Reduction of Ammonia and Turbidity in Wastewater of Pharmaceutical Industry

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Abstract: ***Pharmaceutical industry is one of the major industries causing water pollution. An Attempt has been made in the present project to reduce the ammonia and Turbidity WWTP for Pharmaceutical industry. Different combinations of dosages of ferric alum and non-ferric alum with cationic and anionic polyelectrolyte of were used at Primary clarifier. It was found that there is substantial reduction in turbidity using 3 ppm non ferric alum and 0.35 ppm cationic polyelectrolyte dose. One more additional dose is applied at secondary clarifier of Non ferric Alum & Cationic Polyelectrolyte which effective. After sedimentation, Effluent passed through Sand and Carbon filter and Turbidity reduced from 86.5 NTU to 3.5 NTU. A new concept of ammonia removal from waste water solution by physical method such as, aeration, oxidation by sodium hypochlorite adsorption by carbon followed by ion exchanger zeolite was study in this work. In the first mode aeration was given to waste water sample and dissolved oxygen was increased at aeration stage. In the second mode absorbed ammonia removed by oxidation by sodium hypochlorite and adsorption by granular activated carbon. In the third mode zeolite was used for zeolite was used for removal of ammonia. Ion exchange is more competitive than the other methods because of its relative simplicity and its economy in application & operation. The Turbidity and Ammonia removal efficiency of WWTP were 96%, and 99 % respectively.***

Keywords: Adsorption, Coagulation, Flocculation, Ion exchange, oxidation, Sedimentation.

1. Introduction

The pharmaceutical industry employs various processes and a wide variety of raw materials to produce an array of final products and needed to fulfill national demands.

Now a day's world is becoming small village as new technologies are emerging every day. With increasing globalization the health problems of people are growing in faster rate this leads to growth of pharmaceutical industry. Pharmaceutical industry represents a range of industries with operation and processes as diverse as its product. Hence effluents coming from pharmaceutical industries vary from industry to industry. The wastewater from pharmaceutical manufacturing industries is problematic as it requires different treatment methods due to it's diverse nature". Additionally, as is the case in other industrial manufacturing sectors, water is a critical ingredient in pharmaceutical and chemical manufacturing operations; consistent and high-quality supplies are needed for a range of purposes including production, material processing, and cooling. As disruptions in raw water supply represent a significant concern, more companies are turning to water efficiency initiatives to help mitigate water scarcity-related risks.

Limited water resources and increasing industrialization require a more advanced technology to preserve water quality. One of the important factors affecting water quality is the enrichment of Turbidity and Ammonia in water bodies. Wastewater with high levels of Turbidity and Ammonia several problems, such as Corrosion Scaling to Utility Equipment. It is therefore, necessary to remove these substances from wastewaters to achieve the Zero liquid Discharge.

In Many of the effluent ammonia is present in low concentrations and the quantity of discharge may be low. However Ammonia-containing wastewater cannot be utilized because creates serious problems like corrosion on equipment if the treated water is reused.

NH4-N removal is one of the fundamental aims in wastewater treatment. It can be carried out by Physical, biological, chemical precipitation, Oxidation, ion exchange, air striping, and biological nitrification. Ammonia is generated using different types of molecules during the manufacturing of product that may content Nitrogen. The segregation, separation or replacement or substitution of this product is quiet difficult at the source.

There is scaling, deposition and corrosion on the Utility Equipment due to the high content of Ammonia. Higher Ammonia content will be the higher risk to breakdown of utility Equipment. The traditional method for ammonia removal from pharmaceutical waste water is based on biological treatment. As treated water recycle limit is stringent, ion exchange, adsorption and oxidation become more interesting as possible treatment methods. One ion exchanger with a high affinity for ammonia ion is clinoptilolite, a naturally occurring zeolite. Earliest studies have shown that clinoptilolite, and certain other natural zeolites can be effective in removing ammonia from waste water. Clinoptilolite is reported to have a classified aluminosilicate cage like structure and therefore exhibits significant macro porosity. Ammonia stripping refers a simple process utilized to lower the ammonia contact of waste water. Ammonia stripping is often easier and less expensive to conduct when removing nitrogen from waste water (in the form of ammonia)

