CHAPTER 3

THE RESEARCH METHODOLOGY

3.1 DESCRIPTION OF THE SBR PLANT

3.1.1 Introduction

The SBR research plant is located at the Kuala Lumpur International Airport, Sepang, Selangor. The plant was designed to serve a population equivalent (PE) of about 76,700 or 17,250 m^3/day. It was commissioned on 15 February 1998, and currently serves a population equivalent of about 15,000 to 20,000. Inflows to the SBR plant ranged from 3,000 - 4,700 m^3/day during the study period. The SBR plant was designed to treat domestic wastewater so that the treated effluent discharged will achieve a minimum Standard B effluent quality.

The SBR plant operates two parallel clusters, one for industrial and one for domestic wastewater. The clusters are operated in parallel. Each cluster comprises two SBR tanks, thus the plant has four SBR tanks in all.

The performance of the SBR plant was investigated from 23rd June 1999 to 22nd December 1999, a total of six months. At that time the plant was still under the commissioning period, and was operated and managed by the Contractor, Messrs. Johnson Fluid Engineering Sdn Bhd.

During the duration of investigation, only two out of four SBR tanks were operated at any one time. While two tanks were being used, the third tank would undergo cleaning and maintenance work. The fourth tank was kept empty and unused for the time being since the low inflow did not warrant use of the fourth tank.

3.1.2 Process Flow Description

The schematic flow chart for the plant is given in **Figure 3.1.**

The overall plant comprises the following unit processes or components:

- inlet works (screening, grit chamber),
- SBR tanks (aeration, settling),
- disinfection of effluent, and
- sludge treatment and disposal.

Wastewater was conveyed to the plant via underground sewers to the inlet works where the raw wastewater undergoes pre-treatment. Coarse solids were removed from the raw wastewater with the help of mechanical bar screens. Grit and grease were also removed in the grit and grease removal tank. The wastewater was then held in an equalization tank (capacity 1,750 m^3) before being pumped into the SBR tank.

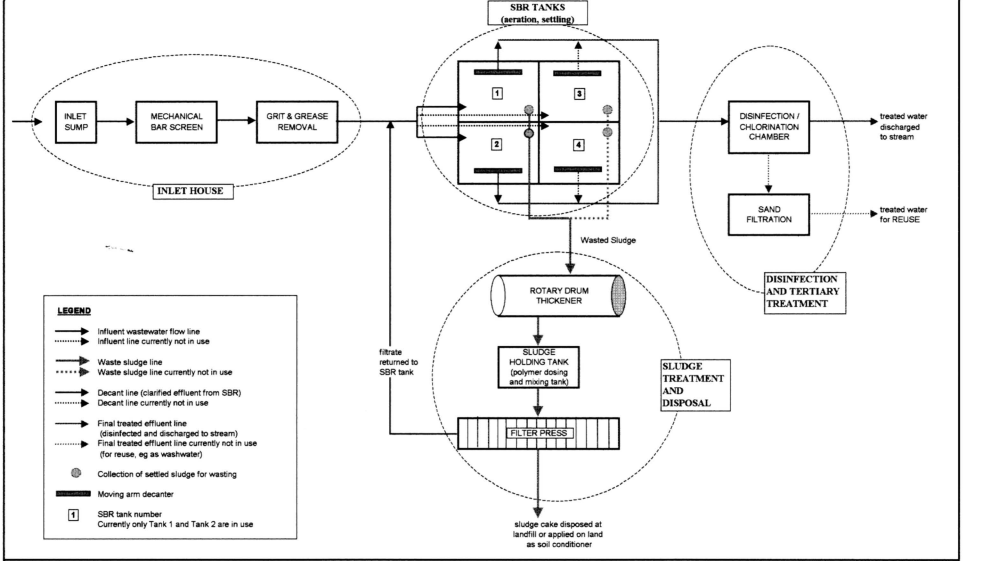

Figure 3.1 : Schematic Flow Diagram for SBR Plant at Kuala Lumpur International Airport (KLIA)

3.2 PROJECT RATIONALE

The objective of this research project is to determine the performance of the SBR with respect to reduction in BOD, COD, SS, ammoniacal nitrogen, nitrates and phosphorus, when operating under different cycle times.

The BOD, COD and SS are the basic parameters tested for performance data, and effluent quality limits are specified by the Environmental Quality Act 1974, in the Environmental Quality (Sewage and Industrial Effluents) Regulations 1978. These effluent quality limits are reproduced in **Appendix A**. Ammoniacal nitrogen, nitrates and phosphorus concentrations give an indication of the level of nutrients in the wastewater effluent. Nitrogen and phosphorus are both essential for the growth of algae, and where both nitrogen and phosphorus are plentiful, algal blooms occur which may produce a variety of nuisance conditions (Sawyer *et al.*, 1994).

Studies in other parts of the world have shown that the SBR is a good system for biological nutrient removal (Ketchum, 1997). Typically, each tank operates between one to eight cycles a day, or in other words total cycle time could range from 3 hours to 24 hours per cycle (Tchobanoglous and Burton, 1991).

