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Waste Water Treatment: Design and Develop Waste Water Disposal Method for Pharmaceutics Laboratory

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Abstract: *Waste water is any water that has been adversely affected in quality by anthropogenic influence. Many pharmaceutical industries is responsible to generates toxic effluent. The waste water generated from these industries possess solids, biodegradable and nonbiodegradable organic compounds. Pharmaceutical compounds typically produced in batch process leading to the presence of a wide variety of products in waste water which are generated in different operations. Various sources of pharmaceutical industries are different sectors of Active pharmaceutical ingredients (API), bulk drugs, and formulation department. Pharmaceutical residues and/or metabolites are usually detected in the environment at trace levels but even that low concentration levels but can induce toxic effects. Pharmaceutical waste water if disposed with insufficient treatment may leads to great damage to the environment and ground resources. Need of waste water treatment is to remove organic and inorganic matter this would otherwise cause pollution, to remove pathogenic disease-causing organism, in order to protect the environment and human health. The treatment of waste water is divided into three parts physical, biological and chemical. Waste water treatment process may reduce suspended solids, biodegradable organics, and pathogenic bacteria. Sand filtration, followed by chemical treatment is a proven procedure to treat the the pharmaceutical waste water for disposal as well as reuse. Method develop to treat the collected laboratory waste water. Various materials were used to treat this collected pharmaceutics laboratory waste water. With the help of various parameters pharmaceutical waste water were evaluated, parameters use for the evaluation of pharmaceutical waste water are Biochemical oxygen demand (BOD), Chemical oxygen demand (COD), Total dissolved solids (TDS), Total suspended solids (TSS), colour, Turbidity, Microbial anal*ysis.

Keywords*- Waste water, Biochemical oxygen demand, Total dissolved solids.*

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I. Introduction

Water, pre-requisite for life and key resource of humanity is in abundance on earth. Water that exists on the Earth surface is present as a water of oceans, lakes, rivers and glaciers. **[1]**Although India occupies only 3.29 million km^2 geographical area which forms 2.4% of the worlds land area, it supports over 15% of world's population with only 4% of the world's water resources. Waste water is full of contaminant including bacteria, chemicals and other toxins. Its treatment aims at reducing the contaminants to acceptable levels to make the water safe for discharge back into the environment **[2]**

Need to remove organic and inorganic matter this would otherwise cause pollution.
To remove pathogenic (disease causing) organisms.In order to protect:
The environment
Human health **[3]**

Water pollution is of widespread national concern. Industrial activities generate a large number and variety of waste products. The nature of industrial waste depends upon the industrial processes in which they originate. The problem of adequately handling industrial waste waters is more complex and much more difficult because industrial waste water vary in nature from relatively clean rinse waters to waste liquors than are heavily laden with organic or mineral matter or with corrosive, poisonous, inflammable or explosive substances. As a result of rapid industrial growth following World War II, the amount of waste material generated by industries has increased manifold and the treatment/removal of these contaminants from the natural resources such as air and water in which they are released has progressed into a special science, involving chemical, mechanical and biological processes. The impure water containing inorganic salts, organic compounds, microbial contamination and turbidity disturbing the natural hydrologic cycle (water cycle). The hydrologic cycle can be maintained by the removal of toxic chemicals by many scientifically simple yet sometimes technologically very complex methods. [4]

Pharmaceutical manufacturers use water for process operations, as well as for other non-process purposes. However, the use and discharge practices and the characteristics of the wastewater will vary depending on the operations conducted at the facility. Additionally, in some cases, water may be formed as part of a chemical reaction. Process water includes any water that, during manufacturing or processing, comes into direct contact with or results from the use of any raw material or production of an intermediate, finished product, byproduct, or waste. Process wastewater includes water that was used or formed during the reaction, water used to clean process equipment and floors, and pump seal water. Non-process wastewater includes noncontact cooling water (e.g., used in heat exchangers), noncontact ancillary water (e.g., boiler blow down, bottle washing), sanitary wastewater, and wastewater from other sources (e.g., storm water runoff). [5]

