WASTEWATER CHARACTERIZATION, SAMPLING AND ANALYSIS

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WASTER WATER

- Wastewater is the term used for discarded or previously used water from a municipality or industry.
- The wastewater that is produced due to human activities in households is called domestic wastewater i.e. wastewater from the kitchen, bathroom, toilet and laundry.
- Such water usually contains dissolved as well as suspended matter and must be treated prior to its discharge into natural water.

Waste water characterization

Quantitative assessments of the quality of wastewater are made by considering many criteria, including temperature, dissolved oxygen level and concentration of organic as well as inorganic compounds.

SIGNIFICANT WASTEWATER CONTROL PARAMETERS

Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Total Organic Carbon (TOC), Alkalinity, Chlorides, Nitrogen, Oil and Grease, Dissolved Oxygen, pH, Phosphorus, Gases, Sulphur, Solids, Temperature, Metals as well as Micro-organisms.

Domestic wastewater and its characteristic

Wastewater produced due to human activities in households is called domestic wastewater i.e. wastewater from the kitchen, shower, wash basin, toilet, washing machines and laundry. Wastewater is historically called sewage.

Sources of Domestic wastewater

The strength and composition of the domestic wastewater dependent on per capita water usage, habits, diet, living standard and life style.

Effluent discharge standards for sewage treatment plant

Sl. No.	Parameter	Parameter limit
1	pH	6.5-8.5
2	BOD (mg./lit.)	Not more than 10
3	COD (mg./lit.)	Not more than 50
4	TSS (mg./lit.)	Not more than 20
5	NH_4-N (mg./lit.)	Not more than 5
6	N-Total (mg./lit.)	Not more than 10
7	Feed coliform (MPN/100)	Less than 100

Note:

1. These standards will be applicable for discharge in water resources as well as for land disposal.

The standards for Fecal Coliform may not be applied for use of treated sewage in industrial purpose.

Classification of Domestic wastewater

- **yellow water:** human urine
- - **brown water:** human faeces with flushed water
- - **black water:** human faeces (brown water) mixed with urine (yellow water), in general: wastewater from toilets. It contains human waste and can be a public health risk if not treated properly.
- - **grey water:** water used in the kitchen, bathroom including sinks, baths, showers and laundry, etc. or any water that has been used at home, except water from toilets

Components present in domestic wastewater

Component	Of special interest	Environmental effect
Microorganisms	Pathogenic bacteria, virus and worms and eggs	Risk when bathing and eating shellfish
Biodegradable Organic material	Oxygen depletion in rivers and lakes	Fish death, odours
Other Organic material	Detergents, pesticides, fat, oil and grease, colouring solvents and phenols	Toxic effect, aesthetic inconveniences, bio accumulation in in food chain.
Nutrients	Nitrogen, phosphorus, ammonium	Eutrophication, oxygen depletion, toxic effect
Metals	Hg, Pb, Cd, Cr, Cu, Ni	Toxic effect, bioaccumulation
Other inorganic materials	Acids, hydrogen sulfide, bases	Corrosion, toxic effect
Thermal effects	Hot water	Changing living conditior for flora and fauna
Odour (and taste)	Hydrogen Sulphide	Aesthetic inconveniences, toxic effect

Physical quality of Domestic Waste water

- Physically, domestic wastewater is usually grey colour, musty odour and has solids content .
- The solid material is a mixture of faeces, food particles, grease, oil, soap, salts, metals, detergents, sand and grit. The solids can be suspended (about 30%) as well as dissolved (about 70%).
- Dissolved solids can be precipitated by chemical and biological processes.
- The suspended solids can lead to the development of sludge deposits and anaerobic conditions when discharged into the receiving environment.

Chemical quality of Domestic Waste water

- Chemically, wastewater is composed of organic (70%) and inorganic (30%) compounds as well as various gases. Organic compounds consist primarily of carbohydrates (25%), proteins (65%) and fats (10%), which reflects the diet of the people. Inorganic components may consist of heavy metals, nitrogen, phosphorus, acids, alkalis, sulphur, chlorides, toxic compounds, etc.
- Since wastewater contains a higher portion of dissolved solids than suspended, about 85 to 90% of the total inorganic component is dissolved and about 55 to 60% of the total organic component is dissolved.
- Gases, commonly dissolved, are hydrogen sulphide, methane, ammonia, oxygen, carbon dioxide and nitrogen. The first three gases result from the decomposition of organic matter present in the wastewater.

Biological quality of Domestic Waste water

- Biologically, wastewater contains various microorganisms but the ones that are of concern are those classified as protista and plants. The category of protista includes bacteria, fungi, protozoa, and algae.
- Plants include ferns, mosses, seed plants and liverworts. Invertebrates and vertebrates are included in the animal category.
- Also, wastewater contains many pathogenic organisms which generally originate from humans who are infected with disease or who are carriers of a particular disease.
- Typically, the concentration of faecal coliforms found in raw wastewater is about several hundred thousand to tens of million per 100 ml of sample.

Definition and measurement of wastewater parameters

1. Solids:

- Solids typically include inorganic matter such as silt, sand, gravel, and clay, and organic matter such as plant fibres and microorganisms from natural and man made sources.
- Classified by their size and state, chemical characteristics, solids can be dispersed in water in both suspended and dissolved forms. In regards to size, solids in wastewater can be classified as suspended, settleable, colloidal, or dissolved.
- They are also characterised as being volatile or non-volatile.
- There are different analytical procedures for analysing solids in wastewater such as settling, filtration, and evaporation; because of their different particle sizes

Total solids

- **Total solids (TS)** in wastewater is the amount of all solids, which are determined by drying a known volume of the sample in a pre-weighed crucible dish at 105 °C. After cooling in an exsiccator, the crucible dish is again weighed. TS is determined by using the following formula:
- TS = (M1–M2)/V
- *with*
- *M1 : mass of crucible dish after drying at 105 °C (mg)*
- *M2 : mass of initial crucible dish (mg)*
- *V : Volume of sample (L))*

Volatile solids

- **Volatile solids (VS)** are the amount of solid that volatilises when heated at 550 °C. This is a useful estimation for organic matter present in wastewater and is determined by burning the total solid at 550°C for about 2 hours in a muffle furnace. After cooling in an exsiccator to room temperature, it is weighed.

