PERFORMANCE EVALUATION STUDY OF AN EFFLUENT TREATMENT PLANT IN PHARMACEUTICAL INDUSTRY

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ABSTRACT

Pharmaceutical industry represents a range of industries in operation and processes as diverse as its product. Hence effluents coming from pharmaceutical industries vary from industry to industry. Thus, it is almost impossible to describe a typical pharmaceutical effluent because of such diversity. Considering the above stated implications an attempt has been made in the present project to evaluate the efficiency of ETP.

The present study has been undertaken to evaluate the performance efficiency of an effluent treatment plant. The pharmaceutical industry considered for study, is engaged in manufacturing of various bulk drugs and Active Pharmaceutical Ingredients (API). Wastewater samples were collected at different stages of treatment units and analyzed for the major water quality parameters, such as pH, Total Suspended Solids (TSS), Total Dissolved Solids (TDS), Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD) and Oil & Grease (O&G).

Inlet values of pH ranged 4.5 – 5.5, TSS is of 100 mg/l (Max.) & 60 mg/l (Min.), TDS is of 2720 mg/l (Max.) & 2200 mg/l (Min.), BOD is of 5800 mg/l (Max.) & 5200 mg/l (Min.), COD is of 9000 mg/l (Max.) & 8100 mg/l (Min.) and O&G is of 8 mg/l (Max.) & 6 mg/l (Min.).Outlet values of pH ranged 5.5 - 9.0, TSS is of < 30 mg/l, TDS is of <2000 mg/l, BOD is of < 70 mg/l, COD is of < 230 mg/l and O&G is of < 3.0 mg/l. The TSS, TDS, BOD, COD and O&G values of the treated effluent reduced significantly comparing with influent values before treatment, where as overall percentage reduction of TSS, TDS, BOD, COD and O&G are 85%, 33%, 98%, 99% and 76%. All the parameters evaluated were in the permissible limits of Andhra Pradesh Pollution control Board (APPCB) standards. Hence, the treated effluent is used for irrigation purpose.

Keyboard: Pharmaceutical effluent, TSS, COD, TDS, BOD

1. INTRODUCTION

Water is the main component which is used in all type of the Industries. Water is used for different processes in the industries. It may be used for washing, dilution, formation and condensing the steam. But all water used in the different industry is not totally consumed. Generally, almost all the industries generate waste water that needs urgent attention. Water use in industry is a double-edged sword. On one hand it puts immense pressure on local water resources. On the other, wastewater discharged from the industry pollutes the local environment. Water is required, often in large volumes, by industries as process inputs in most industries. In other cases, like food and beverage and chloro-alkali industry, water is used as a raw material: turned into a manufactured product and exported out of the local water system. However, in most industries it is essentially used as input and mass and heat transfer media. In these industries a very small fraction of water is actually consumed and lost. Most of the water is actually meant for non-consumptive process uses and is ultimately discharged as Effluent.

1.1 Effluent Treatment Plant

Industrial wastewater treatment covers the mechanisms and processes used to treat water that have been contaminated in some way by anthropogenic industrial or commercial activities prior to its release into the environment or its re-use. Most industries produce some wet waste although recent trends in the developed world have been to minimize such production or recycle such waste within the production process. However, many industries remain dependent on processes that produce wastewaters.

So, industries produce wastewater, otherwise known as effluent, as a bi-product of their production. The effluent contains several pollutants, which can be removed with the help of an effluent treatment plant (ETP). The "clean" water can then be safely discharged into the environment [23].

Advantages of wastewater systems

Manufacturers face strict regulations on discharge and waste. Non-compliance can lead to expensive fees and operations interference. A wastewater treatment skid will help you:

- Stay in compliance
- Reduce hauling and off-site treatment costs
- Eliminate municipal fees
- Reduce supply costs by recovering production materials out of the waste-stream for re-use
- Eliminate unnecessary water usage during processing

1.2 Planning an Effluent Treatment Plant: Factors to Consider

Certain factories are required by law to install an ETP but deciding what type of ETP to install, what components it should contain and how it is best managed can be quite complicated. This chapter aims to present some simple ideas about treatment plants and offers practical advice on how to choose the most suitable one for a particular factory.

