



PERFORMANCE STUDY ON ZERO LIQUID DISCHARGE TREATMENT PLANTS

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Abstract:

The country's economic development depends on industrialization. Water resources are becoming scarce because of increased industrial growth. Water pollution, however, is also a significant problem. People have long been trying to find cost-effective and dependable wastewater treatment methods where recycling or reusing treated water has become a necessity. ZLD is a system made up of discrete operations or processes, or their combination, that prevents the discharge of liquid effluent from a process plant, industry, or other location. The reality that the effluent is effectively treated, recycled and reused indicates that there is no liquid discharge using ZLD. In general, zero liquid discharge is achieved by concentrating the wastewater using a variety of technologies, including membrane-based and MEE-based systems. In the present study, treatment of industrial wastewater from two different fertilizer industries was investigated for 8-month period using two different ZLD treatment plants. Quality parameters including pH, TSS, TDS, COD, BOD, O&G, ammoniacal nitrogen, chloride, sulphate, fluoride, and nitrate were used to evaluate the two ZLD processes.

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1. INTRODUCTION:

Industries generate wastewater, often known as an effluent which is by-product of various manufacturing processes (Singh Jaidev, 2012). The effluent contains a number of pollutants that an ETP can remove. The "clean" water can then be released into the environment in a secure manner (Govindasamy P *et al.*, 2006).

ZLD is a system made up of discrete operations or processes, or their combination, that prevents the discharge of liquid effluent from a process plant, industry, or other location. The reality that the effluent is effectively treated, recycled and reused indicates that there is no liquid discharge using ZLD (Ranade Vivek V. and Vinay M. Bhandari, 2014). In general, zero liquid discharge is achieved by concentrating the wastewater using a variety of technologies, including membrane-based and MEE-based systems. ZLD consists of:

- Reusing treated water from the industry
- Eliminating wastewater stream from the industry
- Creating a standard for minimal liquid pollutants (Ahirrao Shrikant, 2014).

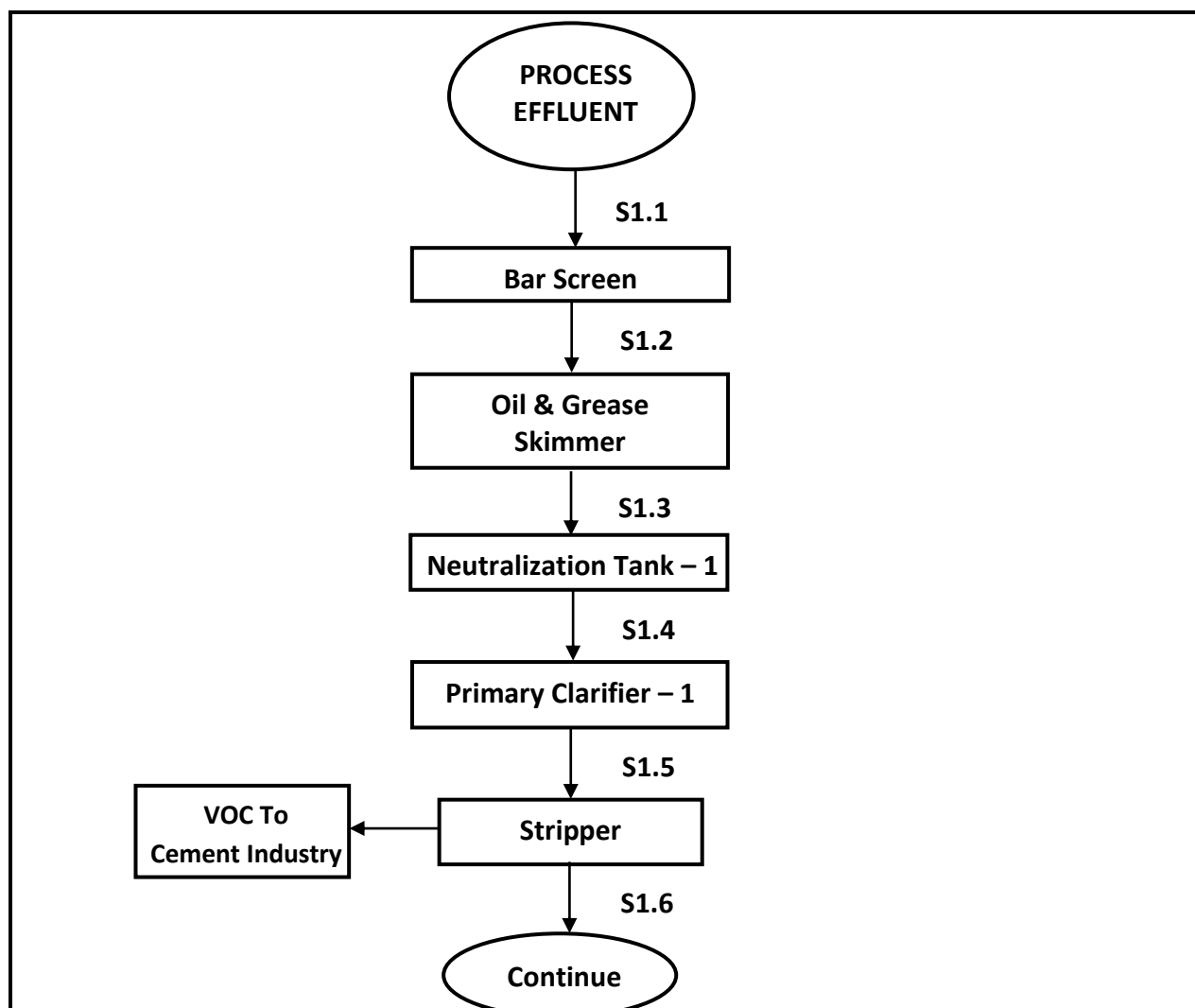
Application of treated effluent

The potential application for the reuse of recycled water:

- In cooling towers, especially big scale industries
- Suitable for use in gardening to water plants and lawns
- In toilet flush
- As cleaning medium in a water scrubber
- For preparing lime slurry for ETP
- Different industrial washing operations
- Water used as boiler feed

2. DESCRIPTION OF ZLD TREATMENT PLANTS

In this study, two ZLD treatment plants were selected and named as P-1 and P-2. The main sources of effluent generation from the plants were segregated into two types viz. process effluents (organic and inorganic in nature) and non-process effluents (from boiler blowdowns and cooling towers). The effluents were treated in two ZLD treatment plants as described in Figures 2.1 & 2.2.