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It was therefore proposed that the SBR plant concerned be operated under these two commonly used cycle times. All other design parameters, facilities, equipment, operating procedures and instrumentation remained same. The quantity and quality of influent wastewater, however, would vary, but not too drastically. The performance of the plant operating under these two cycle times could then be compared. Selected parameters of the effluent was also checked against Standard B effluent quality limits, which is the regulatory requirement (Environmental Quality Act, 1974).

3.3 EXPERIMENTAL DESIGN

The two cycle-times studied were the 8-hour cycle mode and the 6-hour cycle mode. In the 6-hour cycle mode, each SBR tank operated four cycles a day, while in the 8-hour cycle mode, each SBR tank operated three cycles a day.

In the SBR tank the wastewater underwent biological treatment, settling and clarification, in the sequence shown in **Table 3.1**.

Table 3.1: Cycle Times for 8-Hour and 6-Hour Cycle Modes

Process	8-hour cycle mode	6-hour cycle mode
Fill & Anoxic-Anaerobic Mix	2.0 hours	1.0 hour
Fill & Aerate	0 hours	1.0 hour
Aerate	3.5 hours	2.0 hours
Settle	1.5 hour	1.0 hour
Decant & Sludge Wasting	1.0 hour	1.0 hour

Each SBR tank had a volumetric capacity of 2,220 m^3. Out of this volume about 1,480 m^3 to 1,665 m^3 was retained in the tank as activated sludge buffer, while inflow and outflow from each tank during one cycle was about 555 m^3 to 740 m^3.

No Idle time was allocated in the process design for the SBR. The total fill time was not half of the total cycle time as would have been expected in a 2-tank SBR plant. This is because an equalization tank was provided to hold incoming sewage prior to biological treatment. In the typical SBR, the Idle sequence was based on the need for flow equalization and uncertainty of flow rate fluctuations. It can be near zero for peak design flows. When necessary, sludge was wasted during the idle sequence.

The wasted sludge was thickened at the sludge treatment facility, using rotary drum thickener, and dewatered using filter press to 20%-30% solids (wt/wt). The sludge cake was carted away and disposed at the designated sanitary landfill. The sludge treatment process was operated for 6 batches per day and 10 tonnes of sludge cake is

disposed every day. Some of the sludge was used for land application in landscaping of the ground around the Sewage Treatment Plant.

The Tertiary Treatment and Reuse plants were not commissioned at the time of the study due to low hydraulic loading (3 ML/day).

8-Hour Cycle Mode

In the 8-hour cycle mode, the cycle started with the Fill sequence which lasted 2.0 hours. During Fill, the contents of the SBR tank were slowly mixed in anoxic-anaerobic conditions. Mixing enhanced the dispersion of the feed substrate throughout the activated sludge content which was rich in micro-organisms. Under anaerobic conditions, some of the carbonaceous BOD and COD were removed due to consumption by anaerobic micro-organisms in the activated sludge. Phosphorus release occurred. During the anoxic-anaerobic mix, nitrates and nitrites were converted to nitrogen gas which was released to the atmosphere. This process is known as anoxic denitrification or anaerobic denitrification. It has been observed that incorporation of an anoxic or anaerobic phase enhanced the nutrient removal ability of the plant (Tchobanoglous and Burton, 1991).

After two hours of Fill and Mix, the SBR went into its next sequence which was the Aerate phase. The Aerate sequence lasts 3.5 hours, and no Filling was carried out during this phase. During this time, the contents of the SBR were aerated using fine-bubble diffusers, which also caused the contents to be thoroughly mixed. Thus the

contents of the SBR tank were also known as mixed liquor suspended solids (MLSS). Under aerobic conditions, the aerobic micro-organisms in the MLSS consumed much more of the carbonaceous feed substrate, thus reducing the concentration of BOD and COD in the wastewater significantly. During the aerobic phase of the treatment, reduction of biological nutrients was expected to occur. The wastewater underwent nitrification whereby ammoniacal nitrogen was oxidised to nitrites and nitrates by nitrifiers in the wastewater. Phosphorus was expected to be consumed during the aerobic phase.

The Settle sequence was next, set to last 1.5 hours. During this time, the sludge blanket settled in the SBR tank under truly quiescent conditions (i.e. no mixing, no filling, no aeration, no decanting, no sludge wasting), leaving a clear supernatant. Observations at the plant indicated that sludge settled to about half the volume of the tank within the first half-hour of the Settle sequence, after which settling proceeded more slowly due to filamentous growth and sludge bulking hindering the settling process.

The Decant sequence which followed was set to proceed for 1.0 hour. Once the Decant sequence was activated, the stainless steel moving arm decanter, comprising a weir and effluent pipes, was slowly lowered onto the surface of the supernatant at a specified rate to ensure minimal turbulence at the weir. The clear supernatant above the sludge blanket flowed over the weir into the effluent pipes and was conveyed to the disinfection chamber before final discharge. The decanter was lowered at a very

low speed, so that effluent flow rates were low and turbulence at the point of draw-off was minimised. Draw-off rates ranged from 62.5 to 83.3 l/lin.m-min (liters per linear meter per minute). The linear meter here refers to the length of the decanter. This draw-off was equivalent to about 90 to 120 m^3/m.day.

