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## Treatment of Yarn Dyeing Wastewater Using Different Coagulants Followed by Activated Carbon Adsorption

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### Abstract

Yarn dyeing industry consumes large volumes of water and chemicals for wet processing of yarn. The chemical reagents used are very diverse in chemical composition, ranging from inorganic and organic compounds to polymers. The major environmental problem of colorant manufacturing is the removal of dyes from effluents. In this study, the potential of using different coagulants such as alum, ferric chloride, and magnesium chloride were investigated for the treatment of dyeing wastewater. Alum, magnesium chloride and ferric chloride were tested. Lime, cationic and anionic polymers were used as a coagulant aid. To achieve better performance post treatment using granule activated carbon (GAC) was applied as an adsorbent. The use of 500 mg/l alum aided with 400 mg/l lime and 0.35 mg/l cationic polymer achieved the highest removal rate followed by magnesium chloride then ferric chloride. The removal rates of COD, TSS, and turbidity were 69.5 %, 70.7 % and 96.9 %, respectively. Post treatment using GAC enhanced the color and COD removals. Their removal rates reached 94.6% and 77.5%, respectively. Based on the laboratory studies, design, construction and implementation of a full scale pilot plant was executed. The treated effluent satisfied the international regulatory standards of such industry for wastewater discharge into the public sewage network.

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## **1. Introduction**

Textile industries are one of the biggest users of water and complex chemicals during textile processing at various processing stages [1]. The unused materials from the processes are discharged as wastewater that is high in color, biochemical oxygen demand (BOD), chemical oxygen demand (COD), pH, temperature, turbidity and toxic chemicals. The direct discharge of this wastewater into the water bodies like lakes, rivers, etc. pollutes the water and affects the flora and fauna. Effluent from textile industries contains different types of dyes, which because of high molecular weight and complex structures, shows very low biodegradability [2]. Also, the direct discharge of this industrial effluent into sewage networks produces disturbances in biological treatment processes [3]. These effluents produce high concentrations of inorganic salts, acids and bases in biological reactors leading to the increase of treatment costs [4].

Many methods have been reported for removing color from dye wastewater, among which coagulation is a widely used process. Coagulation–flocculation is a commonly used process in water and wastewater treatment due to its high efficiency and low cost, and it has been proved effective in dye removal, especially dissolved dyes in wastewater [5]. Besides, dyes can be removed by coagulation–flocculation without decomposition, which may produce more harmful compounds [6]. Coagulation–flocculation process, is active for suspended matter, colloidal type of very small size, their electrical charge gives repulsion and prevent their aggregation. Adding electrolytic products in water, such as aluminum sulfate, ferric sulfate, ferric chloride, giving hydrolysable metallic ions or organic hydrolysable polymers (polyelectrolyte) can eliminate the surface electrical charges of the colloids. Normally the colloids bring negative charges so the coagulants are usually inorganic or organic cationic coagulants (with a positive charge in the water) [7]. Also, the metallic hydroxides and the organic polymers can help the particle aggregation into flocks, thereby increasing the sedimentation.

To date, various coagulant categories have been commercially available, including inorganic coagulants, synthetic polymer flocculants and inorganic–organic dual-coagulants [8]. Aluminum salts are the most widely used inorganic coagulants due to their high treatment efficiency and low cost [1]. According to the double layer theory, electrical repulsion forces prevent from the colloid aggregation. In order to achieve an effective agglomeration, the compression of the thickness of the electrical double layer or a charge reduction of the particles has to be carried out. This implies the zeta potential reduction [9]. In this way the colloids can be settled. Jar-tests are a valuable tool in wastewater treatment to evaluate the efficiency of the physicochemical treatment [8]. The optimum operating conditions (pH, chemical concentrations) are determined by means of these experiments.

In order to achieve a good quality effluent, post treatment is required. Adsorption is the most used method in physicochemical wastewater treatment after coagulation. This process can mix the wastewater and the porous material powder or granules, such as activated carbon or clay. In this study, treatment of dyeing and finishing ready garment wastewater was elaborated using different coagulants followed by activated carbon adsorbent.

The ultimate goal is to have a treated effluent complying with the national regulatory standards for wastewater discharge into the public sewage network.

