

CHAPTER IV

EXPERIMENTAL PROCEDURES

The study was to investigate the effects of several combinations of lead and zinc on organic removal efficiency, biokinetic coefficients, nitrification and metal distribution of the completely mixed activated sludge process in relation to metal dose concentrations. Two to four bench scale continuous flow reactors with internal cell recycle system (CFSTR) were utilized to conduct 16 steady state experiments as shown in the proposed experimental conditions, Table 9. A control unit, undosed with metals, was partly operated by another student in Laboratory and was utilized to obtain baseline data. These biological reactor systems were operated at room temperature of $30 \pm 5^{\circ}\text{C}$ in Laboratory of Sanitary Engineering Department, Chulalongkorn University, Bangkok, Thailand.

For ease of presentation, descriptions of the laboratory apparatus, the feed solution, initial start up, system operation, sampling procedure and analytical techniques are presented separately.

Laboratory Apparatus

The experimental activated sludge unit with internal cell recycle and schematic diagram of experimental apparatus utilized in this research are shown in Figure 4 and 5, respectively. Reactor was constructed of 1/8 inch Polyglass Acrylic Sheet and have a total operating volume of 10 liters. An aeration chamber and sedimentation chamber contained 8.44 and 1.56 liters, respectively. An adjustable baffle separated reactor into the aeration chamber and sedimentation

TABLE 9
THE PROPOSED EXPERIMENTAL CONDITIONS

| Metal studies | Approx. influent COD (mg/l) | Approx. influent metal concentration | | Operating θ_c (days) |
|---------------------------------|--------------------------------|--------------------------------------|----------------------------|--------------------------------------|
| | | Pb ⁺⁺ (mg/l) | Zn ⁺⁺ (mg/l) | |
| Non, i.e., Control | 500 | 0 | 0 | 4 Values (vary from 3 to 15 days) |
| Combination of lead and zinc | 500 | 1 | 1 | 4 Values (vary from 3 to 15 days) |
| Combination of lead and zinc | 500 | 5 | 1 | 4 Values (vary from 3 to 15 days) |
| Combination of lead and zinc | 500 | 1 | 5 | 4 Values (vary from 3 to 15 days) |

