CHAPTER 4
MATERIALS AND METHODS

The investigations were carried out to realise the study objectives include

1. Physico-chemical analysis of ground water samples exposed to effluent of low cost sanitation systems to study the impact of low cost sanitation on ground water quality.

2. Water quality indices for ground water samples of study areas to assess its potability and to summarise large amounts of water quality data into simple terms.

3. Physico-chemical analysis of soil samples exposed to effluent of low cost sanitation systems to study the impact of low cost sanitation on soil quality.

4. Laboratory experiments on soil column exposed to effluent of low cost sanitation system to assess the Physico-chemical changes in soil characteristics and behaviour of percolated effluent at different depths of soil column.

4.1 WATER SAMPLING:

People are often insufficiently aware of environmental factors effecting their drinking water sources (Lee and Basterneijer, 1991). Water resources, the relief and ethnic groups and their cultural habits should be considered for low cost sanitation installations in suburban areas (Macia et al., 1986; Sadashivaourthy et al., 2004). National level
research institutes like CBRI, NEERI developed a different technical as well as economical solutions to reduce pollution problems due to on-site sanitation system (please refer section 2.4 for details). Hence, surveys were conducted physically during May and June 2001 for selection of sites (Plate No. 4.1, 4.2 and 4.3). Information regarding the available and existing on-site sanitation systems was collected by house-to-house survey. Based on information procured, forty wells from individual houses identified in Jodugullapalem and Arilova Colony study sites, which are having well and ILCS Units nearby and within the home. The entire study of data collection was carried from May 2001 to February 2004. The field survey data collected at Jodugullapalem and Arilova Colony study areas are presented in Table No’s 4.1 and 4.2 respectively.

Few research workers observed no significant deterioration even in the vicinity of the town that have been densely developed using properly operated low cost on-site sanitation systems (Lowe and Miner, 1990; Bounds, 1997; Madeleen and Teresia, 1997; Philippi et al., 1999). Some authors studied the protection of wells from onsite sanitation systems (Canter and Sabatini, 1994; Kauzeni and Norconsult, 1981; Lloyd, 1990; Cotton et al., 1995). But, several research workers reported that the pollution of ground water due to on-site sanitation systems (Gondwe et al., 1997; Nadeem Afzal, 2002; Barrett and Nalnega, 1999; Jacks et al.,
Plate No.4.1: ILCS Unit near to well in Jodugullapalem

Plate No.4.2: ILCS Unit near to well in Arlova Colony

Plate No.4.3: ILCS Unit not in use
Table No. 4.1: Field survey data collected at Jodugulapalem

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Sampling Well Location (Door No. of house)</th>
<th>Age of the LCS Unit</th>
<th>No. of Users</th>
<th>Distance between leach pit and well</th>
<th>Type of wells</th>
<th>Depth of well</th>
<th>HZS Paper Strip Test*</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1-67</td>
<td>2 years</td>
<td>5</td>
<td>5m</td>
<td>Open</td>
<td>6m</td>
<td>+</td>
</tr>
<tr>
<td>2</td>
<td>1-170</td>
<td>2 years</td>
<td>4</td>
<td>4m</td>
<td>Open</td>
<td>6m</td>
<td>+</td>
</tr>
<tr>
<td>3</td>
<td>1-126</td>
<td>2 years</td>
<td>6</td>
<td>13m</td>
<td>Open</td>
<td>6m</td>
<td>+</td>
</tr>
<tr>
<td>4</td>
<td>1-183</td>
<td>2 years</td>
<td>5</td>
<td>7m</td>
<td>Open</td>
<td>6m</td>
<td>+</td>
</tr>
<tr>
<td>5</td>
<td>1-110</td>
<td>2 years</td>
<td>4</td>
<td>10m</td>
<td>Bore</td>
<td>12m</td>
<td>+</td>
</tr>
<tr>
<td>6</td>
<td>1-159</td>
<td>2 years</td>
<td>5</td>
<td>10m</td>
<td>Open</td>
<td>6m</td>
<td>+</td>
</tr>
<tr>
<td>7</td>
<td>1-112</td>
<td>2 years</td>
<td>4</td>
<td>15m</td>
<td>Open</td>
<td>6m</td>
<td>+</td>
</tr>
<tr>
<td>8</td>
<td>1-137</td>
<td>2 years</td>
<td>5</td>
<td>12m</td>
<td>Open</td>
<td>6m</td>
<td>+</td>
</tr>
<tr>
<td>9</td>
<td>1-145</td>
<td>2 years</td>
<td>4</td>
<td>8m</td>
<td>Open</td>
<td>6m</td>
<td>+</td>
</tr>
<tr>
<td>10</td>
<td>1-179</td>
<td>2 years</td>
<td>6</td>
<td>6m</td>
<td>Open</td>
<td>6m</td>
<td>+</td>
</tr>
<tr>
<td>11</td>
<td>1-122</td>
<td>2 years</td>
<td>6</td>
<td>8m</td>
<td>Bore</td>
<td>10m</td>
<td>+</td>
</tr>
<tr>
<td>12</td>
<td>1-127/2</td>
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<td>4</td>
<td>20m</td>
<td>Open</td>
<td>6m</td>
<td>+</td>
</tr>
<tr>
<td>13</td>
<td>1-666</td>
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<td>4</td>
<td>1.5m</td>
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<td>6m</td>
<td>+</td>
</tr>
<tr>
<td>14</td>
<td>1-176</td>
<td>2 years</td>
<td>5</td>
<td>1m</td>
<td>Open</td>
<td>6m</td>
<td>+</td>
</tr>
<tr>
<td>15</td>
<td>1-113</td>
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<td>5</td>
<td>20m</td>
<td>Open</td>
<td>6m</td>
<td>-</td>
</tr>
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<td>Public Well</td>
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<td>4</td>
<td>5m</td>
<td>Open</td>
<td>6m</td>
<td>+</td>
</tr>
<tr>
<td>17</td>
<td>1-149</td>
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<td>4</td>
<td>15m</td>
<td>Open</td>
<td>6m</td>
<td>+</td>
</tr>
<tr>
<td>18</td>
<td>1-121</td>
<td>2 years</td>
<td>6</td>
<td>10m</td>
<td>Open</td>
<td>6m</td>
<td>+</td>
</tr>
<tr>
<td>19</td>
<td>1-12</td>
<td>2 years</td>
<td>4</td>
<td>12m</td>
<td>Bore</td>
<td>12m</td>
<td>+</td>
</tr>
<tr>
<td>20</td>
<td>School, Near house No. 1-67</td>
<td>2 years</td>
<td>4</td>
<td>20m</td>
<td>Bore</td>
<td>15m</td>
<td>-</td>
</tr>
</tbody>
</table>

