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Assessing the Effectiveness and Environmental Sustainability of Reactive Dyes for Their Structural Diversity

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Article

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ABSTRACT

This study aims to assess the environmental impact of different chemical classes of reactive dyes and their treatment. To attain this outcome, four different types of reactive dyes (Procion Blue PX-3R (Monochlorotriazine), Levafix Blue CA (Bi-functional heterogeneous), Remazol Brilliant Blue R (Vinylsulphone) and Remazol Brilliant Blue RR (Bi-functional homogeneous) on the cotton fabric were analyzed. The potential cytotoxic effect of different reactive dyes was evaluated using the growth inhibition test according to AATCC 22662 by exposing the culture over 72 hours at 21 °C. The dyeing operation was performed in a labrotex sample dyeing machine, and a data color spectrophotometer was administered to analyze the strength percentage of dyes in post-dye liquor. Among the reactive dyes, Levafix Blue CA exhibited better effluent characteristics compared to others, lowering the threat to the environment. The dyed wastewater was directly disposed of and treated in a pilot plant containing lime and ferrous sulfate as coagulant and polyacrylamide as polyelectrolyte, which resulted in a significant reduction of BOD₅ (64.97-74.67%), COD (61.03-70.28%), TDS (55.02-61.97%) and TSS (47.07-50.19%) with improved pH and DO values. This investigation reflects the specific selection of a reactive dye leading to a lower environmental impact of a dyeing process coupled with wastewater treatment processes.

KEYWORDS

reactive dyes, pollution, effluent treatment plant (ETP), dye structure, environmental sustainability

INTRODUCTION

Though the textile industry is well-known for its anthropogenic activities, it is highly accountable for a broad list of adverse impacts to the surroundings [1]. This industry releases an enormous amount of wastewater each year from different steps of textile wet processing, including pretreatment, dyeing, and finishing operations [2-4]. According to a study, untreated effluents discharged from textile industries normally comprised 80% of the total outflows delivered by this industry. The dyeing sector is one of the worst polluters of the environment as textile dyeing processes extensively use synthetic dyestuffs, which produce highly colored effluents with toxic wastes [5]. Ultimately this heavily polluted

wastewater easily finds its way into the water bodies [6] and rapidly spreads, ruining the natural environment.

Among all of these colors, reactive dyes are considered as the foremost widespread and compelling class of advanced synthetic dyes, thanks to their splendid fastness properties, convenient application due to the formation of strong covalent bonds with fabrics, and a variety of excellent bright shades on cellulosic fibers [7]. However, reactive dyes are prone to hydrolysis reaction with water, during which a huge number of dyes are segregated from the fabrics. The rate of hydrolysis can be significantly reduced by using bi- or multifunctional reactive dyes, but the problem still exists due to containing a large amount of colorant, some inorganics, lower DO, higher BOD₅, COD, TSS, and TDS in effluents [8-10]. For instance, it was found that the colors associated with reactive dyeing in the wastewater are responsible for aesthetic damage to the water bodies [11]. The dye-contaminated wastewater blocks the light from penetration and complicates oxygen transfer into the water bodies, hence affecting the entire aquatic plant and animal life as well as other organisms [12-14]. Some of the research showed that the presence of reactive dyes even at a lower concentration (<1 mg/l) may lead to serious threats for the aquatic life.

To combat this problem of reactive dye pollution, the industrial effluent must be treated before being discharged into the environment. However, the removal of the reactive dyestuffs is a very difficult task as these dyes are designed to resist biodegradation due to their origin, complex aromatic structure, and high molecular weight [15]. Consequently, they remain in the environment for a long period of time. For example, the half-life of the hydrolyzed dye Reactive Blue 19 is about 46 years at neutral pH and 25 °C [16].

Effluent treatment plants are commonly used in the textile industries to control the quality of wastewater. Several primary, secondary, and tertiary treatment processes, such as chemical oxidation, flotation, biological methods, trickling filters, and electro-dialysis have been used to treat these effluents [14, 17-24]. However, the effectiveness of these treatments varies significantly, depending on specific dyes and chemicals used, and has limitations in terms of financial support and other complexities [3, 25]. As a result, the adverse impact on the environment is unavoidable but reducible at different levels when we use reactive dyes for dyeing cotton fabric.