Sludge wasting was carried out simultaneously with Decant. The base of the SBR tank sloped slightly down towards the inlet portion of the tank. Sludge collected in the sludge trough along the inlet wall of the SBR tank. Sludge pumps located in the sludge trough conveyed the wasted sludge to the waste sludge treatment facility.

6-Hour Cycle Mode

In the 6-hour cycle mode, the sequence was slightly modified to include a Fill-Aerate phase after the Fill-Anoxic Mix phase. The cycle began with Fill and Anoxic-Anaerobic Mix sequence which proceeded for 1 hour. After 1 hour, the aerators were activated, and filling was continued for another 1 hour. Thus, filling and aeration occurred simultaneously during this phase.

Filling was carried out for a total of two hours (from the start of the cycle – same as for the 8-hour mode). After the two hours of filling, the feed valve was closed so that filling stops. However, aeration was continued for another two hours after filling was stopped. Thus, aeration proceeded for a total of three hours during the 6-hour mode, half an hour less than for the 8-hour mode.

The aerobic phase was followed by the Settle sequence which lasted one hour during the 6-hour cycle mode (half an hour less than the 8-hour mode).

As for the 8-hour mode, the Decant sequence proceeded for one hour. Sludge wasting was carried out simultaneously with Decant.

3.4 METHODOLOGY

In order to determine the performance of the SBR plant, sampling and monitoring instruments were located at different sections of the plant as follows:

- Inlet sump (to monitor influent quality) - non-composite samples, and
- Effluent discharge (to monitor effluent quality) - composite samples.

Four refrigerated automatic samplers were provided for water sampling at the plant. The sampler program flowchart used is provided in **Figure 3.2a** and **Figure 3.2b.** Two samplers (one sampler for domestic stream and one sampler for industrial stream) were installed at the inlet house. The other two samplers were used for taking effluent samples, one after Re-use Water Chlorination tank and the last one after Effluent Chlorination Tank. The final effluent was sampled using composite sampler.

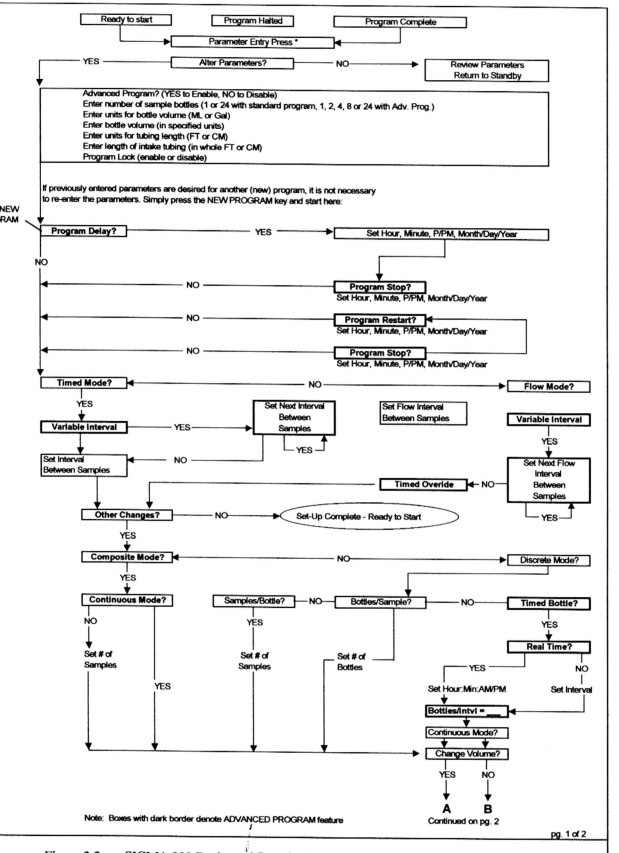

Figure 3.2a: SIGMA 900 Registered Sampler Program Flowchart (1)

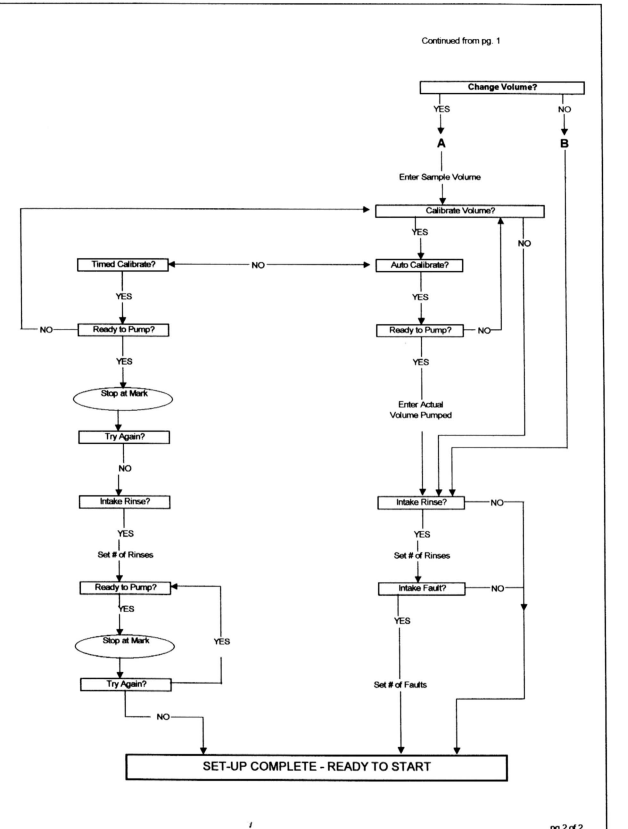

Figure 3.2b: SIGMA 900 Registered Sampler Program Flowchart (2)