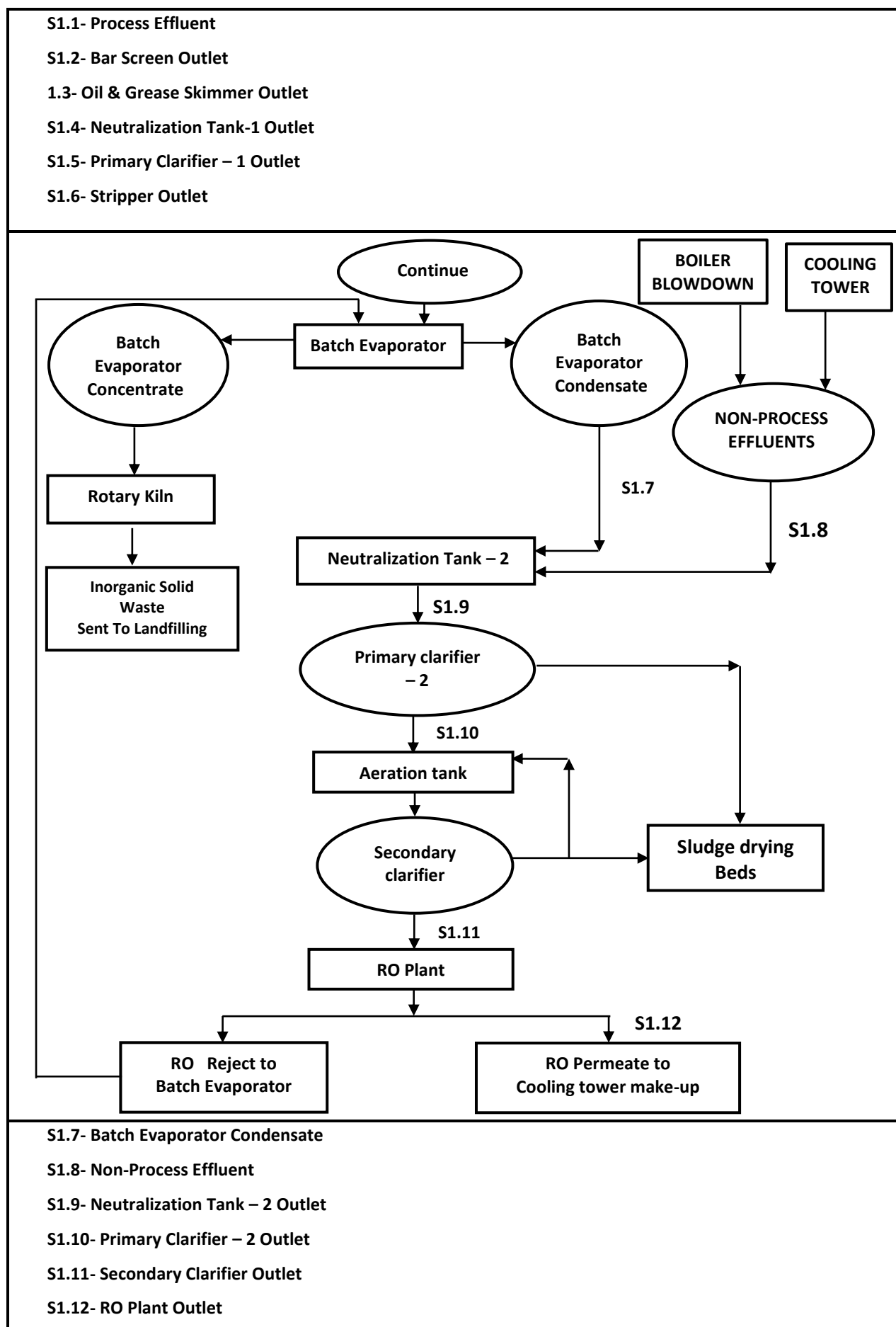
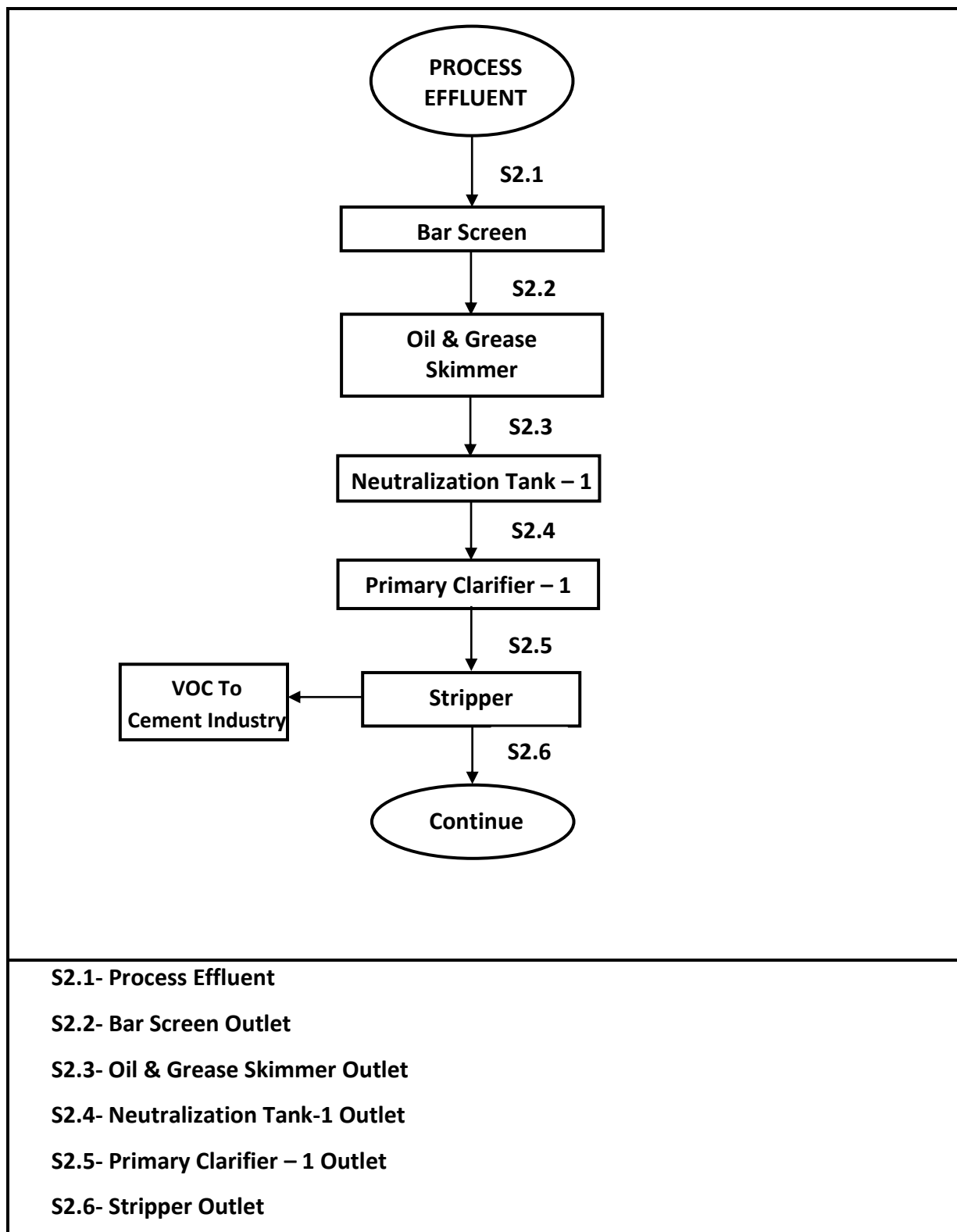


Figure 2.1: Schematic flow diagram of ZLD treatment plant P-1



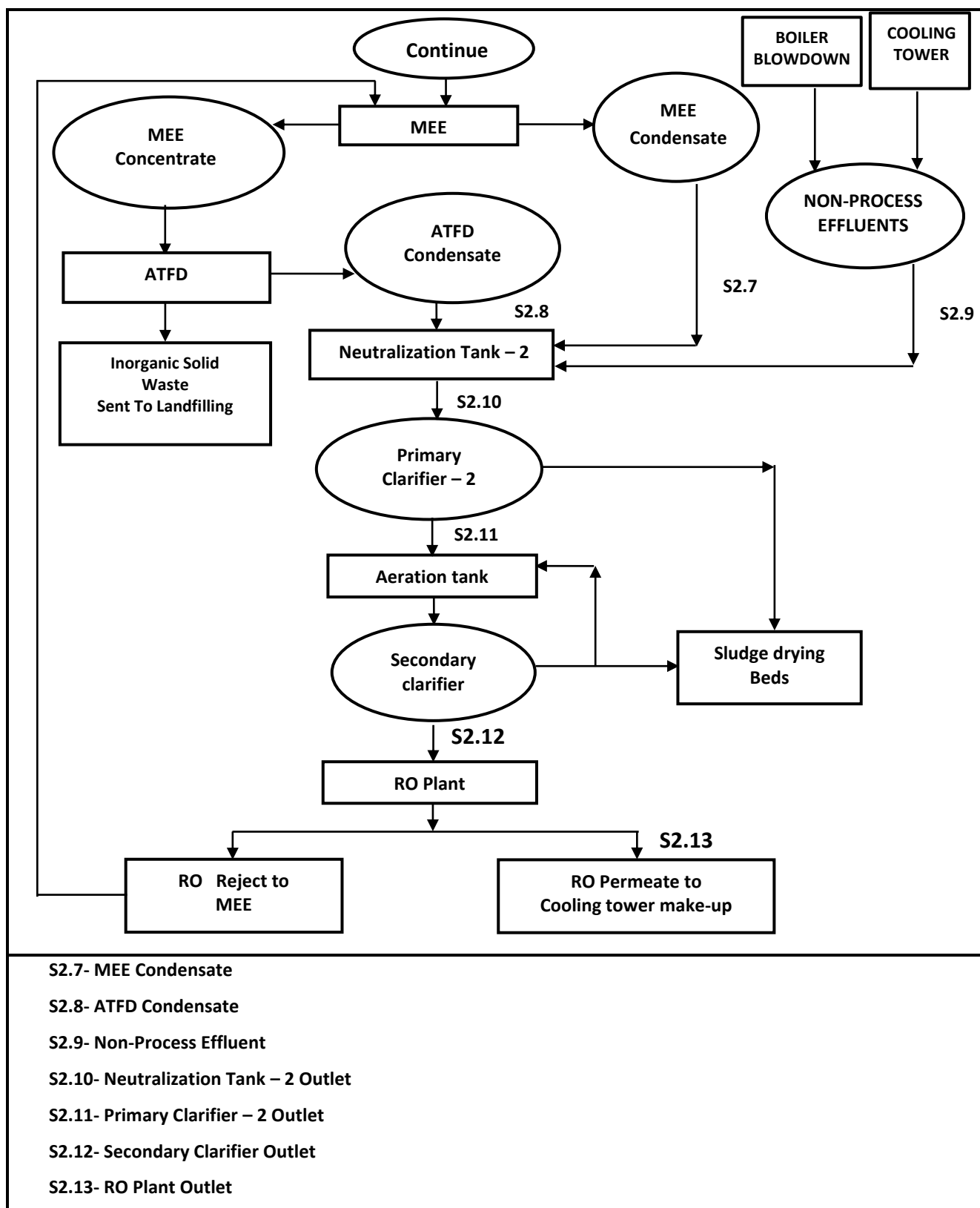


Figure 2.2: Schematic flow diagram of ZLD treatment plant P-2

3. MATERIALS AND METHODS

3.1 Materials

3.1.1 Chemicals used:

Potassium dichromate, sulphuric acid, ferrous ammonium sulphate hexahydrate, O-phenanthroline monohydrate, ferrous sulphate heptahydrate, sodium thiosulphate, sodium

hydroxide, starch, magnesium sulphate, sodium iodide, sodium azide, hydrochloric acid, zinc sulphate heptahydrate, EDTA, mercuric iodide, potassium iodide, potassium chromate, silver nitrate, glycerol, isopropyl alcohol, sodium chloride, acetic acid, barium chloride crystals, 1, 2-cyclohexylene diamine tetra acetic acid, aluminum

sulfate octa decahydrate, boric acid, sulfamic acid, silver sulphate and distilled water used in this study were of analytical grade.

