CHAPTER - III

MATERIALS AND METHODOLOGY
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To meet the objectives of the present research work, a detailed experimental programme was worked out and is discussed in this chapter. The issues considered to carry out research work include:

- Adsorbents used and their preparation.
- Metals studied and preparation of aqueous solution.
- Experimental variables.
- Analysis of parameters.
- Experimental setup.

3.1 ADSORBENTS USED

Four adsorbents were used for experimentation and were Saw dust, Wheat Bran, Sugarcane leaves and Coconut husk. An attempt has been made to evaluate the efficiency of removal of metals using two grades of each adsorbent mentioned.

3.1.1 PREPARATION OF ADSORBENTS

Based on the literature review, the procedure adopted by the other researchers for preparation of adsorbents was used to prepare the adsorbents in the present research work.

The saw dust collected from nearby saw mill was washed with several times with distilled water. Washed saw dust was then treated with 0.1 M aqueous solution of Disodium hydrogen phosphate for 24 hours. Further, the treated saw dust was filtered and washed several times with distilled water till no phosphate is released in the washing. It was then dried at 40°C in a oven and was used for experimentation (Bilquees et.al., 1999).

The coconut husk was cut into small pieces, blended and extracted with hot water several times until the supernatant was colorless. It was then dried at 70°C. The bio material was sieved to obtain required particle sizes and this was stored in clean air tight containers (Oyedeji et.al., 2010). The coconut husk procured from a
local coir factory was treated with distilled water and then dried. About 25 gm of this sample was powdered and treated with 25 ml concentrated sulphuric acid and heated to 150°C for 6 hrs. The carbonized material was washed with distilled water and repeatedly soaked with 1% sodium bicarbonate solution to remove any residual acid. It was then filtered and washed to make it free from bicarbonates. The material was dried, sieved to get bicarbonate treated coconut husk (Anirudhan and Sreedhar : 1998). Further, based on pilot scale studies, the procedure proposed by Anirudhan and Sreedhara which showed consistently better performance was used for experimentation.

The procedure proposed by Artinigam and Rama (2003) was used for preparation of raw sugarcane leaves as adsorbent. Raw sugarcane leaves pieces were cut to sizes 1 cm long were soaked in distilled water for 24 hrs and then washed with distilled water several times and then completely dried under solar light. Further, the required grades were prepared by sieving.

Wheat brawn was sieved through IS sieves to get required grades and washed several times with distilled water and then they were treated with 0.1 M aqueous solution of disodium hydrogen phosphate for 24 hours. Further, they were filtered and washed several times with doubled distilled water till no phosphate was released in washing. Again, it was dried at 40°C and stored in desiccators and then used for experimentation whenever necessary (Singh, K.K., et.al., 2004).

3.2 METALS STUDIED

As mentioned in the objectives, four metals namely Lead, Nickel, Copper and Zinc were considered for study.

3.2.1 PREPARATION OF AQUEOUS SOLUTIONS

A synthetic nickel solution of 5 gm/l was prepared by dissolving 5 gm of analytical grade commercially available pure metal of nickel in 20 ml hot concentrated HNO₃, cooked and diluted with double distilled water upto 1 liter mark. The stock solution was further diluted with distilled water to concentration for obtaining the test solutions.
Copper stock solutions were prepared from an analytical grade copper sulphate pentahydrate using distilled water. pH values of aqueous solution were kept below pH 5.5 (except for pH study) in order to prevent the precipitation of hydroxyls. Further, 0.1 M sulphuric acid was used to adjust the required pH.

Stock solution of 1000 mg/l concentration was prepared by dissolving analytical grade Pb (NO₃)₂ with de-ionized water in 1% HNO₃ solution and this stock solution was diluted with de-ionized water to obtain the working standard solution.

Analytical grade zinc chloride was used for preparing aqueous solution of zinc by dissolving metal chloride in de-ionized water. The pH of the solution was adjusted by adding a small amount of 0.1 M HCl or NaOH.