Composite samples from the SBR plant were analysed when it was operating under 6-hour cycle mode from 23 August to 22 October 1999 (2 months). Similarly, composite samples were taken from the SBR plant operating under 8-hour cycle mode, from 23 June - 22 July 1999 (one month) and again from 23 November to 22 December 1999 (one month).

The composites comprised six one-hourly samples mixed on the basis of flow rate. The samples were collected daily and analyzed for eight parameters: temperature, pH, SS, BOD, COD, ammoniacal nitrogen, nitrate nitrogen, and phosphorus. The laboratory tests used are summarised in **Table 3.2**.

Table 3.2: Laboratory Tests

No.	Laboratory Equipment (Hach method no.)	Test	Range
1	Thermometer	Temperature	-20 – 105°C
2	pH meter (method no. 8156)	pH	-2 – 19.99
3	Gravimetric (method no. 8158)	Suspended solids	–
4	DO meter (method no. 8043)	BOD_5	0-20 ppm
5	Spectrophotometer (method no. 8000)	COD	0-150 ppm
6	Nessler method (method no. 8038)	NH_3-N	0-2.5 ppm
7	Cadmium method (method no. 8039)	NO_3-N	0-30 ppm
8	Acid Persulfate digestion method (method no. 8190)	Total Phosphorous	0-3.5 ppm

The analysis of the selected parameters was carried out using the HACH Method which is accepted by the USEPA, and equivalent to the relevant Standard Methods for Examination of Water and Wastewater (HACH, 1997). The preparation of the sample followed the procedures described in detail in the HACH Water Analysis Handbook (HACH, 1997). Where appropriate the DR/2010 Spectrophotometer was used for taking the readings.

Taking the measurements using the DR/2010 Spectrophotometer was carried out in the following way.

(i) The instrument was turned ON, and the Constant-On mode was selected. The instrument was left on for about 5 minutes to warm up.

(ii) The stored program number for absorbance was entered (a different number for a different test)

(iii) The wavelength dial was rotated until the small display showed the approximate wavelength of interest (upon entering the stored program number, the appropriate wavelength number is displayed).

(iv) The sample and blank was prepared for analysis. The appropriate sample cells (10-ml or 25-ml cells, 2-ml COD vials) were filled with the sample solutions and blank.

(v) The blank was placed in the cell holder in the DR/2010 Spectrophotometer and the light shield was closed.

(vi) The ZERO button was pressed, and the display read "Zero". This is called "zeroing".

(vii) The blank was then removed from the cell holder and replaced with the prepared sample, and the light shield was closed.

(viii) The READ button was pressed to read the absorbance. The measurement of the sample absorbance is automatically converted to concentration in mg/l.

All samples were collected in clean 1-liter polyethylene bottles and analysed immediately or within 24 hours. If not analysed immediately, the appropriate preservation procedures were used, followed by refrigeration (stored at 4°C). The procedures used for analysing the selected eight parameters are described in the following sub-sections.

3.4.1 Temperature

Temperature in degrees Centigrade was measured using a Zeal thermometer, with a measuring range from -20 to 105°C. The thermometer has a scale marked for every 0.1°C. Readings were taken after immersing the thermometer in the sample long enough to permit complete equilibration. Results are reported to the nearest 0.1°C.

3.4.2 pH

The method used is called the Hach One Combination pH Electrode Method (Hach Method 8156) with an EC10 Portable pH Meter. Samples were collected in clean plastic bottles, which were filled completely and capped tightly.

The pH meter was first calibrated. The POWER key was pressed on and pH mode was selected. The CAL key was pressed, then the meter went into measure mode with a "P1" in the display. The electrode was placed into a pH 4 buffer solution. When the display stabilized, the meter beeped and showed the temperature corrected value for the pH 4 buffer and a ready indicator. The YES button was pressed to accept this value. The electrode was rinsed with deionized water and blotted dry. The electrode was placed in the pH 7 buffer and the dispenser button was pressed. When the display stabilized, the meter beeped and showed the temperature corrected value for the pH 7 buffer and "ready". This value was accepted by pressing YES. The slope appeared in the display (59.0 $\pm$ 3 mV/decade). This slope was accepted by pressing YES. The electrode was rinsed with deionized water.

The electrode was then placed into the sample and the dispenser button pressed to measure the sample pH. It was ensured that the entire sensing end was submerged and that there were no air bubbles under the electrode. The pH value was recorded when the display stabilized. Finally, the electrode was rinsed thoroughly with deionized water and blotted dry.

