SEGREGATION, CHARACTERIZATION AND TREATMENT FOR RECYCLING OF WASTEWATER GENERATED FROM DYES MANUFACTURING INDUSTRIES, DYE INTERMEDIATES MANUFACTURING INDUSTRIES AND TEXTILE DEING AND PRINTING INDUSTRIES OF SACHIN INDUSTRIAL AREA, GUJARAT, INDIA.

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ABSTRACT

In the present study the characteristics of segregated untreated wastewater generated from dyes and dye intermediates manufacturing industries and textile dyeing and printing industries were studied. Wastewater from 10 units manufacturing different type of dyes and dye intermediates and 10 units involved in textile dyeing and printing, was used for this study. The study shows that the wastewater generated from dyes and dye intermediates manufacturing industries can be segregated at source in two streams concentrated stream and dilute stream. Wastewater was analyzed for pH, Chemical Oxygen Demand (COD), Biochemical Oxygen Demand (BOD), Total Dissolved Solids (TDS), Suspended Solids (SS), Chloride, Ammonical Nitrogen, Phenolic Compounds, Hexavalent Chromium, Total Chromium, Nickel, Oil & Grease parameters The study confirms that it is possible to recycle effluent and the treated effluent can be reused for different manufacturing stages, which ultimately reduces the waste water discharges and ultimately helps to conserve precious fresh water resources.

In addition to that, concentrated stream can be used to manufacture spent acid and ferrous sulphate, which are potential sources of income for such centralized facility. Entire utility waste water (i.e. waste water generated from boiler, cooling tower) generated from dye intermediate manufacturing industry can be mixed with textile dyeing and printing industry effluent and dyes manufacturing industry effluent and can be treated in conventional CETP, which is operational at Sachin industrial area.

Key Words: Dye Industries, Segregation of wastewater, Manufacturing process, concentrated stream, dilute stream, spent acid, ferrous sulphate, recycling.

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INTRODUCTION

We cannot imagine life without colour. Colour can be imparted to the particular object by using various types of dyes. The dye may be natural or synthetic origin. Typically, the dye material is put in a pot of water and then the textile fibers/yarns to be dyed are added to the pot, which is heated and stirred until the colour is transferred. The discovery of man-made synthetic dyes in the mid-19th century triggered the end of the large-scale market for natural dyes. Synthetic dyes, which could be produced in large quantities, quickly superseded natural dyes for the commercial textile production enabled by the industrial revolution. Most of the industries use dyes and pigments to colour their products. Among various industries the biggest consumers of dyes are textile, tannery and paper industry. Similarly, dyeing and printing industries uses various types of dyes to impart colour on fabric. The textile industry utilizes large volumes of water in its wet processing operation, and thereby, generates substantial quantities of wastewater[1-2]. While manufacturing dyes and dye intermediates, wastewater is generated in significant amount. This is important point, since wastewater is the principle route by which dyestuff enters into the environment. For example, of the 450,000 tons of dyestuffs produced in 1978, 41,000 tons were lost to waste streams from textile processing operations and 9000 tons constitute the estimated loss from dye manufacturing facilities[3]. As far as the cost parameters is concerned, it is beyond question that avoiding, or at least minimizing, and reusing wastewater is preferable to wastewater treatment, as direct or indirect discharge of wastewater certainly changes the water quality of the receiving body[4].

AIMS & OBJECTIVES

Present study is taken up on the dyes manufacturing units, dye intermediates manufacturing units and textile dyeing and printing units. This study has been undertaken to understand the pollution problems faced by the industries manufacturing dyes, dye intermediates and dyeing and printing units of Sachin industrial area and to propose suitable solutions.

MATERIAL AND METHODS

Wastewater from 10 units manufacturing different type of dyes and dye intermediates and 10 units involved in textile dyeing and printing, was used for this study. The production capacity of dyes and dye intermediates manufacturing units is about 6 MT/Month to 125 MT/Month depending upon the capacity of their installed machineries. These units work for about 330 to 340 days per year. While, the textile processing units of Sachin area have an average production capacity of about 40,000 to 1,00,000 meters per day depending upon the capacity of their installed machineries. The raw materials required for manufacturing of dyes and dye intermediates are oleum, sulphuric acid, hydrochloric acid, α - naphthol, β-naphtol, caustic soda, soda ash,sodium nitrite, liquor ammonia, ammonium sulphate, benzene and its derivatives like phenol, aniline, chlorobenzene etc. While the necessary raw material for textile dyeing and printing units is man made art silk (polyester) cloth commonly named as "grey". This is obtained from the weavers. The job work is done for dyeing and printing of this grey cloth. Other raw materials required for the processing of art silk cloth are dyes, caustic soda, printing gum, acetic acid, sodium hydrosulphite and other auxiliaries. The quantity of water consumed per day for dye and dyes intermediate industries varies from 8 KL /day to 40 KL/day. While, the water consumed per day for textile dyeing and printing units varies from 880 KL/day to 2200 KL/day. The amount of wastewater generated by dyes and dye intermediate industry ranges from 6 KL/day to 36 KL/day, while the amount of wastewater generated by textile dyeing and printing units ranges from 780 to 1950 KL/day.