The ultimate goal of this research is to develop a simple but innovative approach to confront dyeing industry water pollution by minimizing the presence of reactive dyes in effluents in order to enhance environmental sustainability. The objectives are designed in threefold- i) to investigate appropriate reactive dyes based on the functionality and structures by evaluating their presence in post-dye liquor solution, ii) to determine the deteriorating impacts of waste dyes on ecological parameters (algal growth inhibition, BOD₅, COD, TDS, TSS, DO, and pH values) and iii) to treat wash liquor from the dyeing

unit in a pilot plant and discover the synergistic effect of dyeing with specific dyes to enhance the condition of wastewater.

Table 1. Discharge quality standards for classified industries composite textile plants [26] (Bangladesh standard)

Parameters	Acceptance Limits
pH	6.5-9
Dissolved oxygen (DO)	4.5-8
Biological Oxygen Demand (BOD ₅)	150 mg/l
Chemical Oxygen Demand (COD)	200 mg/l
Total Dissolved Solids (TDS)	2100 mg/l
Total Suspended Solids (TSS)	150 mg/l
Wastewater flow	100 l/kg of fabric processing

Figure 1. Chemical structure of different classes of reactive dyes based on their reactive group

EXPERIMENTAL

Materials

Pretreated (scoured, bleached, and bio-polished) 100%-cotton single jersey knit fabric was taken for the research work, with the yarn count of 36's, and GSM (gram per square meter) of 150. During this study, four different types of dyes, namely Procion Blue PX-3R (PB), Levafix Blue CA (LB), Remazol

Brilliant Blue R (RBr), and Remazol Brilliant Blue RR (RBrr), were selected and purchased from Hangzhou Tiankun Chem Co. Ltd. (China). Different types of dyeing auxiliaries, namely glauber salt ($\text{Na}_2\text{SO}_4 \cdot 10 \text{H}_2\text{O}$), caustic soda (NaOH), wetting agent, sequestering agent, and leveling agent were used for different purposes. Lime (Ca(OH)_2) and ferrous sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) were used for the treatment of wastewater as coagulants and cationic polyacrylamide as polyelectrolyte. All of these chemicals were purchased from a local chemical shop, Mitali Scientific Store. Four different types of reactive dyes, namely Procion blue PX-3R, Levafix blue CA, Remazol Brilliant Blue R, and Remazol Brilliant Blue RR, were used based on their reactive group and chemical structure and were collected from Dye star. The molecular structures of the dyes are illustrated in Figure 1.

Pilot plant

After the dyeing process, the generated dyed wastewater was treated with lime and ferrous sulfate slurry in a batch mode pilot plant (Figure 2). The first part of the plant consisted of a textile wastewater generation section, which carried out the dyeing operation by an infrared sample dyeing machine. The dyeing operation was carried out according to the recipe and the sequence mentioned in Table 2 and Figure 3. After completing the dyeing processes, the equalization basin (T1) was loaded with approximately 2 L of post-liquor. Then the wastewater was homogenized through an air compressor and a diaphragm. Next, the wastewater got to a fast-stirring tank (T2) from the equalization basin (T1) and was rapidly mixed (at 200 rpm) with coagulants (800 mg/l lime and 800 mg/l ferrous sulfate mixture) for approximately 1 to 2 min. Afterwards, the wastewater samples were slowly mixed with coagulants at 20 rpm in a slow-stirring tank (T3) for 20 min. Subsequently, 10 mg/l cationic polyacrylamide was used as polyelectrolyte in the slow-stirring tank (T3) in order to improve effluent quality. Finally, the wastewater reached the sedimentation tank (T4), where the suspended and dissolved solid particles settled down with the coagulants and polyelectrolyte (settling time - 60 min). In order to examine the water quality, the post-dyeing samples were collected as influents before entering the equalization basin, and the samples from the discharge of the pilot plant were as effluents. All of the sample solutions were stored at 4 °C in closed glass bottles before the test and all the experiments were conducted within 7 days of collecting the samples.

