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Physico-Chemical and Heavy Metals Characteristics of Textile Dyeing Industry Effluent of Haridwar, Uttarakhand, India

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Abstract—*Wastewater is a significant deterrent in the evolvement of textile industry. The textile dyeing industry uses large quantum of water for different processes and at the same time generates huge quantities of wastewater. The amount and composition of wastewater depends upon the type of fabric used and processing required. The amount and composition of wastewater depends upon the type of fabric used and processing required. Wastewater is a significant deterrent in the evolvement of textile industry. The textile dyeing industry uses large quantum of water for different processes and at the same time generates huge quantities of wastewater. The amount and composition of wastewater depends upon the type of fabric used and processing required. The amount and composition of wastewater depends upon the type of fabric used and processing required. The wastewater is often high in oxygen demand, complex chemicals, dyes, color and heavy metals posing a gigantic risk to the environment if released untreated. The present study has been conducted to evaluate the physico-chemical characteristics and the heavy metals present in the textile dyeing wastewater. The physico-chemical parameters such as color, pH, turbidity, solids, alkalinity, total hardness, chloride, sulfate, biochemical and chemical oxygen demand (BOD, COD) were assessed. The heavy metals including chromium, zinc, manganese, copper, cadmium, lead, nickel and iron have also been determined. A total of 12 samples were taken from the equalization tank of the textile industry effluent treatment plant and were analyzed on monthly basis. The effluent was found to be brown-black in color and had a pungent odor. The COD concentration of 2461.54±48.45 mg/L with a very high sulfate concentration of 6620.83±7.22 mg/L has been assessed respectively. The heavy metals concentration in terms of iron was found to be 17.45±0.01 mg/L. The present study signifies that the wastewater emanating from textile industry is extremely polluted and should be discharged in the environment after thorough treatment.*

1. Introduction

Industrialization has changed the face of the Earth with long lasting implications. Industrial pollution is one of the major causes for the degradation of the environment. Textile industries are considered to be one of the most polluting owing to their diversity. Textile industry can be classified into three categories viz., cotton, woolen, and synthetic fibers depending upon the used raw materials[1]. Textile industries consume large volumes of water and chemicals for processing of textiles.

The chemical reagents used, ranging from inorganic compounds to polymers and organic products, are very diverse in chemical composition. Wastewater is generated from different steps in the dyeing and finishing processes[2]. Wastewater from printing and dyeing units is usually rich in color, containing residues of reactive dyes and chemicals, such as complex components, high chroma and high oxygen demand as well as much more hard-degradation materials[3]. The textile industry uses a variety of chemicals and a large amount of water for all of its manufacturing steps. However, the exact quantity of water required varies from industry to industry depending on the dyeing process and the type of fabrics produced. The textile dyeing processes and the nature of pollutants released are shown in figure 1.

2. Materials and Methods

2.1 Sample Collection and Analysis

Samples were collected in plastic containers washed with 10% HNO_{3} and rinsed with deionized water prior to usage. During sampling, sample bottles were rinsed with water which was sampled.

