if we go through the last slide you can see these absorption features are quite broad and it is difficult to identify the small absorption features or very very specific absorption features which are responsible or which are basically absorb which are basically arising from absorption by different components which are present into the soil.

So, that is why we require different types of spectral pre processing. So, you can see here we are doing different types of spectral pre processing this is called these are some common spectral pre processing we call it first derivative of reflection spectra and this is called second derivative of reflectance spectra.

So, in the first derivative of reflectance spectra we generally take the first derivative of the reflectance values as you know base in comparison to the wavelength. And in the second derivative we are taking second derivative of the reflectance values in comparison to the wavelength. So, basically what are these? These are basically the reflectance values individual reflectance values which we have recorded for each of these wavelength starting from 350 to 2500 nanometre.

For each of these wavelengths we have basically recorded the we have basically recorded their reflectance values and when we are plotting these reflecting values you know these are called the spectrum.

So, this spectrum contains this broad absorption features and to remove this is or to further get higher resolution of the spectral features we are doing different types of spectral pre processing. Just you can see here we are doing a first derivative and second derivative.

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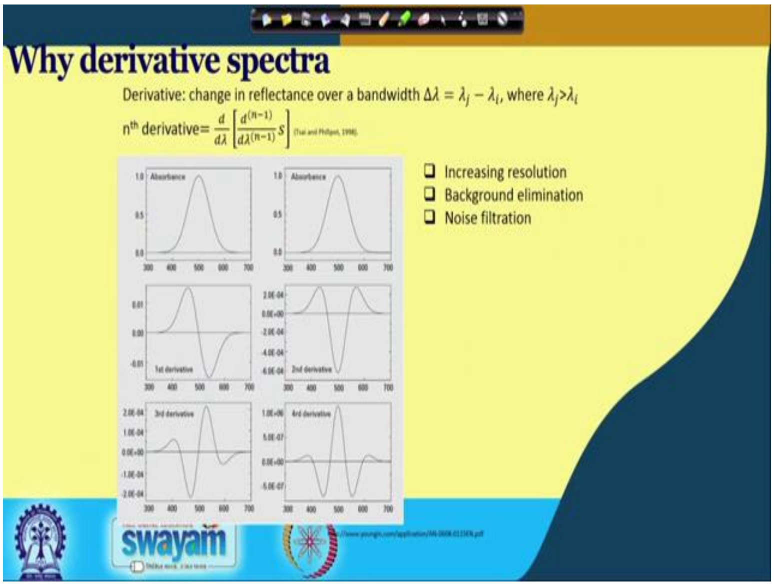

Why derivative spectra

Derivative: change in reflectance over a bandwidth $\Delta\lambda = \lambda_j - \lambda_i$, where $\lambda_j > \lambda_i$

$$n^{th}\text{ derivative} = \frac{d}{d\lambda}\left[\frac{d^{(n-1)}}{d\lambda^{(n-1)}} s\right]$$ (Tsai and Philpot, 1998)

- ☐ Increasing resolution
- ☐ Background elimination
- ☐ Noise filtration

Now the question comes why derivative spectrum? Obviously, the derivative is basically change in reflectance over a bandwidth which is denoted by δλ where, δλ is equal to (λj - λi) where, λj > λi. So, basically we take the nth derivative on the basis of this λ you know in comparison to the λ or reflectance values.

And you can this is an original reflectance feature and from this original reflectance feature which we are taking different types of derivative first derivative. This is obviously, this is the second derivative, this is third derivative and again further higher order derivative like fourth derivative. So, you can see different derivatives you are taking. So, what is the reason behind that?

Obviously, there are couple of reasons; first of all increasing resolution, then background elimination you can see there are several background features which we can eliminate through you know by taking the higher derivatives and also, we can take some noise filtration.

For example, when you are taking the spectral scan in the field there may be or in the lab there may be some ambient light source and from those ambient light they can come and they can you know they can create some interferences. So, this type of interferences and you know can be nullified by using some special prepossessing.

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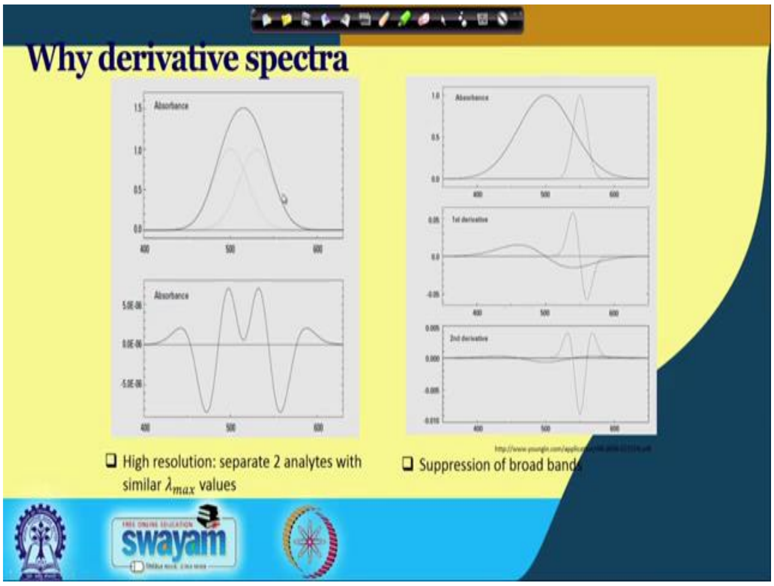

Why derivative spectra

❑ High resolution: separate 2 analytes with similar λ_{max} values

❑ Suppression of broad bands

So obviously, again also you can see higher resolution, this is a broad reflectance spectrum or absorbance spectrum which is basically you know of which is basically masking this two important spectral features. So, when you are going from this original absorbance to higher order derivatives we can see these 2 analytics 2 analytes can we separate with similar λ max values. So, by increasing the you know basically you can see how you are increasing the resolution to get more information about the more analytes which are present in the soil.

