CHAPTER - V

MATERIALS AND METHODS
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5.1 MATERIALS

5.1.1 Glasswares

The glasswares used in the present work were manufactured by M/s Borosil Glass Works Ltd., Bombay and marketed under the brand name of 'Borosil'. These were washed with chromic acid solution and rinsed thrice with tap water followed by distilled water before putting them to use.

5.1.2 Chemicals

The chemicals used were analytical reagent grade (A.R.), guaranteed reagent grade (G.R.) and laboratory reagent grade (L.R.). Details of the chemicals used in the present research work have been given in Table 5.1.

Table 5.1: Chemicals and Reagents Used

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Name of Chemicals</th>
<th>Grade</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Ammonium Chloride (NH₄Cl)</td>
<td>L.R.</td>
<td>Ranbaxy Laboratories Ltd., Bombay</td>
</tr>
<tr>
<td>2.</td>
<td>Ammonium Ferrous Sulphate (NH₄)₂SO₄·FeSO₄·6H₂O Mol. weight=392.14(99%)</td>
<td>A.R.</td>
<td>Ranbaxy Laboratories Ltd., Bombay</td>
</tr>
<tr>
<td>No.</td>
<td>Chemical Name</td>
<td>Supplier</td>
<td>Grade</td>
</tr>
<tr>
<td>-----</td>
<td>---------------------------------------</td>
<td>-----------------------------------</td>
<td>--------</td>
</tr>
<tr>
<td>3.</td>
<td>Calcium Chloride (CaCl₂)</td>
<td>L.R. Ranbaxy Laboratories Ltd.</td>
<td>L.R.</td>
</tr>
<tr>
<td>4.</td>
<td>Calcium Carbonate (CaCO₃)</td>
<td>Commercial L.R. Tayal Chemicals &amp; Mineral India</td>
<td>Commercial</td>
</tr>
<tr>
<td>6.</td>
<td>Ferric Sulphate Fe₂(SO₄)₃·9H₂O</td>
<td>L.R. Radial Chemicals India</td>
<td>L.R.</td>
</tr>
<tr>
<td>7.</td>
<td>Ferrous Sulphate FeSO₄·7H₂O</td>
<td>L.R. Ranbaxy Laboratories Bombay</td>
<td>L.R.</td>
</tr>
<tr>
<td>8.</td>
<td>Mercuric Sulphate (neutral) HgSO₄</td>
<td>L.R. Sarabhai M. Chemicals, Baroda</td>
<td>L.R.</td>
</tr>
<tr>
<td>9.</td>
<td>Magnesium Sulphate MgSO₄·7H₂O (99%)</td>
<td>L.R. (BDH) A Division of Glaxo Laboratories Ltd. Bombay</td>
<td>L.R.</td>
</tr>
<tr>
<td>10.</td>
<td>Manganese Sulphate (Monohydrate) MnSO₄·7H₂O</td>
<td>L.R. Sarabhai M. Chemicals, Baroda</td>
<td>L.R.</td>
</tr>
<tr>
<td>11.</td>
<td>Potassium Dichromate K₂Cr₂O₇ 99.9%</td>
<td>A.R. Radial Chemicals India</td>
<td>A.R.</td>
</tr>
<tr>
<td>12.</td>
<td>Potassium Dihydrogen phosphate KH₂PO₄</td>
<td>L.R. Ranbaxy Laboratories Ltd., Bombay</td>
<td>L.R.</td>
</tr>
<tr>
<td>13.</td>
<td>Polyacrylamide Mol. Weight = 5 x 10⁵</td>
<td>L.R. BDH Chemicals Ltd. Poole, Bombay</td>
<td>L.R.</td>
</tr>
<tr>
<td>14.</td>
<td>Polyacrylamide (BC-1)₅ Mol. weight 5.2 x 10⁵</td>
<td>Laboratory prepared Monomers Acrylamide &amp; Sodium acrylate</td>
<td>Laboratory prepared Monomers Acrylamide</td>
</tr>
<tr>
<td>15.</td>
<td>Polyacrylamide (SC-2) Mol. weight 3x10⁵</td>
<td>Laboratory prepared Monomers Acrylamide &amp; Sodium acrylate</td>
<td>Laboratory prepared Monomers Acrylamide</td>
</tr>
</tbody>
</table>
16. Polyacrylamide (SC-3)  
Mol. Weight = 4.5 x 10^5  
Laboratory prepared Monomers  
Acrylamide & Sodium acrylate.

