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Investigation on Ecological Parameters of Textile Effluent Generated After Dyeing With Mono and Bi-Functional Reactive Dyes

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Abstract:*This study is confined to the analysis of environmental parameters of waste water generated afterdyeing of cotton single jersey fabric with three different synthesized reactive dyes, including mono functional, homo bi-functional and hetero bi-functional dyes under four dye concentrations. Textile effluents were collected from reactive dyeing on two stages, including dyeing without and with additional fixing treatment. In this work, comparatively least pH values are recorded for the effluents collected from the second stage whereas mostDO,BOD and COD values are obtained inthe textile effluents taken from the first stage. In addition to this, the effluents containing mono functional and homo bi-functional reactive dyes show the most and the least DO, BOD and COD values, respectively for all dye concentrations. Besides, waste water containing high dye concentration resulted least DO, andmost BOD and COD values while compared to low dye concentrations in case ofselected reactive dyes.*

Keywords:*Reactive dye, Textile effluent, Dissolve Oxygen (DO), Biological Oxygen Demand (BOD), Chemical Oxygen Demand (COD).*

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I. Introduction

Textile industries consume and produce huge volumes of wastewater from different steps of textile wet processing, including pretreatment, dyeing and finishing processes [1-3].Textile effluent are known to exemplify strong color and higher BOD, COD values, since more than 15% of the textile dyes are usually went to the stream of wastewater during the dyeing operation [4]. Cotton provides an eco-friendly textile, but the wastewater from a typical cotton dyeing industry is characterized by color,less DO but more BOD, COD and pH values due to the requirements of huge amount of salt and alkali by the process when the cellulose fiber is dyed with reactive dyes. However, the favored concentrations of BOD and COD should be less than 30 mg/l and 250 mg/l, respectively for the disposal into surface water [1, 5]. Over 50% of cotton products are colored with reactive dyes which offer good proportion of the total market ranges from 20% to 30% [6]. The reason behind this popularity of reactive dyes for dyeing of cotton fiber is that its molecules, containing one or several reactive groups, chemically react with the fiber polymers to form a stable chemical linkage (covalent bond) between the dye molecules and fiber polymer [7-9].The dyestuff thus becomes a part of the fiber and renders better color fastness with brilliant color. Reactive groups are of two main types, (i) those reacting with the cellulose by nucleophilic substitution of a chlorine, fluorine, methyl sulphone or nicotinyl leaving group activated by an adjacent nitrogen atom in a heterocyclic ring;

$$\text{Dye-X+ Cell-OH} \longrightarrow \text{Dye-O-Cell + HX}$$

(ii) those reacting with cellulose by nucleophilic addition to a carbon-carbon double bond, usually activated by an adjacent electron-attracting sulphone group[10];

$$\text{Dye-SO}_2\text{-CH=CH}_2 + \text{Cell-OH} \longrightarrow \text{Dye-SO}_2\text{-CH}_2\text{-CH}_2\text{-O-Cell}$$

Among all, the utmost used chromophore group is the azo (R–N=N–R), followed by the anthraquinone group that constitute above half of worldwide production, approximately 65% [11-15]. In the case of triazine group,necleophilic substitution reaction takes place where anecleophilic addition reaction takes place in case of vinyl sulphone group based on chemical reaction, i, and ii.simultaneously, there is a competitive reaction between dye molecules and water which results dye hydrolysis and becomes responsible for poorer fixation rate (less than 70%),and inadequate utilization of dyes as well [16].

The following reaction shows hydrolysis of halogen containing reactive dye,
Dye-Cl + H-OH $\longrightarrow$ Dye-OH + H-Cl
The following reaction shows hydrolysis of activated vinyl compound holding dye,
Dye-CH_2-CH_2-OSO_3H + H-OH $\longrightarrow$ Dye - CH_2-CH_2-OH + H_2SO_4