Similarly, you can see this broad absorption feature is suppressing this small absorption features. So, when we are going from original absorption to first derivative and second derivative how these are you know getting separated. And then how we are getting much more you know important information. So, these are some reason that is why we have why you use the you know spectral pre processing.

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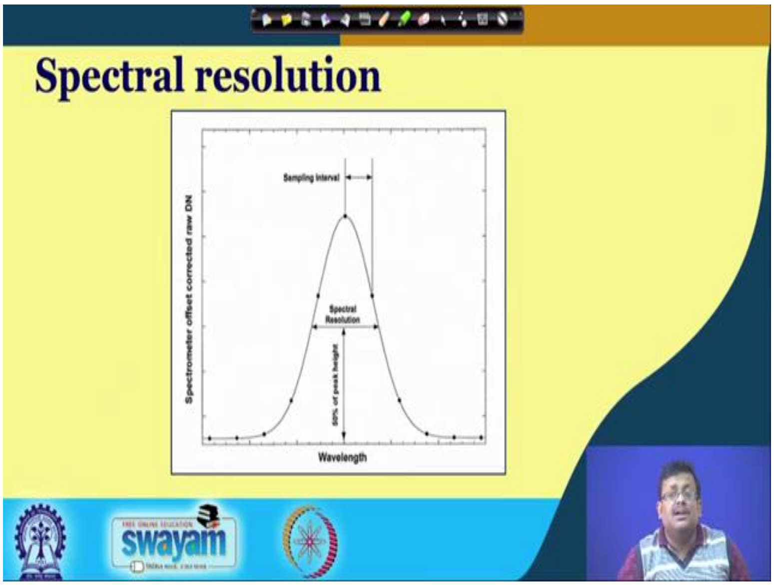

Spectral resolution

Spectral resolution is also very important, the spectral resolution suppose this is a peak spectral peak. So obviously, these difference is called the spectral resolution and this the 50 percent of the peak height. So, if this is the total peak height the 50 percent of the peak height we call it full width half maximum or full width stands for full width half of maximum. So, it is a half of maximum.

So, that shows the spectral resolution. Higher the resolution; that means, you can you know higher the resolution means lower the distance or in other words you can better resolute the minor spectral features using the spectra radiometer. And these consecutive points are basically shows the sampling interval. So, this is a representation of spectral resolution in the spectral curve.

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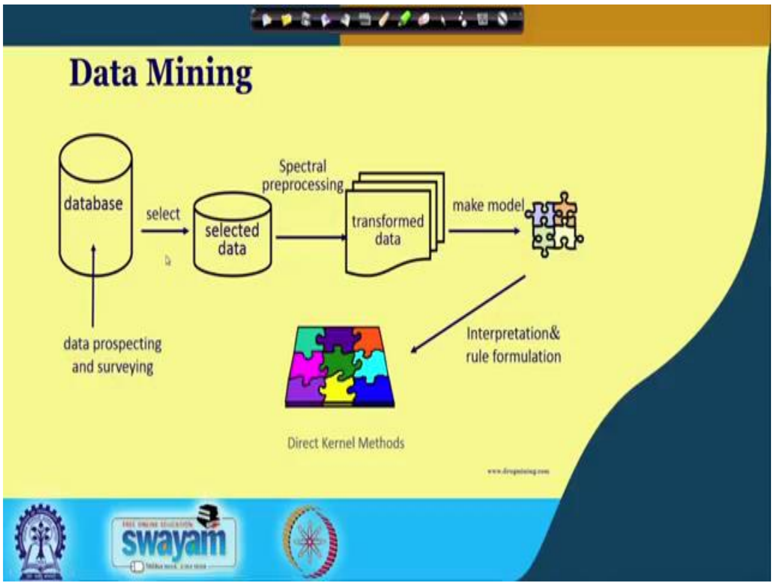
Data Mining

So, again once so, the idea is once we have gathered this database of spectra. For examples and we have thousands of samples and we can gather the thousands of spectra for those thousands of samples. And once we gather the thousands of samples spectra from these we will create a database and from this database we will do some spectral pre processing.

And from this spectral pre processing will be transformed the data and from using the transformed data we will create some predictive modelling. And from this predictive modelling we will do some interpretation and rule formation. And we will and that is why we use this method extensively now a days for predicting different soil properties. And we will see that in the next topics where, we discuss about the diffuse reflectance spectroscopy for application for soil.

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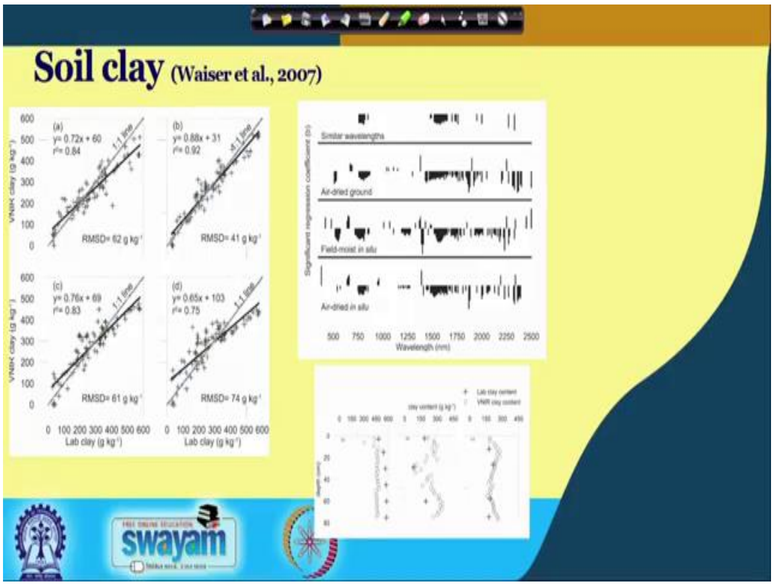