* A ready - to - use qualitative test kit
+ Presence of coliforms and faecal contamination in water
- Fit for drinking
Table No. 4.2: Field survey data collected at Arilova Colony

<table>
<thead>
<tr>
<th>No.</th>
<th>Sampling Well Location (Door No. of house)</th>
<th>Age of the LCS Unit</th>
<th>No. of Users</th>
<th>Distance between leach pit and well</th>
<th>Type of wells</th>
<th>Depth of well</th>
<th>H₂S paper Strip Test*</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>12-240, Balaji Nagar</td>
<td>5 years</td>
<td>5</td>
<td>10m</td>
<td>Bore</td>
<td>10m</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>16-205, Operation Colony</td>
<td>5 years</td>
<td>4</td>
<td>1m</td>
<td>Bore</td>
<td>12m</td>
<td>+</td>
</tr>
<tr>
<td>3</td>
<td>12-298, Balaji Nagar</td>
<td>4 years</td>
<td>5</td>
<td>4m</td>
<td>Bore</td>
<td>10m</td>
<td>+</td>
</tr>
<tr>
<td>4</td>
<td>16-205, Operation Colony</td>
<td>9 years</td>
<td>4</td>
<td>1m</td>
<td>Bore</td>
<td>10m</td>
<td>+</td>
</tr>
<tr>
<td>5</td>
<td>12-311, Balaji Nagar</td>
<td>5 years</td>
<td>3</td>
<td>15m</td>
<td>Bore</td>
<td>10m</td>
<td>-</td>
</tr>
<tr>
<td>6</td>
<td>13-1417, Balaji Nagar</td>
<td>4 years</td>
<td>2</td>
<td>20m</td>
<td>Bore</td>
<td>10m</td>
<td>-</td>
</tr>
<tr>
<td>7</td>
<td>13-1074, Apsara Colony</td>
<td>4 years</td>
<td>3</td>
<td>30m</td>
<td>Bore</td>
<td>10m</td>
<td>-</td>
</tr>
<tr>
<td>8</td>
<td>12-341, Balaji Nagar</td>
<td>5 years</td>
<td>4</td>
<td>4m</td>
<td>Bore</td>
<td>10m</td>
<td>+</td>
</tr>
<tr>
<td>9</td>
<td>13-1230, Vivekananda Colony</td>
<td>4 years</td>
<td>5</td>
<td>15m</td>
<td>Bore</td>
<td>15m</td>
<td>-</td>
</tr>
<tr>
<td>10</td>
<td>15-497, Sivaji Nagar</td>
<td>5 years</td>
<td>6</td>
<td>2m</td>
<td>Bore</td>
<td>12m</td>
<td>+</td>
</tr>
<tr>
<td>11</td>
<td>15-320, Sivaji Nagar</td>
<td>4 years</td>
<td>5</td>
<td>1m</td>
<td>Bore</td>
<td>10m</td>
<td>+</td>
</tr>
<tr>
<td>12</td>
<td>13-1420, B.C. Colony</td>
<td>4 years</td>
<td>5</td>
<td>12m</td>
<td>Bore</td>
<td>18m</td>
<td>-</td>
</tr>
<tr>
<td>13</td>
<td>13-1374, P. H. Colony</td>
<td>5 years</td>
<td>5</td>
<td>2m</td>
<td>Bore</td>
<td>28m</td>
<td>+</td>
</tr>
<tr>
<td>14</td>
<td>12-418/2, Anna Nagar</td>
<td>5 years</td>
<td>10</td>
<td>5m</td>
<td>Bore</td>
<td>12m</td>
<td>+</td>
</tr>
<tr>
<td>15</td>
<td>13-1469, Balaji Nagar</td>
<td>5 years</td>
<td>5</td>
<td>10m</td>
<td>Bore</td>
<td>10m</td>
<td>-</td>
</tr>
<tr>
<td>16</td>
<td>12-310, Balaji Nagar</td>
<td>5 years</td>
<td>2</td>
<td>12m</td>
<td>Bore</td>
<td>10m</td>
<td>-</td>
</tr>
<tr>
<td>17</td>
<td>16-317, Sivaji Nagar</td>
<td>3 years</td>
<td>5</td>
<td>5m</td>
<td>Bore</td>
<td>10m</td>
<td>+</td>
</tr>
<tr>
<td>18</td>
<td>12-224, Balaji Nagar</td>
<td>4 years</td>
<td>2</td>
<td>1m</td>
<td>Bore</td>
<td>10m</td>
<td>+</td>
</tr>
<tr>
<td>19</td>
<td>12-199, Balaji Nagar</td>
<td>4 years</td>
<td>3</td>
<td>2m</td>
<td>Bore</td>
<td>18m</td>
<td>+</td>
</tr>
<tr>
<td>20</td>
<td>13-1342, Rajiv Nagar</td>
<td>4 years</td>
<td>2</td>
<td>12m</td>
<td>Bore</td>
<td>12m</td>
<td>-</td>
</tr>
</tbody>
</table>