3.4.3 Suspended Solids (SS)

The determination of suspended solids is extremely valuable in the analysis of polluted waters. It is one of the major parameters used to evaluate the strength of domestic wastes and to determine the efficiency of treatment units. From the viewpoint of

stream pollution control, the removal of suspended solids is as important as BOD removal. Suspended solids determination is also used to control the solids in the activated sludge process. (Sawyer *et al.*, 1994).

Usually, the sample size is limited to 50ml or less because of difficulties encountered in filtration of larger samples. The weight of solids removed seldom exceeds 20 mg and is often less than 10 mg. Sufficient sample should be filtered to yield an increase in weight of about 10 mg. This often requires the filtration of 500 ml or more of samples of biologically treated wastewaters. Suspended solids are reported in terms of milligrams per liter (mg/l). (Sawyer *et al.*, 1994)

Procedure

The SS was measured using the Gravimetric Method (Hach Method 8158) for suspended solids.

A 47-mm filter disc was placed in the filter holder with the wrinkled surface upward. Tweezers were used to handle the filter discs as fingers add moisture which subsequently cause a weighing error. The filter holder assembly was placed in the filtering flask and 100 ml of deionized water was added. Vacuum was applied to the flask until all the water was drawn through the filter. The disc was then removed from the filter holder and transferred to a watch glass. This was placed in a drying oven at 103°C for one hour. The drying oven was preheated to 103°C to ensure adequate drying of the filter disc during the one-hour period.

After the one-hour drying period, the disc with the watch glass was removed from the oven and placed in a desiccator, where it was allowed to cool to room temperature. Metal tongs were used to transfer the watch glass and filter disc from the oven directly into the desiccator, and the desiccator was covered immediately.

After cooling, the disc was removed from the desiccator and weighed to the nearest 0.1mg using an analytical balance. This was the weight of the disc without residue (B). The watch glass and disc was removed from the desiccator as a unit and placed beside the analytical balance. Plastic tweezers were used to remove the disc from the watch glass and to transfer the disc to and from the weighing pan of the balance.

Again the disc was placed in the filter holder/flask assembly with the wrinkled surface upward. The disc was wetted with deionized water to ensure adhesion to the holder. The 100 ml of the well-mixed representative water sample was filtered by applying vacuum to the flask. This was followed with three separate 10 ml washings of deionized water. The vacuum was slowly released from the filtering system and the filter disc was gently removed from the holder. The disc was placed on a watch glass. The filtrate was inspected to ensure that proper trapping of solids was accomplished on the disc. Again the watch glass and filter was placed in a drying oven at 103°C for one-hour. The watch glass and filter was removed from the oven after drying and placed in a desiccator to cool to room temperature. The disc was carefully removed from the desiccator and weighed to the nearest 0.1 mg using an analytical balance.

This was the weight of the disc with residue (A). Care was taken when removing the lid of the desiccator so as not to disturb the dried suspended matter on the disc. The disc was then discarded. The SS was then calculated from the Equation 4 as follows, and tabulated.

$$SS = \frac{A - B}{\text{Sample volume in liters}} \text{ mg/l} \qquad \text{(Equation 4)}$$

where:

A = weight of disc with residue, mg

B = weight of disc without residue, mg

3.4.4 Biochemical Oxygen Demand

BOD or biochemical oxygen demand is a regulatory parameter used by the Department of Environment (DOE) of Malaysia to monitor water quality. BOD is usually defined as the amount of oxygen required by bacteria while stabilizing decomposable organic matter under aerobic conditions.

BOD removal from a wastewater by a biological sludge may be considered as occurring in two phases (Eckenfelder, 1989a). An initial high removal of suspended, colloidal, and soluble BOD is followed by a slow progressive removal of remaining soluble BOD. Initial BOD removal is accomplished by one or more mechanisms, depending on the physical and chemical characteristics of the organic matter. These mechanisms are:

- Removal of suspended matter by enmeshment in the biological floc. This removal is rapid and is dependent upon adequate mixing of the wastewater with the sludge
- Removal of colloidal material by physicochemical adsorption on the biological floc.
- A biosorption of soluble organic matter by the micro-organisms. There is some question as to whether this removal is the result of enzymatic complexing or is a surface phenomenon and whether the organic matter is held to the bacterial surface or is within the cell as a storage produce or both. The amount of immediate removal of soluble BOD is directly proportional to the concentration of sludge present, the sludge age, and the chemical characteristics of the soluble organic matter.

These three reactions occur immediately on contact of sludge with wastewater. The colloidal and suspended material must undergo sequential breakdown to smaller molecules in order that it may be made available to the cell for oxidation and synthesis (Eckenfelder, 1989a).

Procedure

The BOD was measured using the Dilution Method (Hach Method 8043). The five-day BOD measurement at 20°C is an empirical measurement of the oxygen requirements of municipal and industrial wastewaters and sewage. The test results are

used to calculate the effect of waste discharges on the oxygen resources of the receiving waters. The BOD was performed by incubating a sealed wastewater sample (or a prepared dilution) for the standard five-day period and then determining the change in dissolved oxygen content. The BOD value was then be calculated from the results of the dissolved oxygen tests.

