CHAPTER 3



METHODOLOGY

The methodology in this experiment was performed following the ANSI/ASCE-2-91 standard (1993), "The measurement of oxygen transfer rate in clean water" (ASCE, 1993). The oxygen transfer tests were conducted with:

- 2 sizes of 3 blades propellers diameter which were 10.7 cm and 15 cm
- 3 surfactant concentrations: 0 mg/L, 5 mg/L and 10 mg/L
- 4 power inputs: 0.5 hp/1000 ft³, 1 hp/1000 ft³, 1.5 hp/1000 ft³, and 2 hp/1000 ft³ (13.2 watt/m³, 26.3 watt/m³, 39.5 watt/m³, and 52.7 watt/m³)

A suitable power input in aeration process is necessary to adjust when surfactant is presented. In this research, one focuses on the oxygen transfer rate (K_La) by varying the power input in the presence of surfactant. The K_La values, which vary with the power input, are calculated by DO parameter estimation program. The guidelines for oxygen transfer measurements in clean water (ASCE, 1993) allow the simulation of the oxygen transfer rate of aeration systems in wastewater via an addition of 5 mg/L of an arbitrary anionic surfactant into clean water. The oxygen transfer tests are conducted with the common unsteady state absorption method (addition of sodium sulfite and cobalt salt as a catalyst oxidation).

The experimental procedure was shown in Figure 3.1. The details in each step were described in this chapter.



Figure 3.1 Experimental design

3.1 Materials and Apparatus

3.1.1 Tank

In the experiment, approximately 500 liters of baffled tank was used as a reactor as shown in Figure 3.5. The dimension of tank was 1.1 m height and 0.65 m tank diameter. Four baffles were installed extending approximately one-tenth in tank diameter out from the wall to eliminate vortex conditions. The depth of the propeller was set with the tank standard, 0.8 < depth/diameter < 1.2 (Metcaft and Eddy, 2003).

3.1.2 Water

For determination of a standard oxygen transfer rate, the water to which oxygen is transferred was added with surfactant. The unsteady-state clean water test was occasionally conducted in clean water and clean water with surfactant addition in an effort to mask the effect of trace contaminants in tap water or to roughly simulate transfer in municipal wastewater (ASCE, 1993).

Tap water was used as clean water. Dodecyl Sodium Sulfate (DSS) was used as a surfactant and the optimum oxygen transfer coefficient in the presence of surfactant was tested. The concentrations of surfactant were 0 mg/L, 5 mg/L and 10 mg/L, respectively.

3.1.3 Oxygenation Device

The mechanical high-speed surface aerator was installed in the tank for this experiment. The depth of the two sizes of propeller, 10.7 cm and 15 cm, were determined. Preliminary tests to find the optimum depth for each propeller were performed. The optimum depth below the water surface was 5 cm for both sizes of propellers. The results of preliminary test were shown in Appendix A.

For surface aerator, three-phase induction motor, 1 hp footmounted motor, was used to perform these tests. The converter, T-verter, E2-201-H1, was used to control RPMs.

3.1.4 Dissolved Oxygen Measurement

Membrane electrode measurement of DO, CONSORT model Z921T, was used to measure the DO concentration.



Figure 3.2 DO Probe, CONSORT model Z921T

3.1.5 Temperature Measurement

Water temperature was measured by the thermometer in the DO probe.

3.1.6 Total Dissolved Solid and pH Measurement

Portable conductivity meter, Hatch model sension 5, was used to measure total dissolved solid. The pH value was measured by using Benchtop pH meter, Hatch model sension 5.



Figure 3.3 Portable conductivity meter, Hatch model sension 5



Figure 3.4 Benchtop pH meter, Hatch model sension 5

3.1.7 Deoxygenation Chemicals

3.1.7.1 Sodium Sulfite

Analytical reagent grade, sodium sulfite (Na_2SO_3) was used for deoxygenation in accordance with ASCE standard method (ASCE, 1993).

3.1.7.2 Cobalt Catalyst

Analytical reagent grade of cobalt chloride hydrate, $CoCl_2 \cdot 6H_2O$ was used to catalyze the deoxygenation reaction in accordance with ASCE standard method (ASCE, 1993)

3.1.7.3 Surfactant

Analytical reagent grade of Dodecyl Sodium Sulfate was used as a surfactant in this experiment.