Any factory needing to install an ETP has to consider several factors. For example, information about the wastewater from the factory is required, including quantity and quality. To get this information the factory will have to take samples and have them analysed at a reputable laboratory [25].

Some of the factors to be considered are presented as follows:

What national or international standards must you comply with?

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Choosing an Effluent Treatment Plant

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What volume of effluent do you have?

↓

What chemicals does it contain?

↓

At what concentrations?

e.g. 30m3/hour with COD of 500ppm, and pH of 11.5

↓

Do you plan to increase production?

↓

Will this increase the amount of effluent to be treated?

↓

How much can you afford to spend on constructing an ETP?

↓

How much can you afford to spend on running an ETP?

↓

How much land do you have available, or can you buy, on which to build the ETP?

↓

Which ETP expert or designer should be used?

↓

What type of plant will best suit your requirements?

(the answers that you give to the above questions will help you and

the designers to decide this).

↓

What capacity do you have in your factory to manage the ETP?

Do you need to hire more staff or train existing

staff

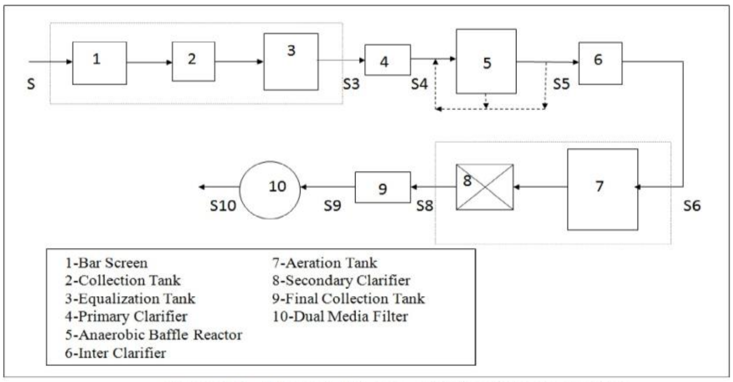

Figure 1.1: Schematic Diagram of Effluent Treatment Plant

S-Raw Effluent
S3-Equalization Tank Outlet
S4-Primary Clarifier Outlet
S5-Anaerobic Baffle Reactor Outlet
S6-Intermittent Clarifier Outlet
S8-Secondary Clarifier Outlet
S9-Final Collection Tank Outlet
S10-Dual Media Filter Outlet (Treated Effluent)

2. MATERIALS AND METHODS

2.1 Reagents and chemicals used:

Analytical grade chemicals were used for the analytical experiments. The chemicals required were purchased from Qualigens, Merck and Fischers.

2.2 Sample handling and preservation:

- Samples are collected in glass bottles. Use of plastic containers is permitted if it is known that there are no organic contaminants present in it.
- Biologically active samples should be tested as soon as possible. Samples containing settable material should be well mixed, preferable homogenized, to permit removal of presentative aliquots.
- Samples should be preserved with sulphuric acid to a pH < 2 and maintained at 4^0C until analysis. Do not allow the samples to freeze.
- Deionized water was used for all the dilutions.

2.3 Testing of sample

In the study period, samples at different stages of treatment units of ETP were collected and analyzed for evaluation of ETP. The collected samples were analyzed for parameters viz., pH, Total Dissolved Solids (TDS), Total Suspended Solids (TSS), Chemical Oxygen Demand (COD), Biochemical Oxygen Demand (BOD) and Oil and Grease(O&G) .

2.3.1 pH

The pH is determined by measurement of the electromotive force of a cell comprising an indicator electrode (an electrode response to hydrogen ions such as glass electrode) immersed in the test solution and a reference electrode contact between the test solution and the reference electrode is usually achieved by means of a liquid junction, which forms a part of the reference electrode. The emf of this cell is measured with pH meter. This is a high impedance electrometer calibrated in terms of pH. 100ml of the sample was taken in a beaker. The electrodes were dipped in it and the pH was recorded.

2.3.2 TDS

- ✓ 50ml of well-mixed sample was filtered through glass fiber filter.
- ✓ Then 10ml of distilled water was allowed to wash for complete drainage between washing and suction was continued for about 3 minutes after filtration is complete.
- ✓ Filtrate was transferred to an empty weighed (W_1) crucible and evaporated on hot plate hot water bath.
- ✓ Crucible was transferred into hot air oven for dryness at 105^0C ± 2^0C for atleast one hour.
- ✓ Then the crucible was cooled in a desiccator and weighed. The process of drying and cooling and weighing was repeated until a constant weight (W_2) was obtained.

