Environmental Quality:
Monitoring and Analysis
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Lecture No. 22
Analysis Methods – Organics in Water

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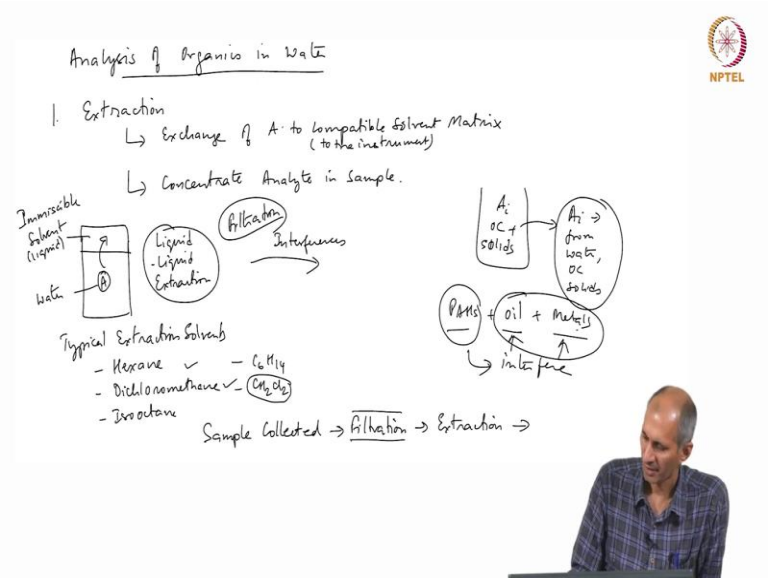

So, we look at analysis of organics in water. So, the first thing is the extraction, we look about extraction. Extraction means we are talking about something which is at very low levels typically we are talking about nanogram per liter or microgram per liter that level of concentration. So, we want to pull it out. So, as if you recall the objectives of extraction are use of compatible solvent exchange of A, compatible solvent matrix that is compatible means it to the instrument.

Second, it gives you an opportunity to concentrate analyte in the sample. So you can do this in multiple ways. So, one of the common methods in which people do this is one is your water sample which contains A they add another solid solvent. This is a liquid another liquid and then it will transfer to this. So, this is directly using what is called us liquid - liquid extraction. So the second solvent liquid that we use, by definition if you are going to do liquid - liquid extraction it must be immiscible in water predominantly.

So, use an immiscible when we say immiscible we are not talking if I am environmental point of view, all of them have some solubility, but bulk in solubility is very small. So, that you if you add to 1 liter of water, if you add 100 ml of 50 ml of solvent, you can recover most of it 40 ml you can recover at least. So, we did that calculation last time. So, this is one of the reasons you can only recover some of it. You cannot recover all of it because some of it may have already dissolved in it.

So, that is possible we did not worry about that. We are goal is to extract us much as possible. So, typically immiscible solvent which can now has a greater solubility of A in it. So, if some inorganic some organic solvent that can hold a in a larger quantity, so, typical extraction solvents are hexane, Dichloromethane these are 2 very commonly used you also use Isooctane and things like that, but, typically these 2 most of the methods.

You will see these 2 Dichloromethane is CH_2Cl_2 hexane is C_6H_{14} this is a very hazardous compound so, I said chlorinated organic solvent this but it is one of the strongest solvents that there is many of the chlorinated solvents organic solvents are very powerful they will extract a lot of things. But they also are listed among the sugar and look at the priority chemicals of concern organic you will find some of them there.

You will find some of them there because they are used in large quantities so there is an opportunity of exposure is very high. So, industry and all the people use solvents for all kinds of things they use for painting, cleaning of things it is used because it is an extract valuable extract and so you want to clean something it will come out very easily. So, for this kind of extraction, we are not using large quantities we are using small quantities.

And that is where you have a section in your analysis method is called waste management which means you will end up at the end of your extraction you will have some Dichloromethane, hexane which is not supposed to be you can revert right down the down the drain. You have to dispose it properly. This this is 1 point in which you have to do method selection properly. So, there is a waste management associated with it because you are introducing some hazardous chemicals.

And you are understand this principle when you are we are not discussing it in this course but when you are discussing treatment processes or remediation you remember this that maybe sometimes it is very effective to use some chemical for treatment or clean up but then that you are creating more problems and mental problems. But here the usage is so small quantities are very small and the purpose is to analyze. So, you see, it is not considered as we will search for methods which are more sustainable, but if it is not, so we do whatever is the best available for us.

