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Comparative Study of Treatment of Reactive Super Black Textile Dye Effluent Using Different Chemical Coagulants

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Abstract

Color is the most obvious contaminant to be noticed in textile dye wastewater. A numbers of methods and materials have been used in textile dye effluent treatments. Generally, the chemical approaches are mostly utilized by employing coagulation and flocculation mechanisms. The efficiency of dye removal depends on the type of dye, pH, coagulant type and dosage. The amount of the unfixed dyes lost into effluent is variable with reactive dyes reported to be highest, ranging from 20-50%.

A comparative study was therefore conducted to optimize treatment of Reactive Super Black dye effluent from a textile factory using Alum, Polyferrous Sulphate (PFS) and Polyalluminium Ferric Chloride (PaFC) coagulants at varied pH and dosages. Treatment efficiency was assessed by analysis of total dissolved solids, conductivity, salinity, COD, colour concentrations, turbidity and quantities of sludge generated.

It was found that Alum clarified the dye waste water at pH 6,7 and 10, Polyferrous Sulphate at pH 6-8 and Polyalluminium Ferric Chloride at pH 4- 8. Treatment of the effluent at the optimized pH ranges showed that Alum clarified the dye at 120 g/L and PFS at 80 mg/L. Gradual addition Polyalluminium Ferric Chloride from 140-280 mg/L however gave insignificant clarification of the dye. On the other hand, Polyalluminium Ferric Chloride gave the lowest values of TDS, conductivity, salinity and sludge generation compared to PFS and Alum whose results were relatively similar. Therefore, while Polyalluminium Ferric Chloride indicated to be the best coagulant for the reactive super black dye waste water coagulation by the chemical parameters analysed, it performed poorly in terms clarification of the dye both from visual observation and UV-Vis Spectrophotometer analysis results.

Keywords: Dye effluent; Alum; Polyferrous sulphate; Polyalluminium ferric chloride; pH; Coagulant dosage

Introduction

Textile industries are considered as one of the biggest sources of water pollution due to the utilization of different chemicals during textile processing and eventual discharge. The effluents from different processes are discharged as wastewater that contains turbidity, colour, high level of toxic chemicals, Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), salts, metal elements, pH, dissolved and suspended solids which have direct and indirect effects on aquatic fauna, flora and human health [1,2]. Dyeing contributes to most of the metals and almost all of the salts and colour present in the overall textile effluent. For some dyeing processes like cotton with reactive dyes, about 70% to 75% of the salts end up in the wastewater [3,4,5,6].

Color is the most obvious contaminant to be recognized in textile wastewater. Dye effluents are more difficult to treat because their synthetic origin are mainly complex aromatic molecular structures, often synthesized to resist fading on exposure to sweat, soap, water, light or oxidizing agents. This renders them more stable and difficult to biodegrade. Resistance of dyes to degradation make color removal from textile wastewaters difficult. Due to high thermal and photo stability of dyes, they can persist in the water environment for an extended period of time if left untreated. Limiting the re oxygenation capacity of the receiving water and prevention of sunlight transmission disturbs the photosynthetic activities of plants in the aquatic system [7]. These industrial discharges if released to sea, river and/or lakes without proper treatment, adversely affect the environment, aquatic and human life [8,9]. The extent of loss of unfixed dyes into effluent is variable and ranges from 20-50% for reactive dyes, 30-40% for sulphur dyes, 5-10% for azoic dyes, 5-20% for vat and direct dyes, 7-20% for acid and disperses dyes, 2-3% for basic dyes and 1-2% for pigment dyes [2]. The loss of dyes to effluent can be estimated to be 10 % for deep shades, 2 % for medium shades and minimal for light shades. Dyes are present in the effluent at concentrations of 10 mg/L to 50 mg/L with 1 mg/L being visible to the naked eye [10].

Recently, therefore, dye removal from textile waste water has become a research area of increasing interest, as government legislation concerning the release of contaminated effluents has become more stringent. Textile industries have therefore been faced with more stringent effluent treatment regulations and are required to remove hazardous chemicals in their wastewater before discharge into the surface water.

