Performance enhancement of textile effluent treatment processes using electron beam method

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Abstract

This work explores the e-beam treatment method for treating wastewater that contains reactive dyes, Reactive Red 198 (RR 198) and Reactive T. Blue 21 (RB 21) in combination with salts. The effects of e-beam treatment were compared and analyzed for changes in pH, chemical and biological oxygen demands (COD and BOD), colour and toxicity for particular reactive dye solutions and reactive dye effluent. Biodegradability enhancement with e-beam treatment was assessed in terms of the increased ratio of BOD/COD. It was found that the BOD/COD ratio of reactive mixed dye effluent rose by 13%, 54% and 61% respectively, when 5,10 and 20 kGy e-beam dose was applied. This rate is comparatively less than that of red and blue dye effluent. This difference observed is due to the presence of a stable and complex structure of chromophores in the effluent. The structural alterations in the irradiated samples were examined using UV-visible absorbance and FTIR spectra analysis, which confirmed the dye breakdown through chromophore sites. The fish toxicity studies verified that the byproducts generated after the irradiation were less toxic than non-irradiated dyehouse effluent. Furthermore, for simulated dye house effluent, the effectiveness of coagulation and flocculation with and without e-beam pretreatment was assessed using Jar test method. This recommends e-beam pre-treatment prior to coagulation-flocculation, which results in a 20% high rate of COD reduction. Overall study showed that electron beam method can be used as pretreatment option for coagulation-flocculation as well as for biodegradation in textile ETP plant to enhance the performance in terms of COD reduction efficiency, chemical saving in coagulation-flocculation and residence time reduction in biodegradation process.

Key words:

E-beam treatment, textile industry effluent, coagulation-flocculation, biodegradation, BOD/COD ratio

Citation

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1.0 Introduction

Textile industry is a major contributor towards global economy, but, at the same time, it is also a major water consuming and pollution contributing sector. To produce about 1 Kg of finished fabric approximately 300litres of fresh water is consumed and same get converted to wastewater which includes pollutants like dyes, surfactants and other auxiliaries–[1], [2], [3]Based on the survey carried out in India under the project on 'Analysis of Eco management in Indian Textile Industries, major concerns observed with the textile effluent treatment plant (ETP) processes are in terms of controlling parameters such as strong colour, high concentration of suspended solids, a widely swinging pH, temperature variations, high chemical and biochemical oxygen demand'[4]. Knit, woven and yarn dyeing processors contribute COD in the range of 400-2700ppm and after the treatment, it reduces to 50-500ppm range' [4].This high organic pollutant-loaded effluent can cause adverse effects to aquatic ecosystem with decreasing dissolved oxygen, leading to bioaccumulation of toxic chemicals which ultimately disturbs the food chain.[5], [6] Hence there is a need to improve ETP working by adapting new techniques.

Numerous methods of treating textile effluent have been documented and are also employed in the industrial sector. The treatment techniques for textile effluent differ since it releases a range of contaminants. Textile effluent treatment

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plant (ETP) mainly involves chemical treatment followed by the biodegradation process to treat textile effluent. Among these, chemical coagulation and flocculation are most common to remove colloidal and suspended particles. Tertiary coagulation, adsorption (Activated Carbon filtration), membrane filtration, oxidation, and ozonation can be used to degrade dyes and other hazardous compounds[7], [8]. While many of these techniques are very effective, their main disadvantages are that they consume excessive amounts of chemicals, which can lead to sludge development and disposal issues, high running costs, and lack of discoloration with some dyestuffs[9]. The biological method, which is the second stage of treatment in the typical wastewater treatment process, uses microorganisms to break down organic contaminants into simpler compounds. The most widely employed microorganisms are fungus and bacteria because of their capacity to remediate wastewater contaminated with dyes. Nevertheless, the traditional chemical and microbiological treatments typically fail to break down the complex structure of organic molecules and are ineffective at removing colour from dyestuffs [10]. The most effective method in this situation is to employ an advanced oxidation process, which produces extremely reactive hydroxyl radicals while converting the dissolved organic contaminants to CO_2 and H_2O. Scientists have embraced AOPs for treating wastewater using UV, UV/H_2O_2, Fenton oxidation, and other processes [11], [12]. E-beam treatment also comes in the class of advanced oxidation processes. High-energy electron beam irradiation over water can produce hydroxyl radicals (Oxidation potential 2.8eV) as one of the active species through water radiolysis. This is one among the strongest oxidizing agents available, reacting instantly and unselectively with all non-degradable contaminants.

A radiation treatment method has been developed to effectively remove color and heavy metals from dye-contaminated water. The decolorization and mineralization of reactive dyes in simulated aqueous solutions by E-beam method has been extensively researched[13], [14]. However, in dyehouse effluent, dye substances are present with other auxiliaries such as dye fixing and exhausting agents[15]. Therefore, contrary to what is stated in the literature, decolorization is not frequently the outcome. Therefore, in this context, objective of this research is to study the application of an e-beam accelerator to enhance the efficacy of current effluent treatment processes, including coagulation-flocculation and biodegradation processes for wastewater from dye houses. Accordingly, in present study, the impact of E- beam irradiation (10MeV) towards dyeing effluent and simulated dye solutions were measured by means of decolorization percentage, biodegradability change, toxicity, pH, and COD removal.

2.0 Materials and Methods:

Reactive dyes; Corazol red RB 133% and Corazol Turq. Blue G were procured from Colourtex India PvtLtd . Along with these dyes, sodium sulphate and sodium carbonate were added in the reactive dyeing bath which acts as dye exhaustion and dyeing fixing agent respectively. Sodium sulphate, 99% (Loba Chemie) and anhydrous sodium carbonate ,99% (Loba Chemie) were used as received. Ferrous sulphate, 99% (Merck), Ferric Chloride hexahydrate (Loba Chemie), Alum (Loba Chemie), and Lime (calcium hydroxide, 99-102%, Fisher Scientific) were used as coagulants to carry out coagulation-flocculation process.