Although, reactive dyes in the beginning contained only one functional group in its molecule, but well along the number of functional groups in dye molecules increased to reduce the hydrolysis problem. Thus, reactive dyes can be classified as mono, bi, or tri-functional depending on the number of groups capable to react with the fiber [9], but it can easily be assessed that dyes having two or more identical (homo functional) or different (hetero functional) reactive groups unveil a more fixation yield than dyes having single group[17].The main problem with conventional reactive dyes is that its dyeing efficacy always stands less than 100%, because of imperfect fixation on the fiber, and it is not more than 70% even in the case of textile printing. The removal of 30% or more unfixed dyes during the soaping treatment is arduous and time consuming, whereas the application procedures of reactive dyes are relatively simple [18]. The rate of hydrolysis decreases with the use of multifunctional reactive dyes, but the pollution problem still exists due to having fewer proportions of unreacted hydrolyzeddyes. Hence, those hydrolyzed dyes become unable to react with the fiber and leads to possibility of obtaining highly polluted effluent [8], containing gigantic amount of color, more BOD and COD. Because of the higher BOD, the untreated textile wastewaters make a rapid depletion of dissolved oxygen if it is directly discharged into the surface water sources [19]. High levels of COD are expected from dyeing section due to the nature of chemicals employed in the processing and high color of the effluent renders the water unfit for use at downstream of the disposal point [20].Thus, lower DO and higher BOD, COD of surface water creates a glacial ecosystem for the aquatic lives to sustain.

From a research report coveringphysiochemical analysis of textile dye effluent, BOD and COD values in the collected effluents were recorded to a range from 50 to 100 ppm and 200 to 400 ppm [21], respectively which commonly represents four times higher COD values than the BOD values in the effluent. In general, high levels of COD is generated during the reactive dyeing methods employed in cotton wet processing industries [22], and a published research work also indicates that greater values of BOD and COD in the effluents can be declinedwithsurrogatingby comparatively low impact reactive dyes. According to the authors of another research paper, low impactdyesdeliver greater ecological benefits for the reduction of COD, BOD and pH valuesin effluent considerably [23].Furthermore, an additional research work illustrates the same and suggests using eco-friendly dyes and chemicals in wet processing to minimize the environment pollution to a large extent [24].However, the first part of this paper focused on the preparation of the effluent with three reactive dyes, comprisingmono functional Reactive Blue-19,homo bi-functional Reactive Black-5 and hetero bi-functional Reactive Red-195 with four concentrations 0.5%, 2.5%, 4.5% and 6.5%, respectively with the aim to obtain the effluent characteristics. And the second part underscored on the estimation of DO, BOD, COD and acidity/alkalinity in textile effluents collected from two stages, including dyeing without andwith additional fixing treatment.

II. Experimental Details

2.1 Sample specification

In this research work, pretreated Single Jersey knit fabric was selected. The whole study was carried out with 100% cotton single jersey fabric which is specified as yarn count of 36’s, WPI (wales per inch) of 38, CPI (course per inch) of 52 and GSM (gram per square meter) of 160.

2.2 Dyeing process

Dyeing of selected sample was carried out with 0.5%, 2.5%, 4.5% and 6.5% concentrations of mono functional (VS) Reactive Blue-19, homo bi-functional (VS/VS) Reactive Black-5 and hetero bi-functional (MCT/VS)Reactive Red-195dyes. The chemical structures with molecular formula of those particular dyes are shown in the following Fig.1, Fig.2 and Fig.3, respectively.

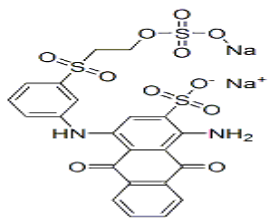

Fig.1: Chemical structure of Reactive Blue-19 [25]
Molecular formula: $C_{22}H_{16}N_2Na_2O_{11}S_3$ [26]

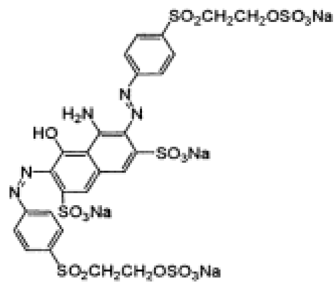

Fig.2: Chemical structure of Reactive Black-5 [27]
Molecular formula: $C_{26}H_{21}N_5Na_4O_{19}S_6$ [28]

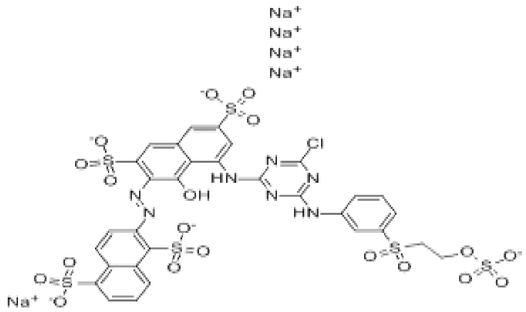

Fig.3: Chemical structure of Reactive Red-195 [29]
Molecular formula: $C_{31}H_{19}ClN_7O_{19}S_6.5Na$[29]

**VS-Vinyl sulphone (mono functional)*
**VS/VS-Vinyl sulphone/Vinyl sulphone (homo bi-functional)*
**MCT/VS-Monochlorotriazine/Vinyl sulphone (hetero bi-functional)*

The dyeing process with three reactive dyes listed above was carried out in IR (infra-red) sample dyeing machine. Two dye recipes were selected forcotton dyeing with three dyes; one recipe was used for mono functional and homo bi-functional reactive dyes, and another recipe for hetero bi-functional reactive dyes. The dye recipes which were used in dyeing with four concentrations are stipulated in the Table 1 and Table 2, respectively.