2.3.3 TSS

- ✓ The Filter paper disk was taken and dried at 105°C for an hour to remove (any water) moisture adhering to its surface.
- ✓ Then it was cooled in a desiccator and its weight was taken accurately on a precision balance [W_1 (g)].
- ✓ Put the membrane filter on filter holder and wet it with water. 50ml of sample was filtered through it (to get a residue of 200mg) under vacuum.
- ✓ Filter membrane was filtered and dried at 103 - 105°C in an oven.
- ✓ Then the membrane filter was cooled in a desiccator and weighed. The process of drying, cooling and weighing was repeated until a constant weight (W_2) was obtained.

2.3.4 COD

- ✓ 50ml of the sample was taken in a round bottom flask.
- ✓ 1 gm of $HgSO_4$ and some broken porcelain pieces were added to it.
- ✓ The flask was immersed in cold water and slowly 75ml of silver sulfate reagents was added with continuous shaking. (As this is an exothermic process, the flask is immersed in cold water).
- ✓ 25ml of $K_2Cr_2O_7$ (0.25N) was added to this solution and the contents were mixed.
- ✓ Reflux condenser was attached and refluxed for 2 hours.
- ✓ The condenser was washed with distilled water into the flask. Then it was cooled and diluted to 300ml by distilled water.
- ✓ 2-3 drops of ferrion was added as an indicator and was titrated against 0.25N Mohr's salt solution till the end point i.e. from blue to wine red.
- ✓ The volume of Mohr's salt solution used was recorded and let it be X ml.
- ✓ Blank titration was performed using distilled water in place of sample solution. For this, 50ml of distilled water was taken in round bottomed flask and the same amounts of reagents were added and refluxed for two hours. It was titrated in the same way as it was done with the sample water.

- ✓ The volume of Mohr's salt solution used was recorded and let it be Y ml.

2.3.5 BOD

- ✓ Four 300 mL glass BOD bottles with stopper (two for the sample and two for the blank) were taken.
- ✓ 10 mL of the sample was added to each of the two BOD bottles and the remaining quantity was filled with the dilution water.
- ✓ The remaining two BOD bottles were for blank, to these bottles dilution water was added alone.
- ✓ After the addition immediately the glass stopper was placed over the BOD bottles and the numbers were noted on the bottle for identification.
- ✓ Now one blank solution bottle and one sample solution bottle were preserved in a BOD incubator at 20ºC for five days.
- ✓ The other two bottles (one blank and one sample) were analyzed immediately.
- ✓ Any kind of bubbling and trapping of air bubbles were avoided.
- ✓ Then 2mL of manganese sulfate and 2mL of alkali-iodide-azide reagent were added to the BOD bottle by inserting the calibrated pipette just below the surface of the liquid.
- ✓ The pipette was dipped inside the sample while adding the above two reagents. If the reagent was added above the sample surface, we will introduce oxygen into the sample.
- ✓ It was allowed to settle for sufficient time in order to react completely with oxygen.
- ✓ When this floc has settled to the bottom, the contents were shaked thoroughly by turning it upside down.
- ✓ Then 2 mL of concentrated sulfuric acid was added via a pipette held just above the surface of the sample.
- ✓ Carefully stopper was placed and was inverted several times to dissolve the floc.
- ✓ 203 mL of the solution was measured out from the bottle and transferred to an Erlenmeyer flask.
- ✓ The contents were transferred to Erlenmeyer flask and were titrated immediately.
- ✓ The solution was titrated with standard sodium thiosulphate solution until the yellow color of liberated Iodine is 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 during titration was noted.
- ✓ The titration was repeated for concordant values.
- ✓ After five days, the bottles were taken out from the BOD incubator and the sample and the blank were analysed for DO.

