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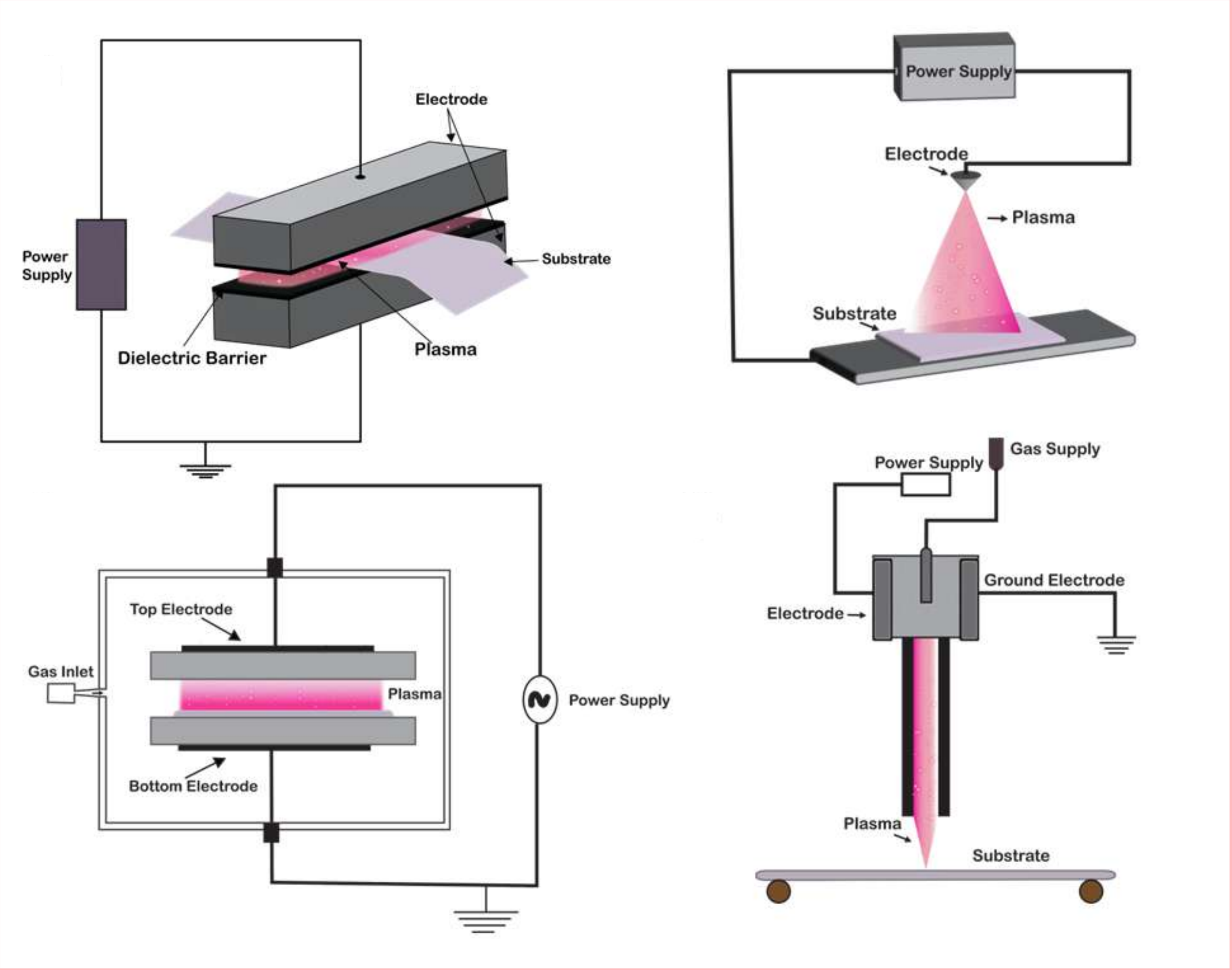

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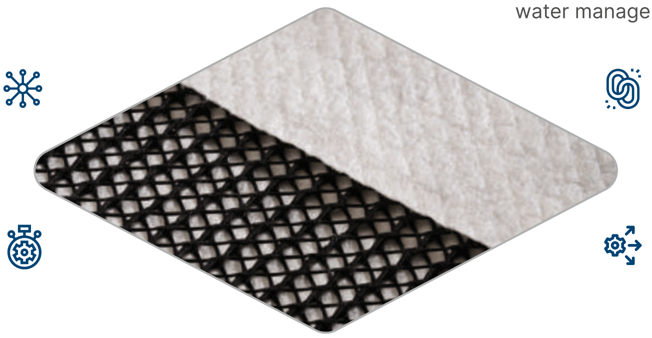

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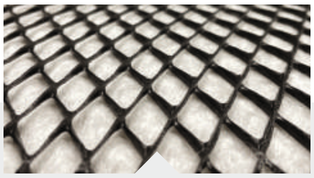

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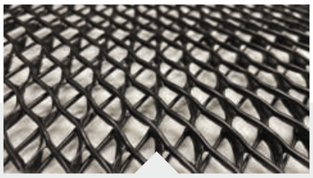

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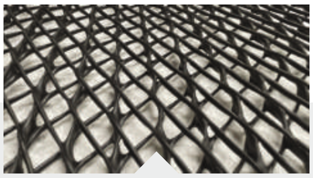

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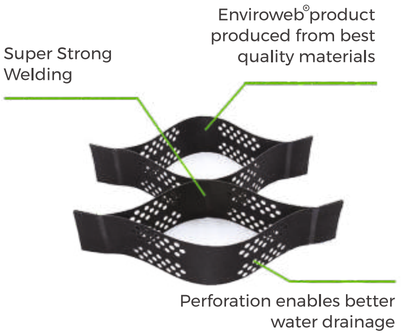

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EDITOR'S DESK

Dear Readers,

Greetings!!

Research with persistent and focused efforts lead to a positive result. Fostering research and providing a platform to publish quality research papers and related articles has been a continuous effort of BTRA Scan. We are working hard to help the journal in climbing up the ranking ladder. In continuation to this effort, I am delighted to present to our readers the 1st issue of 55th Edition of BTRA SCAN.

This issue has 3 papers from the different domains such as Performance enhancement of textile effluent treatment processes using electron beam method, Metal Organic Frameworks And Conductive Polymers For Energy Storage and Plasma-Assisted Surface Engineering of Textiles and Polymers for Improved Flame Retardancy : A Comprehensive Review. Now we are open for authors from outside so researchers can send their original articles, case studies, research reviews or empirical contributions for publication in our journal.

I thank my entire publishing team for all their support. Together we would work towards making the journal a truly influential publication. Comments and suggestions are always welcome.

Our sincere thanks to all the reader and contributors for their support and interest.

T V Sreekumar, PhD
Director, BTRA

Contents

Advertisement Index :

- **STRATA INDIA -** Front Cover Inside
- **TECHFAB INDIA INDUSTRIES LTD. -** Back Cover Page
- **ENVIRO GEOSYSTEMS -** Inside Page
- **GEOTECH INDUSTRIES PVT. LTD. -** Back Cover Inside

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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.

$$\text{COD Removal Efficiency}\ (\%) = \frac{\text{COD}_\text{i} - \text{COD}_\text{t}\ \ \times 100}{\text{COD}_\text{i}}$$

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.

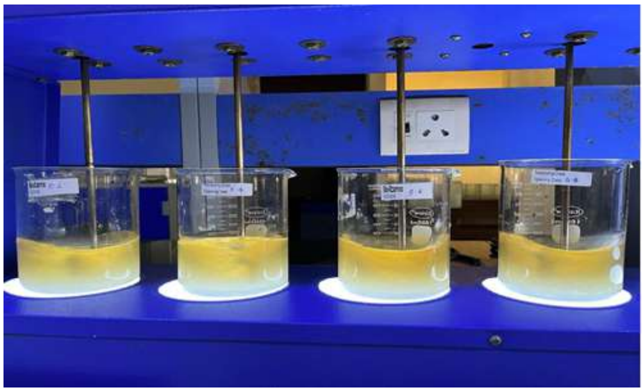

Fig 1: Jar test Apparatus to carry out coagulation-flocculation treatment on dye effluent

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.