2.1 Preparation of Dye solutions and effluent:

Three samples each of reactive dye solutions and reactive dyeing industrial effluent were prepared separately. In the case of reactive dyeing solutions, 0.5 gram per liter(g/L) concentration of three dyestuff solutions is prepared in

Table1 Sample preparation details

Sr. No.	Sample Name	Dyestuff used with C.I. Number and concentration	Other assisting chemicals	Function in dying process
1	Reactive Red solution (RS)	Corazol red RB 133% (Reactive red 198)- 0.5g/L	-	colouration
2	Reactive Blue solution (BS)	Corazol Turq. Blue G (Reactive Blue 21)- 0.5 g/L	-	colouration
3	Reactive Mixed solution (MS)	Corazol red RB 133% - 0.25g/L + Corazol Tur Blue 0.25 g/L	-	colouration
4	Reactive red effluent (RDE)	Corazol red RB 133% (Reactive red 198)- 0.5 g/L	Sodium sulphate- 10g/L Sodium carbonate- 1g/L	Dyes: Colouration Sodium sulphate: Transfer dyestuff to fabric (for dye exhaustion) Sodium carbonate: Produces covalent bond between dyestuff and fabric (Dye fixing agent)
5	Reactive Blue effluent (BDE)	Corazol Turq. Blue G (Reactive Blue 21)- 0.5 g/L	Sodium sulphate- 10g/L Sodium carbonate- 1g/L	
6	Reactive mixed effluent (ME)	Corazol red RB 133% (Reactive red 198)- 0.25g/L + Corazol Turq. Blue G (Reactive Blue 21)- 0.25g/L	Sodium sulphate- 10g/L Sodium carbonate- 1g/L	

distilled water. Dyestuffs used here are Reactive dyes Corazol red RB 133% (C.I. Reactive red 198) and Corazol Turq. Blue G (C.I. Reactive T Blue 21). In case of mixed dye solution, 0.25g of reactive red 198 dye and 0.25g of Reactive T. Blue 21 dye are dissolved in 1liter of distilled water. After preparing these dye solutions, the dyehouse effluent was prepared in a similar way considering the industrial reactive dyeing process on cotton fabric. In this dyehouse effluent, 10g/L of sodium sulfate and 1g/L sodium carbonate were added as dye exhausting agents and fixing agent respectively[15]. Information regarding the concentration of dyestuff and other assisting chemicals used during the dyeing process is reported in Table1, along with their functions in the dyeing processes. Sample names were simplified for easy understanding and labeled as Red solution (RS), Blue Solution (BS), Mixed Solution (MS), Red Dye Effluent (RDE), Blue Dye Effluent (BDE), and Mixed Effluent (ME)

2.2 E-beam Irradiation:

A 10 MeV RF accelerator from EBC-Kharghar, Navi Mumbai, was used to carry out E-beam treatment. E-beam treatment over the reactive dye solution and effluent samples was performed at room temperature. The impact of E-beam treatment on textile wastewater samples was analyzed to improve the performance of conventional effluent treatment processes, such as coagulation-flocculation and biodegradation. This analysis was conducted in terms of changes in color and biodegradability with respect to the E-beam dose.

2.3 Jar Test Method: This method simulates the process of coagulation-flocculation with differing the types and concentration of coagulants. E-beam treated and untreated samples were subjected to coagulation-flocculation process in 6-stirrer Jar test apparatus viz Flocculator Jar Test- TR-50 (Fig.1). Various coagulants, including alum, lime, ferrous sulphate, and ferric chloridewere selected and each one of them with concentration of 1g/L is added in mixed dye effluent separately. Addition of coagulant with rapid mixing, to the tune of 200rpm speed, was maintained for 1minute duration. The speed of the mixer is then reduced slowly to 20rpm for 10 minutes duration which enhances the floc formation. Finally, the mixture was allowed to undergo sedimentation for 20minutes. The performance of coagulation flocculation is then assessed in terms of COD removal % which is calculated as follow.

$$\text{COD Removal Efficiency (\%)} = \frac{\text{COD}_i - \text{COD}_t \ \times 100}{\text{COD}_i}$$

Where, COD_i and COD_t represents the COD values initially and after the coagulation-flocculation process'[16]. Highest COD removal percentage value decides the best suitable coagulant for the effluent. In a similar way, optimum dose required for coagulation flocculation is calculated using best coagulant.

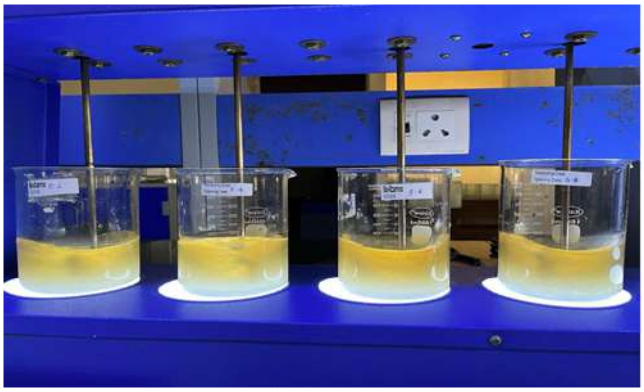


Fig 1: Jar test Apparatus to carry out coagulation-flocculation treatment on dye effluent

2.4 Other Analysis Methods:

The effect of e-beam treatment on the biodegradability of textile wastewater simulation is measured by examining the variations in both chemical and biochemical oxygen demand in relation to the e-beam dosage. The standard test procedure of IS 3025-part 58 and part 44 was used to determine chemical oxygen demand (COD) and biochemical oxygen demand (BOD) of untreated and treated samples. The pH values of solution and effluent samples were measured on a pH meter from Labindia (Model- Pico+). Decolourization of reactive dye solution and effluent samples was evaluated by the same method as described in our previous work'[17].