Pharmaceutical manufacturers generate process wastewater containing a variety of conventional parameters (e.g., BOD, TSS, and pH) and other chemical constituents.Many pharmaceutical industries are responsible to generate toxic effluent as a consequence of their operation. The waste water generated from these industries possess solids, biodegradable and nondegradable organic compounds etc. **[6]**

In general, the numerous unit operations and processes to remove wastewater contaminants are grouped together to provide various levels of treatment. The treatment of water is divided into 3 parts:

1. Physical - primary methods are referred to physical or physical-chemical unit Operations. e.g. filtration, adsorption, air flotation, flocculation and sedimentation.
2. Biological- secondary referred to biological operations. e.g. aerobic, anaerobic and activated sludge.
3. Chemical- advanced or tertiary referred to chemical or to combinations of all three e.g. Thermal oxidation (combustion), Chemical oxidation, Ion exchange, Chemical precipitation, incineration. The use of conventional water and wastewater treatment processes becomes increasingly challenged with the identification of more and more contaminants, rapid growth of population and industrial activities, and diminishing availability of water resources.**[22]**The actual state performance of conventional methods is clearly not suitable to treat toxic, non-biodegradable organic pollutants and new improved treatments must be developed and tested. To overcome the inconveniences of conventional treatment methods, Advanced Oxidation Techniques (AOP's) have emerged in the last decades, for the treatment of industrial wastewaters.**[7]**

Waste water treatment processes may reduce -Suspended solids Biodegradable organics

Waste water treatment process of converting waste water or water which is no longer for use and needed or no longer suitable for use and which cannot be discharged into the environment. Conventional waste water treatment consists of a combination of physical, chemical and biological processes and operations to remove solids, organic matter and sometimes nutrient from waste water.

Pharmaceutical wastewater if disposed with insufficient treatment may leads to great damage to the environment and groundwater resources. General treatment cannot be used for every pharmaceutical waste water due to its variable composition. Therefore, specific treatment is required for specific type of waste water. Sand filtration, followed by chemical treatment is a proven procedure to treat the waste water for disposal as well as reuse. Studies suggested the similar methods for treatment of pharmaceutical waste water addition to the granular activated carbon adsorption. In present study physicochemical waste processes such as coagulation, flocculation, sedimentation and sand filtration and activated carbon adsorption were evaluated. Common coagulants alum, ferric chloride, and ferrous sulphate were used for the preferable removal of suspended solids, biochemical oxygen demand, chemical oxygen demand, and turbidity. **[8]**

II. Experimental Work

Pharmaceutical Waste Water Treatment Plan

We have chosen pharmaceutics laboratory for the collection of pharmaceutical waste water. The six-month (October-March) study was performed. In a one month five days were selected. In a single day at different timing (11.00 AM, 2.00 PM, 5.00 PM) pharmaceutical waste water was collected.

The collected pharmaceutical waste water was evaluated before and after the treatment. Pharmaceutical waste water treatment method. The pharmaceutical waste water was treated (Filtered) with the help of sand, alum, charcoal, flocculating agent, hydrogen peroxide etc. Stepwise procedure followed to treat pharmaceutical waste water.

The evaluation parameter of pharmaceutical waste water is:

Biochemical oxygen demand (BOD)
Chemical oxygen demand (COD)
Total dissolved solids (TDS)
Total suspended solids (TSS)
Turbidity
Microbial analysis
pH

Color
Odor
Methods for the evaluation parameter of pharmaceutical waste water
2.1 Biochemical Oxygen Demand (BOD)
Titrimetric method used to measure the biochemical oxygen demand and titrant use was sodium thiosulphate.

$$\text{Calculation of BOD} = \frac{(D0\text{-}D5\text{-}BC) * \text{volume of diluted sample}}{\text{Volume of sample taken}}$$

D0- Dissolved oxygen at the 1st day
D5- Dissolved oxygen at the 5th day
BC- Blank correction (C0-C5) [9]

2.2.Chemical Oxygen Demand (COD)