Figure 2. Pilot plant setup for the treatment of textile wastewater using lime and ferrous sulfate

Table 2. The recipe for dyeing on cotton fabric with different chemical classes of reactive dyes

Reactive Dyes	Procion Blue PX-3R (PB)	Levafix Blue CA (LB)	Remazol Brilliant Blue R (RBr)	Remazol Brilliant Blue RR (RBrr)
Reactive groups	Monochloro- triazine	Bi-functional heterogeneous	Vinyl-sulphone	Bi-functional homogeneous
Dye concentration (%) on the wt. of fabric	1.5	1.5	1.5	1.5
Fabric weight (g/m)	5	5	5	5
Salt (Na ₂ SO ₄ .10 H ₂ O) (g/l)	40	40	40	40
Soda (g/l)	10	10	10	10
Wetting agent (ml/l)	1	1	1	1
Sequestering agent (g/l)	1	1	1	1
Leveling agent (ml/l)	1	0.5	1	0.5
Temp (°C)	80	60	60	60
Time (min)	45	45	45	45
Material to Liquor ratio (M:L)	1:10	1:10	1:10	1:10

Figure 3. Dyeing operation curve and post-washing curve for (a) PB and (b) LB, RBr and RBrr

Characterization of water quality

The collected wastewater samples before and after entering the ETP plant were analyzed, for several wastewater quality parameters, including temperature, pH, dissolved oxygen (DO), chemical oxygen demand (COD), biological oxygen demand (BOD₅), total dissolved solids (TDS), and total suspended solids (TSS). All analysis was carried out according to the Standard Methods [27]. Dye hydrolysis rates were analyzed by estimating the strength of the dye concentration in the hot and cold wash liquor solution using a Data Color 650 TM Spectrophotometer.

RESULTS AND DISCUSSION

Dye release in post-dye liquor solution

After a complete dye fixation on the dyed cotton fabric, the samples were washed in hot water (80°C for 5 min) or cold water (25°C for 5 min) in order to remove any of the unwanted residual dyes present in the fabrics. Moreover, due to the hydrolysis reaction and the incomplete fixation of dye molecules in the cotton fabrics, segregation of dye molecules may arise. The study showed that the rate of dye fixation and hydrolysis reaction (segregation rate) mostly depends on the fabric character, dye structure, and also operating conditions (pH, dyeing recipe, fixation time, liquor ratio, and many more) [28]. Consequently, the percentage of residual dyes left in the post-dye solution is a serious concern, as they remain in the wastewater and eventually get into the environment. Figure 4 shows that the concentration or strength of dyes in post-hot wash liquor is significantly higher than in the post-cold wash liquor, but it varies from dye to dye significantly. The spectrophotometric analysis demonstrates that PB hydrolyzed more than the other dyes in both post-hot and cold wash and was 51% and 33%, respectively. That was due to the weaker covalent bonding and less functional groups in PB dye than in other dyes resulting in the release/hydrolysis of the dye molecules from cotton fabric. LB and RBrr

dye molecules contain two functional groups that bind with the reactive sites, leading to the lower release of the bonded dye. LB dye has a new aliphatic vinyl-sulphone system and a fluoro-triazine group. Both of them contribute to ensuring better fixation under similar operating conditions decreasing the dye hydrolysis rate. The strength percentage of this dye in post-hot wash and cold wash liquor is, respectively, 36% and 17% for LB dye, while for RBr, it is 43% and 27%.

Figure 4. Dye concentration (%) of different classes of dye in the post-hot and cold wash liquor

Table 3. Textile wastewater characteristics collected from the dyeing unit after the dye fixation

Name of reactive dyes	pH	DO (mg/l)	BOD ₅ (mg/l)	COD (mg/l)	TDS (mg/l)	TSS (mg/l)
PB	8.2±0.3	0.013±0.005	155±14.4	710±24.5	3720±35.4	265±25.5
LB	7.5±0.4	0.025±0.003	115±15.5	457±26.3	2950±44.3	165±23.6
RBr	7.4±0.2	0.011±0.003	135±16.4	631±15.5	3460±39.9	205±23.3
RBrr	7.8±0.4	0.014±0.003	120±13.7	562±13.9	3230±40.8	190±22.8