Turbidity is the cloudiness or haziness of a fluid caused by large numbers of individual particles that are generally invisible to the naked eye, similar to smoke in air. The measurement of turbidity is a key test of water quality. Waste water can contain suspended solid matter consisting of particles of many different sizes. While some suspended material will be large enough and heavy enough to settle rapidly to the bottom of the container if a liquid sample is left to stand (the settable solids), very small particles will settle only very slowly or not at all if the sample is regularly agitated or the particles are colloidal. These small solid particles cause the liquid to appear turbid. In waste water, the higher the turbidity level, frequency of filter chocking frequently occurred. There is generation of Turbidity in the form of colloidal and suspended form. Removal of colloidal turbidity is one of the serious challenges in the Waste water treatment. Tablet powder is dissolved in waste water in the form of fine particle. These fine particles if not removed from waste water, will deposit on the membrane of RO. Various methods are available for determination of turbidity such as Physic chemical treatment -screening; Pre sedimentation; Coagulation; Flocculation, Sedimentation, Filtration Settling & Decanting.

Need of Technology of Waste Water Treatment:

Rapid industrialization has resulted in the rise of pollution. Developing low cost technology for wastewater treatment offers an alternative and has been found to be most effective for treatment of industrial wastewater. New Environment friendly, Cost effective Technology is required to reduce the concentration of Ammonia and Turbidity of waste water. This project deals with the treatment of Waste water generated by Pharmaceuticals Industry, mainly manufacturing plant of tablet and Research & Development activities. An objective of Study is to reduce the Ammonia and Turbidity through appropriate treatment processes so that maximum treated water can be recycled to Utility and Boiler. This project is giving feasible solution above two challenges.

2. Material and Method

Waste water treatment aims at the removal of unwanted components in waste waters. This can be achieved by using physical, chemical and biological means, either alone or in combination. Physical treatment methods such as screening, sedimentation, and skimming remove floating objects, grit.

Chemical treatment methods such as precipitation, pH adjustment, coagulation, oxidation, and reduction and colloidal impurities. Tertiary treatment methods are used for further purification and for reuse of treated wastewater for various purposes. Dissolved organics are removed by biological.

Monitoring of WWTP and its performance evaluation:

Samples were collected from WWTP at different. Sampling points of WWTP and characterize for parameters BOD, COD, pH, TSS, TDS, Turbidity and Ammonia etc. Samples were collected and analyzed for the parameters shown in Table. Samples for BOD, COD, Turbidity and Ammonia etc. were analyzed in accordance with the procedure laid down in Standard Methods for the Examination of Water and Wastewater (APHA 1996)

Sampling procedures

The reliability of the results of analysis of waste Water samples depend up on the proper collection of a true representative sample from a large volume of waste water. The sample after collection was transported to the laboratory in a preserved condition. So that it will represent fairly and accurately conditions when the sample was collected.

Sampling Schedule and frequency

Composite samples were collected. Samples comprising of Equalization tank, Primary settling tank, Aeration tank, Secondary settling tank, Sand and Carbon filter, Oxidation treatment by Sodium Hypochlorite and Ion exchange treatment. Samples for BOD, COD, TDS, Turbidity and Ammonia etc were analyzed in accordance with the procedure laid down in Standard Methods for the Examination of Water and Wastewater (APHA 1996)

Methods for Removal of Turbidity

Turbidity is mainly in two from colloidal and suspended. Suspended form Turbidity can be removed easily by physical method such as screening, simple filtration. Colloidal Turbidity removal is challenge in Pharmaceutical industry as tablets are dissolved in water resulted into colloidal particle as well as suspended particle. Waste water content particle's size below 5 micron. It is too difficult to remove the fine particle from filtration system. This study involve to use diffident type of coagulant and flocculants with different type of concentrations dosage and sedimentation followed by Duel media flirtation i.e. Sand and carbon filter. Two stage chemical dosing used to reduce the turbidity. At the initial stage to equalization and second stage to secondary clarifiers. Usually there is only single Chemical treatment after the equalization.