So we extract when you extract water as it is raw water, sometimes you will get everything you will get all the interferences in your sample and this is this is true mostly for samples that are coming from sewage water treatment plants or sewage treatment plant or some such thing you want to know what is the concentration there you may find some other material there and that may also get extracted and that will introduce interferences.

One example is this so, when I normally say I collect a water sample of water from a very turbid lake, I did not do anything I just add hexane or dichloromethane with an extract it will extract everything it will extract from the organic carbon there will be organic carbon in the water it will extract all of that. So, if you if the original water contains A + organic carbon plus some other solids say let us not call it a we call it a bunch of A, I will call it as Ai it means there are lots of chemicals.

It is not only extracting this Ai from water, it is also extracting water from OC and from the solids and Ai will contain a few things. So, here comes the definition of an interference very interesting. Now, the value of your goal is to find out what is there in the water whatever is there in the water is going to come out and none of it is really an interference is very specifically defined for 1 analyte or a group of analyte that you are focusing on.

For example, if I have PAHs plus oil plus some other things say let us say metals in the water. I am interested in all of this. How much of PAHs are there although I did not, but if I am analyzing PAHs specifically, these 2 may interfere with my analysis of pH, the oil and the metals will

interfere. So in my method for analysis of PAHs if this is seen as an interference, these are interference. So I must do something with these 2 before I go on the analysis PAHs.

So, you have to interpret the interference like that so there is no absolute interference is relative to whatever you are trying to analyze. All of these are important for analysis. So, the other interference is from what we talked about last class. When I am analyzing water I want competition concentration of A, in water alone. If this is associated with solids, and I am really not I know I am getting the wrong information. So, before this you have to before you do liquid - liquid extraction, if you did not want that information, you have to filter the samples.

So filtration must be done before LLE. So normal sequence of events that you will see is when the sample is collected then we move on from there. After this we do the next step.

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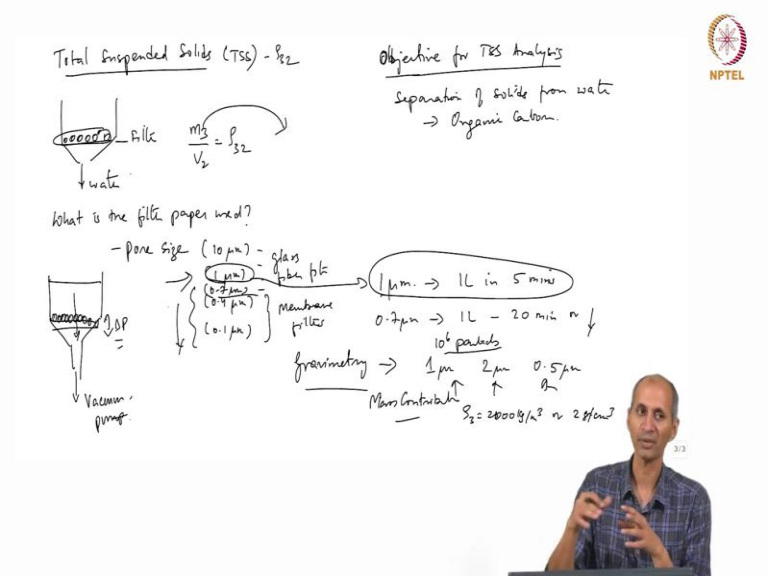

I want to sidestep a little bit here and talk about filtration a little bit. So, we have discussed something called total suspended solids. I think we have not discussed it in detail. So, is it a good base to do this now, when you say total suspended solids it is we are taking a water sample and filtering it through we are putting a filter paper and all the solids are trapped on here and you get water and the mass of filter that is collected here divided by the volume of water we give you row 32 this total suspended solids, row 32.

There is 1 piece of information that is needed here is what is the filter paper that we use? Have you discussed this in the class? No, I did not think so, what filter paper will use. So, you if you go and open a catalog for filter papers, you will find a lot of papers you have useful to papers from high school for various things. This what is the definition of the filter paper that we use here? What kind of filter paper do we use? What is the characteristic of filter papers?

Typically when you say filter paper, what is a pore size predominantly is a pore size is a microns. So, it will say 10 microns or it will say 1 micron you can go up 0.4 microns, 0.2 microns, 0.1 microns all of them are there different kinds up to about 0.7 or 0.4 microns of up to 1.7 microns you can do you have glass fiber filters from somewhere around here you have membrane filters and your more specialized filters.