Soil clay (Waiser et al., 2007)

So, let us see some specific examples of diffuse reflectance spectroscopy. You can see here Waiser et al in 2007 they have used this diffuse reflectance spectroscopy and they have predicted the soil clay. So, you can use that spectra visible near infrared diffuse reflectance spectrometer for predicting the soil clay. And they have got an R^2 values of 0.92 varies 0.84 and 0.92, 0.75 and 0.83 for different conditions. Not only they got good results, but also they have identified what are the important wavelengths, which are responsible for producing these high you know prediction results.

So, you can see for different conditions like air dried ground condition, field moist in situ condition, air dried in situ condition; all these condition they have identified these important spectral wavelengths which are responsible for producing high you know which are responsible producing high accuracy. So, also by using their model they have seen the good correlation or good conformity between lap clay content as well as the visionary predicted clay content. So, you can see soil clay can be predicted by using this technique.

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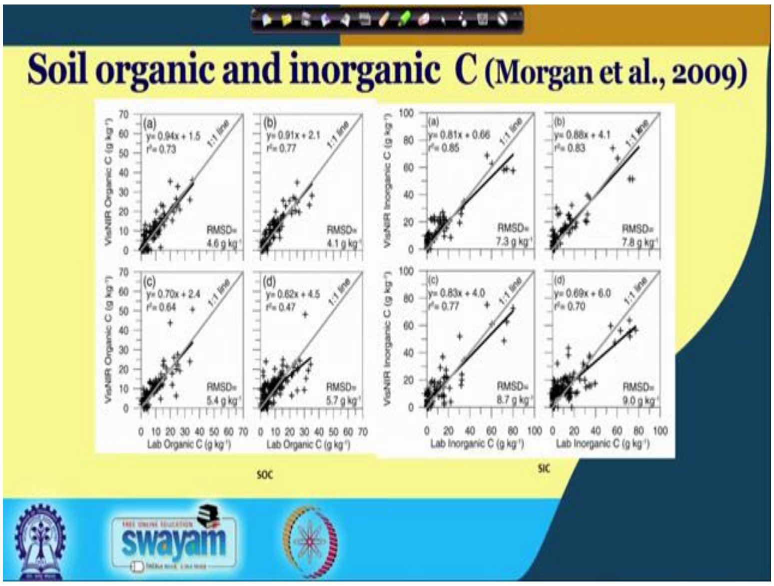

Soil organic and inorganic C (Morgan et al., 2009)

Another important aspect of soil is organic carbon and you know the inorganic carbon. And these scientist Morgan et al they have proved that you can use this visible near infrared spectroscopy, diffuse reflectance spectroscopy to produce the high you know to produce the; to produce the quantification. Or, you can predict both organic carbon and inorganic carbon you can see organic carbon can be predicted with an accuracy R^2 values of 0.77 that mean 77 percent accuracy.

And in case of organic carbon or solid organic carbon basically there you can predict it with an R^2 values of 0.85. So, that shows that this instrument can be used for predicting soil organic carbon and soil inorganic carbon without any consumables or without you know any you know chemical used.

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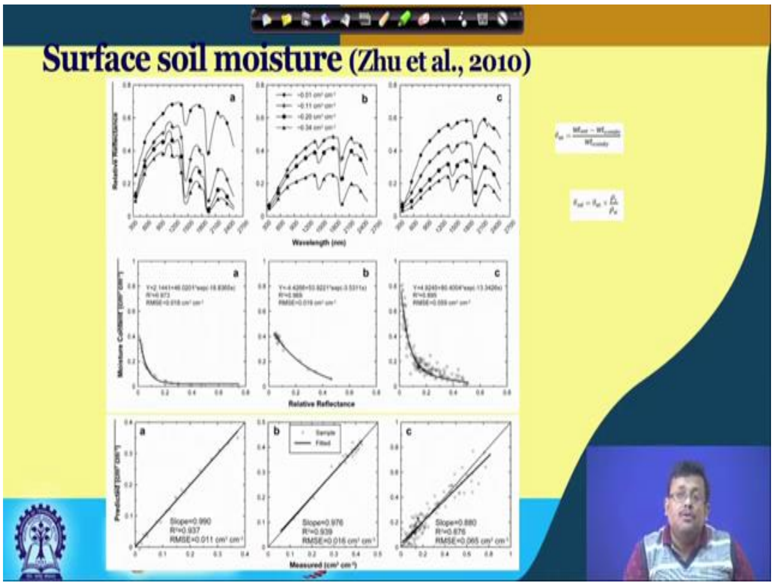

Surface soil moisture (Zhu et al., 2010)

Also you know Zhu et al in 2010 they have proved that you can use this technology for measuring surface soil moisture. Now, surface soil moisture is generally you can calculate using some gravimetric measurement, if this is an gravimetric measurement. And you can convert into volumetric content of water volumetric water content by multiplying with the bulk density ratio.

And you can see that this you know this water content can be predicted, the surface soil water content can be predicted by using the scan results from this diffuse reflectance spectroscopy.