Freshwater microalgae growth inhibition test

The algal growth inhibition test was carried out on the green microalgae species: *Pseudokirchneriella subcapitata* [29]. This test was conducted to estimate the impact of dyes on the aquatic environment at various contamination doses. Before starting this test, the growth inhibition experiments, the algal cells were cultured for three days (in similar conditions for the test) to ensure that they were in the exponential growth phase when used to inoculate the test suspensions. This experiment was conducted by following OECD 2011 guideline [30]. In brief, microalgae species were exposed and cultured in several concentrations (10, 50, 100, 500, 1000, and 2000 mg/l) of dye liquor solution over a period of 72 h at 25°C under continuous illumination of fluorescent tubes (OSRAM Fluora). A control-

cultured sample was assessed (without the dye solution) in order to estimate the normal growth and compare it to algal growth in the dyed sample [31]. All the samples (studied concentrations) were evaluated in triplicates with six controls. Figure 5 shows the algal growth inhibition rate relative to the control (rtc) at various dye concentrations for each of the reactive dyes. It also shows that the growth rate of the culture was inhibited more with the increase in the concentration of each of the dye classes, and an almost similar adverse effect resulted, as a higher dye contamination leads to higher turbidity of the liquor, which prevents the light passing through the aquatic solution and reduces photosynthesis rate as well as the elimination of the dissolved oxygen. Among all of the dye cultured solutions, PB exhibited maximum inhibition rtc % at each of the dye concentrations. According to the literature, the coefficient of variation of average percentile growth inhibition rate rtc must not exceed 7-10% [29]. According to this study, the inhibition rate exceeds the permeable limit in a cultured media with a higher dye concentration (more than 50 mg/l). Theoretically estimated from Figure 5, the effluent from textile waste should be of a lower dye concentration, with PB at 22 mg/l, LB at 26 mg/l, RBr at 28 mg/l, and RBrr at 32 mg/l in order to prevent the algal growth rate.

In this study, the concentration of dyes in the post-liquor and treated effluent was measured for individual dyes, which was summarized in Table 4. For each dye, the initial concentration was kept the same (1.5%) and the dyeing liquor was prepared with other necessary auxiliaries required for dyeing as mentioned in Table 2. After the completion of dyeing, it was apparent from the dye concentration in the post-dye liquor that the percentage of LB dyes in the post-liquor solution was 36%, while PB, RBr, and RBrr dyes were respectively 55%, 52%, and 47%; a similar observation was found by Irfan et al. [28]. The percentage of dyes present in the remaining solution differed due to the characteristics and nature of the reactive group. After the treatment in a FeSO₄-based ETP plant, the concentration of each dye in the effluent exhibited that the removal percentage of PB, LB, RBr, and RBrr dyes were, respectively, 82, 89, 83, and 86. Moreover, after the ETP treatment process, only 59 and 95 mg/l of LB and RBrr remained in the wastewater, which was close to the theoretical dye concentration (26 and 32 mg/l for LB and RBrr) for the prevention of algal growing rate. Optimizing the ETP plant operations (pH, contact time, FeSO₄ dose, etc.) may solve this problem.

Figure 5. Growth inhibition rate of *Pseudokirchneriella subcapitata* (cytotoxicity test) exposed to dye solutions at various concentrations for 72 h

Table 4: Dye concentration in the effluent collected after the dye fixation and after the coagulation with FeSO_4 in the ETP plant

Dye name	Initial concentration (mg/l)	Concentration after the dye fixation (mg/l)	Concentration after the ETP treatment (mg/l)
PB	1500	825	148
LB	1500	540	59
RBr	1500	780	133
RBrr	1500	705	95

Wastewater treatment in the ETP pilot plant setup

After the dye fixation and a complete post-washing (hot and cold washing) of the reactive dye on the cotton fabric, the waste liquor solution and the washing water were considered as effluents, and the effluent wastewater quality parameters such as pH, DO, BOD_5 , COD, TDS, and TSS were assessed and recorded in Table 2. Those results indicate numerous findings, like variation of all parameters with the addition of different reactive dyes at a similar concentration, which may be due to the dye fixation ability on cotton fabrics for each of the dyestuffs. Furthermore, the wastewater also contains a vast amount of dye molecules (due to hydrolysis and the unreacted dye) which will be responsible for the death of aquatic life, specifically algae as per the cytotoxicity test report (the dye concentration more than 30 mg/l). Moreover, water quality parameters, such as the DO, BOD_5 , COD, TDS, and TSS values, from each of the dyed effluents crossed their maximum permeable limit, as summarized in Table 3 [32].

