

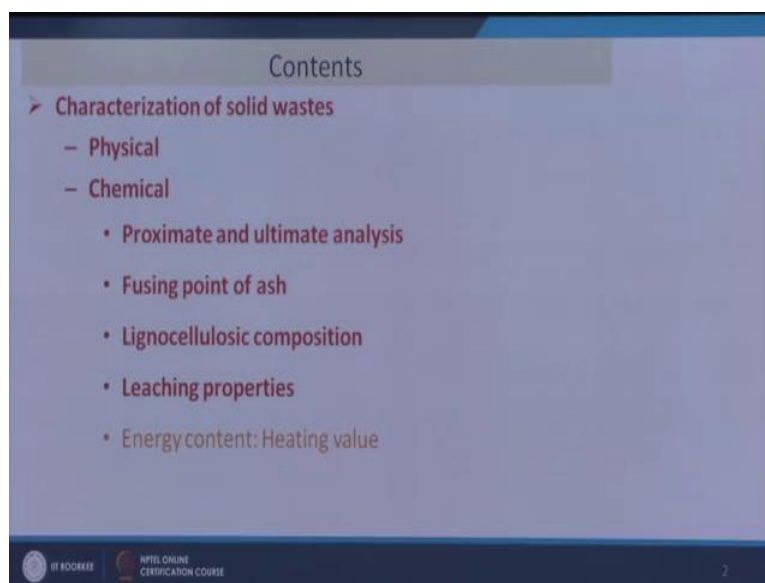
Waste to energy conversion
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Lecture - 03
Characterization of Wastes – 1

Good morning. Now we will start discussion on the module Characterization of Wastes. In the introductory module we have discussed that all type of wastes are not suitable for energy production, and we have also come to know that those waste which are suitable for energy production can also produce energy through different routes not with the same routes; that is why to understand whether a particular waste will be suitable for energy production through a particular route, we need to understand the properties of that waste.

So, characteristics of the waste is very important and this module will be used to describe the processes which are used to kept characterize the waste, and how the properties are determined. So, basically we have solid waste and liquid that is waste water. So, we will be dividing the whole characterization module in parts, the first two parts will describe the characterization of solid waste, and the third part will describe the characterization of liquid that is waste water.

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So, in this part we will be discussing most of the important properties of solid waste except that energy content or heating value. So, will be discussing the physical and

chemical properties and mention their proximate and ultimate analysis fusing point of ash, then cellulosic composition and leaching properties of the solid waste .

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Important physical properties

- Specific weight or density
- Particle size and size distribution
- Permeability or hydraulic conductivity
- Moisture content

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So, what are the physical properties of solid waste? Some important physical properties are mentioned here that is specific weight or density, then particle size and size distribution, permeability or hydraulic conductivity and moisture content.

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Specific weight or density

Specific weight/ density is defined as the weight of a material per unit volume (kg/m^3)

There are three densities associated with powders.

Absolute density (also termed the **true or skeletal density**): it excludes the volumes of pores and of the inter particle spaces.

Envelop density (sometimes called the **apparent density**) includes the pore volume but excludes inter particle spaces; and

Bulk density : it includes both pore volume and inter particle spaces. Its value changes with degree of compaction

Condition	Density (kg/m^3)
Loose MSW, no processing or compaction	90-150
In compaction truck	355-530

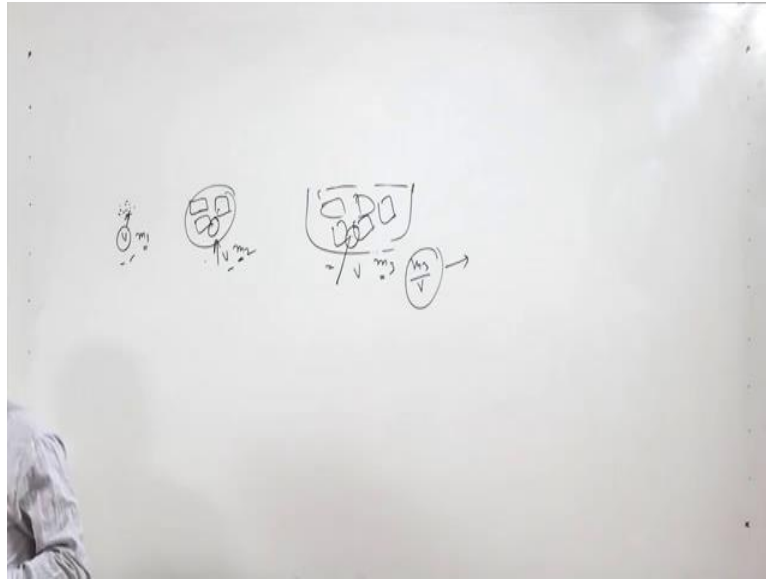
Measurement of density is done by true density and tap density analyzer (Pycnometer) normally by employing Archimedes' principle of fluid (gas) displacement and the technique of gas expansion.