  VS is determined by using the following formula:

  VS = (M1–M3)/V

  with

- *M1 : mass of crucible dish after drying at 105 °C (mg)*
- *M3 : Mass of crucible dish after ignition at 550 °C (mg))*
- *V : Volume of sample (L))*

Fixed solids & Suspended solids

- **Fixed solids (FS)** are the amount of solid that does not volatilise at 550 °C. This measure is used to gauge the amount of mineral matter in wastewater. It is the difference between TS and VS. It can be divided in a suspended and a filterable fraction.
- **Suspended solids (SS)** are the solids retaining in a filter and is usually determined by filtration using glass fibre filters. In all analytical procedures for determination of suspended solids, weighed filters are used for sample filtration, the filters are dried at about 105°C after filtration, cooled in an exsiccator to room temperature and the weight of the loaded filter is determined. SS is determined by using the following formula:

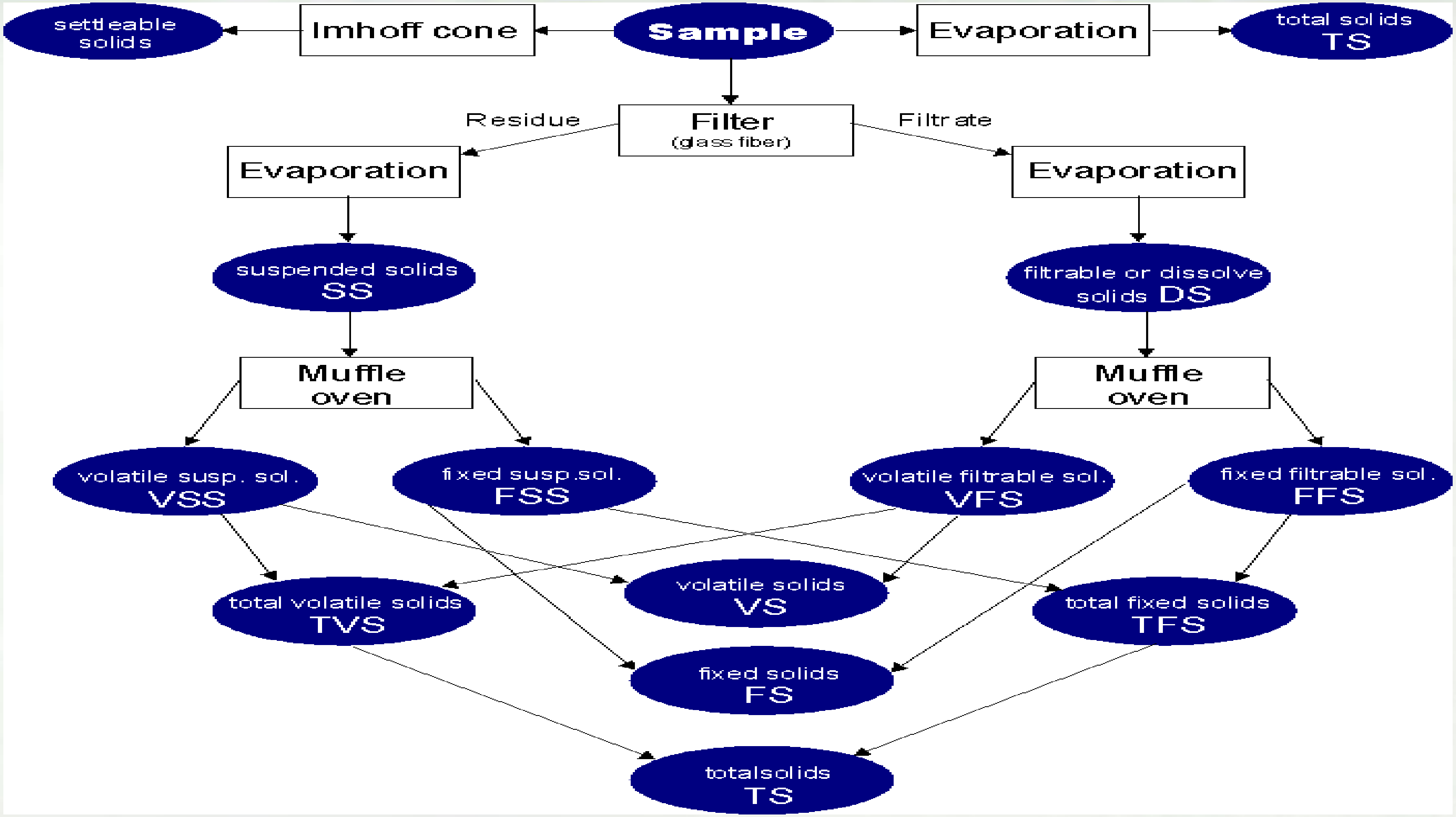
$$SS = (M4 - M5)/V$$

- *M4 : mass of filter after drying at 105 °C (mg), M5 : mass of initial filter (mg) , V : Volume of sample (L))*

Volatile suspended solids

- **Volatile suspended solids (VSS)** are, one portion of SS which are defined as that part of SS which can be removed by heating the solids at 550°C in a muffle furnace. The suspended solids is burned at 550°C for 2 hours in a muffle furnace and weighed after cooling in an exsiccator to room temperature. VSS is determined by using the following formula:
- VSS=(M4–M6)/V
- *M4 : mass of filter after drying at 105 °C (mg)*
- *M6 : mass of filter after ignition at 550 °C (mg)*
- *V : Volume of sample (L))*

- **Fixed suspended solids (FSS)** are the solid that are unburnable at 550 °C and is determined by subtracting VSS from SS.
- **Dissolve solids (DS)** or **filterable solids** can be determined by subtracting SS from TS. The solids passing through the filter consist of colloidal and dissolved solids.
- **Settable solids** are those solids that will settle to the bottom of an Imhoff cone (a cone shaped container) in one hour and determined by allowing a wastewater sample to stand for one hour in an Imhoff cone which enables to read the volume of the settled solids. It is expressed as mL/L and is important, because it is related to the efficiency of sedimentation tanks.


settleable solids
Imhoff cone
Sample
Evaporation
total solids TS
Residue
Filter (glass fiber)
Filtrate
Evaporation
Evaporation
suspended solids SS
filtrable or dissolve solids DS
Muffle oven
Muffle oven
volatile susp. sol. VSS
fixed susp.sol. FSS
volatile filtrable sol. VFS
fixed filtrable sol. FFS
total volatile solids TVS
volatile solids VS
total fixed solids TFS
fixed solids FS
totalsolids TS

COLOUR

In wastewater, colour is an indicator of the condition of the wastewater. Condition refers to the age of the wastewater, which is determined qualitatively by its colour and odour. Fresh wastewater is a light brownish-grey colour. The colour of wastewater changes sequentially from grey to dark grey and ultimately to black as the travel time in collection system increases (flow becomes increasingly more septic) and more anaerobic conditions develop.

ODOUR

- In wastewater, odours are of major concern, especially to those who reside in close proximity to a wastewater treatment plant. These odours are generated by gases produced by decomposition of organic matter or by substances added to the wastewater. Odour from fresh wastewater is less objectionable than the odour from wastewater that has undergone anaerobic decomposition. The most characteristic odour of stale or septic wastewater is that of hydrogen sulphide (H2S), which is produced by anaerobic microorganisms that reduce sulphate to sulphide.
- The malodorous compounds responsible for producing objectionable odours in water can be detected by diluting a sample with odour free water until the least detectable odour level is achieved. This is recorded as TON (Threshold Odour Number). The concentration of malodorous gases such as hydrogen sulphide, ammonia, mercaptans etc. emitted into the air from wastewater can be measured by any commercially available gas monitor.