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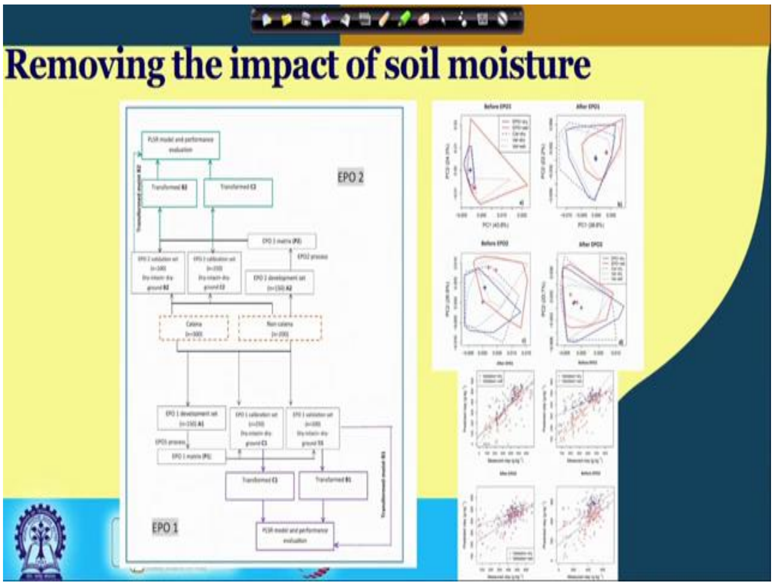

Removing the impact of soil moisture

So, also although it you know we have although the scientists have produced this high correlation between the moisture content and this diffuse reflectance spectra. These moisture content sometimes produce problem for producing higher prediction accuracy. Because, most of the time this moisture due to the absorbance of near infrared radiation by moisture these you know the features the spectral features which are you know the spectral features coming from other soil components are massed.

And as a result of that there are some separation and as a result of that there is reduction of accuracy of the modelling. So, scientist have used a new technique called EPO or External Parameter Orthogonalization technique for removing the effect of moisture. So, now a days you do not have to worry about the moisture content in the soil, you can directly take the scan in the field.

And you can use some mathematical techniques to remove the moisture impact by this EPO method or external parameter orthogonalization technique and by using this technique you can directly predict organic carbon as well as clay without any moisture influence. So, this is the advancement of research in this near infrared spectroscopy or visible and infrared diffuse reflectance spectroscopy.

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Indian applications of DRS: soil As

Dhapa Landfill, Kolkata

And also some Indian application I would like to show you, you can see here you know our group has used this technology for measurement of arsenic content in landfill side soil. So, we use this technology for measurement of soil arsenic concentration. So, we went to a landfill area where municipal land waste or municipal solid waste are dumped for a quite a long time. And around this landfill area there has been a huge amount of agriculture lands which grow the leafy vegetables which accumulate which has the potential for accumulating these heavy metals which are coming through this municipal solid waste.

So, we thought that we should use this DRS technique for predicting the arsenic concentrations. So, we went and collected several soil samples from there. As we can see from this is a study area and we call it the you know this is a landfill site called Dhapa landfill site situated in Kolkata city of India and these are water bodies. So, these are the sampling points.

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Scanning with DRS

So, we collected the samples and then we scan using these two techniques; one is the mid infrared spectrometer we call a drift or diffuse reflectance infrared fourier transform technique another is visible near infrared diffuse reflectance spectroscopy.

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Sequential extraction

And after scanning these using these two methods we also simultaneously measured their properties like you know total arsenic content. And also not only the total arsenic content but, also we used a specialised technique called sequential extraction to extract the individual pools of arsenic which is present in the soil.

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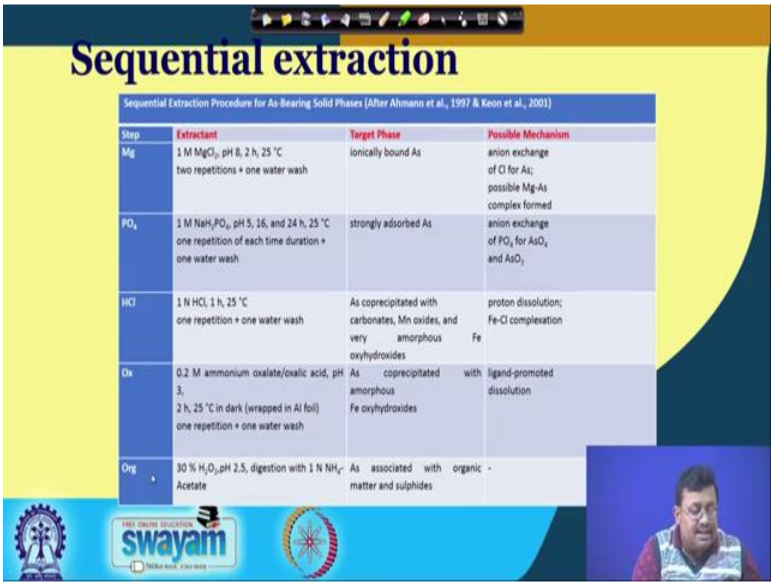

Sequential extraction

Sequential Extraction Procedure for As-Bearing Solid Phases (After Ahmann et al., 1997 & Keon et al., 2001)