- A ready-to-use qualitative test kit
+ Presence of coliforms and faecal contamination in water
- Fit for drinking
1999; McLarin et al., 1999; Ravi Mishra and Richariya, 1999; Deepanjjan and Gupta, 2000; Scandura and Sobsey, 1997; Odai and Dugbantey, 2003; Robertson and Harman, 1999)(please refer section 2.3 for details).

The ground water sampling has been done in a scientific manner during the period of study (March 2002 to February 2004) to assess the ground water contamination from LCS (second objective of study). Seasonal variation of ground water quality indicates the deterioration of ground water quality (Linda, 1999; Ramaraju et al., 1998; Singh et al., 2000; Sundari et al., 2004). Hence, the climate of the study area has been classified into three seasons viz., summer (March to July), rainy (August to October) and winter (November to February). Water samples were collected bi-monthly from all the 40 sampling stations from the two study sites during study period. Sample collection was usually completed during morning hours between 8a.m to 10a.m every time.

Water samples were collected separately in pre-cleaned 2lit capacity polythene cans for Physico-chemical analysis. Sampling of water was done as per standard methods (APHA, 1995; NEERI, 1988). Samples for bacterial assessment were collected in H2S paper strip test bottles at the sampling station itself.
4.2 ANALYSIS OF WATER SAMPLES:

The ground water samples collected from the two study areas were analysed for various physico-chemical parameters by using standard methods (APHA, 1995; NEERI, 1988).

4.2.1 pH: Electrodes were removed from storage solutions and rinsed with distilled water. The electrodes were gently dried by blotting with a soft tissue paper. The instrument was standardized with electrodes by using buffer solutions of pH - 4 and pH - 9.2. The electrodes were removed from the buffer solutions and thoroughly rinsed and blotted with tissue paper. Equilibrium between electrodes and sample was established by stirring the sample and the pH of the sample was measured. The reading was taken after the indicated value remained constant for about a minute.

4.2.2 Electrical Conductivity: The cell of conductivity meter was calibrated with standard 0.1N KCl solution of conductivity 14.12mS/cm. The cell was thoroughly rinsed with deionised distilled water and carefully wiped with a tissue paper. The cell was dipped into the sample solution and the solution was swirled and waited up to one minute for a steady reading.

4.2.3 Total Hardness: 25 ml of well mixed sample was taken in a conical flask and one ml of buffer solution was added. A pinch of Erichrome Black - T indicator was added and titrated with standard EDTA (0.01M)
till wine red colour changed to blue. The volume of EDTA consumed (A) was noted down and total hardness was calculated by using following formula:

**Total Hardness as CaCO₃ mg/l = A * 1000/ ml of Sample**

### 4.2.4 Chlorides:
25 ml of well mixed sample was taken in a conical flask and the pH was adjusted to 7 - 8. 1ml of potassium chromate indicator was added to get light yellow colour and titrated with standard silver nitrate solution till colour changed to brick red from yellow. The volume of silver nitrate consumed (A) was noted down and chloride content was calculated by using the following formula:

**Chloride mg/l = A*N*35.46 * 1000/ ml of Sample**

Where N = Normality of AgNO₃.