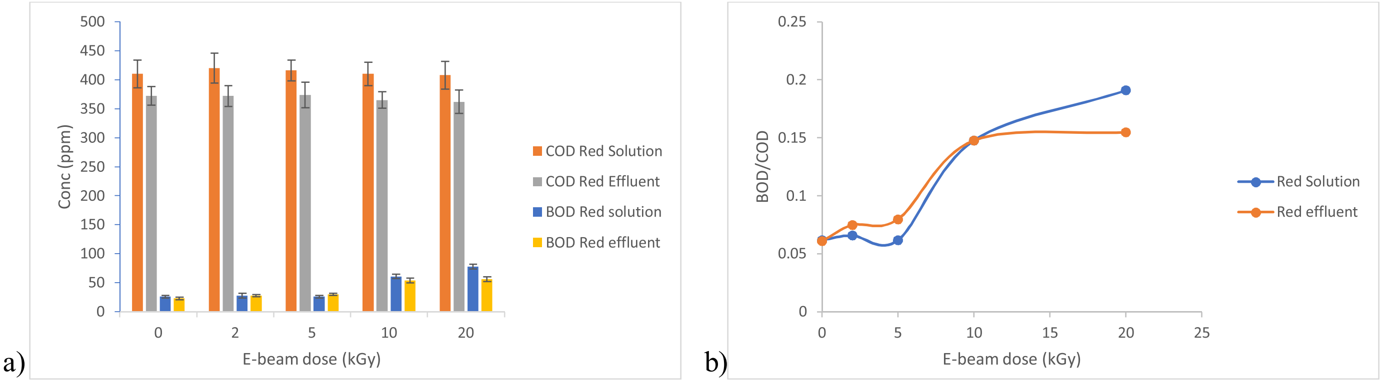

Fig. 2 Pre-treatment with EB: impact onbiodegradability (COD. BOD and BOD/COD ratio) of reactive red dye solution and effluent

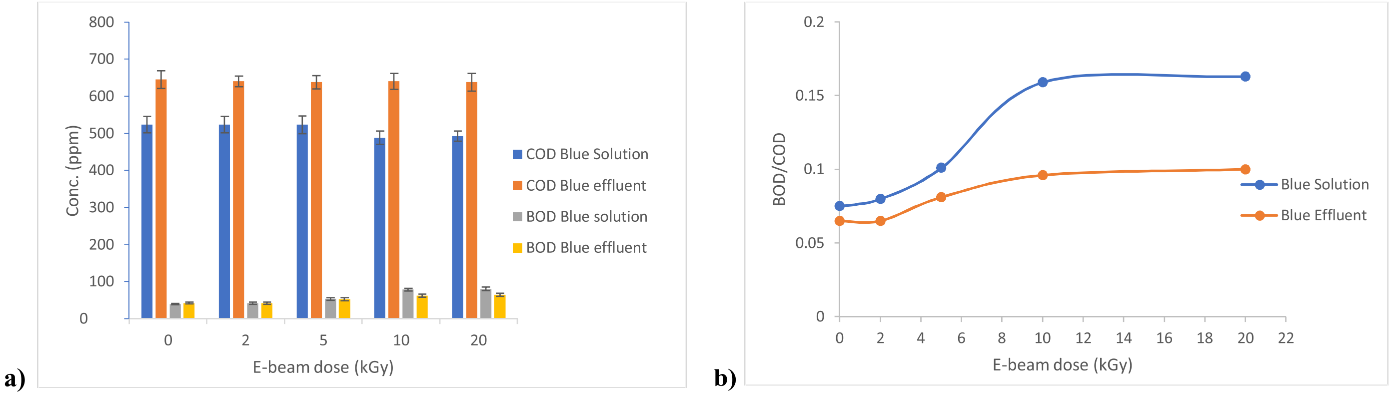

Fig. 3 Pre-treatment with EB: impact on biodegradability (COD. BOD and BOD/COD ratio) of reactive blue dye solution and effluent

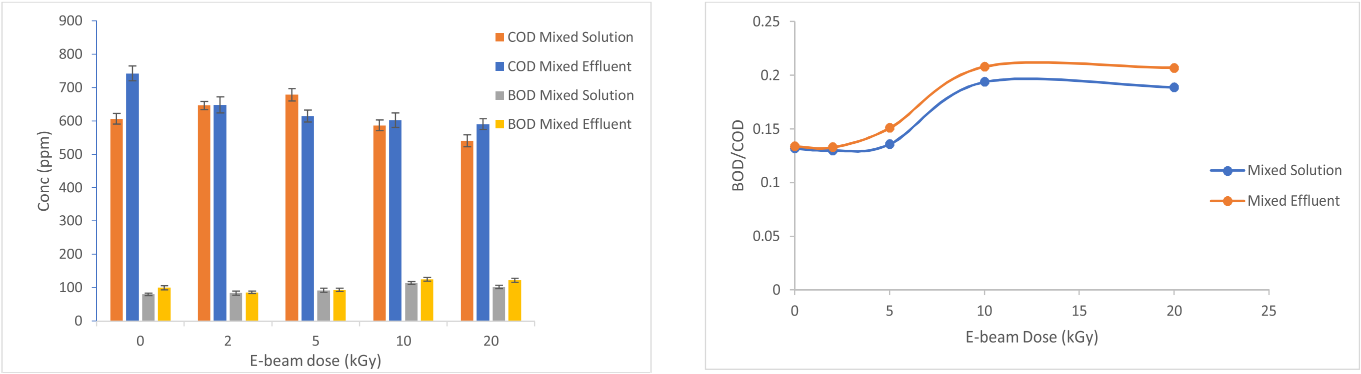

Fig. 4 Pre-treatment with EB: impact on biodegradability (of mixed dye solution and effluent

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.