Step	Extractant	Target Phase	Possible Mechanism
Mg	1 M $MgCl_2$, pH 8, 2 h, 25 °C two repetitions + one water wash	ionically bound As	anion exchange of Cl for As; possible Mg-As complex formed
PO_4	1 M NaH_2PO_4, pH 5, 16, and 24 h, 25 °C one repetition of each time duration + one water wash	strongly adsorbed As	anion exchange of PO_4 for AsO_4 and AsO_3
HCl	1 N HCl, 1 h, 25 °C one repetition + one water wash	As coprecipitated with carbonates, Mn oxides, and very amorphous Fe oxyhydroxides	proton dissolution; Fe-Cl complexation
Ox	0.2 M ammonium oxalate/oxalic acid, pH 3, 2 h, 25 °C in dark (wrapped in Al foil) one repetition + one water wash	As coprecipitated with amorphous Fe oxyhydroxides	ligand-promoted dissolution
Org	30 % H_2O_2,pH 2.5, digestion with 1 N NH_4-Acetate	As associated with organic matter and sulphides	-

So, you can see there are five different pools of arsenic which is present into the soil; one is called ionically bound arsenic, then strongly absorbed arsenic, then arsenic co precipitated with carbonates manganese oxide and vary amorphous iron oxyhydroxides. Arsenic co precipitated with amorphous iron oxy hydroxide, arsenic associated with organic matter and sulphides and we measured them individually.

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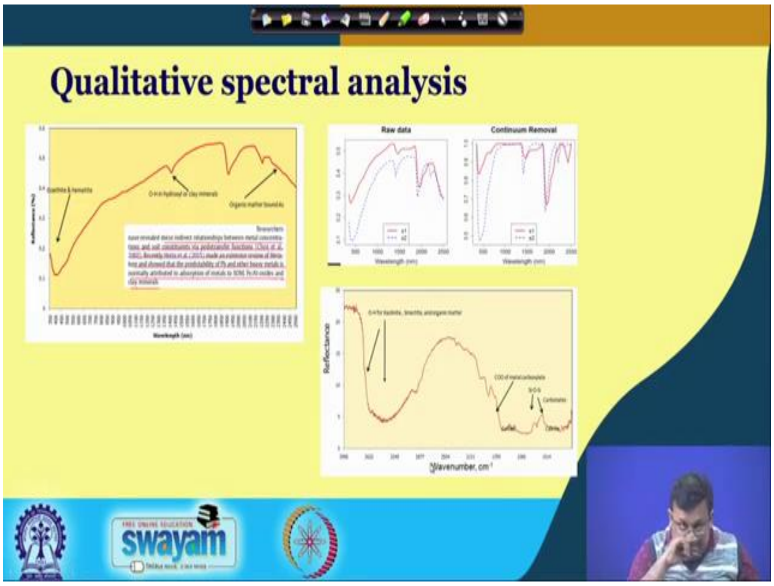

Qualitative spectral analysis

And then using some qualitative spectral analysis we tried to identify whether we can identify those individual features or individual pools. So, this is the actually the visible near infrared reflectance spectrum and this is a mid infrared reflection spectrum of the collected soil sample. So, you can see we have identified the goethite and hematite which are important iron based minerals.

And, then we can identify the hydroxyl of clay minerals and organic matter bound arsenic here. And also identified the kaolinites smectites in organic matter hydroxyl here and then other features like CW of metal carboxylates and then calcite and carbonates and all this things.

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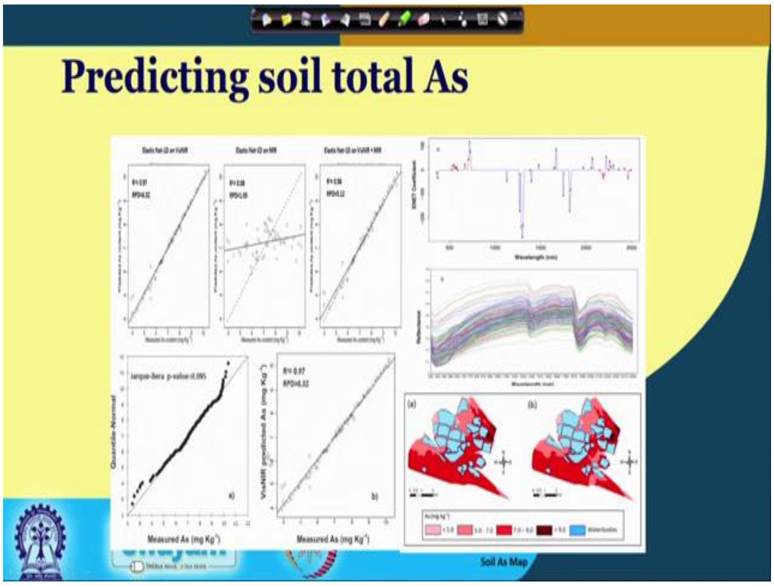

Predicting soil total As

Not only we have done the qualitative analysis, but also we predicted the total arsenic concentration and we got an very high R^2values of 0.97. And the beauty of it you can use this technology for directly mapping into the field also or directly producing the contamination map also. So, you can see using this model you can use the geostatistical interpolation which we covered in the last lecture.

So, geostatistical interpolation can be used to use this model predicted values to produce the arsenic contamination map. So, you can see we are getting arsenic contamination maps and for that area showing different levels of arsenic contamination starting from low to very high ok.