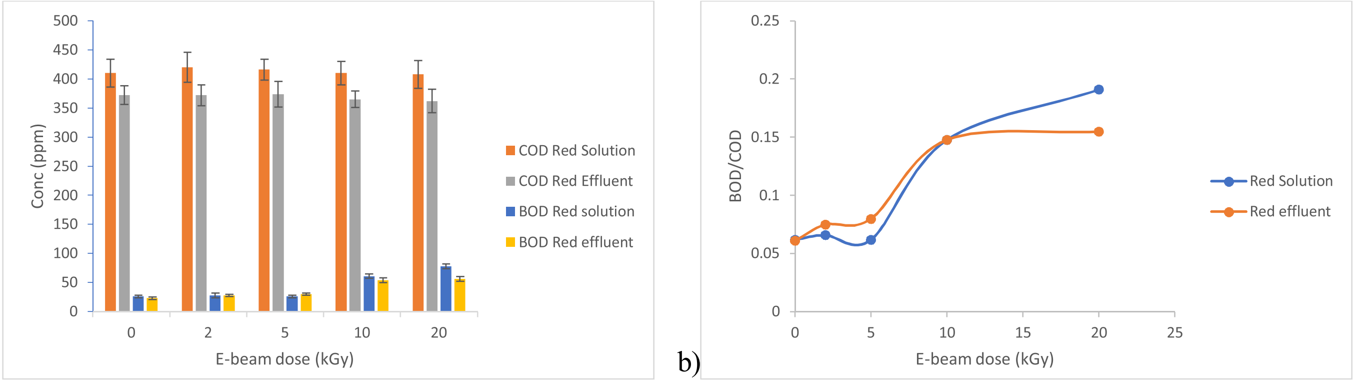
Structural changes in e-beam irradiated samples were tried to analyzed by FTIR spectroscopy. The ATR-FTIR spectra were measured with 8scans on Perkin-Elmer spectrometer (model system FTIR-Spectrum Two) in the range of 4000-650cm^{-1} with resolution of 4cm^{-1} scans. Toxicity assessment was done for the e-beam irradiated and untreated mixed dye effluent samples in terms of fish acute toxicity as per OECD 203 using fish species of Danio rerio (Zebrafish)[18]. The fishes are exposed to the variable concentrations of effluent samples, for 96 hours, and then mortalities are recorded in terms of percentage. The specific concentration which kills 50% of fish is then determined as lethal concentration LC50 value. Lower LC50value indicates the more toxic nature of effluent.

3.0 Results and Discussion:

3.1 Biodegradability enhancement with E-beam Treatment at low doses:

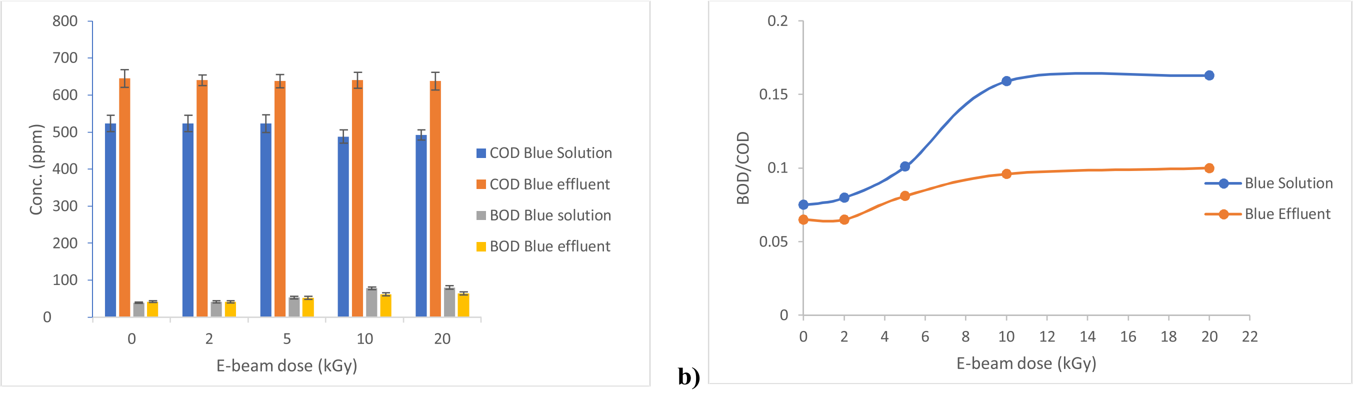
The concentrations of COD and BOD in the simulated red dye solutions are 410 and 26 mg/L respectively, while in the red effluent are 372 and 23 mg/L (Fig.2a). Stated otherwise, the BOD/COD ratios for the solution and effluent are same, at 0.06, suggesting that both samples are non-biodegradable wastewater. Initially this solution and effluent samples were irradiated with low doses of e-beam (i.e. up to 20kGy) to see the biodegradability enhancement. However, it was found that in both cases up to 5kGy doses there is no significant change in BOD/COD ratio, whereas after 10 kGy dose for simulated red dye solution and effluent, the BOD/COD ratio increases significantly by 146% in both the cases (Fig.2a& b). Similar trend was observed for reactive blue solution and effluent (Fig. 3a&b).In case of blue solution and effluent, up to5kGy e-beam dose slight improvement was observed, however10kGy e-beam dose leads to significant improvement in biodegradability i.e. by 114% and 60% respectively.(Fig.3b)

During the dyeing process in textile industry, many dyes are utilized and unfixed dyes are released in through wastewater. Therefore, in this paper separate dye solutions, mixed dye solution and mixed dye effluent with salts and carbonates were studied. The reactive mixed dye solution and effluent were subjected to 2, 5, 10, and 20 kGy dose of e-beam irradiation. The changes in COD and BOD observed after irradiation are reported in Fig. 4. From this, it can be interpreted that, as like in earlier case, in mixed dyes solution and effluent, significant improvement in degradability, 46 % and 54 % respectively, was observed for 10kGy e-beam doses. However, the rate of biodegradability improvement in mixed form is slower than that of single components. This slower rate of biodegradability improvement may be attributed to the presence of two types of chromophore sites from related two types of dyes i.e -N=N- from reactive red dye and Cupper phthalocyanine by reactive blue dye.



a) b)