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So, at first we will discuss on specific weights or density; we know that specific weights or density is defined as the weight of any material per unit volume. So, that is kg per

meter cube, there is a unit in a system and the density may be of basically 3 types: absolute density, apparent density and bulk density. Say if we have 1 material say we have some wood piece of wood, so if we get the saw dust from this the wood dust from it, so the particular size will be smaller or you we can keep more number of chips and store in a truck.

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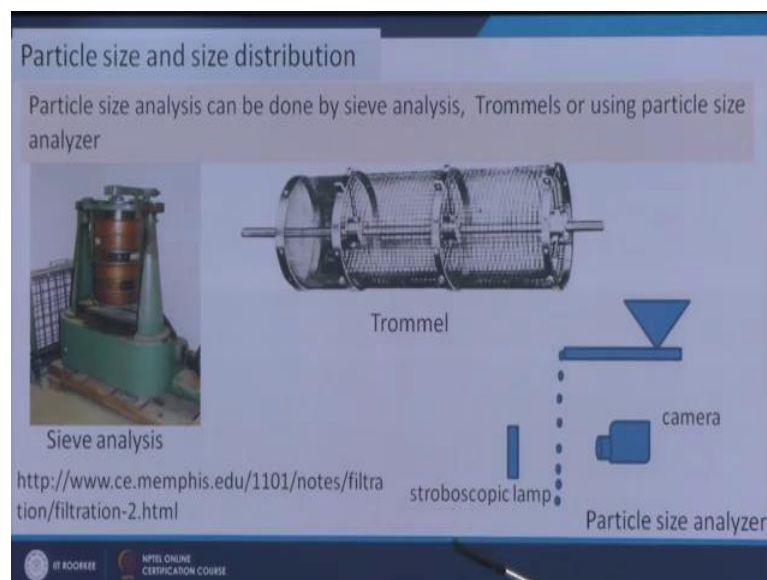
So, in this 3 situation if we use the smaller particles or we use some chips here, and we can use it store it in a very big volume like this.

So, if we consider 1 volume v here same in this if we consider volume v , and here also if we take any volume v say here we are taking v . So, these 3 v are same volume is same. So, mass m_1 , m_2 and m_3 this m_1 , m_2 and m_3 will not be same So, here the m_1 will be more than this and then this; that means, the less m or less mass in this unit volume or volume v is because of the incorporation of inter particle wide space and the wide space inside the particulars. So, here also the inter particle portion is less the contribution of inter particle wide space is less and here both are less the particles are considered only. So, this is absolute or skeletal or true density and this is apparent density and this is our bulk density.

So, these are 3 types of density are used to characterize the solid waste and on application point of view the solid waste are basically having relatively bigger size. So, bulk density and apparent density will be much important, but when will be using some

catalyst for the processing of the solid waste at that time. This true density of the catalyst will also be important. So, how can we measure the density? The density can be measured by Pycnometer. So, there are 2 types of arrangement that is true density and tap density analyzer and the principle is the Archimedes principle of replacement of volume of the gas. So, as discussed just now that this is the m/v these value can be increased by compression by. So, some example is given here if we take loose (Refer Time: 05:46) solid waste no processing or compaction if the density is say 90 to 150 kg per meter cube, but this can be increased to 355 to 530 kg per meter cube by compaction in the truck.