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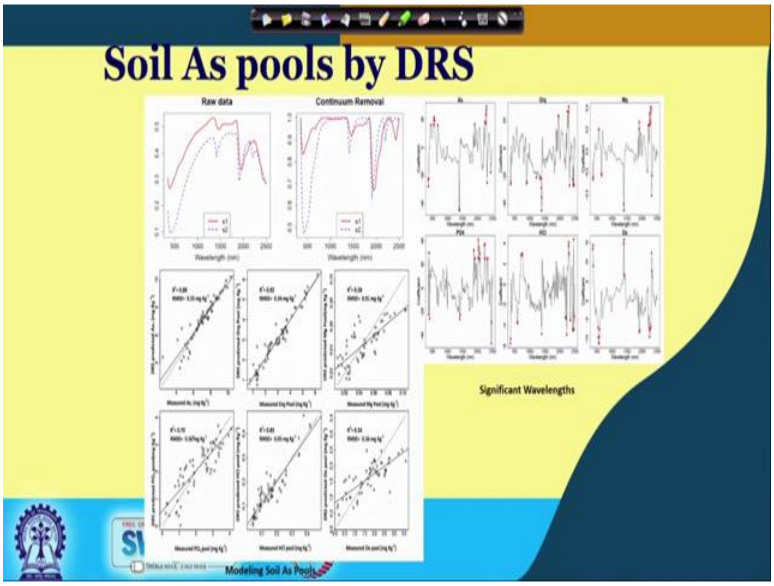

Soil As pools by DRS

Not only that we also produce this prediction model for individual soil arsenic pools and produce to that you can predict this individual soil arsenic pools by using this diffuse reflectance spectroscopy. And also we have identified the individual significant wavelengths by this diffuse reflectance spectroscopy.

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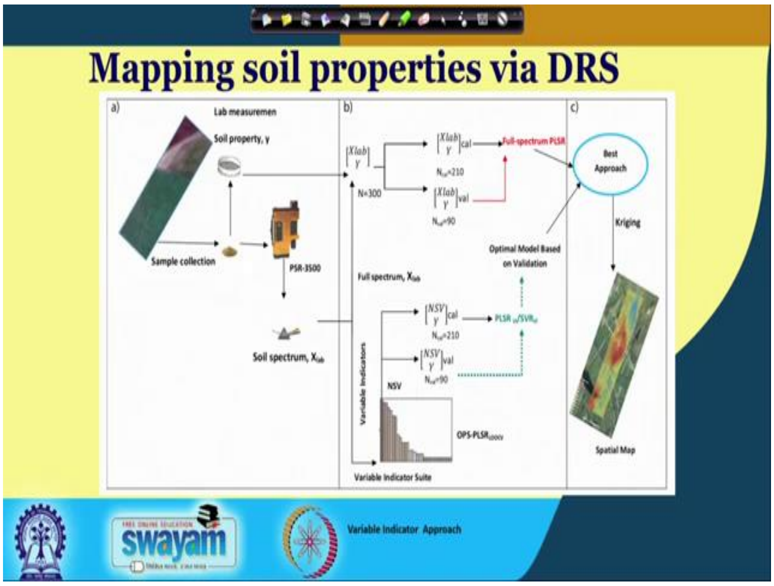

Mapping soil properties via DRS

Now, we are also produce the online mapping of soil properties or onsite mapping of soil properties by using some variable indicators. So, variable indicators uses some specialised variables between this 350 to 2500 nanometre. So, these 350 to 2000 nanometre shows the whole range of spectral variables or spectral wavelengths. Now, we are selecting some spectral wavelengths from this whole range of spectral wavelengths. And then we are efficiently utilising them in using some model to predict particular soil properties.

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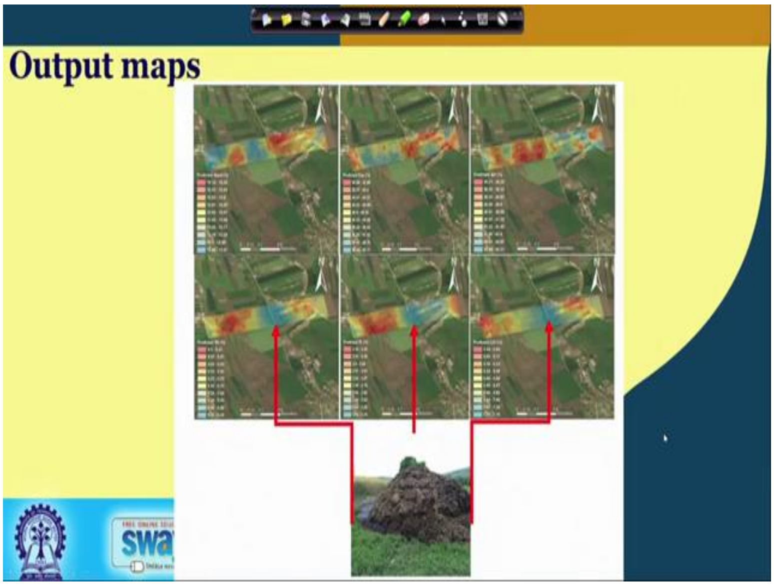

Output maps

So, you can see here we are producing the spatial variability maps or kriging maps of particular soil property, actually we have produced maps of total carbon. And the you know this is a map of sand and then map of clay and then map of shield and then total nitrogen total carbon and loss on ignition method of soil organic carbon. So, you can see we have produced all of this map using this combine predictor, combine variable indicators as well as this mapping geostatistical mapping techniques.