Fig. 2 Pre-treatment with EB: impact onbiodegradability (COD. BOD and BOD/COD ratio) of reactive red dye solution and effluent



a) **b)**

Fig. 3 Pre-treatment with EB: impact on biodegradability (COD. BOD and BOD/COD ratio) of reactive blue dye solution and effluent

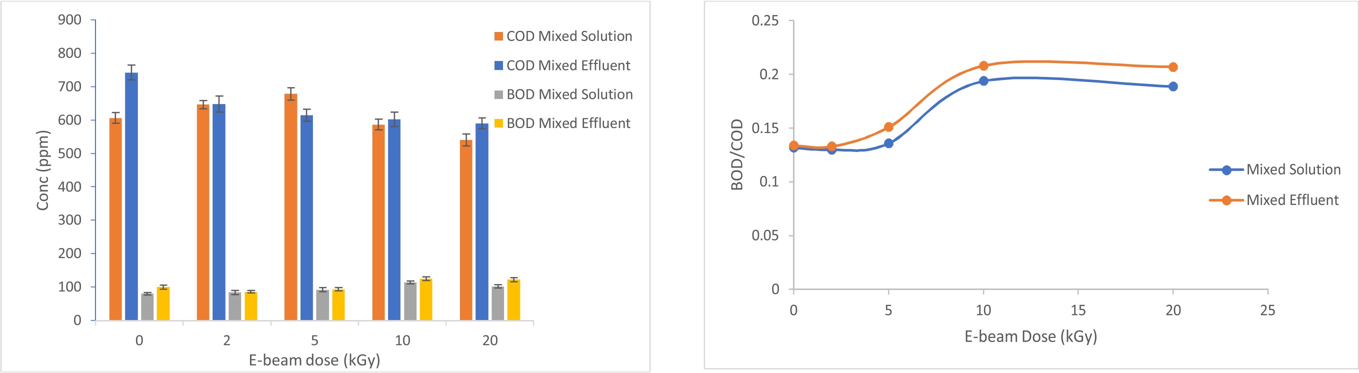


Fig. 4 Pre-treatment with EB: impact on biodegradability (of mixed dye solution and effluent

Overall, this study reveals that,10kGy e-beam dose towards dye house effluent can significantly improves its biodegradability and can be able to enhance the performance of any existing biodegradation process in terms of improving COD reduction from existing biodegradation facility or by lowering the residence time in biodegradation process.

3.2 Decolorization of dye house effluent with E-beam Treatment at higher doses:

After degradability improvement, our next objective was to observe the effect of high doses of E-beam treatment on complete decolorization and mineralization of dye house effluent. This study basically reveals the possibility of complete minimization of other treatment processes in textile ETP. Accordingly, simulated red dye, blue dye and mixed dye effluent were then irradiated by electron beam at high doses such as 20kGy, 50kGy, 100kGy, 150kGy and 200kGy to get complete mineralization and decolorization. Visual colour change observed in related red dye, blue dye and mixed dyes solution and effluent are shown through photographic images given in Fig.5.

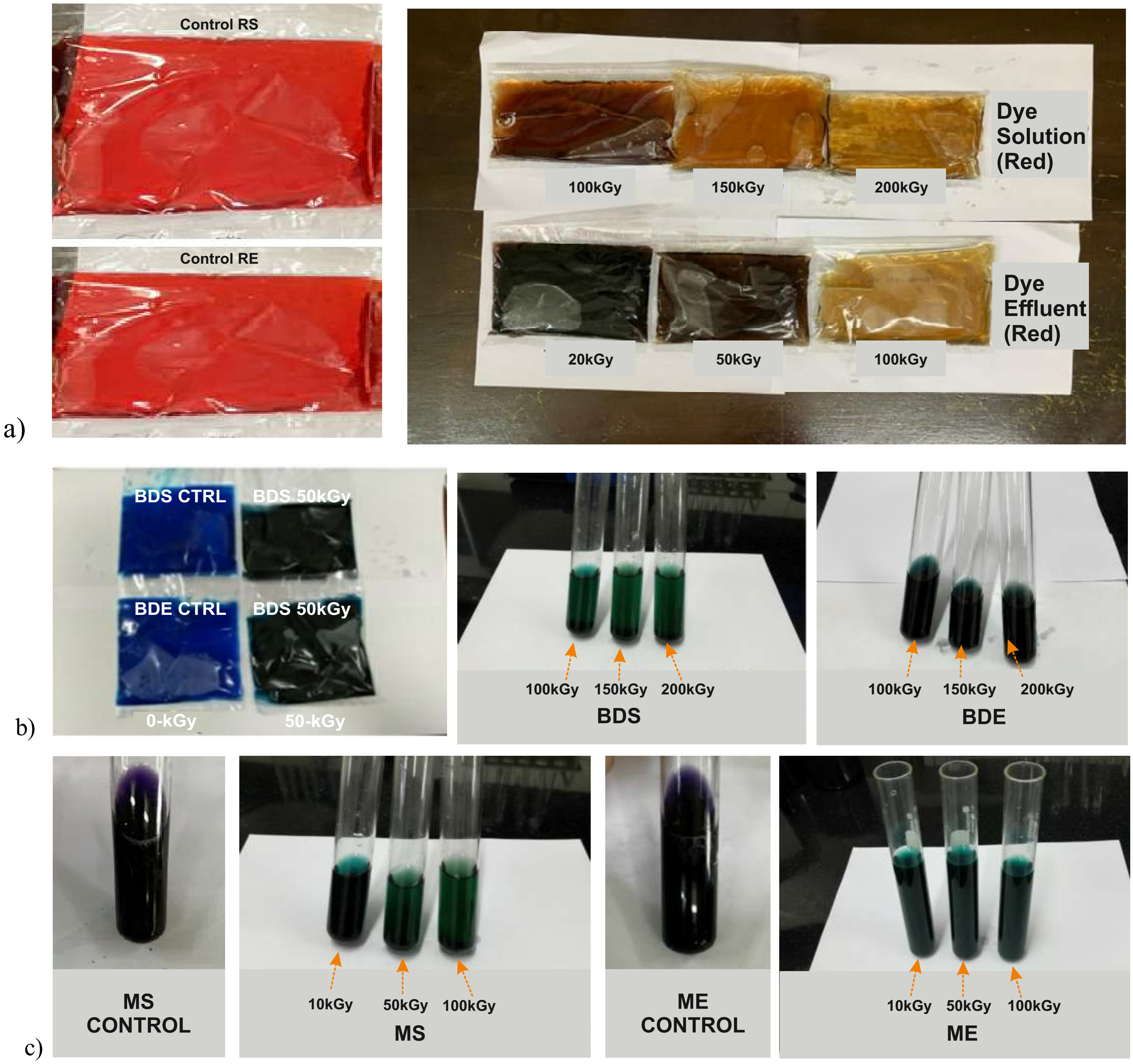


Fig. 5 Photographs of E-beam treated a) red dye b) blue dye and c) mixed dye samples at variable doses of E-beam