### 4.2.5 Nitrates:
50 ml of sample was taken in a conical flask and 50ml of silver Sulphate solution was added to remove chlorides and heated slightly and AgCl precipitate was removed by filtration. The filtrate was evaporated in a porcelain dish to dryness and cooled. The residue in porcelain dish was dissolved in 1ml Phenol Disulphonic Acid and diluted to 10ml with distilled water. The contents were transferred into 50ml volumetric flask and 3ml of liquid ammonia was added to develop the yellow colour and made up to the mark with distilled water. The absorbance of colour developed was measured at 410nm with a light path of 1cm. The concentration of nitrate was calculated from the
standard curve by using the following formula (The standard curve was prepared using suitable aliquots of the standard nitrate solution in the range of 5 to 50 mg/L following the above procedure).

\[
\text{Nitrate (mg/l)} = C \times 1000 / \text{ml. of sample.}
\]

Where \( C \) = Concentration from Graph.

4.2.6 Nitrites: 50ml of clear sample was taken and the pH was adjusted to 7.0. One ml of sulphanilamide solution was added to this after 5 minutes. 1ml of NED-dihydrochloride solution was added and thoroughly mixed immediately. Absorbance was measured after 10 minutes (but before 2 hours) at 543nm. A blank was prepared in the same way by using distilled water instead of sample. Calibration curve was prepared by pipetting suitable volumes of standard nitrite solution following the above procedure. The concentration of nitrite was calculated from the standard curve by using the following formula:

\[
\text{Nitrite (mg/l)} = C \times 1000 / \text{ml. of sample.}
\]

Where \( C \) = Concentration from Graph.

4.2.7 Ammonia: Ion selective meter was calibrated by using ammonia standards of 1ppm and 10 ppm and TISAB solution. Electrode slope was checked with ion meter. 50 ml of sample was transferred to 100 ml plastic beaker and 0.5 ml of TISAB solution was added. The electrode was rinsed, blotted dry and placed in the sample. The contents of the
plastic beaker are stirred thoroughly and the steady reading on the meter
was noted down directly.

4.2.8 Sulphates: Suitable volume of sample was taken in a volumetric
flask and 2ml of conditioning reagent was added. The contents were
mixed well and 1 gm of BaCl₂ crystals were added while stirring. The
stirring was continued for 1 minute after addition of BaCl₂ crystals. The
absorbance was measured at 420nm after complete dissolution of BaCl₂
crystals. A blank was prepared in the same way by using distilled water
instead of sample. Calibration curve was prepared by pipetting suitable
volumes of standard Sulphate solution in the range of 40 mg/l following
the above procedure. The concentration of Sulphate was calculated from
the standard curve by using the following formula:

\[
\text{Sulphate (mg/l)} = C \times 1000 / \text{ml. of sample.}
\]

Where C = Concentration from Graph.

4.2.9 Phosphates: Suitable volume of sample was taken in a volumetric
flask and 50ml of distilled water was added. To this 4 ml of ammonium
molybdate and 0.5 ml of stannous chloride were added and made up to
the mark with distilled water. Absorbance was measured at 690nm
between 10-12 minutes after the development of the blue colour. A blank
was prepared in the same way by using distilled water instead of sample.
Calibration curve was prepared by pipetting suitable volumes of standard
phosphate solution in the range of 30 mg/l following the above
procedure. The concentration of phosphate was calculated from the standard curve by using the following formula:

\[
\text{Phosphates (mg/l)} = \frac{C \times 1000}{\text{ml. of sample}}.
\]

Where \( C \) = Concentration from Graph.

4.2.10 Fluoride: Ion selective meter was calibrated by using ammonia standards of 1 ppm and 10 ppm and TISAB solution and electrode slope was checked with ion meter. 50 ml of sample was transferred to 150 ml plastic beaker and 50 ml of TISAB solution was added. The electrode was rinsed, blotted dry and placed in the sample. The contents of the plastic beaker were stirred thoroughly and the steady reading on the meter was noted down directly.

4.2.11 Sodium: The water samples were filtered through Whatman filter paper No. 42. The filtrate was transferred in 100 volumetric flasks. First distilled water was aspirated to flame photometer and adjusted to zero. The instrument was calibrated by aspirating the standards of sodium and adjusted the galvanometric reading to desired mark. The sample was aspirated and the galvanometric reading was noted down directly.

4.2.12 Potassium: The water samples were filtered through Whatman filter paper No. 42. The filtrate was transferred in 100 volumetric flask. First distilled water was aspirated to flame photometer and adjusted to
zero. The instrument was calibrated by aspirating the standards of potassium and adjusted the galvanometric reading to desired mark. The sample was aspirated and the galvanometric reading was noted down directly.