## 2. Materials And Methods

### 2.1 Description of the industrial dyeing process.

Wastewater was collected from a factory located in an industrial city eastern Cairo, Egypt. It produces different types of sewing or sewing of garments, leather wear, embroidery, polyester / cotton core th read and polyester core-thread. The production rate is 1800-2040 ton thread/year. The factory has 21 dyeing machines with capacities, ranged from 7L to 800 L. In the dyeing process several chemicals are used such as dispersed dyes, hydrogen peroxide, acetic acid, caustic soda, sodium hydrosulphite, and sodium chloride and leveling agents. The wastewater produced from manufacturing processes is 600 m<sup>3</sup>/day and discharged directly into the sewerage network without treatment. Fig. 1 illustrates the manufacturing process and the wastewater resulted from each step.

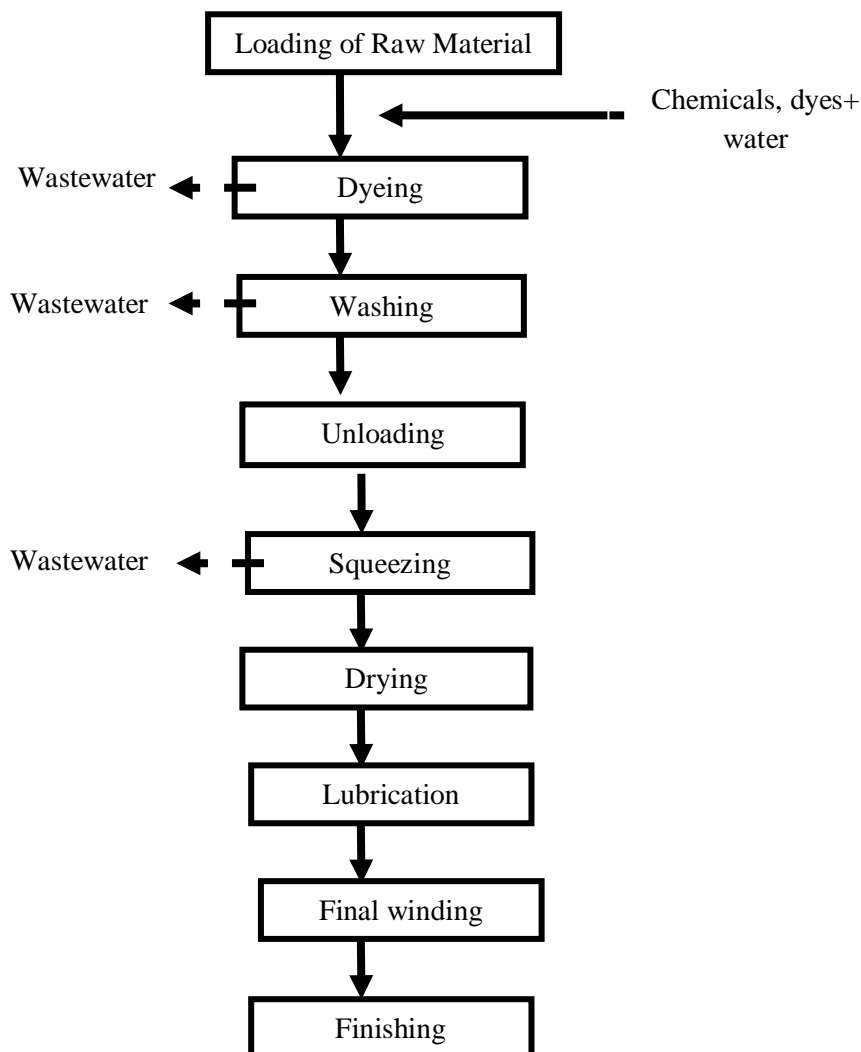


Fig.1: Dyeing manufacturing processes

## **2.2 Collection of samples and analysis**

Due to the great variations in the quantity and the quality of wastewater produced during the working day , a continuous monitoring program was carried out to identify the quality and quantity of wastewater discharged. Composite samples were collected during the working shifts for almost four weeks and then were subjected to physic-chemical analysis according to "Standard methods for water and wastewater, APHA, 2005 [10]. The analysis included, pH, color, total suspended solids (TSS) settleable solids, chemical oxygen demand (COD), biological oxygen demand (BOD), total phosphorous (T.P), total Kjeldahl nitrogen (T.K.N), oil and grease, total sulfide (H<sub>2</sub>S) and ammonia-nitrogen.