Dye Effluent Treatment

A numbers of methods and materials; Electrocoagulation, constructed wetlands, hybrid ion exchange materials, modified bentonite, membrane filtration, activated carbon, ozonation, photo oxidation, adsorption and reverse osmosis have been used in industrial effluents treatments. All these treatments technologies are playing significant role in the management of industrial effluents. However, they are expensive and produce massive amounts of toxic sludge [11]. Generally, the chemical approaches are utilized for elimination of organic contaminants by employing coagulation as well as flocculation approaches. Coagulation/flocculation of suspended particles and colloids result from different mechanisms including electrostatic attraction, sorption (related to protonated amine groups) and bridging (related to polymer high in molecular weight). Chemical coagulants are capable of dissociating polyelectrolytes when in solution. The polyelectrolytes will neutralise the negatively charged dye molecules to form particle-polymer-particle complexes. These contaminants will be precipitated in the form of chemical sludge when the treated water is further added with flocculants [9,12]. Chemical coagulation/flocculation is usually employed to remove the suspended colloidal particles within the 0.1–1 µm and possibly any dissolved particles which cannot be easily removed by the application of other conventionally applied physical separation processes such as gravity settling, filtration.

Chemical coagulants can be classified into the following categories:

- Hydrolyzing metallic salt coagulants (FeCl3, FeSO4, MgCl2 and alum)
- Pre-hydrolyzing metallic salt coagulants: (Polyaluminium Chloride (PaC); Polyaluminium Ferric Chloride (PaFC); Polyaluminium Sulphate (PaS) and Polyferrous Sulphate (PFS)
- Synthetic cationic polymers (aminomethyl polyacrylamide, polyalkene, polyamine, polyethylamine).
- Commonly used coagulants are lime, magnesium, iron and aluminium salts.Coagulation by alum and MgCl2 generates large quantity of sludge and usually not acceptable [13,9].

PaC is preferred for the following reasons: coagulation can be done under wider pH range (7-10); lower dose of coagulant is required because the aluminium content is 3 times in comparison to alum, contains chloride instead of sulphate and produces larger and heavier flocs. PaC shows better colour removal efficiency in a wider pH range of 7-10[14]. Likewise, PFS is also an efficient highly soluble coagulant that works under broad pH range with the formation of rapidly settling flocs. PaC and PFS exhibit similar mechanisms of coagulation which are adsorption and charge neutralization. Prehydrolysed coagulants such as PaC, PaFC and PFS are reported to give better colour removal even at low temperature and may also produce lower volume of sludge. PaFC combines the advantages of aluminium and iron salts [15]. These chemicals however stay in wastewater after treatment and sludge and may cause health and ecological complications. The generated sludge contains a considerable amount of chemical residue such as iron and aluminium salts that are hazardous to human health and environmental. The efficiency of dye removal depends on the type of dye, coagulant type and dosage, and the sample pH.

A comparative study was therefore conducted to assess efficiency of dye waste water treatment using alum, Polyaluminium Ferric chloride (PaFC) and Poly Ferrous Sulphate (PFS).

Physicochemical Characteristics of Dye Effluents

Apart from dyes, different auxiliary chemicals are used that also end up in the wastewater. Typical pollutants generated in the dyeing step are colour and different auxiliaries, such as organic acids, fixing agents, defoamers, oxidising/reducing agents, and diluents [3,4,5,6]. Dye effluents are usually high in color, pH, total dissolved solids, chemical oxygen demand, biochemical oxygen demand, salts.

- Colour; Colour may result from the presence of metallic ions such as iron and manganese from dyes, industrial wastes inform of colloidal or suspended material among others. Residual colour is mainly due to insoluble dyes, pigments and other coloured compounds with a low biodegrability.
- Chemical Oxygen Demand (COD); It is used to measure the oxygen equivalent to organic material in the wastewater that can be oxidized chemically using dichromate in an acid solution. It comprises both the biodegradable and non-biodegradable portions of live bacterial attack, but it can be oxidized by strong chemical oxidants. High COD levels imply toxic conditions and the presence of biologically resistant organic substances
- Biochemical Oxygen Demand (BOD); the amount of oxygen consumed during a certain time for biochemical degradation of organic material and for oxidising inorganic material salts. Its measurement concerns the determination of the degradation of organic substances by microorganisms.
- Salinity; salinity is the total amount of salts. These salts may include sodium chloride, magnesium sulphates, potassium nitrates, and sodium bicarbonate.
- Total Dissolved Solids; Total dissolved solids is a measure of the combined content of all inorganic and organic substances contained in a liquid in molecular, ionized or micro-granular suspended form.
- Electrical Conductivity; a measure of the ability of water to conduct an electric current. It depends on the dissociation of ions, their concentration, and temperature. It is sensitive to variations in dissolved solids, mostly mineral salts. EC is directly

Proportional to the total dissolve solids (TDS) concentration [16,17].

