***Chapter Three***

***Experimental Design***

**3.1 Introduction**

The materials and experimental procedures that have been used in this study are presented in details in this chapter. To achieve the objectives of the current research, two main sectors were investigated. The aeriation of the sewer system and the seeding process coupled with aeriation sectors had been studied. Information about the experimental setup and the specifications of the used pipes system with other constructions used to simulate sewer system is presented in the current chapter. The experiments conditions and the used aeriation parameters, the used methods in measuring BOD5, COD, SS, VSS was also presented. The method used for calculating the degradation kinetics and the overall mass transfer coefficient for aeriation process was further described in the current chapter. The overall flow diagram for the research methodology is shown in Figure 3.1.

**3.2 Experimental Procedure**

**3.2.1 Pipes System Design**

The actual sewer system that connects [Al-Adhamiya](https://twitter.com/adhamiyahnews?lang=ar) district and Al-Wazireya neighborhood was experimentally simulated in the current study. PVC pipes of nominal size of 75.0 mm (3 inches) were used in constructing the system. The pipe system was designed as a square shape with four equal sides. The side length was designed as 4 m and a settling tank was fitted at each corner of the square design to simulate manholes in the real sewer system. The tanks have a cylindrical shape with a total

volume of 250 L supplied with transparent level measuring pipe and fitted to the system via quarter gate valve. An extra tank was connected to one of the level tanks and rested outside the square system to be used for delivering the activated sludge to the system throw pipe connection with a valve. More specifications about the pipes used in the system can be seen in Table 3.1.

|  |
| --- |
| **Table 3.1** Specifications of pipes system |
| Outer diameter  | 81.5 mm (3.21”) |
| Inner diameter  | 75.0 mm (3.0”) |
| Length (m) | 50 |
| Area (cm2) | 45.6 |
| Manning Coefficient | 0.01 |
| Number of 90º bends | 4 |
| Number of 180º bends | 8 |
| Gate valves half opening | 4 |

Each side of the square shape was designed to include three pipe branches with length of 4 m connected with 180O bend pipe. So the distance of the pipes inside each side was set to be 12 m. Each pipe branch was fitted with a slop of 1.1% in order to simulate the inclination in real sewer system that assures the gravitational flow. Due to this inclination, the total head between the entrance of water in the feeding tank and the deliverance point was set to be 42.0 cm, (Appendix D) at the deliverance point, with halve opening of gate valves to maintain partial flow of 0.5 d/D. A pump was used to raise the wastewater above the head back to the entrance tank. Beside the pipes, 90º elbow (in manhole tanks) and 180º bend pipes were used to join the pipes together

and construct the square shape system. A schematic diagram for the system can be seen in Figure 3.1, while photographic pictures for the pipe system, tanks, and aeration method were presented an Appendix A.

 

I.L.

+0.14

I.L.

I.L.

I.L.

+0.28

±0.00

+0.42

**Submersible Pump**



**Figure 3.1.** Schematic diagram for the designed system

**3.2.2 Aeration Design**

 In order to supply air to the system, air was pumped via air compressor shown in Appendix A. Air was fed into the system in two ways, thin metal pipes used to deliver air to air nozzles distributed in terms of three nozzles attached to the outlet pipe from each tank and the other method of aeration had been adopted expressed in supplying air via

nozzles to the tanks itself as tanks were always partially filled with wastewater. These two procedures were adopted together to ensure the deliverance of air to the microorganisms presented in the system.

**3.2.3 Activated Sludge Seeding Process**

 The activated sludge used for seeding the raw wastewater in the pilot scale system was the returned sludge collected from Al-Rustamiyah sewage treatment plant.

The returned activated sludge was allowed to settle for one hour just after arrival to the pilot scale system site and the supernatant decanted in order to increase the suspended solid concentration, the biomass concentration was 4500 mg/l. The seeded experimentation was started with 30/70, 40/60, 50/50 and 40/60 (v/v) activated sludge to wastewater mixed in feeding tank.

**3.3 Experimental Design Procedure**

 The process of aeration experiments was conducted as the feeding tank filled with raw wastewater (screened) delivered from Al-Adhamiyah pumping station. The water was first examined in terms of BOD5, COD, SCOD, SS and VSS values. Quarter gate valve was opened and the wastewater let to flow due to gravity reaching to the following tank and accumulated. When the water level reached to an appropriate value, the gate valve was set to be opened and sewage water flowed to the next tank. This process continued until a wastewater reached to the deliverance point with the consideration of adding more sewage water to the feeding tank in necessity to maintain water levels in all tanks with the range of partially filled. At this point, submersible

pump was started to raise water to next tank and all valves were opened and circulation of water in the system started.