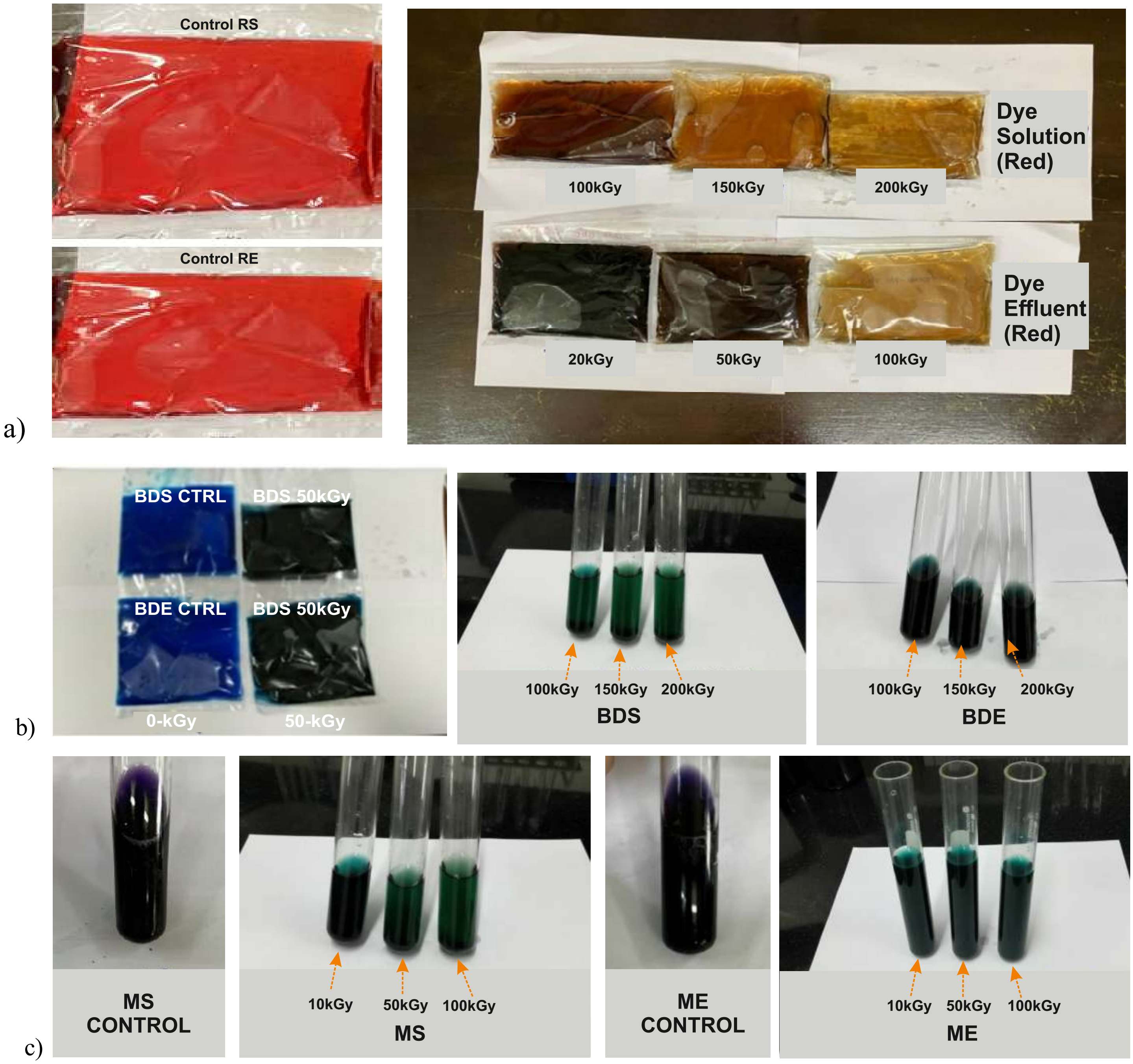

Fig. 5 Photographs of E-beam treated a) red dye b) blue dye and c) mixed dye samples at variable doses of E-beam

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

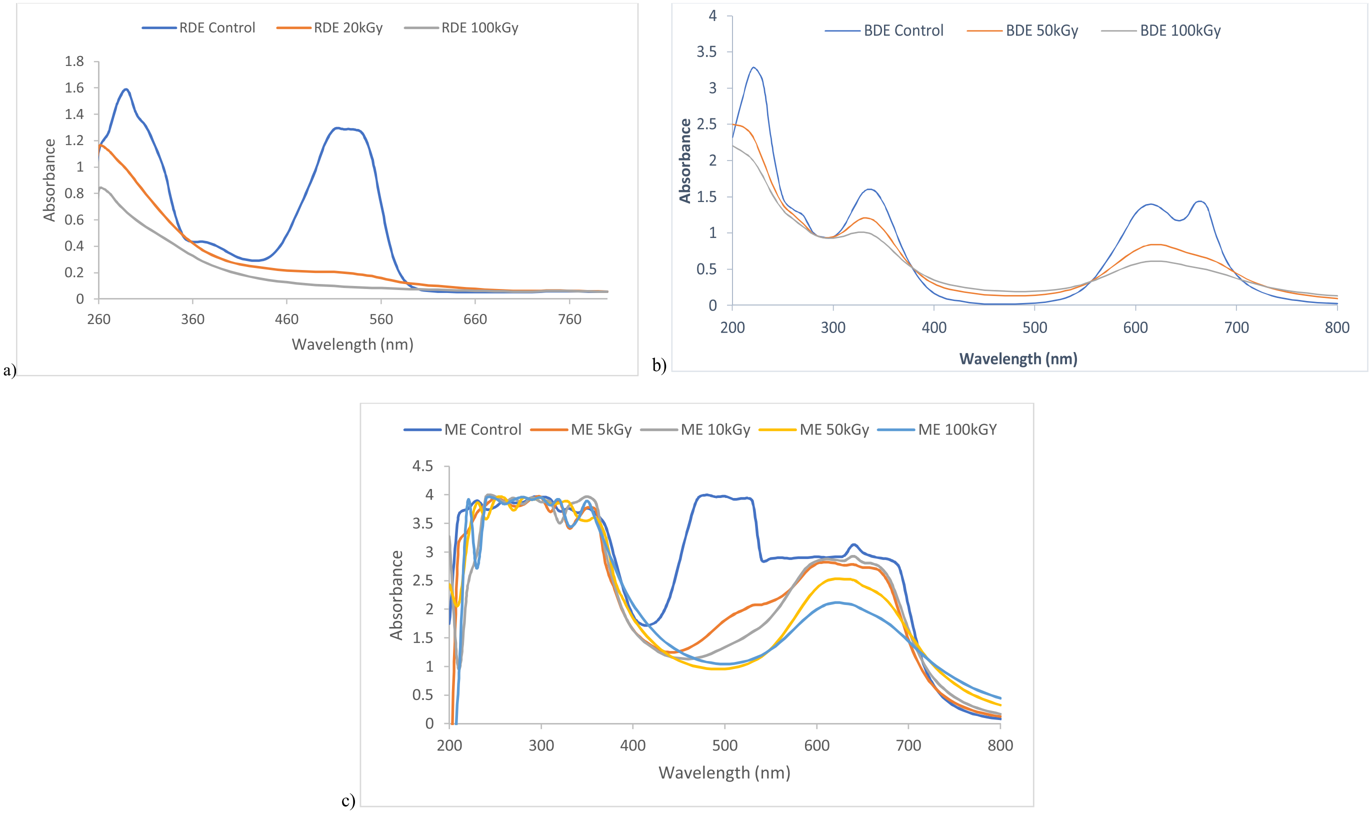

Fig. 6:UV visible spectrum of E-beam treated a) Red, b) Blue and c)mixed dye effluent

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.

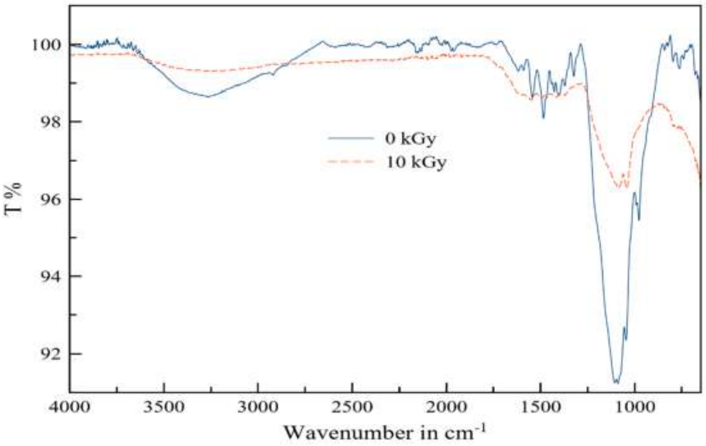

Fig. 7 : FTIR Spectrum of Reactive red dye effluent

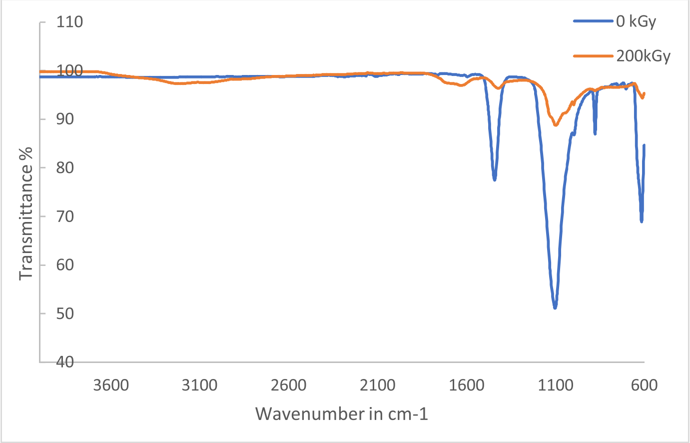

Fig. 8 : FTIR Spectrum of Reactive blue dye effluent in overall range

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).