Table 5. Characteristics results of the treated textile wastewater (treated with lime and ferrous sulfate and polyelectrolyte)

Name of the reactive dye	pH	DO (mg/l)	BOD ₅ reduction (%)	COD reduction (%)	TDS reduction (%)	TSS reduction (%)
PB	6.8±0.2	0.155±0.005	64.97	70.28	60.00	50.19
LB	6.4±0.2	0.35±0.008	68.01	65.43	55.02	47.27
RBr	6.6±0.3	0.147±0.006	70.04	64.03	61.97	57.07
RBrr	6.9±0.2	0.126±0.008	74.67	61.03	56.11	45.26

Figure 6. Water quality test after treatment with lime and ferrous sulfate along with polyelectrolyte in the pilot plant setup

Dissolved oxygen (DO) is the amount of molecular oxygen dissolved in water and plays a crucial role in determining water quality. This value provides information about water quality in rivers, streams, and lakes as a minimum range of DO values is required for the survival of aerobic aquatic organisms [33]. Lower values of DO, less than 1-3 mg/l, would indicate poor water quality and would make the survival of microorganisms that can degrade organic pollutants in the wastewater difficult [26]. The textile wastewater contained extremely low DO concentration levels, 0.013, 0.025, 0.011, and 0.014 mg/l for PB, LB, RBr, and RBrr, respectively, all of which were lower than the standard value set for Bangladesh at 4.5-8 mg/l [26]. Due to a high temperature during the post-hot washing operation, molecular oxygen was dislodged from the solution, ultimately reducing the DO values. A significant improvement of DO values was achieved after the treatment of each of the dyed wastewater samples in the ETP pilot plant setup shown in Table 5.

Biochemical Oxygen Demand (BOD₅) is the amount of dissolved oxygen used by aerobic microorganisms to degrade organic pollutants. It is an important parameter for the determination of water quality, providing a biological index to assess the effect of the wastewater on the environment [32]. A higher BOD₅ value represents the requirement for more oxygen for the decomposition of organic pollutants and indirectly denotes the presence of organic pollutants in the aquatic environment. Moreover, higher BOD₅ in the water body reduces the dissolved oxygen resulting in fish mortality and changes in species composition in the long term [34]. The textile wastewater containing reactive dyes had a BOD₅ concentration from 115 to 155 mg/l, which was close to the BOD₅ value set by the Bangladesh government for the industrial wastewater at 150 mg/l [26]. However, BOD₅ values were significantly improved after the treatment with coagulants, which can remove organic pollutants from the wastewater as it reduces the demand for oxygen for aerobic microorganisms for the degradation of organic pollutants. This study observed that BOD₅ values of 64.97-74.67% were reduced after the treatment to BOD₅ values of 30.4-54.3 mg/l, which were within the permeable limit of 150 mg/l.

Chemical oxygen demand (COD) represents the capacity of water to consume oxygen during the degradation of organic pollutants and the oxidation of inorganic chemicals. Here, the COD values of the textile wastewater were estimated within the range of 457 to 710 mg/l, which were higher than the maximum permeable limit of COD in the wastewater set for the industrial wastewater at 200 mg/l [26]. Maximum COD value was found for the PB reactive dye-based wastewater due to the lower dye-fixing ability of the monochloro-triazine group present in the PB dye and releasing more unreacted dyes in the waste liquor. On the other hand, the LB dye has a strong affinity towards the fabric, which is due to two functional groups (monofluoro-triazine and vinyl-sulphone) in its molecular structure, and is able to make strong covalent bonds with fabrics, resulting in a lower dye release in the effluent from the dye bath [35]. Moreover, inorganic salts, wetting agents, and leveling agents were also responsible for increasing COD values. After the pilot plant treatment, COD values reduced about 61 to 70% (Figure 6 and Table 5) due to the removal of organic and inorganic matters from effluents. Similar COD removal efficacy was also observed by the coagulation treatment of the textile wastewater with lime: ferrous sulfate (1:1) [36] and *Moringa oliefera*: alum (1:1) [37].