- Pipette 20ml of sample in 250 ml of refluxing flask.
- Add 10 ml of potassium dichromate by pipette.
- 30 ml of conc. sulfuric acid reagent by measuring cylinder. Acid should be added in controlled manner with mixing the sample.
- Add approximately 400mg of mercuric sulphate.
- If the sample colour changes to green, dilute the sample and repeat the procedure for diluted sample
- Connect the reflux through the condenser and reflux for a minimum period of 2 hrs at 150°C.
- Add 80 ml of distilled water through the condenser cool it to room temperature and titrated with standard sulphate using 2-4 drops of ferroin indicator.

$$\text{COD} = \frac{\{(B\text{-}V) * N(FAS) * 8000\}}{\text{Volume of sample (ml)}}$$

B –Volume of titrant require for the blank titration
V – Volume titrant require for titration of sample [10]
2.3. Total Suspended Solids (TSS)
Simple filtration with the help of selective filters and evaporating the residue in the oven at 103˚C.
Calculation of total suspended solid
1. Weight of the clean filter paper (gm) = X gm
2. Weight of clean filter paper and residue (gm) =Y gm
3. Weight of residue (gm) = Y-X gm
4. Volume of sample 10 ml [11]
2.4. Turbidity
1. Prepare the hydrazine sulphate reagent (1gm in 100ml).
2. Prepare the hexamethylenetetramine reagent (10 gm in 100ml)
3. Standard 4000NTU solution –Mix 5 ml of hydrazine sulphate and 5ml of hexamethylenetetramine solution in 100ml volumetric flask.
4. With the help of above mention stock solution calibrate the turbidity meter
5.Testing of sample- To the sample cells, add the sample water up to the mark, wipe gently with soft tissue paper and place it in turbidity meter such that the vertical mark in the sample cell should coincide with the mark in the turbidity meter and cover the sample cell.
6. Check the reading in the turbidity meter [12]
2.5. Total Dissolved Solids (TDS)
1. Total dissolved solids were measured with the help of HM DIGITAL TDS meter.
2. In the sample the meter was inserted up to the mark and then it directly displays the reading in PPM

2.6Microbial Analysis
Pour the sample on the agar plate an incubate it for 24 hrs. After the period of incubation scrap the upper surface of plate dilute it with water and prepare slide using simple stain and examine under the microscope.

2.7 Colour and Odour
Visually colour of a sample was identified, and odour also checked manually.

III. Result and Discussion

3.1 Results of untreated pharmaceutics laboratory graywater

Table 1

Sr. No.	Parameter	Day 1			Day 2			Day 3			Day 4			Day 5			Average
1	BOD(mg/l)	5.6	10.1	12.2	10.4	11.5	17.4	10.3	12.6	18.9	9.4	13.4	16.2	11.7	12.9	18.9	12.76
2	COD(mg/l)	12.7	26.4	47.4	24.1	_	38.4	14.4	_	54.7	14.4	_	47.5	16.1	61.1	42.1	34.99
3	TDS(PPM)	1932	3047	1606	2019	1053	1678	1932	2052	2104	2090	2042	1069	3210	1832	1094	133.26
4	TSS(gm)	0.212	0.123	0.190	1.130	1.012	0.292	0.129	0.323	0.291	0.321	0.431	1.004	0.423	0.624	0.132	0.43
5	Turbidity (NTU)	0989	1072	1292	1142	1953	1063	2092	1932	1772	1321	1032	0952	0962	0992	1105	619.13
6	Microbial Analysis	++	+++	+++	+	+++	+++	+	+++	++++	+	+++	++++	+	+++	++++	
7	Colour	Dirty white	Pale yellow	white	Brownish	Purple	Off white	pink	orange	yellow	browish	Fresh red	Charcoal black	Off white	Slight pink	bluish	
8	Odour	Peppermint	Oily	camphorous	Iodine	Surfactant	camph	Menthol	Tinctury	oily	iodine	iodine	Odourless	peppermint	formulatory	surfactant	
9	Ph	5.64	6.63	6.93	7.52	5.82	8.62	6.83	7.63	9.43	4.63	9.53	6.91	4.62	7.73	8.42	7.126