3.1.2 Equipment's used:

pH meter, drying oven, desiccator, analytical balance, COD digester, water bath, BOD incubator, spectrophotometer, fluoride ion concentration meter and nitrate ion-selective electrode were the equipment's used for the quality study and performance evaluation of the two ZLD processes.

3.2 Methods

3.2.1 Sample collection and preservation

- The effluent samples of the two ZLD processes namely P-1 and P-2 were collected in the glass bottles throughout the study. The collected samples were denoted as shown in the Figures 2.1 & 2.2.
- Samples containing settable material should be well mixed, preferable homogenized, to permit removal of representative aliquots.
- Distilled water was used for all the dilutions.

3.2.2 Evaluation of quality

3.2.2.1 pH

pH was determined by measuring the EMF of a cell comprising an indicator electrode (an electrode response to hydrogen ions such as glass electrode) immersed in the testing sample. Before use, remove electrode from storage solution wash the glass electrode with distilled water and clean slowly with a soft tissue. Take 100 mL of the sample in a beaker and mix thoroughly before the measurement. Dip the electrode into the testing sample (the electrode immersion depth approximately 4 cm) and wait until the value on the display stabilizes and record the pH value. After the measurement rinse carefully the electrode with distilled water and dip it back into the storage solution.

3.2.2.2 TDS

50 mL of well-mixed sample was filtered through glass fiber filter. Then 10 mL of distilled water was allowed to wash for complete drainage between washing and suction was continued for about 3 min. after filtration is completed. The empty crucible was dried at $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for 1 h, cooled in a desiccator and weighed (W_1). Then, filtrate was transferred to an empty weighed crucible and evaporated on hot plate. Later, crucible was kept in a hot air oven at $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for one hour. After one hour, the crucible was cooled in a desiccator and weighed. The process of drying, cooling and weighing was repeated until a constant weight (W_2) was obtained.

3.2.2.3 TSS

A filter paper was taken and dried at $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for an hour to remove moisture adhering to its surface. It was then cooled in a desiccator and its weight was taken accurately on a precision balance (W_1). The weighed filter paper was placed over the funnel and wetted it with water. 50 mL of sample was filtered through it. The filter paper was then dried at $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$ in an oven. It was then cooled in a desiccator and weighed. The process of drying, cooling and weighing was repeated until a constant weight (W_2) was obtained.

3.2.2.4 COD

Culture tubes & caps were washed with 20% sulphuric acid before using to prevent contamination. 2.5 mL of sample was taken in a culture tube, and 1.5 mL of potassium dichromate solution was added. 3.5 mL of sulfuric acid reagent was carefully run down to the above solution, an acid layer was formed under the sample-digestion solution. Caps of the tubes were kept tightly and inverted each several times to mix completely. After mixing, the culture tubes were placed in COD digester which was preheated to 150°C and reflux for 2 h behind a protective shield. Later, the culture tubes were cooled to the room temperature and placed them in a test tube rack. After cooling 1 to 2 drops of Ferroin indicator was added and stirred rapidly on magnetic stirrer while titrating with standardized 0.25 N ferrous ammonium sulphate. The end point is a sharp color change from blue-green to reddish brown, although the blue green may reappear within minutes. In the same manner a blank containing the reagents and a volume of distilled water equal to that of the sample was refluxed, titrated and the titrant value was noted to calculate the COD of the sample.

3.2.2.5 BOD

Four 300 mL glass stoppered BOD bottles (two for the sample and two for the blank) were taken. 10 mL of the sample was taken in each of the two BOD bottles and the remaining quantity of the bottles were filled with distilled water. The other two BOD bottles filled with distilled water alone were kept as blank. After adding the glass stopper was placed immediately over the BOD bottles and the numbering was given on the bottles for identification. A blank and sample solution bottles were preserved in a BOD incubator at 20°C for five days. The other two bottles (one blank and one sample) were analyzed immediately. Care should be taken to avoid bubbling and trapping of air bubbles. 2 mL of manganese sulphate and 2 mL of alkali-iodide-azide reagent were added to the BOD bottle by inserting the calibrated pipette just below

the surface of the liquid to prevent the sample from receiving oxygen. Now, it was allowed to settle for sufficient time in order to react completely with oxygen. As the floc was settled at the bottom, it was shaken thoroughly by turning it upside down. 2 mL of concentrated sulfuric acid was added with a pipette just above the surface of the sample. The stopper was carefully placed and inverted several times to dissolve the floc. 203 mL of the solution was taken from the bottle into a flask and titrated immediately with standard sodium thiosulphate solution until the yellow color of liberated iodine was almost faded out (pale yellow color). 1 mL of starch solution was added and the titration was continued till the blue color disappeared to colorless. The volume of sodium thiosulphate solution added was noted. After five days, the BOD bottles were taken out from the incubator and analyzed for DO in the same manner as analyzed for the initial blank and sample. BOD was calculated.

3.2.2.6 O&G

An evaporating dish was weighed (initial weight) and kept aside. To 100 mL of sample 0.5 mL of conc. hydrochloric acid was added for acidifying it to get pH 2. This solution was taken in a separating funnel and 5 mL of the petroleum ether was added to it which was shaken well for at least 2 min. It was then allowed to rest for 2 min. Two separate layers were observed, one ether and another sample. Lower layer was collected in a beaker and ether layer was collected in an evaporating dish. The ether layer which was collected in evaporating dish was placed in an oven and allowed it to evaporate until all visible water has been removed. It was then cooled and weighed until a constant weight (final weight) was obtained.

3.2.2.7 Ammoniacal nitrogen

100 mL of sample was taken in a beaker. To it 1 mL zinc sulphate solution and 0.5 mL sodium hydroxide were added to obtain pH 10.5. The suspended solids in the sample were allowed to settle and the supernatant was filtered through filter paper. To 50 mL of filtrate, 1 drop of EDTA was added and mixed well. Then 3 mL of Nessler reagent was added and makeup to 100 mL. After 10 min, the sample was analyzed using UV-Vis spectrophotometer at 550 nm.

3.2.2.8 Chloride

25 mL of sample was taken in a conical flask and pH was adjusted between 7 to 10. 1.0 mL potassium chromate indicator was added to it. Later, it was

titrated with standard silver nitrate solution until an end point, pinkish yellow was observed. The titrant volume was noted and the chloride concentration was determined.

3.2.2.9 Sulphate

5 mL of conditioning reagent was added to 100 mL sample and mixed it by placing on a magnetic stirrer. To it 1 spatula of barium chloride crystals were added with continuous stirring for 1 min. The optical density was measured using UV-Vis spectrophotometer at 420 nm.

3.2.2.10 Fluoride

In a beaker, 100 mL of sample and 50 mL of TISAB were thoroughly mixed. Using fluoride ion concentration meter, the concentration of Fluoride was determined.

3.2.2.11 Nitrate

Equal volumes of sample and buffer were stirred well in a beaker. The nitrate concentration was determined using a nitrate ion-selective electrode.

Overall percentage removal efficiency of each parameter of the two ZLD treatment plants over an 8-month period was also calculated.

4. RESULTS & DISCUSSION

4.1 Evaluation of quality parameters

4.1.1 pH

Extreme pH levels in wastewater are frequently unacceptable because they make aquatic life harder to survive. Irrigation water should not have a pH that is either high or low. Most metals become soluble and accessible at low pH levels, possibly threatening the ecosystem, whereas at high pH levels, most metals become insoluble and build up in sediments and sludge.

pH of the individual samples for the two ZLD processes namely P-1 & P-2 were measured immediately after its collection. Before treatment, the effluents pH ranged from 9.3 to 9.8, while after treatment it ranged from 6.4 to 7.5 for all the two ZLD processes.

According to Figure 4.1, the overall removal efficiency of pH ranged from 19 to 26% for P-1 & P-2.

Sankar Raja P and Rajesh S, 2015 studied on ZLD plant of dyeing industry and observed that pH value of final recovery water was 6.5.