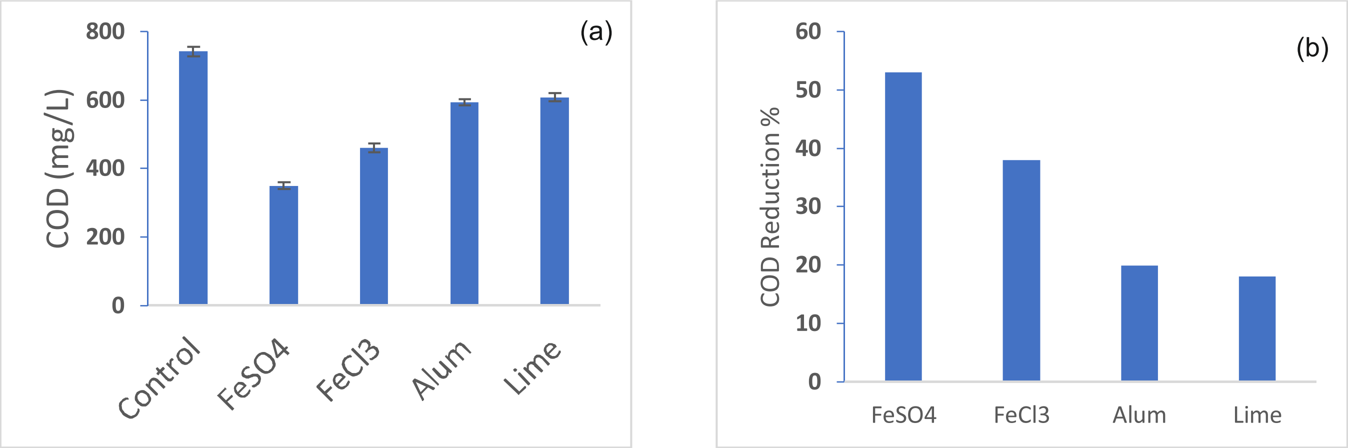

Fig.9 : Coagulant suitability study by Jar test method for treating mixed dye effluent a) in terms of COD reduction and b) % COD reduction

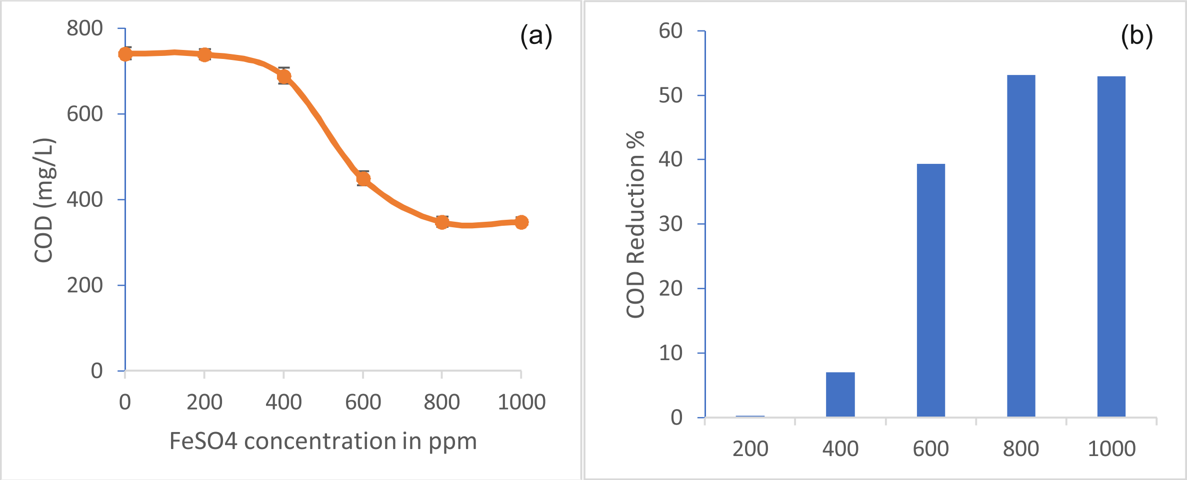

Fig. 10 : FeSO$_4$ dose optimization study by Jar test method for treating mixed dye effluent a) in terms of COD reduction and b) % COD reduction

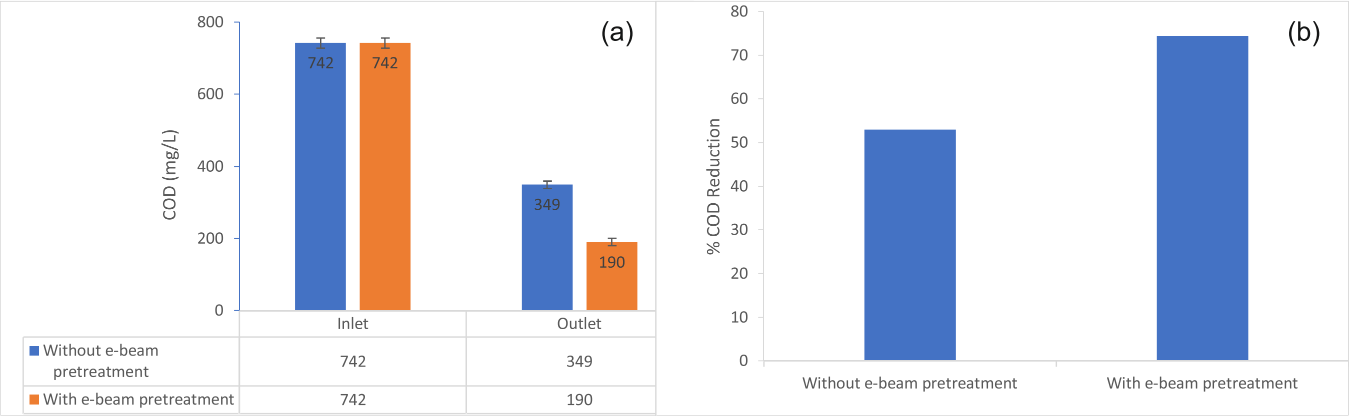

Fig. 10 : FeSO$_4$ dose optimization study by Jar test method for treating mixed dye effluent a) in terms of COD reduction and b) % COD reduction

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

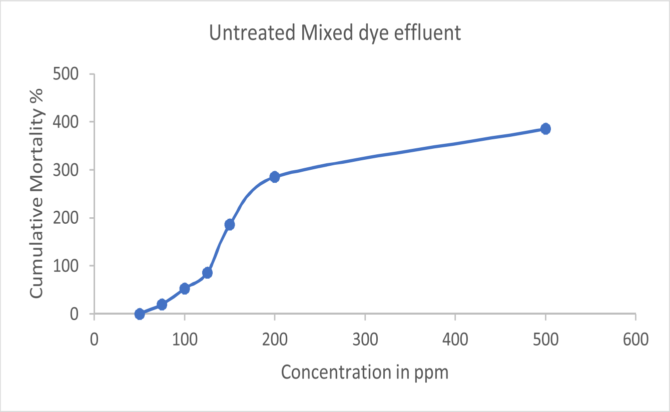

Fig. 12 : Acute Fish toxicity measurements of untreated mixed dye effluent samples