Table 1 shows Biochemical oxygen demand was slightly increase at the time of 5.00PM. Chemical oxygen demand was maximum than the normal range at 2.00PM. Different sample at the time interval have different values of Turbidity, pH, Total dissolved solids, and Total suspended solids. Also, different sample have assorted color and odor. Microbial load was maximum at the time of 5.00 PM

3.2 Results of treated pharmaceutics laboratory graywater

Table 2

Sr. No.	Parameter	Day 1			Day 2			Day 3			Day 4			Day 5			Average
1	BOD(mg/L)	0	0.9	3.2	1.8	5.9	7.4	1.8	9.2	11.2	2.6	4.1	6.9	9.7	10.8	11.4	5.79
2	COD(mg/L)	5.4	14.9	29.14	13.4	56.1	23.8	9.8	64.1	28.1	8.5	35.4	24.3	3.1	30.4	34.1	25.36
3	TDS(PPM)	1120	1930	1060	1410	743	977	13250	1050	1293	1920	1410	1250	982	1810	1680	89.6
4	TSS(gm)	0.005	0.045	0.041	0.393	0.007	0.01	0.08	0.03	0.133	0.131	0.272	0.038	0.107	0.12	0.15	0.104
5	Turbidity (NTU)	419	512	796	524	857	658	854	760	986	970	386	774	467	329	468	492.1
6	Microbial Analysis	_	+	+	_	+	_	_	_	_	_	_	+	_	_	_	
7	Colour	Brown	Off White	Off white	Pink	Colourless	Colourless	Off white	pink	Off white	Fresh brown	peach	Oily yellow	Dirty white	Pale peach	Brownish	
8	Odour	Fishy	Rotten egg	Surfactant Like	Mentholike	Odourless	Unpleasaant	Baodourless	odourless	oily	Odourless	calamine	Camphorlike	Menthol like	Calamine like	Bad	
9	pH	5.70	5.82	5.96	8.46	6.76	6.00	4.04	1.83	6.99	5.32	6.99	6.29	10.29	6.42	3.7	6.038

Table 2 shows Biochemical oxygen demand was decrease after the treatment of pharmaceutics laboratory graywater but it slightly maximum at the time of 5.00 PM. Chemical oxygen demand was decreased after the treatment of pharmaceutics laboratory graywater. Total dissolved solids, Total suspended solids, pH was decrease after the treatment of pharmaceutics laboratory graywater. Microbial load was also decrease after the treatment.

IV. Discussion:

Treatment Plan of Pharmaceutics Laboratory Greywater

4.1 Aeration

Aeration allows for the intimate exposure of water and air by intensely mixing the air and water so that chemical reactions can occur between the air and water in the aerators. The primary objective of aeration is to improve water quality by eliminating tastes and odour producing substances such as hydrogen sulphides and carbon dioxide. In this work marketed Fish Pond aerator was used.

4.2 Sedimentation

Sedimentation is a physical treatment process that utilizes gravity to separate suspended solids from water. This was the next stage of the treatment processes, used to remove turbidity causing particles after coagulation and flocculation. The treatment was carried out in an intermittent tank with a hopper at the bottom where sludge was deposited and removed. The specification of sediment filter is 5µ.

4.3 Filtration

Filtration is the process of passing water through a porous medium with the expectation that the filtrate has a better quality than the influent, the medium is usually granular bed, such as sand, anthracite, garnet, or activated carbon. Filters can be classified according to the medium type as single (mono.) medium filters, dual media filters, and mixed-media filters. The last stage was gravity filtration. The specification of this filter (polypropylene) is 0.5µ.

4.4 Coagulations and flocculation

Coagulation is a complex process, involving many reactions and mass transfer steps. As practiced in water treatment the process is essentially three separate and sequential steps: coagulant formation, particle destabilization, and inter-particle collisions. These processes had been achieved by adding chemical material. These chemicals involved in coagulation are known as coagulants or coagulant aids. Choice of specific coagulants and coagulant aids depend on the nature of the solid– liquid system to be separated.