2.3.6 O&G

- ✓ 100ml of sample was taken in a beaker.
- ✓ pH of the sample was acidified using dil. HCl.
- ✓ Then the evaporating dish was weighed (initial weight).
- ✓ Then the sample was added to the separating funnel.
- ✓ 5ml of the petroleum ether was added to it and was shaked well for atleast 2 min. Then it was allowed to rest for 2 min. We will find two separated layers, one ether layer and another sample layer. Lower layer was collected to same sample beaker.
- ✓ Ether layer was added to evaporating dish.
- ✓ The above procedure was repeated for 2 more times. Then the ether layer which was collected in evaporating dish was placed in oven and allowed it to evaporate to constant weight. Then allow it to cool. It was weighed again (final weight).

3. RESULTS & DICUSSION

ETP wastewater samples were collected for a period of 4 months. The effluent samples collected were denoted as:

S-Raw Effluent

S3-Equalisation tank

S4-Primary Clarifier Outlet

S5-Anaerobic Baffle Reactor Outlet

S6-Intermittent Clarifier Outlet

S8-Secondary Clarifier Outlet

S9-Final Collection Tank Outlet

S10-Dual Media Filter Outlet (Treated Effluent)

3.1 Evaluation of parameters

3.1.1 pH

Extreme of pH of wastewater are generally not acceptable as extreme of pH causes problems to survival of aquatic life. It also interferes with the optimum operation of wastewater treatment facilities. Water with high or low pH is not suitable for irrigation. At low pH most of the metals become soluble and become available and therefore could be hazardous in the environment. At high pH most of the metals become insoluble and accumulate in the sludge and sediments.

pH of the individual samples were measured immediately after its collection by a pH meter. Initially the pH value of the influent was as low as 4.5 and as high as 5.1 before the treatment and the pH value of the treated effluent was as low as 7.5 and as high as 7.9. The pH value was increased from S to S10 as shown in Table 5.1 and Figure 5.1 to bring the effluent pH to neutral stage.

Hashmi Imran (2005) reported that the pH of wastewater samples is generally towards acidic side. Here the effluent treated was taken from pharmaceutical industry. The pH value of the influent before the treatment was 4.5 and the pH value of effluent after treatment was 9.0.

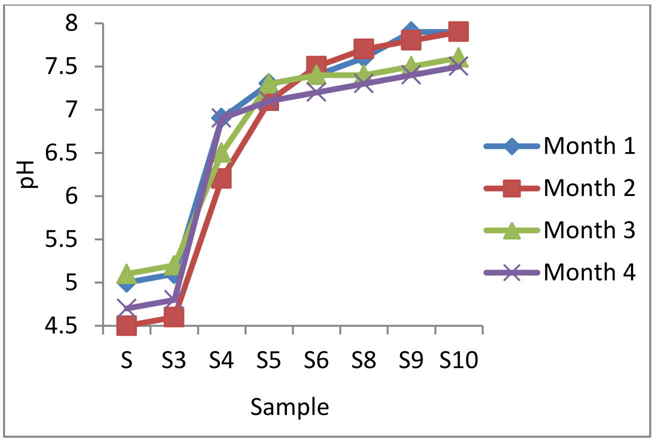

Figure 3.1: Variation of pH

3.1.2 TDS:

High TDS in treated effluent is a widespread problem in many common effluent treatment plants. It was observed that salinity, primarily due to salts of sodium, was the primary contributor to the high TDS problem, as high TDS was almost invariably accompanied by high chlorides and sodium concentration. The TDS concentration of the wastewater was mainly due to the inorganic ions in the water supply and those added during the use of water.

The values for TDS were reduced from S to S10 as shown in Figure 5.2. In the present study, it was found that the TDS value of raw effluent ranged from 2200-2720 mg/l and the treated effluent was ranged from 1800-2000 mg/l.

V Krishna Murthy Roshan Makam, 2012 reported on physico-chemical analysis of effluents from pharmaceutical industry where we can observe that the raw effluent value of TDS was 2272 mg/l and the treated effluent value of TDS after treatment was 2132 mg/l.