4.2.13 Iron: Suitable volume of sample was taken in a conical flask and 2ml of Conc. HCl followed by 1 ml of hydroxylamine hydrochloride solutions were added and 2 – 3 glass beads were added and boiled for 20 – 25 minutes to ensure dissolution of iron, cooled and transferred to 50 ml volumetric flask. To this 10 ml of ammonium acetate and 3 ml of 1, 10-phenanthroline solutions were added and made up to the mark with distilled water. The absorbance of colour developed was measured at 510nm between 10 – 15 minutes. A blank was prepared in the same way by using distilled water instead of sample. Calibration curve was prepared by pipetting suitable volumes of standard iron solution in the range of 1000–4000μg/l following the above procedure. The concentration of iron was calculated from the standard curve by using the following formula:

$$\text{Iron (mg/l)} = C \times 1000 / \text{ml. of sample}.$$ 

Where C = Concentration from Graph.

4.2.14 B.O.D: Dilution water was prepared by adding 1 ml each of phosphate buffer, MgSO₄, CaCl₂, and FeCl₃ to 1 litre of distilled water which was already aerated for 1 – 2 days. Four B.O.D bottles were taken
and they are marked as S₀, S₅, B₀ and B₅. Water samples of proper dilution were taken in S₀ and S₅ and only dilution water in B₀ and B₅.

Proper care was taken to see that no air bubbles in B.O.D bottles. Dilution water was sewage seeded. First the bottle S₀ was taken and 2 ml of MnSO₄ was added following by addition of 2 ml of mixture of KI, NaOH and NaN₃. Then the bottle was stoppered and shaken well. The precipitate was formed and settled. Then 2 ml of Conc. H₂SO₄ was added to it. 203 ml of this solution was transferred into a conical flask and it was titrated against 0.025N Hypo until yellow colour changes to pale yellow. The starch indicator was added. Then the solution was turned to blue colour. Titration was continued until blue colour changes to colourless. The burette reading was noted down as D.O S₀. Now the bottle B₀ was taken and the above steps were repeated and D.O. B₀ was noted down. Now the bottles S₅ and B₅ were taken and kept in B.O.D. incubator for 5 days at 20°C and then D.O S₅ and D.O. B₅ were noted down following the above procedure. The B.O.D. was calculated using the following formulae:

\[
\text{B.O.D (mg/l)} = Y \times \frac{\text{Volume of bottle (ml)}}{\text{Volume of sample (ml)}}
\]

\[
Y = D.O \ S₀ - D.O \ S₅ - D.O \ B₀ + D.OB₅
\]

4.2.15 C.O.D: 50 ml of the sample was taken in a round bottom flask, 25 ml of K₂CrO₇ and 75 ml of Conc. H₂SO₄ mixed with Ag₂SO₄ were added. The mixture was refluxed for 2 hours and cooled. The condenser
was washed with distilled water. The contents were transferred into conical flask and made up to 350 ml with distilled water. The excess dichromate was titrated against standard Ferrous Ammonium Sulphate solution using ferroin as indicator. At the end point, the colour changed from bluish green to reddish blue. The Ferrous Ammonium Sulphate solution consumed was noted down (A). The procedure was repeated with distilled water instead of the sample and the Ferrous Ammonium Sulphate solution consumed was noted down (B). Then C.O.D was calculated by using the following formula:

\[
\text{C.O.D. mg/l} = (A-B) \times N \times \frac{8000}{\text{ml of Sample}}
\]

Where \(N\) = Normality of Ferrous Ammonium Sulphate.

4.2.16 \(H_2S\) Paper Strip Test: A ready-to-use qualitative test kit is based on the detection of \(H_2S\) producing bacteria whose presence is consistently associated with the presence of coliform bacteria and faecal contamination in water. Furthermore it also detects enteric bacteria such as Salmonella, Proteus, Citrobacter, Klebsiella, and Clostridium. The water to be tested was filled up to 'Fill line' and replaced the cap, kept at room temperature (at 25° to 30°C) for 24 - 48 hours. The change in colour (blackening) was observed of the medium. If medium turned black water was not fit for drinking.
4.2.17 Water Quality Index (WQI):

The WQI was calculated for various beneficial uses considering seasonal mean values of the thirteen parameters based on their degree of importance for the relevant use. Parameters selection has a great importance for the calculation of water quality index. Since various parameters depend on the intense use of water, physico-chemical parameters namely pH, EC, TH, Cl, SO\textsubscript{4}, NO\textsubscript{3}, NO\textsubscript{2}, Fe, F, NH\textsubscript{3}, Na, B.O.D and C.O.D were used to estimate water quality index. Procedure suggested by Guruprasad (2003) was used to calculate WQI. The unit weights for various parameters were assumed to be inversely proportional to the standard norms for corresponding parameters i.e.

\[ W_i = K / S_i \]

Where, \( W_i \) = Parameter unit weight

\[ K = \text{Proportionality constant} \]

\[ S_i = \text{Standard recommended for } i\text{th parameter } P_i \]

\[ i = 1, 2 \ldots \ldots A(13 \text{ in the present case}) \]

\[ \sum w_i = k \sum (1 / S_i) - 1 \]

\[ i=1 \quad i=0 \]