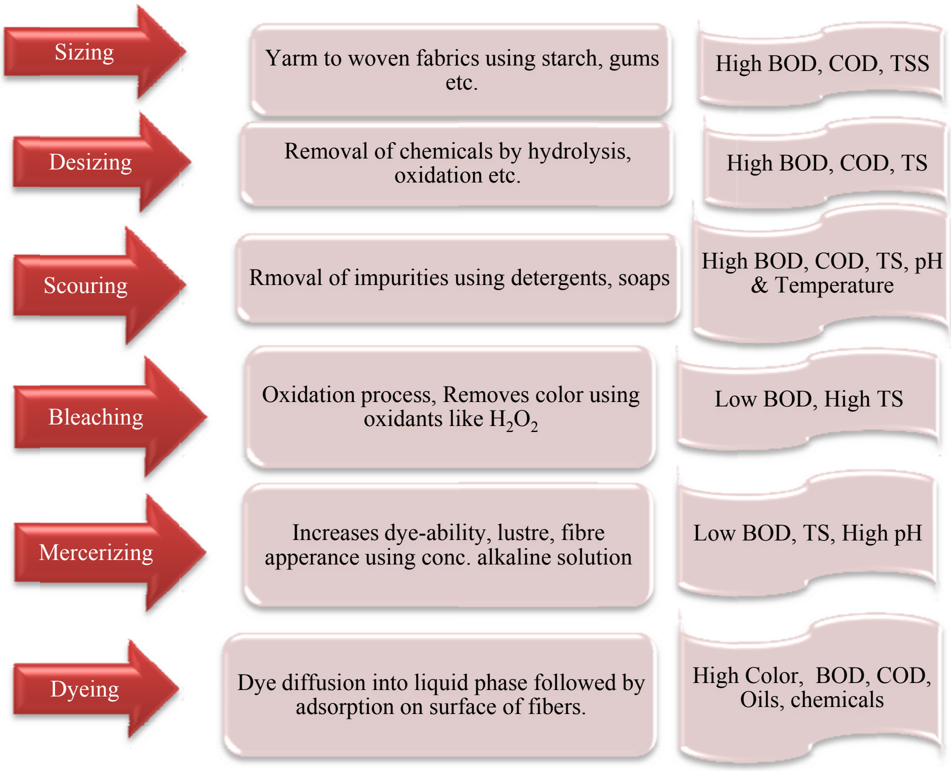

Figure 1: Textile Dyeing Process and Wastewater Characteristics

The samples were collected before treatment from the equalization tank of the textile dyeing industry in triplicates. The chemicals used were of AR grade. Double distilled water was used for the preparation of solutions and reagents. All the parameters were analyzed according to Standard Methods for the Examination of Water and Waste Water, APHA *et al.,* (2012)[4].

3. Results and Discussion

The results of the Physico-chemical characteristics and heavy metals concentration of textile dyeing industry effluent are shown in table 1 and 2 resp. The variation of physico-chemical characteristics is shown in figure 2.

3.1 Statistical analysis

The monthly (April 2018 - March 2019) analysis data presented in the study was mean and standard deviation of three replicates. Pearson correlation was used to show the interaction of the physico-chemical and heavy metal parameters of the effluent by using the MS Excel 2013.

3.2 Variation of Physico-Chemical Parameters

Color is the first contaminant to be recognized in wastewater[5].The effluent was brown black in color and had an unpleasant odor. The dark color of the effluent might be due to the various dyes and pigments used in the manufacturing processes in the industry. Banat *et al.,* (1997), reported that textile and other dye effluents are produced at relatively high temperatures[5]. In the present study, temperature of the effluent was recorded at a minimum of 54.67±0.58°C in April and maximum at 56.33±0.58°C in January. pH of the effluent was observed at a minimum of 9.63±0.01 in January and maximum at 9.87±0.01 in April. The high pH of the effluent can be attributed to the addition of bases, lime, and surfactants etc. which are added during the textile dyeing process[6]. Rao *et al.,* (1993) also observed the pH of textile industry effluent varied from 5.0 to 11.0[7]. Conductivity in water is affected by the presence of dissolved ions such as sodium, potassium, calcium, magnesium, iron, chloride, nitrate, sulfate, phosphate etc. The conductivity was recorded at a minimum of 1506.67±11.55 µS/cm and maximum at 2906.67±5.77 µS/cm. This was found to significantly higher which might be due to the presence to dissolved solids in the effluent.

Turbidity is an important water quality parameter that can indicate the presence of dispersed suspended solids, algae and other microorganisms, organic material and other minute particles. Turbidity was recorded at a minimum of 4.07±0.06 NTU in

January and maximum at 14.27±0.06 NTU in July. Total dissolved solids (TDS) is the term used to describe the inorganic salts and small amounts of organic matter present in dissolved form. Total dissolved solids were observed at a minimum of 996.67±5.77 mg/L in April and a maximum at 1806.67±11.55 mg/L in September. Textile industries show higher TDS value than the other industries due to the bleaching, fixing, dyeing agents, etc used during the processing of the fabrics[6]. Total suspended solids were recorded at a minimum of 86.67±11.55 mg/L in January and maximum at 410.00±17.32 mg/L in July. The increased amount of TSS is primarily due to increased chemical dosing, dye fixation and partial dissolution of fibre materials[6].

Hardness is mainly due to presence of calcium and magnesium salts. It can be a nuisance due to the mineral build-up on plumbing fixtures and poor soap and detergent performance. Total hardness was observed at a minimum of 71.97±2.06 mg/L in July and maximum at 648.34±2.40 mg/L in November. The chemical oxygen demand is the most widely used parameter for wastewater characterisation. Chemical oxygen demand was recorded at a minimum of 853.66±52.81 mg/L in April and maximum at 2461.54±48.45 mg/L in July. High COD levels imply toxic condition and the presence of biologically resistant organic substances. The higher value of COD is due to the nature of chemicals used in the dying process[8].