Materials and methods

Textile dye waste water sample was collected from Rivatex East Africa Limited, a vertically integrated textile factory that processes cotton located along Kipkaren Road, Eldoret, Kenya. The effluent was sampled at the jig dyeing machine drainage point using a sterile polyethylene container. Sampling was done according to standard sampling principles and guidelines outlined in American Public Health Association (APHA, 1998). The container was washed and rinsed twice with distilled water and then the effluent to be sampled before sampling. The recipe that had been used for the dye process was as follows;

Reactive Super Black R. 4%, Sodium Sulphate 80 g/l, Sodium Carbonate 30 g/l, Wetting Agent 2 g/l, Acetic Acid 1 g/l.

The sample was transported immediately in a cooler box and refrigerated at 40 C awaiting analysis and treatment. The sample was analysed based on the following parameters; pH, electrical conductivity, salinity, total solids, total dissolved solids, total suspended solids, COD, BOD and turbidity. Temperature, pH, electrical conductivity, salinity and total dissolved solids were analysed using BIOBASE precision Ph/Ion meter. Turbidity was determined by Turbidimeter (ums-UK-Water Quality Analyzer). Chemical oxygen demand was analysed using COD- 571 Analyzer. Biochemical Oxygen Demand (BOD) was determined by incubating the sample at 21 °C for 5 days using OxiTop –IDS bottles. Colour concentrations analysis was done by UV-Vis spectrophotometer (UV-1900 Shimadzu) at 600 nm.

Chemical coagulation-flocculation of the dye effluent was done using Polyaluminium Ferric Chloride (PaFC), Polyferrous Sulphate (PFS) and Alum. Coagulation-flocculation experiments were conducted using jar test procedure with five beakers of one Litre and a stirrer [18,19]. Commercial grade Alum, PaFC and PFS coagulants were used as received.

Optimization of pH and Dosage Treatments

60 grams of Alum, 20 mg/L of PaFC and PFS each were separately added into three sets of five beakers and Ph respectively adjusted to 4,6,7,8 and 10 using 1 N HCl and 40% NaOH. Experiments for each of the three coagulants were run in triplicates. Control samples were prepared using the same procedure without Ph adjustment. The samples were stirred rapidly at 200 rpm for 2 minutes followed by slow mixing at 30 rpm for 20 minutes to allow flocculation then allowed to settle for one hour. Supernatant samples were withdrawn for further analysis [18]. The sludge from each of the coagulant treatments was dried in beakers overnight in an oven at 105 oc or until constant weights were and respective percentage weights differences calculated. The same procedure was repeated but varying the coagulants dosages at the optimized pH for each of the coagulants respectively. COD and Turbidity for the clarified samples at optimal and maximum (optimal) coagulants dosages was tested. Results from each set of treatments were compared with those of the untreated sample.

Results and discussion

Physicochemical Analysis

Table 1: Physicochemical characteristics of the untreated waste water sample.

Parameter	Value
pH	10.23
Colour	Black
Electrical conductivity (EC) (mS/cm)	17.43
Salinity (psu)	10.22
Total Solids (ppm/mg/L)	89.710
Total dissolved solids (TDS) (ppm/mg/L)	81.575
Total Suspended Solids (ppm/mg/L)	8.135
Turbidity	17.52
COD (mg L-1)	1486
BOD (mg L-1)	4.2

Table 2: Effect of pH and dosage variation on treated dye waste water parameters.

Coagulant	pH	Sludge (g)	TDS (ppm)	Conductivity (mS/cm)	Salinity (psu)	Turbidity (NTU)	COD(mg L-1)
Alum	4	3.03	11.81	22.43	13.93		
	6	18.27	9.88	21.55	11.82	5.09	892
	7	17.47	11.94	21.67	14.64	2.49	467
	8	20.12	11.27	21.76	13.31		
	10	16.89	11.42	21.38	13.23		
PFS	4	7.29	11.68	23.33	14.16		
	6	7.55	11.67	23.30	14.14	12.98	4425
	7	7.84	12.14	24.53	15.09	4.06	2137
	8	7.12	11.86	23.20	14.45		
	10	6.63	11.67	22.20	13.82		
PaFC	4	3.13	8.32	11.6	9.69		
	6	5.75	6.86	10.47	7.75	4.06	6395
	7	1.20	7.73	10.66	8.10	0.39	7345
	8	1.06	7.17	11.27	8.35		
	10	1.81	7.85	12.55	9.18		
Coagulant	**Dosage**	**Sludge (g)**	**TDS (ppt)**	**Conductivity (mS/cm)**	**Salinity (psu)**	**Turbidity (NTU)**	**COD (mg L-1)**
Alum	30	8.21	21.87	43.6	26.26		
	40	16.01	27.35	54.625	32.54		
	50	17.65	34.29	68.55	41.30		
	60	22.64	46.72	93.2	56.6		
	70	29.73	49.48	94.4	56.72	30.3	3123
	80	33.45	50.1	100	59.52	24.47	1590
PFS	10	1.42	8.44	16.81	9.87		
	20	3.50	12.03	24.1	15.59		
	30	5.56	17.78	35.59	20.95		
	40	9.97	27.08	51.5	29.99	1.14	6206
	50	11.62	26.55	53.21	33.66	0.95	6081
PaFC	70		12.25	24.4	14.85		
	80		13.45	26.87	15.39		