## **2.3 Chemical coagulation of the end-off-pipe**

Chemical coagulation of the end-off-pipe was carried out using different coagulants such as alum, ferric sulfate and ferric chloride. Lime, cationic and anionic polymer are used as a coagulant aid. Organic coagulants including poly electrolytes synthetic polymers and natural polymers were also used for coagulation process. Determination of the optimum doses and pH of each coagulant was determined.

## **2.4 Jar test**

Jar testing is a method of simulating a full-scale wastewater treatment process, so operators can use jar testing to help in determination of optimum operating conditions to get the best results. Jar testing entails adjusting the amount of chemicals and the sequence in which they are added to samples of raw water held in jars. The sample is then stirred so that the formation, development and settlement of flocs can be watched just as it would be in the full-scale treatment plant. A series of tests were performed to compare the effects of different amounts of flocculating agents at different pH values. The procedure was performed several times so that the optimum pH and dose of coagulant can be calculated. After each run the supernatant was withdrawn for analysis.

## **2.5 Post treatment using granule activated carbon (GAC)**

Post treatment was applied after coagulation by using GAC as adsorbent. Studies were conducted at room temperature ( $25 \pm 0.1^{\circ}\text{C}$ ) to determine the effects of GAC sorbent dosage and contact time on the adsorption of residual pollutants and color removal. Each experiment was conducted using a mechanical shaker at 200 rpm. Samples were taken at different time intervals for analysis. All samples were filtered through Whatman filter paper (No. 42).

# **3. Results and Discussions**

## **3.1 Characterization of wastewater**

Analyses of wastewater from End-off-pipe are recorded in Table 1. The results indicated that the quality of effluent is not complying with both the national regulatory standards and the factory international regulation for discharge into public sewerage network. The concentration of chemical oxygen demand (COD), ranged from

1020 to 1382 mgO<sub>2</sub>/l. While biological oxygen demands, (BOD) reached 570 mgO<sub>2</sub>/l. The total suspended solids ranged from 14.7 to 28 mg/l. In addition, the concentration of oil and grease were ranged from 70 to 191 mg/l, respectively. This violating value mainly attributed to the colored materials extracted by chloroform.

Table-1: Characteristics of raw wastewater

Parameters	Unit	Min	Max	Average	International standards
pH-value	--	7.2	9.4	--	6-9
Color	Pt-Co	3600	4500	3900	---
Chemical Oxygen Demand	mg/l	1020	1382	1189.4	500
Biochemical Oxygen Demand	mg/l	420	570	490	300
Total Suspended Solids	mg/l	14.7	28	20.04	400
Oil , Grease and all Extractable matters by Chloroform	mg/l	70	191	116.56	20
Total Kjeldhal Nitrogen	mg/l	24	60.4	37.56	--
Total Phosphorous	mg/l	2.8	4.1	3.56	--
Total Sulphide	mg/l	6	166	55.4	1
Ammonia	mg/l	1.6	8	4.825	--

### 3.2 Effect of using different coagulants

A comparative study between the three coagulants under investigation was carried out to obtain the best coagulant. Trial jar test experiments for each coagulant using different doses were carried out at their optimum pH. Each coagulant was aided with lime to accelerate the coagulation process. The pH was raised to 11 by adding 400 mg/l of lime, then lowered to the optimum pH of each coagulant. The COD and pH were measured for each coagulant. Fig. 2 shows the effect of different coagulants in the quality of treated effluent at their optimum conditions. It is obvious from Fig. 2 that the using of alum aided with lime gave the best removal efficiency.

In Coagulation process, Al<sup>+3</sup> starts to hydrolyze and forms monomeric Al hydrolyzed species. Hence, soluble cations such as Al<sup>+3</sup>, Al (OH)<sup>+2</sup> and Al (OH)<sup>2+</sup> are predominant in the solution. They play an important role in destabilizing the negatively charged dye particles via charge neutralization [11]. At around neutral pH, Al (III) has limited solubility because of the precipitation of amorphous hydroxide, which leads to the possibility of sweep flocculation as impurities (i.e., dye particles) enmesh in the growing precipitates and are effectively removed [12]. The disadvantage of uses magnesium chloride (MgCl<sub>2</sub>) was its high price and it needs for pH adjusting before disposal as it produced treated effluent with a pH value of 12. This is probably due to the strongly hydrated free Mg ions that predominate in the neutral pH range and the decrease in concentration of these species as pH and the concentration of the hydrolyzed species of Mg<sup>+2</sup> increase [11]. Magnesium ions in the Mg(OH) form reduce the negative charges on dye particles and eventually reverse the negative charge to

positive as Mg(OH) precipitates on the dye particle surface. Therefore, the percentage of COD removal increases as pH increases from 9.0 to 11.0 and the most effective coagulation-flocculation process can be achieved in the pH range of 10.5 to 11. Moreover, the use of ferric chloride as a coagulant causes corrosion for the pipeline of the treatment plant.