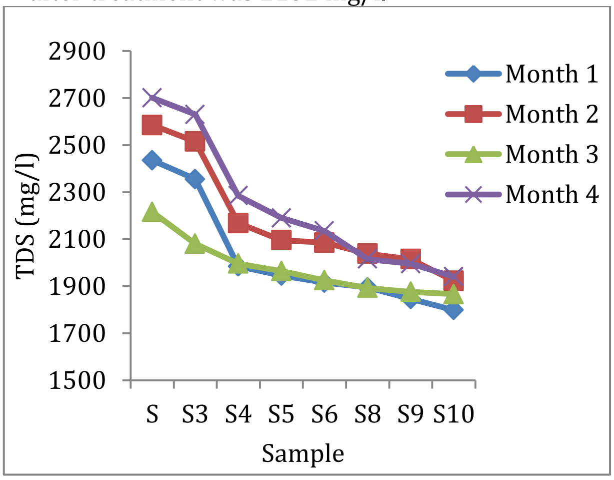

Figure 3.2: Variation of TDS in mg/l

3.1.3 TSS:

Total suspended solids play an important role in water and wastewater treatment. Suspended solid do not mean that they are floating matters and remain on top of water layer. They are under suspension and remain in water sample. So, their presence in water sample causes depletion of oxygen level.

The values for TSS were reduced from S to S10 as shown in Table 5.3and Figure 5.3. In the present study, it was found that the TSS value of raw effluent has a maximum value of 90 mg/l and the treated effluent has <30 mg/l.

Asith K A, 2012 reported on physico-chemical analysis of effluents from pharmaceutical industry and its efficiency study. This paper presents that the raw effluent value of TSS before treatment was 100 mg/l and the treated effluent value of TSS after treatment was 94mg/l.

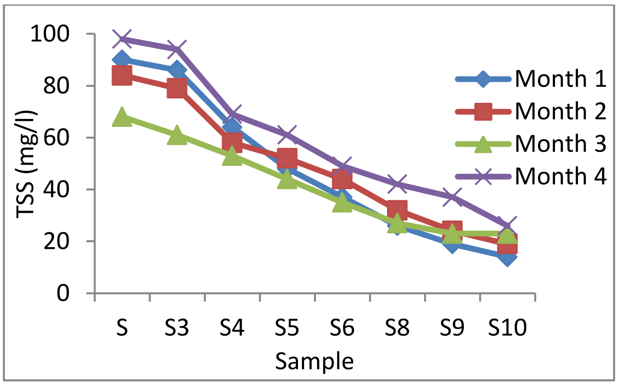

Figure 3.3: Variation of TSS in mg/l

3.1.4 COD:

COD determines the oxygen required for chemical oxidation of organic matter with the help of strong chemical oxidant. COD is a test which is used to measure pollution of domestic and industrial waste. The waste is measure in terms of equality of oxygen required for oxidation of organic matter to produce CO2 and water. It is a fact that all organic compounds with a few exceptions can be oxidizing agents under the acidic condition. COD test is useful in pinpointing toxic condition and presence of biological resistant substances.

For COD determination, samples were preserved using H_2SO_4 and then processed. The COD value was reduced from S to S10 as shown in Table 5.4 and Figure 5.4. In the present study, the range of COD in the raw effluent was 8100-9000 mg/l and the treated effluent was reduced to a range 190-230 mg/l.

Ketan A.Salunke, 2014 reported on evaluation and the performance of common effluent treatment plant in a chemical industry. This paper presents that the raw effluent value of COD before treatment was 1200 mg/l and the effluent value of COD after treatment was 180mg/l.