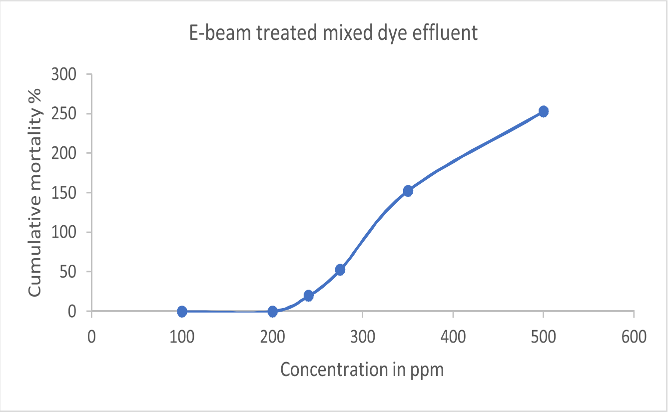

Fig. 13 : Acute Fish toxicity measurements of e-beam treated mixed dye effluent samples

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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Metal Organic Frameworks And Conductive Polymers For Energy Storage

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Abstract

The escalating demand for sustainable and flexible energy storage systems in wearable technology has driven research into advanced electrode materials. This mini-review explores the synergistic potential of metal-organic frameworks (MOFs) and conductive polymers (CPs) as hybrid electrode materials for next-generation smart textiles. While conducting polymers like polyaniline (PANI) and polypyrrole (PPy) offer high conductivity and flexibility, they suffer from mechanical fatigue and instability during cycling. Conversely, MOFs provide ultrahigh surface area and tunable porosity but are limited by poor intrinsic electrical conductivity. The hybridization of these materials creates hierarchical architectures that combine the pseudocapacitive behavior of CPs with the structural regularity of MOFs, significantly enhancing electrochemical performance. This review synthesizes recent progress in synthesis techniques, including in situ polymerization, layer-by-layer assembly, and electrochemical deposition, which are critical for integrating these composites into flexible fibers and fabrics. Detailed analysis of MOF@PEDOT and MOF@PPy hybrids highlights their superior specific capacitance, rate capability, and mechanical robustness under deformation. Despite these advances, challenges such as long-term stability under washing and scalable manufacturing remain significant hurdles. The paper discusses emerging solutions like core-shell nanocoatings and continuous processing methods to address these limitations. Ultimately, this review identifies future research directions for developing durable, high-performance energy storage textiles capable of powering autonomous wearable systems

Keywords

Metal-Organic Frameworks (MOFs), Conductive Polymers, Flexible Supercapacitors, Smart Textiles, Energy Storage, Hybrid Composites, Wearable Electronics, Electrochemical Performance

Citation

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1.0 Introduction:

The use of fossil fuels has led to severe environmental challenges, particularly climate change and global warming, thereby accelerating the demand for sustainable and low-carbon energy technologies. Among various solutions, renewable energy sources such as solar, wind, hydro, and biomass have gained prominence; however, their intermittent nature necessitates efficient, reliable, and scalable energy storage systems to ensure a stable power supply [1]. In this context, electrochemical energy storage devices, especially supercapacitors and rechargeable batteries, have emerged as indispensable components for portable electronics, wearable systems, and next-generation smart grids due to their high efficiency, fast response, and design flexibility [2].For advanced energy storage, the performance of the device is largely dictated by the properties of the active electrode materials, which are expected to combine high electrical conductivity, large accessible surface area, mechanical robustness, and chemical stability within a single platform. Conventional materials such as porous carbons, metal oxides, and metal sulfides have been extensively explored, yet they often suffer from intrinsic drawbacks, including limited conductivity, sluggish ion transport, or structural degradation upon cycling, which restrict their practical energy and power densities [3]. These limitations have driven intensive research toward emerging classes of functional materials, particularly conductive polymers (CPs) and metalorganic frameworks (MOFs), and their rationally designed hybrids.

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Conductive polymers, such as polyaniline, polypyrrole, and PEDOT, are attractive for supercapacitor electrodes owing to their high electrical conductivity, tuneable redox activity, and processability into flexible films, fibers, and coatings compatible with textile substrates. They exhibit pronounced pseudocapacitive behaviour, where fast and reversible faradaic reactions at the polymer–electrolyte interface enable high specific capacitance and power capability, making them suitable for lightweight and deformable devices. However, pure CPs often undergo volumetric changes and mechanical fatigue during repeated cycling, resulting in capacity fading and poor long-term stability, especially under bending and stretching conditions relevant to smart textiles [4, 5].

MOFs, composed of metal ions or clusters bridged by organic linkers, offer an exceptionally high surface area, structural diversity, and tuneable pore architecture, making them promising precursors or hosts for energy storage materials. Although pristine MOFs frequently exhibit low intrinsic electrical conductivity, their porous frameworks facilitate ion diffusion and can serve as scaffolds for the growth or confinement of conductive species, thereby enabling hierarchical architectures with improved electrochemical response. By judiciously selecting metal nodes, organic linkers, and post-synthetic modifications, MOF-derived structures can be engineered to balance porosity, conductivity, and mechanical integrity, which are crucial for integration into flexible and wearable devices [6].

Hybridization of CPs with MOFs has recently emerged as a powerful strategy to synergistically couple the pseudocapacitive characteristics of polymers with the high surface area and structural regularity of MOFs. In such composites, the CP component can impart electrical pathways and mechanical flexibility, while the MOF framework enhances ion-accessible surface, stabilizes the polymer chains, and mitigates volumetric changes during cycling, resulting in improved capacitance, rate capability, and cycling stability. Moreover, the morphology of these hybridswhether core–shell structures, interpenetrating networks, or MOF coated fiberscan be finely tuned by controlling parameters such as surfactant type, reactant concentration, reaction temperature, and polymerization time, which directly influence charge transport and ion diffusion [7].

Smart textiles demand energy storage units that are not only electrochemically efficient but also lightweight, breathable, mechanically flexible, and capable of withstanding repeated deformation without performance loss. CP/MOF-based electrodes can be conformally deposited onto fibers, yarns, or fabrics, enabling the realization of flexible supercapacitors that can be woven, sewn, or laminated into garments while maintaining stable operation under bending, twisting, and stretching. Such textile-integrated devices are particularly attractive for powering wearable sensors, health-monitoring systems, and human–machine interfaces, where on-body energy storage must be safe, unobtrusive, and durable [8].

This mini-review focuses on recent progress in the design and fabrication of flexible supercapacitors based on MOFs, conductive polymers, and their composites for smart textile applications. Emphasis is placed on material design principles, hybridization strategies, structure–property relationships, and device-level performance metrics relevant to wearable energy storage. Remaining challenges such as long-term mechanical and electrochemical stability under real-use conditions, large-area scalable processing, washability, and integration with other textile componentsare discussed, together with future research directions toward truly autonomous and multifunctional energy storage textiles.

2.0 Properties of Metal-Organic Frameworks (MOFs)

MOFs offer tuneable porosity, ultrahigh surface areas (>5000 m^2 g^{-1}), and modular structures formed by metal nodes and organic linkers, distinguishing them from conventional porous materials for supercapacitor applications. These features enable precise control over pore hierarchies (micro- to macropores) for rapid ion diffusion and abundant pseudocapacitive sites, with conductivities enhanced via redox-active metals (Ni, Co) or thiophene linkers. Diverse routes solvothermal, microwave-assisted, or mechanochemical allow substitution of nodes/ligands, nanosizing, or flexible linker incorporation to boost mechanical resilience against cycling-induced volume changes, critical for textile electrodes. Derivatives like MOF-derived carbons or thin 2D nanosheets (<10 nm thick) further optimize electron pathways while preserving hierarchical porosity [9].Despite advantages in ion-accessible surfaces, pristine MOFs face poor intrinsic conductivity (<10-6 S cm^{-1}), pore collapse under repeated charge–discharge, and pH sensitivity, limiting capacitances to 100-500 F g^{-1} and cycling retention. Stability improves with high-valent metals (Zr4+) paired to hard bases or divalent soft ions, alongside scalable, low-cost synthesis from mineral salts. Hybridization with conductive polymers addresses these gaps, synergistically enhancing rate capability (>90% retention over 5000 cycles) and flexibility for smart textiles.