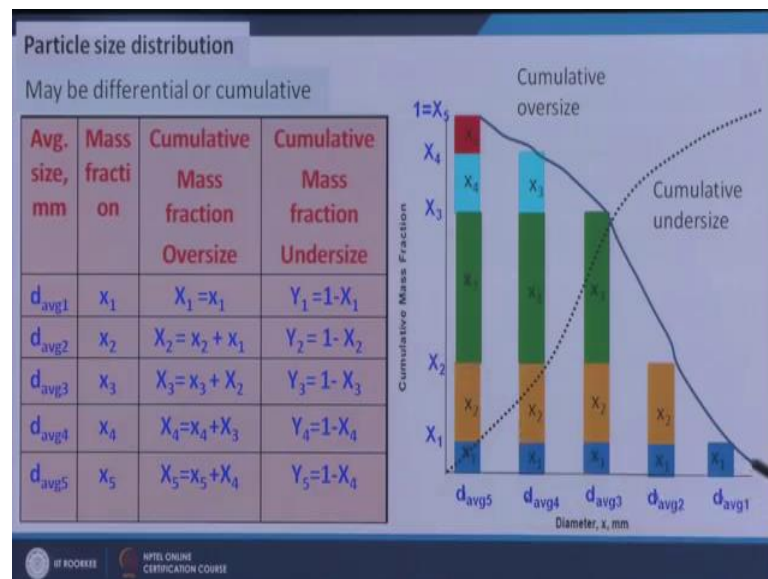
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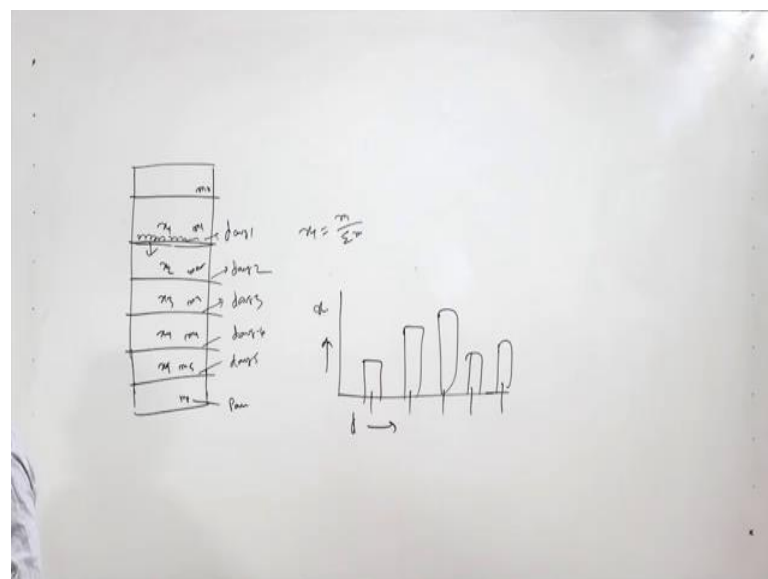
Next property is particle size and its distribution; particle size is very very important because different type of reactors we will see in later modules that the reactors have some capacity to handle certain range of particles. So, particle size is important to understand whether this will be suitable for using in a particular process or not, and the determination of that particle size can be done by basically three approaches as shown here; now 1 is the trammel when the particle size is relatively bigger the trammels are used and this is very suitable for (Refer Time: 06:43) solid waste having particular bigger particle size, intermediate particle size materials can be the particle size can be determined using sieve analysis and when the particles are very very fine particles then we can go for it is particle size analyzer using some camera.

So, now we will discuss particle size distribution. So, we have come to know that these are different approaches through which can determine the particle size, but in a mixture of solid waste all the particles will not be having the same dimension or same size there will be some range all the particles may have different size, but very similar or within certain range.

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So, the distribution is also very important. So, 1 example is given here now what do we mean by distribution. Say if we use 1 chip (Refer Time: 07:36). So, say d_v average d

average say $d_{\text{average } 1}$, $d_{\text{average } 2}$, $d_{\text{average } 3}$, $d_{\text{average } 4}$, $d_{\text{average } 5}$. So, we have different screens pan and there is some other top also. So, this is the screens just as shown here as shown here different screens are there. So, after certain time we will get the material into different screens. So, here we will getting some m_1 say here m_0 , m_1 , m_2 , m_3 here we will get m_4 , here we will get say m_5 like this.

So, correspondingly we will get the total and this is for m_{pan} . So, we get the total if we ignore this 1 and this one. So, that will be our total m_1 , m_2 , m_3 , m_4 , m_5 of corresponding x_1 , x_2 , x_3 , x_4 , x_5 that is mass fraction. So, x_1 is equal to m_1 divided by sum of m . So, this is the mass fraction. So, that way it is reported here that mass fraction is this one. So, $d_{\text{average } 1}$, $d_{\text{average } 2}$, $d_{\text{average } 5}$ we are getting different mass fraction.

Now with this mass fraction we can plot like this see x_1 just we can plot. So, $d_{\text{average } 3}$, $d_{\text{average } 4}$, $d_{\text{average } 5}$. So, $d_{\text{average } 1}$ say this one like this, this is x , so the average the value this direction, and y axis x . So, that has that this difference will plot the same information we can present in that way or the same information we can present in this way also. So, one is cumulative oversize and cumulative undersize. So, this is called cumulative presentation of the data and this one is called differential presentation of the data. So, both way we can present the data for further understanding or better understanding of the distribution of the particles in into different fractions.

So, here oversize cumulative oversize means when we will consider one screen, the material which will be on 8 written on 8 oversize which will be passing through 8 undersize; so that way we will be getting this type of nature for oversize and this is for undersize cumulative

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Hydraulic conductivity

- Hydraulic conductivity: It is a measure of the ability of porous media to conduct liquid. Although testing of hydraulic conductivity is simple in theory, movement of water through a medium is very complex.
- Factors affecting hydraulic conductivity:
 1. Particle size
 2. Fabric
 3. Degree of saturation
 4. Pore geometry
 5. Properties of the test fluid
 - However, it is difficult to characterize MSW samples based on these factors due to heterogeneity of MSW

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Now hydraulic conductivity is another property of the solid waste, because it helps the percolation of water through it. So, this is tested because to know in landfill leachate how the water is going through the layers under the earth. So, to understand this the hydraulic conductivity is measured and the factors which affect this hydraulic conductivity are particle size, fabric, degree of saturation, pore geometry, properties of the test fluid, but it is not so easy to measure this one for the (Refer Time: 11:33) solid waste because of its heterogeneity nature.