Source of samples and frequency of sample collection and detailed study of manufacturing process

Initially a study was carried out, to identify initial details of industries like their production capacity, source of water, water consumption, present wastewater treatment and disposal scheme. In this study, it was found that the present wastewater treatment scheme is resulting in the generation of high volume of sludge. Industries were facing problems in disposal of this sludge. After that, manufacturing process was studied in detail and sources of wastewater generation at various stages of manufacturing process were identified. This study was carried out to identify wastewater streams which are having similar characteristics, so that wastewater having similar characteristics can be segregated from different industries and then mixed, and can be treated effectively. It was found that wastewater was mixed by the industries from all the wastewater sources at one place, which makes it difficult to establish effectiveness in wastewater treatment.

Table -1 Details – type of industries and their products

Industry	**Type of Industry**	**Products / Process**
Industry – 1	Dye intermediate manufacturing	Schaeffer’s Acid, K Acid, Sulfo Tobias Acid
Industry – 2	Dye intermediate manufacturing	G Salt, Sulfo Tobias Acid
Industry – 3	Dye intermediate manufacturing	Schaeffer’s Acid, G Salt
Industry – 4	Dye intermediate manufacturing	G Salt, Amino G Salt, K Acid
Industry – 5	Dye intermediate manufacturing	Sulfo Tobias Acid, Schaeffer’s Acid
Industry – 6	Dye intermediate manufacturing	Aniline 2:5 di sulphonic acid, Sulfo Tobias Acid
Industry – 7	Dyes manufacturing	Acid Dyes, Direct dyes
Industry – 8	Dyes manufacturing	Acid Dyes, Solvent dyes

Industry – 9	Dyes manufacturing	Solvent Dyes
Industry – 10	Dyes manufacturing	Reactive Dyes, Direct Dyes
Industry – 11	Textile	Dyeing
Industry – 12	Textile	Dyeing and printing
Industry – 13	Textile	Dyeing and printing
Industry – 14	Textile	Dyeing
Industry – 15	Textile	Dyeing
Industry – 16	Textile	Dyeing
Industry – 17	Textile	Dyeing
Industry – 18	Textile	Dyeing and printing
Industry – 19	Textile	Dyeing and printing
Industry – 20	Textile	Dyeing

The untreated wastewater samples from each industry were collected three times a year during year 2010. Segregated wastewater samples were collected three times a year during year 2011 to identify characteristics of segregated wastewater. For the study of characterization and treatment of segregated wastewater, samples were collected during year 2012. The characteristic parameters of the polluted wastewater were determined according to the standard procedures, described in the standard methods, i.e APHA and IS standards[5-6]. Samples were preserved after sampling as tabulated below:

Table -2 Wastewater sample parameters with preservation methods

Sr. No.	**Parameters**	**Method for sample preservation**
1	pH	Measure within 0 to 4 hours
2	Chemical Oxygen Demand (COD)	Add H_2SO_4 (1:1) to pH 2, and refrigerate at 4 ^{0}C.
3	Biochemcial Oxygen Demand (BOD)	Store at 4 ^{0}C temperature, analyze as soon as possible
4	Total Dissolved Solids (TDS)	Store the sample at 4 ^{0}C.
5	Total Suspended Solids (TSS)	Store the sample at 4 ^{0}C.
6	Chloride	Store the sample at 4 ^{0}C.
7	Ammonical Nitrogen	Store the sample at 4 ^{0}C.
8	Phenolic Compounds	Acidify with H_3PO_4 to pH 4, and add 1 gm $CuSO_4.5H_2O$ per liter of sample
9	Hexavalent Chromium	Acidify with HNO_3 to pH 2, store at 4 ^{0}C.

10	Total Chromium	Acidify with HNO_3 to pH 2, store at 4 ^{0}C.
11	Nickel	Acidify with HNO_3 to pH 2, store at 4 ^{0}C.
12	Oil and Grease	Add 5 ml H_2SO_4 (1:1) per liter of sample.