 As the circulation process started, air compressor switched on and air had been delivered to both pipes and tanks. The process continued for 8 successive working hours and samples were withdrawn at intervals of 1, 2, 3, 4, 6, and 8 hours measured from the aeriation starting and stored in plastic containers in dark place in order to be analyzed.

 The feed stock was changed to be a mixture of activated sludge with the real wastewater in different mixing ratio and the same procedure was followed.

**3.4 Flow Parameters Calculations**

 According to the design parameters and slope chosen to incline the pipes to assure gravitational flow, the volumetric flow rate across the pipes can be calculated using Manning formula:

$Q=VA=\frac{1}{n}A\left(\frac{A}{P}\right)^{\frac{2}{3}}\sqrt{S}$ …….………3.1

where:

Q: volumetric flow rate (m3/s).

V: velocity (m/s).

A: flow area of the pipe (m2).

P: wetted perimeter which is the portion of the circumference that is in contact with water (m).

S: channel slope (m/m).

n: Manning factor.

 The terms used in the current formula and the calculated volumetric flow and velocity were presented in Table 3.2.

**Table 3.2** Terms used to calculate Manning formula

|  |  |  |
| --- | --- | --- |
|  | **Full Flow** | **Partial Flow** |
| **Diameter (m)** | 0.075 | 0.0375 |
| d/D | 1.0 | 0.5 |
| Ɵ | 0.0º | 180º |
| A (cm2) | 45.6 | 22.8 |
| P (cm) | 23.94 | 11.97 |
| R (cm) | 1.90 | 1.90 |
| S (m/m) | 0.011 | 0.011 |
| n | 0.01 | 0.0125 |
| V (m/s) | 0.75 | 0.60 |
| Q (L/s) | 3.41 | 1.36 |
| Volume (L) | 228 | 114 |
| Circulations per hour | 54 | 43.2 |

 According to the calculated velocity, the sewage water can travel about 2.70 km per working hour in the designed system and about 21.6 km for the whole exposure time of 8 hrs.

**3.5 Wastewater Analytical Methods**

 Influent and effluent samples were stored at 4 OC and all the analysis were carried out within 24 hours according to (Standard Methods, 1989).

Adscription of the testes is given in Table 3.3.

**Table 3.3** Analytical methods used (Standard |Method, 1989)

|  |  |
| --- | --- |
| Parameters | Methods |
| BOD5 (ATU) | Dilution Method, DO by modified Winkler Method |
| DO | Electrode |
| Soluble COD | Closed reflux, titrimetric Method |
| SS | Total SS dried at 105 OC |
| Total COD | Open reflux, titrimetric Method |
| VSS | Fixed and Volatile Solid ignited at 550 OC  |

**3.5.1 Calculation of Removal Efficiencies**

 The COD and SCOD removal efficiencies calculated at the start of the experiment and after 8 hours operation time. The removal efficiency is given by:

R= (So – Sf / So) \*100 ………………..3.3

where

R= COD or SCOD removal efficiency (%)

So= Initial COD or SCOD concentration (mg/l)

Sf= Final COD or SCOD concentration (mg/l)

**3.6 Degradation kinetics**

Most reactions that take place in sewage treatment process are slow and the consideration of their kinetics is crucial. The most usual reaction orders found in sewage treatment are zero order and first order. Second order reactions may occur with some specific industrial wastewaters.

**3.6.1 First Order Reaction Kinetics**

 The system can be considered as a plug flow reactor with very long length. The rate constant k can be considered constant along the reactor for the biodegradable substrate with first order reaction kinetics. Yet the concentration decreases gradually as the waste water flow along the reactor. At the inlet point (feeding tank), the concentration is high, which cause the removal rate to be high (as rate is proportional to concentration for first order reaction). At the end of the reactor, the concentration is reduced so the reaction rate is low.

 The governing equation for first order reaction is as follows:

$$\frac{dC}{dt}=-kC ………3.4$$

Where:

C: is the concentration of reactant.

k: reaction rate constant.