Through these images it can be clearly see that in case of red dye samples decolorization can be seen at e-beam dose of 50kGy (for dye solution) and above (for dye effluent). Whereas in case of reactive blue and mixed samples, visually no decolorization observed up to 200kGy and 100kGy dose respectively. These findings imply that the blue dye's structure is more stable than the red dye's. The dyes' chromophore structures may be responsible for this. In this instance, the red dye's chromophore is azo type, and the blue dye's is cu-phthalocyanine type . As a result, samples of mixed and blue dye degrade less quickly than samples of red dye. To confirm this data, structural changes happened after e-beam treatment were analyzed by UV-visible spectroscopy and FTIR investigations.

The UV-Visible absorption spectra of simulated red, blue and mixed dye effluent with respect to applied dose then measured with uv-visible spectrophotometer and presented in Fig. 6. Two distinctive peaks of the red dye effluent (at 290 nm and 535 nm) can be seen in Fig. 6a, and they show a considerable decrease with increasing doses. The decolouration of dye effluent was calculated by monitoring the decrease in absorbance at 535 nm. About 85% decolouration of red effluent was achieved at 20kGy whereas the effluent was 93% decolorized at 100kGy dose. Similarly, in case of blue dye effluent, characteristic peaks observed as doublet peak at 620 and 660nm and singlet peaks at 340 and 220nm (Fig. 6b). Out of this, doublet peaks represent the chromophore site of reactive blue dye and it was found that with the irradiation, peak area at this wavelength significantly reduces and represents the decolorization of 53% at 50kGy dose and 62% at 100kGy dose. The UV visible spectrum of mixed dye effluent represents the characteristic peaks at 520 and 640nm related to red and blue dye chromophores (Fig.6c). UV visible spectrum of this mixed effluent clearly shows the disappearance of related peak at 520nm with e-beam dose which is attributed by red colour chromophore, however, no significant change was observed in peak at 640nm.

This data supports the earlier investigation that the more stable structure of the blue dye results in a slower degradation rate compared to the red dye. To observe further structural changes caused by e-beam treatment, the FTIR spectrum was analyzed for both red and blue dye effluents. The characteristic peaks in the red dye effluent's FTIR spectrum appear at 3259cm^{-1}, 1540cm^{-1}, 1485cm^{-1}, 1089cm^{-1}, and 978 cm^{-1}. These peaks correspond to the O-H/N-H stretch in amine, the C=C aromatic stretch, the azo group, the C-N stretch in amine, and the -C=C stretch in alkene, respectively. The intensity of these peaks significantly decreased after e-beam treatment (Fig. 7). Notably, the peaks at 1540 cm^{-1} and 1485 cm^{-1} which are related to the C=C aromatic stretch and N=N stretch, respectively, disappeared after a 10 kGy dose. This confirms that the red dye molecules' chromophore (azo -N=N) site was broken by the e-beam. In the case of blue dye effluent, the FTIR spectrum showed characteristic peaks at 1440 cm^{-1}, 1104 cm^{-1}, 876 cm^{-1}, and