	90		14.64	29.15	16.69		
	100		16.26	32.57	18.48		
	120		17.03	31.87	18.15	1.17	7371
	140		21.92	43.84	24.8	0.65	7287

Effect of ph and coagulant dosage variation on total dissolved solids (tds)

PaFC gave the lowest values of TDS in both pH and dosage variations. Alum yielded the highest TDS values that increased with increase in the coagulant's dosage. Alum and PFS gave similar results in TDS and their values remained constant with pH variation. Coagulant dosage variation of PaFC and PFS gave relatively lower values compared to those of Alum. Overall, PaFC was found to be the most effective in reduction of TDS at pH 6 and 280 mg/L. Figures 3.1 (a & b) show the effect of pH and coagulant dosage variations on the treated waste water TDS.

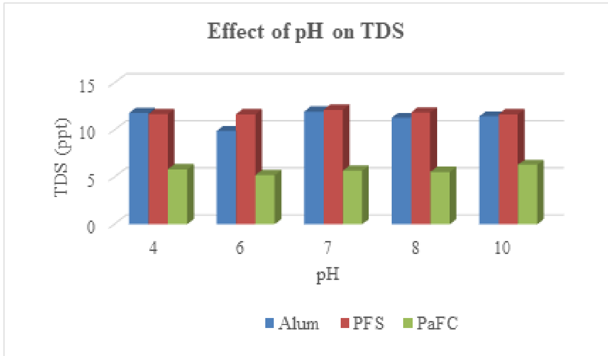

Figure 1(a): Effect of pH variation on TDS.

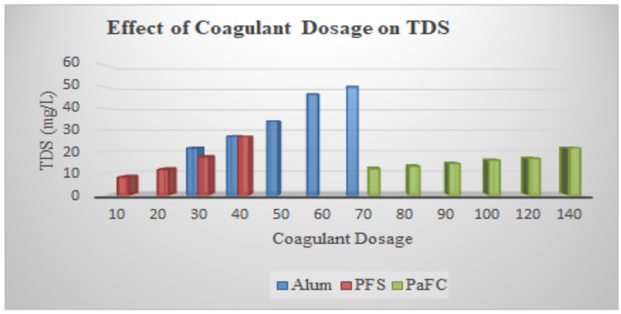

Figure 1(b): Effect of coagulant dosage variations on TDS.

Effect of ph and coagulant dosage variation on conductivity

PaFC gave the low values of conductivity across the pH range with the lowest 10.5 and 10.6 mS/cm being recorded at pH 6 and 7 respectively. Alum and PFS gave relatively similar conductivity results across the pH range. PaFC also had the lowest readings of conductivity values with dosage variations compared to Alum and PFS. Conductivity increased with dosages for all the coagulants. Figures 3.2 (a & b) show the effect of pH and coagulant dosage variations on the treated waste water conductivity.

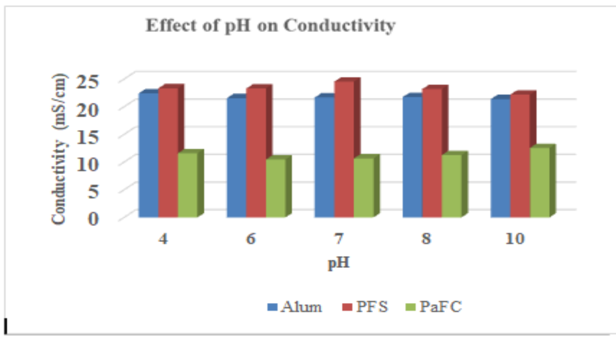

Figure 2(a): Effect of pH variation on conductivity.

Effect of ph and coagulant dosage variation on salinity

PaFC gave the lowest values of salinity across the pH and dosages compared to Alum and PFS. Alum and PFS on the other hand gave relatively similar salinity results across the pH range. The salinity values increased proportionally with Alum and PFS dosages as shown in Figure 3.3 (a & b).

Effect of ph and coagulant dosage variation on sludge generation

Alum generated the highest sludge that increased with rise in pH, while PFS generated comparatively high sludge but the amounts remained relatively constant across the pH range. Comparatively, PaFC gave lowest amounts of sludge with the highest value of 5.75 being recorded at pH 6 as shown in Table 3.2. Figure 3.4 illustrates comparison of the sludge generation for the three coagulants. The amount of sludge generation by Alum and PFS increased proportionally with the coagulants dosages. Sludge generated by variation of PaFC dosage was negligible hence not determined.