3.1.8 Computer Program

ASCE DO parameter estimation program (DO_PAR) (Lee *et al.*, 1997) was used to analyze data to estimate DO parameter. After inputting the data, the graph between DO versus power input per unit volume was created by the program. The example of DO parameter estimation input and output from DO_PAR was shown in the Appendix B.

3.2 Procedure

3.2.1 Test Tank Geometry and Aerator Placement

Appropriate configurations for tank were prepared for ease of distribution of deoxygenation chemicals, sampling requirements and mixing patterns. The placement of aerator was established in the middle of tank. The depth of propeller was considered to obtain to optimum depth for each propeller (see Appendix A) (ASCE, 1993).



Figure 3.5 Surface Aerator Placement

Surface Aerator Setup

3.2.2 **Power Inputs**

The power inputs, which were measured, had to be considered RPMs because the same power input in various types of devices could give the different RPM. The range of power input and Reynolds number were shown in Appendix C. In this research, four values of power input, 0.5 hp/1000 ft³, 1.0 hp/1000 ft³, 1.5 hp/1000 ft³, and 2.0 hp/1000 ft³ were selected to perform the tests. The RPMs of motor were set to compiling this calculation. Table 4.1 and 4.2 below showed the values of power inputs that were used in the experiments.

Power Input			Motor Revolution			Reynolds No.	Mean Velocity Gradient
hp/1000ft ³	Watts/m ³	Watt/Tank vol.	RPSs	RPMs (Tank Vol.)	RPMs per Unit Vol	(Re)	G (1/s) (tank vol.)
0.5	13.2	5.3	10.6	632	859	135687	121.6
1.0	26.3	10.5	13.3	798	1082	170954	172.0
1.5	39.5	15.8	15.2	914	1239	195694	210.7
2.0	52.7	21.1	16.7	1005	1363	215389	243.3

 Table 3.1
 Selected Values of Power Inputs for 10.7 cm Diameter Propeller

 Table 3.2
 Selected Values of Power Inputs for 15 cm Diameter Propeller

Power Input			Motor Revolution			Reynolds No.	Mean Velocity Gradient
hp/1000ft ³	Watts/m ³	Watt/Tank vol.	RPSs	RPMs (Tank Vol.)	RPMs per Unit Vol	(Re)	G (1/s) (tank vol.)
0.5	13.2	5.3	6.0	362	489	151859	121.6
1.0	26.3	10.5	7.6	455	616	191330	172.0
1.5	39.5	15.8	8.7	519	705	219018	210.7
2.0	52.7	21.1	9.5	571	776	241061	243.3

3.2.3 Water Quality

3.2.3.1 General and Total Dissolved Solids

The water supplied for the initial test was tap water. Subsequent tests were conducted in the same water, and, because of the addition of deoxygenation chemicals, the total dissolved solids concentration (TDS) would increase. Repetitive testing may be conducted in the same water, providing that the TDS did not exceed 2,000 mg/L (ASCE, 1993).

3.2.3.2 Temperature

Water temperature measurement should be between 10°C and 30°C. Low temperatures slow the deoxygenation reaction, and this may introduce some error. It is recommended that a standard θ , temperature correction factor, of 1.024 was employed to adjust for temperature. Water temperature shall not change by more than 2°C during a single unsteady-state test (ASCE, 1993).

3.2.3.3 Water Quality Analyses

Initial analyses: Prior to the beginning of the testing program, a representative sample of the water from the test tank shall be analyzed for the TDS and the temperature. Addition analyses during experiment for parameters of concerns, such as pH, temperature and total dissolved solid will be performed.

Water quality analyses during testing: When repetitive tests are conducted on the same water, the water shall be analyzed for various constituents and properties at frequency. The cobalt concentration was added to achieve a soluble cobalt concentration between 0.1 and 0.5 mg/L so as to assure complete oxidation of the sulfite (ASCE, 1993).

3.2.4 System Stability

The aeration system should be operated to achieve state hydraulic conditions prior to start the oxygen transfer evaluation. For this system, the volume of water under aeration should not vary by more than $\pm 2\%$ during any one test (ASCE, 1993).