The quality rating \( Q_i \) for a quality parameter \( P_i \) may be obtained by the relation

\[ q_i = 100 / (V_i / S_i) \]
Vi is observed value and Si is recommended standard for the ith parameter. The sub-index Si for the ith parameter is given by

$$SI = q_i \cdot wi$$

Finally overall WQI was calculated after computing all the sub-index.

$$WQI = \frac{q_i Wi}{W_i}$$

(Unit weights for corresponding parameters are given in Table No. 4.3)

4.3 SOIL SAMPLING:

Traveling distance of pollutants from on-site sanitation system depends on the physico-chemical quality of the soil (Whelan and Barrow, 1984; Chauhan, 1984; Sinton, 1986; Robertson et al., 1991; Harman et al., 1996; Tole, 1997) (please refer 2.2 for details). Hence, soils from low cost sanitation system implemented sampling stations were collected and used for Physico-chemical analysis. Soil samples collected randomly at different depths from the field in five replicates and air dried for 72hrs, powdered, sieved through 2mm sieve. The soil samples were collected seasonally i.e., summer (March to July), rainy (August to October) and winter (November to February). From the two study sites, one typical sampling station in each study site was considered for seasonal soil sampling to determine typical physical properties of soils in study area. Seasonal soil sampling is done during March'2002, August'2002, November'2002, March'2003, August'2003 and November'2003
Table No. 4.3: List of parameters and unit weights used for Water Quality Index

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Parameters</th>
<th>Standard [SI]</th>
<th>Recommending agency</th>
<th>Unit weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>pH</td>
<td>7.0 8.5</td>
<td>ICMR</td>
<td>0.117</td>
</tr>
<tr>
<td>2.</td>
<td>E.C (micromhos/cm)</td>
<td>250</td>
<td>WHO</td>
<td>0.004</td>
</tr>
<tr>
<td>3.</td>
<td>Total Hardness</td>
<td>300</td>
<td>ICMR</td>
<td>0.0033</td>
</tr>
<tr>
<td>4.</td>
<td>Chlorides</td>
<td>200</td>
<td>ICMR</td>
<td>0.005</td>
</tr>
<tr>
<td>5.</td>
<td>Sulphates</td>
<td>200</td>
<td>ICMR</td>
<td>0.005</td>
</tr>
<tr>
<td>6.</td>
<td>Nitrates</td>
<td>20</td>
<td>ICMR</td>
<td>0.05</td>
</tr>
<tr>
<td>7.</td>
<td>Nitrites</td>
<td>0.5</td>
<td>E.U</td>
<td>2</td>
</tr>
<tr>
<td>8.</td>
<td>Fluoride</td>
<td>1.0</td>
<td>ICMR</td>
<td>1</td>
</tr>
<tr>
<td>9.</td>
<td>Iron</td>
<td>0.1</td>
<td>ICMR</td>
<td>10</td>
</tr>
<tr>
<td>10.</td>
<td>Ammonia</td>
<td>0.5</td>
<td>B.U</td>
<td>2</td>
</tr>
<tr>
<td>11.</td>
<td>Sodium</td>
<td>200</td>
<td>WHO</td>
<td>0.005</td>
</tr>
<tr>
<td>12.</td>
<td>BOD</td>
<td>2</td>
<td>C.P.C.B</td>
<td>0.5</td>
</tr>
<tr>
<td>13.</td>
<td>COD</td>
<td>20</td>
<td>C.P.C.B</td>
<td>0.05</td>
</tr>
</tbody>
</table>
4.4 ANALYSIS OF SOIL SAMPLES:

The collected soil samples were analyzed for various physical properties such as permeability, bulk density and dry density as per IS 2720 – 1973. The sieve analysis was also done for all six seasons and soil grade curves are drawn to find out the coefficient of curvature (Cc) and coefficient of uniformity (Cu) to know the type of soil. The tests were carried out in Geotechnical Engg. Laboratory of Civil Engineering dept.

Preparation of Soil Samples:

Soil samples collected from study areas were crushed in an iron mortar. The grounded soil was sieved through 2 mm sieve. 250 g of the sieved soil sample was placed in a cardboard box for analysis.

Physico-chemical analysis of Soil Samples:

4.4.1 pH: The pH of the soil was determined in 1:5 soil-water suspension using a digital pH meter (Systronics).

4.4.2 Electrical Conductivity: EC was determined in terms of mS/cm for 1:5 soil-water suspensions by a digital conductivity bridge with a cell constant value of 1.0.

4.4.3 Organic Carbon: This was determined by wet digestion method of Walkley Black method and expressed as percentage. 1 gm of soil sample was taken in a 500ml conical flask and treated with 10ml of 1N potassium dichromate and 20 ml of Conc. H₂SO₄. The contents of the flask were mixed and the reaction was allowed to proceed for 30 minutes.
After cooling, 200 ml of distilled water and 5 drops of ferroin indicator were added while stirring. The solution was titrated against 1N Ferrous ammonium Sulphate to get the colour changed from greenish brown to dark green. Volume of Ferrous ammonium Sulphate consumed (T) was noted down. A blank was also run simultaneously and volume of Ferrous ammonium Sulphate consumed (B) for blank was noted down. The percentage of organic carbon was derived using the formulae:

\[
\text{% Organic Carbon} = \frac{(B - T) \times F}{F} = \frac{(3/7 \times N/W)}{
\]

Where \( N \) = Normality of Ferrous ammonium Sulphate, \( W \) = weight of soil sample (g).