The sample dilution water was prepared using a BOD Nutrient Buffer Pillow. For 3 liters of dilution water, 1 pillow (Catalog No. 14861-98) was used. The BOD Nutrient Buffer Pillow comprises calcium chloride, ferric chloride, magnesium sulfate and phosphate buffer in powder form. The pillow was shaken, cut open and the contents added to a jug containing the proper amount of 20°C distilled water. The jug was capped and shaken vigorously for one minute to dissolve the slurry and to saturate the water with oxygen.

With a serological pipet, a graduated series of four portions of well-mixed sample was measured and transferred to separate 300 ml glass stoppered BOD bottles. The sample sizes selected for influent samples was 2, 4, 6 and 8 ml, while sample sizes selected for effluent samples was 15, 30, 45 and 60 ml. The sample was stirred with the pipet before pipetting each portion. One of the BOD bottles was left as the dilution water blank. Two shots of Nitrification Inhibitor (approximately 0.16 g) was added to each bottle; this was meant to inhibit the oxidation of nitrogen compounds.

Each BOD bottle was filled to just below the lip with the dilution water. When adding the water, it was allowed to flow slowly down the sides of the bottle to prevent formation of bubbles. The bottle was stoppered, then inverted several times to mix. The initial DO value was determined, using DO meter (YSI Model 55).

Enough water was added to the lip of the BOD bottle to make a water seal. A plastic overcap was placed over the lip of each bottle and the bottles were placed in an incubator at 20 $\pm$ 1 °C. The samples were incubated in the dark for five days.

When the five-day incubation period was completed, the DO content was determined (dissolved oxygen remaining) in each bottle, by using the DO meter. The BOD was calculated using the following equation (Equation 5):

$$BOD_5, \text{ mg/l} = \frac{(D1 - D2) - (B1 - B2) \times \text{Volume of Bottle}}{\text{Volume of sample used}} \qquad \text{(Equation 5)}$$

where,

D1 = DO of diluted sample immediately after preparation, mg/l

D2 = DO of diluted sample after 5 day incubation at 20 °C, mg/l

B1 = DO of initial blank

B2 = DO of final blank

3.3.5 Chemical Oxygen Demand (COD)

The chemical oxygen demand or COD test is widely used as a means of measuring the organic strength of domestic and industrial wastewater. This test allows measurement of a waste in terms of the total quantity of oxygen required for oxidation to carbon dioxide and water. It is based on the fact that all organic compounds, with few exceptions, can be oxidized by the action of strong oxidizing agents under acid conditions. While the COD values are generally greater that BOD values, the COD test is unable to differentiate between biologically oxidizable and biologically inert organic matter, and does not provide any evidence of the rate at which the biologically active material would be stabilized under conditions that exist in nature. (Sawyer *et al.*, 1994).

Procedure

The COD was measured using the Dichromate Reactor Digestion Method (Hach Method 8000). The mg/l COD results are defined as the mg of O_2 consumed per liter of sample under conditions of this procedure.

In this procedure, the sample was heated for 2 hours with a strong oxidizing agent, potassium dichromate. Oxidizable organic compounds react, reducing the dichromate ion ($Cr_2O_7^{2-}$) to green chromic ion (Cr^{3+}). In the 0-150 mg/l colorimetric measurement used here, the amount of Cr^{6+} remaining was determined. The COD reagent also contains silver (a catalyst) and mercury ions (used to complex chloride interferences).

For storage up to 28 days, the samples were treated with sulphuric acid to a pH of less than 2 (about 2 ml per liter) and refrigerated at 4°C. All samples containing solids were homogenized to assure representative samples.

The sample of volume 100ml was mixed, then homogenized for 30 seconds in a blender. The homogenized sample was poured into a 250 ml beaker and gently stirred with a magnetic stir plate. The COD Reactor was turned on and preheated to 150°C. A plastic shield was placed in front of the reactor as protection from splattering should reagent leaking occur. The COD Digestion Reagent Vial for the low range (0 to 150 mg/l) was selected, and the cap removed. The vial was held at a 45-degree angle, and 2.00 ml of the sample was pipetted into the vial. The vial cap was then replaced tightly. The outside of the COD vial was rinsed with deionized water and wiped clean with a paper towel. The vial was held by the cap over a sink and inverted gently several times to mix the contents. The vial was then placed in the preheated COD Reactor.

A blank was prepared in the same way as above, substituting 2.00 ml of deionized water for the sample. For each set of samples, one blank was run.

The vials were heated for 2 hours in the COD Reactor, after which the reactor was turned off. The vials were allowed to cool for about 20 minutes to 120°C or less. Each vial was inverted several times while still warm, then placed into a rack, and cooled to room temperature.

The COD was then measured using the Colorimetric method, on the Hach DR/2010 Spectrophotometer. For the low range (0 to 150 mg/l COD), the stored program number (430) was entered. The wavelength dial was rotated until the small display showed 420 nm. The COD Vial Adapter was placed into the cell holder with the marker to the right. The outside of the blank was cleaned with a towel. The blank was placed into the adapter with the Hach logo facing the front of the instrument and the cover was placed on the adapter. The ZERO button was pressed for "zeroing" and **0 mg/l COD LR** displayed.