2021
Vol.12 No.9:43

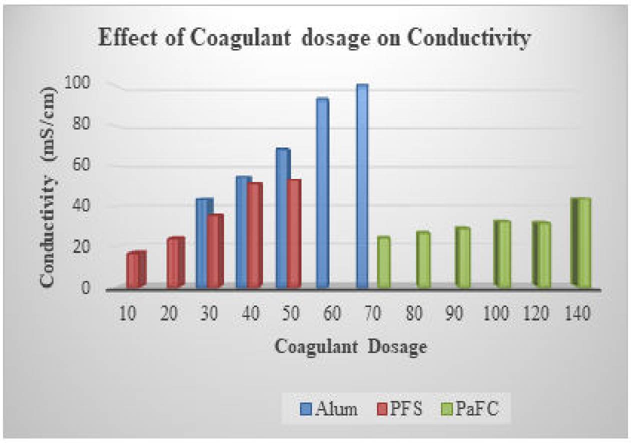

Figure 2(b): Effect of coagulant dosage variation on conductivity.

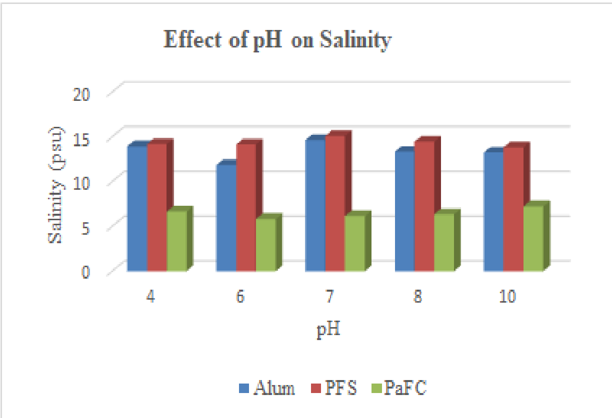

Figure 3(a): Effect of pH variation on salinity.

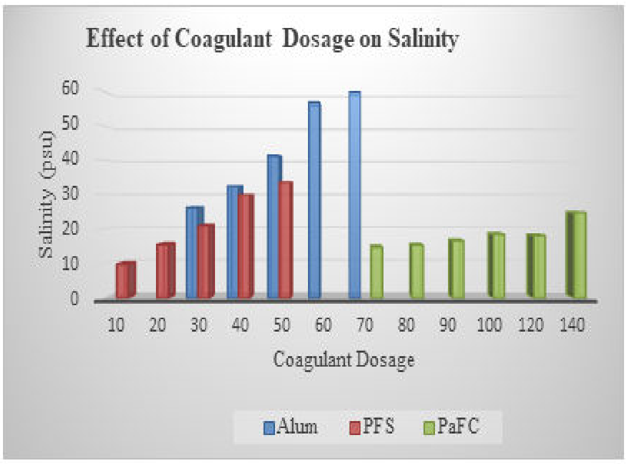

Figure 3(b): Effect of coagulant dosage variations on salinity.

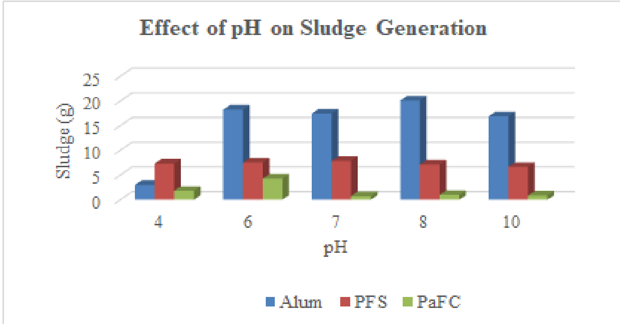

Figure 4(a): Effect of pH variation on sludge generation.

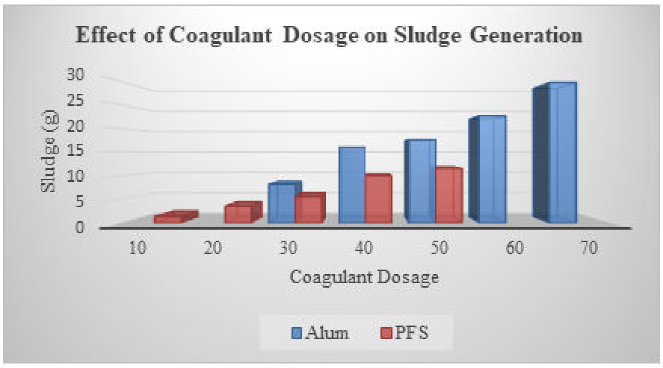

Figure 4(b): Effect of coagulant dosage variation on sludge generation.