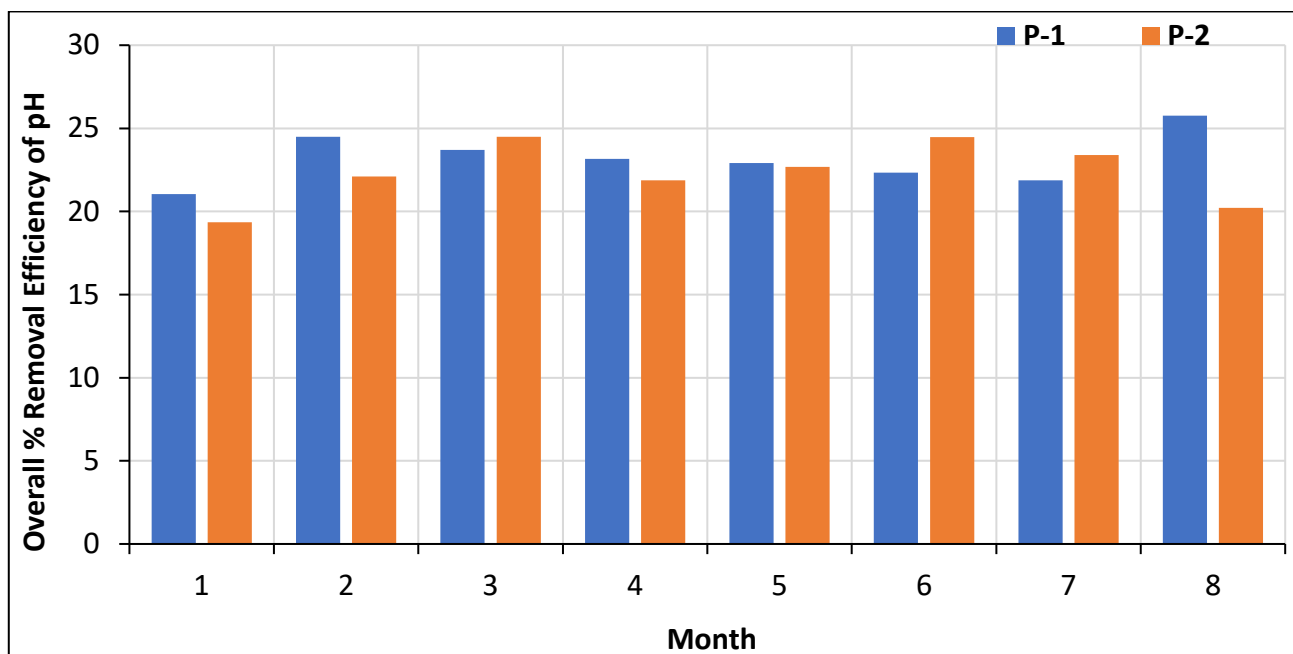


Figure 4.1: Comparison of overall percentage removal efficiency for the two ZLD processes on pH

4.1.2 TDS:

High TDS in treated effluent of wastewater treatment plants is hazardous to aquatic species and also produces sore throat & itchy feeling in humans. This is mostly caused by salinity which was primarily brought on by sodium salts. The TDS level of the effluent was also caused by the inorganic ions in the water supply and those added during water consumption.

The process effluent TDS values of the two ZLD processes ranged from 46,000 – 49,300 mg/L and

after treatment the reduction in TDS values were in between 50-200 mg/L.

From Figure 4.2, it was observed that overall percentage removal efficiency of TDS ranged from 99.58 - 99.75% for P-1& P-2.

Reddy Sareddy Ravi Sankar *et al.*, 2020 studied on treatment of effluents containing high TDS and concluded that TDS were reduced by more than 98% over the course of the whole study period, with a maximum average TDS reduction of 98.77%.

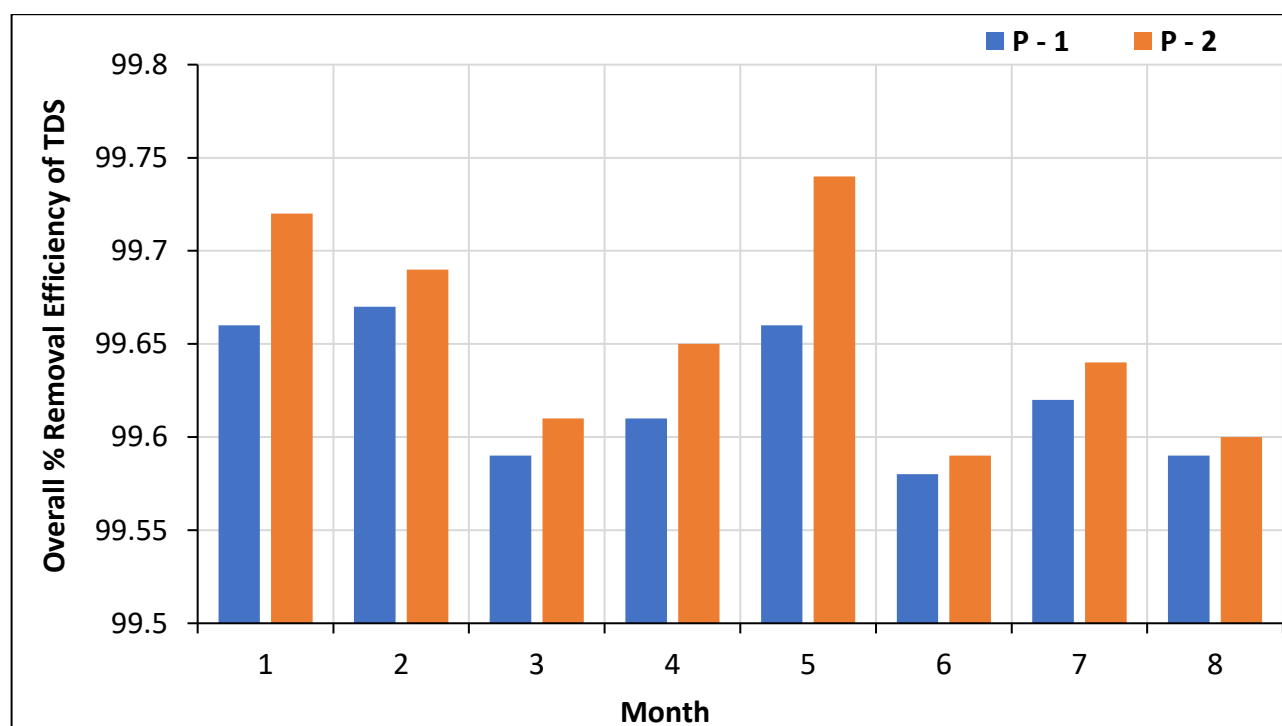


Figure 4.2: Comparison of overall percentage removal efficiency for the two ZLD processes of TDS

4.1.3 TSS:

TSS, which primarily consists of inorganic materials, is essential for the treatment of wastewater. The total dissolved oxygen content declines and the temperature of the wastewater was drastically raised because of the high TSS levels. This was due to the fact that suspended particles absorb more heat than typical water molecules. The TSS values for the two ZLD processes from the current study observations demonstrate that process effluent TSS values ranged from 320 to 350

mg/L and reduced TSS value of treated effluent was less than 10 mg/L.

According to Figure 4.3, the overall percentage removal efficiency of TSS ranged from 97.25 to 98.30% for P-1 & P-2.

Maheswara Uma *et al.*, 2015 concluded that the system intended for recycling effluents demonstrated high-quality permeate production and the overall decrease in TSS from the ZLD plant in API manufacturing unit was 100%.