4.4.4 Available Phosphorus: Phosphorus was determined following molybdenum blue method (APHA, 1995). 1 gm of soil sample was extracted with 50 ml of 2.5% acetic acid and the extract was filtered and the filtrate was used for the determination of available phosphorus (Stewart et al., 1974). Suitable aliquots of soil sample extract were taken in 50 ml nessler tubes. To this 2 ml of ammonium molybdate and 5 drops of stannous chloride solutions were added. The optical density of the blue colour was measured at 690 nm against a blank using a UV-Visible spectrophotometer (Systronics-118). Readings on the spectrophotometer were recorded in between 10 – 12 minutes after the
addition of the last reagent. The concentration of phosphate was computed from a standard curve, using the following formula:

\[ P(\%) = \frac{C \text{ (mg)} \times \text{solution volume (ml)} \times \text{sample weight (g)} \times 10 X \text{aliquot (ml)} }{10 X \text{ sample solution (ml)}} \]

Where \( C = \text{mg of P obtained from the graph.} \)

**4.4.5 Potassium:** Potassium content in the soil was determined by the method described by Stewart et al. (1974). 25 grams of air-dried and sieved soil was taken in 500 ml of Erlenmeyer flask. To this 125 ml of 1M ammonium acetate solution (pH 7.0) was added while shaking to completely wet the soil and the contents were allowed to stand overnight. The contents were filtered through a Whatman filter paper No. 42 through a Buchner funnel under light suction using multiple washings. The leachate was transferred to 250 ml of volumetric flask, brought to volume with 1M ammonium acetate and homogenized.

Potassium in the solution was determined by Flame photometric method (APHA, 1995), using the following formula:

\[ \text{Potassium (\%) = C \times D.F. \times extracted sample solution (ml) / 10^4 \times sample weight} \]

Where \( C = \text{Concentration of Potassium in diluted sample} \)

\( \text{D.F. = Dilution factor} \)

**4.4.6 Exchangeable Calcium and Magnesium:** These were determined by EDTA titration following Stewart et al. (1974). 2 grams of sieved soil
sample was taken in a 250 ml beaker and incubated in 100ml of ammonium acetate buffer solution for overnight. The contents were filtered through a Whatman filter paper No. 42 and the filtrate was used for the determination of calcium and magnesium. 25 ml of filtrate was taken in a conical flask and 1 ml of NaOH solution was added to raise pH to 12. A pinch of murexide indicator was added and titrated immediately with standard EDTA (0.01M) till pink colour changed to purple. The volume of EDTA consumed (A) was noted down and calcium was calculated using following formula:

\[ \text{Calcium (mg/g)} = A \times 400.4 \times \frac{V_1}{V_2} \times X \times 10,000 \]

Where \( A \) = Vol. of EDTA consumed for Ca  
\( V_1 = \) Total Vol. of extract prepared  
\( V_2 = \) ml of filtrate taken  
\( X = \) Weight of soil taken

Now 25 ml of filtrate was taken in another conical flask and 1 or 2 ml of ammonium buffer solution was added. A pinch of Erichrome Black-T indicator was added and titrated immediately with standard EDTA (0.01M) till wine red colour changed to blue. The volume of EDTA consumed (B) was noted down and magnesium was calculated using following formula:

\[ \text{Magnesium (mg/g)} = (B - A) \times 440.4 \times \frac{V}{V_1} \times 1.646 \times X \times 10,000 \]

Where \( B \) = Vol. of EDTA consumed for Ca and Mg.
\[ A = \text{Vol. of EDTA consumed for Ca.} \]

\[ V = \text{Total Vol. of extract prepared.} \]

\[ V_1 = \text{ml of filtrate taken} \]

\[ X = \text{Weight of soil taken} \]

**4.4.7 Chlorides:** Chlorides were determined by Chromate indicator method of Piper. The chloride of the soil was determined in 1:5 soil-water suspension. The contents were filtered through a Whatman filter paper No. 42 and the filtrate was used for the determination of chlorides. 25 ml of filtrate was taken in a conical flask and 1ml of potassium chromate indicator was added to get light yellow colour and titrated with standard silver nitrate solution till colour changed to brick red from yellow. The volume of silver nitrate consumed \( A \) was noted down and chlorides were calculated by using the following formula:

\[
\text{Chloride (mg/g)} = \frac{X \times \text{Vol. of soil extract}}{10 \times \text{weight of soil}}
\]

Where \( X = \text{Vol. of AgNO}_3 \times 0.0141 \times 35.46 \times 1000 / \text{Vol. of aliquot taken.} \)