Treatment of the dye waste water at varied pH (4-10) with Alum cleared the dye at pH 6, 7 and 10. PFS samples clarified the at pH 6- 8 while those treated with PaFC clarified at pH 4-8 as shown in Figures 3.4, 3.5 and 3.6 respectively. These results relate with those reported by Gao, Yue and Miao (2001) indicating that PFS and PaFC are efficient coagulants that work under broad pH range with the formation of rapidly settling flocs.

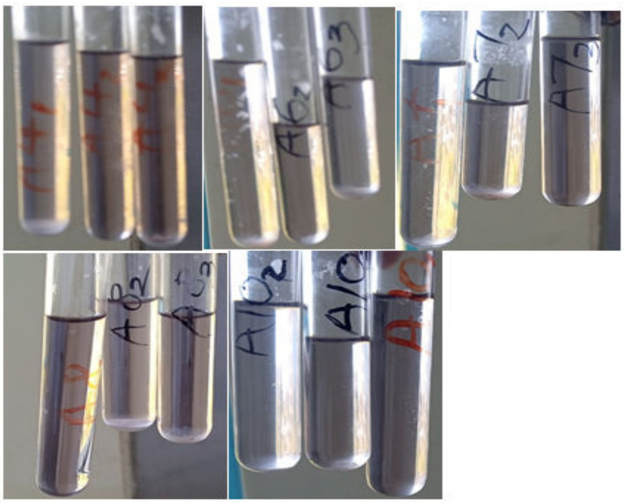

Figure 4: Treatment of Reactive Super Black dye waste water with Alum at varied pH.

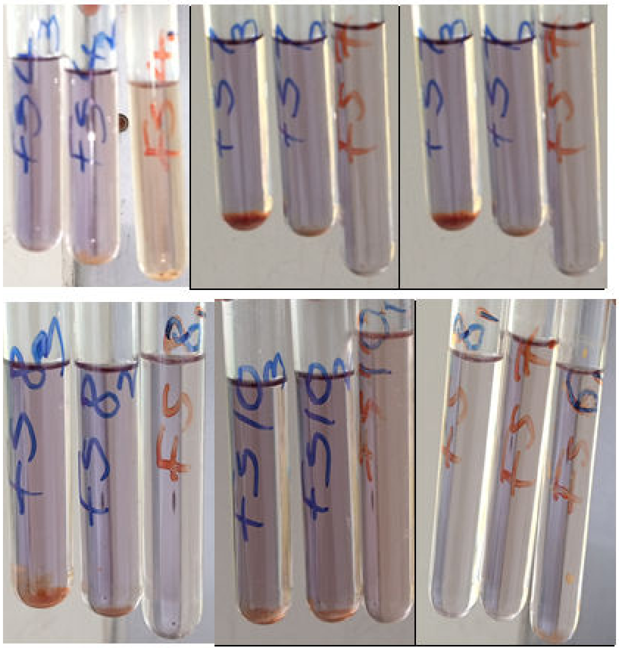

Figure 5: Treatment of Reactive Super Black dye waste water with PFS at varied pH.

Figure 6(b): Treatment of Reactive Super Black dye waste water with PaFC at varied pH.

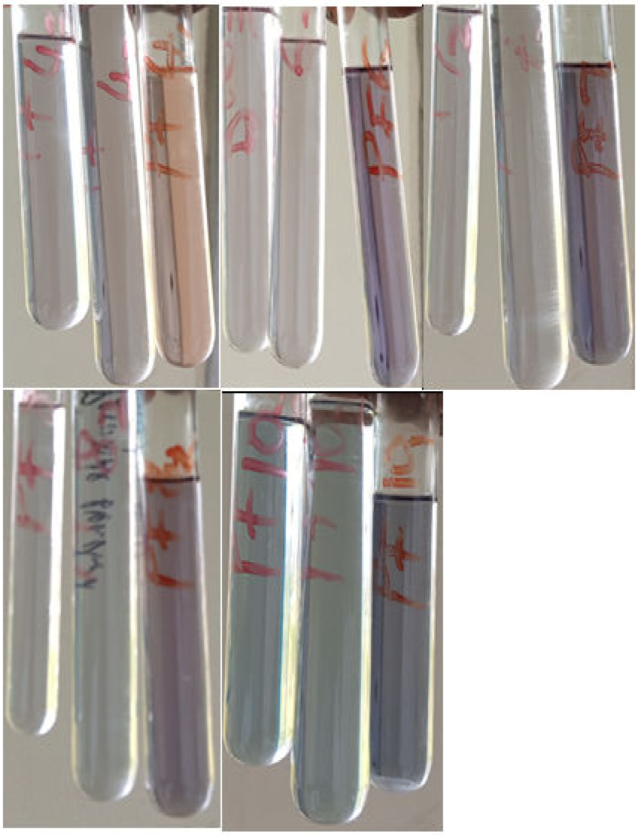

Figure 6(a): Treatment of Reactive Super Black dye waste water with PaFC at varied pH.