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Moisture content

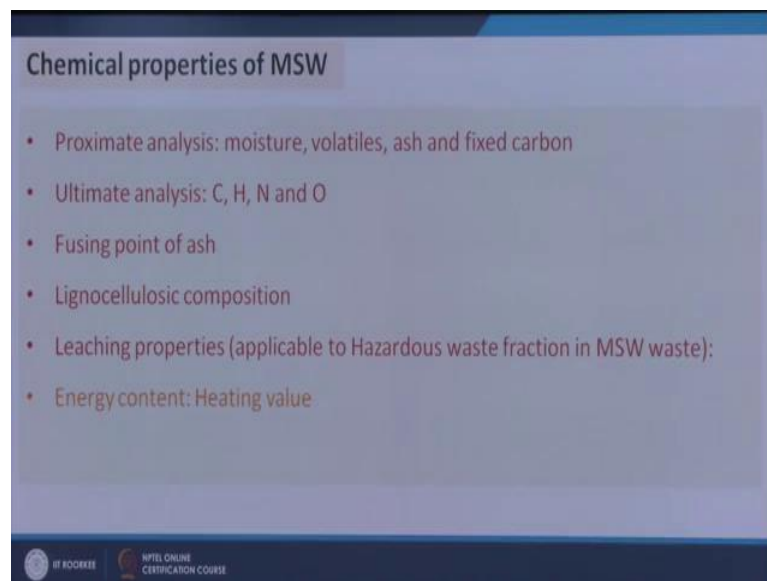
- It is determined by using a hot air oven, where the material is heated at 105°C till constant weight is achieved.
- It is calculated by following formula:
$$\text{Percentage moisture} = \frac{\text{Loss in weight}}{\text{Weight of material taken}} \times 100$$



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The moisture content can be measured by heating certain amount of solid waste at certain temperature and that temperature is equal to 105 degree centigrade and till constant weight. So, all the water will be vaporized and then will get the remaining materials. So, loss in weight is proportional to the weight of the moisture content of it. So, that is percentage moisture is equal to loss in weight divided by weight of material taken into 100.

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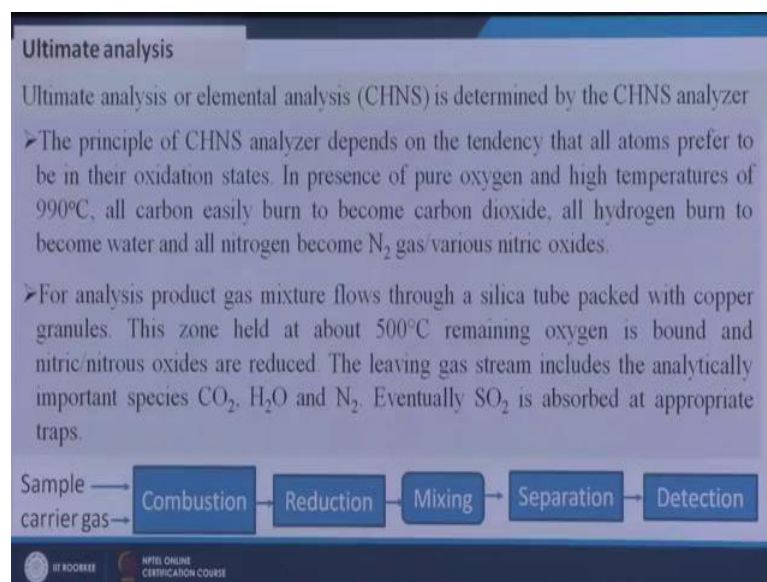


Now we are coming to chemical properties of the MSW municipal solid waste or any other solid waste. So, their proximate analysis ultimate analysis at last we discuss the proximate analysis. So, in case of proximate analysis we can determine fixed carbon, we can ash content, we can moisture determine moisture content and volatiles. So, when we take some solid waste and we heat it at very high temperature in absence of oxygen then the volatiles will blow off, but will not allow for a longer period of heating very small period of heating at high temperature.

So, volatiles will blow off and the mass loss will be proportional to volatiles, and then we will use the material and heat it under certain condition for longer period in presence of oxygen. So, that all the material is burnt and the metals which are present in it that will be converted to ash, and the direct amount of material present that will be proportional to that ash content; and then moisture content already we have discussed. So, the remaining will be the fixed carbon, so fixed carbon equal to 100 minus moisture

minus volatiles content minus ash content. So, at first the moisture content is determined same sample is used for volatiles determination, and then for ash determination and by difference fixed carbon.

