



IIT ROORKEE



NPTEL ONLINE
CERTIFICATION COURSE

WASTE TO ENERGY CONVERSION

CHARACTERIZATION OF WASTES-1

PRASENJIT MONDAL
CHEMICAL ENGINEERING DEPARTMENT



Contents

- **Characterization of solid wastes**
 - **Physical**
 - **Chemical**
 - **Proximate and ultimate analysis**
 - **Fusing point of ash**
 - **Lignocellulosic composition**
 - **Leaching properties**
 - **Energy content: Heating value**
- **Characterization of waste water**



Important physical properties

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



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

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.

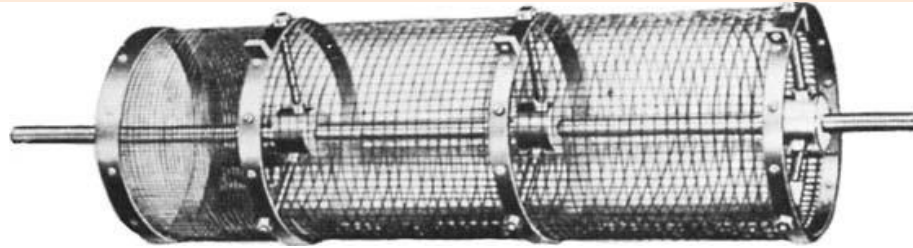
Condition	Density (kg/m^3)
Loose MSW, no processing or compaction	90-150
In compaction truck	355-530

Particle size and size distribution

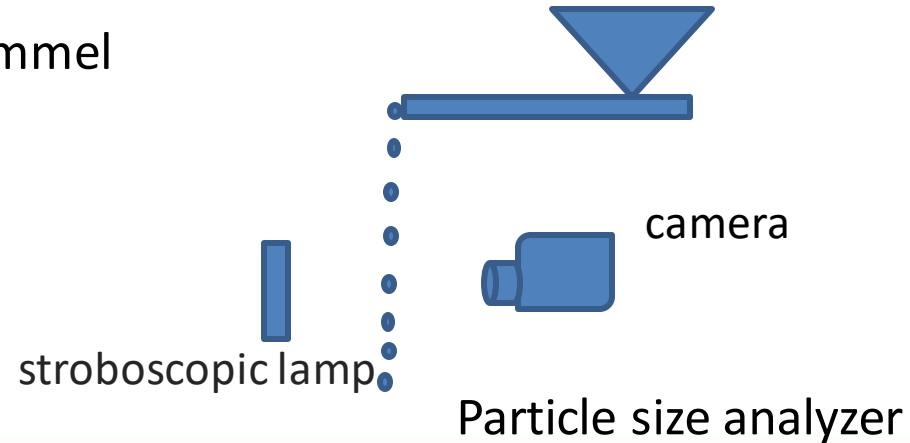
Particle size analysis can be done by sieve analysis, Trommels or using particle size analyzer