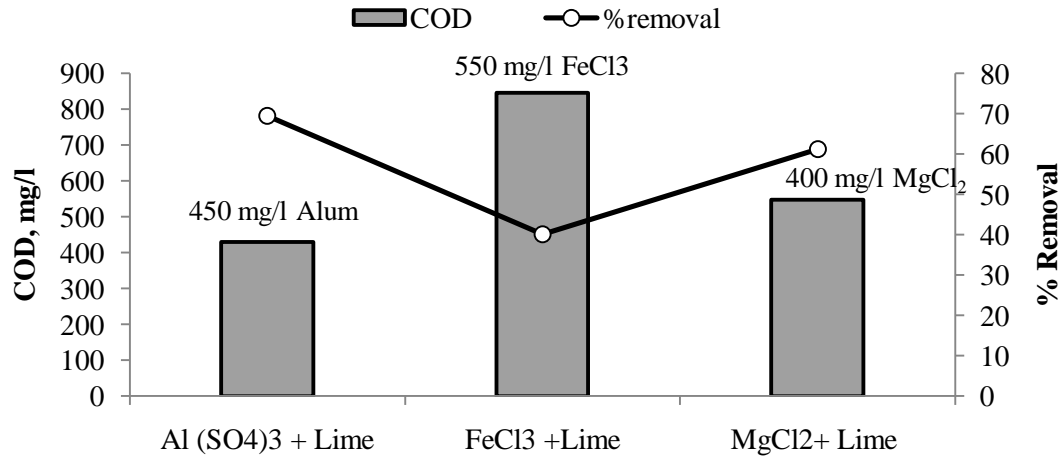


Fig.2: Effect of different coagulants on cod removal

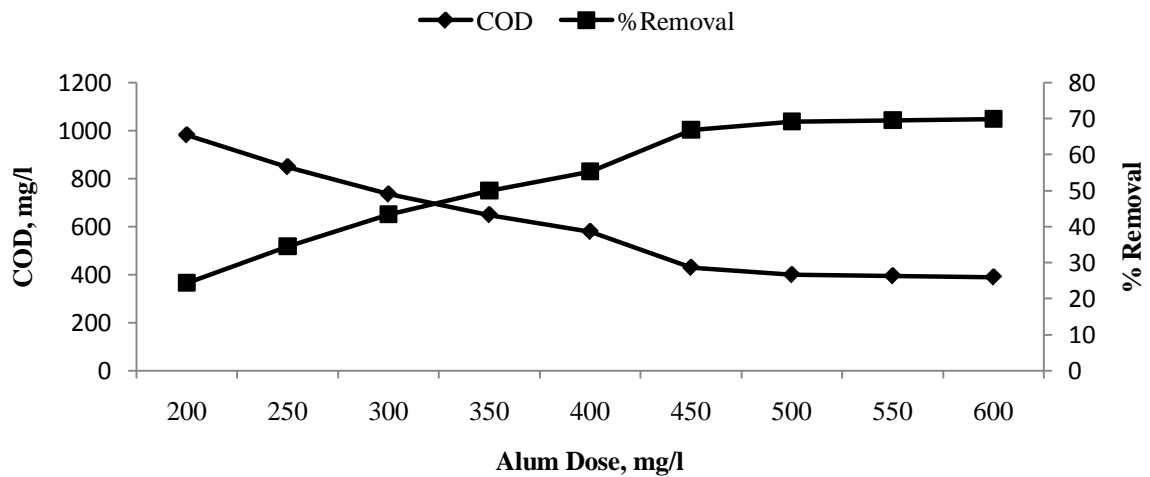


Fig.3: Effect of different doses of alum on COD removal

Accordingly, more investigation using alum was carried out. To select the optimum dose of alum, which can be used for the treatment, a series of runs were carried out. They were done at a fixed dose of lime (400 mg/l) and different doses of alum ranged from 200 mg/l to 650 mg/l. Fig.3 shows the effect of using different doses on the removal of COD. It is obvious that the optimum dose of alum that gave the best removal efficiency of COD was 500 mg/l at pH 7.5. However, the sludge volume was high (154 ml/l).