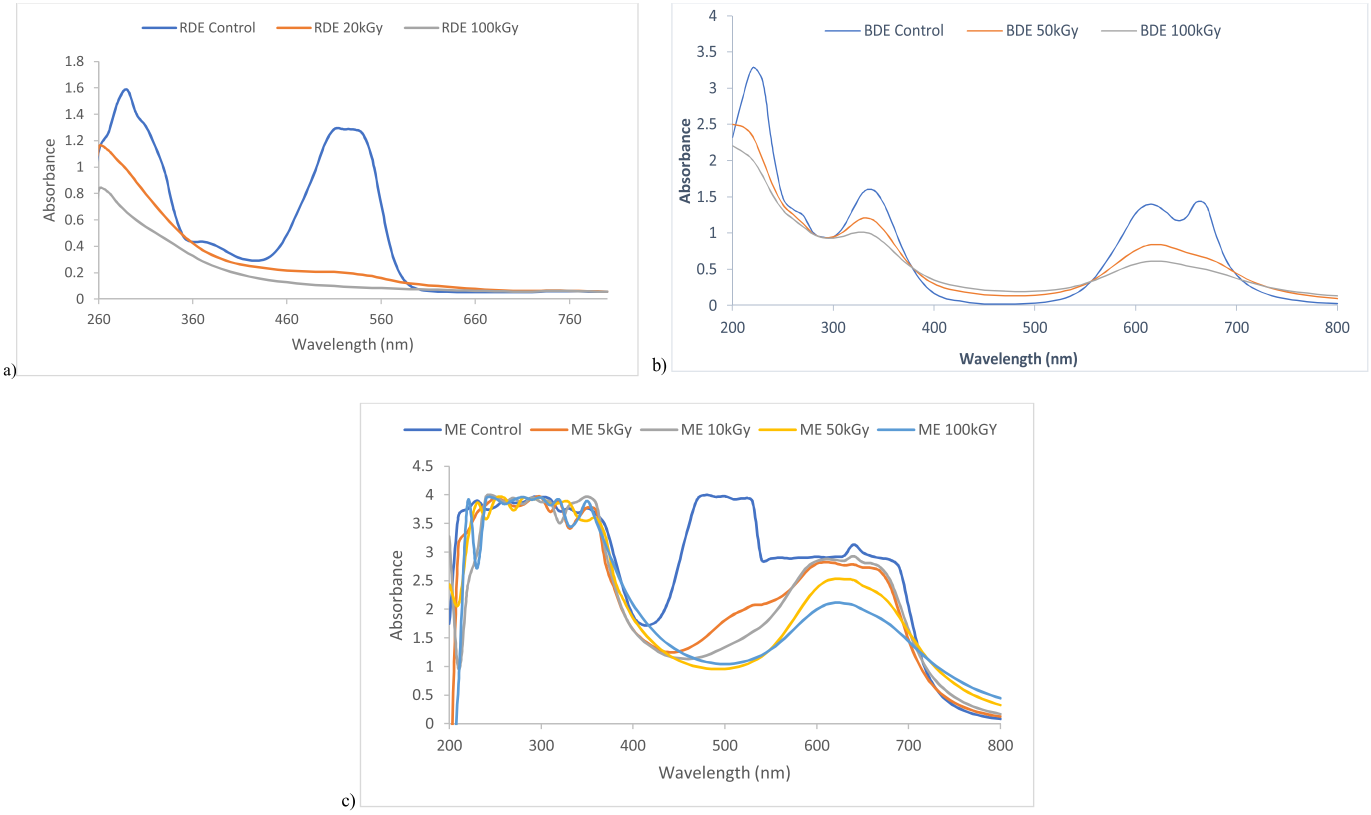


Fig. 6:UV visible spectrum of E-beam treated a) Red, b) Blue and c)mixed dye effluent

610 cm^{-1}, corresponding to aromatic C=C stretch, C-N stretching vibrations in amine, =C-H bending in alkene, and C-Cl stretch, respectively (Fig. 8). After irradiation with the e-beam at a 200 kGy dose, the peaks related to aromatic C=C stretch and C-N stretching in amines were significantly reduced, indicating degradation of the blue effluent through the C-N sites. Overall, the FTIR data confirms the degradation of reactive dyes via their chromophore sites, namely azo groups in red dye and Cu-phthalocyanine in blue dye effluent.

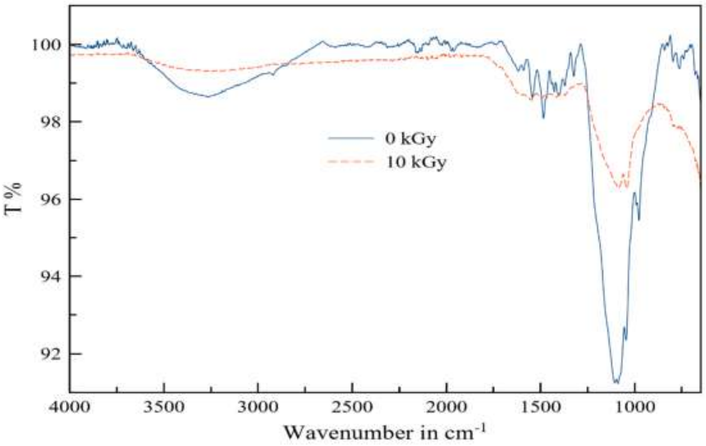


Fig. 7 : FTIR Spectrum of Reactive red dye effluent

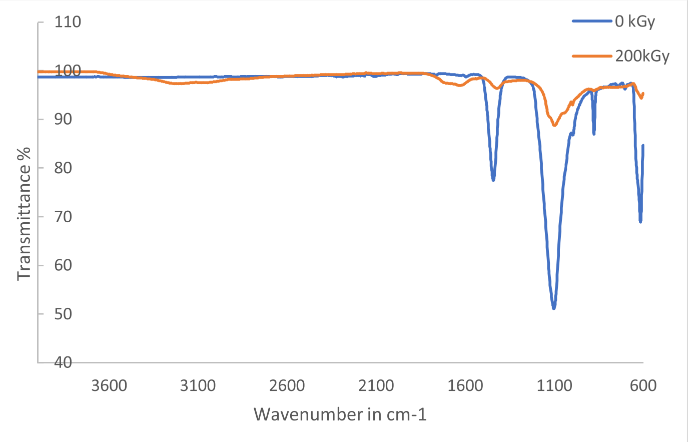


Fig. 8 : FTIR Spectrum of Reactive blue dye effluent in overall range

The pH of the unirradiated dye solutions and effluents were observed and reported in Table2. In case of red dye solution, at 20, 50 and 100kGy e-beam doses, effluent's pH reduces to 3.7, 3.2 and 3.04 respectively from initial value of 6.99. In case of effluent, it decreases from the initial value of 10.93 to 10.8, 10.55 and 10.05 respectively. Similar kind of trend for decrease in pH with irradiation is observed for blue as well as mixed solutions and effluents. The drastic pH decrease upon irradiation is attributed to the fragmentation of complex dye molecules to smaller components which ultimately leads to formation of some organic acids or carbonic acids formed due to complete mineralization of dye molecule[14]. The reduction of pH in dye effluents are comparatively very low as that of dyes solutions because of the suppression of mineralization which occurs in the presence of other contaminants in effluent such as salts and carbonates–[9], [19].

Table2E-beamimpact on pH of dye solutions and effluent samples

E-beam dose (kGy)		0	2	5	10	20	50	100
pH Values	Red Dye solution	6.99	6.91	6.85	6.3	3.7	3.23	3.04
	Red Dye effluent	10.93	10.8	10.71	10.55	10.39	10.39	10.05
	Blue Dye solution	7.14	7.03	6.44	5.43	4.64	3.92	3.05
	Blue Dye effluent	10.93	10.83	10.71	10.67	9.87	9.05	8.33
	Mixed Dye solution	8.01	6.96	5.21	3.49	2.86	2.61	2.58
	Mixed Dye effluent	11.36	11.04	10.94	10.68	10.55	10.41	9.81

3.3 E-beam treatment to enhance coagulation-flocculation performance in textile effluent treatment plant.

In textile effluent treatment processes (ETP), coagulation and flocculation are commonly employed as primary treatment methods to reduce COD, BOD and colour from wastewater. The addition of coagulants such as ferrous sulphate, alum, lime, and ferric chloride reduces the zeta potential of the effluent, thereby aiding in the removal of colloidal suspensions [20]. At the laboratory scale, the efficiency and optimum dosage of coagulants can be determined using a jar test apparatus. The treatment performance is typically evaluated in terms of percentage COD reduction, as per IS 3025-Part 58.