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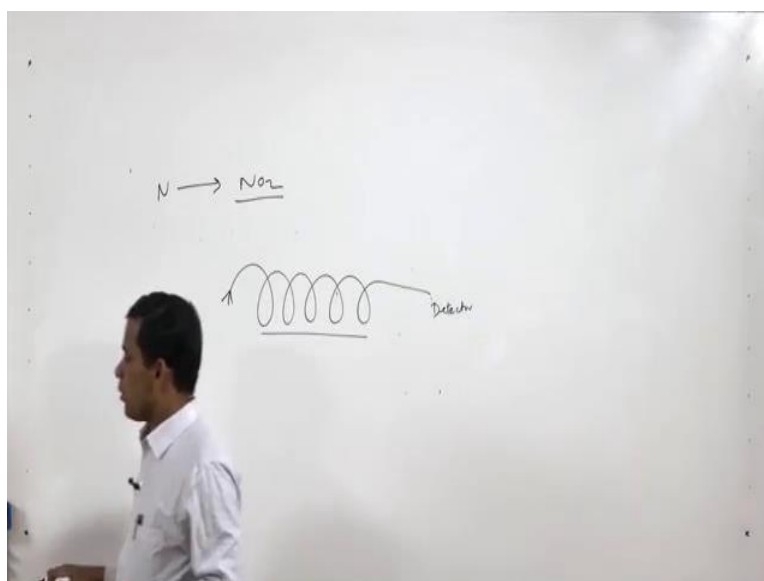


So, ultimate analysis that is CHNS analyzer the basic principle of this CHNS analyzer is that all the elements carbon, hydrogen nitrogen sulfur will be converted to oxides. So, C will be converted to CO₂, H will be converted to H₂O. So, S will be converted to SO₂, N will be converted to NO₂, but this NO₂ conversion is not so easy as the temperature maintained where is around 990 or 1000 degree centigrade.

So, part of the nitrogen is converted to NO₂. So, the first job of the determination of CHNS is the combustion. So, sample will be combusted at higher temperature and then the flu gas will be produced in oxide form CO₂, H₂O, NO₂, SO₂ etcetera. Then that will be correct by some carrier gas and will be passed though some reduction zone, some reduction means some parts some tubes are there filled with proper granules. So, proper granules will use the oxygen and reduce the Knox which are generated. So, nitrogen will be produced.

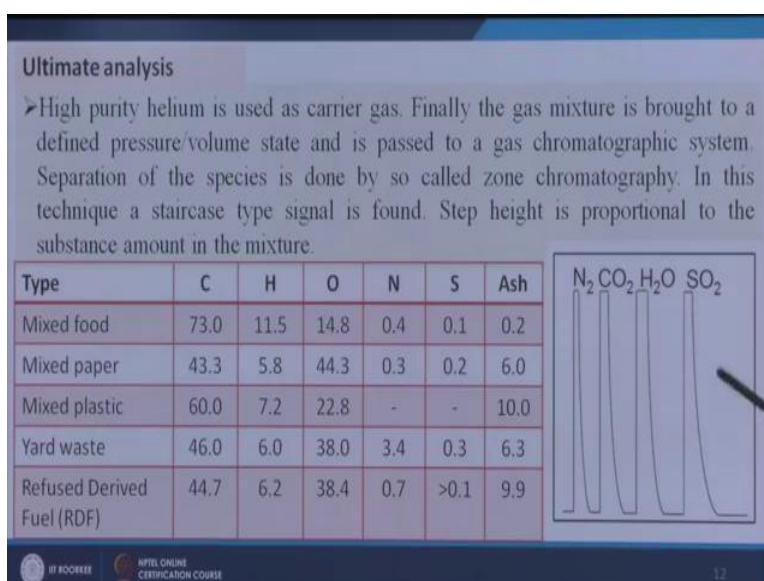
After that will be some mixture is required for the proper mixing of different gas components here, then separation of the gas components this separation is basically done using some chromatographic column say gas chromatographic column. So, depending upon the nature of these gas molecules, the retention time will be different say.

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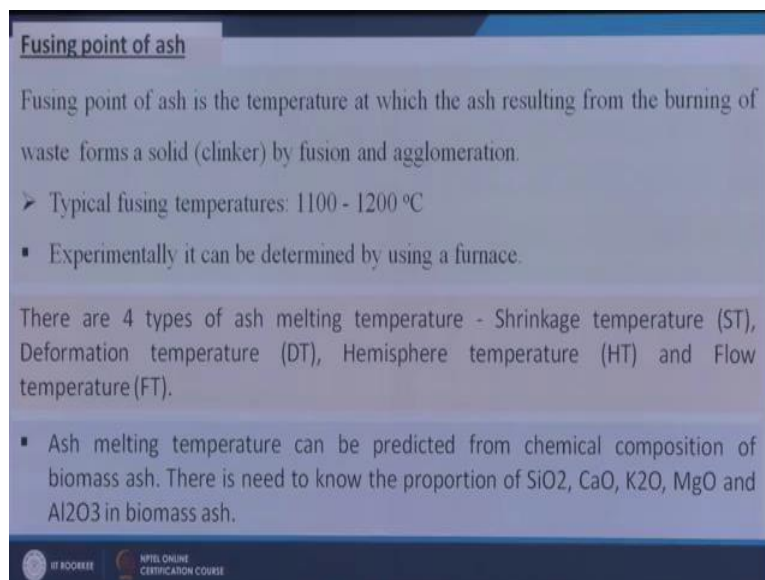
If we have one column in this column say gas is going having all the components CO₂, H₂O, N₂ and SO₂ and this column is filled with some adjuvants. So, that materials will be having different capacity and different attachment capacity, show the less reactive materials will come first, then the lesser one then the lesser one. So, so retention period will be different then when the material the gas is coming out we will be using some detector. So, that detector will detect what is the component is coming in what is the concentration. So, this is the mechanism for the determination of ultimate analysis of the CHNS content in it.