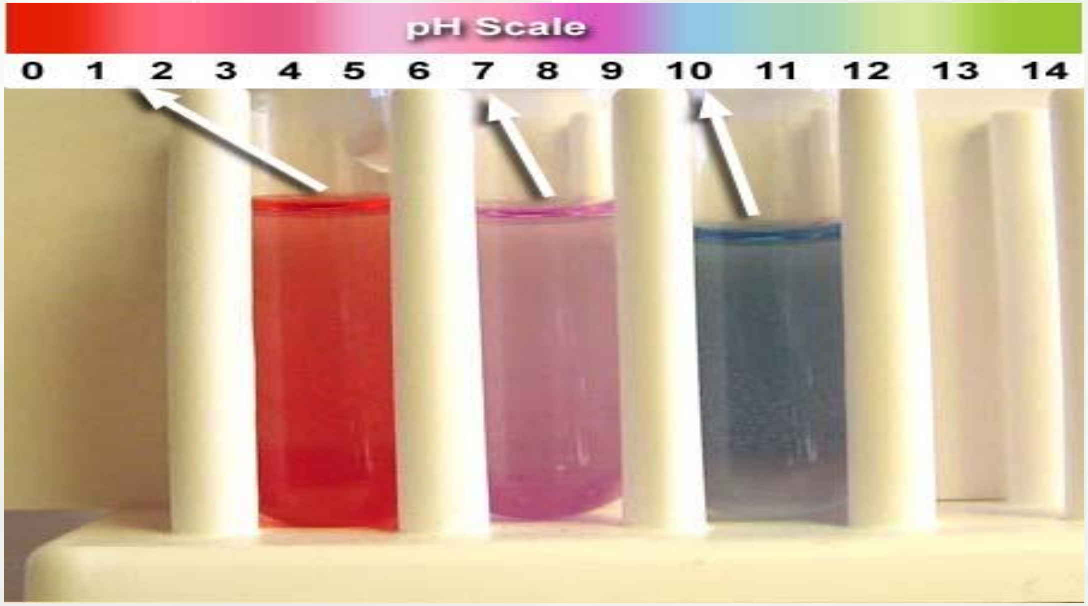
TEMPERATURE

- Temperature affects the efficiency of the biological process. Most biota are acclimated to the specific temperature ranges in their immediate environment. While fishes and aquatic plants can thrive within a naturally occurring variation of temperatures, artificially induced temp changes effect their metabolism. The dissolved oxygen concentration decreases with higher temperatures.
- Growth and propagation are impaired when a change in temp of about 3^{0} is artificially induced by the discharge of water at an elevated temperature.

pH

pH describes intensity of acidity or alkalinity of water solution or sample. Mathematically, it is the negative logarithm of the Molar Hydrogen ion concentration. Ions are chemical units with electrical charges.

Hydrogen ions (H+) make a water solution acidic, hydroxyl ions (OH-) make the solution alkaline. Very few organisms survive at pH below 3.0 and above 11.0. he tolerance level of organisms is between 6.5 and 8.5.



ALKALINITY

- Alkalinity is the capacity of water to neutralise acids. It results from the presence of hydroxides, carbonates, and bicarbonates of elements such as calcium, magnesium, sodium, potassium, or ammonia.
- It is determined by titrating against a standard acid and the results are expressed in terms of calcium carbonate $CaCO_3$, mg/l as $CaCO_3$.
- Alkalinity plays an important role in the treatment of wastewater, as it indicates the buffer capacity of water. This affects the growth and activity of microbes present in activated sludge, which are responsible for the treatment of wastewater. It is also an essential parameter to be estimated to design and implement the corrosion and odour control processes.

Dissolved oxygen

- Dissolved oxygen is the amount of molecular oxygen dissolved in water. It is required for the respiration of aerobic microorganisms. However, oxygen is only slightly soluble in water. The actual quantity of oxygen (other gases too) that can be present in solution is governed by;

❖ The solubility of gas

❖ The partial pressure of the gas in the atmosphere

❖ The temperature.

❖ The concentration of the impurities in the water (e.g., salinity, suspended solids, etc.).

The concentration of DO in water is small and therefore precarious from ecological point of view .

The Dissolution process:

O_2 (gas)⇔O_2 (Dissolved)

The amount of DO decreases with increasing water temperature. So a cool or cold water can contain much more DO than the warm water. As a result, aquatic life, in streams and lakes is placed under more oxygen stress during summer months than during the other seasons.

Organic compounds

Definition:

All organic compounds contain carbon in combination with one or more elements.

General Characters:

- Usually combustible
- Have lower melting and boiling points
- Less soluble in water
- Have very high molecular weight
- Most organic compounds can serve as a source of food for micro-organisms

Source(s):

•Nature: fibres, vegetable oils, animal oils and fats, cellulose, starch, sugar.

•Synthesis: A wide variety of compounds and materials prepared by manufacturing processes. E.g. DDT, polyvinyl chloride.

•Fermentation: Alcohols, acetone, glycerol, antibiotics, acids.

Classification of Organic mater:

- Biodegradable organics
- Non-biodegradable organics

Biodegradable organics:

Food for micro-organisms

Fast and easily oxidized by micro-organisms

e.g. starch, fat protein, alcohol, human and animal waste.

Non-biodegradable organics :

Difficult and much more longer to biodegrade

Or toxic to micro-organisms

e.g. PVC, pesticide, industrial waste, cellulose, phenol, lignic acid.

Effect(s):

- Depletion of the dissolved oxygen in the water
- Destroying aquatic life
- Damaging the ecosystem

Some organics can caused cancer:

Trihalomethane (THM-carcinogenic compound) are produced in water and wastewater treatment plants when natural organic compounds combine with chlorine added for disinfection purposes.

Normally, wastewater has high organic content. The organic content are measured by Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD) and Total organic carbon (TOC)

Biochemical Oxygen Demand

- This test is a measurement of the amount of oxygen required, in a 5-day period by the microorganisms in consuming the organic material in the Waste water.
- BOD is simply stated, the amount of oxygen used by the microorganisms as they biologically decompose organic matter in wastewater. The larger the organic contamination, the larger the amount of oxygen that will be needed by these organisms. So BOD is an indirect measure of the concentration of organic contamination in water.
- It has a widest application in measuring water loading to treatment plants and in evaluating the efficiency (BOD removal) of treatment systems.
- BOD analysis does not tell all of the organic matter present in the waste but only the organic matter that is biochemically degradable (oxidisable) during n days time period at 20°C.. The day period is given as index in BODn.

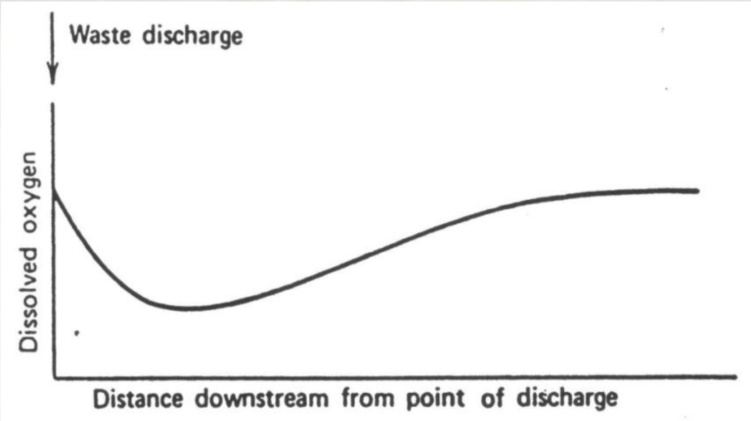
Continuation of BOD

- BOD5 is the most widely used parameter of organic pollution applied to wastewater and is used:
- To determine the approximate quantity of oxygen that will be required to biologically stabilise the organic matter present,
- Too measure the efficiency of some treatment processes.
- The standard for usual measurements is a 5-day period.
- High BOD value = high organic-matter concentration = poor water quality.
- Decomposition of organic matter is a slow process
- In 20 days, 95 to 99% of organic matter decomposes.
- In 5 days, 60 to 70% of organic matter decomposes.