3.2.5 Deoxygenation Chemicals

Analytical reagent grade sodium sulfite (Na₂SO₃) was used for deoxygenation. The concentration of cobalt in the sulfite should be sufficiently low so that the concentration in the test tank following repetitive testing does not exceed limits set. Sodium sulfite was added in solution. This was accomplished by dissolving the sulfite in a separate mixing tank prior to its addition to the test tank. Saturated solutions contain 2.23 lb/gal (26.72 kg/m³) at 20°C and 3.00 lb/gal (35.94 kg/m³) at 30°C.

The sulfite deoxygenation reaction is catalyzed by cobalt. The cobalt source utilized was cobalt chloride hydrate, $CoCl_2 \cdot 6H_2O$, or cobalt sulfate, $CoSO_4$, reagent of technical grade. In this experiment, cobalt chloride hydrate, $CoCl_2 \cdot 6H_2O$, was used. The cobalt was dissolved prior to its addition to the test tank. Solubility of $CoCl_2 \cdot 6H_2O$ is approximately 50 g/100 ml of water at 20°C and increases with temperature (ASCE, 1993).

3.2.6 Addition of Deoxygenation Chemicals 3.2.6.1 Cobalt Addition

The cobalt catalyst should normally be added once for each test water. A solution of cobalt salt shall be added to the test tank to achieve a soluble cobalt concentration between 0.10 mg/L and 0.50 mg/L in the test water.

The cobalt solution was added prior to the beginning of oxygen transfer testing with the aeration system operating. The solution should be uniformly distributed into the test tank. The cobalt solution should be dispersed throughout the tank by operating the aeration system for approximately 30 minutes (ASCE, 1993).

3.2.6.2 Sulfite Addition

The theoretical sodium sulfite (Na_2SO_3) requirement for deoxygenation is 7.88 mg/L per 1.0 mg/L DO concentration. Sulfite additions were made in excess of stochiometric amounts. The amount excess is dependent on the oxygen transfer rate of the aeration system and the size of the test tank. The amount of excess varies from 20 to 250% and increases for high transfer rate systems.

Sodium sulfite was dissolved in mixing tanks outside the test tank and distributed uniformly into the test tank (ASCE, 1993).

3.2.7 Determination of Dissolved Oxygen at Various Points in the Tank During the Unsteady-State Test

Dissolved oxygen concentrations were determined at various points in the tank and at various times during the unsteady-state test.

This determination was carried out by in situ measurement of dissolved oxygen in the tank by membrane probes.

3.2.7.1 Location of Dissolved Oxygen Determination Points

The number and the location of dissolved oxygen determination points were dictated by the size of the test tank, aerator placement, and mixing patterns in the tank.

Two determination points were used as shown in Figure 3.5. One was at a shallow depth, in the middle of the upper half of the tank. Another one was at the middle of the lower half of the tank. Figure 3.5 showed the location of two probes.

The determination points should be located in an attempt to have each sense an equal portion of the tank volume and should be distributed vertically and horizontally to best represent the tank contents. DO probes were secured by appropriate means to minimize movement due to water currents generated by operation of the aeration system during the tests (ASCE, 1993).

3.2.7.2 Times of Dissolved Oxygen Determinations

DO Probes with Continuous Recorders: Dissolved oxygen concentrations were determined at various times during the unsteady-state test using DO probes with continuous recorders. A continuous record of DO values versus time was available at each determination point. A minimum of 21 discrete data values was selected from the continuous record for analysis. The values selected were meet the timing criteria outlined which was described below. *Timing Criteria*: The purpose of these criteria is to ensure that the data points are representative of the reaeration curve, and that an adequate number of points are obtained in sensitive regions of the curve. The rising part of the curve, up to about 85% of saturation, is sensitive for estimation of K_La, and the stationary part of the curve, above 90% saturation, is sensitive for the estimation of C_{∞}^{*}. Consequently, the timing of DO values used in the data analysis should meet the following criteria for range and balance after preparation and possible low-end truncation as described in Appendix **C**. The lowest DO value should be not greater than 20% of C^{*}_{∞}. The highest DO values should be not less than 98% of C^{*}_{∞}.