Sieve analysis



Trommel

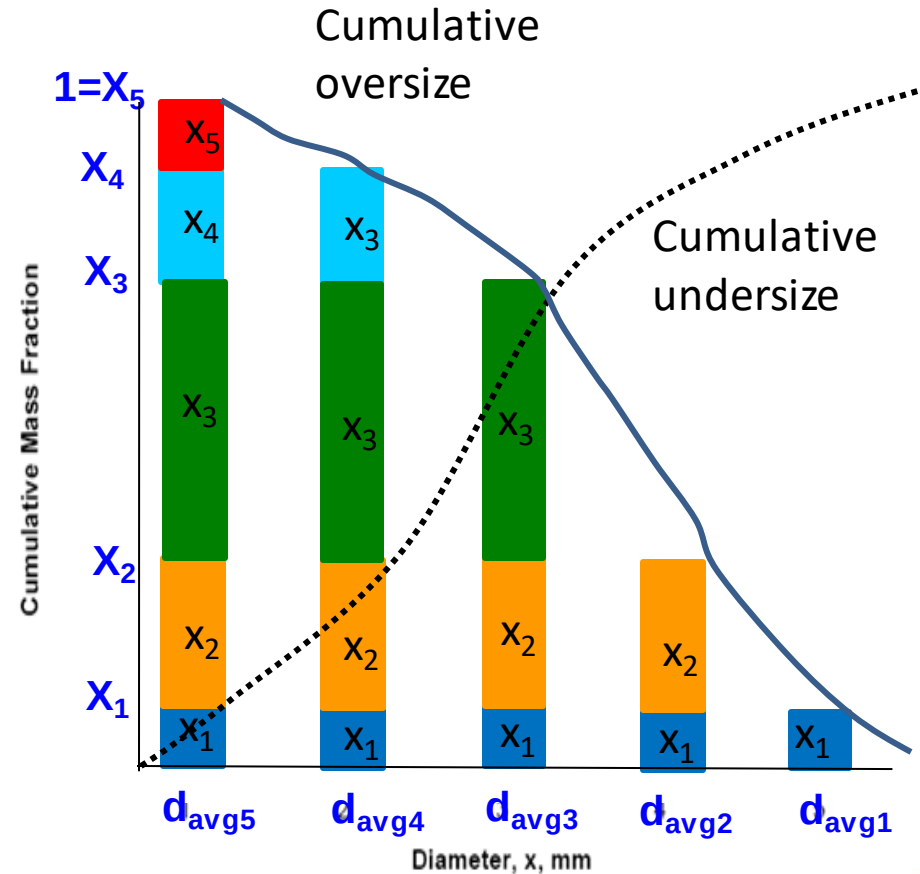


<http://www.ce.memphis.edu/1101/notes/filtration/filtration-2.html>

Particle size distribution

May be differential or cumulative

Avg. size, mm	Mass fraction	Cumulative Mass fraction Oversize	Cumulative Mass fraction Undersize
d_{avg1}	x_1	$X_1 = x_1$	$Y_1 = 1 - X_1$
d_{avg2}	x_2	$X_2 = x_2 + x_1$	$Y_2 = 1 - X_2$
d_{avg3}	x_3	$X_3 = x_3 + X_2$	$Y_3 = 1 - X_3$
d_{avg4}	x_4	$X_4 = x_4 + X_3$	$Y_4 = 1 - X_4$
d_{avg5}	x_5	$X_5 = x_5 + X_4$	$Y_5 = 1 - X_4$



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



Moisture content

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



Chemical properties of MSW

- Proximate analysis: moisture, volatiles, ash and fixed carbon
- Ultimate analysis: C, H, N and O
- Fusing point of ash
- Lignocellulosic composition
- Leaching properties (applicable to Hazardous waste fraction in MSW waste):
- Energy content: Heating value



Proximate analysis

- **Moisture:** Loss in weight of sample due to drying at 105°C for 24h
- **Volatile matter:** Loss in weight of moisture free sample ignite in muffle furnace at 950 °C for 7 minutes in the absence of oxygen in an close crucible
- **Ash:** weight of residue left after combustion of dry sample in muffle furnace at 750 °C for half an hour in the presence of oxygen in an open crucible
- **Fixed carbon:** Obtained by subtracting moisture, volatile matter and ash percent from 100.

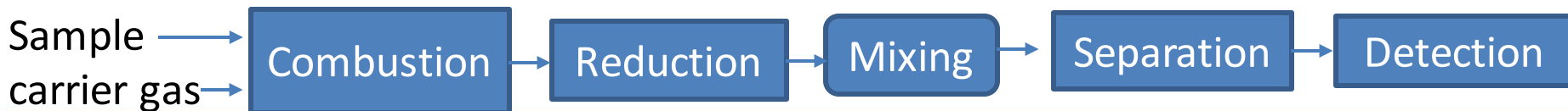
Types of waste	Moisture (%)	Volatiles (%)	Fixed Carbon (%)	Ash (%)
Mixed food	70.0	21.4	3.6	5.0
Mixed paper	10.2	75.9	8.4	5.4
Mixed plastics	0.2	95.8	2.0	2.0
Yard waste	60.0	42.3	7.3	0.4
Residential MSW	21.0	52.0	7.0	20.0



Ultimate analysis

Ultimate analysis or elemental analysis (CHNS) is determined by the CHNS analyzer

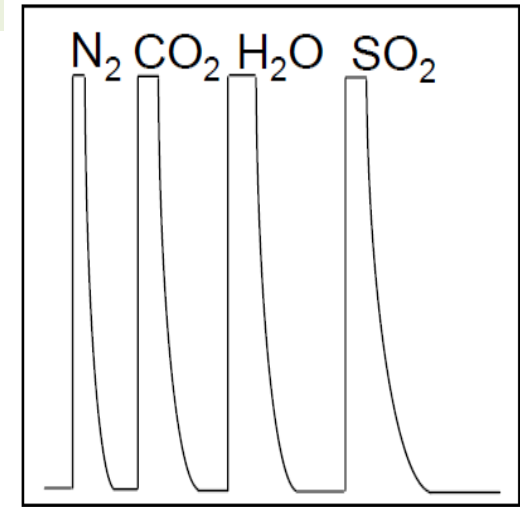
- The principle of CHNS analyzer depends on the tendency that all atoms prefer to be in their oxidation states. In presence of pure oxygen and high temperatures of 990°C , all carbon easily burn to become carbon dioxide, all hydrogen burn to become water and all nitrogen become N_2 gas/various nitric oxides.
- For analysis product gas mixture flows through a silica tube packed with copper granules. This zone held at about 500°C remaining oxygen is bound and nitric/nitrous oxides are reduced. The leaving gas stream includes the analytically important species CO_2 , H_2O and N_2 . Eventually SO_2 is absorbed at appropriate traps.