Methods for Removal of Ammonia

Ammonia is one of the impurities where much more attention is not provided. It is substantial hazard having corrosive property in built. It has an adverse impact on the performance of specially Utility Equipment. Some ppm levels of ammonia have potential to have scaling and corrosion inside the equipment.

The study involves the identification of ammonia generating stream, to check the feasibility of Segregations of the ammonia stream with feasible solution of treatment method, Air striping to Equalization and Aeration effluent. Oxidation by Sodium Hypochlorite using different concentration dose. Adsorption of Ammonia by Dual media filer using different type of Granular Activated carbon (GAC) of different Iodine value. Study also involves using Ion exchange of clinoptilolite to reduce the ammonia.

3. Results and Discussion

Industry is manufacturing Tablet, Injectable and Central Research and Development. The waste water generated from following some streams.

Table 3: Sources of the effluent:

Stream - 1:	Effluent is from manufacturing, Ware House and Quality Control
Stream - 2:	Effluent is from Utility and Boiler House
Stream - 3:	Effluent is from R & D
Stream - 4:	Effluent is from manufacturing Unit III facility
Stream - 5:	Effluent is from Washrooms and Toilets

Effluent characteristics

The physico-chemical characterization of effluent from of individual stream is given in Table - 1. The key pollutants in the wastewater from pharmaceutical based formulation industry are inorganic compounds and suspended solids.

Table 3.1: Analysis report of stream 1 to 5- (Average Values)

Parameters	Stream 1 (Warehouse and QC)	Stream 2 (Cooling Tower & Boiler)	Stream 3 (R&D)	Stream 4 (Unit III)	Stream 5 (Washrooms & Toilet)	Mean
pH	7.4	7.7	8.2	7.7	7.8	7.76
TSS	500	220	123	180	200	244.6
TDS	1667	1193	1267	3453	605	1637
COD	748	300	4043	411	340	1168.4
BOD	127	70	740	94	30	212.2
Turbidity	95	91	63	72	27	89.6
Ammonia	0	0	48	0	72	60

(All values expressed in tables are in mg/ltr. --Except Turbidity & pH)

The Table 3.1 indicates that the ammonia concentration is present in Stream 3 (R&D) and Stream 5 (Washrooms, Toilet). It was advisable to segregate the Stream No--5 containing high Ammonia and which was also feasible solution. It was recommended to have sewage treatment facility where Stream No 5 effluent would be treated. The effluent from equalization tank, Primary settling tank and Secondary Settling tanks were analyzed.

Table 3.2: Analysis report- Before the mix up of Sewage stream and after the segregation of the Sewage Stream

Parameters	Before Segregation of Sewage stream			After Segregation Sewage stream		
	E	P	S	E	P	S
pH	7.5	7.5	7.4	7.6	7.5	7.5
TSS	515	320	140	520.7	335	150
TDS	1900	1880	1430	1898.3	1862	1392
COD	824	500	324	834.0	512	315
BOD	140	60	45	145.3	65	38
Turbidity	86.5	45	35	89.3	47	32
Ammonia	70	40	30	50	40	25

(All values expressed in tables are in mg/ltr. (Except Turbidity & pH)

Note E= Equalization Tank, P = Primary Settling Tank S= Secondary Settling Tank

After segregation of Stream No -5 which was generated by Washrooms and Toilets, it was found that Initial Ammonia content was 70 ppm with mix- up of all five stream and after segregation of Stream -5, it was come down to 50 ppm at the stage of Equalization without any chemical treatment.

Air Stripping

Ammonia stripping refers to a simple process utilized to lower the ammonia content of a wastewater stream

Table- 3.3: Analysis report- Air Stripping at the Equalization stage

Parameters (mg/l)	Before Air stripping Sample from Equalization Tank				After Air stripping Sample from Equalization Tank			
	S1	S 2	S 3	S.D.	S1	S 2	S 3	S.D.
Ammonia	50	45	55	5.0	40	30	35	5.0

Note- Sample 1=S1, Sample 2=S2, Sample 3=S3 S.D.- Standard Deviation

By doing the Air stripping, the initial Ammonia 50 ppm and after air stripping it was decreased to 35 ppm.