### 3.3 *Effect of polymer type*

In order to improve the quality of the effluent as well as settlability of the sludge, cationic and anionic polymers were added with the coagulants. Studying the influence of different polyelectrolyte on the settling performance was represented in Table 2.

Table2: Effect of type of polyelectrolyte on quality of treated wastewater

<b>Parameters</b>	<b>Unit</b>	<b>Alum + Lime</b>	<b>Alum + Lime + Cationic polymer</b>	<b>Alum + Lime + Anionic polymer</b>
Dose of polymer	mg/l	--	0.3	0.35
pH-value	--	7.5	7.16	7.25
Turbidity	NTU	16	1.8	1.6
Color	Co-Pt	1320	1200	980
COD	mg/l	430	500	410
Oil and grease and all Extractable matters by Chloroform	mg/l	56	50	35
Volume of Sludge	ml /l	154	90	80

From the results depicted in Table 2, the use of anionic polymer (0.35 mg/l) was more efficient in settling performance and also in the reduction of COD concentration and color than cationic polymer. Also, the use of anionic polymer decreased the sludge volume to 74 ml/l.

### 3.4 **Post-treatment using granule activated carbon**

Post treatment using GAC was used to improve the removal efficiency of residual organic matter as well as color. The adsorption of organic compounds from treated effluent by activated carbon (AC) may result from solute hydrophobicity [13].

#### 3.4.1 *Effect of sorbent time*

At equilibrium, time is one of the important parameters for selecting a wastewater treatment system. As shown in Fig. 4, the COD removal was rapid at the first 30 min. and equilibrium was reached within 45 min. Therefore,

the period of 45 min. was considered as the optimum time, where the residual value of COD reached 270 mg/l while 94% of color was removed.

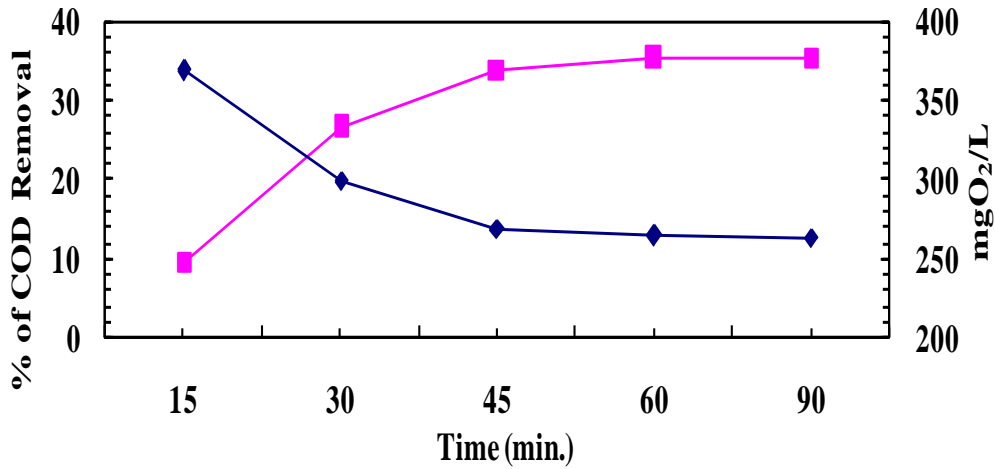


Fig. 4: Effect of contact time on the removal of cod by adsorption onto GAC

### 3.4.2 Effect of sorbent dosage

Several experiments were carried out using different doses of GAC (0.1 - 0.5 g) with a fixed volume of 100 ml of chemically treated effluent. At the equilibration time of 45min (Fig.5). The percentage removal of COD varied linearly with the amount of the adsorbent and amount of adsorbate. The increase in adsorption rate (A %) with an increase in adsorbent dosage is due to the availability of larger surface area and more adsorption sites. At adsorbent dosage >3g the incremental of COD removal became very low. Thus, increased adsorbent dosage did not enhance the COD removal percentage.