- $BOD_5 = DO_5 - DO_0$
- where DO_0 = DO before incubation (day 0) DO_5 = DO after 5 days of incubation at 20ºC (day 5)
- BOD_5 for domestic sewage = several hundreds mg/L
- BOD_5 for industrial sewage = several thousands mg/L
- when the sewage is discharged to water, the Dissolved oxygen will be depleted quickly.

Initial stage :

DO curve drops (i.e. rate of O_2 consumption by bacteria > rate of re-aeration with atmosphere)



At the point where [DO] = minimum,
Rate of consumption of O_2 = rate of re-aeration.

Beyond Minimum Point:
rate of consumption < rate of re-aeration (DO level eventually returns to normal

This sequence is called **"natural self-purification of water"**

Chemical Oxygen Demand

- ❖Chemical Oxygen Demand is measure of the amount of oxygen needed to decompose all the organic materials, in the wastewater, in to Carbon dioxide and water.
- ❖The equivalent amount of oxygen required to oxidise organic matter present in a water sample to CO2 by means of a strong chemical oxidising agent is called chemical oxygen demand (COD). COD is used to measure the content of organic matter of wastewater.
- ❖The COD values include the oxygen demand created by biodegradable as well as non-biodegradable substances. As a result, COD values are greater than BOD.
- ❖In comparison with BOD5, COD measurement has an advantage in that it requires a short digestion period of about 3 hours rather than incubation of 5 days period required for BOD5 measurement.

For many types of wastes, it is possible to correlate COD with BOD. Once the correlation has been established, COD measurements can be used as good advantage for treatment-plant control and operation.

Total organic carbon

- Another means for measuring the organic matter present in water is the TOC test, which is especially applicable to small concentrations of organic matter. Wastewater content of carbon bound in organic molecules is the TOC (total organic carbon). Organic carbon comprises nearly all carbon compounds except a few carbon species which are looked at as inorganic (carbon dioxide, hydrogen carbonate, carbonate, cyanide, which are not commonly found in wastewaters)

Interrelationship between BOD, COD and TOC

Typical values for the ratio of BOD/COD for untreated municipal wastewater are in the range from 0.3 to 0.8. If the BOD/COD ratio for untreated wastewater is 0.5 or greater, the waste is considered to be easily treatable by biological means. If the ratio is below/ about 0.3, either the waste may have some toxic components or acclimated microorganisms may be required in its stabilization. The corresponding BOD/TOC ratio for untreated wastewater varies from 1.2 to 2.0. In using these ratios it important to remember that they will change significantly with the degree of treatment the waste has undergone.

Comparison of ratios of various parameters used to characterize wastewater

Type of wastewater	BOD/COD	BOD/TOC
Untreated	0.3 – 0.8	1.2 – 2.0
After primary settling	0.4 – 0.6	0.8 – 1.2
Final effluent	0.1 – 0.3	0.2 – 0.5

Nitrogen compounds

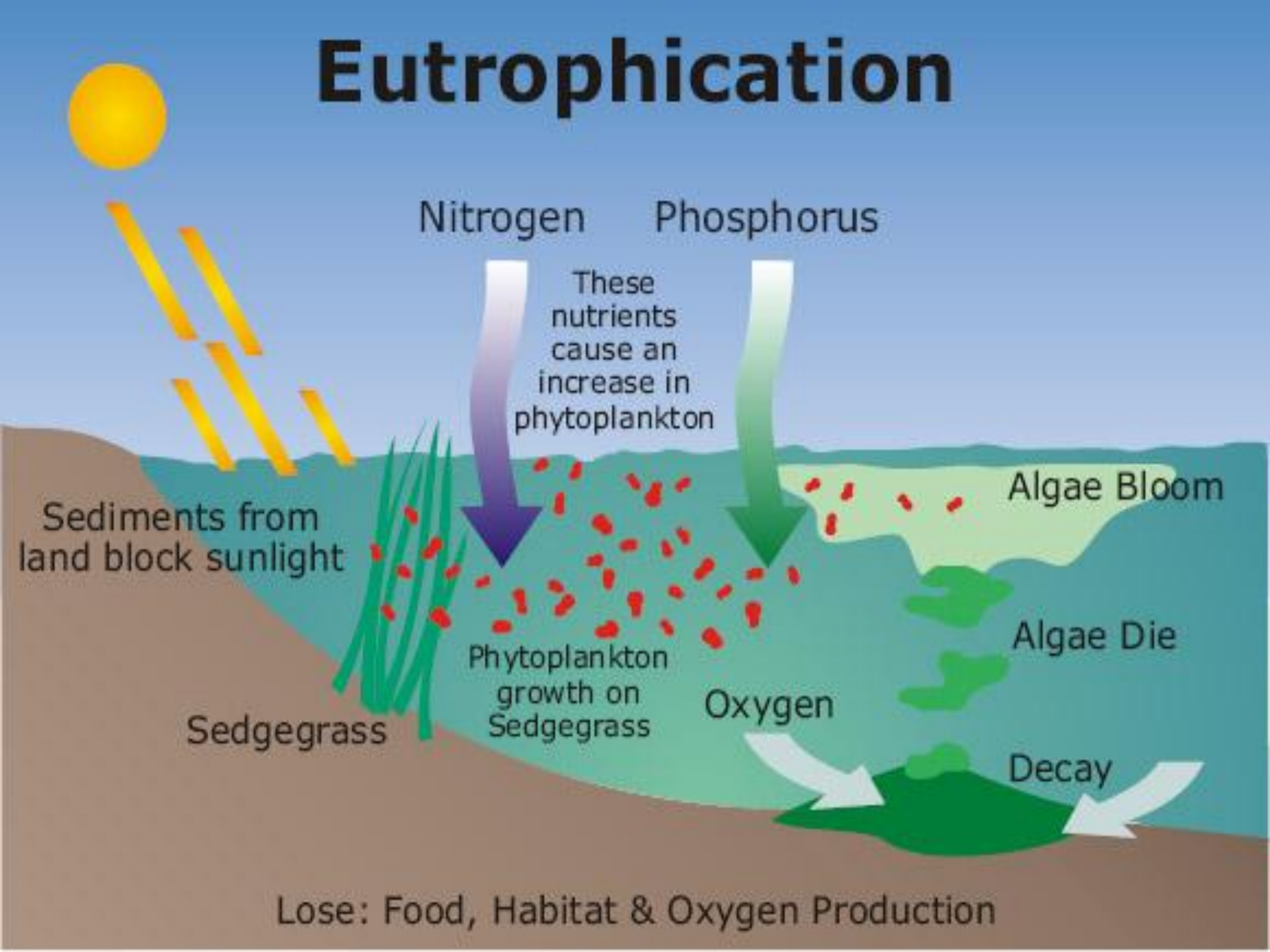
Nitrogen compounds, with environmental relevance frequently analyzed in wastewater, are ammonia, nitrite, nitrate, and Kjeldahl nitrogen. Ammonia discharged to surface water can be nitrified in the aqueous environment if nitrifying microorganisms are present. The nitrifying bacteria consume dissolved oxygen for this process, thus depleting the oxygen content of the surface water with the consequence of massive dying of fish. Moreover, if the pH of the surface water is in the alkaline range, NH3 is formed which is toxic towards fish. The nitrate ion represents a nutrient leading to eutrophication of surface water, and nitrite is toxic and can react with amines (formed e.g. from amino acids of proteins) to yield N-nitosoamines which represent powerful carcinogens. Kjeldahl nitrogen is a sum parameter of compounds containing the nitrogen atom with an oxidation number of -3 (ammonia, amines and many other organic nitrogen compounds). It thus comprises organic nitrogen compounds besides ammonia nitrogen. This is also an important nitrogen parameter, because organic nitrogen compounds can be metabolized to ammonia.