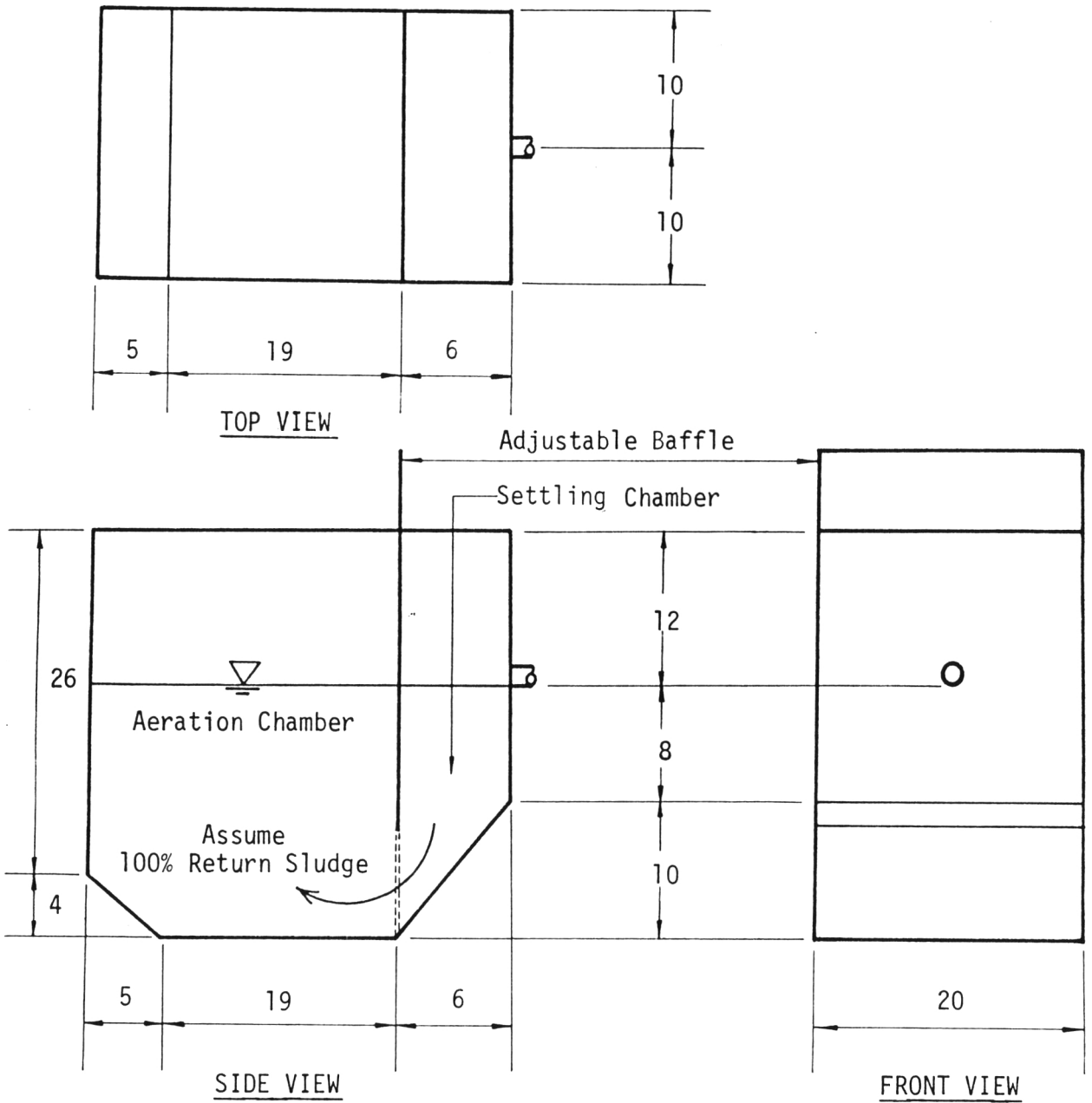


Figure 4. Experimental Activated Sludge Unit with Internal Cell Recycle
 (All dimensions are in Centimeter)