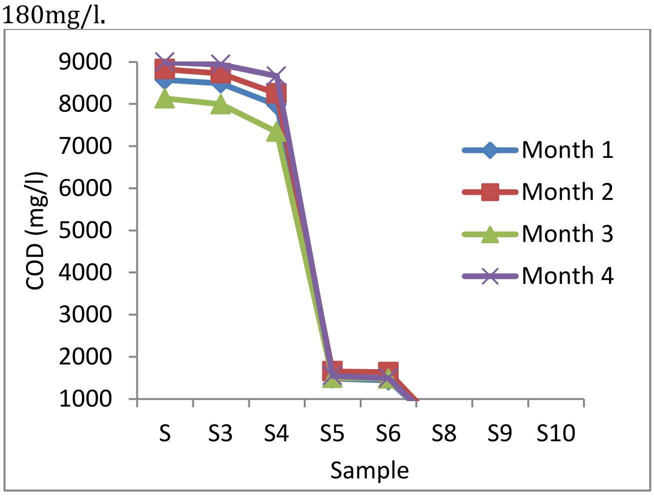

Figure 3.4: Variation of COD in mg/l

3.1.5 BOD:

The amount of oxygen required by the microbial activity to oxidize and stabilize the decomposable organic matter is called BOD. BOD determination is an empirical test in which standardized laboratory procedures are used to determine relative oxygen requirements of wastewater. The test has widest application in measuring waste loading to treatment plants and evaluation of BOD removal efficiency of such treatment systems. The test has its limitations but still used extensively and is useful for determining approximately how much oxygen will be removed from water by an effluent or how much may be required for treatment and to estimate size of the treatment plant needed.

For BOD, samples were immediately processed after collection for the determination of initial oxygen and incubated at 20°C for 5 days for the determination of BOD at 5^{th} day. The BOD value was reduced from S to S10 as shown in Table 5.5 and Figure5.5. In the present study, it was found that the BOD value of the raw effluent varied from 5200-5800 mg/l and the treated effluent varied from 35-70 mg/l.

Prashant.P.Bhave, 2014 reported on performance status of common effluent treatment plant in a pharmaceutical industry. This paper presents that the raw effluent value of BOD before treatment was 500 mg/l and after treatment it was reduced to 50mg/l.

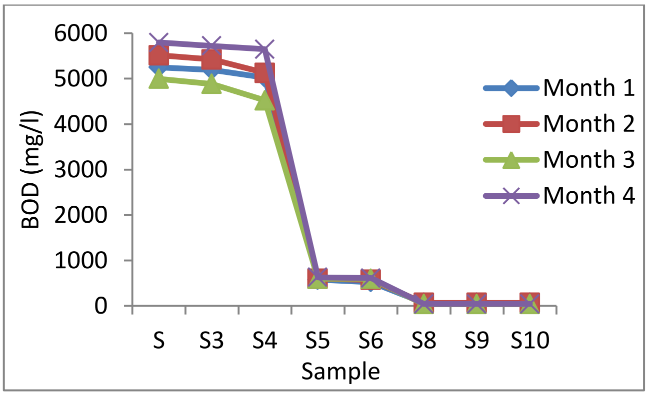

Figure 3.5: Variation of BOD in mg/l

5.1.6 O&G:

Oil and grease are organic in nature and contribute BOD and COD. Their presence in high quantities in wastewater results in the formation of scum on the surface, which interferes with the penetration of solar radiations and hence biological activity. High oil and grease content also interferes with the performance of pumps. If grease is not removed before discharge of treated wastewater, it can interfere with the biological life in the surface water and create unsightly films.

Table 5.6 and Figure 5.6 represent the concentration of oil & grease at different sampling points and here the O&G value was reduced from S to S10. The concentration of oil & grease in raw effluent ranged from 6-8 mg/l and treated effluent ranged from 1.5-2.5 mg/l.

N. V. Srikanth Vuppala, 2012 reported on treatment process of effluent in Bulk drug industry. This paper presents that the raw effluent value of O&G before treatment was 25 mg/l and the treated effluent value of O&G after treatment was 9 mg/l.

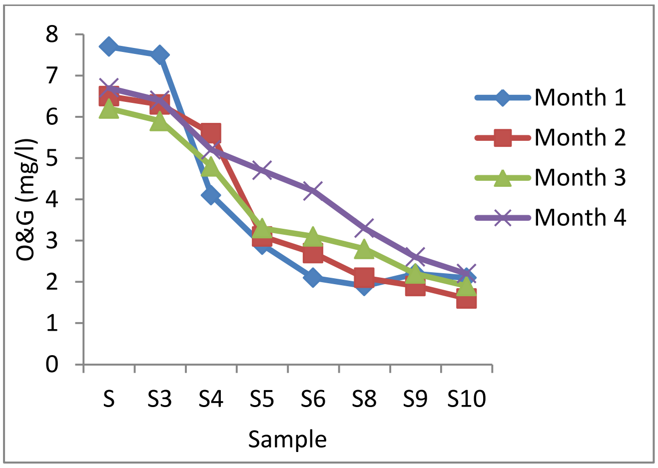

Figure3.6: Variation of O&G in mg/l

3.2 Comparison of Treated Effluent Parameter Values with APPCB Standards:

It is important for the industry to develop its own wastewater treatment system before discharging the effluent in order to meet the Andhra Pradesh Pollution control Board (APPCB) standards. Reduction of pollutants in the wastewater down to permissible concentrations is necessary for the protection of ground water and the environment.