Wastewater samples were collected from equalization tanks of selected industries. Each sample was analyzed for the parameters listed in Table-2 above. The analysis results for dyes and dye intermediate industries show that pH value ranges from 1.04 to 5.12, COD value ranges from 35200 to 88250 mg/L, BOD value ranges from 6650 to 16700 mg/L, TDS value ranges from 95500 to 180240 mg/L, TSS value ranges from 6950 to 14800 mg/L, Chloride values ranges from 25500 to 150000 mg/L, Ammoniacal nitrogen ranges from 52 to 180 mg/L, Phenolic compound ranges from 47 to 80 mg/L, Hexavalent Chromium value ranges from 0.012 to 0.204 mg/L, Total chromium value ranges from 0.048 to 4.258 mg/L, Oil and grease value ranges from 52 to 205 mg/L, Nickel value ranges from 0.01 to 0.02 mg/L, while the results for textile dyeing and printing industries show that pH values for the selected industries ranges from 7.24 to 8.92, COD values range from 802 to 1002 mg/L, BOD values range from 310 to 510 mg/L, TDS values range from 4750 to 7500 mg/L, TSS values range from 102 to 195 mg/L, Chloride values range from 2850 to 4100 mg/L, Ammoniacal nitrogen range from 82 to 105 mg/L, Phenolic compounds range from 0.25 to 0.57 mg/L. Hexavalent Chromium values range from 0.024 to 0.104 mg/L, Total chromium values range from 0.092 to 0.212 mg/L, Oil and grease values range from 9 to 21 mg/L, Nickel was ranging from ND to 0.017 in wastewater. It is observed that wastewater collected from dyes and dye intermediate manufacturing industries have high COD, BOD, TDS, oil and grease and phenolic compounds, the wastewater also contains very low amount of heavy metals like chromium and nickel, the wastewater collected from the textile dyeing and printing industries also having high COD, BOD and TDS values but the values of the same are very low as compared to the dyes and dye intermediate manufacturing industries' wastewater. On the other hand the quantity of wastewater generated from the textile dyeing printing industries is very high as compared to the dyes and dye intermediates manufacturing industries.

There are two Common Effluent Treatment Plants (CETPs) for Sachin GIDC area. All industries selected for this study are member of either of these CETPs, as one CETP receives wastewater from only textile dyeing and printing industries, while another CETP receives wastewater from dyes and dye intermediate manufacturing industries[7]. Members of the dyes and dye intermediate CETP are sending their wastewater after providing primary treatment[8-11] (i.e. bringing pH value of the wastewater to Neutral range).It was observed that large amount of sludge was generated during neutralization of the wastewater. Both of the CETPs are located nearby each other in Sachin

Industrial area. Wastewater was collected during 3 times a year from equalization tank of dyes and dye intermediate industries and mixed in a pre-calculated ratio. 1L of this combined and equalized wastewater was given primary treatment with 10% solution of hydrated lime and 5% solution of Ferrous Sulphate. pH of wastewater was raised to 11 to 12 with lime solution and neutralized to bring 7 to 8 pH value with Ferrous Sulphate solution. This treatment generates chemical sludge which was filtered by a filter cloth and kept in an oven for drying at 70 ^{o}C for 24 hours. After drying this sludge was collected and weighed. The treated wastewater was analyzed for the same parameters as of untreated wastewater. The volume of sludge generated was found in the range of 482 to 502 mg/L.

Water resources are drastically reducing and the discharge norms are getting stringent day by day, government agencies like MoEF and CPCB are encouraging industries to reduce wastewater either by recycling or any other process changes. Considering the quality and quantity of wastewater generated from the industries located in the GIDC, Sachin, it was decided to carry out a study which can help industries to plan a recycling based treatment system at Sachin GIDC. After studying manufacturing process of dye intermediates, manufactured at industrial unit of Sachin GIDC, it was found that four stages are involved in the process of dye intermediate manufacturing these are: 1) Sulphonation, 2) Drawing 3) Filtration and 4) Centrifuge. The wastewater generated is from 1) Filtration and 2) Centrifuge. The manufacturing process also indicated that during manufacturing of all products involves sulphonation but Schaeffer's Acid, G Salt and Amino G Salt products requires either ammonia liquor or caustic flakes during isolation stages, so the wastewater generated from filtration and centrifuge stage is mild acidic streams (designated as Stream -1), while K –Acid, Sulfo Tobias Acid and Aniline 2:5 Di Sulfonic acid are isolated by pouring reaction mass on Ice bath, therefore the wastewater stream from these products is resulting in highly acidic stream at the centrifuge stage (designated as Stream -2). Therefore it was decided to collect wastewater streams generated from each product, regardless to manufacture at same industry or not.

Wastewater samples of Stream-1 and Stream -2 were collected from Filtration stage and Centrifuge stages from the plant based on product manufactured from all industries and then it was mixed stream wise, based on ratio identified from manufacturing capacity and wastewater quantity generated from each industry. Manufacturing process of dyes produced by industrial unit was studied. It was found that the dyes manufactured are Azo dyes and which is produced by coupling of dye intermediates so, Wastewater was generated from filtration / centrifuge stages of dye manufacturing process, which is indicated as Stream -3. However, from all industries (dyes intermediate, dyes manufacturing industries, textile dyeing and printing industries) wastewater is also generated from utilities like boiler, cooling tower and while washing of reactors, spillages etc., this stream was collected from each industries and was mixed based on ratio identified from manufacturing capacity and wastewater quantity of each industry, which is indicated as Stream-4.