**4.4.8 Available Iron:** The available iron was extracted with 3% oxalic acid following Stewart et al., 1974. A suitable aliquot of soil extract was taken in 125 ml conical flask and available iron present was determined by following the procedure as described in analysis of water, using following formula:

\[
\text{Iron (mg/g)} = \frac{C \times \text{Vol. of extract}}{1000 \times \text{ml. of aliquot} \times \text{Wt. of Soil.}}
\]

Where \( C = \text{Concentration from Graph.} \)
4.4.9 Microbial Biomass: It was estimated by the method of Jenkinson and Powlison. Sieved soil was taken in a petri plate and it was moistened with distilled water. 50 ml of 0.1N KOH was taken in a 100 ml beaker and 5ml of 50% BaCl₂ and 2–3 drops of phenolphthalein were added. The beaker was placed on the petri plate. The whole setup was placed on a tile and 1000 ml beaker was placed upside down. Grease was applied to make it intact so that air does not enter. After 24 hours, the solution was titrated with 0.1N HCl till the colour changed from pink to colourless indicating the end point. A blank was prepared in the same manner without soil sample and the volume of 0.1N HCl consumed for blank was noted down. The microbial biomass was calculated by using the following formula:

\[ \text{CO}_2 \text{ released (mg/100g)} = (B - S) \times \text{normality of acid} \times 22 \]

Where \( B = \) Vol. of 0.1N HCl consumed for Blank.

\( S = \) Vol. of 0.1N HCl consumed for Sample.

4.4.10 Available Nitrogen: 5 grams of soil was taken in distillation flask. 20 ml of distilled water and 100 ml of 0.32% KMnO₄ were added to the flask. 25 ml of N/50 H₂SO₄ was taken in a conical flask and 2–3 drops of methyl red indicator were added. The end of distillation flask was dipped into the conical flask. 100 ml of 2.5% NaOH was poured in distillation flask and it was closed immediately. Distilled ammonia was completely absorbed by H₂SO₄ present in the conical flask. Distillation
was continued till the litmus paper colour changing is stopped. The excess $\text{H}_2\text{SO}_4$ was titrated against $N/50 \text{NaOH}$ until colour changed from pink to yellow and the volume of NaOH consumed ($X_a$) for titration was noted down. Available nitrogen was calculated by using the following formula:

$$\text{% of Available Nitrogen} = \frac{25 - X_a}{0.00028} \times 100 / \text{Wt. of Soil}.$$ 

4.5 SOIL COLUMN STUDIES:

Soil column experiments have to be conducted to study the infiltration of liquid domestic waste into soil (Francois, 1975;). Laboratory experiments are useful to study the sorption capabilities of soil and to understand the mechanism of pollutants' removal (Zarnett, 1976; Chowdhry, 1979; Alhajjar et al., 1990; Gross and Mitchell, 1990; Ramaraju et al., 1999; Zhang and Shan, 1999; Gopalasamy et al., 2002) (please refer section 2.5 for details). Soil column experiments are also useful to study the mechanism of soil clogging for developing techniques that would allow more efficient use of soil (Prasad, 1977).

A circular perspex column (10 cm X 120 cm) having three outlets has been designed and fabricated as per NEERI guidelines (Olaniya et al., 1998) to study the changes in characteristics of sewage of low cost sanitation latrines, due to movement through the soil by conducting soil column experiments (third objective of study). The outlets were at the heights of 25, 50 and 90cm (Plate No. 4.4). The column was filled up to
Plate No. 4.4: Soil Column Experimental Set Up
1m height with soil collected at typical sampling station (Plate No. 4.5) and was well compacted by giving wetting and drying cycle as per standard guidelines (OECD, 2002) to achieve natural condition.

The soil used for column study was analysed for various physico-chemical characteristics viz., pH, EC, Cl, Calcium, Magnesium, PO₄, Fe, K, Na and Organic Carbon (O.C). Sewage from leach pit of ILCS unit at typical sampling station was collected (Plate No. 4.6) and analysed for various physico-chemical characteristics like pH, E.C, T.H, S0₄, Cl, F, NO₃, NO₂, PO₄, Na, K, Fe, B.O.D, C.O.D and NH₃ and applied to soil column @ 500ml per week. The experiment was designed for 75 days duration. The percolated leachate was collected every fifteenth day in PVC bags without aeration from outlets at three different depths. It was then analysed for various physico-chemical parameters. Soil samples were collected from soil column at three different depths at the end of the study and analysed for pH, E.C, Cl, O.C, PO₄, microbial biomass and available nitrogen.

4.6 STATISTICAL ANALYSIS:

Statistical analysis such as correlation are useful to determine the major contributing pollutants (Verma et al., 2003; Patil and Khaire, 2004; Hudak, 2005; Shivashankara and Sharmila, 2004). Hence, the data obtained in the present study were subjected to analysis for statistical treatments.
Plate No. 4.5: Collection of soil sample for column studies

Plate No. 4.6: Collection of sewage from leach pit of ILCS unit for column study