Phosphorus

- Phosphorus is essential to the growth of algae and other biological organisms. The amount of phosphorus compounds present in wastewater discharge has to be controlled in order to avoid noxious algal blooms occurred in surface water. The usual forms of phosphorus found in aqueous solutions include the orthophosphate, polyphosphate, and organic phosphate.

EUTROPHICATION

After seeing the picture of children swimming in a sea of seaweed, you will surely wonder what strange phenomenon has hit the coast of Qingdao in eastern China. It is an abnormal growth of algae, a clear manifestation of a process called eutrophication.



Eutrophication
Nitrogen
Phosphorus
These nutrients cause an increase in phytoplankton
Algae Bloom
Sediments from land block sunlight
Algae Die
Phytoplankton growth on Sedgegrass
Oxygen
Sedgegrass
Decay
Lose: Food, Habitat & Oxygen Production

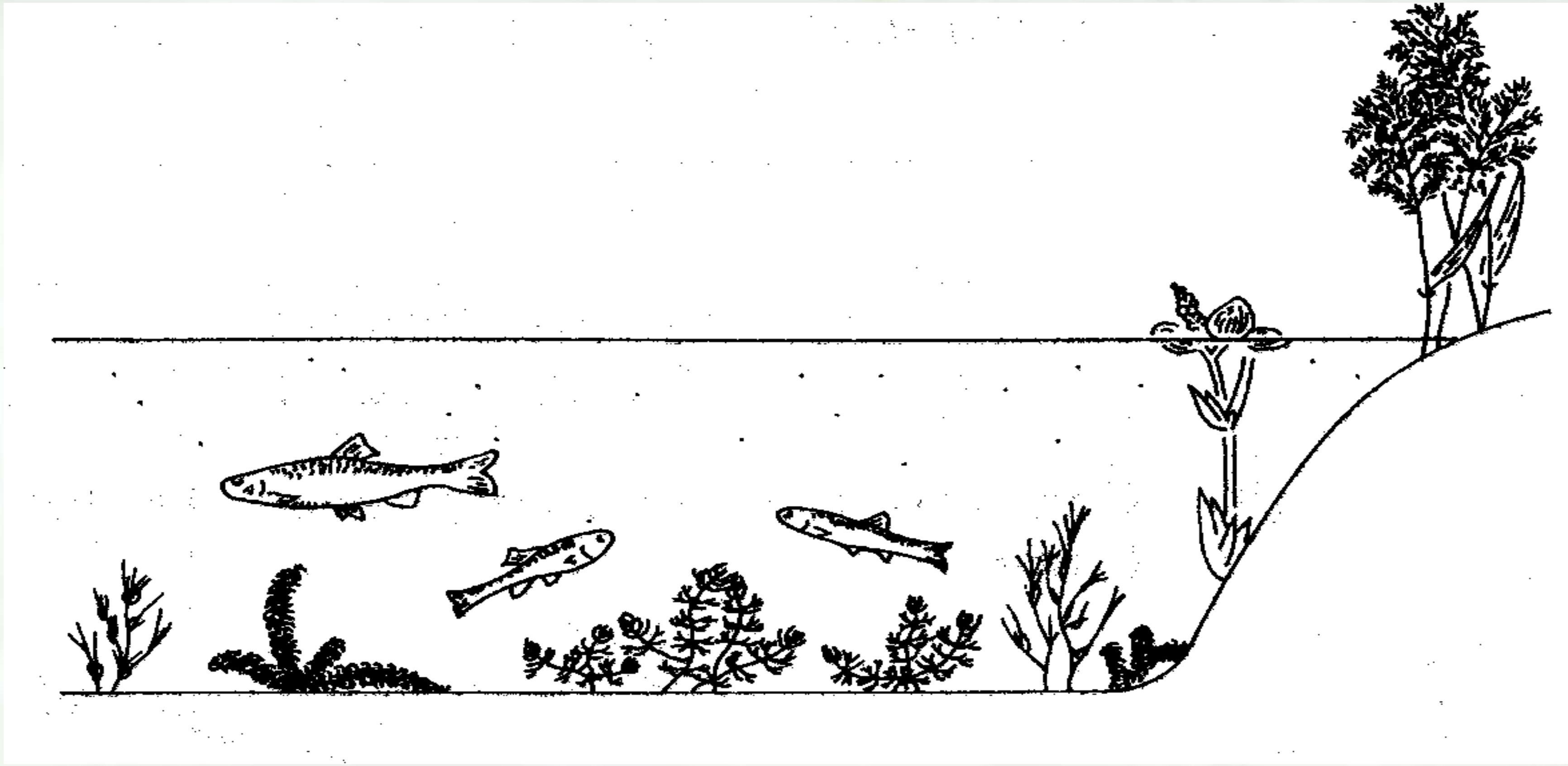
Eutrophication is an enrichment of water by nutrient salts that causes structural changes to the ecosystem such as increased production of algae and aquatic plants, depletion of fish species, general deterioration of water quality and other effects that reduce and preclude use".

According to the Survey of the State of the World's Lakes, a project promoted by the International Lake Environment Committee, eutrophication affects 54% of Asian lakes, 53% of those in Europe, 48% of those in North America, 41% of those in South America and 28% of those in Africa.