From textile dyeing and printing industry samples were collected from main wastewater collected tank where wastewater from entire manufacturing plant is received, this stream is indicated as Stream -5.

Table -3 Industry wise products

Stream No.	Collected from Industry	Products / Stages
Stream -1	Industry -1, Industry -2, Industry – 3, Industry – 4, Industry – 5	Schaeffer’s Acid, G Salt, Amino G Salt
Stream -2	Industry - 1, Industry – 2, Industry – 4, Industry – 5, Industry – 6	K –Acid, Sulfo Tobias Acid, Aniline 2:5 Di Sulfonic acid
Stream -3	Industry – 7 to 10 (i.e. Dyes manufacturing industries)	Filtration and Centrifuge stage
Stream -4	Industry -1 to 10 (i.e. Dye intermediates manufacturing industries)	Utility section such as boiler, cooling and washing
Stream -5	Industry -11 to 20 (i.e. Textile dyeing and printing industries)	Main wastewater collection tank.

Therefore, the identified streams can be summaries as below: Stream -1 Wastewater sample collected from filtration and centrifuge stage from dye intermediate manufacturing industries which manufacture either of Schaeffer’s Acid, G Salt , Amino G Salt. Stream -2 Wastewater sample collected from filtration and centrifuge stage from dye intermediate manufacturing industries which manufacture either of K –Acid, Sulfo Tobias Acid, Aniline 2:5 Di Sulfonic acid. Stream -3 Wastewater samples collected from filtration and centrifuge stage from dyes manufacturing industries. Stream -4 Wastewater samples collected from boiler, cooling and washing streams from dyes and dye intermediate manufacturing industries. Stream -5 Wastewater samples collected from textile dyeing and printing industries.

Treatment of segregated wastewater:

It was observed that during stream wise segregation wastewater characteristics were almost identical during a whole year, as the streams for segregation was identified after studying the products manufactured by each selected industries. The manufacturing process of each product and the possible characteristics of the wastewater generated from each industry were also studied.

Considering above facts, following were decided:

a) The wastewater of Stream -1 is acidic in nature and also as evident from COD and BOD values it also contains dissolved organic material. Therefore, it was decided that this wastewater stream needs to be evaporated, this will separate dissolved organic matter and the condensate water can be recycled back. Following method was used to evaporate wastewater Stream -1. 0.5 L of Stream -1 wastewater sample was taken in laboratory scale distillation flask of 1L capacity. Water condenser was attached with this distillation flask. The distillation flask was heated with caution and condensate water was collected in previously washed and dried beaker of 1 L capacity. The distillation flask was heated till complete evaporation of the residue. The condensate water thus collected was measured for volume collected. The distillation flask was cooled and little amount of distilled water was added to dissolve solid residue remained in the flask. This distilled water was transferred into previously weighed beaker. This beaker was put on water bath to facilitate evaporation of distilled water. After complete evaporation the glass beaker was weighed again. The amount of solid residue obtained was noted. The data of this experiment is tabulated below.

Table -4 Quantity of distilled water and solid residue generated from evaporation of Stream -1 wastewater.

No.	Particulars	Unit	Amount
1	Quantity of wastewater (Stream -1)	Liter	0.5
2	Quantity of solid waste remaining	Gram	65.2412
3	Final quantity of wastewater stream (i.e. condensate water recovery after distillation)	Liter	0.45

During this treatment, distilled water will be generated which is having high purity and can be used in critical operations of manufacturing process. After distillation /evaporation, the solid material generated can be reused or disposed off to TSDF site.

b) Wastewater stream -2, can be used to produce ferrous sulphate by addition of Iron. For this the wastewater needs to be checked for pH and acidity only. Process wastewater of stream -2 of dye intermediates manufacturing units can be collected and analyzed for its acidity, based on acidity the entire wastewater can be sold as Spent acid to end users. If the acidity is lower, then the wastewater can be treated with waste iron to produce ferrous sulphate which can be used as wastewater treatment chemicals and resulting wastewater can be treated along with either Stream-1 wastewater for evaporation. During this recycling process due to implementation of segregation, by-products like Ferrous sulfate, Spent acid were recovered, which are potential sources of income for centralized facility such as CETPs[12].

Table -5 Quantity of iron scrap required to produce ferrous sulphate from stream -2 wastewater.