Table 1: Dyeing recipe withmono functionaland homo bi-functional reactive dyes

Dye concentration (%)	0.5	2.5	4.5	6.5
Fabric weight (gram)	5	5	5	5
Glauber salt (gram/litre)	15	45	65	85
Sodaash (gram/litre)	7	14	17	20
*Sequestering agent(gram/litre)	1	1	1	1
**Wetting agent(g/l)	1	1	1	1
Dyeing temperature (°C)	60	60	60	60
Dyeing time (min)	45	45	45	45
Liquor ratio	1:30	1:30	1:30	1:30

Complexant P-H/C, Dynotex MH$_1$D*

Table 2: Dyeing recipe withhetero bi-functional reactive dyes

Dye concentration (%)	0.5%	2.5%	4.5%	6.5%
Fabric weight (gram)	5	5	5	5
Glauber salt (gram/litre)	12	40	60	80
Soda ash (gram/litre)	5	5	5	5
Caustic soda 50% (ml/litre)	0.7	1.2	1.6	2
*Sequestering agent (gram/litre)	1	1	1	1
**Wetting agent (gram/litre)	1	1	1	1
Dyeing temperature (°C)	60	60	60	60
Dyeing time (min)	45	45	45	45
Liquor ratio	1:30	1:30	1:30	1:30

Complexant P-H/C, Dynotex MH_1D*

2.3 After-treatment process

The dyeing process of cotton sample was carried outat 60^{0}C for 45 minutes with four concentrations of reactive dyes having three chemical structures. Total 24 fabric samples were dyed and distributed into two groups; 12 dyed samples went to drying step directly after the dyeing process, leaving other 12 samples to after-treatment process (include additional fixing after dyeing). For after-treatment 12 dyed samples were cold rinsed first andsubsequent hot rinsing was done at 60°C for 10 minutes. Colored cotton samples were neutralized by acid wash later in where 1 ml/litre of acetic acid was used at room temperature and continued for 10 minutes. After that, hot wash is given on the neutralized samples with l gram/litre of soaping agent at 70°C temperature for 15 minutes. Sequentially, fixation process was carried out at 40°C for 10 minutes exactly with 1 gram/litreof cationic dye fixing agent in where pH is kept between 5.0 and 6.0 with the addition of 0.5 g/l of acetic acid.Finally, samples were dried at 105°C temperature for 10 minuteswith the help of a sample dryer. The pictorial views of 24 colored samples are shown in the following Fig.4.

Type of Dye	***Condition***	***Dye Concentration***			
		0.5%	***2.5%***	***4.0%***	***6.5%***
Reactive Red-195 hetero bi-functional (MCT/VS)	After dyeing				
	After dyeing and fixing				
Reactive Black-5 homo bi-functional (VS/VS)	After dyeing				
	After dyeing- and fixing				
Reactive Blue-19 Mono-functional (VS)	After dyeing				

	After dyeing and fixing				

Fig.4: Pictorial views of samples dyed with three reactive dyes under various conditions

2.4 Effluent collection

Textile effluents were collected from reactive dyeing on two stages for each dye structure under four concentrations. On first stage, the waste water was collected just after dyeing of cotton sample with reactive dyes without adopting any after-treatment (rinsing, neutralization, soaping and fixing) process. On second stage, the waste water was taken after the treatment of dyed samples with a fixing agent. Hence, total 24 effluent samples were collected from dyeing and dyeing-fixing stages for four different concentrations of three dyes and well-preserved in the following bottles for the assessment of DO, BOD, COD and pH later.

Reactive Red-195
hetero bi-functional (MCT/VS)

Reactive Black-5
homo bi-functional
(VS/VS)

Reactive Blue-19
Mono-functional
(VS)

Fig.5: Bottles containing textile effluents collected from dyeing and dyeing-fixing stages.