Clari ication of the dye samples a ter treatments with the three coagulants was observed to be proportional to the coagulants' dosages as shown in Figure 3.7 (a, b & c). Alum and PFS clari ied the dye at 120 g/L and 80 mg/L respectively. PaFC indicated slight clari ication at 280 mg/L, gradual addition of the coagulant dose did not give signi icant dye clari ication. This inding relates with that reported by Ashtekar et al. (2015), who observed that raising coagulant dose for PaFC had a negligible effect on reactive dye colour removal.

Figure 7(a): Effect of Alum dosage variation on dye removal.

Figure 7(b): Effect of PFS dosage variation on dye removal.

Figure 7(c): Effect of PaFC dosage variations on dye removal.

Consequently, COD for the clarified samples at optimal pH 6 and 7 and maximum (optimal) coagulants dosages was obtained as shown in Figure 3.8 (a & b). PaFC gave the highest COD values followed by PFS. pH 7 gave relatively lower values compared to pH 6 across all the coagulants. COD values decreased with rise in coagulant dosages.

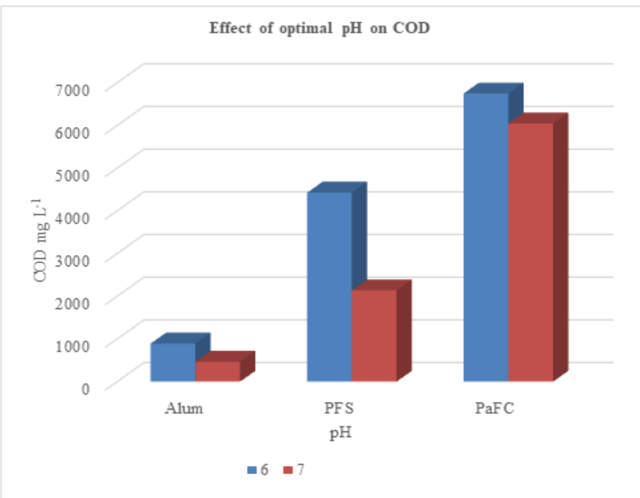

Figure 8(a): Effect of optimal pH on COD.

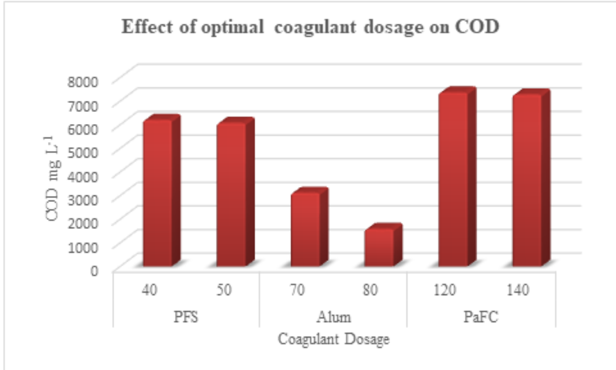

Figure 8(b): Effect optimal coagulant dosage on COD.

Turbidity for the optimal/clarified samples at pH 6 and 7 and corresponding coagulants optimal dosages were as shown in Figure 3.9 (a & b). pH 7 was found to give the lowest turbidity for all the coagulants as shown Table 3.2.

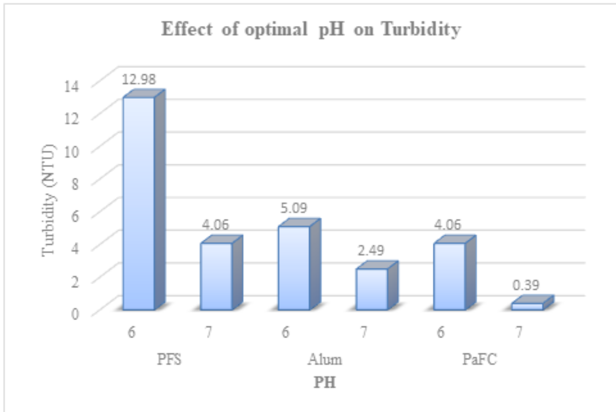

Figure 9(a): Effect of optimal pH on turbidity.