4.5 Biochemical Oxygen Demand (BOD):

The BOD of the sewage is the amount of oxygen required for the biochemical decomposition of biodegradable organic matter under aerobic conditions. The oxygen consumed in the process is related to the amount of decomposable organic matter. The general range of BOD observed for raw sewage is 100 to 400 mg/L. The range of BOD of untreated pharmaceutics laboratory grey water - 9-19.13 mg/l and treated pharmaceutics laboratory grey water 4-13 mg/l % reduction 55-32 (fig 8)

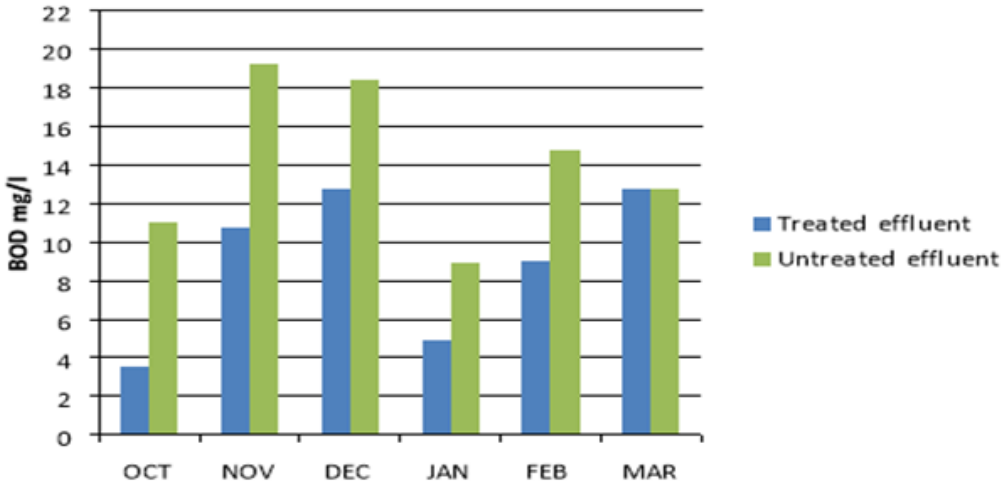


Fig. 8 Biochemical Oxygen Demand Histogram

4.6 Chemical Oxygen Demand (COD)

The COD gives the measure of the oxygen required for chemical oxidation. It does not differentiate between biological oxidisable and nonoxidisable material. However, the ratio of the COD to BOD does not change significantly for particular waste and hence this test could be used conveniently for interpreting performance efficiencies of the treatment units. In general, the COD of raw sewage at various places is reported to be in the range 200 to 700 mg/L. In COD test, the oxidation of organic matter is essentially complete within two hours, whereas, biochemical oxidation of organic matter takes several weeks. In case of wastewaters with a large range of organic compounds, an extra difficulty in using BOD as a quantitative parameter is that the rate of oxidation of organic compounds depends on the nature and size of its molecules. Smaller molecules are readily available for use by bacteria, but large molecules and colloidal and suspended matters can only be metabolized after preparatory steps of hydrolysis. It is therefore not possible to establish a general relationship between the experimental five-day BOD and the ultimate BOD of a sample, i.e., the oxygen consumption after several weeks. For sewage (with k=0.23 d-1 at 20o C) the BOD5 is 0.68 times of ultimate BOD, and ultimate BOD is 87% of the COD. Hence, the COD /BOD ratio for the sewage is around 1.7. 13.1.8

The range of COD of untreated pharmaceutics laboratory graywater26 -225mg/l and for treated pharmaceutics laboratory grey water -10-30mg/l % reduction in October the reduction was almost 90% and in remaining months the reduction was 25% (fig 9)