2.5 Methods of Assessment

2.5.1 Assessment of DO and BOD in textile effluent

IS: 3025 (Part 44)-Reaffirmed 2003 test method was selected for the assessment of DO and BOD in effluent samples collected from dyeing and dyeing with fixing stages. According to this method, the samples are filled in airtight bottles and incubated at specific temperature for 5 days. The dissolved oxygen (DO) content of the samples is determined before and after five days of incubation at 20°C and the BOD is then calculated from the difference between Initial and final DO and recorded in the following Table 3.

2.5.2 Assessment of COD in textile effluent

IS: 3025 (Part 58)-Reaffirmed 2006 test method was used for the assessment of COD in effluent samples collected from two stages. Based on this method, the organic matters present in effluent samples gets oxidized completely by potassium dichromate ($K_2Cr_2O_7$) in presence of sulphuricacid (H_2SO_4), silver sulphate ($AgSO_4$) and mercury sulphate($HgSO_4$) to produce CO_2 and H_2O. Then, those samples are refluxed with a known amount of potassium dichromate ($K_2Cr_2O_7$) in the sulphuric acid medium and the excess potassium dichromate ($K_2Cr_2O_7$) is determined by the titration against ferrous ammonium sulphate, using ferroin as an indicator. The dichromate consumed by each sample is equivalent to the amount of O_2 required to oxidize the organic matter and recorded in the following Table 3.

2.5.3 Assessment of acidity/alkalinity in textile effluent

With the help of a digital pH meter, acidity or alkalinity of effluent samples were evaluated and recorded in the Table 3.

III. **Results And Discussion**

After the assessment of four environmental parameters (DO, BOD, COD and pH values)of textile effluents generated from dyeing without and with additional fixing treatments, the data were recorded in the following table 3. Comparison of those data under various conditions elicited several imperative findings.

Table 3: DO, BOD, COD and pH values in textile effluents collected after dyeing with0.5%, 2.5%, 4.5% and 6.5% concentrations of reactive dyes

Sl. No.	Parameters of textile effluents evaluated	Stage of effluent collection	Dye concentration (%)											
			Reactive Red-195 hetero bi-functional (MCT/VS)				Reactive Black-5 homo bi-functional (VS/VS)				Reactive Blue-19 mono-functional (VS)			
			0.5	2.5	4.5	6.5	0.5	2.5	4.5	6.5	0.5	2.5	4.5	6.5
1.	DO_i(mg/l)	After dyeing	6.2	9.5	13.6	18.5	9.3	16.0	25.0	32.0	3.1	6.3	10.9	14.5
		After dyeing with fixing	2.1	4.5	8.5	12.5	3.0	7.4	9.8	15.2	1.6	3.9	7.2	10.7
2.	DO_f(mg/l)	After dyeing	1.3	3.9	5.7	6.0	4.2	7.5	15.5	18.0	1.3	2.0	3.6	4.2
		After dyeing with fixing	0.2	0.5	1.7	2.4	0.6	1.2	2.8	3.3	0.1	0.3	0.8	1.2
3.	BOD (mg/l)	After dyeing	490	560	780	1250	513	850	950	1400	180	430	730	1030
		After dyeing with fixing	190	395	683	1011	240	620	703	1196	150	359	644	952
4.	COD (mg/l)	After dyeing	1499	2669	3111	3333	1999	3499	3999	4498	889	1999	2666	3127
		After dyeing with fixing	889	1777	2444	3117	1111	2888	3114	3555	667	1556	2374	2889
5.	pH	After dyeing	9.5	10.1	10.5	11.2	9.1	9.8	10.3	10.7	9.2	9.8	10.5	10.8
		After dyeing with fixing	7.2	7.4	7.6	7.9	7.1	7.2	7.2	7.3	7.1	7.3	7.6	7.8

**DO_i–Initial dissolvedoxygen under light condition*
**DO_f–Final dissolved oxygen under dark condition*

3.1 DO values (under light and dark conditions)in textileeffluents generated from dyeing without and with additional fixing treatment

Overall analysis of Dissolve Oxygen (DO_i at light and DO_fat dark) with different concentrations after dyeing and dyeing with additional fixing shows most and the least DO values in case of homo bi-functional Reactive Black-5 and mono functional Reactive Blue-19; DO values for dyeing with hetero-bi functional Reactive Red-195 remain in between the values of other two dyes. Identically DO_i values in the effluents under light condition are found to be more due to the growth of micro-organisms on dark stage after preserving at 5 days, which mainly soak up the oxygen in this long period of time and causes decrease in DO_f at dark. The central reason behind more concentrated deep shade solution than the medium or light shade solution is that containing extremely large amount of salt, alkali, hydrolyzed and unfixed dye molecules and vice-versa. Aftermath circularly it happens in case of dyeing-fixing and in terms of shade percentage also. Reactive Blue-19 and Reactive Red-195 show close DO_f values at 0.5% and 2.5% shade.