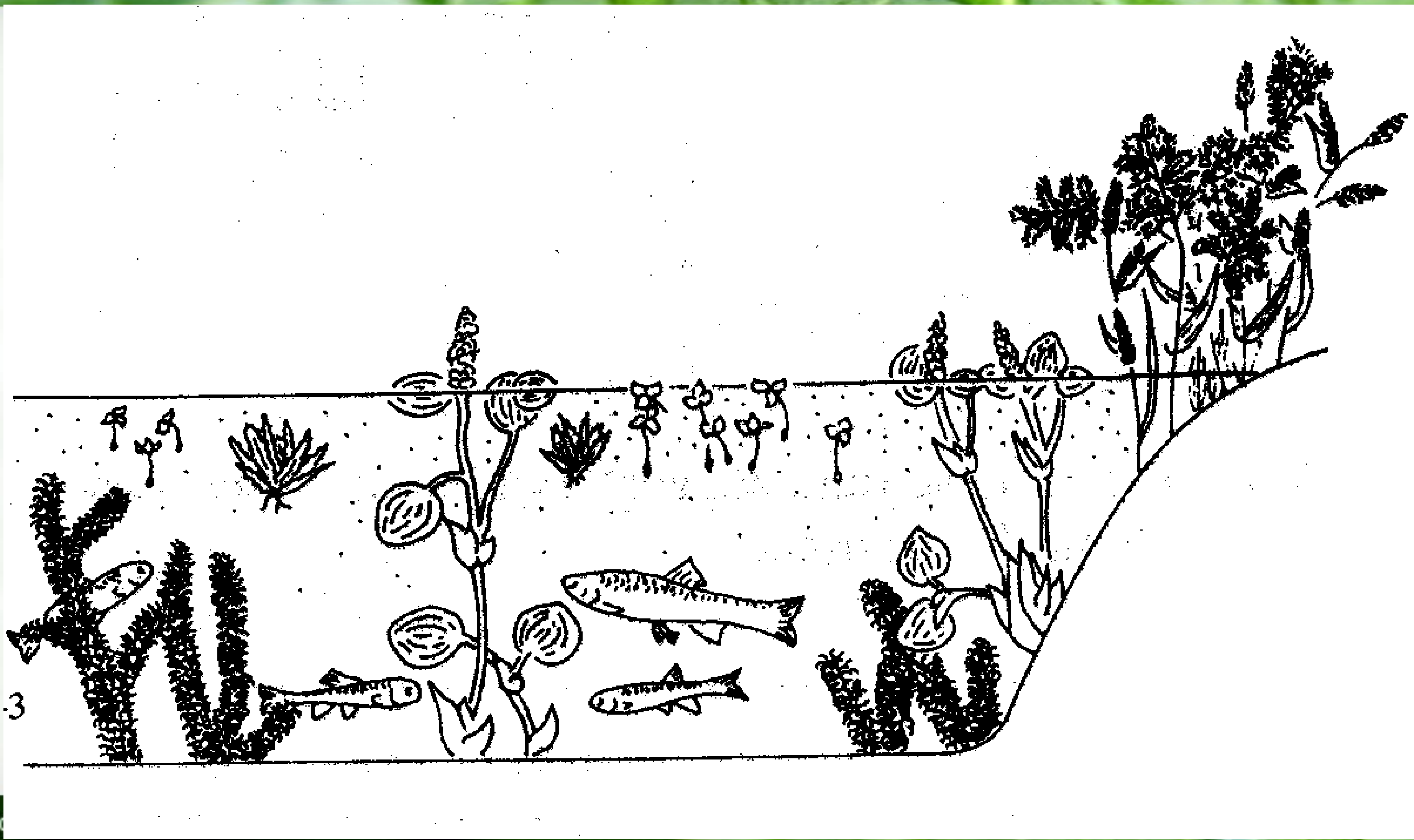
Three Factors:

- ❖Usage of fertilizers.
- ❖Discharge of un treated waste water in to water bodies.
- ❖Reduction of self purification capacity of lakes.