The outside of the sample vial was then cleaned with a towel, then placed into the adapter with the Hach log facing the front of the instrument. The adapter was covered, then the READ button was pressed to show the result in mg/l COD.

3.4.6 Ammoniacal nitrogen (NH_3-N)

In wastewater treatment, ammoniacal nitrogen analysis is important in determining whether sufficient available nitrogen is present for biological treatment. Reduced forms of nitrogen are oxidized in natural waters, thereby affecting the dissolved-oxygen resources. Autotrophic conversion of ammonia to nitrites and then to nitrates requires oxygen, so the discharge of ammoniacal nitrogen and its subsequent oxidation can seriously reduce the dissolved oxygen levels in rivers and estuaries, especially where long residence times required for the growth of the slow-growing nitrifying

bacteria are available. Their discharge together with the treated effluent can cause rapid nitrification to occur in waterways. (Sawyer *et al.*, 1994)

Procedure

The ammoniacal nitrogen concentration in the samples was determined using the Nessler Method (Hach Method 8038). The stored program number for ammoniacal nitrogen (380) was entered in the DR/2010 Spectrophotometer. The wavelength dial was then rotated until the small display showed 425 nm.

A 25-ml graduated mixing cylinder was filled to the 25-ml mark with the prepared sample. Another 25-ml graduated mixing cylinder was filled with deionized water (the blank).

Three drops of Mineral Stabilizer was added to each cylinder, which was then inverted several times to mix. The Mineral Stabilizer complexes hardness in the sample. Then, 3 drops of Polyvinyl Alcohol Dispersing Agent was added to each cylinder, and again the cylinders were inverted to mix the contents. The Polyvinyl Alcohol Dispersing Agent aids the colour formation in the reaction of Nessler Reagent with ammonium ions. Next, 1.0ml of Nessler Reagent was pipetted into each cylinder, which was stoppered and inverted to mix. If ammonia was present, a yellow colour developed. The reagent caused a faint yellow colour in the blank. The yellow colour was formed in proportion to the ammonia concentration.

The SHIFT and TIMER buttons on the DR/2010 Spectrophotometer were pressed, to begin a one-minute reaction period. The prepared solution from each cylinder was poured into a sample cell. Upon beeping of the timer, the blank was placed into the cell holder, and the light shield was closed. The ZERO button was pressed, for "zeroing" – and the display showed **0.00 mg/l NH_3-N Ness**. Next the prepared sample was placed into the cell holder, and the light shield was closed. The READ button was pressed to display the result in mg/l of ammoniacal nitrogen.

3.4.7 Nitrate Nitrogen (NO_3^--N)

The determination of nitrate nitrogen in water is important in determining whether water supplies meet U.S. Environmental Protection Agency maximum contaminant levels for the control of methemoglobinemia in infants. For this reason the U.S. Environmental Protection Agency has set a maximum contaminant level requiring that the nitrate-nitrogen concentration not exceed 10 mg/l in public water supplies. (Maxcy, 1950).

Procedure

The nitrate nitrogen concentration in the samples was measured using the Cadmium Reduction Method (Hach Method 8039). This method uses the fact that cadmium metal reduces the nitrates present in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt. This salt couples to gentisic acid to from an amber-coloured product.

The samples were collected in clean plastic bottles, stored at 4 °C or lower and analysed within 24 to 48 hours. The samples were warmed to room temperature before running the test.

The stored program number (355) for the high range of nitrate nitrogen was entered. The wavelength dial was then rotated until the small display showed 500 nm.

The sample was prepared as follows: The sample cell was filled with 25 ml of sample. The contents of one NitraVer 5 Nitrate Reagent Powder Pillow was added to the cell and the cell was stoppered. The SHIFT and TIMER buttons on the DR/2010 Spectrophotometer were pressed. The cell was shaken vigorously until the timer beeped in one minute. When the timer beeped, the buttons SHIFT and TIMER were pressed again, to begin a five-minute reaction period. An amber colour developed if nitrate nitrogen was present.

Another sample cell was filled with 25 ml of deionised water (the blank). A reagent blank must be determined on each new lot of NitraVer 5.

When the timer beeped after the 5-minute reaction period, the blank was placed into the cell holder and the light shield was closed. The ZERO button was pressed, to perform "zeroing" – the display showed **0.0 mg/l NO_3^--N HR**. Next, the prepared sample was placed into the cell holder, and the light shield closed. Finally, the READ

button was pressed and the display showed the result in mg/l nitrate nitrogen. The sample cell was immediately rinsed after use to remove all cadmium particles.

3.4.8 Total Phosphorus

Phosphorus is essential to the growth of organisms and can be the nutrient that limits the primary productivity of a body of water. In instances where phosphate is a growth-limiting nutrient, the discharge of raw or treated wastewater to that body of water may stimulate the growth of photosynthetic aquatic micro- and macro-organisms in nuisance quantities. Phosphorus occurs in the wastewater almost solely as phosphates. They occur in solution, in particles or detritus, or in the bodies of aquatic organisms. Organic phosphates are formed primarily by biological processes. They are contributed to sewage by body wastes and food residues, and also may be formed from orthophosphates in biological treatment processes or by receiving water biota. Phosphates also occur in bottom sediments and in biological sludges, both as precipitated inorganic forms and incorporated into organic compounds. (Sawyer *et al.*, 1994).