Alum was found to give the highest turbidity values compared to PFS and PaFC. Turbidity from all the coagulants decreased with the dosage increase as shown in Figure 3.9 (b).

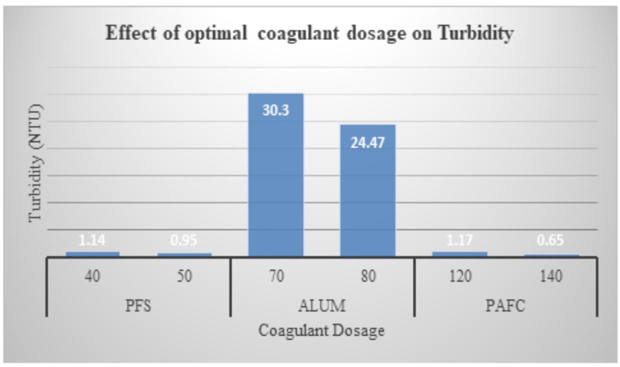

Figure 9(b): Effect of optimal coagulant dosage on turbidity.

Colour concentration tests were done by analyzing the treated samples using UV-Vis spectrophotometer at 600 nm.

Figure 3.10 (a & b) shows the comparative analysis of the dye concentrations of treated samples at varied pH and coagulant dosages using UV-Vis Spectrophotometer. The dye concentration was found to be minimal at pH 7 for PFS while Alum gave lowest concentrations at pH 10 and PaFC at pH 4 as shown in Figure 3.10 (a).

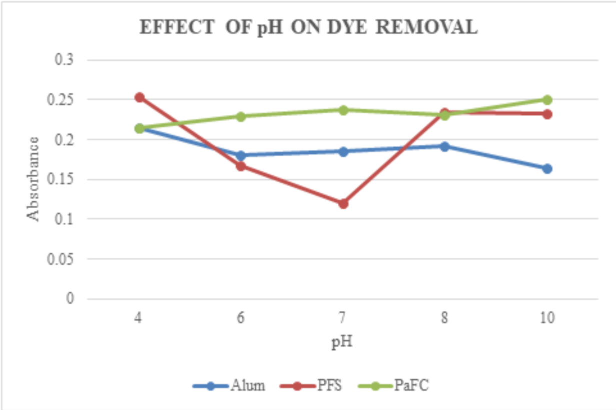

Figure 10 (a): Effect of pH on dye removal.

The dye concentration reduced with increase in all the coagulants dosages as shown by Fig 3.10 (b), which is confirmed by the photos in Figure 3.7 (a, b & c).

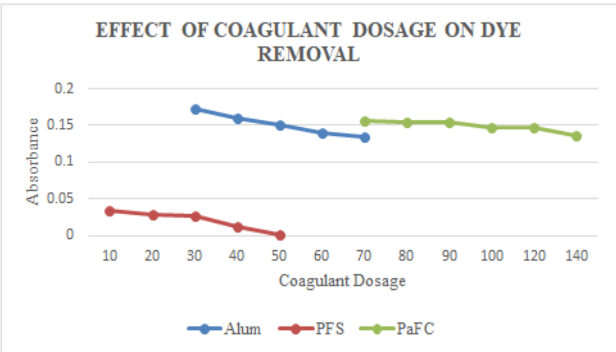

Figure 10(b): Effect of Coagulant Dosage on Dye Removal.

Conclusion

Treatment of Reactive Black textile dye waste water with variation of pH and coagulant dosages showed that PaFC gave the lowest values of TDS, conductivity, salinity and sludge generation compared to PFS and Alum which gave relatively similar results. As reported by [18], PaFC generated negligible sludge compared to Alum and PFS. Alum however, gave the highest amount of the sludge generated with variation of both the pH and coagulant dosages. PaFC also gave the lowest readings of turbidity for both optimal pH and dosages treatments compared to PFS and Alum. While PaFC was found to give the highest COD at the optimal dosage and pH treatments, Alum on the other hand gave the lowest COD at the optimal pH treatments. These findings relates with those reported by [20,21]. that Prehydrolysed coagulants such as Polyaluminium Chloride (PaC), Polyaluminium ferric chloride (PaFC), Polyferrous Sulphate (PFS) and Polyferric Chloride (PaFC) seem to give better colour removal even at low temperature and may also produce lower volume of sludge. On the contrary, while PaFC indicated to be the best coagulant for the Reactive Super Black dye waste water coagulation by the chemical parameters analysed, it performed poorly in terms clarification of the dye both from visual observation and UV-Vis Spectrophotometer test results.

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