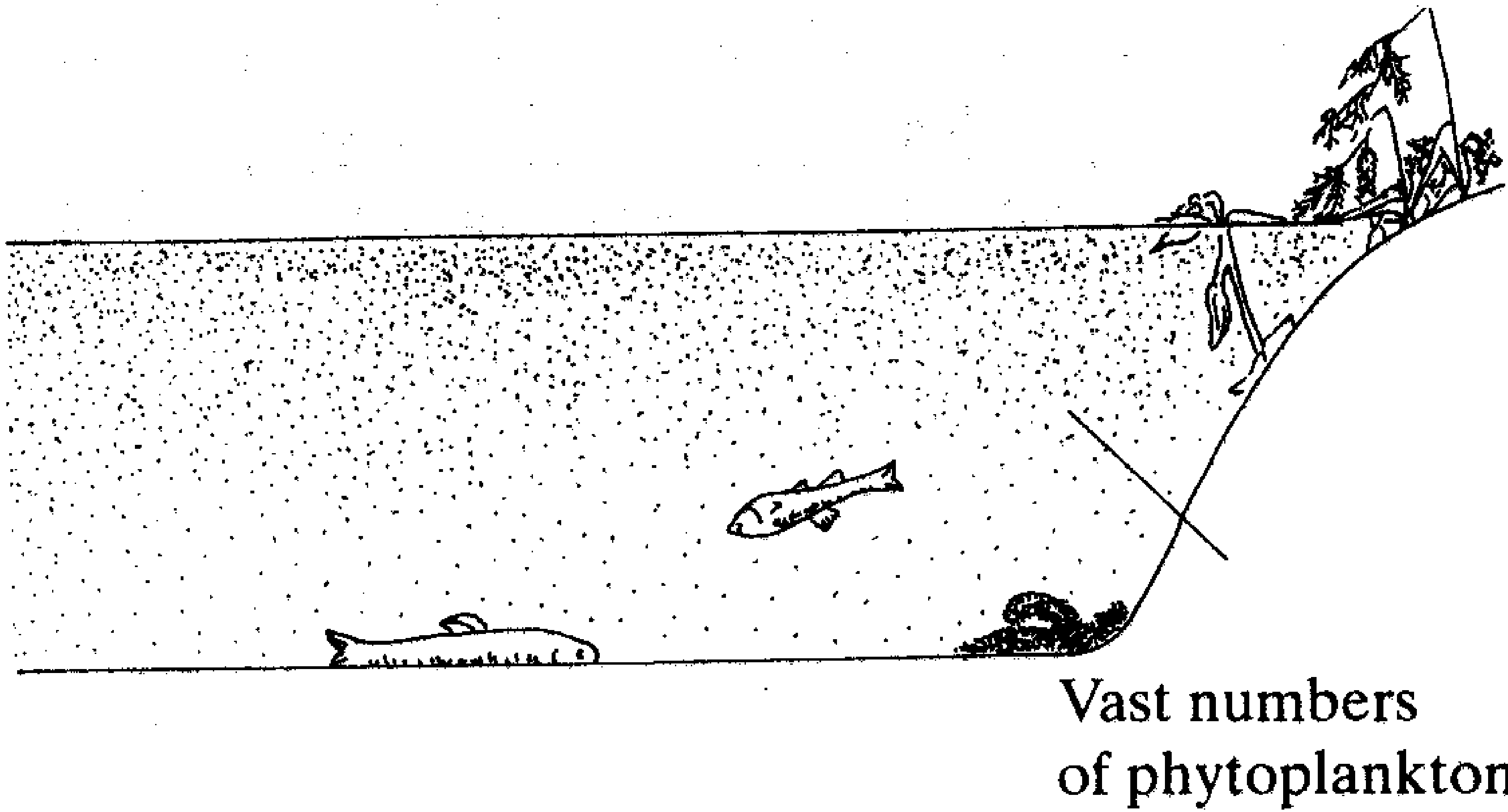
Before increases in nutrients

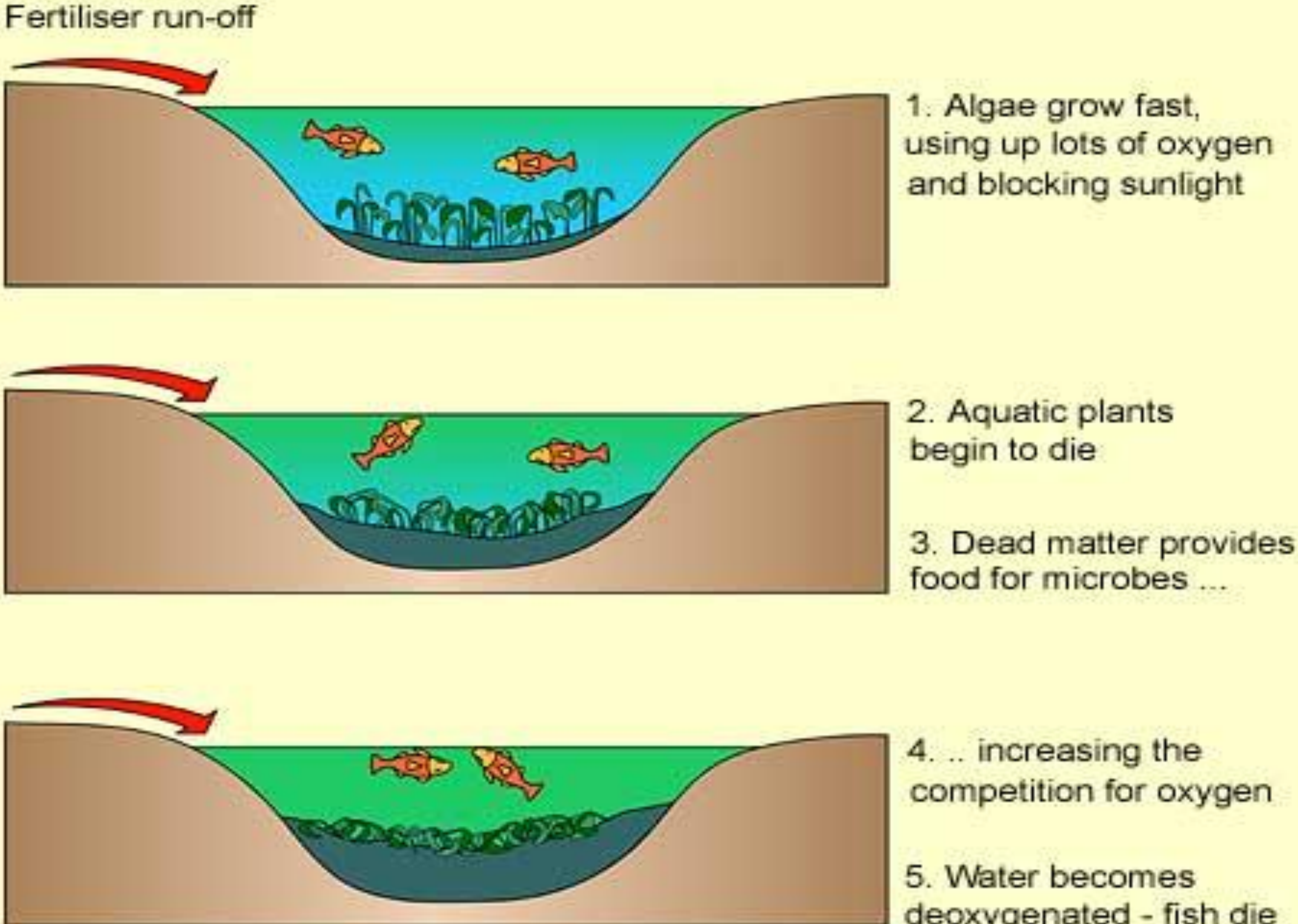


Acceleration of eutrophication :



Results of eutrophication :




Fertiliser run-off
1. Algae grow fast, using up lots of oxygen and blocking sunlight
2. Aquatic plants begin to die
3. Dead matter provides food for microbes ...
4. .. increasing the competition for oxygen
5. Water becomes deoxygenated - fish die

Oil and Grease

- Oils, fats, waxes and fatty acids are the major constituents included in this category in domestic wastewater. The presence of a significant amount of oil and grease in wastewater hinders the transportation of wastes through pipelines. It causes scum in aeration basins of activated sludge plants, which interferes with the biological oxidation of wastes and produces a low quality settling sludge.
- Oil and Grease form a thin layer on top of the water surface which effectively interferes with the oxygen transfer mechanism between air and water body. Grease and Oil also dries up and forms unsightly deposits on beaches, ports and harbors. Grease and Oil of mineral origin are relatively resistant to biodegradation and will require removal by methods other than biological treatment

Gases

Gases commonly found in untreated wastewater include nitrogen (N_2), oxygen (O_2),carbon dioxide (CO_2), hydrogen sulfide (H_2S), ammonia (NH_3), and methane (CH_4). The first three are common gases of the atmosphere and will be found in all waters exposed to air. The latter three are derived from the decomposition of the organic matter present in wastewater

Sulphur

- The sulphate ion occurs naturally in most water supplies and is present in wastewater as well.
- Sulphate is reduced biologically under anaerobic conditions to sulphide, which in turn can combine with hydrogen to form hydrogen sulphide (H_2S).
- The following generalised reactions are typical.

Organic matter + SO_4^{2-} by bacteria gives S^{2-} + H_2O + CO_2

❖ S^{2-} + $2H^+$ gives H_2S (gas)

- The H_2S accumulated in sewers can be oxidised to sulphuric acid, which is corrosive to sewer pipes.

HEAVY METALS

- **LEAD** is a substance commonly used in paint preparation, drinking water pipes, various metallic implement such as gasoline additive.
- **COPPER** is toxic to algae, marine plants, invertebrates and fishes. This is the reason why copper compounds are often used to control algal growth in cooling towers. Copper has a lesser effect on mammals. Aside from toxicity, copper imparts an undesirable taste to water. While a concentration of 0.2mg/L can already be toxic to some fishes, the recommendation limit is 1.0 mg/L for effluent discharges because of expected dilution effects.

HEAVY METALS

- **HEXAVALENT CHROMIUM** is highly toxic although the trivalent from is less toxic. It is commonly used for chrome plating and highly toxic to all life forms.

- **MERCURY** is perhaps the most widely known poison among the heavy metals due to the Minamata diseases discovered in Japan. Organo-metallic or methyl mercury has the highest toxicity while free mercury used in thermometer has the lowest toxic level of the various forms of mercury.

COLIFORMS

- Coliform organisms (or E. Coli) are the indicator organisms of fecal contamination in any water body. The presence of coliform organisms may signify fecal contamination from warm blooded animals.
- Normal raw sewage contains up to 1,000,000 MPN/100 ml. MPN is the most probable number.
- Coliforms are removed from the effluent by chlorination. However, chlorine gas reacts readily with most organic substance to form chlorinated hydrocarbon which may have higher environmental and health related problems.

Collection and Preservation of Water and Wastewater Samples

The objective of sampling is to collect a portion of material small enough in volume to be transported conveniently and yet large enough for analytical purposes while still accurately representing the material being sampled.

General Requirements

1. Meat the requirements of the sampling program.
2. Handle sample so that it does not deteriorate or become contaminated or compromised before it analyzed.
3. Ensure sampling equipments are clean and quality assured before use.
4. Use sample containers that are clean and free of contaminants.