Table 6.13 is showing minimum and maximum values of parameters during the study period and their comparison with APPCB standards.

3.3 Overall Percentage Removal Efficiency

3.3.1 TDS

The overall percentage reduction of TDS in the Effluent Treatment Plant has been shown in the Table 5.8 and Figure 5.7. During the study, it has been observed that overall removal efficiency of TDS ranged from 15-33%.

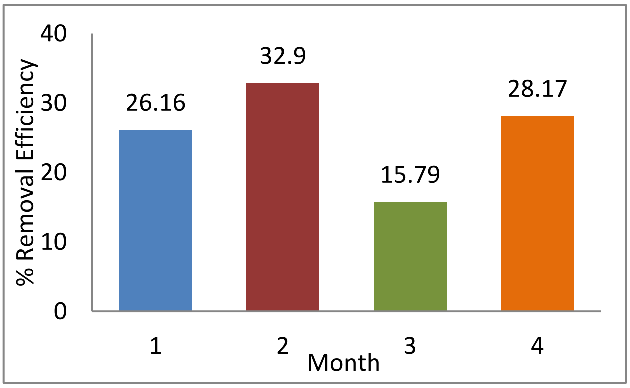

Figure 3.7: Percentage Removal Efficiency of TDS (%)

3.3.2 TSS

The overall percentage reduction of TSS in the Effluent Treatment Plant has been shown in the Table 5.9 and Figure 5.8. During the study, it has been observed that overall removal efficiency of TSS varied from 73-85%.

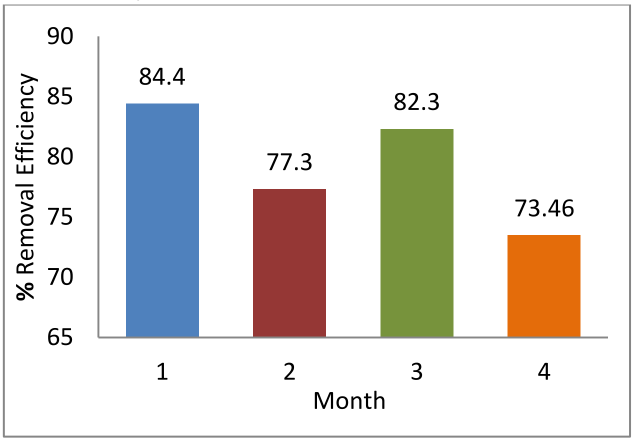

Figure 3.8: Percentage Removal Efficiency of TSS (%)

3.3.3 COD

The overall percentage reduction of COD in the Effluent Treatment Plant has been shown in the Table 5.10 and Figure 5.9. During the study, it has been observed that overall removal efficiency of COD was found to be in the range 97-98%.

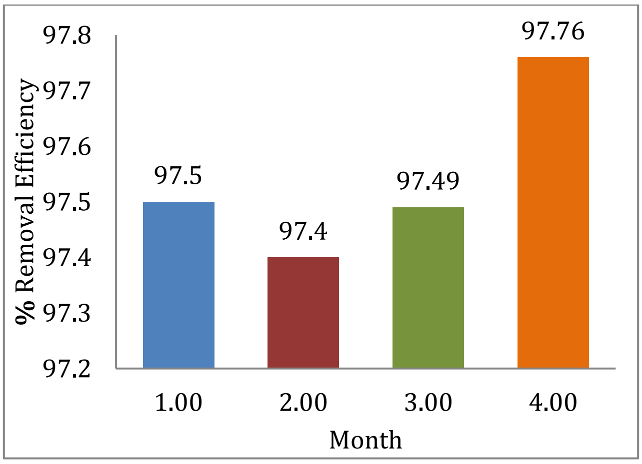

Figure 3.9: Percentage Removal Efficiency of COD (%)

3.3.4 BOD

The overall percentage reduction of BOD in the Effluent Treatment Plant has been shown in the Table 5.11 and Figure 5.10. During the study, it has been observed that overall removal efficiency of BOD was found to in the range 98-99.50%.