Biochemical oxygen demand is a measure of the quantity of oxygen used by microorganisms (aerobic bacteria) in the oxidation of organic matter. Biological oxygen demand ranged was recorded at a minimum of 267.22±30.66 mg/L in April and maximum at 793.33±63.74 mg/L in July. The ratio of BOD: COD was also studied and if the BOD: COD ratio was found to be less than 0.5, it indicates that the effluent contains a large proportion of non-biodegradable matter. The ratio of the BOD: COD obtained from the results of the present study was found to be in the range of 0.31 to 0.36. This indicates that the effluent contains a large portion of non-biodegradable matter. The BOD_5: COD ratio values ranged from 0.2 – 0.4 indicates the low biodegradability of the textile effluents[9].

3.3 Major cation chemistry

Nitrogen can be present in different forms. Ammonia sources are printing, coating, preparation, and dyeing processes[10]. Ammonical nitrogen was recorded to be minimum at 36.91±0.11mg/ L in April and maximum at 176.79±0.11mg/L in February. The level of sodium was found to be minimum at 21.67±0.58 mg/L in March and maximum at 144.33±0.58 mg/L in September. Whereas the minimum level of potassium was recorded as 1.13±0.06 mg/L in April and maximum at 57.87±0.12 mg/L in July. The higher concentration of the sodium was due to the sodium compounds used in wet processes. Sodium compounds are preferred than that of potassium. Therefore, the potassium is in lower concentration[11]. Calcium was found to be minimum at 15.67±0.94 mg/L in April and maximum at 162.52±0.32 mg/L in November. Magnesium was found to be minimum at 7.75±0.00 mg/L in May and maximum at 58.82±0.39 mg/L in November.

3.4 Major anion chemistry

Carbonates show its presence in water at pH more than 8.3, and hence, in the present study, no bicarbonate alkalinity could be expected as pH was higher than 8.3. The total alkalinity therefore of the water sources could be considered almost entirely due to the presence of carbonates. In textile processing, wastewaters from preparation processes such as mercerising and caustic scouring have high alkalinity levels. After being processed in baths with high alkalinity, fabrics have to be rinsed thoroughly and neutralised to assure that the residual alkalinity in the fabric is low[5].

Alkalinity was recorded to be minimum at 160.82±2.44 mg/L in April and maximum at 429.92±2.12 mg/L in November. Chloride ions are abundantly present in textile wastewater. In bleaching, amongst other chemicals chloride containing compounds are frequently used. Sodium chloride is used in large quantities in reactive dyeing[12]. Chloride was recorded to be minimum at 298.23±4.07 mg/L in April and maximum at 691.21±2.56 mg/L in September. Phosphates are used in different steps in textile wet processing including buffers, scouring, water conditioners, surfactants, and flame-retardant finishes[13].

Phosphate for the effluent was recorded to be minimum at 2.11±0.05mg/L in April and maximum at 28.48±0.02mg/L in February. In textile wastewaters, dye bath effluents containing phosphate buffers are the main source of phosphorus[14]. Sulfate is an important anion imparting hardness of water. It may undergo transformation to sulfur or hydrogen sulfide depending largely upon the redox potential of water. Sulphate was recorded to be minimum at 950.00±12.50mg/L in March and maximum at 6620.83±7.22 mg/L in November. It is generated due to mercerizing and dyeing operations[15].

3.5 Metals

Metals enter the wastewater in many ways: the incoming water supply, metal parts (like pumps, pipes, valves, etc.), oxidising and reducing agents, electrolyte, acid and alkali, dyes and pigments, certain finishes, herbicides and pesticides, and maintenance chemicals[11]. The main source of heavy metals is the dyeing process. Most used dyes contain chromium, cadmium, zinc or other metal atoms[12]. The results of heavy metals are shown in table 2.