3.0 Synthesis Techniques for MOF–Polymer Composites

In situ polymerization, layer-by-layer (LbL) assembly, and electrochemical deposition enable intimate integration of MOFs with conductive polymers (CPs) like polyaniline (PANI), polypyrrole (PPy), or PEDOT, ensuring uniform coatings, hierarchical porosity, and synergistic conductivity enhancement for flexible supercapacitors.

3.1.1 In Situ Polymerization

MOF crystals serve as templates where monomer polymerization occurs directly on surfaces or within pores, yielding core–shell or interpenetrating CP@MOF architectures. Parameters such as oxidant concentration (e.g., APS for PANI), pH, temperature (20–80°C), and reaction time (1–24 h) control polymer thickness (10–100 nm) and loading, boosting specific capacitance by 2–3× (e.g., PPy–ZIF-67 hybrids achieving 650 F g^{-1}) while mitigating MOF brittleness [10].

3.1.2. Layer-by-Layer Assembly

Alternating deposition of anionic MOF nanosheets and cationic polymer layers via electrostatic interactions produces freestanding films (5–50 nm per bilayer) with precise thickness control and >90% capacitance retention after 5000 bends. Ideal for textiles, this method preserves MOF porosity (BET >2000 m2g^{-1}) and imparts CP-mediated conductivity (>10 S cm^{-1}), as demonstrated in PANI–UiO-66 membranes [11].

3.1.3. Electrochemical Deposition

One-step electro-polymerization on MOF-coated electrodes or conductive fabrics yields conformal CP coatings (20–200 nm) with tailored pseudocapacitive sites. Cyclic voltammetry (-0.2 to 1.0 V) or chronoamperometry optimizes film uniformity, enabling fiber-shaped devices with 300–500 F g^{-1} and stability under 180° bending [12].

3.1.4 Diffusion Method

The diffusion method gradually transports reactants into contact to promote controlled crystal growth, primarily yielding single crystals suitable for X-ray diffraction analysis, especially for low-solubility MOFs. In solvent liquid diffusion, two density-stratified layers precipitant solvent and product-laden solventare separated by an intermediate solvent layer; crystals nucleate at the interface as the precipitant diffuses slowly. Alternatively, physical barriers (e.g., nested vials) or gels slow diffusion further, preventing bulk precipitation and enabling high-quality MOF nucleation for composite precursors [13].

3.1.5 Hydrothermal/Solvothermal Method

Hydrothermal/solvothermal synthesis drives self-assembly of MOFs from soluble precursors in sealed autoclaves under autogenous pressure (80-260°C), with cooling rate dictating crystallization kinetics. Though reaction times span days to weeks, this approach excels in morphological control, producing uniform nanocrystals ideal for polymer infiltration or coating in composites. Microwave assistance accelerates the process to hours, generating seeding conditions akin to solvothermal routes while enabling precise tuning of particle size and shape for scalable supercapacitor electrodes [14].

3.1.6 Electrochemical Method

Electrochemical synthesis generates MOFs directly on conductive supports by in situ metal ion release, bypassing aggressive anions (e.g., nitrates) and enabling rapid, low-temperature (<solvothermal) growth with minimal thermal cracking from mismatched expansion coefficients. Voltage tuning or pulsed signals fine-tune nucleation near the substrate, reducing bulk aggregation and favouring thin films or membranes for polymer hybridization. This industrially viable route produces phase-pure MOF powders or coatings with hierarchical porosity, enhancing CP integration for flexible textiles.Microwave-assisted in situ growth accelerates synthesis (<30 min) for scalable CP–MOF yarns; 3D printing extrudes MOF–PEDOT inks into porous architectures; and plasma-enhanced polymerization grafts ultrathin CPs onto MOFs for breathable textiles. These innovations achieve >85% retention over 10,000 cycles and areal capacitances >1 mF cm^{-2}, addressing scalability for smart wearables.

3.2 MOF@CP Hybrid Strategies

MOF@CP hybrids integrate metal–organic frameworks with conductive polymers through strategies that maximize interfacial synergy, preserving MOF porosity while enhancing conductivity and mechanical flexibility for textile supercapacitors. These methods address CP volumetric instability during cycling and MOF powder processability, yielding freestanding electrodes with π–π-stabilized charge transport.

3.2.1 MOF Hybrids with Poly(3,4-ethylenedioxythiophene) (PEDOT)

PEDOT stands out for its exceptional conductivity (up to 1000 S cm^{-1}), optical transparency, and stretchability (>4% strain), making it highly suitable for flexible MOF hybrids in smart textile supercapacitors. Hybrids leverage PEDOT's film-forming ability to create binder-free, breathable electrodes with enhanced cycling stability and rate performance.

Table.1.Supercapacitor performance of MOF@PEDOT hybrids and their comparison of electrochemical properties.

Hybrid material	Gravimetric/ Areal Capacitance	Current density/ Scan rate	Capacity retention (cycles)	Energy density/ Power density	Electrolyte (Potential window)	Ref
PEDOT:PSS@ HKUST-1	31 F g^{-1}	1 A g^{-1}	90.1 % (10 000) at 0.2 A g^{-1}	-	1M Na_2SO_4 (0.35 V)	15
UiO-66/GO/CNTF/ PEDOT	30 mF cm^{-2}	5 mV s^{-1}	89% (1000)	0.0022mWh cm^{-2}; 0.2 mW cm^{-2} at 0.4 mA cm^{-2}	PVA/H_3PO_4 (1 V)	16
HKUST-1/GO/CNTF/PEDOT	37.8 mF cm^{-2}	5 mV s^{-1}	89.8% (2000)	0.051 mWh cm^{-3}; 2.1 mW cm^{-3} at 0.4 mA cm^{-2}	PVA/H_3PO_4 (1 V)	17

Table 2. Electrochemical Performance of Selected MOF@PPy Hybrids

Hybrid material	Gravimetric/ Areal Capacitance	Current density/ Scan rate	Capacity retention (cycles)	Energy density/ Power density	Electrolyte (Potential window)	Ref
PPy–CPO-27-Ni PPy–CPO-27-Co PPy–HKUST-1	354 F g^{-1} 263 F g^{-1} 185 F g^{-1}	-	42.3% (2500) 68.3% (2500) 27.1% (2500)		1 M KCl (1 V)	18
PPy@UIO-66@CT	565 F g^{-1}	0.8 mA cm- 2	90% (500)	38.2 Wh kg^{-1}; 37.7 W kg- 1	1 M H2SO4/0.4 M hydroquinone (0.8 V)	19
Cu-TCPP/PPy	240 F g^{-1}	2 A g^{-1}	68.5% (3000) 5 A g^{-1}	0.29 mWh cm^{-3}; 270 mW cm^{-3}	PVA/H2SO4 (0.9 V)	20
(NiCo-MOF@PPy)//AC	132 F g^{-1}	0.5 A g^{-1}	79.1% (10 000)	41.2 Wh kg^{-1}; 375 W kg^{-1}	2 M KOH (1.5 V)	21
Cu-CAT -NWAs/PPy	252.1 mF cm^{-2}	1.25 mA cm^{-2}	90% (8000) at 100 mV s- 1	22.4 µWh cm^{-2}; 1.1 mW cm^{-2}	LiCl/PVA gel electrolyte (0.8 V)	22

3.2.2 MOF Hybrids with Polypyrrole (PPy)

Polypyrrole (PPy) is widely employed in MOF hybrids due to its facile electro-polymerization, high pseudo-capacitance (from reversible redox), and biocompatibility, effectively enhancing MOF conductivity and mechanical adhesion to textiles. PPy coatings (20–150 nm) suppress MOF collapse while enabling flexible, aqueous-stable electrodes with improved rate capability.

