

Determination of the Physiochemical Properties of Sediment Samples

Fresh sediment samples were used for determination of pH, temperature, total organic carbon, available phosphorus, organic Nitrogen, salinity, Nutritive salts and particle size. Acidity, chloride content, sulphates, nitrates, dissolved oxygen and nitrite five days, biochemical oxygen demand (BOD₅) etc were determined in water samples.

pH

This was determined at the point of collection using pH – meter embedded in multi combi meter.

Temperature

Fast changing parameter like this was measured in the field using thermometer embedded in multicombi meter.

Available Phosphorus

The estimation of phosphorus was carried out using the (Effiong, 2005) method about 0.5g of finely ground well mixed sediment samples were weighed into different 100ml Telfon beaker. About 5ml conc. H₂SO₄ was added and swirled gently. 3ml of 30% H₂O₂ was added in 0.5ml portions and swirled vigorously. When the reaction subsided 1ml of hydrofluoric acid was added in 0.5ml portions and swirled gently. The beaker was placed on a hot plate at 150°C for 10-12mins to eliminate excess H₂O₂. After cooling, the contents of the flasks were transferred quantitatively to a 50ml quantitative flask, passing it through a quantitative filter paper. Two additional washings of the beaker were made each with 10ml distilled water. These were filtered and made up to exact volume.

Determination of Total Organic Carbon

The carbon content of the sediment samples was determined using Walkely and Black (1934) method. 1.0g of each sediment was weighed into 500ml conical flask each. 10ml of 1N K₂Cr₂O₇ and 20ml of conc. H₂SO₄ was added. The flasks were swirled carefully and allowed to stand for 30minutes. 200ml of distilled water and 10ml H₂PO₄ was slowly added. 1ml of diphenylamine indicator was added and titrated against 0.5N ferrous ammonium sulphate solutions until green colour started to appear indicating the end point.

Calculation:

$$\text{Organic Carbon (\%)} = \frac{10 (B - S) \times 0.39 \times \text{mcf}}{B \times W} \quad \text{Equation 3}$$

Where:

B = ml of ferrous ammonium sulphate solution used for blank

S = ml of ferrous ammonium sulphate solution used for the sample

mcf = Moisture correction factor

W = sample weight (g)

0.39 = conversion factor (including a correction factor for a supposed 70% oxidation of organic carbon).

Determination of Chloride Content

Chloride content in sediment was determined titrimetrically by silver nitrate method or Argentometric method (APHA, 1998). The method requires titrating an aliquot portion of sediment suspension in the presence of potassium chromate as indicator to brick-red end point with silver nitrate that was standardized with sodium chloride.

Determination of Nitrate Content in Sediment

Nitrate content in sediment and water samples were determined calorimetrically by UV/visible HACH spectrophotometer (APHA, 1998) the method is based on a yellow complex formation between brucine sulphate and nitrate in the presence of sulphuric acid. The measurement was taken at a wave length of 400nm. The spectrophotometer was calibrated with working standards from neat potassium nitrate salts.

Determination of Salinity in Sediment Samples

The sediment samples were air-dried and percentage salinity determined from silver thiourea (AgTu) extracts and 0.1M. Silver nitrate (AgNO₃) titration using potassium chromate as indicator and calculated as total water soluble salts (chloride + sulphates) (APHA, 1998).

Determination of Sulphates Content

The sulphate content of sediment was determined turbidimetrically with UV/visible HACH spectrophotometer at wavelength of 425nm (APHA, 1998). The method is based on precipitation of sulphate ion in an acetic acid medium with barium chloride (precipitating agent). The reaction formed barium sulphate crystals of uniform size light absorbance of the barium sulphate suspension was measured and the SO₄²⁻ concentration in Mg⁻¹ was determined by comparison with standards.

Carbonates Determination

Bicarbonates (CO₃²⁻) was determined by titrimetric method (APHA, 1998). A known quantity of the sediment sample was measured into conical flasks and 3 drops of bromocresol green indicator added. This was titrated with 0.01N solution of sulphuric acid. The titre values of the titration were used to calculate the bicarbonate concentration in sample using the relationship:

$$\text{HCO}_3 = \frac{V \times E \times N \times 1000}{\text{Volume of sample}} \quad \text{Equation 4}$$

Where, V = titre value

E = equivalent weight of bicarbonate

N = Normality of sulphuric acid

Determination of Particle Size in Sediment Samples

The hydrometer method of APHA (1998) was used to determine the grain size of sediment. The method requires the determination of particles size, larger than 63μm by sieving and particle sizes smaller than 63μm were determined using hydrometer with calgon (sodium hexametaphosphate) as the displacing agent. The hydrometer was calibrated so that the corrected reading gives the grams of sediment materials in suspension. The sand settled to the bottom of the cylinder within 40 seconds. Therefore, the 40 seconds hydrometer reading gave the amount of silt and clay in suspension. The weight of sand in the sample was obtained by subtracting the corrected hydrometer reading from the total weight of the sample.

$$\% \text{ sand} = \frac{\text{weight of sand}}{\text{Weight of sample}} \times 100 \quad \text{Equation 5}$$

To determine the percentage clay in the sample, the suspension was re-shaken and the hydrometer reading was taken at the end of 2 hours and corrected. The corrected hydrometer reading therefore represents the gram of clay in the sample.

% sand = 100% - (% sand + % clay).