Increases in DO:

Sample from Secondary Settling Tank approx. 5 ltr is taken, dissolved oxygen of the consecutive 3 samples is checked, which is mentioned in below table. Then DO was increased up to 5 ppm level. The ammonia content was analyzed.

Table 3.4: Analysis report- Increase in DO at the Aeration stage

Parameters (mg/l)	Before increase in DO in Secondary Settling Tank					After increase in DO Secondary Settling Tank				
	S1	S2	S3	S.D.	Avg.	S1	S2	S3	S.D	Avg.
DO	1.5	1.5	1.6	0.06	1.5	5	4.8	5	0.12	4.9
Ammonia	25	20	22	2.52	22.3	15	18	15	1.73	16

Note- Sample 1=S1, Sample 2=S2, Sample 3=S3

From Table 3.4, it was observed that initial ammonia content was 22.3 ppm average and after increasing the DO in secondary settling tank, it was reduced to 16 ppm. There was a 28 % reduction in Secondary settling tank.

Reduction of ammonia by oxidation method using Sodium Hypochlorite process.

Samples from Secondary Settling Tank were taken and Sodium Hypochlorite doses were given. The results obtained were tabulated in below Table 4.6 Oxidation by Sodium Hypochlorite after secondary settling tank. The Sodium Hypochlorite was added in doses form of 0.2 ppm, 0.25 ppm, 0.28 ppm and 0.3 ppm

Table 3.5-Analysis report using Sodium Hypochlorite dose

Sodium Hypochlorite dose (ppm)	*Ammonia content(ppm)*
0.2	12
0.25	10
0.28	08
0.3	06

There was reduction in Ammonia from 12 ppm to 6 ppm by oxidation using Sodium Hypochlorite (Table 3.5)

Adsorption method: Reduction of ammonia by using Granular activated charcoal:

The bed of Granular Activated carbon having Iodine value of 900, 1000 and 1100 are prepared Sample from after oxidation by hypochlorite was poured on the three beds. Results were mentioned below table.Carbon adsorption has numerous applications in removing pollutant from water stream.

Stage 1 the effluent after treatment by Sodium Hypochlorite passed through to Carbon bed of 900 Iodine Value

Table 3.6: Analysis report- Passing through carbon bed

Iodine value	Ammonia content after passing through carbon bed (ppm)
900	5
1000	4
1100	3.5

Reduction of ammonia by using Ion Exchange

Waste water containing ammonia to be treated is passed through zeolite. A series of experiments was conducted to examine the effects of solution adsorbent dosage. The effluent after treatment by 0.3 ppm Sodium Hypochlorite and passed through to Carbon bed of 1100 Iodine Value is subjected to Ion Exchange treatment and following results observed.

Table 3.7: Analysis report- Sodium Hypochlorite dose 0.3 ppm, using carbon bed of Iodine value 1100 and passing through Natural Zeolite by Ion exchange

S1	S2	S3	S4	S5	S6	Mean	S.D.
0.17	0.18	0.2	0.17	0.20	0.17	0.17	0.01

Note- Sample 1=S1, Sample 2=S2, Sample 3=S3, Sample 4=S4, Sample 5=S5, sample6=S6

The desired ammonia content in treated effluent achieved by taking the following measures

a. Segregation of effluent stream containing ammonia
b. Air stripping in equalization tank.
c. Increase in DO in Aeration tank
d. Oxidation- by Sodium hypochlorite solution (0.3 ppm)
e. Adsorption through carbon bed of Iodine value 1100.
f. Ion exchange.

There is 99 % reduction in ammonia content of Waste water due to applying the above methods.

Study on Turbidity Removal:

Stage 1: Coagulant agent - Ferric Alum and Flocculent agent – Cationic Polyelectrolyte

It is found from table 4.8 that using ferric alum 3.0 ppm and cationic Polyelectrolyte 0.35 ppm turbidity reduced to 11.3 NTU.

Stage 2: Coagulant agent - Ferric Alum and Flocculent agent – Anionic Polyelectrolyte

From Table 4.8 reveals that using ferric alum 3.0 ppm and Anionic Polyelectrolyte 0.35 ppm turbidity reduced to 10.7 NTU.