3.2 BOD values in textile effluents generated from dyeing without and with additional fixing treatment

Analysis on textile effluent after dyeing and dyeing-fixing stage with different concentrations show the highest BOD values for homo-bi functional Reactive Black-5 and least values for mono functional Reactive Blue-19; BOD values for dyeing with hetero-bi functional Reactive Red-195 remain in between the values of other two dyes.Reactive Blue-19 and Reactive Red-195 show closest BOD values only at 4.5% shade after dyeing. Three types of reactive dyes show closest values for 4.5% shade and merely closer values for all shade % after dyeing-fixing. In addition to this, BOD values in case of deep and medium dye concentrations are found to be more than the BOD values for light concentration after dyeing without or with additional fixing treatment. In the course of dyeing, heavily concentrated waste water are produced by the reaction of fibers with a mixture of reactive dyestuffs, salt, alkali, other auxiliary chemicals and correspondingly diluted waste water was produced after the fixing treatment.

3.3 COD values in textile effluents generated from dyeing without and with additional fixing treatment

Analysis on textile effluent after dyeing and dyeing-fixing stage with different concentrations show the highest COD values for homo-bi functional Reactive Black-5 and least values for mono functional Reactive Blue-19. Besides, COD values for dyeing with hetero-bi functional Reactive Red-195 remain in between the values of other two dyes, Reactive Black-5 and Reactive Blue-19. In addition to this, COD values in case of deep and medium dye concentrations are found to be more than the COD values for light concentration after dyeing without or with additional fixing treatment. After dyeing process the highly concentrated waste water are found in the waste water mixture containing mainly hydrolyzed and unfixed reactive dyestuffs, salt, alkali and auxiliary chemicals. The waste water collected after fixing treatment becomes comparatively diluted while compare to the concentration of waste water after dyeing stage.

3.4 pH values in textile effluents generated after dyeing without and with additional fixing treatment:

The nature of the effluents collected from dyeing without and with additional fixing treatment for 0.5%, 2.5%, 4.5% and 6.5% concentrations of three dyes, was found to be always alkaline. Because, the dyeing processes with reactive dyes were carried out in alkaline medium. In case of all dyes and for each concentration, pH values in the textile effluents after dyeing (include basic alkali fixing) were found to be more than those values in the effluents generated after dyeing with additional fixing (include after-treatment with a fixing agent) treatment. This is because of removal of alkali from fabric samples after the subsequent rinsing, soaping and neutralization processes.

IV. Conclusion

This research work was intended and conducted to assess four ecological impacts DO, BOD, COD and pH of the textile effluents by dyeing cotton fabric with four different concentrations (0.5%, 2.5%, 4.5% and 6.5%) of reactive dyes having three reactive groups (mono functional VS: Reactive Blue-19, homo bi-functional VS: Reactive Black-5 and hetero bi-functional MCT- VS: Reactive Red-195) in its structure mainly at two different stages; stage-1: after dyeing without fixing (only mandatory internal alkali fixing during exhaustion reaction) and stage-2: after dyeing with additional fixing (fixing next to after-treatment).Effluents from the first stage represented less DO, but more BOD, COD, pH compared to the effluents of second stage for all dye structures and dye concentrations. Among four dye concentrations 6.5% was more responsible in generating effluents having the minimum DO and maximum BOD, COD. So, amount of dissolve Oxygen (DO) can be increased or demand of Oxygen (BOD, COD) can be decreased by maintaining lower concentration of shade %, minimizing the use of alkali and other auxiliaries. In addition to this, dyeing with bi-functional (homo bi-functional Reactive Black-5 and hetero bi-functional Reactive Red-195) reactive dyes discharge effluents with the highest BOD and COD compared to mono functional (Reactive Blue-19) reactive dye. Respectively for all dye concentrations among all Reactive Black-5 was responsible for highest BOD, COD and lowest DO values; whereas totally opposite condition was shown by Reactive Blue-19 and values of Reactive Red-195 remain in between. Hence, this research work becomes vital to identify the environmental effects of textile dyeing with reactive dyes having different chemical structures at various concentrations. This work will also be supportive to the dye house people selecting suitable dyes during the dyeing process in terms of ecological aspect and comprehend the characteristics of textile effluent under various conditions.

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