Balance: The balance of data and timing should meet these criteria below.

- The DO values are measured so that about two-thirds (58%-75%) of the values are evenly distributed over the rising portion of the curve between 20% and 86% of saturation, and about one-third (25% - 42%) of the values are evenly distributed over the stationary portion between 86% and 98% of saturation.
- When 21 or more DO values are used, the values can be evenly spaced at approximately equal time internals over the entire range between the first and last DO value (ASCE, 1993).

3.2.7.3 Test Duration and Dissolved Oxygen Saturation

Dissolved oxygen data should be obtained over as wide range as possible. Even though low-end values may be truncated to avoid lingering effects of the deoxygenation technique prior to analysis, it is important to collect low dissolved oxygen data so that these effects can be detected. The low dissolved oxygen data shall be established that the sulfite addition has depressed the DO concentration to less than 0.5 mg/L at all determination points. All tests were continued for a time so that the last measured DO value at each determination point is equal to or greater than 98% of C_{α}^{*} for that determination point.

3.2.8 Dissolved Oxygen Measurements

Measurement by In Situ DO Probes: The in situ DO probes were fast-response probes with a probe time constant of less than $0.02/K_La$. In these tests, the velocity of flow past the probe was established and maintained to provide an accurate response from the probe because the water sample was agitated by the aerator enough and not necessary to use an agitator. Each probe was equipped by the digital recorder in the probe. It permitted reading of the DO concentration at appropriate intervals with a precision of ± 0.05 mg/L. Dissolved oxygen probes were calibrated in each test. The considerable care and attention to provide continuous reliable data were required (ASCE, 1993).

3.2.9 Data Preparation

3.2.9.1 Preparation of Data for Analysis

The purposes of data preparation are:

- To obtain discrete numerical values from continuous probe reading, to plot and examine the data to determine if lowend truncation is necessary,
- To truncate low dissolved oxygen values, and
- To assure that the data values to be analyzed meet the timing criteria (see in Appendix D) (ASCE, 1993).

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3.2.9.2 Parameter Estimation

The purpose of the parameter estimation procedure is to determine the best estimates of the three model parameters, K_La , C^*_{∞} and C_0 so that the model given by equation 2.1 and 2.2 best describes the variation of DO with time at each determination point location in the tank. Parameter estimates were performed for the data at each determination point by the nonlinear regression method.

This method based on nonlinear regression of the model (Equation 2.2) through the DO-versus-time data as prepare for analysis. The best estimates of the parameters, K_{La} , C_{∞}^{*} and C_{0} were selected as the values that drive the model equation through the prepare DO concentration-versus-time data points with a minimum residue sum of squares. Here, a residual refers to the difference in concentration between a measured DO value at a given time and the DO value predicted by the model at the same time.

A computer was required to apply for this method which gave the least square estimates, standard deviations of K_La , $C^* \propto$ and C_0 , along with an output of the data, fitted values, and residuals. Because of using this method, the prepared data for a particular determination point were entered along with initial approximations of the three parameters. An initial estimate of the three parameters was obtained by examining a plot of DO versus time. The computer then adjusted these initial approximations to minimize the residual sum of squares and reports the final parameter estimates.

For adequate convergence, the initial parameter estimates for C_0 were within 2 mg/L of the final estimate. Frequently, data was taken with time zero defined early in the test sequence, and this could correctly result in large negative predicted values of C_0 . In these cases, the initial parameter estimate for C_0 might be facilitated by redefining time zero so that C_0 was approximately zero. This redefinition of time zero did not affect the model predictions for K_La , and $C^* \propto$.

The values of DO predicted by the model based on the final parameter estimates should be plotted along with the measured values and examined. A visual examination of such a plot indicated any gross mistakes or false convergence occasionally occurs and could be easily remedied, improving the initial parameter estimates (ASCE, 1993).

3.3 Data Analysis

Collected data from the digital recorder in the probe were transfer in to DO_PAR program. The program procedures were performed as describe previously. K_La , values and C^*_{α} were obtained. The relationship between K_La , power input, turbulent condition and surfactant concentrations were analyzed and discussed in the next chapter.