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So, as show here SO₂, H₂O, CO₂, and N₂ this type of a spectrum we can get in this see depending upon the different retention period. Some examples are CHNS analyses of different materials are shown in this table.

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Fusing point of ash

Fusing point of ash is the temperature at which the ash resulting from the burning of waste forms a solid (clinker) by fusion and agglomeration.

- Typical fusing temperatures: 1100 - 1200 °C
- Experimentally it can be determined by using a furnace.

There are 4 types of ash melting temperature - Shrinkage temperature (ST), Deformation temperature (DT), Hemisphere temperature (HT) and Flow temperature (FT).

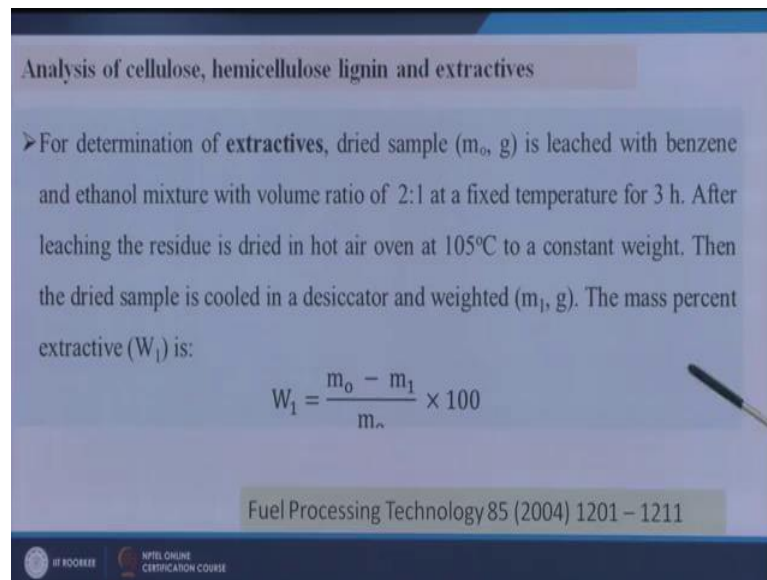
- Ash melting temperature can be predicted from chemical composition of biomass ash. There is need to know the proportion of SiO₂, CaO, K₂O, MgO and Al₂O₃ in biomass ash.

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Now, fusing point of ash; so fusing point of ash that is the temperature at which the ash content of the solid will be fused. So, if we think about the mechanism of this. So, basically we will get number of steps; the first is shrinking of the solid waste that is shrinkage temperature, then further heating deformation temperature if we give more heat than hemisphere temperature; that means, the solid ash gas is gradually going to be molten and this is intermediate stage when hemispherical form appears and then if we increase the temperatures further, it will be flowing that is flow temperature.

So, these are the temperature which have been reported and step by step this situation comes; and for the determination these can be determined experimentally by using some furnace and people have tried to develop some mathematical relationships that is empirical models through which this can also be determined and for that the composition of the ash is required. What are the metal oxides present in it like say SiO₂, CaO, K₂O, MgO and Al₂O₃ what are the composition on it if we can understand, then some empirical formulas are available. So, that can be used to calculate the ash fusing temperature.

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Analysis of cellulose, hemicellulose lignin and extractives

➤ For determination of **extractives**, dried sample (m_o , g) is leached with benzene and ethanol mixture with volume ratio of 2:1 at a fixed temperature for 3 h. After leaching the residue is dried in hot air oven at 105°C to a constant weight. Then the dried sample is cooled in a desiccator and weighted (m_1 , g). The mass percent extractive (W_1) is:

$$W_1 = \frac{m_o - m_1}{m_o} \times 100$$

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Next we will discuss on the determination of the lignocellulosic components. So, that is lignocellulosic biomass are having lignin, cellulose and hemi cellulose. So, we have to determine the concentration of these and this also the lignocellulosic biomass mass also contains some amount of volatiles, so our objective here to determine the extractives, cellulose, hemi cellulose and lignin. So, how can we determine the extractives as the term indicates there is it (Refer Time: 18:53) it is extracted from the solid by using some solvent. So, that solvent is basically benzyl and ethanol in 2 is to 1 ratio. So, we add some certain amount of solid waste in the solvent and keep certain time and provide certain conditions and then the volatiles will come out to the solvent, then the condition is here at fixed temperature for 3 hours agitation and then after leaching the residue is dried in hot air oven at 105 degree centigrade. So, that all moisture is going out then the dried sample cooled in desiccators and weighted.