For mixed dye effluent, preliminary jar tests were conducted using various coagulants—ferrous sulphate, ferric chloride, alum, and lime—with a 1 g/L concentration to identify the most effective agent. Among these, ferrous sulphate exhibited the highest COD removal efficiency, achieving a reduction of 53% (Fig. 9). To determine the optimum dosage, ferrous sulphate concentration was varied from 200 to 1000 ppm. The maximum COD reduction (53%) was observed at 800 ppm (Fig. 10).

Subsequently, the study was extended to include electron beam (e-beam) pretreatment at a dose of 5 kGy, followed by coagulation–flocculation using 800 ppm of ferrous sulphate. The results demonstrated a significant improvement in treatment efficiency, with COD reduction increasing to 74%—an enhancement of 21% compared to coagulation–flocculation alone (Fig. 11).

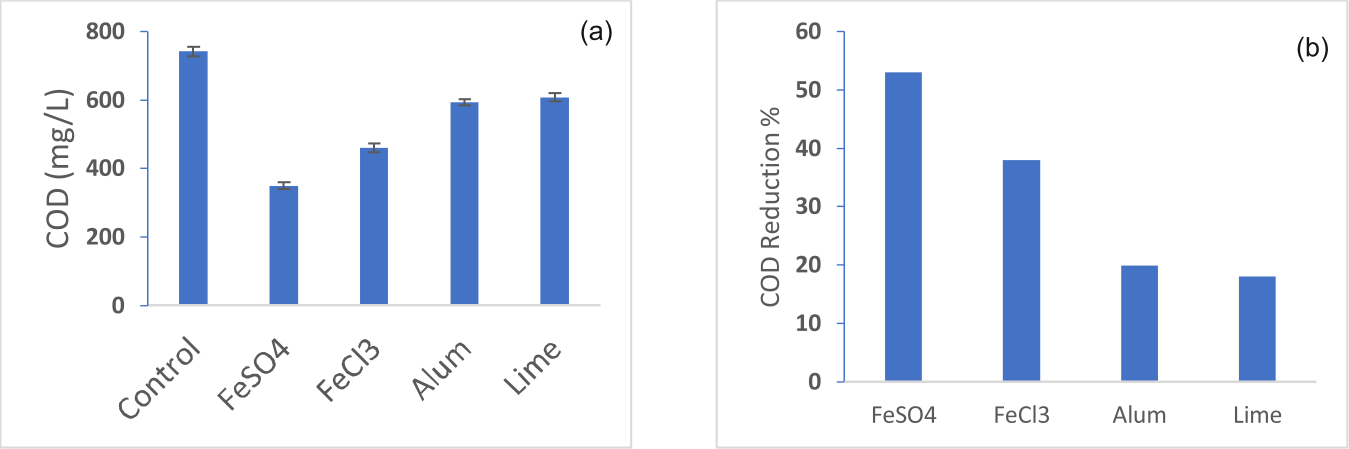


Fig.9 : Coagulant suitability study by Jar test method for treating mixed dye effluent a) in terms of COD reduction and b) % COD reduction

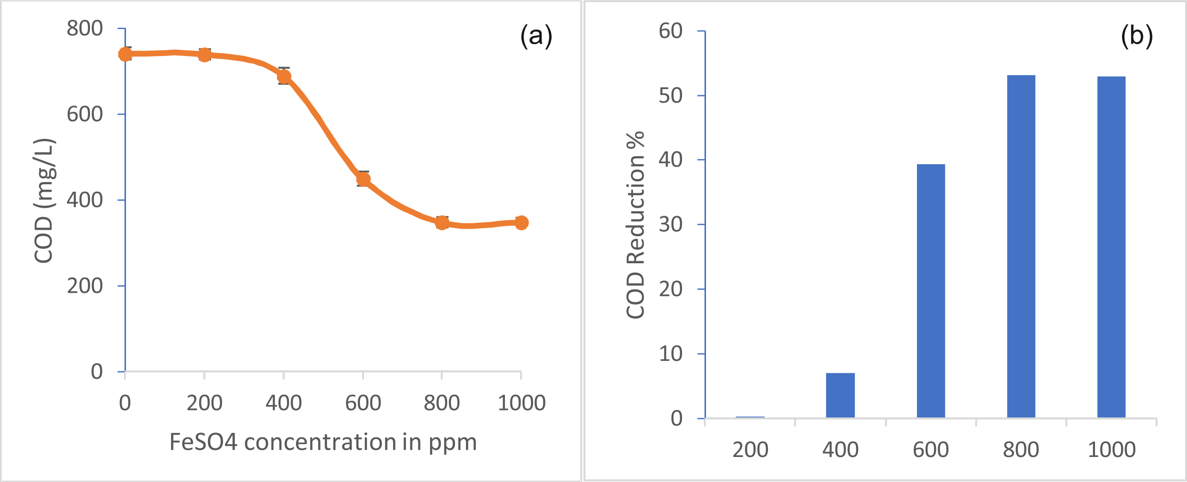


Fig. 10 : FeSO$_4$ dose optimization study by Jar test method for treating mixed dye effluent a) in terms of COD reduction and b) % COD reduction