No.	Particulars	Unit	Amount
1	Quantity of wastewater (stream -2)	Liter	2.0
2	Quantity of Iron Scrap consumed	Gram	704
3	Final quantity of wastewater stream (i.e. ferrous sulphate slurry)	Liter	2.248

This slurry can be directly used to provide primary as well as tertiary treatment to the mixed stream a) above.

c) Mix Stream -3, Stream -4 and stream -5 and give primary treatment followed by secondary treatment with activated sludge process and tertiary treatment with Fenton reagent., this wastewater than passed through lab scale sand and carbon filter and then was analyzed and compared with the recommended water quality suitable for washing off processes[13]. The analysis results are represented in below table:

Table -6 Characteristic of mixed wastewater stream after tertiary treatment

No.	Parameters	Unit	Mixed Wastewater Stream -3, 4 and 5				GPCB norms for discharge in rivers, creeks	Recommended Water Quality Suitable For Washing Off Processes
			Untreated	Primary treated	Secondary treated	Tertiary treated		
1.	pH	pH Unit	8.41	8.12	8.15	8.11	**5.5 to 9.5**	**7.0 – 8.5**
2.	Chemical Oxygen Demand	mg/L	950	615	215	175	**250**	**200**
3.	Biochemical Oxygen Demand	mg/L	410	305	45	28	**30**	**Not applicable**
4.	Total Dissolved Solids	mg/L	3380	2435	2070	1960	**2100**	**Not applicable**
5.	Total Suspended Solids	mg/L	104	26	10	7	**100**	**Not applicable**
6.	Chloride	mg/L	4220	2095	1160	920	**1000**	**500 -2000**
7.	Ammoniacal Nitrogen	mg/L	94	83	11	5	**50**	**Not applicable**
8.	Phenolic Compounds	mg/L	0.62	0.51	0.39	0.24	**1**	**Not applicable**
9.	Hexavalent Chromium	mg/L	0.12	0.079	0.067	ND	**0.1**	**Not applicable**
10.	Total Chromium	mg/L	0.22	0.140	0.119	0.09	**2**	**0.9**
11.	Nickel	mg/L	ND	ND	ND	ND	**3**	**Not applicable**

12.	Oil & Grease	mg/L	17	13	3.21	1.05	**10**	**Not applicable**

Note: ND = Parameters analyzed but Not Detected

RESULTS AND DISCUSSION

Comparison of removal efficiency in percentage (%) for each parameter for primary, secondary and tertiary treatment of mixed wastewater stream 3, 4 & 5 is provided below.

- % removal in COD values ranges from 18.60 to 65.04%, maximum % removal of 65.04 % was obtained during secondary treatment.
- % removal in BOD values ranges from 25.61 to 85.25%, maximum % removal of 85.25 % was obtained during secondary treatment.
- % removal in TDS values ranges from 5.31 to 27.96 %, maximum % removal of 27.96 % was obtained during primary treatment.
- % removal in TSS values ranges from 30.00 to 75.00 %, maximum % removal of 75.00 % was obtained during primary treatment.
- % removal in Chloride values ranges from 20.69 to 50.35 %, maximum % removal of 50.35 % was obtained during primary treatment.
- % removal in Ammoniacal nitrogen values ranges from 11.70 to 86.75 %, maximum % removal of 86.75 % was obtained during secondary treatment.
- % removal in Phenolic compounds values ranges from 17.74 to 38.46 %, maximum % removal of 38.46 % was obtained during tertiary treatment.
- Hexavalent chromium was not detected after tertiary treatment however; maximum % removal of 34.17 % was obtained during primary treatment.
- % removal in Total chromium values ranges from 15.00 to 36.36 %, maximum % removal of 36.36 % was obtained during primary treatment.
- % removal in Oil and Grease values ranges from 23.53 to 75.31 %, maximum % removal of 75.31 % was obtained during secondary treatment.

CONCLUSION

The present treatment scheme is shown in Figure -1. As shown, in the figure, large amount of sludge is generated during treatment of wastewater of textile dyeing and printing industries as well as during treatment of wastewater of dyes and dye intermediates manufacturing industries. Also, it is evident that there is no reuse or recovery of resources.

The study confirms that it is possible to recycle wastewater and the treated wastewater can be reused for different manufacturing stages, which ultimately reduces the wastewater discharges and ultimately helps to conserve precious fresh water resources.

Entire quantity of utility wastewater stream, wastewater stream generated from textile dyeing and printing industries and dyes manufacturing industries can be mixed together and treated in conventional CETP, which is operational at Sachin industrial area. For this purpose process modification, i.e. installations of tertiary treatment units are required in addition to present treatment units at CETP of Sachin industrial area.

Sludge generated during the treatment can be reused by cement industry as alternate raw material[14-17], as well as can be reused as alternative to cement in the manufacturing of non-structural building materials.