 The equation can be put in terms of SCOD concentration and integrated for the initial condition at t=0, SCOD=SCODO to yield:

$$SCOD=SCOD^{o}e^{-kt} ………..3.5$$

 This equation can be used to fit the experimental data to find the value of k. yet the equation is nonlinear, so nonlinear curve fitting procedure will be used.

**3.6.2 Fitting the Experimental Data**

 Nonlinear least square method had been invested in fitting the experimental data to the proposed model of reaction kinetics to find k value. Nonlinear least squares method is the form of [least squares](https://en.wikipedia.org/wiki/Least_squares) analysis used to fit a set of observations with a model that is

nonlinear. It is used in some forms of [nonlinear regression](https://en.wikipedia.org/wiki/Nonlinear_regression) and the basis of the method is to approximate the model by a linear one and to refine the parameters by successive iterations. There are many similarities to [linear least squares](https://en.wikipedia.org/wiki/Linear_least_squares_%28mathematics%29) with also some [significant differences](https://en.wikipedia.org/wiki/Least_squares#Differences_between_linear_and_nonlinear_least_squares). Nonlinear least squares problem is unconstrained minimization problem with the form:

$$minimize f\left(x\right)=\sum\_{i=1}^{m}r\_{i}^{2} ………3.6$$

For a nonlinear relation in the form of:

$$y=f\left(x\right) ……..3.7$$

The term r can be defined as:

$$r\_{i}=f\left(x\_{i}\right)-y\_{i} ………..3.8$$

 Computer program designed by MATLAB 8.3 (R2014a) was used to calculate k values according to the proposed kinetics model and the programs used were listed in Appendix B.

 Predicted SCOD values can be calculated via applying the estimated k values to the model equation (3.5). The model validity can be examined by calculating coefficient of determination R2 using the real and predicted SCOD values as follows:

$$SSM= \sum\_{}^{}\left(\overbar{y}-\hat{y}\_{i}\right)^{2} ……….3.9$$

$$SSE= \sum\_{}^{}\left(y\_{i}-\hat{y}\_{i}\right)^{2} ………..3.10$$

$$SST=SSM+SSE ………..3.11$$

$$R^{2}=\frac{SSM}{SST} ……….3.12$$

where:

SSM: is the sum of squares mean.

SSE: is the sum of squares error.

SST: is the sum of squares total.

$\overbar{y}$ : is the mean value of the experimental data.

$\hat{y}\_{i}$ : is the predicted data.

R2: is coefficient of determination.

**3.7 Mass Transfer Coefficient Estimation**

 In order to investigate the amount of oxygen delivered to the living biomass presented in sewage water during the treatment for all experiments conducted in the current research, mass transfer coefficient KLa had been estimated.

**3.7.1 Dissolved Oxygen Measurement**

 The amounts of oxygen dissolved in sewage water samples were measured using probe type DO measurement instrument for water samples. The air was pumped into each samples (at 10, 20 and 30 OC) for 30 min at rate of 20 cm3/s to insure reaching to saturation condition. DO concentration and pH values were measured each minute and DO saturation concentration was identified.

**3.7.2 Mass Transfer Coefficient Calculation**

Estimation of KLa for sewage water samples was calculated via model equation used to calculate the predicted dissolved oxygen for each time point in a single experimental run, (Molder, *et. al.,* 2009), Equation (3.13) represented the adopted model for predicting DO concentration:

$$C\_{pr}=C\_{sat}-\left(C\_{sat}-C\_{o}\right)e^{-K\_{La} t} ……….3.13$$

where:

Cpr : nonlinear regression predicted DO concentration (mg/L).

Csat : saturated DO concentration (mg/L).

CO : initial DO concentration (mg/L).

KLa : mass transfer coefficient min-1).

t : elapsed time (min).

The previous equation had been used to calculate the square error combing with the real values of DO as:

$$S=(C-C\_{pr})^{2} ……….3.14$$

where C is the measured DO concentration in mg/L each time interval.

Nonlinear regression was used to fit a model to the experimental data in order to obtain the best fit values of the parameters in that model. Nonlinear least square method with suitable initial guess was invested in order to solve equation (3.14) to estimate KLa value.

The estimated KLa value had been used in equation (3.13) to calculate the predicted DO values. Coefficient of determination R2 had been calculated in order to validate the model using equations (3.12).