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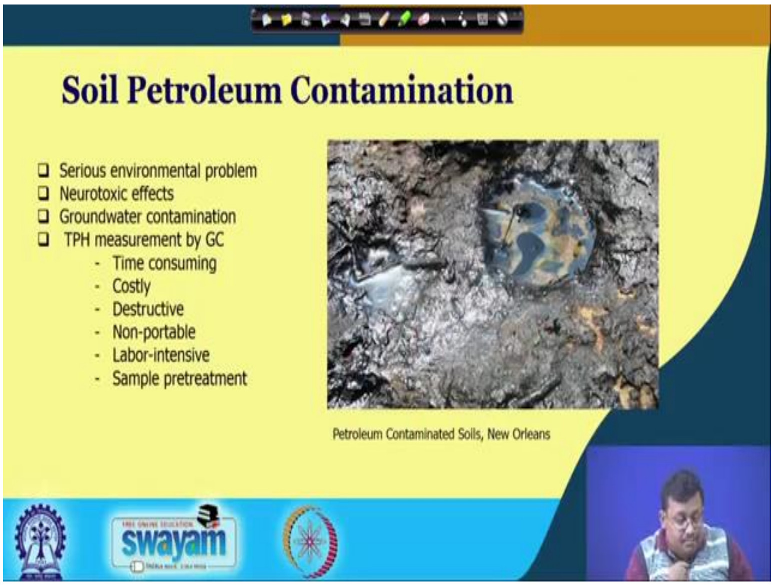

Soil Petroleum Contamination

- ☐ Serious environmental problem
- ☐ Neurotoxic effects
- ☐ Groundwater contamination
- ☐ TPH measurement by GC
  - Time consuming
  - Costly
  - Destructive
  - Non-portable
  - Labor-intensive
  - Sample pretreatment

Petroleum Contaminated Soils, New Orleans

Also we approved the usefulness for mapping the soil petroleum contamination, you know soil petroleum contamination is a serious environmental problem because, it produces the neurotoxic effects.

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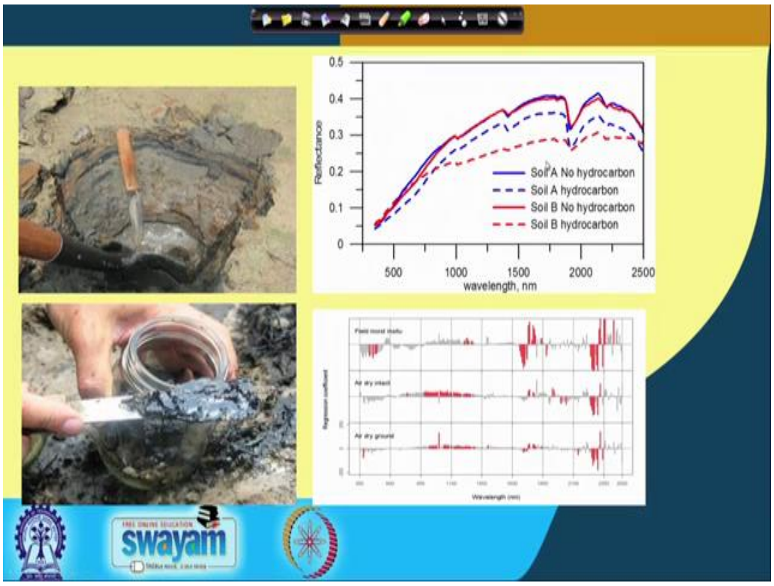

It is so, if we collected several contaminated samples from different areas and we plotted their you know their reflectance spectrum. You can see the differences between the reflectance spectrum originated from the soil which has got no hydrocarbon or petroleum and also the from the soil which are having hydrocarbon on petroleum. So, you can see we have collected two different soils and then we can see the differences in their reflectance pattern. Also we have identified these red are the basically important significant wavelengths which are responsible for identifying these petroleum hydrocarbons.

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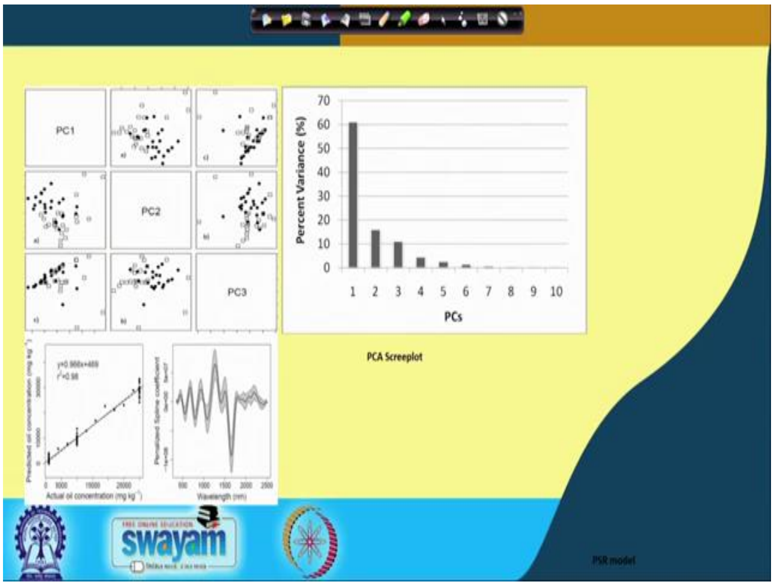

PCA Screeplot

PSR model

So, we produced several types of maps on several types of models. So, we saw that we can predict this petroleum hydrocarbon contaminated soil using great accuracy with an R^2 values of 0.98 you can see here.