Ultimate analysis

- High purity helium is used as carrier gas. Finally the gas mixture is brought to a defined pressure/volume state and is passed to a gas chromatographic system. Separation of the species is done by so called zone chromatography. In this technique a staircase type signal is found. Step height is proportional to the substance amount in the mixture.

Type	C	H	O	N	S	Ash
Mixed food	73.0	11.5	14.8	0.4	0.1	0.2
Mixed paper	43.3	5.8	44.3	0.3	0.2	6.0
Mixed plastic	60.0	7.2	22.8	-	-	10.0
Yard waste	46.0	6.0	38.0	3.4	0.3	6.3
Refused Derived Fuel (RDF)	44.7	6.2	38.4	0.7	>0.1	9.9



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 SiO_2 , CaO , K_2O , MgO and Al_2O_3 in biomass ash.



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:

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

Fuel Processing Technology 85 (2004) 1201 – 1211



➤ 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:

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

Fuel Processing Technology 85 (2004) 1201 – 1211



- 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

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

Fuel Processing Technology 85 (2004) 1201 – 1211



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 multiphasic wastes.

8 Heavy Metals

- ❖ Arsenic
- ❖ Barium
- ❖ Cadmium
- ❖ Chromium
- ❖ Lead
- ❖ Mercury
- ❖ Selenium
- ❖ Silver

12 Organic Solvents

- ❖ Benzene
- ❖ Carbon tetrachloride
- ❖ 2,4-dinitrotoluene
- ❖ Hexachloroethane
- ❖ Methyl ethyl ketone
- ❖ Nitrobenzene
- ❖ Tetrachloroethylene
- ❖ Trichloroethylene
- ❖ Pyridine
- ❖ Chloroform
- ❖ Vinyl chloride
- ❖ Cresol(s)



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

17 Pesticides

- ❖ Chlordane
- ❖ 2,4-Dichlorophenoxyacetic acid
- ❖ Cchlorobenzene
- ❖ 1,4-dichlorobenzene
- ❖ 1,2-dichlorethane
- ❖ 1,1-dichloroethylene
- ❖ Endrin
- ❖ Heptachlor

- ❖ Hexachlorobenzene
- ❖ Lindane
- ❖ Hexachlorobutadiene
- ❖ Methoxychlor
- ❖ Pentachlorophenol
- ❖ Toxaphene
- ❖ 2,4,5-trichlorophenol
- ❖ 2,4,6-trichlorophenol
- ❖ 2,4,5-TP (Silvex)

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

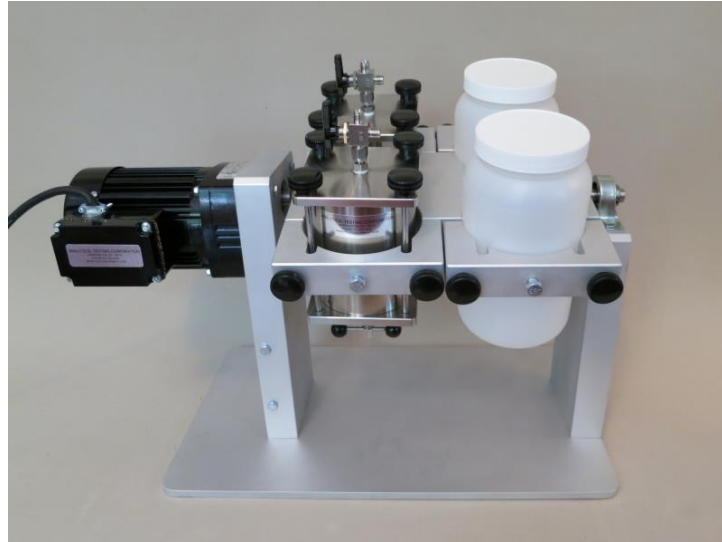
Zero-Headspace Extraction Vessel (ZHE):

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>)

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>

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 .

Thanks...