Procedure

The Total Phosphorus (also called Organic and Acid Hydrolyzable) content of the samples was determined using the Acid Persulfate Digestion Method (Hach Method 8190). This procedure was immediately followed by the Ascorbic Acid Method (Hach Method 8048) to determine the orthophosphate or reactive phosphorus content of the sample.

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate.

Samples were collected in 1-liter plastic bottles that had been cleaned with 1:1 Hydrochloric Acid solution and rinsed with deionised water. Samples were tested immediately after collection for best results. However, if this was not possible, the samples were preserved up to 28 days by adjusting the pH to 2 or less with sulphuric acid (about 2 ml per liter) and storing at 4°C. The samples were warmed to room temperature before testing.

First, 25 ml of the sample was measured into a 50-ml erlenmeyer flask using a graduated cylinder. All glasswares used were rinsed with 1:1 Hydrochloric Acid solution, then rinsed again with deionised water. The contents of one Potassium Persulfate Powder Pillow was added to the flask, and the flask was swirled to mix thoroughly. Next, 2.0 ml of 5.25 N sulphuric acid solution was added to the mixture using a 1-ml calibrated dropper. The sample in the flask was concentrated to less than 20 ml for best recovery. After concentration, the volume was maintained near (but not exceeding) 20 ml, by adding small amounts of deionised water.

The flask was then placed on a hot plate, and boiled gently for 30 minutes. After boiling, the sample was cooled to room temperature. Next, 2.0 ml of 5.0 N sodium hydroxide solution was added using the 1-ml calibrated dropper provided, and the mixture was swirled to mix. The sample was poured into a 25 ml graduated cylinder, and the volume was made up to 25 ml by adding deionized water rinsings from the flask.

The reactive phosphorus test described below was then carried out. The results of the reactive phosphorus test at this point includes the phosphate plus the orthophosphate and the acid-hydrolyzable (condensed) phosphate.

The PhosVer 3 (Ascorbic Acid) Method (Hach Method 8048) was used to determine the reactive phosphorus content. The orthophosphate reacts with molybdate in an acid medium to produce a Phosphomolybdate complex. Ascorbic acid then reduces the complex, giving and intense molybdenum blue colour.

The stored program number for this method (490) was entered. The wavelength dial was rotated until the small display showed 890 nm.

A 10-ml Cell Riser was inserted into the cell compartment. A 10-ml sample cell was filled with 10 ml of sample. The contents of one PhosVer 3 Phosphate Powder Pillow for 10-ml sample was added to the cell (the prepared sample), and the cell was swirled immediately to mix. A blue colour formed if phosphate was present.

The SHIFT and TIMER buttons were pressed to start a ten-minute reaction period. Meanwhile, a second 10-ml sample cell was filled with 10 ml of deionised water (the blank). When the timer beeped, the blank was placed in the cell holder and the light shield was closed. The ZERO button was pressed to carry out "zeroing" – the display showed **0.0 mg/l PO_4^{3-} PV**. Next the prepared sample was placed into the cell holder, and the light shield was closed. On pressing the READ button, the results were displayed in mg/l PO_4^{3-}. This measurement gives for the total phosphorus concentration of the sample.

3.4.9 Statistical Analysis of Data

Data obtained was analysed statistically to obtain arithmetic mean and standard deviation, as well as minimum and maximum values for each run.

For significance testing, a student's two-tailed t-test was applied to determine whether the means from two sets of sample were equal. For example, the t-test was applied to determine whether the concentration of BOD in the influent was significantly different from the concentration of BOD in the effluent for any given run. Similarly, the t-test was applied to determine whether the concentration of BOD in the effluent of one run (say the June-July 1999, 8-hour mode run) was significantly different from the concentration of BOD in the effluent of a different run (say the September-October 1999, 6-hour mode run). In the same way, the t-test was applied for the other selected parameters – COD, SS, ammoniacal nitrogen, nitrate nitrogen and phosphorus.

A correlation analysis was also applied to determine if there was any correlation between parameters within the same run.

The detailed results of the statistical analysis are summarised in **Appendix E.**

3.5 LIMITATION OF THE STUDY

This study is subject to the following limitations:

- The study was carried out in equatorial climate, ambient atmospheric temperature between 26°C and 33°C.
- The type of wastewater studied is municipal or domestic wastewater only.
- The temperature of the influent flow is between 26°C and 30°C.
- The quantity of influent is between 1,800 m^3,day and 5,100 m^3/day.
- Coarse solids, grit and grease is removed from the wastewater before entering the SBR tank
- An equalization tank is provided so that no idle time is required.
- At least 1 hour of Anoxic-Anaerobic Mix is provided.
- At least 3 hours of Aerate is provided.
- At least 1 hour of Settle is provided.
- At least 1 hour of Decant is provided.
- Provide sufficient ventilated space around the SBR tanks.