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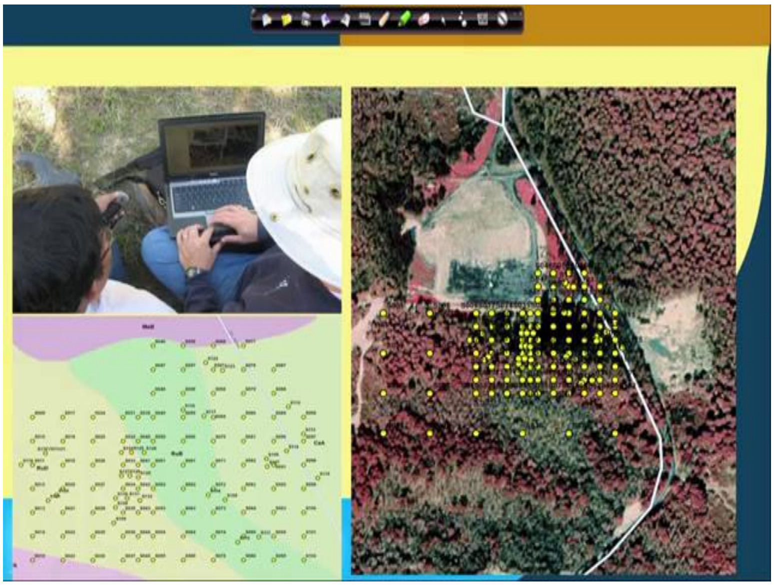

And not only that we use this technology in the directly into the field for mapping as a petroleum spilled area. So, you see there is a spilled area where there is a petroleum you know petroleum oil spill; it is a actually hilly area we are looking from the top and actually the oil moved through these area. So, this in an swell area so, we collected several soil samples from this area these are the soil sampling points.

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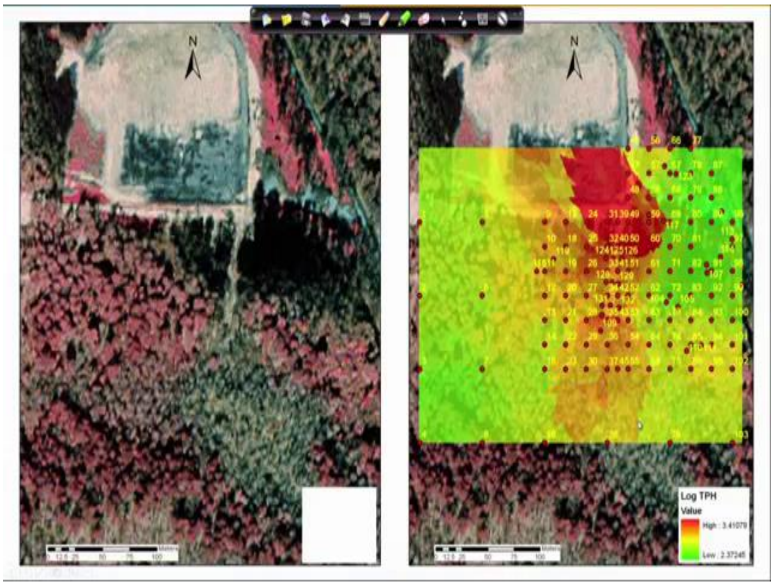

After collecting the soil sample points we model them using our we scan them using our spectro radiometer we model them and this is the spatial visibility map as you can see. Obviously, there are concentration high concentration of the petroleum is consented at this area and it moves through this area. So, that shows the better accuracy of our modelling.

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BP Deepwater Horizon oil spill

And not only that we also collected the contaminated soil samples from BP Deepwater Horizon oil spill you know. There is a big Deepwater Horizon oil spill in 2010 in the in United States. So, we went there and we collected several soil samples starting from very clean sand to very highly contaminated sand. And these are basically the petroleum which is mixed with the sand sample. So, we actually collected the sand samples which and this petroleum actually came from the sea and it actually contaminated the whole land as well as the beaches.

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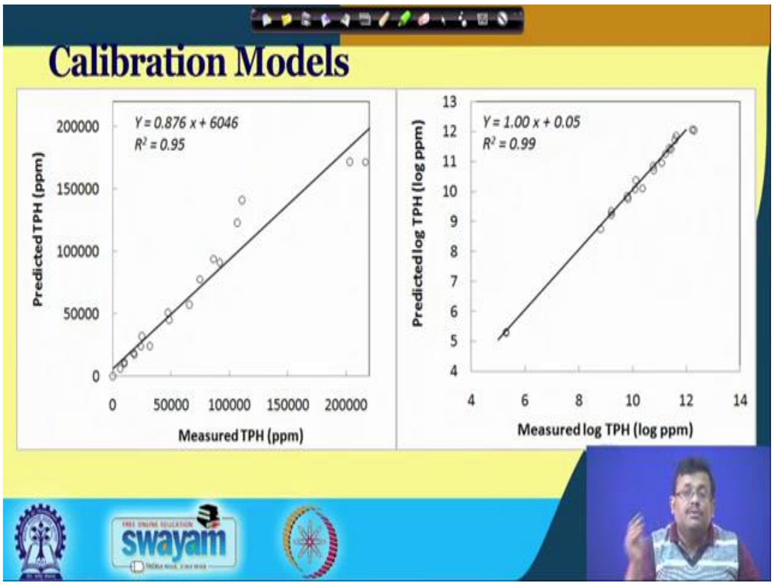

Calibration Models

So, we went there and we collected the samples from the beaches. These are the actual samples, after connecting them we scanned them with our spectra radiometer and we used the prediction model. And you can see we are getting the R^2values of 0.95 to 0.99. So, that shows the you know that shows that you know effectiveness of our technology.

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On-site Prediction

And also we gave some on site prediction for predicting the soil arsenic soil petroleum contamination. So, guys it is now I hope that you have got some basic overview of this technique how it works. Obviously, it you know it is not possible to cover all these aspects within this time frame, but I would request you to go ahead and read some literature. This is a burning topic now a days and this the most advanced techniques that soil scientists are using now a days for different types of soil research.

And it can literally curve down all the efforts of soil of soil based estimation. So, you can do some more advanced research; those who are who are pursuing some research in the field of soil science you can do this research on diffuse reflectance spectroscopy based modelling of different soil properties. So, guys thank you; let us wrap up here and then in the last lecture we will be talking about another important technology that is portable X-ray fluorescence.

Thank you.