3.6 Correlation matrix analysis

The degree of linear association between the important physico-chemical parameters and the major cations and anions is represented in the correlation matrix. A correlation coefficient (*r*) of + 1 indicates that two variables are perfectly related in a positive linear sense, but $r \leq -1$ indicates a negative linear correlation. The correlation can be considered as strong when $r > 0.50$, good when $r \leq 0.50$ and poor when $r < 0.50$[16]. Thus, correlation analysis reveals the nature of the relationship between the water quality parameters of the textile dyeing industry effluent. The correlation matrix is shown in table 3.

pH has a strong negative correlation with temperature (r≤ -0.91). Turbidity has a strong positive correlation with total suspended solids (r≤ 0.96) while a good negative correlation with ammonical nitrogen, NH_4^+ as N (r≤ -0.86) and PO_4^{3-} (r≤ -0.75). Electrical conductivity bears a strong positive correlation with total dissolved solids (r≤ 0.95), Cl^- (r≤ 0.74), chemical oxygen demand (r≤ 0.69), biological oxygen demand (r≤ 0.67) and Na^+ (r≤ 0.55). Total dissolved solids bear good positive correlation with Cl^- (r≤ 0.74), chemical oxygen demand (r≤ 0.57), biological oxygen demand (r≤ 0.55), Na^+ (r≤ 0.46). Total suspended solids have a positive correlation with chemical oxygen demand (r≤ 0.52), biological oxygen demand (r≤ 0.52) and K^+ (r≤ 0.44).

Alkalinity correlates positively with total hardness (r≤ 0.91), Ca^{2+} (r≤ 0.90), Mg^{2+} (r≤ 0.90),Na^+ (r≤ 0.71), SO_4^{2-} (r≤ 0.69), Cl^- (r≤ 0.68). Total hardness correlates positively with Ca^{2+} (r≤ 1.00) and Mg^{2+} (r≤ 0.99), SO_4^{2-} (r≤ 0.77), Cl^- (r≤ 0.67) and Na^+ (r≤ 0.62). Chloride bears a positive correlation with Na^+ (r≤ 0.79), Ca^{2+} (r≤ 0.68), Mg^{2+} (r≤ 0.65), SO_4^{2-} (r≤ 0.56), chemical oxygen demand (r≤ 0.55), biological oxygen demand (r≤ 0.55), PO_4^{3-} (r≤ 0.48), Ammonical nitrogen, NH_4^+ as N (r≤ 0.36). Chemical oxygen demand bears a positive correlation with biological oxygen demand (r≤ 1.00), K^+ (r≤ 0.57), Na^+ (r≤ 0.30), SO_4^{2-} (r≤ 0.25). Biological oxygen demand correlates positively with K^+ (r≤ 0.56), Na^+ (r≤ 0.28), SO_4^{2-} (r≤ 0.22). Ammonical nitrogen bears a positive correlation with PO_4^{3-} (r≤ 0.96), Ca^{2+} (r≤ 0.30), Mg^{2+} (r≤ 0.20) while a negative correlation with SO_4^{2-} (r≤ -0.21) and K^+ (r≤ 0.09).

4. Conclusion

Wastewater is a significant deterrent in the evolvement of textile industry. The volume of water used in the textile dyeing industry is humungous and the volume of wastewater generated is also high. Textile wastewater is characterized by the diversity of chemicals used depending upon the industry and the type of fiber. The minimum and maximum BOD concentration of 267.22±30.66 mg/L observed in April and 793.33±63.74 mg/L observed in July respectively. The minimum and maximum ammonical nitrogen concentration of 36.91±0.11mg/ L has been observed in April and 176.79±0.11mg/L observed in February respectively. Characterization of the effluent is a crucial step in choosing an end of pipe treatment method for the wastewater. Majority of pollutants are released from the dyeing operations. It is pivotal to identify the prospective sources of wastewater and release the effluent only after significant treatment.

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Table 1: Physico-Chemical Characteristics of the Textile Dyeing Industry Effluent