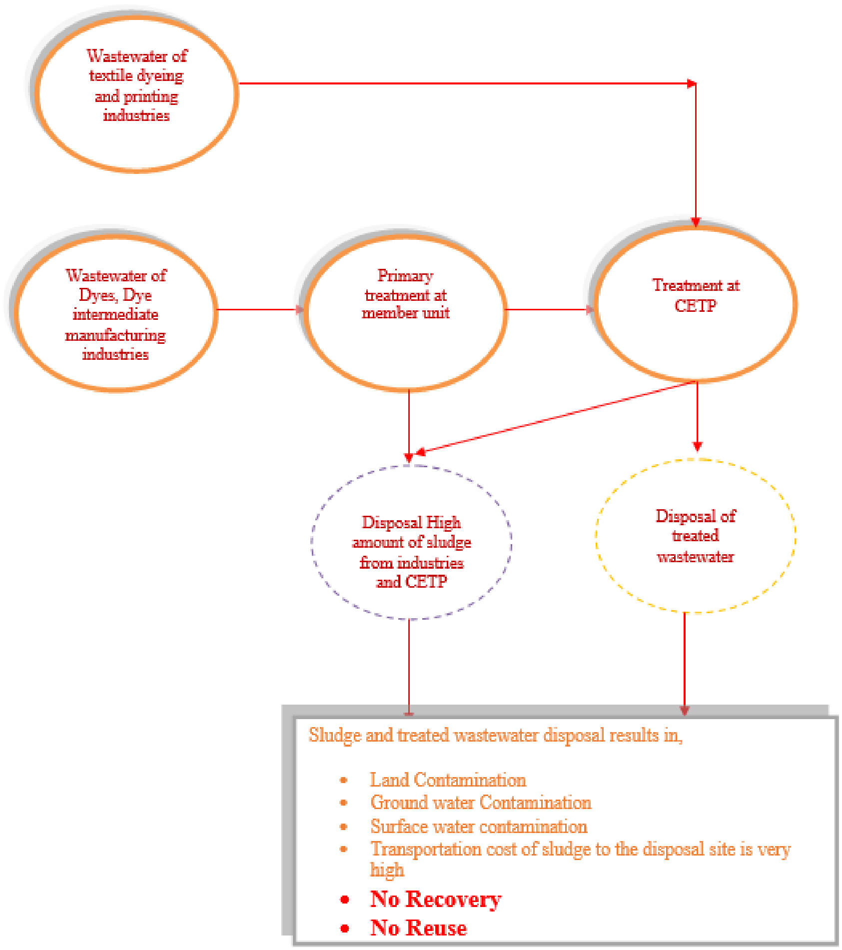
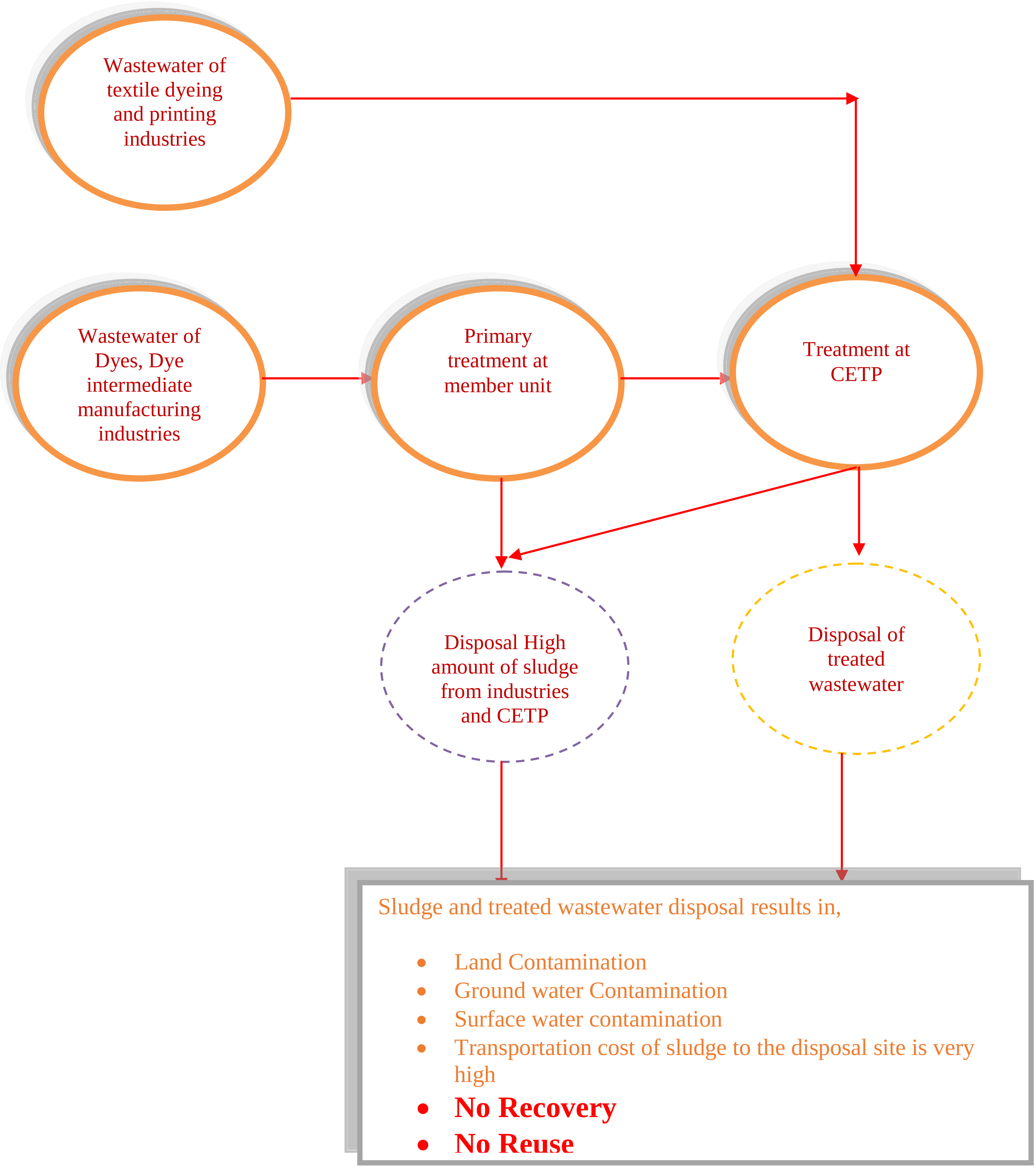