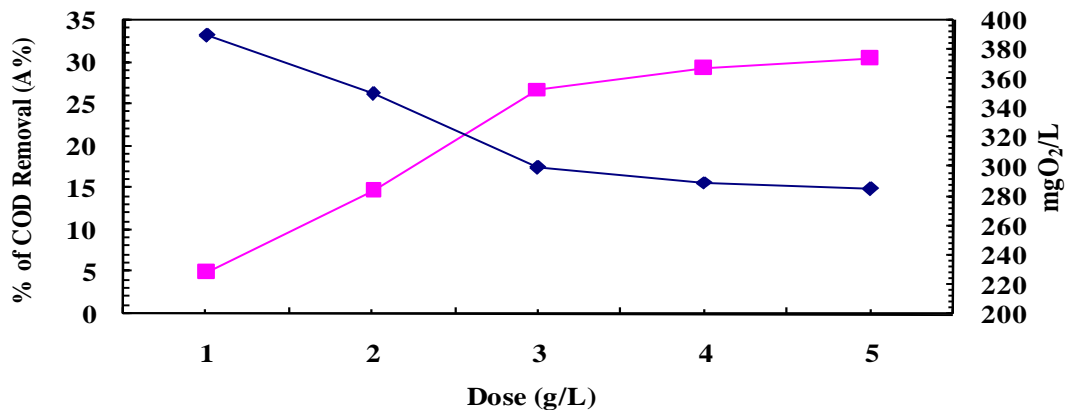


Fig.5: Effect of adsorbent dose on the removal of COD by GAC

### 3.5 Overall efficiency of the treatment system



The results obtained indicated that the quality of effluent is complying with both the national and international environmental legislation. The final effluent was treated using chemical coagulation techniques followed by adsorption on activated carbon. Table 3 shows the quality of the final effluent.

Table-3: Overall treatment efficiency of the integrated treatment module

Parameters	Unit	Raw Wastewater	Treated Effluent	% Removal	International standards
pH-value	--	8.54	7.5	--	6-9
Turbidity	NTU	48	1.6	96	--
Color	Co-Pt	3900	207	94.6	--
Chemical Oxygen Demand	mg O <sub>2</sub> /l	1189.4	270	77.5	500
Biochemical Oxygen Demand	mg O <sub>2</sub> /l	490	130	70	300
Total Suspended Solids	mg/l	20.04	8	71	400
Oil , Grease and all extractable matters by chloroform	mg/l	116.56	16.7	84	20
Total Sulphide	mg S/l	55.4	0.6	97	1
Total Kjeldhal Nitrogen	mg/l	37.5	5.6	85	-
Ammonia	mg/l	4.8	N.D	100	-

### **3.6 The full scale plant design and construction**

#### **3.6.1 Design Consideration**

The main objective of the laboratory scale study was to develop the information needed for the design of a full scale treatment system. Based on extensive laboratory scale test findings, a full scale plant (600 m<sup>3</sup>/day) has been designed. Our goal was to design a system capable of producing effluent meeting the requirements for wastewater discharge into the public sewage network. A flow diagram of the treatment plant is shown in Fig. 6.

#### **3.6.2 Optimum operating conditions and design criteria**

Based on the results of the laboratory scale, optimum operating conditions and design criteria for full scale treatment plant were developed as presented in Table 4.