Parameter / Month	April	May	June	July	August	September	October	November	December	January	February	March
Temp (°C)	54.67±0.58	55.33±0.58	55.67±0.57	56±0.00	55.67±0.5	55.67±0.58	56±0.00	55.67±0.58	55.33±0.58	56.33±0.58	55.33±0.58	55.67±1.15
pH	9.87±0.01	9.83±0.01	9.78±0.01	9.75±0.01	9.78±0.01	9.77±0.01	9.72±0.01	9.75±0.01	9.80±0.01	9.63±0.01	9.81±0.01	9.71±0.01
Turbidity (NTU)	9.97±0.15	12.13±0.12	12.23±0.06	14.27±0.06	8.17±0.06	5.17±0.06	8.13±0.06	12.13±0.06	5.07±0.06	4.07±0.06	5.13±0.06	5.17±0.06
E.C. (µS/cm)	1507±11.55	1897±5.77	2007±11.55	2013±11.55	2513±11.55	2907±5.77	1697±5.77	1813±11.55	1673±5.77	1807±5.77	1647±5.77	1583±5.77
TDS (mg/L)	997±5.77	1207±11.55	1210±17.32	1090±17.32	1787±23.09	1807±11.55	1093±11.55	1113±11.55	1007±11.55	1193±11.55	1010.±17.32	1007±11.55
TSS (mg/L)	207±11.55	293±11.55	343±11.55	410±17.32	153±5.77	107±11.55	137±5.77	307±11.55	107±11.55	86.67±5.77	107±11.55	107±11.55
Alkalinity (mg/L)	161±2.44	173±3.23	178±1.60	253±2.19	284±2.40	351±4.28	406±3.49	430±2.12	389±4.49	368±2.39	305±1.61	171±1.61
Hardness (mg/L)	71.97±2.06	81.69±0.77	89.08±0.76	141±2.45	368±2.20	397±4.20	525±1.47	648±2.40	494±4.98	338±2.34	260±3.43	270±2.22
Chloride (mg/L)	298±4.07	372±2.55	444±1.97	462±2.56	637±3.51	691±2.56	569±5.10	545±5.41	488±1.94	508±2.49	470±3.90	434±1.97
C.O.D (mg/L)	854±52.81	1029±52.41	1923±49.70	2462±48.45	2355±47.98	2042±45.94	1074±46.50	2210±46.69	1145±46.13	1175±46.25	1016±46.31	931±46.07
B.O.D (mg/L)	267±30.66	334±11.09	654±27.13	793±63.74	750±94.94	645±54.54	359±14.04	712±50.48	370±8.30	426±47.89	341±13.60	321±14.45
A. Nitrogen (mg/L)	36.91±0.11	56.98±0.21	64.88±0.21	68.46±0.21	107±0.11	152±0.21	59.32±0.11	69.20±0.11	151±0.11	169±0.11	177±0.11	161±0.11
Phosphate (mg/L)	2.11±0.05	8.16±0.02	8.41±1.67	12.74±0.02	20.01±0.02	24.26±0.04	7.83±0.04	14.71±0.02	20.58±0.02	26.79±0.02	28.48±0.02	25.08±0.05
Sulphate (mg/L)	2717±7.22	1133±7.22	1050±12.50	1092±7.22	2617±7.22	5042±19.09	5504±7.22	6621±7.22	2367±7.22	1575±12.50	1329±14.43	950±12.50
Sodium (mg/L)	25.33±0.58	31.33±1.15	43.67±0.58	62.17±0.58	59.67±0.58	144.33±0.58	122±0.58	70.33±0.58	84.33±0.58	51.33±1.15	48.67±1.15	21.67±0.58
Potassium (mg/L)	1.13±0.06	1.23±0.06	1.37±0.06	57.87±0.12	1.43±0.06	28.67±0.58	1.83±0.06	1.57±0.06	1.33±0.06	1.37±0.06	1.27±0.06	1.23±0.06
Calcium (mg/L)	15.67±0.94	19.91±0.31	22.78±0.92	42.84±0.86	91.95±0.88	103±1.68	127±0.59	163±0.32	126±2.64	83.15±0.83	69.66±1.37	73.85±2.63
Magnesium (mg/L)	7.97±0.19	7.75±0.00	7.81±0.74	8.33±0.20	33.49±0.00	33.88±0.00	50.13±0.00	58.82±0.39	43.56±0.61	31.58±0.19	20.81±0.00	20.82±1.06