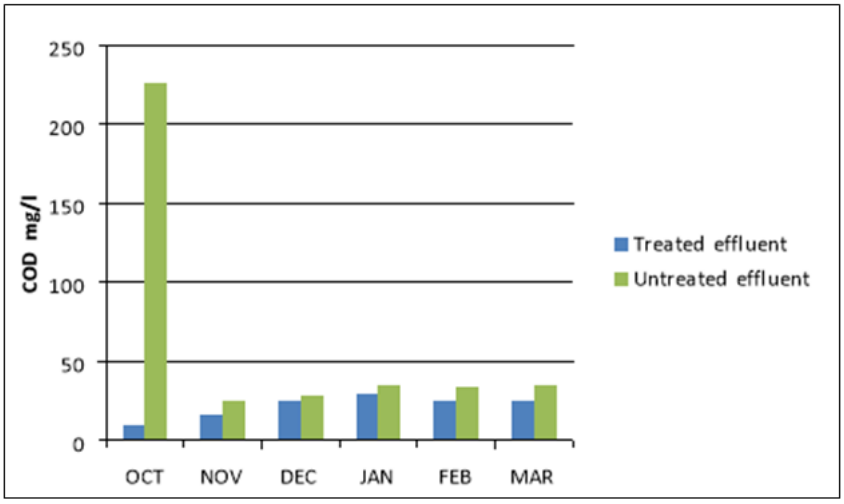


Fig. 9 Chemical Oxygen Demand Histogram

4.7 Total Dissolved Solids (TDS)

The total solid concentration in waste effluent represents the colloidal form and dissolved species. The probable reason for the fluctuation of value of total solid and subsequent the value of dissolved solids due to content collision of these colloidal particles. The rate of collision of aggregated process is also influenced by PH of these effluents. The TDS content in the effluent varied from 1837 to 1858 mg/l before treatment whereas after physical and biological treatment the values obtained were 1459 and 1229 mg/l respectively. The percentage reduction of 33% was achieved. The range of total dissolved of untreated pharmaceutics laboratory grey water 2-245 ppm and for treated waste water 78-110ppm % reduction in October the reduction was 68% and in remaining months the reduction was 30-36% (fig 10)

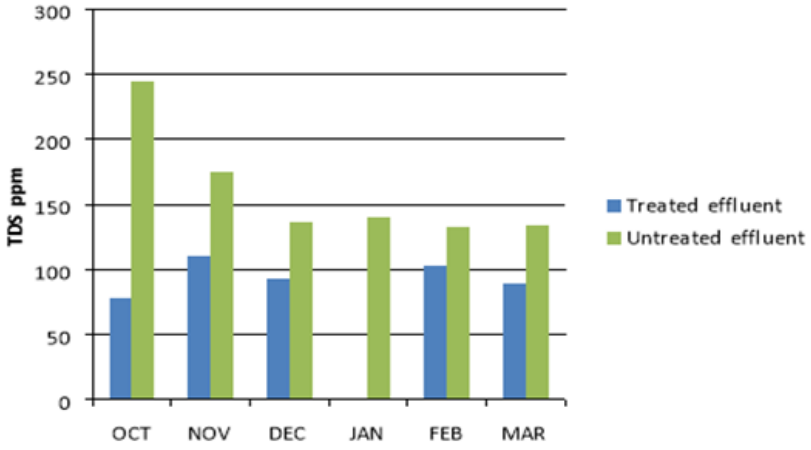


Fig. 10 Total Dissolved Solids Histogram

4.8 Total Suspended solids (TSS)

Determination of Total Suspended Solids (TSS) -Wet a filter paper with a small volume of distilled water, placed it in the oven and dry for 1 hour at 103°C. Then cooled and stored in Desiccator until needed. Weighted immediately before use for initial weight. Filtered a measured volume of well mixed sample through the filter paper by sucking with vacuum pump. Washed with three successive 10 ml volumes of distilled water, allowed complete drainage between washings. Carefully removed the filter paper from filtration apparatus and transferred to the oven for drying. Dried for 1 hour at 103°C in the oven. Cooled in a desiccator to balance temperature and weight. The range of total suspended solids of untreated pharmaceutics laboratory graywater 0.181-0.43gm and the range for treated pharmaceutics laboratory graywater 0.06-0.27 gm % reduction 66-37(Fig 12)