4.0 Challenges and Opportunities in MOF-Based Smart Textiles

MOF@CP hybrids face critical hurdles for practical smart textile deployment, including cycling-induced structural degradation, limited intrinsic conductivity, and processing scalability. Opportunities lie in targeted material engineering and manufacturing innovations to unlock commercial viability. Stability Challenges: Repeated deformation and laundering cause MOF pore collapse and CP delamination, reducing capacitance by 20–40% after 5000 wash cycles; aqueous electrolytes exacerbate linker hydrolysis in acid/base conditions. Mechanical fatigue under >30% strain leads to microcracks, compromising breathability and skin comfort. Scalability Barriers: Lab-scale solvothermal synthesis yields grams, not kilograms; high-cost linkers (e.g., BTC >\$50 kg^{-1}) and solvent-intensive processes hinder textile-yardage production. Uniform coating over km-scale fabrics remains inconsistent, with edge effects causing 30% performance variation. Conductivity and Metrics Gaps: Bulk MOF conductivity (<10^{-5} S cm^{-1}) demands >30 wt% CP, diluting capacitance density; areal metrics lag textiles (<1 mF cm^{-2} vs. >5 mF cm^{-2} needed). Energy density (20–50 Wh kg^{-1}) falls short of Li-batteries for all-day wearables.Emerging Solutions: Core–Shell Nanocoating: Atomic layer deposition of hydrophobic silanes protects MOFs, achieving 95% capacitance retention post-100 laundry cycles.Continuous Roll-to-Roll Processing: Microwave/plasma-assisted synthesis coats 100 m fabrics/min at <10% cost premium.Conductive MOF Engineering: Thiophene-linker or Ni/Co-node doping reaches 10 S cm^{-1}, minimizing CP loading to 10 wt%.

5.0 Conclusion

This paper reviews the latest breakthroughs in MOF–conductive polymer research, offering strategies for designing and optimizing materials tailored for wearable energy storage systems. These insights aim to drive innovation in next-generation smart textile technologies. Comparative research on synthesis techniques and their outcomes can clarify the mechanisms behind hybridization improvements. Several strategies exist to enhance current materials. One approach involves novel conductive MOFs, particularly bimetallic variants with superior electrochemical properties. Another focuses on CP nanostructures (dendrimers, nanofibers, nanotubes) that provide higher surface area and optimized MOF/CP/electrolyte interfaces. These advances may necessitate new MOF@CP preparation methods. Further research should maximize CP mechanical properties to create robust, bendable, stretchable materials for organic electronics. Finally, practical efforts must prioritize long-term stability and durability testing to enable market-ready MOF@CP hybrids.

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Plasma-Assisted Surface Engineering of Textiles and Polymers for Improved Flame Retardancy: A Comprehensive Review

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Abstract

Flame-retardant materials are increasingly critical across textiles, polymers, electronics, and construction, yet conventional chemical finishing often compromises fabric integrity, durability, and environmental safety. Plasma-assisted surface modification has emerged as a sustainable alternative, selectively functionalizing the outer nanolayers of substrates without altering bulk properties. This review highlights the principles and types of plasma, particularly non-thermal methods such as dielectric barrier discharge (DBD), corona, and glow discharge, and examines their role in enhancing adhesion, durability, and performance of flame-retardant coatings on cotton, polyester, polypropylene, and polyamide. Compared with traditional chemical, sol–gel, or inherently flame-resistant approaches, plasma treatment offers superior surface specificity, reduced chemical usage, and eco-friendliness. Despite challenges in cost and process optimization, plasma-assisted strategies represent a versatile route for next-generation, durable, and environmentally responsible flame-retardant materials.

Key words:

Plasma treatment, Flame retardancy, Surface modification, Dielectric barrier discharge (DBD).

Citation

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1. Introduction:

The increasing demand for materials exhibiting enhanced flame-retardant performance has significantly increased interest in the development of efficient, sustainable, and technologically advanced strategies for improving fire resistance across a broad spectrum of industrial sectors. In applicationslike electronics, textiles, transportation, and construction, fire safety is not only a regulatory requirement but also a critical factor influencing product performance, durability, and human safety. Conventionally, flame-retardant treatments have relied heavily on halogenated compounds and other chemically intensive systems. Although effective, these materials are associated with significant drawbacks, including the release of corrosive and toxic gases during combustion, which pose serious harm to living beings and the environment [1]. In response to these limitations, plasma-based surface modification techniques have emerged as promising alternatives, offering a viable route to meet both performance and sustainability requirements [2,3].

From a textile flame-retardancy perspective, plasma treatment functions primarily as a surface-engineering tool that enhances fibre–chemical interactions rather than as a bulk modification technique.In flame-retardant finishing applications, plasma is valued for its ability to generate highly reactive surface sites that promote wettability, adhesion, and chemical anchoring of flame-retardant systems.. These interactions can significantly alter surface energy, introduce reactive functional groups, and improve wettability, thereby facilitating improved adhesion or grafting of flame-retardant agents [3]. In contrast to conventional dip-coating or chemical soaking processes, plasma treatments are inherently dry, require minimal chemical consumption, and enable precise control over surface modification. As a result, they reduce chemical waste, improve process uniformity, and enhance reproducibility, making them attractive for advanced material processing.

Over the past decade, extensive research has developed the efficiency of plasma-assisted treatments in improving flame retardancy across a wide range of substrates, including polymers, textiles, foams, and composite materials. Notably,

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atmospheric pressure plasma treatments have been shown to enhance the uptake and retention of phosphorus-based flame-retardant systems in polyester and polyamide fabrics [4,5]. These surface-induced modifications contribute to measurable improvements in key fire-performance parameters such as Limiting Oxygen Index (LOI), thermal stability, and char formation, all of which are essential for passive fire protection assessment. Importantly, such enhancements are typically achieved without compromising critical end-use properties, including mechanical strength, flexibility, or wearer comfort, which is particularly relevant for consumer-oriented textile applications [6].

Beyond simple surface activation, plasma-treated substrates also provide a favorable platform for subsequent graft polymerization processes. In such systems, flame-retardant monomers can be covalently bonded directly onto the substrate surface, significantly improving the durability and permanence of the flame-retardant functionality. Several studies have reported that plasma-induced grafting leads to sustained flame-retardant performance even after repeated laundering cycles, representing a substantial advancement over traditional coating-based approaches. In this context, dielectric barrier discharge (DBD) plasma has been widely employed as a pretreatment technique to enhance surface capillarity and wettability, thereby promoting deeper penetration and stronger fixation of flame-retardant formulations applied in subsequent finishing steps [6,7].

In addition to performance benefits, plasma-assisted flame-retardant treatments are increasingly recognised for their environmental compatibility. Unlike conventional wet-chemical processes, plasma-based techniques operate with little to no solvent usage, leading to a significant reduction in wastewater generation and residual chemical discharge. This characteristic aligns plasma processing with the principles of green chemistry and sustainable manufacturing, making it particularly attractive for industries facing strict environmental regulations and increasing consumer urging for eco-friendly products [8].