So, if that is m_1 , m_o minus m_1 by m_o , m_o minus m_1 by m_o that is initial mass how much we have taken now how much we have present. So, that is the loss m_o minus m_1 by m_o into 100 that is the extractive percentage.

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➤ For determination of **hemicellulose** the residue m_1 after extractive is put in a flask with 150 ml NaOH solution (20g/l). Then the mixture is boiled for 3.5 h with recycled distilled water. After that the residue is washed with distilled water till no sodium ion present in wash liquor and dried it to a constant weight. The dried sample is cooled in a desiccator and weighted (m_2 , g). The mass percent of hemicellulose (W_2) is:

$$W_2 = \frac{m_1 - m_2}{m_o} \times 100$$

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Next are hemicelluloses. So, hemicellulose can be dissolved in alkaline solution. So, this property is exploited to determine the hemicellulose content and certain strength of NaOH solution not very high strength it is 150 ml NaOH solution 20 gram per liter, by 2 solution is taken then in this case 3.5 hour recycled is done with distilled water and after that the residue is washed with distilled water till no sodium ion is present in the washed water; that means, it is the material is made free from the sodium hydroxide with the dried sample is cooled in a desiccator and weighted, and the amount if it is say m_2 then W_2 is equal to m_1 minus m_2 divided by m_o ; that means, after the separation of the extractives the sample is taken. So, m_1 minus m_2 divided by m_o , this is the percentage of the hemicellulose in the solid sample.

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➤ For determination of **lignin** about 1g of residue after extractive analysis is dried to a constant weight. Then the sample is cooled and weighted (m_3 , g). After that the sample is mixed with 30 ml sulfuric acid of 72 percent (v/v) concentration and kept at 8-15°C for 24 h. The mixture is diluted with 300 ml of distilled water and boiled for 1h with recycled distilled water. After cooling and filtration the residue is washed with distilled water till no sulfate ion present in liquor, which is detected by 10% barium chloride solution. Then the residue is dried to a constant weight and cooled in a desiccator and weighted (m_4 , g). The mass percent of lignin (W_3) is :

$$W_3 = \frac{m_4(1 - W_1)}{m_3} \times 100 \quad \text{where } w_1 = W_1/100$$

➤ **Determination of cellulose** The mass percent cellulose (W_4) was calculated as per equation

$$W_4 = 100 - (W_1 + W_2 + W_3)$$

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Next is lignin determination. So, lignin is not soluble in acid and alkaline, it is it is very less solubility it is having so, but cellulose can be dissolved. So, sulfuric acid is added here the concentrated sulfuric acid that is 72 percent, concentrated acid is added at 8 to 15 degree centigrade for 24 hour, and the mixture is diluted with 300 ml of distilled water and boiled for 1 hour with recycled distilled water. So, this is the procedure for the digestion of the materials in the (Refer Time: 21:37) super media. So, that cellulose will come out and then lignin will be in the solid phase. So, after separating it after the cleaning then it is washed with the water and then the residue is dried to a constant weight and cooled in a desiccator and weight. So, this weight is say m_4 . So, the lignin content is equal to m_4 into $1 - w_1$ divided by m_3 into 100.

So, what is this w_1 , small w_1 is equal to capital W_1 by 100 thus capital W_1 is extractive percentage, so extractive percentage by 100. So, $1 - w_1$ is m_4 divided by m_3 into 100 is the lignin content as reported by this fuel fusing technology, this journal in 2004 it is reported, now determination of cellulose. So, cellulose is determined by with a hundred minus W_1 minus W_2 minus W_3 . So, this is the determination of different lignocellulosic components in solid waste.

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Leaching test (*Toxicity Characteristic Leaching Procedure or TCLP*)

The *TCLP* mimics conditions found in landfills when groundwater percolates through buried materials.

The *TCLP* is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid and multiphase wastes.

8 Heavy Metals	12 Organic Solvents	
❖ Arsenic	❖ Benzene	
❖ Barium	❖ Carbon tetrachloride	❖ Pyridine
❖ Cadmium	❖ 2,4-dinitrotoluene	❖ Chloroform
❖ Chromium	❖ Hexachloroethane	❖ Vinyl chloride
❖ Lead	❖ Methyl ethyl ketone	❖ Cresol(s)
❖ Mercury	❖ Nitrobenzene	
❖ Selenium	❖ Tetrachloroethylene	
❖ Silver	❖ Trichloroethylene	

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Now, we will discuss how to determine the leaching property of the solid waste. So, leaching property is necessary because this is not desirable yes from solids if some proxy chemicals come out that is not desirable. So, some conditions are produced which are very similar to the conditions which favor in landfills. So, different types of toxic chemicals have been investigated their leaching capacity from the waste like say 8 heavy metals 12 organic solvents.