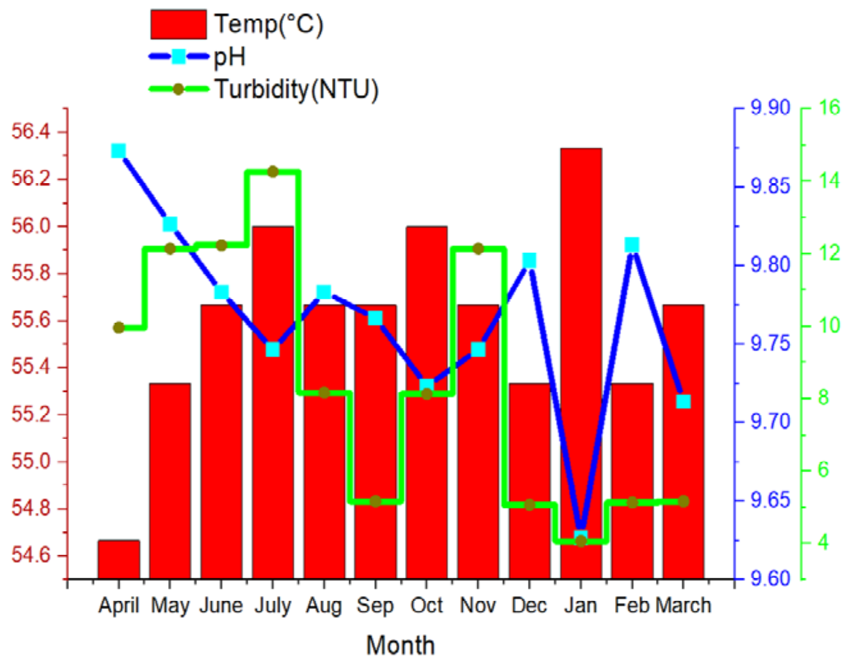
Table 2: Heavy Metals Concentrations of Textile dyeing Industry Effluent

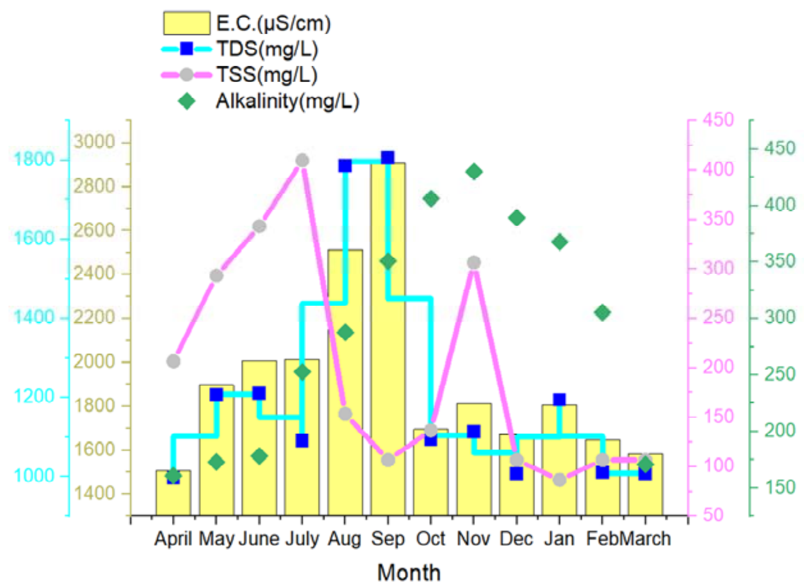
Metals / Month	**April**	**May**	**June**	**July**	**August**	**September**	**October**	**November**	**December**	**January**	**February**	**March**
Chromium as Cr^{3+}	N.D.	N.D.	N.D.	N.D.	0.02±0.00	0.02±0.01	0.02±0.00	0.04±0.01	0.04±0.00	0.05±0.00	0.05±0.00	0.04±0.00
Zinc as Zn^{2+}	0.22±0.00	0.22±0.01	0.58±0.01	1.09±0.01	1.10±0.01	1.91±0.01	1.25±0.01	0.88±0.01	0.96±0.01	1.30±0.01	2.59±0.00	2.55±0.01
Manganese as Mn^{2+}	N.D.	N.D.	N.D.	N.D.	N.D.	0.18±0.00	N.D.	N.D.	0.10±0.00	0.12±0.00	0.09±0.01	0.33±0.01
Copper as Cu^{2+}	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
Cadmium as Cd^{2+}	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
Lead as Pb^{2+}	N.D.	N.D.	N.D.	0.18±0.00	0.22±0.01	0.25±0.01	0.38±0.01	0.49±0.01	0.56±0.01	N.D.	1.18±0.00	0.22±0.01
Nickel as Ni^{2+}	N.D.	N.D.	N.D.	0.53±0.01	0.77±0.01	0.84±0.00	0.22±0.01	0.17±0.00	0.15±0.00	0.15±0.00	0.68±0.01	0.94±0.01
Iron as Fe^{2+}	3.42±0.00	3.65±0.00	3.88±0.00	4.23±0.01	11.63±0.01	12.81±0.01	7.12±0.01	6.33±0.00	5.23±0.01	7.46±0.01	17.45±0.01	10.80±0.00