17. 1,10, Phenanthrolin  
(Monohydrate)  
C_{12}H_{8}N_{2}H_{2}O  
G.R.  
Emerck Pvt. Ltd., Bombay

18. Sulphuric Acid  
H_{2}SO_{4}  99.5%  
L.R.  
Ranbaxy Laboratories Ltd., Punjab.

19. Sulphuric Acid  
H_{2}SO_{4}  99.9%  
A.R.  
Ranbaxy Laboratories Ltd., Punjab.

20. Sodium Thiosulphate  
Na_{2}S_{2}O_{3}. 5H_{2}O  
99.5%  
A.R.  
(BDH) A Division of Glaxo Laboratories Ltd., Bombay

21. Sodium Hydroxide  
(NaOH) Pallets 96%  
L.R.  
Ranbaxy Laboratories Ltd., Bombay

22. Sodium Azide  
NaN_{3}  99%  
L.R.  
LOBA Chemie Ind. Corp., Bombay.

23. Silver Sulphate  
Ag_{2}SO_{4}  
L.R.  
The Central Drug House, Delhi.

24. Soluble Starch  
(Potato)  
L.R.  
S.D. Fine Chem. Pvt. Ltd., Boisar

5.2 Equipment used

The equipments used in the present investigation are as follows:

5.2.1 Chemical Balance

Sartorius Electric Balance, Model 2432, type fabr.
with a range of 0.0001 gm to 200 gm manufactured by Sartorius Gmbh (Germany).

5.2.2 Mercury Thermometer

Thermometers used in the present work were:

1. Verix ASTM 3c thermometer having a range of 50°C to 400°C with a least count of 1°C was used for the measurement of the temperatures in higher range.

2. Jenson make, having a range of -10°C to 110°C with a least count of 1°C was used for the measurement of medium range temperature.

5.2.3 Nylon Tube

Nylon tube with 1.0 cm diameter was used as the connecting line between the condensers and filtration flask to vacuum pump.

5.2.4 Digital pH Meter

Digital pH meter 'Philips' model PP 9.46 with combination electrode Pt - 100 probe, electrode stand with following -

I. Philips PP 9046 digital pH meter.

II. Philips PV 9038 combination electrode with 1 m cable and BNC connector 0-14 pH/0-100°C for use with PP 9046.
III. Philips PV 9451 fast response stand Pt - 100 O.D. 3 mm with 1 mm cable and 2 x 4 mm banana plug for use with PP 9046.

5.2.5 Hot Air Oven

Tempo model, TI 130 FAS, with forced Air Circulation.

5.2.6 Air Dryer

35 x 35 x 35 cm Tempo model TI 125 Air Circulation type was used at temperature 100 ± 10°C.

5.2.7 Heater

Six heater Assembly for COD. Each heater was 1.0 KW capacity.

5.2.8 BOD Incubator

Tempo Model TI-91, 88.2 x 57 x 55.8 cm was used at 20°C.

5.2.9 Water Distillation Apparatus

Table Model, 'CEW' make, manufactured by CPN Industries, New Delhi, boiler and condensor both S.S. yield, 4 litre per hour, power rating, 3 KW, 220 V, Cat.No. TD 559.
5.2.10 Multiple Stirrer

M.B. stirrer having six paddles Model No. MB-FL-6, rpm range from 0 to 160, supplied by M/s Dass Scientific Corporation, Kanpur.

5.2.11 Vacuum Pump

Canco Hyvae Vacuum Pump manufactured by Central Scientific Co. Scientific Laboratories Instruments Apparatus, Chicago, USA, with maximum speed of 600 rpm and capacity 0.0003 mm was used for filtration.

5.2.12 Stop-watch

Racer Brand, antimagnetic, 7 jewels, made at Ellora Industries, Delhi having a range of 0.1 second to 15 minutes with a least count of 0.1 second stop watch was used.

5.3 EXPERIMENTAL METHODS

Following four types of polyelectrolytes and one inorganic coagulant were studied in the present investigation.

1. Straight chain polyacrylamide (SC-1) (Molecular Weight = $5.0 \times 10^5$) (Commercial)
2. Straight chain Poly[acrylamide-co-sodium acrylate SC-2] (Molecular weight = $3.0 \times 10^5$)

\[
\text{f CH}_2 - \text{CH} \text{CH}_2 - \text{CH} \text{CH}_2 - \text{CH} - \text{CONH}_2
\]

3. Straight chain Poly[acrylamide-co-sodium acrylate (SC-3)] (Molecular weight = $4.5 \times 10^5$)

\[
\text{f CH}_2 - \text{CH} \text{CH}_2 - \text{CH} \text{CH}_2 - \text{CH} - \text{CONH}_2
\]

4. Branched chain Polyacrylamide (BC-1)

Molecular weight = $5.2 \times 10^5$

5. Ferric chloride (Anhydrous) neutral in nature, an inorganic coagulant used for coagulation.

**Jar Test**

Jar test method was used to conduct various experiments to determine the effectiveness of various coagulants and flocculants, optimum dosage of coagulation and optimum pH. In this test upto six number of samples can be simultaneously subjected to varying chemical parameters under identical physical conditions of temperature, mixing time, mixing speed, sedimentation time etc. This six jar assembly has a multiple stirrer unit having varying speed. Various chemical doses are applied to waste water sample on a multiple stirrer unit followed by intensive mixing and
thereafter slow stirring for specific period to promote flocculation. The samples are allowed to stand, supernatant liquid is decanted and analysed for various parameters. In principle main features of this (90) test are -

i. Identical and reproducible stirring conditions in a number of samples.

ii. Two fixed stirring speeds; one fast, one slow.

iii. Simultaneous addition of chemicals in all the samples.

iv. Accommodation of samples of approximately 1000 ml capacity.