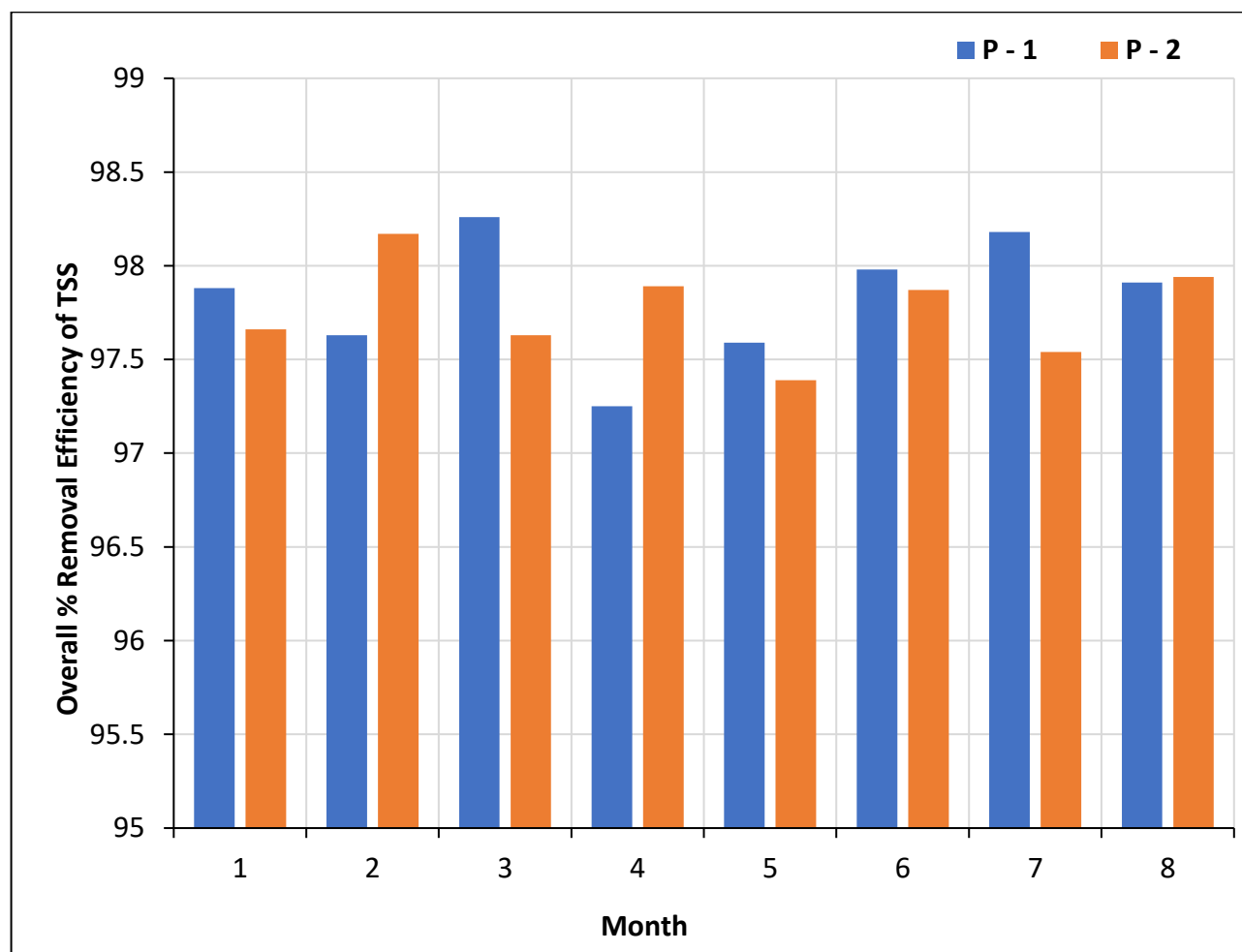


Figure 4.3: Comparison of overall percentage removal efficiency for the two ZLD processes of TSS

4.1.4 COD:

For determining the level of contamination in both household and commercial waste, a test known as COD uses a potent chemical oxidant to assess the quantity of oxygen needed for chemical oxidation of organic compounds. Majority of organic compounds can act as oxidizing agents in an acidic environment. To identify harmful conditions and the existence of biologically resistant chemicals, COD tests were helpful.

COD of the process effluent ranged from 72,000 to 75,000 mg/L and the treated effluent was reduced to less than 50 mg/L.

During the study, it has been observed that overall percentage removal efficiency of COD was ranged from 99.930 - 99.960 % for P-1 & P-2 as shown in Figure 4.4.

Nibe R L *et al.*, 2022 studied on wastewater treatment by ZLD. This study concluded that ZLD treatment procedure can be used as an advanced wastewater treatment to produce biodegradable products and lowered COD up to 100% from initial to final effluent.

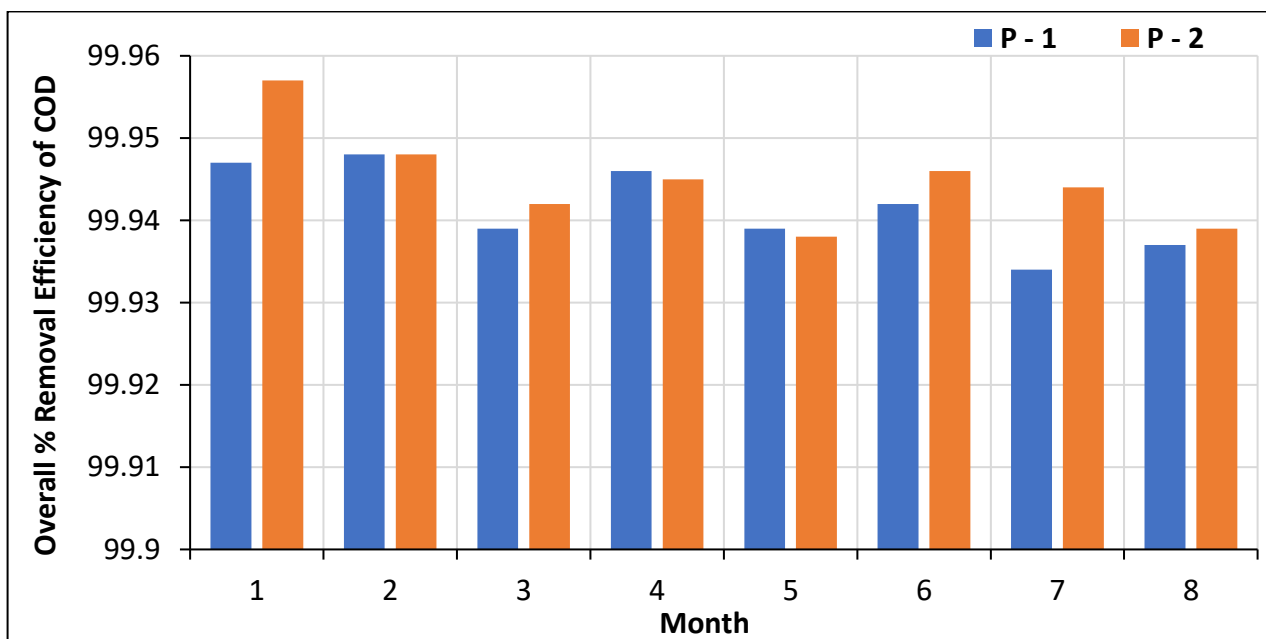


Figure 4.4: Comparison of overall percentage removal efficiency for the two ZLD processes of COD

4.1.5 BOD:

BOD is a crucial factor in refining wastewater because it drains the receiving waters of oxygen which harms the aquatic environment. Hence, it is important to control the level of BOD in every stage of the treatment process and to remove as much as possible in the final stages. BOD is the measurement of the amount of oxygen needed by microbial activity to oxidize and stabilize the decomposable organic matter. This test is most frequently used to gauge how much waste is being loaded into treatment facilities and to assess how effectively these facilities remove BOD.

It was observed during the current investigation the BOD value of the process effluent differed between

26,000 to 30,000 mg/L and the treated effluent was reduced to less than 30 mg/L.

During the study, it was observed that overall percentage removal efficiency of BOD was ranged from 99.890 - 99.920% for P-1 & P-2 as depicted in Figure 4.5.

Nibe R. L *et al.*, 2022 studied on Wastewater Treatment by ZLD. This study concludes that ZLD treatment procedure can be used as an advanced wastewater treatment aspect to produce by-products that were easier to biodegrade and that lower BOD up to 100% from initial to final effluent.

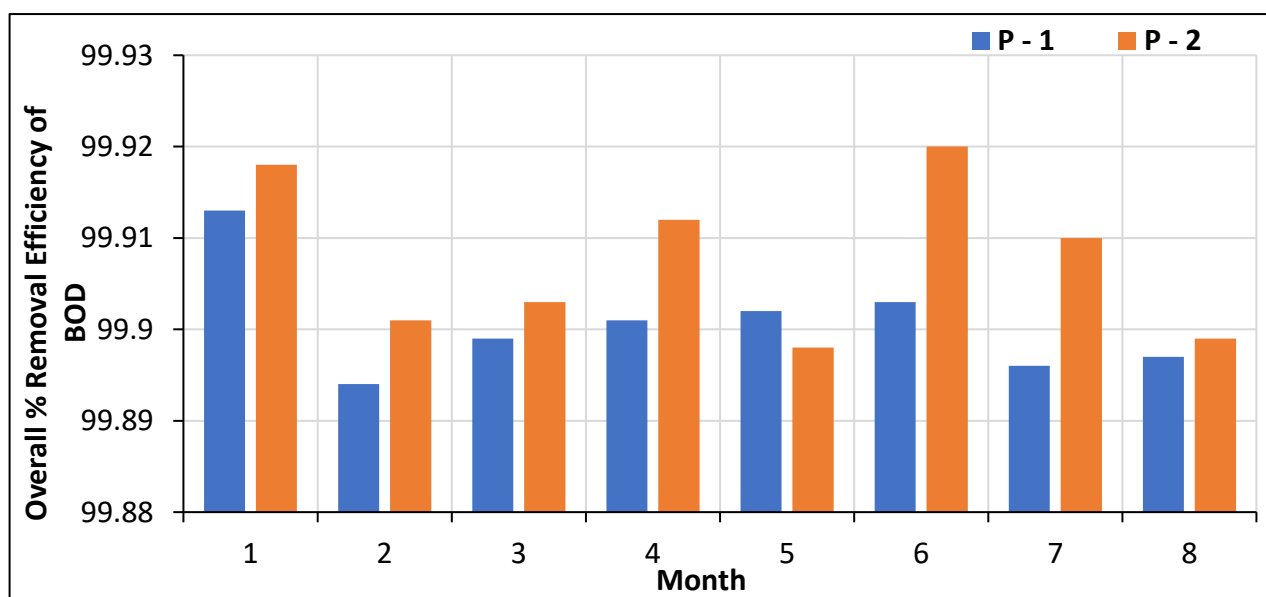


Figure 4.5: Comparison of overall percentage removal efficiency for the two ZLD processes of BOD

4.1.6 O&G:

O&G are natural organic compounds that generate BOD and COD. High levels of O&G in wastewater produce scum that accumulates on the surface and prevents sunlight. This interfere with the biological life in the surface water and leave undesirable films if it is not removed prior to the discharge of treated wastewater.