Stage 3: Coagulant agent – Non Ferric Alum and Flocculent agent – Anionic Polyelectrolyte

Initial Turbidity was 25.3 NTU and using Maximum 3 PPM dose of Non ferric Alum, it was reduced to 10.7 NTU.

Stage 4: Coagulant agent – Non Ferric Alum and Flocculent agent – Cationic Polyelectrolyte

It is found from table 4.8 that using ferric alum 3.0 ppm and cationic Polyelectrolyte 0.35 ppm turbidity reduced to 8 NTU.

Table 3.8: Analysis report- Stage 1 to Stage 4 experiments

Stage-1			Stage-2			Stage-3			Stage-4		
Ferric Alum Dose (ppm)	Cationic Polyelectrolyte (ppm)	Turbidity (NTU)	Ferric Alum Dose (ppm)	Anionic Polyelectrolyte (ppm)	Turbidity (NTU)	Non Ferric Alum Dose (ppm)	Anionic Polyelectrolyte (ppm)	Turbidity (NTU)	Non Ferric Alum Dose (ppm)	Cationic Polyelectrolyte (ppm)	Turbidity (NTU)
1.5	0.2	29.7	1.5	0.2	23.7	1.5	0.2	25.3	1.5	0.2	19.7
2.0	0.25	22.3	2.0	0.25	17.3	2.0	0.25	19.3	2.0	0.25	12.3
2.5	0.3	16.3	2.5	0.3	13.0	2.5	0.3	15.7	2.5	0.3	9.0
3.0	0.35	11.3	3.0	0.35	10.7	3.0	0.35	10.7	3.0	0.35	8

By using Non ferric alum and cationic polyelectrolyte with different concentrations the turbidity reduced upto 8 NTU then the sample after Secondary clarifier tank was taken and jar test was carried out with following doses Dose of Non Ferric Alum used for experiment are 0.5 ppm, 1 ppm, 1.5 ppm and 2 ppm with cationic polyelectrolyte are 0.1 ppm, 0.15 ppm, 0.2 ppm and 0.25 ppm In these experiments samples were taken from outlet of Secondary Settling Tank and proper settling is given and then the samples were passed through Sand and Carbon filter.

Results of the experiment are tabulated in following table (3.9)

Stage 5: Coagulant agent – Non Ferric Alum and Flocculent agent – Cationic Polyelectrolyte at Secondary clarifier.

Table 3.9: Analysis report-Non Ferric alum doses and cationic Polyelectrolyte at the secondary clarifier. Sample analyzed after passing through Sand & Carbon filter

Non Ferric Alum Dose (ppm)	Cationic Polyelectroly (ppm)	Turbidity (NTU)
0.5	0.1	20
1	0.15	17
1.5	0.2	8
2	0.25	3.5

Additional treatment of non ferric alum and cationic polyelectrolyte was given to the secondary clarifier and turbidity reduced 3.5 NTU of giving 2 ppm Non ferric alum dose and 0.25 ppm of cationic polyelectrolyte dose Table 3.9

The desired turbidity is in treated effluent achieved by taking the following measures

a. Physico-chemical treatment of effluent by using 3 ppm dose of non ferric alum and 0.35 dose of cationic polyelectrolyte in primary clarifier. Initial Turbidity was 86.5 NTU and it was reduced to 8 NTU
b. Additional treatment is given to Secondary settling tank by using 2 ppm dose of non ferric alum and 0.25 dose of cationic polyelectrolyte with proper sedimentation and turbidity reduced up to 3.5 NTU. The limit is 5 NTU for RO water feed.

4. Discussion

As per the study carried out and analysis done for the reduction of Ammonia – () and Turbidity by various method for reducing the Ammonia and Turbidity following results were summarized below.