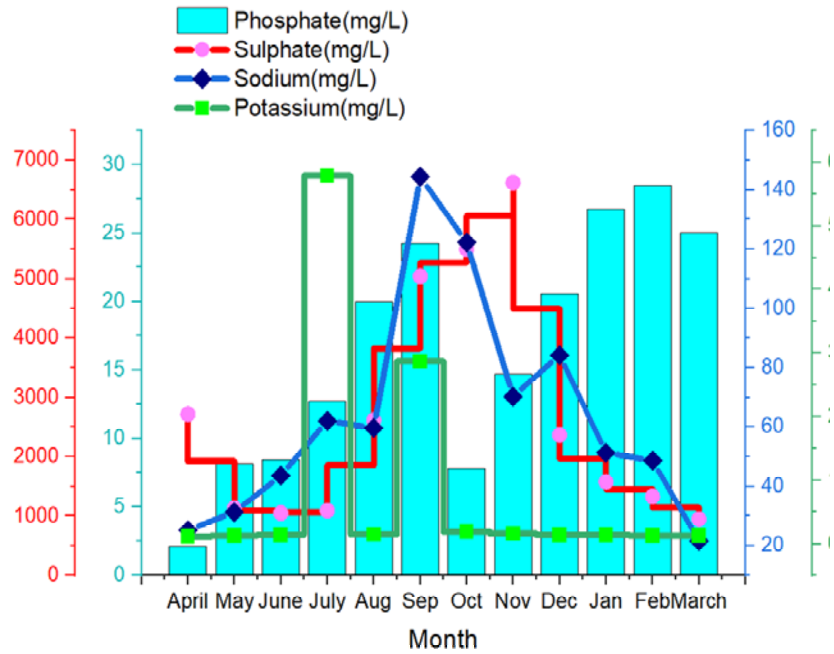
(N.D.-Not Detected)

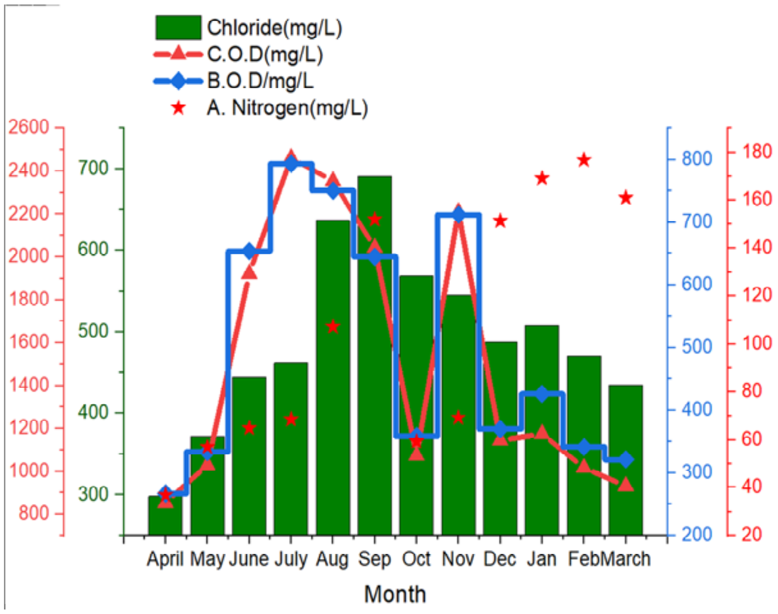
Table 3: Correlation Matrix for textile dyeing industry effluent parameters