The concentration of O&G in process effluent ranged from 100 to 108 mg/L, whereas treated effluent was minimized to less than 5 mg/L.

As outlined in Figure 4.6, it was observed that the overall percentage removal efficiency of O&G during the analysis varied from 96.10 to 98.10% for P-1 & P-2.

Buljan J *et al.*, 2017 showed assessment of performance of ZLD operations of two plants. This study concluded that the two plants concentration of O&G raw effluent ranged from 10-25 mg/L while the treated effluent ranged from 0-0.1 mg/L.

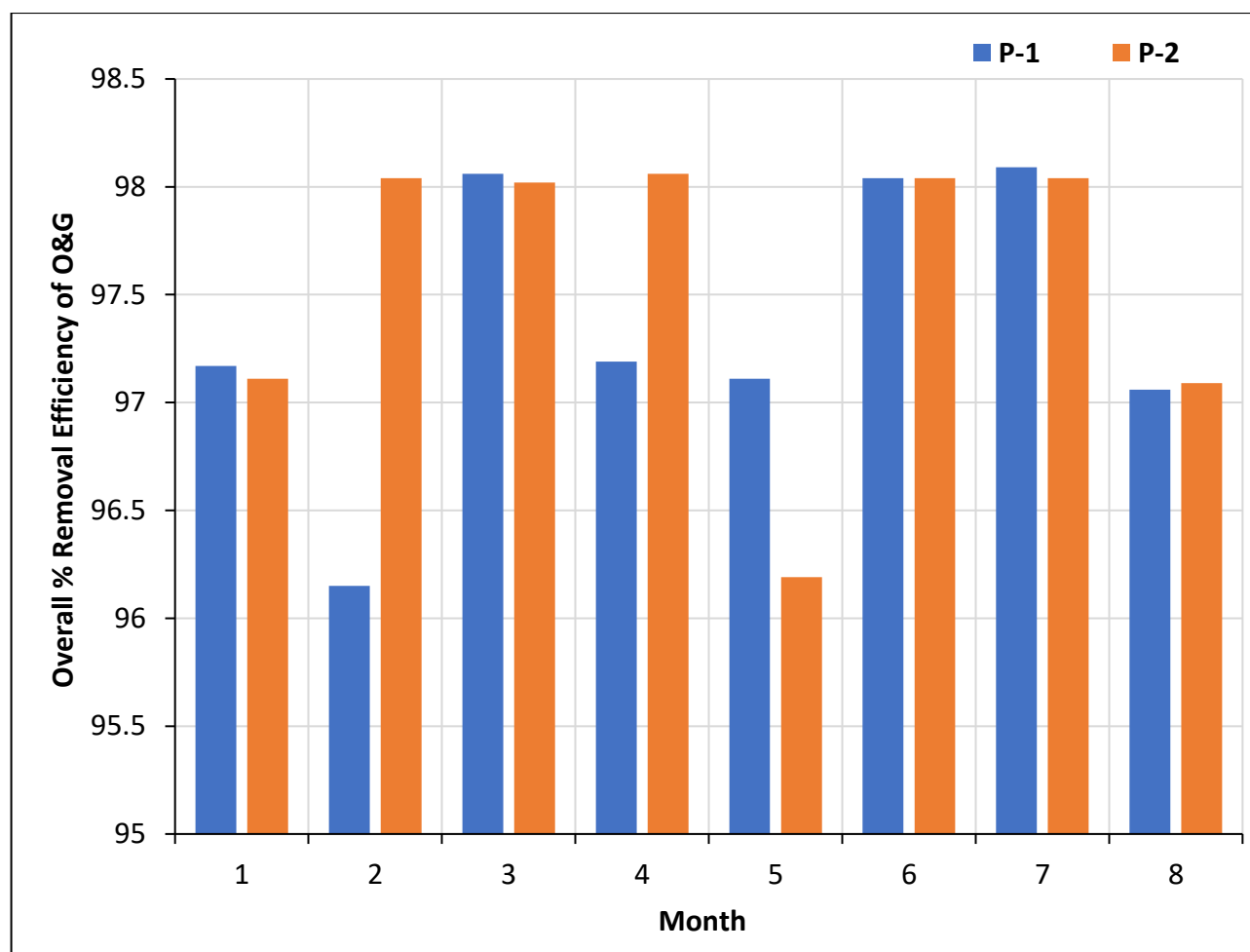


Figure 4.6: Comparison of overall percentage removal efficiency for the two ZLD processes of O&G

4.1.7 Ammoniacal nitrogen:

It is vital to measure the ammonia content of effluent because high concentrations of ammonia can damage the ecosystem. The amount of ammonia in the effluent is determined by its ammoniacal nitrogen content. As effluent is treated, the amount of ammoniacal nitrogen, which represents the initial step of the decomposition of organic matter, will gradually decline.

Ammoniacal nitrogen value of process effluent ranged from 1,800 to 1,860 mg/L, whereas the treated effluent decreased to less than 50 mg/L. Figure 4.7 illustrates the overall ammoniacal nitrogen removal efficiency, which ranged from 97.80 to 98.60% for P-1 & P-2.

Duong, Pham-Hung *et al.*, 2018 discussed about removal of ammoniacal nitrogen in wastewater by using electrochemical method and stated the ammonia removal performance was 99%.

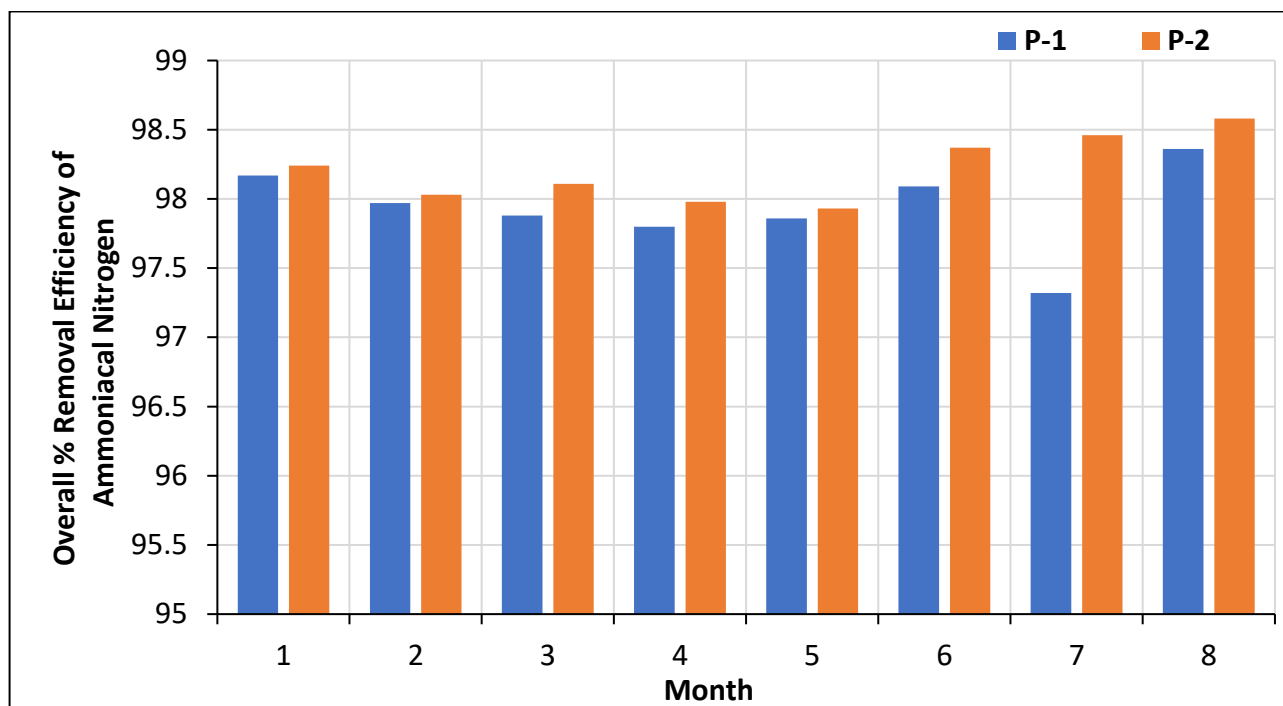


Figure 4.7: Comparison of overall percentage removal efficiency for the two ZLD processes of ammoniacal nitrogen

4.1.8 Chloride

Dissolution of salt deposits, discharges of effluents from chemical industries, oil well operation and seawater intrusion in coastal locations were all causes of chloride in natural waters. The salty taste produced by chloride is determined by the chemical composition of the water. Chlorides are extremely stable and soluble. Chlorides can corrode metals and affect the taste of food products. In surface waterways, excessive corrosion prevents the growth of fish, microbes and plants. Additionally, rising groundwater salinity is now recognized as a severe environmental risk.