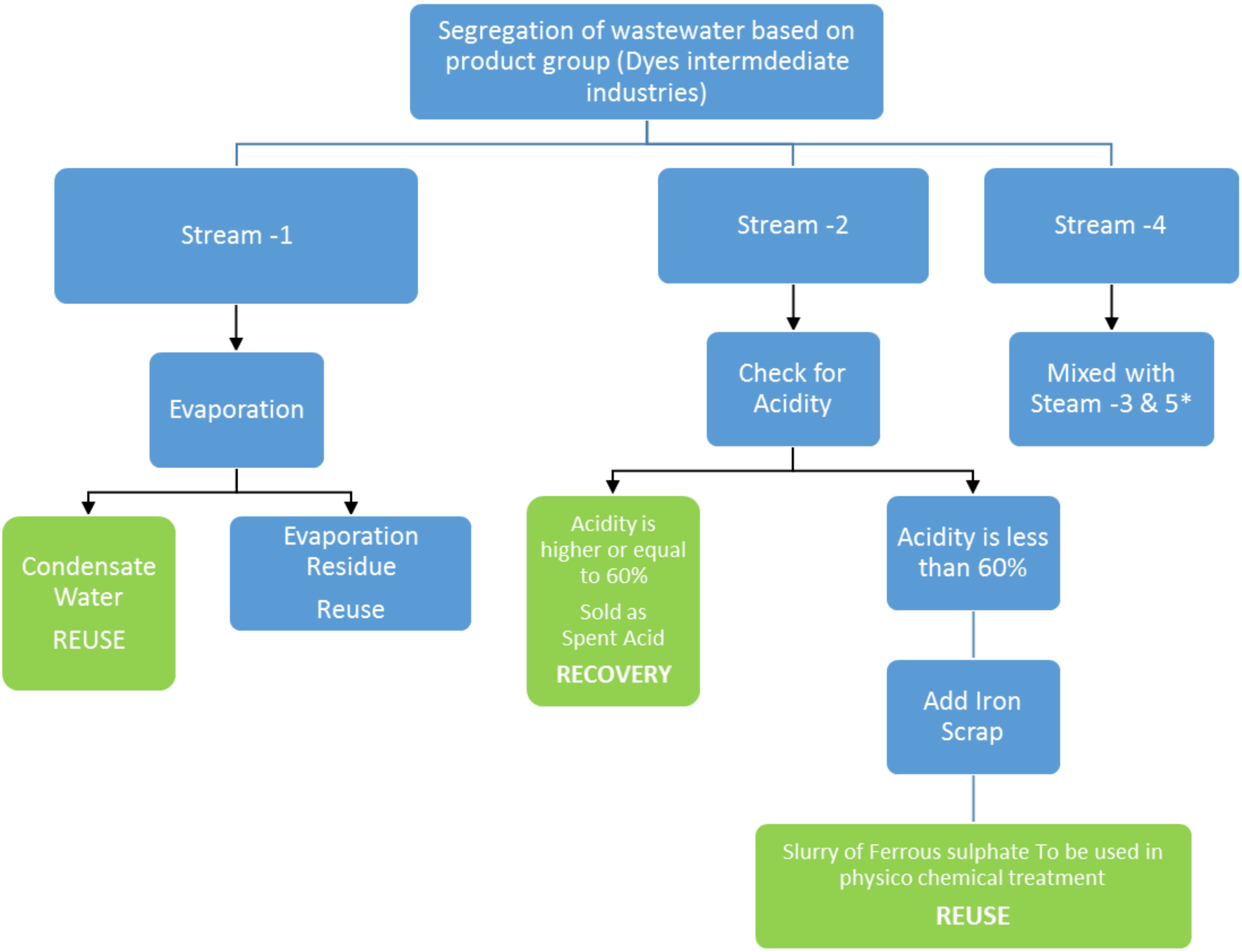