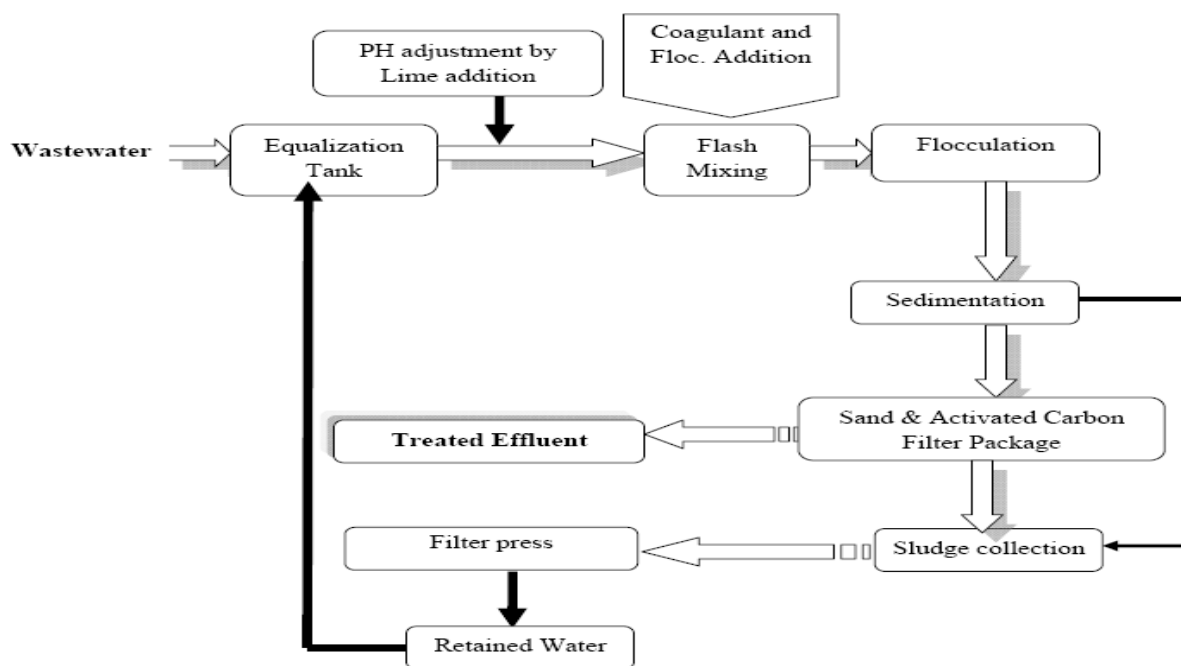


Fig.6: Flow Diagram Of The Treatment Process

Table-4: Optimum operating conditions and design criteria for the waste water treatment plant

Parameters	Results
Wastewater discharge	600 m <sup>3</sup> /day
Flow Rate	25 m <sup>3</sup> /hr
Temperature of end off-pipe effluent	70 °C
pH	Raised to 10 by CaO, then lowered to 7 ±0.2 by alum
Dose of lime	400 g/ m <sup>3</sup>
Dose of alum	500-550 g/ m <sup>3</sup>
Dose of anionic polymer	0.35 g/ m <sup>3</sup>
Settling Time	30 min
<u>Sludge analysis</u>	
Sludge volume	75-80 l/ m <sup>3</sup>
Total Sludge weight	4.8-5.0 g/l
Inorganic Sludge weight	2.14- 2.4 g/l
Sludge Volume Index	15.6-16