So, you have a wide selection of filter paper and filter materials, what will you use? Is there a qualitative rational decision. Why we want to use what is the people who have done filtration will know what is the difference what is your objective is we had all this example analysis problems because for this question what is your objective in doing this in analyzing TSS objectives are very simple answer objectives, separation of solids.

So, what are the solid sizes from what are like separate or fully like separate based on the example in the last week that we go mainly what would you like to separate doing organic analysis? What would you like separate finalizing an organic chemical in the water I would like to separate some component which will accumulate a lot of organic compounds. So mainly I am interested in separating organic carbon for that purpose.

But TSS does not differentiate when organic carbon and the TSS suspended solids, it does not care anything, all particles from whatever size down to the size. So, ideally what would I want to use if I have filter papers available for 0.1, 4 size. Can I use 0.1, 4 size? It will stop everything 0.1 because colloids organic carbon is in the size range of 0.4, 0.2. I will definitely remove all of them. Then why do you have filter papers have 1 micron and 4 microns and 10 microns.

For one is easier to make, that is 1 reason but they are still there in the market and people sell it for one reason. What is the other support size goes down? What is the other consequence in filtration? It takes longer, it takes an enormous amount of pressure drop to push liquid through solid. That so it takes very long to do this second. So let us say I use a filter say I use 1 micron glass fiber filters. I am able to do it in filtration of 1 liter in 5 minutes.

I take 0.7 micron filter, it takes 1 liter I am able to do it in 20 minutes or sometimes depending on the amount of solid subscription I may not even be able to finish it filter will get clogged. I cannot do so the way we do filtration here. If I if you if I want a little filtration if I just put it on a filter paper and leave it the pressure drop across the filter paper itself is so much that the moment the first drop of particles come and collect there. It will not go through by gravity.

So you have to put some negative pressure here so you have to apply a vacuum to do this. You have to otherwise by gravity alone, it will not happen. You have to push you have to have a vacuum to pull the water across a filter, there is a pressure drop that is acting here. So, you have to push it across at using the filter. So, you went with the vacuum pump you are you would not be able to filter it sometimes with 0.7 micron filter.

So, between 1.7 is there is this much difference, I would rather use a 1 micron filter what will be my loss of information if I use a 1 micron filter versus 0.7 let us say that some small particles will get through we go through for 1 microns pore size does not mean all particles below 1 micron will not get filtered. That is not true filtration theory. It is not the fourth size it is a lot of it is much more complicated. So you will get retention of particles much lower than 1 micron.

There may be other particles which we still going on that is one of the basis for which we use 1 micron filters instead of pointing fingers is not really needed pointers on 1 micron is probably good enough to do whatever collide removal you want. But still there is a question when asked to draw a line somewhere and the basis for that is not 1 of this is this that if filtration was very easy 0.5 people would use 0.5 but filtration is not easy.

And so 1 micron filter is used. What is the difference between 19.7 in terms of information lost? What is TSS? How is analysis done? With entry or do you get entry by weighing gravimetry measurement. So, gravimetry if I take particles that are 1 micron versus particles that are 2 microns versus particles that are 0.7 micron 0.5 microns. And let us say there are 10 raise to 6 particles. You calculate what is the mass contribution of this one?

Calculate for 1, take the density of the particle as they say 2000 kilograms, or 2 grams per centimeter cube will turn out that the even if you have a million particles, the contribution to mass is very very small. You would not even probably see it in the balance that you are using the 4 digit balance that you use you may not even see it in terms of contribution to mass. So, while it gives you a lot of separation, lot, little more separation, 1 micron filter will give you 1.7 and below these filters will give you a lot more separation then 1 micron filter.

The information that you get from terms of mass gravimetry is decreasing it is almost negligible. While you do this, you can do the calculation, for yourself convince yourself how much is the loss. So, since the total suspended solids methodology is, is based on gravimetry. It does not really matter then this row, below 1 micron, you are not going to get any additional information and you are also going to get you are going to waste your time by trying to push it through this thing and you are not getting any additional information.

So, 1 micron is to set as standard filter size for TSS to because it is TSS standard filter size when you do water analysis use general TSS analysis which means you are filtering the sample and that filtered sample is what we usually take for analysis of water. Now, it does not mean that the water that is filtered through 1 meter does may contain collide which have organic and for that you have to measure the TOC and then correct that value. That is a separate issue.

And nothing you can do about it in terms of separating, but you can at least to calculate the TOC and say, I know the total concentration. Now I know that TOC I want to find out what is the actual phase concentration based on the calculations we did last week.