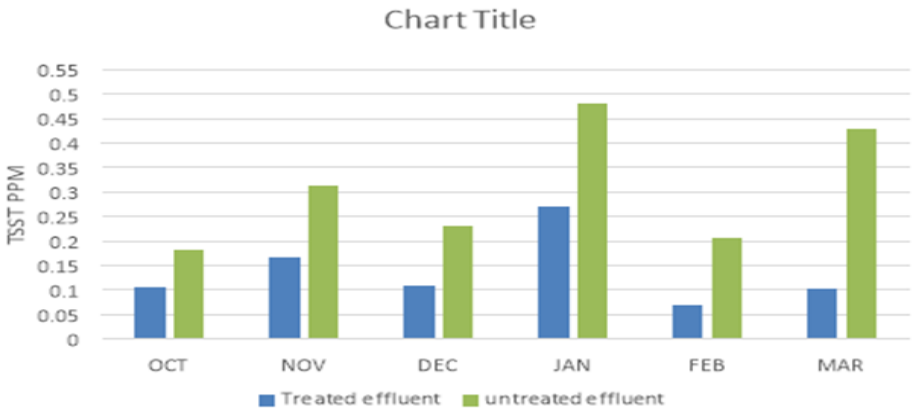


Fig. 11 Total Suspended Solids Histogram

4.9 Turbidity

It is a measure of the light - transmitting properties of water which indicates the quality of wastewater with respect to residual suspended and colloidal matter. Samples of feed and permeate of UF plant was collected to check turbidity using turbidity meter. Turbidity was completely removed by pretreatment with Ultrafiltration, turbidity of untreated pharmaceutics laboratory graywater range 545-800 NTU and treated 2 pharmaceutics laboratory graywater 90-720 NTU % reduction 42.78-10 (Fig 13)

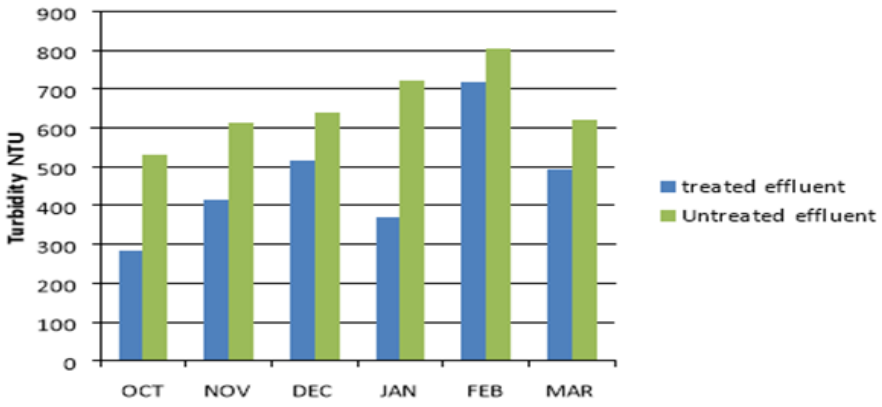
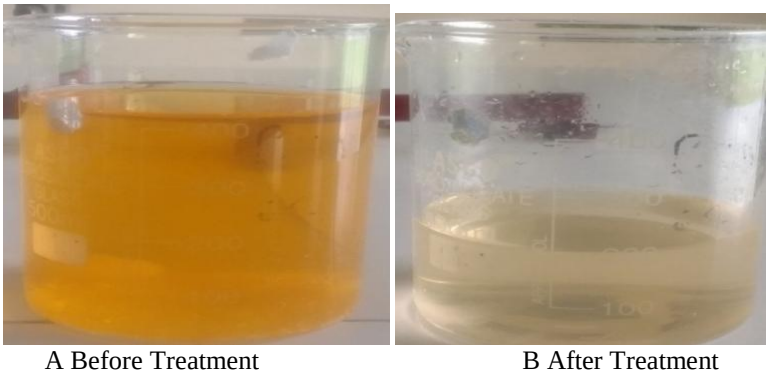


Fig. 12 Turbidity histogram



A Before Treatment B After Treatment

Fig. 13 Sample Example of Pharmaceutics Laboratory Graywater Before and After Treatment

4.10 Microbial Analysis

The water quality is directly related to health and is important for determination of water utility. Assessment of water quality is a critical factor for assessment of pollution levels. The results from the present study clearly pointed out that water obtained after treatment showed no or fewer colonies of microorganisms.

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