Parameters	***Temp***	***pH***	***Turbidity***	***E.C.***	***TDS***	***TSS***	***Alkalinity***	***Hardness***	**Cl^-**	***C.O.D***	***B.O.D***	**NH_4^+ as N**	**PO_4^{3-}**	**SO_4^{2-}**	**Na^+**	**K^+**	**Ca^{2+}**	**Mg^{2+}**
Temp	1																	
pH	-0.91	1																
Turbidity	-0.10	0.31	1															
E.C.	0.25	-0.04	-0.01	1														
TDS	0.20	-0.04	-0.12	0.95	1													
TSS	-0.01	0.22	0.97	0.01	-0.15	1												
Alkalinity	0.44	-0.43	-0.33	0.14	0.13	-0.34	1											
Hardness	0.34	-0.39	-0.35	0.10	0.13	-0.39	0.91	1										
Cl^-	0.54	-0.40	-0.33	0.74	0.74	-0.33	0.68	0.67	1									
C.O.D	0.37	-0.12	0.46	0.69	0.57	0.52	0.20	0.17	0.55	1								
B.O.D	0.42	-0.18	0.45	0.67	0.55	0.52	0.20	0.17	0.55	1.00	1							
NH_4^+ as N	0.25	-0.39	-0.86	0.11	0.13	-0.73	0.29	0.27	0.36	-0.19	-0.17	1						
PO_4^{3-}	0.36	-0.46	-0.76	0.22	0.23	-0.63	0.36	0.34	0.48	0.01	0.03	0.96	1					
SO_4^{2-}	0.08	-0.08	0.01	0.24	0.25	-0.10	0.69	0.77	0.56	0.25	0.22	-0.21	-0.13	1				
Na^+	0.33	-0.19	-0.24	0.55	0.46	-0.26	0.71	0.62	0.79	0.30	0.28	0.12	0.14	0.70	1			
K^+	0.30	-0.10	0.35	0.41	0.17	0.44	-0.01	-0.18	0.18	0.57	0.56	-0.09	-0.01	-0.06	0.30	1		
Ca^{2+}	0.35	-0.40	-0.35	0.11	0.13	-0.38	0.90	1.00	0.68	0.19	0.19	0.30	0.38	0.75	0.62	-0.14	1	
Mg^{2+}	0.32	-0.38	-0.33	0.08	0.13	-0.39	0.90	0.99	0.65	0.14	0.13	0.20	0.27	0.79	0.61	-0.25	0.98	1


Temp(°C)
pH
Turbidity(NTU)
56.4
56.2
56.0
55.8
55.6
55.4
55.2
55.0
54.8
54.6
9.90
9.85
9.80
9.75
9.70
9.65
9.60
16
14
12
10
8
6
4
April May June July Aug Sep Oct Nov Dec Jan Feb March
Month


E.C.(µS/cm)
TDS(mg/L)
TSS(mg/L)
Alkalinity(mg/L)
1800
1600
1400
1200
1000
3000
2800
2600
2400
2200
2000
1800
1600
1400
450
400
350
300
250
200
150
100
50
450
400
350
300
250
200
150
April May June July Aug Sep Oct Nov Dec Jan FebMarch
Month


Phosphate(mg/L)
Sulphate(mg/L)
Sodium(mg/L)
Potassium(mg/L)
April May June July Aug Sep Oct Nov Dec Jan FebMarch
Month


Chloride(mg/L)
C.O.D(mg/L)
B.O.D/mg/L
A. Nitrogen(mg/L)
April May June July Aug Sep Oct Nov Dec Jan FebMarch
Month

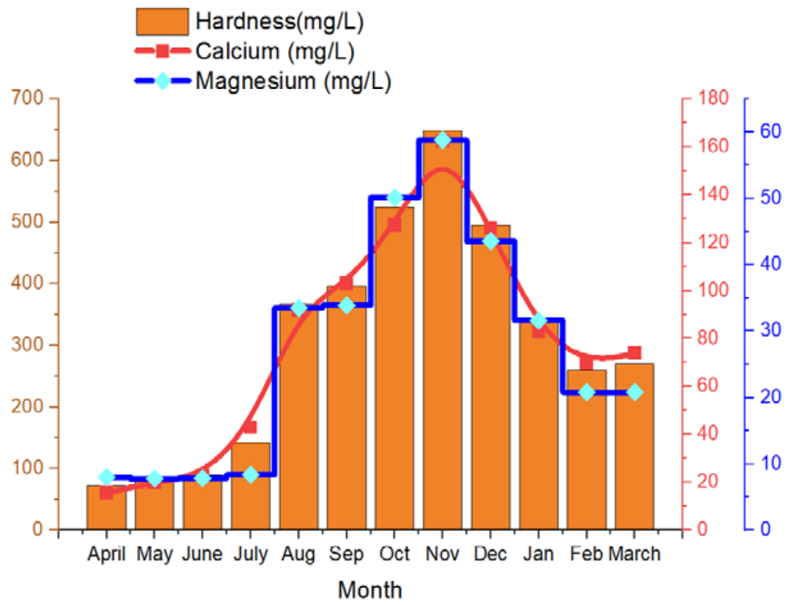


Figure 2: Variation of physico-chemical parameters