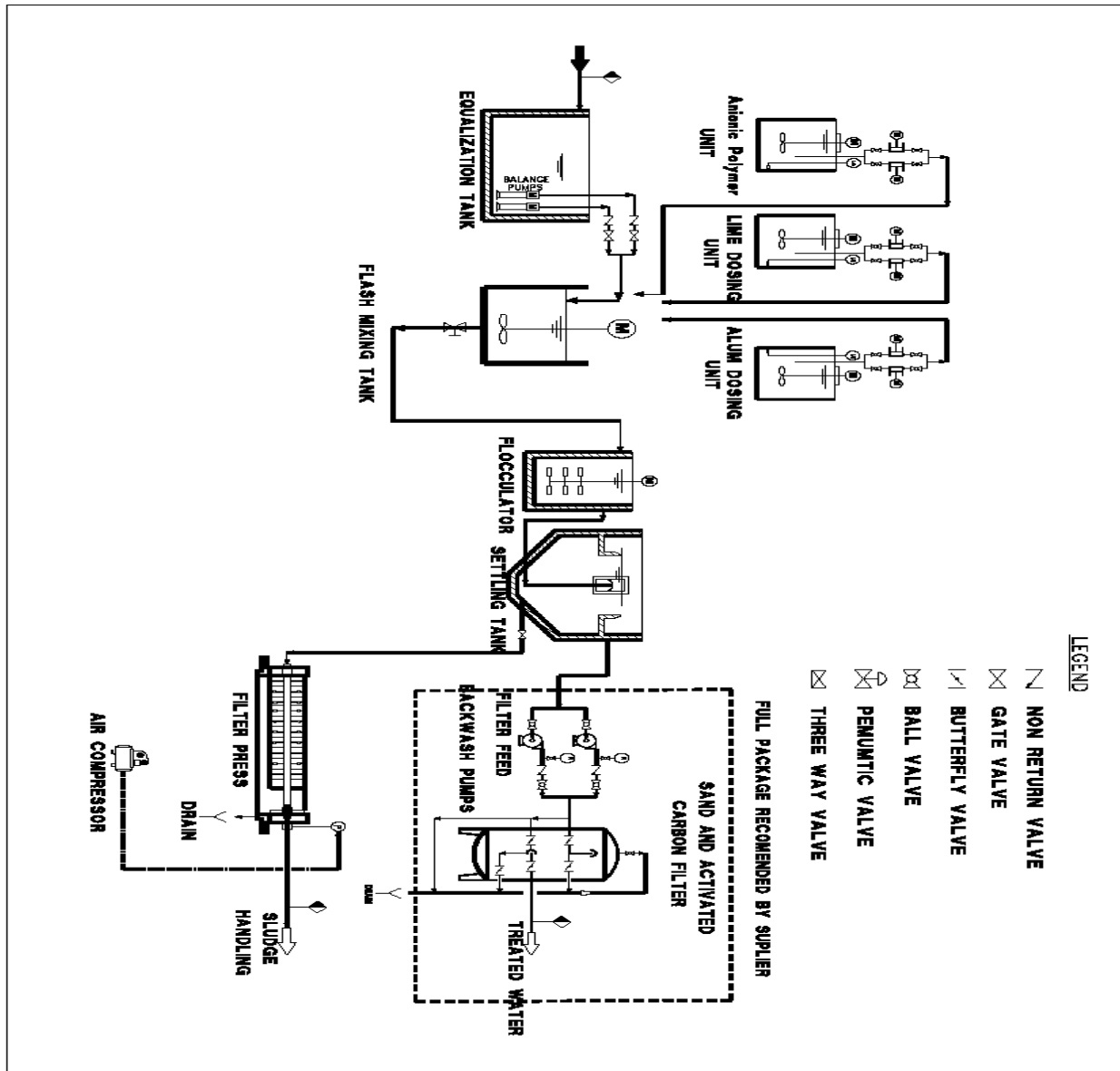


Fig. 7. The main components of the treatment system

### 3.6.3 End - Off -Pipe wastewater treatment components

Both domestic and industrial wastewater (Dying, Washing, Squeezing, Machine cleaning, Floor washing, Laboratories, Municipal wastewater, and Boiler discharges) were collected in existing settling tank which has been utilized to act as equalization tank. Fig.7 shows the main treatment system components. The suggested treatment sequence of the end-off-pipe effluent from the factory comprises the following:

- Equalization tank for wastewater retention to ensure constant quality.
- Clarification tank (flash mixing tank, flocculation tank, sedimentation tank).
- Chemical equipment (anionic polymer, alum sulfate solution, lime solution).
- Sand and activated carbon filter compact package.

- Filtering processes (filter press)

Flocculation is the process that brings the colloid particles together so they can settle in the clarifier. The mixing must be very precise: an inadequate amount of mixing energy will cause settling in the flocculation basin; too much energy will cause the floc particles to break [14]. As the water moves through the basin, the velocity gradient (G) should decrease to prevent the flocs from breaking. Equation (1) describes the velocity gradient. G is a function of volumetric flow rate (Q), head loss in the flocculator ( $\Delta H$ ), water's density ( $\rho$ ), viscosity ( $\mu$ ), and volume of the tank (V). In this system a variable speed motor/gear reducer drives a shaft fitted with wooden paddles cut to the proper size to deliver a range of velocity gradients in the flocculation tank.

$$G = \sqrt{\left(\frac{Q\rho g \Delta H}{\mu V}\right)} \quad \text{Eq.1}$$

The design considered to move the existing settling tank to be operated as an equalization tank to guarantee the full mixing of the domestic wastewater discharges with the industrial wastewater discharges from the different process streams. The collected wastewater flows were pumped to the clarification tank which consisted of flash mixing unit, and coagulation unit and then pumped to the settling tank.

#### 4. Conclusion

Wastewater produced from dyeing yarn blends were highly contaminated with organic and inorganic pollutant as well as color. Treatment of this wastewater using different coagulants indicated that the use of 500 mg/l alum aided with 400 mg/l lime and 0.35 mg/l anionic polymer are capable of removing 69%, 65% and 61% of COD, BOD and color. However, post treatment with granulated activated carbon at 45 minute contact time and 2.5g/l dose improved the quality of treated effluent by 8.5%, 5%, and 33% COD, BOD and color.

The overall removal efficiency of COD, BOD and color reached 77.5%, 70% and 94.2% respectively. Based on the laboratory studies, design, construction and implementation of a full scale pilot plant was executed. The treated effluent satisfied the international regulatory standards of such industry for wastewater discharge into public sewage.

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