These specifications are necessary to produce comparable results between different samples. Various factors such as stirring time, mixing time, flocculation time etc. have marked effects on the observed results. Sample volume less than 1000 ml is likely to lead to difficulties in decanting treated water samples for analysis. It is essential that the treated samples are simultaneously drawn from the same depth.

Sample Collection

Waste water from cotton textile, leather tannery and chrome plating industries were collected around Kanpur and pulp and paper for Lalkuna (Nainital) were collected in HDP
jars for 10 litres capacity from the outlet point. Waste water samples were immediately tested for COD and BOD stored at 15°C for subsequent experiments.

5.4 EXPERIMENTAL PROCEDURE

The experimental procedure is as follows -

5.4.1 Determination of Optimum Coagulant and Flocculant Dosage

(i) A bulk waste water sample collected from various industries was analysed for total solids (TS), suspended solids (SS), chemical oxygen demand (COD), biological oxygen demand (BOD) and pH. The waste water temperature and room temperature were recorded at the time of testing.

(ii) One liter of waste water sample was taken in the beaker and placed on the six jar assembly. A concentrated stock solution of coagulant and flocculant was prepared and a required concentration of this was added to the waste water sample.

(iii) Similarly various concentrations were added to the other beakers keeping one beaker as a control.
(iv) The stirrer of the jar test assembly were set to rotate at the speed of 100 rpm.

(v) After adding flocculants rapid mixing at the speed of 100 rpm was done about one minute.

(vi) After rapid mixing, slow mixing at the speed of 50 rpm was done for about 10 minutes.

(vii) After slow mixing a sedimentation time of 30 minutes was given to allow to settle the flocs.

(viii) After settling time of 30 minutes the supernatant liquid was decanted in number of flask for the subsequent testing of TS, SS, COD and BOD.

The concentration at which the TS, SS, COD and BOD reduction are maximum is the optimum coagulant and flocculant dose.

5.4.2 Determination of Optimum pH

pH of the waste water sample was varied in the range of by adding sodium hydroxide and sulfuric acid. Three pH value in the acidic, basic and neutral range were selects to observe the performance of polyelectrolytes. Samples of different pH values were taken in beakers and optimum dose of polyelectrolytes determined as above was added to each beaker. The experiments were carried out in the above mentioned manner. Blank samples of different pH values were also carried out. pH at which the TS, SS, COD, & BOD reduction are maximum, is optimum pH.
5.4.3 Determination of Optimum Polyelectrolyte Dosage in Presence of Inorganic Coagulant

(i) Sequence of addition of Polyelectrolyte and Inorganic Coagulant -

Inorganic coagulant was added first and it was thoroughly mixed for about one minute at the mixing speed of 100 rpm. After this the polyelectrolyte was added.

(ii) Waste water samples were taken in different beakers and optimum dose of inorganic coagulant determined as above and different concentrations of polyelectrolytes were added to the each beaker. Experiments were conducted in the above mentioned manner. Polyelectrolyte concentration at which TS, SS, COD and BOD reductions are maximum is optimum polyelectrolyte dose.

5.4.4 Determination of Optimum Temperature

Keeping the polyelectrolyte concentration and other factors constant the experiments were conducted at different temperatures in the range of 15°C and 45 ± 2°C and the TS, SS, COD and BOD reductions were determined in the manner as mentioned earlier, temperature at which these reduction are maximum is optimum temperature.
5.4.5 Polyelectrolyte and Coagulant Concentration Range

In the present research work, polyelectrolytes and coagulant at different concentration were used which are as follows:

1. Polyelectrolyte SC-1 (0.01 mg/l - 1.0 mg/l) or $0.40 \times 10^{-10} \frac{g}{mol} - 20.0 \times 10^{-10} \frac{g}{mol}$.

2. Polyelectrolyte SC-2 (0.01 mg/l - 1.0 mg/l) or $0.66 \times 10^{-10} \frac{g}{mol} - 33.3 \times 10^{-10} \frac{g}{mol}$.

3. Polyelectrolyte SC-3 (0.01 mg/l - 1.0 mg/l) or $0.44 \times 10^{-10} \frac{g}{mol} - 22.0 \times 10^{-10} \frac{g}{mol}$.

4. Polyelectrolyte BC-1 (0.05 mg/l - 1.0 mg/l) or $0.96 \times 10^{-10} \frac{g}{mol} - 38.0 \times 10^{-10} \frac{g}{mol}$.

5. Inorganic Coagulant FeCl₃ 20.0 mg/l - 10.0 g/l.

5.5 PARAMETER DETERMINATION

Following parameters were determined for the raw and treated waste water -

i. Total solids (TS)
ii. Suspended solids (SS)
iii. Chemical oxygen demand (COD)
iv. Biological oxygen demand (BOD)
v. pH

These parameters were determined as per ASTM specification (91).