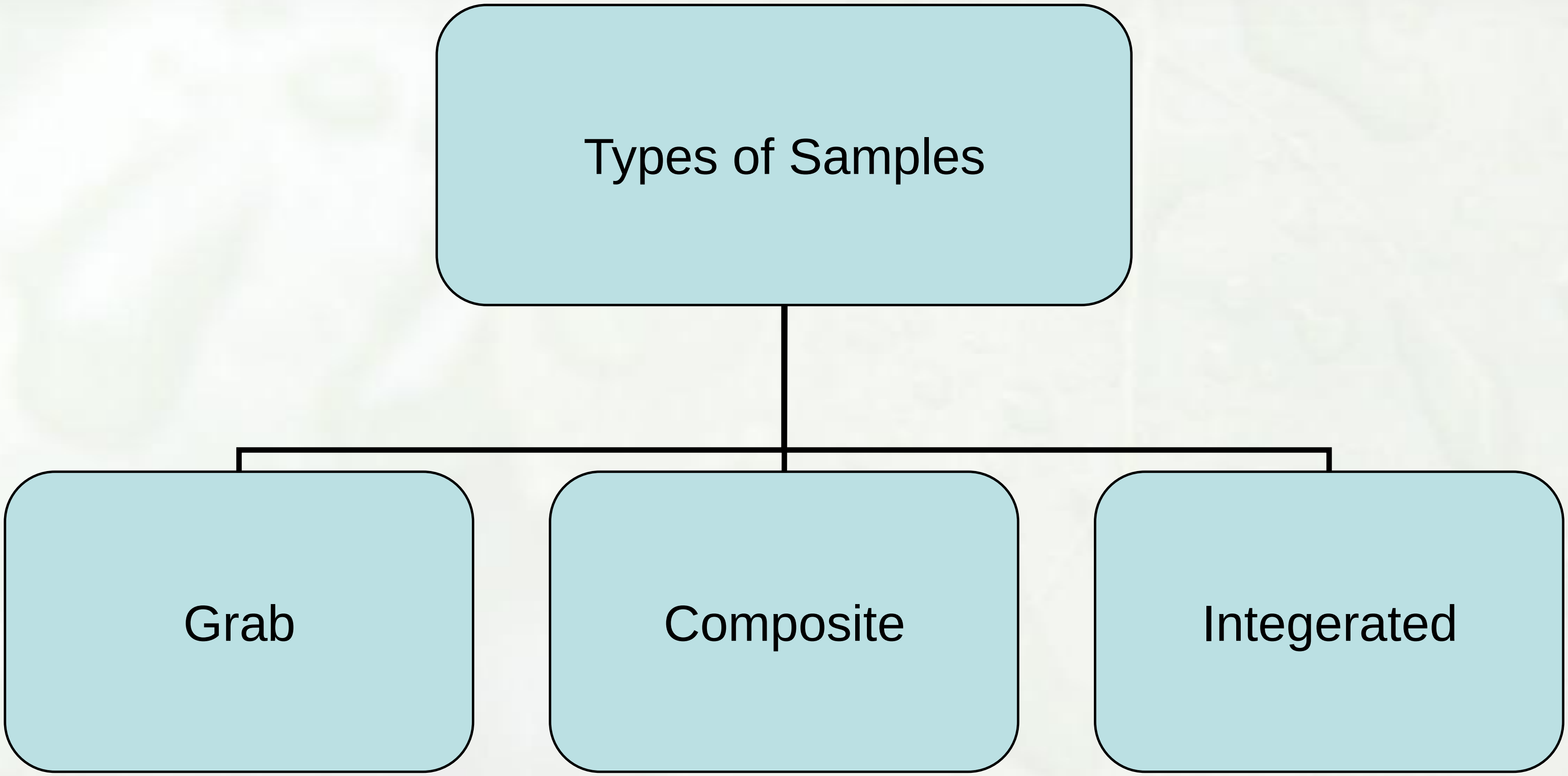
5. Fill sample containers with/or without pre-rinsing with sample.

6. Composite samples can be obtained by collecting over a period of time, depth, or at many different sampling points.

7. Make a record of every sample collected and identify every bottle.

8. Label bottles and document sufficient information for sample identification.

9. Before collecting samples from distribution systems, flush lines with 3-5 pipe volumes (or until water is being drawn from the main source).


Types of Samples
Grab
Composite
Integerated

Grab Samples

Grab samples are single samples collected at a specific spot at a site over a short period of time (typically seconds or minutes).

Composite Samples

Combination of grab samples collected at the same sampling point at different times.

Composite sample can be obtained by combining portions of multiple grab samples or by using specially designed automatic sampling devices.

Provides a more representative sampling of heterogeneous matrices in which the concentration of the analytes of interest may vary over short periods of time and/or space.

Advantages of Composite Samples

- ✓Reduced costs of analyzing a large number of samples .
- ✓more representative samples of heterogeneous matrices,
- ✓and larger sample sizes when amounts of test samples are limited.

Disadvantages of Composite Samples

- Loss of analyte relationships in individual samples.
- potential dilution of analytes below detection levels
- increased potential analytical interferences
- and increased possibility of analytes interactions.

Integrated (discharge-weighed) Samples

Mixture of grab samples collected from different points simultaneously, or as nearly so as possible, using discharge-weighed methods

Chain-of-Custody Procedures

a-Sample labels (including bar-code labels).

b-Sample seals.

c-Field log book.

d-Chain-of-custody record.

e-Sample analysis request sheet.

f-Sample delivery to the laboratory.

g-Receipt and logging of sample.

h-Assignment of sample for analysis.

i-Disposal.

Sample Container

Collect samples in a glass or plastic bottles that have been cleansed and rinsed carefully, given a final rinse with deionized or distilled water, and sterilized.

Volume of the sample should be 1000 ml for physico – chemical analysis and 125 ml. for bacteriological analysis.

Parameters to be determined in Situ :

- Temperature
- Dissolved oxygen.

Parameters to be determined immediately after sample collection:.

- pH
- Electrical Conductivity
- Turbidity

Zero head-space is important in preservation of volatile organic compounds.

Sample Containers Considerations

- Silica , sodium, and boron may be leached from soft glass but not plastic.
- Trace levels of some pesticides and metals may sorb onto the walls of glass containers.
- Use glass containers for all organic analyses such as volatile organics ,semi volatile organics ,pesticides, PCB’s ,and oil and grease.
- Some analytes are light sensitive ,collect them in amber glass containers to minimize photodegradation.Plastic caps also can be a problem.

What may happen during sample transportation ?

Certain cations are subject to loss by adsorption on, or ion exchange with the walls of the glass containers, these includes: Al,Cd,Cr,Cu,Fe,Pb,Mn, Ag,and Zn.

For testing these parameters in water samples, the samples are to be collected in a separate clean bottle and acidified with nitric acid to pH below 2.0 to minimize precipitation and adsorption on container walls.

What may happen during sample transportation ?

Changes in the pH-alkalinity-carbon dioxide balance may cause calcium carbonate to precipitate, decreasing the values of calcium and total hardness.

Hardness can be preserved by adding nitric acid to pH <2

What may happen during sample transportation ?

Biological activity taking place in a sample may change the oxidation state of some constituents

Changes caused by growth of microorganisms are greatly retarded by keeping the sample at a low temperature (<4 C) but above freezing.

What may happen during sample transportation ?

Chlorine will oxidize cyanide.

Dechlorinate sample ,
add NaOH to pH >12,
And refrigerate in dark.

Preservation and Storage

Start microbiological examination of water samples as soon as possible after collection.

Analyze samples on day of receipt whenever possible and refrigerate overnight if arrival is too late for analyzing on same day.

THANK YOU