The chloride concentration data obtained in the current study ranged from 18,300 to 18,800 mg/L for the process effluent of the two ZLD processes while the treated effluent was less than 50 mg/L. From Figure 4.8, the overall percentage removal efficiency of chloride ranged from 99.750 to 99.820% for P-1 & P-2.

Ranganathan Kuppasamy and Shreedevi D. Kabadgi, 2011 investigated the viability of using reverse osmosis technology to treat tannery effluent and determined that the removal efficiency of chloride was between 91 to 99%.

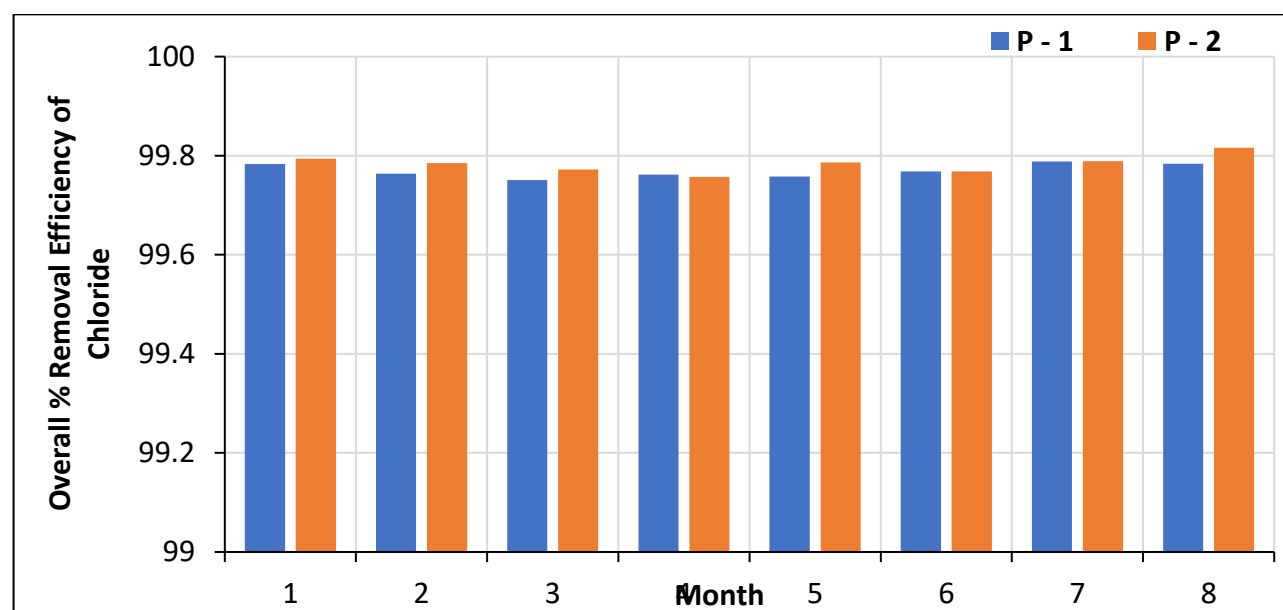


Figure 4.8: Comparison of overall percentage removal efficiency for the two ZLD processes of chloride

4.1.9 Sulphate

Natural water's sulphate concentration must be taken into account when deciding whether it is suitable for use in industrial water systems. When estimating the severity of issues that can result from the conversion of sulphates to hydrogen sulphides, the amount of sulphate in wastewater is a consideration to be concerned about. It is possible to estimate the hydrogen sulphide concentration of the gas produced and the likelihood of sulphide toxicity by knowing the sulphate content of the wastewater.

In the present research, the sulphate concentration value of process effluent for the two ZLD processes ranging from 14,300 to 14,600 mg/L whereas the treated effluent was dropped to less than 55mg/L. As shown in Figure 4.9, it was found that the overall percentage removal efficiency of sulphate during the investigation varied from 99.638 to 99.698% for P-1 & P-2.

Fang Ping *et al.*, 2018 concluded the removal efficiency of high concentration sulphate ions from the wastewater using precipitation method was more than 98%.

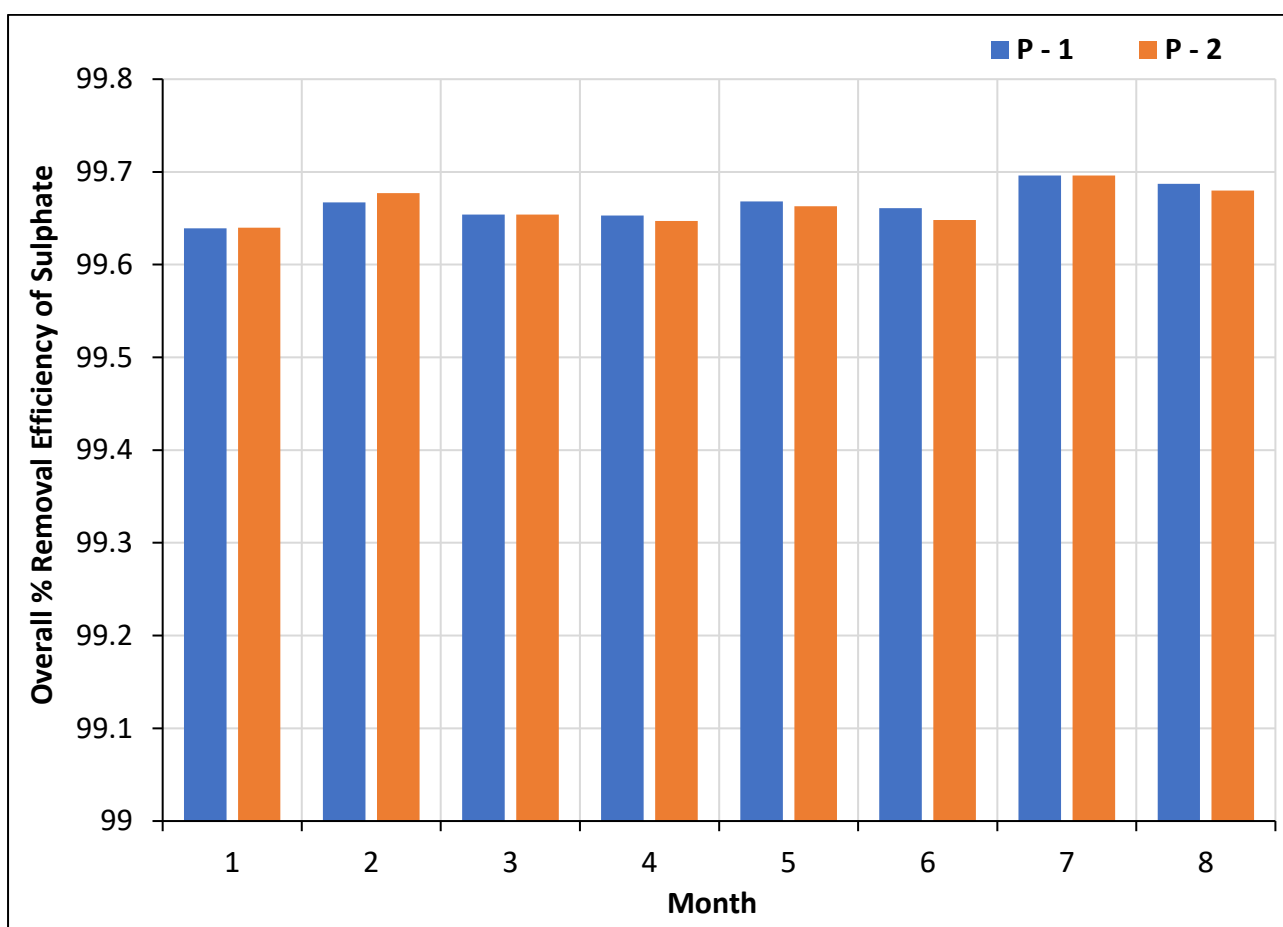


Figure 4.9: Comparison of overall percentage removal efficiency for the two ZLD processes of sulphate

4.1.10 Fluoride

Ions of fluoride are important in water sources in two ways. Fluorosis in the teeth is caused by a high F^- content (disfigurement of the teeth). At the same time, "dental caries" happens at concentrations lower than 0.8 mg/L. Therefore, it is crucial to keep the F^- concentration in drinking water between 0.8 and 1.0 mg/L. Fluoride levels in wastewaters can typically range from 100 mg/L to more than 10,000 mg/L. Fluoride discharge restrictions typically range from less than 20 mg/L for wastewater that can be disposed of into a public sewer system

and less than 5 mg/L for wastewater that must be disposed of into an aquatic environment.