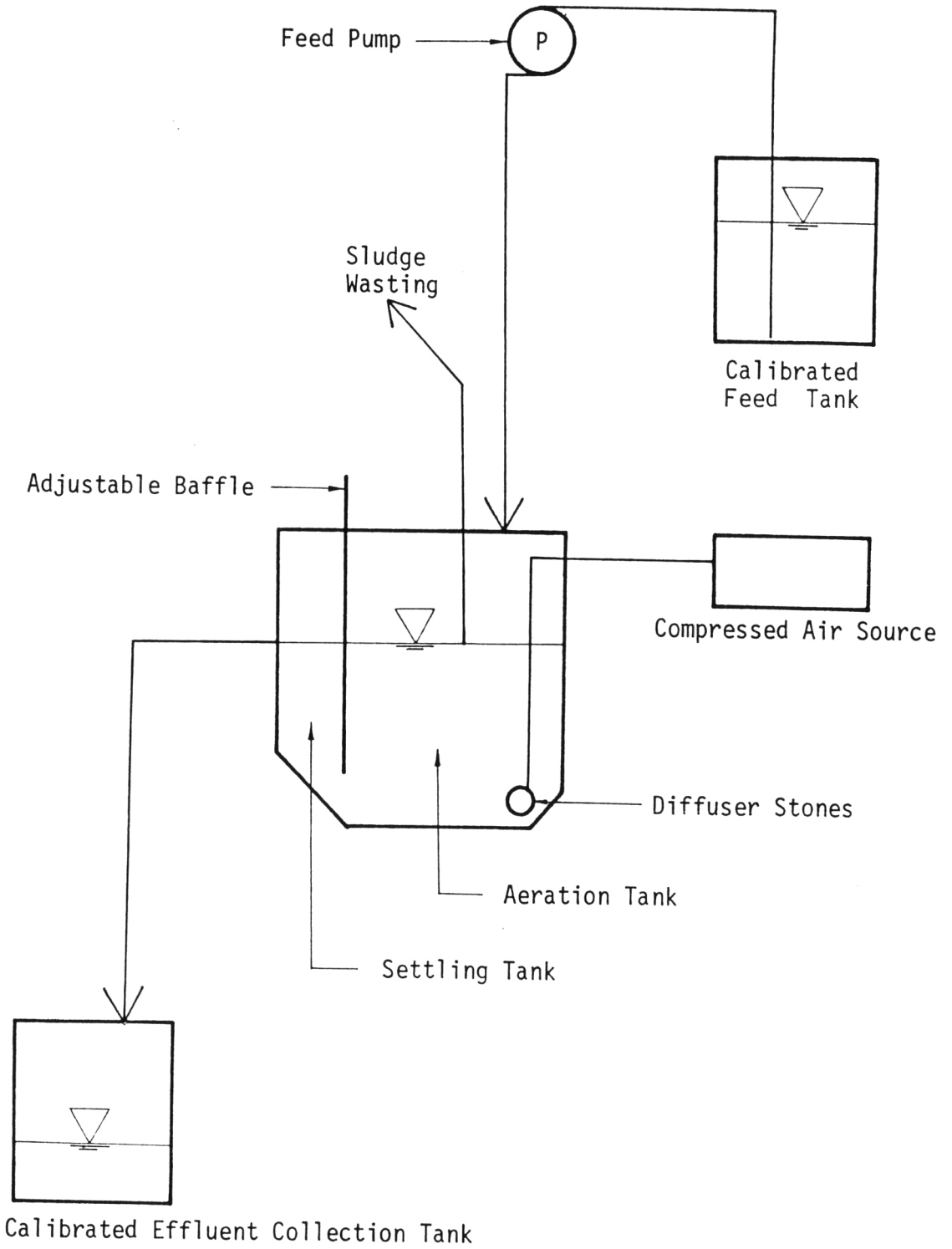


Figure 5. Schematic Diagram of the Experimental Apparatus.

chamber which allowed for internal cell recycle of bacteria cells.

The peristaltic pump and diaphragm pump were utilized to transfer the feed solution to the reactors on a continuous flow basis. The constant feed rate of 18 liters per day provided a hydraulic detention time of 11.25 hours in the aeration chamber and 2.08 hours in the sedimentation chamber. The total hydraulic detention time in the reactor was 13.33 hours.

Feed Solution

Soya-bean solution served as the carbon and energy source and was added to the feed solution in sufficient quantities to yield an approximate chemical oxygen demand of 500 mg/l. Other required nutrients were provided in the feed solution to satisfy microbial growth requirement according to the amounts given in the Table 10. The phosphate buffer was added to the system not only as a source of phosphorus but also for its buffering capacity. Supplemental nitrogen was added to the feed solution using an ammonium sulfate solution. Lead chloride and zinc chloride were added to the feed solution to provide the approximate concentration of lead and zinc, in combinations, as follows : a) 1 mg/l of lead and 1 mg/l of zinc b) 5 mg/l of lead and 1 mg/l of zinc and c) 1 mg/l of lead and 5 mg/l of zinc. No lead and zinc were added to the feed solution for the control reactor.

The calibrated 20 liters plastic container was used to prepare the feed solution daily. The plastic container was first filled with tap water to approximately 15 liters, the feed solution was added into the container with the amount depicted in Table 10. Then the container was filled to 18 liters volume with tap water and mixed thoroughly before connecting to the feed line.

TABLE 10
COMPOSITION OF FEED SOLUTION

| Constituent | Stock Concentration per liter (gm) | Quantity Used per 18 liters (ml) | Final Concentration in feed solution (mg/l) |
|--|--|--|---|
| $\text{FeCl}_3 \cdot 6 \text{H}_2\text{O}$ | 4 | 9 | 2 |
| CaCl_2 | 15 | 9 | 7.5 |
| $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$ | 100 | 9 | 50 |
| $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ | 10 | 9 | 0.5 |
| $(\text{NH}_4)_2 \text{SO}_4$ | 270 | 20 | 300 |
| KH_2PO_4^* | 108 | 50 | 300 |
| K_2HPO_4^* | 252 | 50 | 700 |
| NaHCO_3 | 90 | 100 | 500 |
| PbCl_2 | 1.36 | ** | ** |
| ZnCl_2 | 2.12 | ** | ** |
| Soya-bean solution | *** | 75 - 85 | ≈ 500 mg/l COD |

* Phosphate buffer solution

** Dependent on concentration utilized

*** Nominal COD of 100,000 - 120,000 mg/l

Initial Start Up

The original seed of microorganisms for this study were obtained from the Huay Kwang Wastewater Treatment Plant, Bangkok. The microorganisms were acclimated to the feed solution on a batch basis by a combination of domestic sewage and the feed solution. The amount of domestic sewage was gradually decreased until there was only the feed solution. After each reactor receiving only the feed solution, it was operated on a batch basis until the biomass concentration had build up to approximately 1,800 mg/l. Then transfer it to continuous flow operations. No wasting of biomass was initiated until five to seven days after continuous flow operations had begun. At the end of this period, wasting of the mixed liquor was conducted on a daily basis to achieve the desired mean cell residence time. For the metal dose units, lead and zinc were added to the synthetic wastewater as soon as the reactors were transferred to continuous flow operation.