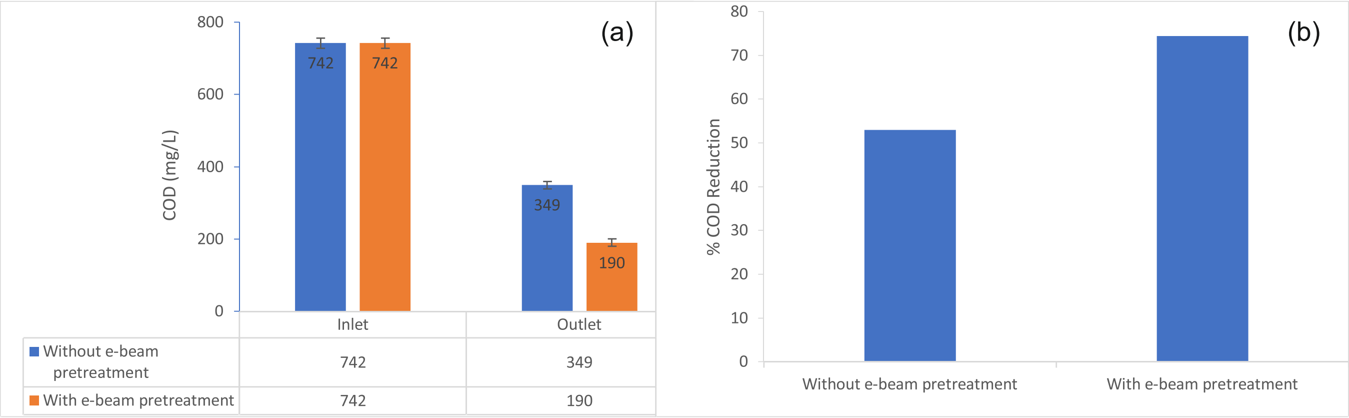


Fig. 10 : FeSO$_4$ dose optimization study by Jar test method for treating mixed dye effluent a) in terms of COD reduction and b) % COD reduction

3.4 E-beam treatment for reducing the toxicity of dyehouse effluent:

To determine how well e-beam radiation eliminated acute toxicity, toxicology tests were conducted for untreated and e-beam-treated textile dyehouse wastewater. Using acute fish toxicity, the toxic nature of the mixed dye effluent was evaluated in terms of lethal concentration (LC50). This concentration is essentially the particular solution concentration at which fish cumulative mortality of 50% has

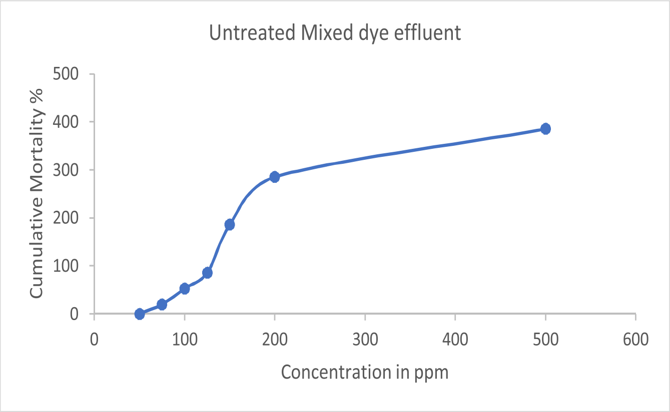


Fig. 12 : Acute Fish toxicity measurements of untreated mixed dye effluent samples

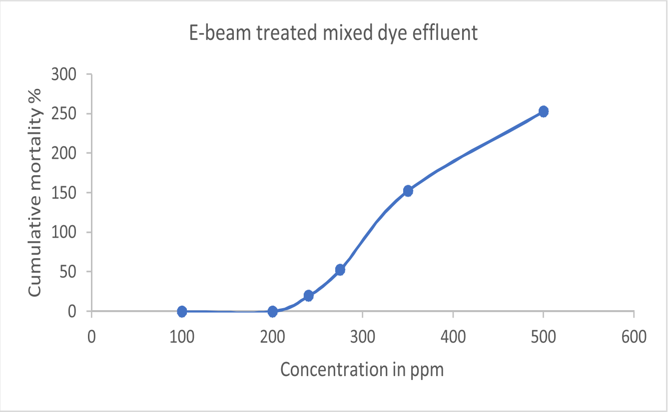


Fig. 13 : Acute Fish toxicity measurements of e-beam treated mixed dye effluent samples

been reported. Five fish were added in order to conduct this investigation in different concentrations of mixed dye effluent samples (without and with e-beam treatment), and the total number of fish that perished or survived was noted. This observation served as the basis for calculating the cumulative mortality percentage. Plotting the cumulative mortality percentage versus the mixed dye effluent concentration yields the LC50 value. This study is represented by Fig 12 and 13 for without irradiated and irradiated mixed dye effluent samples.

This result shows that the Lc50 value obtained for a non-irradiated sample is less than that of irradiated sample with 5kGy dose. With 5kGy e-beam dose, initial LC50 value of mixed dye effluent samples increased from 98ppm to 270 ppm. These results infer that e-beam treatment with a 5kGy dose lowers down the initial toxicity of dye house effluent significantly by 2.8 times that of initial toxicity.

4.0 Conclusion:

The results conclude that E-beam treatment significantly enhances the biodegradability and decolorization of dye house effluent depending on structures of dyes present in the effluent. The BOD/COD ratio, decolorization percentage and pH of the samples were influenced by the effluent composition, especially chromophore structure present in the dye, and applied dose. The BOD/COD ratio and decolorization of red dye effluent found to be improved at higher rate compared to blue dye effluent due to more stable structure of blue dye chromophore. To treat mixed dye effluent, the coagulation-flocculation process in conjunction with E-beam technology was optimized. E-beam irradiation with 5 to 10kGy of dose found to be very effective in improving the efficiency of subsequent coagulation-flocculation and biodegradation processes which reduces the need of chemical, operational cost and retention time in coagulation flocculation and biodegradation process respectively. With 5kGy e-beam dose, the performance of existing coagulation flocculation process is found to be increased by 21% and acute fish toxicity found to be reduced by 2.8 times. Treatment of dye house effluent using 10kGy e-beam dose significantly improved its biodegradability by 56% and enhanced the performance of existing biodegradation process in terms of improving COD reduction from existing biodegradation facility or by lowering the residence time in biodegradation process. So, e-beam seems to be viable solution for treating dyehouse wastewater and also it improves the performance existing ETP processes towards COD removal efficiency, chemical saving and maintenance cost.

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