Figure -1 Present Treatment Scenario at GIDC Sachin


Wastewater of textile dyeing and printing industries
Wastewater of Dyes, Dye intermediate manufacturing industries
Primary treatment at member unit
Treatment at CETP
Disposal High amount of sludge from industries and CETP
Disposal of treated wastewater
Sludge and treated wastewater disposal results in,
Land Contamination
Ground water Contamination
Surface water contamination
Transportation cost of sludge to the disposal site is very high
No Recovery
No Reuse



***Note: Further treatment of Stream- 4 is shown in Figure -3**

Figure -2 Proposed Treatment Scheme (Plant Layout): Wastewater stream segregation, treatment and recycle (PART -1)

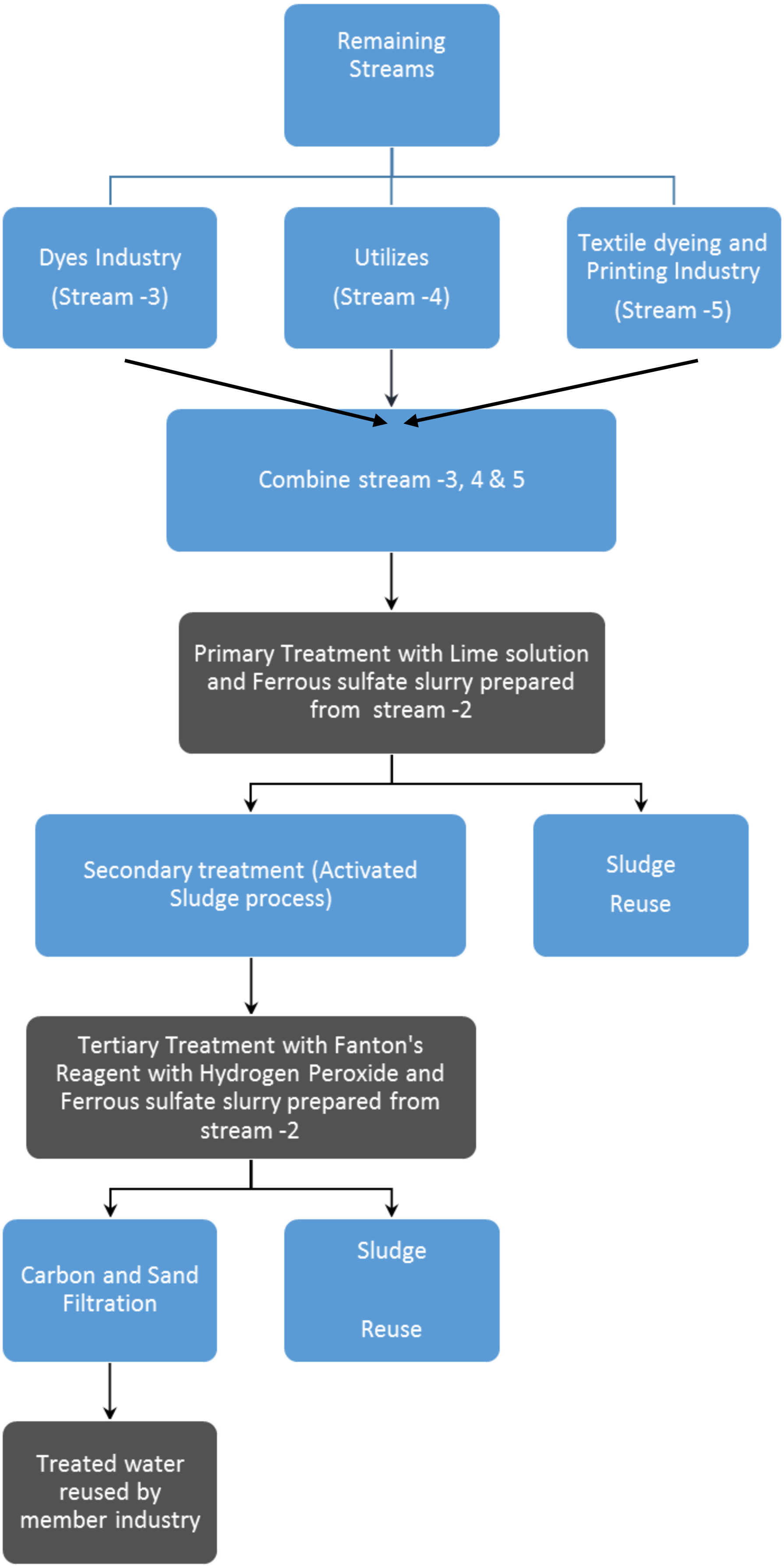


Figure -3 Proposed Treatment Scheme (Plant Layout): Wastewater stream segregation, treatment and recycle (PART -2)

This treatment scheme can be financially beneficial to the industries as well as the CETP as little modification is required at member industries level and at CETP level, and the implementation of this scheme can generate revenue by means of sale of Ferrous sulfate, spent acid and recycling of wastewater, recovery and reuse of sludge as raw material in Cement factory.

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