Total dissolved solids (TDS) are those organic and inorganic substances, such as various salts like chloride, phosphates, carbonates, bicarbonates, and nitrates of calcium, organic matter, sodium, potassium, magnesium and manganese, and other particles [38]. On the other hand, various types of suspended organic and inorganic particles in the textile effluent are considered total suspended solids (TSS). TDS and TSS values of the textile industrial water also depend on the types of dyes and other auxiliary materials (such as salts, the dye to liquor ratio, sequestering agents, the leveling agent used). TSS concentrations over a specific threshold impact water turbidity, which affects the photic zone for

sunlight penetration resulting in the reduction of growth of photonic algae, and indirectly reducing the number of aquatic animals. Hence, water transparency is also impaired, as turbidity is related to water clarity [39].

After completing the dyeing operation, total dissolved solids (TDS) values were found to be 3720, 2950, 3460, and 3230 mg/l for PB, LB, RBr, and RBrr, respectively. Similar TDS values were observed in previous studies in textile effluents [40, 41]. The lower TDS value of LB (compared to other dyed wastewaters) was due to the higher compatibility of the dye on the cotton surface. Moreover, the inclusion of an ETP plant with the dyeing operation significantly reduces the TDS content for 55.02 to 61.97% (the TDS concentration after the treatment was 1315-1488 mg/l), which was within the permeable limit for TDS in the industrial wastewater at 2100 mg/l [26]. Similarly, TSS was also significantly reduced from the dyed wastewater in the range of 45.26-50.19% (the TSS concentration after the treatment was 87-132 mg/l), which was lower than its maximum permeable limit of 150 mg/l [26] and similar results were also published in FeSO₄-based coagulation process for the industrial wastewater treatment process [42, 43]. The obtained data indicated that the present study is comparatively efficient with a comparable removal rate of BOD₅, COD, TDS, and TSS with other treatment processes.

CONCLUSION

This research work demonstrates an opportunity to understand the devastating impacts of reactive dyes on our environment, and at the same time, provides us with facile routes for managing the pollution practically and economically. Comparing different classes of reactive dyes, we found Levafix Blue CA as an environmentally sustainable reactive dye due to its complex bi-functional reactivity and heterogeneous structure compared to other simpler mono-functional and homogenous-natured dyes. Moreover, an ETP pilot plant was designed with cost-effective chemicals to treat the wash liquor from the dyeing unit. The treated wastewater exhibited similar results, in plain, better efficacy in almost all the experiment parameters for Levafix Blue CA compared to the rest, but further detailed studies are still required to answer several key issues that we wish to discuss in our future studies. Our research confirmed that selecting reactive dyes on structural and functional precedence of the reactive groups, combined with an efficient effluent treatment system, can ensure less generation of the waste that degrades the environment. Therefore, as the reactive dyes show a high level of toxicity and deviations from the standard ecological parameters, it is high time to focus on the structural and reactive functional group modification in order to produce reactive dyes with maximum fixation properties and minimum wastage in the future.

Author Contributions

Conceptualization – Saha T and Uddin Z; methodology – Saha T, Islam MD and Islam MN; formal analysis – Saha T and Tahsin AA; investigation – Saha T and Islam MN; resources – Saha T and Islam MD; writing-original draft preparation – Saha T, Islam MD and Islam MN; writing-review and editing – Saha T, Islam MD, Islam MN and Shamsuzzaman M; visualization – Saha T, Uddin Z, Islam MN, Shamsuzzaman M, Tahsin AA and Islam MD; supervision – Uddin Z. All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest

The authors declare no conflict of interest.

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