3.3. VARIABLES CONSIDERED

The variables considered for experimentation in the present research work include pH, Flow rate, particle size of adsorbent and initial concentration of metals. The ranges of these variables considered are summarized in Table 3.1

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Variables</th>
<th>Ranges</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>pH</td>
<td>Lead &amp; Nickel: 2,4,6,8 &amp;10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Copper &amp; Zinc: 3,5,7,9 &amp; 10</td>
</tr>
<tr>
<td>2</td>
<td>Metal concentrations, mg/l</td>
<td>Lead: 2,4,6,8 &amp;10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Nickel, Zinc &amp; Copper: 10,20,30,40 &amp; 50</td>
</tr>
<tr>
<td>3</td>
<td>Flow Rate ,ml/min</td>
<td>20,40,60 &amp; 80</td>
</tr>
<tr>
<td>4</td>
<td>Grades of Adsorbents, mm</td>
<td>0.3 &amp; 1.0</td>
</tr>
</tbody>
</table>

3.4 ANALYSIS OF PARAMETERS

Synthetic metals and treated metal samples were analyzed for metal concentrations employing Atomic Adsorption Spectrophotometer (AAS) as per
standard methods and following manual provided by supplier of AAS. However, characteristics of adsorbents were collected from literature and recorded.

3.5 EXPERIMENTAL SETUP

Metal removal efficiency by various adsorbents considered for study was evaluated for varied experimental conditions using both upflow and downflow columns. The line diagrams of the experimental setups are shown in fig. 3.1 and 3.2. Plates 3.1 and 3.2 represent picturesque views of the experimental setup.

3.5.1 UP FLOW COLUMN STUDIES

The cylindrical Jar was used as column. A hole was drilled at the bottom of a column which was used for upflow studies. The hole was then covered with a fine mesh to take care of adsorbent washout. A small plastic tube was attached to the hole at the bottom. The column was filled with adsorbent upto predetermined depth. For upflow studies, the synthetic sample was fed at varied flow rates by adjusting the speed of Peristaltic Pump. The samples were fed from the over head tank in the case of downflow column studies. Overflow samples from the outlets fixed to the upflow columns were collected and analyzed. On the other hand, the samples were collected from the bottom in case of downflow studies. All experiments were triplicated and average values were tabulated. The results were represented by Graphs / Bar charts, analyzed and inferences were drawn.

3.5.2 DOWN FLOW COLUMN STUDIES

Graduated measuring jar was used as column for down flow experimentation. To collect the treated sample at the bottom, a hole was drilled and was covered with a fine mesh to arrest the washout of adsorbents. A plastic tube was also fixed at the bottom to enable the comfortable collection of treated sample.

Synthetic metal solutions were fed from a glucose bottle fixed to the stand in a inverted position. Drip set was used to adjust the flow rate. Effluent collected from the bottom of the column was analyzed for metal.
3.6 ISOTHERM STUDIES

Sorption equilibrium isotherms are useful for the estimation of amount of sorbent needed for sorbing required amount of sorbate from solution. Thus, an attempt has been made to carry out the experiments needed for development of isotherms.

Upflow columnar studies were carried out for development of isotherms. Studies were carried out for optimum conditions of removal efficiencies obtained from the present study. For copper and zinc studies were carried out at optimum condition, pH – 7. Accordingly the adsorbent used for zinc was saw dust and for copper was sugarcane leaves. Whereas for lead and nickel pH value adopted for isotherm studies was 4. Wheat brawn was used for lead and coconut husk was used for nickel. Equilibrium concentrations of metal (Ce) was recorded and thereby absorption at equilibrium was calculated. Based on the results Ce, Ce/Qe, log Ce and log Qe were calculated for the various metal concentrations. Langmuir isotherms were drawn with Ce against Ce/Qe and freundlich isotherm with log Ce against log Qe. Further based on R² values, the isotherms were validated.
fig: 3.1. Line Diagram of Experimental Setup (Up flow)
fig. 3.2: Line Diagram of Experimental Setup (Down flow)
Plate 3.1 : Experimental Setup (Upflow)
Plate 3.2 : Experimental Setup (Down flow)