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Leaching test (*Toxicity Characteristic Leaching Procedure or TCLP*)

17 Pesticides

❖ Chlordane	❖ Hexachlorobenzene
❖ 2,4-Dichlorophenoxyacetic acid	❖ Lindane
❖ Cchlorobenzene	❖ Hexachlorobutadiene
❖ 1,4-dichlorobenzene	❖ Methoxychlor
❖ 1,2-dichloroethane	❖ Pentachlorophenol
❖ 1,1-dichloroethylene	❖ Toxaphene
❖ Endrin	❖ 2,4,5-trichlorophenol
❖ Heptachlor	❖ 2,4,6-trichlorophenol
	❖ 2,4,5-TP (Silvex)

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- Agitation apparatus: Capable of rotating the extraction vessel at 30 ± 2 rpm
- Extraction vessel:

Zero-Headspace Extraction Vessel (ZHE):
This device is for use only when the waste is being tested for the mobility of volatile analytes like Acetone, Benzene, n-Butyl alcohol, Carbon disulfide, Carbon tetrachloride, Chlorobenzene, Chloroform etc.



(<http://www.tclp-equipment.com/zero.htm>)

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And 17 pesticides have been tested. So, for the testing of this leaching property of the solid waste basically extraction method was used. So, extraction vessel and then extraction vessel are of basically types one is 0 headspace extraction. So, in this case there is no headspace and other is with headspace we are having some headspace.

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Bottle Extraction Vessel: When the waste is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed. Headspace is allowed in this vessel.



<http://www.tclp-equipment.com/zero.htm>

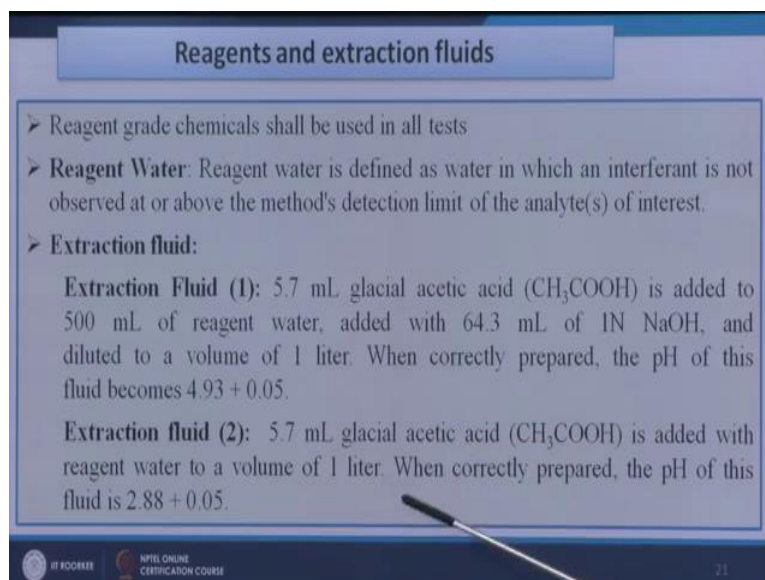
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So, the previous one is important for the case when some volatiles material are present in the solids like say Acetone, Benzene n-Butyl alcohol, Carbon disulfide, Carbon tetrachloride, Chlorobenzene, Chloroform etcetera when these are present in the in the

solids or in the waste. So, this setup is used in other case this setup is used that is bottom extraction vessel.

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Reagents and extraction fluids

- Reagent grade chemicals shall be used in all tests
- **Reagent Water:** Reagent water is defined as water in which an interferant is not observed at or above the method's detection limit of the analyte(s) of interest.
- **Extraction fluid:**
 - Extraction Fluid (1):** 5.7 mL glacial acetic acid (CH_3COOH) is added to 500 mL of reagent water, added with 64.3 mL of 1N NaOH, and diluted to a volume of 1 liter. When correctly prepared, the pH of this fluid becomes 4.93 ± 0.05 .
 - Extraction fluid (2):** 5.7 mL glacial acetic acid (CH_3COOH) is added with reagent water to a volume of 1 liter. When correctly prepared, the pH of this fluid is 2.88 ± 0.05 .

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Now, we need some extract extractive or extraction media. So, what are those requirement reagents and extraction fluids, we need the extraction fluids that is fluid 1 and fluid 2, which is at the pH is higher in the case of 1 that is acetic acid and sodium hydroxide both are added in this case, and here extraction 2 when pH is very less 2.88. So, only acetic acid is added no sodium hydroxide is required. So, 2 types of extractive fluids are used depending upon the nature of the pollutants present in the waste to waste or solid waste stream. So, after this in this part and we will discuss the heating value of solid waste in the second part of this module.

Thank you very much.