System Operation

Total system mean cell residence time was used as the operational control parameter. Microorganisms were wasted daily to achieve desired mean cell residence times. The baffle was removed to allow complete mixing of mixed liquor in the reactor before wasting of mixed suspended solids. The baffle was replaced after wasting of microorganisms. After replacing of baffle into the position, the new feed solution was connected to the feed line and was fed into the reactor. The feed solution containers and the effluent containers were cleaned daily and the feed lines were cleaned on the every third day of operating.

Sampling Procedure

Mixed liquor suspended solids concentration was determined daily to be the parameter indicated steady state condition. As the system reached equilibrium, the sample collection and analysis were initiated to determine the parameters listed in Table 11. The samples were collected daily for approximately six days to obtain the average of steady state data.

Mixed liquor suspended solids concentration and mixed liquor volatile suspended solids concentration in reactor, suspended solids concentration in effluent, alkalinity and pH measurements were performed immediately after sampling. The milliliters of each sample were 25-50 ml for solids concentration determination and 50 ml for alkalinity determination.

A 120 ml sample for chemical oxygen demand determination was preserved by H_2SO_4 (conc.) 2 ml per liter of sample and a nitrogen concentration determination sample of 240 ml was preserved by 40 mg HgCl_2 per liter of sample. HNO_3 (conc.) 5 ml per liter of sample was used to preserve a 60 ml sample for lead and zinc concentration determinations. All of preserved samples were stored at 4°C for later analysis.

The influent feed solution and effluent were mixed thoroughly before being used for sampling. The sample for mixed liquor suspended solids concentration and mixed liquor volatile suspended solids concentration determinations was taken from the wasted sludge. Filtrated sample was obtained by filtering through the 7.0 cm GF/C, glass microfibre filter (Whatman, England).

TABLE 11
PARAMETERS MONITORED DAILY

INFLUENT FEED

Chemical Oxygen Demand
Ammonia Nitrogen Concentration
Total Kjeldahl Nitrogen Concentration
Lead Concentration
Zinc Concentration
pH
Alkalinity as mg/l CaCO_3

FILTERED EFFLUENT

Chemical Oxygen Demand
Ammonia Nitrogen Concentration
Total Kjeldahl Nitrogen Concentration
Nitrate Nitrogen Concentration
Lead Concentration
Zinc Concentration

UNFILTERED EFFLUENT

Chemical Oxygen Demand
Ammonia Nitrogen Concentration
Total Kjeldahl Nitrogen Concentration
Lead Concentration
Zinc Concentration
pH
Suspended Solids Concentration
Alkalinity as mg/l CaCO_3

TABLE 11 (Continued)

BIOLOGICAL REACTORS

Mixed Liquor Suspended Solids Concentration

Mixed Liquor Volatile Suspended Solids Concentration

Lead Concentration

Zinc Concentration

pH

Analytical Techniques

Each of the parameters monitored daily as depicted in Table 11 were conducted in accordance with the procedures outlined in Standard Method for the Examination of Water and Wastewater (1976).

Mixed liquor suspended solids concentration and mixed liquor volatile suspended solids concentration samples were determined on a dry cell weight basis as described in Standard Method. Effluent suspended solids concentration determinations were also performed. All solids concentration samples were filtered through the 7.0 cm. GF/C, glass microfibre filters (Whatman, England). The weight determinations used the Sauter Analytical Balance (GmbH D-7470).

The chemical oxygen demand determinations of unfiltered influent and effluent and filtered effluent samples were performed utilizing the dichromate reflux method as described in Standard method.

Ammonia nitrogen and organic nitrogen were performed on unfiltered influent and effluent and filtered effluent samples. All samples were analyzed on the distillation, digestion and acidimetric titration techniques as described in Standard Method.

Nitrate nitrogen determinations were made on filtered effluent samples utilizing the Brucine Method as described in Standard Method.

Alkalinity determinations of unfiltered influent and effluent samples were used the alkalinity titrations as described in Standard Method through a Beckman pH meter (Zeromatic IV). A pH of 4.5 was used as the endpoint for all alkalinity determinations. All pH values of unfiltered influent and effluent samples were read from the pH meter.

Lead and zinc concentration determinations were made on unfiltered influent, digested reactor solids, filtered effluent and digested unfiltered effluent samples. Mixed liquor suspended solids and unfiltered effluent samples were digested with nitric acid and then diluted to know a volume. All of lead and zinc concentration determinations were measured by an Atomic Absorption Spectrophotometer (Perkins Elmer 4000).