CHAPTER-IV

MATERIALS AND EXPERIMENTAL METHODOLOGY
A bench scale slurry phase batch reactor was used in the present study to treat the soil contaminated with transformer oil with different soil masses.

4.1 Materials

4.1.1 Contaminated Soil Sample Site

Karnataka Power Transmission Co. Ltd. is storing large numbers of transformer and transformers oil containers at FTS Mysore. At the station the transformers are also reconditioned. It is observed that the site in and around the transformer station is contaminated with transformer oil and the soil colour has changed to black. Plate 4.1 and Plate 4.2 shows the contamination of soil in and around the transformer station.

4.1.2 Collection of Sample

The contaminated area of about 1m x 1m was marked. Top layer of soil, which contains debris, was removed and the soil was collected using a tool upto a depth of 15 cm. The contaminated soil was collected in a thick quality polythene bag and immediately transferred to laboratory. In the laboratory the pH and moisture content of soil were measured.

4.1.3 Extraction of PAHs and PCBs from Contaminated Soil

PAH and PCBs were extracted from contaminated soil using Soxhlet extraction method. Acetone was used as an extraction solvent for PAHs and PCBs. The soil sample was mixed with anhydrous sodium sulfate placed into an extractor thimble and extracted using Soxhlet extractor for about 12 to 16 hours. The extracted solvent was concentrated by opening the sample to the atmosphere. Concentrated solvents were analyzed using gas chromatography.
Plate 4.1 Transformer Station and Soil Contamination.

Plate 4.2 Contaminated Soil in Service Station.
4.2 Experimental setup

A slurry phase batch reactor made of plexiglass material of working volume 8L capacity fixed on a stand was placed in the laboratory at room temperature (22^oC to 30^oC) used for the treatment of contaminated soil. Mechanical stirrer was used to keep the soil particles in suspension throughout the treatment period. Aeration was done from the bottom of the reactor just below the stirrer. Compressor motor was used to pass the compressed air to the reactor. Two ports placed at different heights of the reactor were used to collect the sample for analysis. Fig.4.1 and Plate 4.3 shows the schematic representation and photographic view of a bench scale slurry phase reactor respectively.

![Fig. 4.1 Schematic Diagram of Slurry Phase Batch Reactor](image)

Mutnents

- Microbial Culture
- Contaminated soil
- Mechanical stirrer
- Compressed air
- plexiglass material
- Reactor

Fig. 4.1 Schematic Diagram of Slurry Phase Batch Reactor
4.3 Analytical Methods

The performance of slurry phase batch reactor was evaluated by measuring parameters like pH, oxidation-reduction potential (ORP), dissolved oxygen (DO), chemical oxygen demand (COD), polychlorinated biphenyl (PCB), and Polyaromatic hydrocarbons (PAH). Chemical analysis were performed as per Standard Methods for Examination of Water and Wastewater (2005). pH was measured with a digital pH meter. ORP was measured with an ORP meter (platinum with silver chloride as reference electrode, saturated KCl). DO was measured by titrimetric method, COD was measured by closed reflux titrimetric method, Phosphate and nitrate concentrations were measured using spectrophotometer. PAHs and PCBs were analysed using gas chromatography. Sieve analysis and moisture content of soil were carried out as per the methods prescribed by Pumninia and Ashok (1988).
4.4 Gas Chromatography

The instrument used for analysis of PAHs was Varian Star 3400 gas chromatograph equipped with flame ionization detector. The column used in this analysis was BP X – 5 Cap. Col. (25m x 0.22mm) with nitrogen as the carrier gas. The temperature of injector and detector was 290°C and oven temperature was raised up to 240°C with the increment in temperature at the rate of 8°C/min from initial temperature of 160°C. The sample was injected into gas chromatography in micro liters.

For analysis of PCBs 30m x 0.25mm fused silica capillary column was used. The injection port was maintained at temperature of 280°C and the detector at temperature of 300°C, Carrier gas Helium (He) with a flow rate of 6mL/min, make up gas Nitrogen (N₂), flow rate of 20mL/min was maintained. With oven temperature was programmed from 50°C for an initial time of 4 min to 150°C at a rate of 4°C/min during the first stage and it was increased to 280°C with an increase of 10°C/min, during the final stage.

4.5 Experimental Methodology

The contaminated soil diluted to predetermined percentage soil mass was mixed with the acclimated biomass, prepared by mixing the sewage and cow dung was used for experimentation. The nutrients (Urea and DI Potassium OrthoPhosphate) were added into the reactor to maintain required C:N:P ratio.

The slurry was then mixed and aerated throughout the treatment period. The mixing of slurry was done using variable speed mechanical stirrer and speed of the stirrer was adjusted to achieve good mixing. The air was supplied using air compressor and was introduced below the stirrer. The DO concentration in the reactor was adjusted to maintain 2 mg/L for entire treatment period. The mixing was then stopped and solid particles were allowed to settle. Removal efficiency of the reactor for the soil masses of 20%, 25% and 30% for 20 days and 30%, 35% and 40% for 25 days and 45 days were tried.
4.6 Design and Fabrication of the Bio-Slurry Reactor

For laboratory analysis the capacity of reactor was assumed as 8 liters

\[
\text{Volume of reactor is (V)} = 8 \text{ l} \\
= 8/1000 \\
= 0.008 \text{ m}^3
\]

Assume diameter of reactor as 20 cm

\[
\text{Dia of reactor (D)} = 20 \text{ cm} \\
= 0.2 \text{ m}
\]

\[
\text{The area of reactor (A)} = \pi D^2/4 \\
= (\pi \times 0.2^2)/4 \\
= 0.031 \text{ m}^2
\]

\[
\text{Depth of reactor (d)} = \frac{V}{A} \\
= 0.008/0.031 \\
= 0.25 \text{ m}
\]

Provide 10 cm free board

\[
= 0.25+0.1 \\
= 0.35 \text{ m}
\]

The size of the reactor is 0.2 m X 0.35 m (Diameter x Depth)