Ammonia:

- There are five stream of waste water. Stream wise analysis was carried out.
- Average analysis was presented in Table 3.1.It shows that the Stream 3 &-5 was having Ammonia 48 ppm and 72 ppm respectively.Other streams generated from manufacturing and Utility are not having ammonia.
- It is advisable to segregate the Stream No--5 containing high Ammonia and which was also feasible solution to the industry.
- Analysis of Ammonia stream 1 to 5 (Mix-stream) and Stream 1 to 4 (After segregation of sewage) was carried out (Table 3.2). At equalization tank Ammonia was 70 ppm and after segregation of sewage it was reduced to 50 ppm.
- Air striping was done at the equalization sample.Initial Ammonia was 50 ppm and after air stripping it was reduced to 35 ppm. (Table 3.3).
- The next method used was, increase in dissolved oxygen at the aeration stage. In this method Sample from Secondary Settling tank was taken and dissolved oxygen of the samples were checked. DO was increased up to 5 ppm level. The ammonia content was analyzed. It was observed that initial ammonia content was 22.3 ppm average and after increasing the DO in secondary settling tank sample, it was reduced to 16 ppm.
- Oxidation by method Sodium Hypochlorite was used to reduce the ammonia. (Table 3.6).There was reduction in Ammonia from 12 ppm to 6 ppm.
- Adsorption by using Granular activated charcoal was done. (Table 3.7) Initial ammonia was 5 ppm and by passing through Granular activated Carbon (GAC) bed, it reduced to 3.5 ppm.
- Ion Exchange – Natural Zeolite was used to bring down the ammonia. Ammonia was 3.5 ppm. Using Ion exchange Bed, it was reduced to 0.17 ppm. (Table 3.8). Industry has internal limit of the Ammonia is <0.5 ppm.

By using segregation of Sewage stream, Air stripping, increasing in Do in aeration stage, Oxidation- by Sodium hypochlorite solution, Adsorption through carbon bed of Iodine value 1100 and Ion exchange by Natural Zeolite, there was reduction of ammonia 70 ppm to 0.17 ppm.

Turbidity:

- There are five stream of waste water. Stream wise analysis was carried out (Table 4.1)
- Stream No 1 was having turbidity -95 NTU, Stream No -2 was having turbidity- 91 NTU, Stream No-3 was having turbidity -63 NTU, Stream No-4 was having turbidity -72 NTU, Stream No-5 was having turbidity -27 NTU (Table3.1)
- For reducing the turbidity different doses of coagulating and flocculating agents were used. Different combinations of dosages of ferric alum and non-ferric alum (1.5 ppm, 2 ppm, 2.5 ppm and 3 ppm) with cationic and anionic polyelectrolyte of (0.2 ppm, 0.25 ppm, 0.3 ppm and 0.35 ppm) were used to reduce the Turbidity.
- It was found reduction in turbidity using 3 ppm non ferric alum and 0.35 ppm cationic polyelectrolyte dose. (Table 3.8).Turbidity reduced from 8.0 NTU.
- Additional one more treatment introduced at secondary clarifier stage to reduce the turbidity.
- Doses of Non Ferric Alum - 0.5 ppm, 1 ppm, 1.5 ppm and 2 ppm with cationic polyelectrolyte 0.1 ppm, 0.15 ppm, 0.2 ppm and 0.25 ppm were used. In these experiments samples were taken from outlet of Secondary Settling Tank and proper settling (sedimentation) was given. Effluent then passed through Sand and Carbon filter. (Table 3.9).
- The Dose of 2.0 ppm Non ferric Alum and 0.25 ppm Cationic Polyelectrolyte was found effective and turbidity reduced to 3.5 NTU. Turbidity reduced from 86.5 NTU to 3.5 NTU using Chemical treatment at two stages-(Equalization and Secondary clarifier) such as coagulation and flocculation by the combinations of Non Ferric Alum and Cationic Polyelectrolyte and sedimentation. The industry is having 5 NTU limits for RO water feed which is achieved by above study.

5. Conclusion

The present study is aimed to reduce the ammonia and turbidity of waste water and ensure the feed quality of RO plant. This study demonstrates the field operation of pharmaceutical waste water treatments by physicochemical treatment followed by oxidation, adsorption & ion exchange. It was demonstrated that coagulation /flocculation at two stage is capable of removing turbidity and air stripping, oxidation, adsorption followed by ion exchange capable of removing ammonia. The results indicate that above system has great potential in treating the waste water containing ammonia and turbidity. It is possible to reuse or recycle entire effluent to Utility and Boiler by applying above Pre-treatment technologies for removal of Ammonia and Turbidity of waste water in pharmaceuticals industry.

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