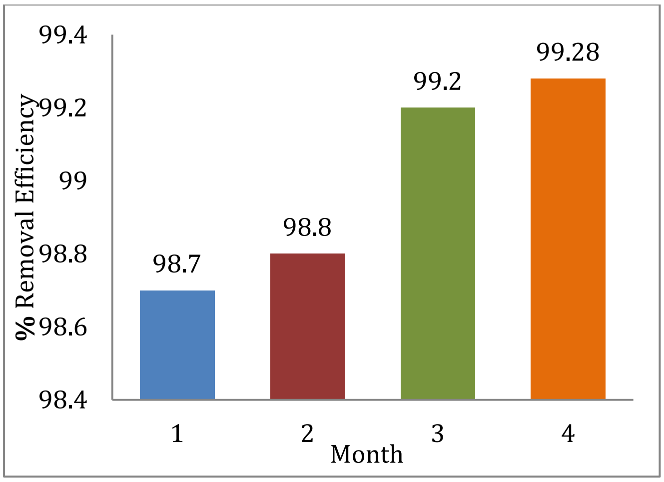

Figure 3.10: Percentage Removal Efficiency of BOD (%)

3.3.5 O&G

The overall percentage reduction of O&G in the Effluent Treatment Plant has been shown in the Table 5.12 and Figure 5.11. During the study, it has been observed that overall removal efficiency of O&G ranged from 67-76%.

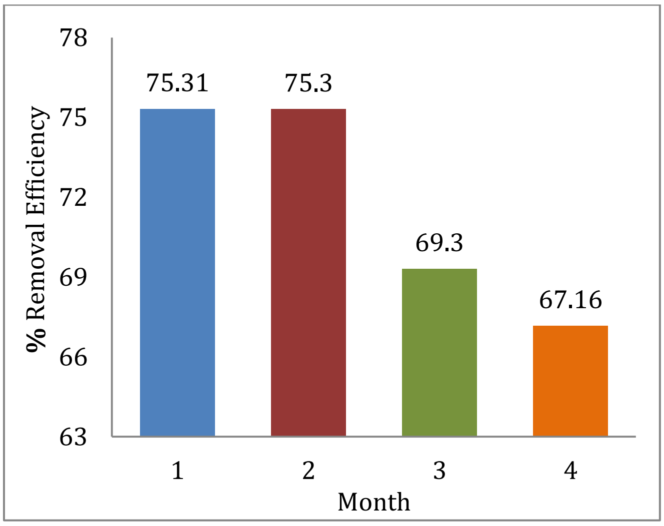

Figure 3.11: Percentage Removal Efficiency of O&G (%)

4. CONCLUSION

Present study was concerned with the performance evaluation of ETP for pharmaceutical industry. Based on the results obtained from this study, the following points were concluded:

- It was found that pH values of raw effluent were ranging from 4.6 to 5.2. The pH values of treated effluent were varying from 7.4 to 7.9.
- It was found that TDS concentration of raw effluent was varying from 2200-2720 mg/L. TDS concentration in treated effluent samples fluctuated from 1800 - 2000 mg/L.
- TSS concentration of raw effluent were ranging from 60 - 100 mg/L. TSS concentration of treated effluent were ranging from 12 - 30 mg/L.
- COD concentration of raw effluent were varying from 8100-9000 mg/L. Maximum and minimum values of COD for treated effluent were 190 mg/L and 230 mg/L respectively.
- BOD concentration of raw effluent was fluctuating from 5200-5800 mg/L. BOD of treated effluent was ranging from 35 - 70 mg/L.
- O&G concentration of raw effluent was fluctuating from 6-8 mg/L. O&G of treated effluent was ranging from 1.5 - 2.5 mg/L.
- The overall percentage reduction of TSS, TDS, BOD, COD and O&G in the Effluent Treatment Plant during the study period of 4 months was found in the range of 73-85%, 15-33%, 97-98%, 98-99% and 67-76%.

The parameters above were in the permissible limits of Andhra Pradesh Pollution control Board (APPCB) standards. Finally, the effluent after the treatment from the ETP can now be used for irrigation purpose.

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