In the present study, it was found that the process effluent of fluoride for the two ZLD processes ranged from 9 to 10 mg/L and the treated effluent was reduced to less than 1 mg/L.

Figure 4.10 showed the overall percentage removal efficiency of fluoride for P-1, P-2 ranging from 91.50 to 95.80%.

Using a neutralising approach, El Diwani *et al.*, 2022 removed 97.64% of the fluoride from the industrial effluent.

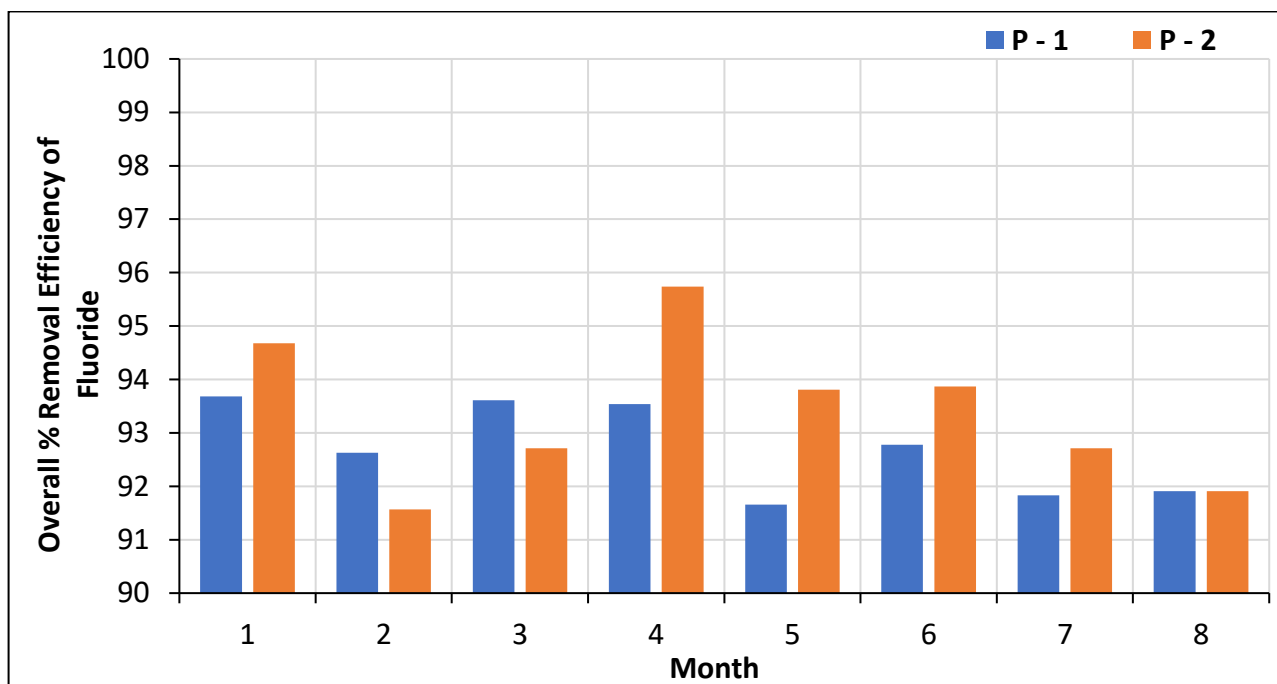


Figure 4.10: Comparison of overall percentage removal efficiency for the two ZLD processes of fluoride

4.1.11 Nitrate

The most highly oxidized form of the nitrogen molecules frequently found in natural waterways is nitrate. Chemical fertilizers, decomposing plant and animal matter, home effluents, sewage sludge disposal to land, industrial discharge, leachates from refuse dump, and atmospheric washout are significant sources of nitrate. These sources have the potential to contaminate lakes, rivers, streams and groundwater depending on the circumstance. Nitrate levels in unpolluted natural water are quite low. Excessive concentrations in drinking water are considered dangerous for new-borns because they are converted to nitrite in the digestive system, resulting in methemoglobinemia.

The process effluent nitrate values of two ZLD processes varied from 45 to 50 mg/L, while the treated effluent's nitrate content was less than 8 mg/L.

During the study, it was observed that overall percentage removal efficiency of nitrate was between 84.20 to 85.70% for P-1 & P-2 as shown in Figure 4.11.

Damrongsri Mongkol and Sutthirut Pruksanan 2007 investigated on the removal efficiency of nitrate from industrial wastewater treatment effluent by activated sludge and estimated the maximum efficiency of nitrate removal was 92%.

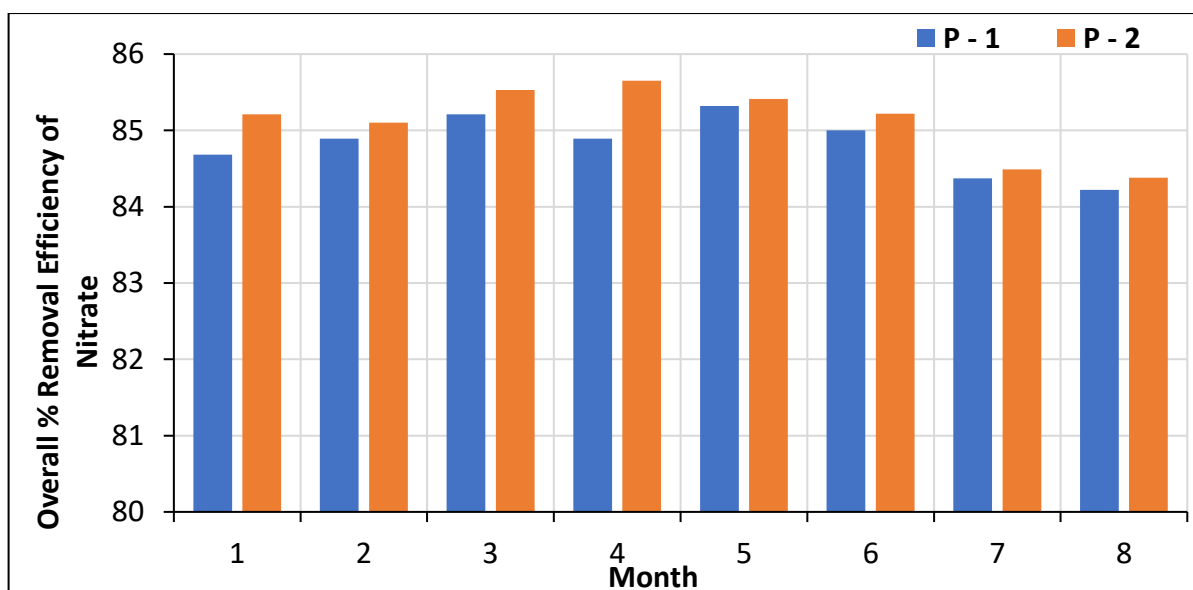


Figure 4.11: Comparison of overall percentage removal efficiency for the two ZLD processes of nitrate

4.1.12 Comparison of treated effluent parameter values with CPCB standards:

Wastewater pollutants must be brought down to acceptable levels for the reuse of industrial effluent. Prior to discharging the effluent, the industry must

establish its own wastewater treatment system to meet CPCB criteria.

Table 4.1 shows minimum and maximum values of parameters after treatment during the study period and their comparison with CPCB standards.

Table 4.1: Comparative statement of treated effluent parameters with CPCB standards

Process Parameter	P - 1		P - 2		CPCB Limit
	Min. value	Max. value	Min. value	Max. value	
pH	7.2	7.5	7.2	7.5	5.5 – 9.0
TDS (mg/L)	154	196	126	194	<2100
TSS (mg/L)	6	9	6	9	<100
COD (mg/L)	38	48	32	46	<250
BOD (mg/L)	24	29	23	29	<100
O&G (mg/L)	2	4	1	4	<10
Ammoniacal Nitrogen (mg/L)	31	40	30	38	<50
Chlorides (mg/L)	38	46	34	45	<600
Sulphates (mg/L)	44	52	44	52	<1000
Fluorides (mg/L)	0.6	0.8	0.4	0.8	<2.0
Nitrates (mg/L)	6.8	7.5	6.6	7.6	<20

5. CONCLUSION

Present study was concerned with the performance study of two ZLD treatment plants. Based on the results obtained from this study, the following points were concluded:

➤ Evaluating the quality parameters:

The overall removal efficiency of pH ranged from 19 to 26%, TDS ranged from 99.58 - 99.75%, TSS ranged from 97.25 to 98.30, COD ranged from 99.930 - 99.960 %, BOD ranged from 99.890 - 99.920%, O&G ranged from 96.10 to 98.10%, ammoniacal nitrogen ranged from 97.80 to 98.60%, chloride ranged from 99.750 to 99.820%, sulphate ranged from 99.638 to 99.698%, fluorides ranged from 91.50 to 95.80% and nitrate ranged from 84.20 to 85.70% for P-1 & P-2. According to the quality analysis, the parameters of treated effluent of the two ZLD processes were reduced to acceptable values in accordance with CPCB standards.

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