Another notable advantage of plasma technology lies in its high degree of tunability. By carefully selecting process parameters such as plasma gas composition, input power, and treatment duration, it is possible to tailor surface chemistry to meet specific application requirements. For example, oxygen or nitrogen plasmas can introduce polar functional groups that enhance the interaction of substrates with phosphorus- or nitrogen-based flame-retardant systems, while inert gases such as argon are commonly used to activate surfaces without introducing additional chemical functionalities or by-products [9]. This level of control provides a versatile platform for designing material-specific and performance-driven flame-retardant strategies.

Furthermore, plasma treatments can be readily integrated with hybrid flame-retardant technologies, including sol–gel coatings and nanoparticle-assisted systems. Such combined approaches enable the development of multifunctional finishes that not only improve fire resistance but also impart additional properties such as water repellency, ultraviolet protection, or antimicrobial activity [10]. For instance, plasma activation of cotton fabrics has been shown to significantly enhance the adhesion and long-term durability of silica-based sol–gel flame-retardant coatings, thereby improving both functional performance and wash resistance [11].

From an industrial perspective, plasma-assisted processes offer substantial advantages in terms of scalability and compatibility with continuous manufacturing operations. Atmospheric pressure plasma systems, in particular, enable roll-to-roll processing of fabrics, polymer films, and foams, facilitating seamless integration into existing production lines. This scalability, combined with reduced resource consumption and enhanced durability of flame-retardant finishes, positions plasma technology as a strong candidate to replace conventional wet-finishing methods in next-generation material processing [12].

Overall, plasma-assisted surface modification emerges as a powerful and sustainable approach for imparting flame retardancy while simultaneously enabling multifunctionality and long-term performance. In light of these developments, this review focuses on the fundamental mechanisms, recent advancements, and future prospects of plasma-assisted flame-retardant enhancement across various material systems.This surface-confined modification strategy is particularly advantageous for textiles, where preservation of mechanical integrity, flexibility, and wearer comfort is essential.

2.0 Types of Plasma

Plasma is widely recognized as the fourth state of matter and is defined as a partially ionized gas comprising a complex ensemble of electrons, ions, excited atoms and molecules, free radicals, and photons [13]. Unlike solids, liquids, or gases, plasma exhibits collective behavior governed by electromagnetic interactions, which allows it to induce unique physical and chemical effects at material surfaces. From a materials engineering perspective, plasma processing is particularly attractive because it enables controlled surface modification without significantly affecting the bulk properties of the substratean advantage that is rarely achieved using conventional wet-chemical or thermal treatments [14].

The interaction of plasma with solid surfaces depends strongly on plasma characteristics such as temperature, electron density, degree of ionization, pressure, and energy distribution. Consequently, plasmas are commonly classified based on their thermal equilibrium and operating pressure, both of which play a decisive role in determining their suitability for different material systems. In general, plasmas are classified into thermal (equilibrium) and non-thermal (non-equilibrium) plasmas [15, 16].

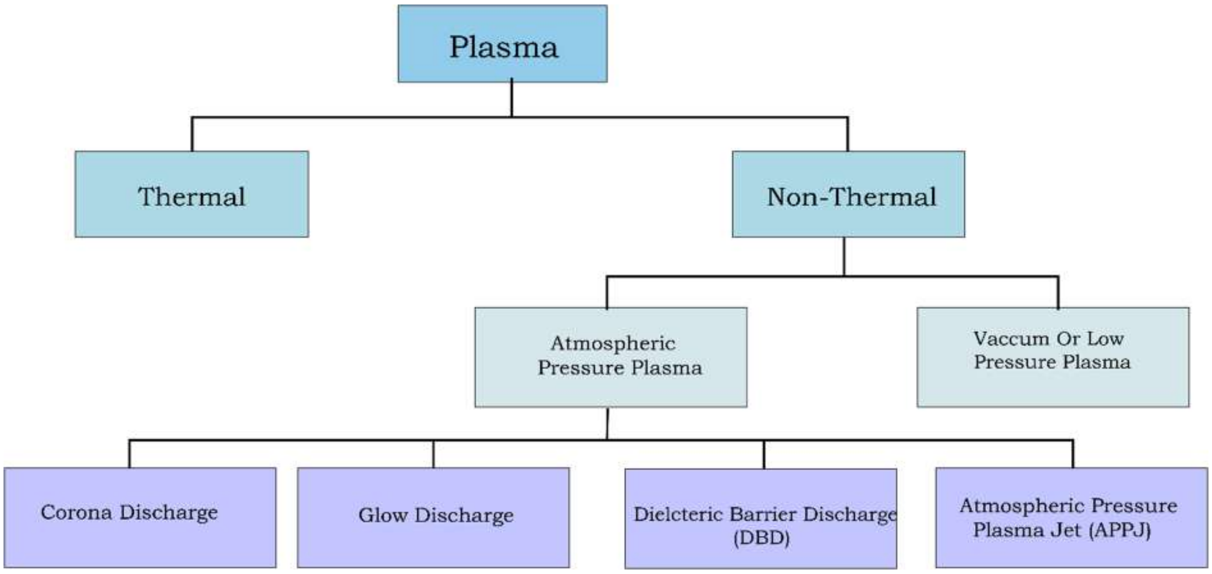

Fig 1. Types of Plasma

2.1. Thermal Plasma

Thermal plasma, also referred to as equilibrium plasma, is characterised by nearly equal temperatures of electrons, ions, and neutral species [17]. These plasmas operate at extremely high temperatures, often exceeding 1000–10,000 K, and are typically generated using arc discharges or plasma torches. Due to their high enthalpy and energy density, thermal plasmas are extensively used in applications such as metal cutting, welding, plasma spraying, and waste treatment.

However, the elevated temperatures associated with thermal plasmas render them unsuitable for heat-sensitive materials such as polymers and textiles. Exposure to thermal plasma would result in severe degradation, melting, or complete destruction of fibrous substrates. As a result, thermal plasma systems have very limited relevance in textile flame-retardant finishing and are not considered viable for surface engineering of organic materials.As a result, thermal plasma systems are not considered viable for textile flame-retardant finishing and are largely restricted to high-temperature industrial applications.

2.2 Types of Non-Thermal Plasma

Non-thermal plasma systems are of particular interest for flame-retardant textile processing because they enable highly reactive surface modification while maintaining the structural integrity of fibrous substrates. In these systems, electrons attain high kinetic energy, while the bulk gas temperature remains close to ambient conditions. This non-equilibrium state enables non-thermal plasmas to induce surface-confined chemical reactions, including bond scission and the formation of reactive functional groups, without causing thermal degradation or compromising the structural integrity of the underlying substrate.

Owing to their low thermal load combined with high surface reactivity, non-thermal plasma systems have attracted significant interest for the modification of polymeric and textile materials, particularly in flame-retardant surface engineering. Such treatments allow precise tailoring of surface chemistry and surface energy while preserving the intrinsic bulk properties of heat-sensitive textile substrates, including natural, synthetic, and blended fabrics. Based on operating pressure, non-thermal plasmas are generally classified into low-pressure (vacuum) plasma [18] and atmospheric-pressure plasma systems, each offering distinct advantages in terms of treatment uniformity, process controllability, scalability, and compatibility with textile manufacturing lines.

In addition to operating pressure, the discharge configuration strongly influences plasma–textile interactions and determines the suitability of a given plasma source for specific textile applications. Among the various non-thermal plasma technologies, dielectric barrier discharge (DBD), corona discharge, glow discharge, and plasma jet systems are the most widely adopted in the textile industry for surface modification and functional finishing. These plasma sources differ in discharge mechanism, energy distribution, penetration depth, and treatment homogeneity, which directly affect their effectiveness in activating fiber surfaces, improving wettability, enhancing coating adhesion, and increasing the durability of flame-retardant finishes.

Specifically, DBD plasma is extensively used in textile processing due to its ability to provide uniform, large-area treatment under atmospheric pressure and its compatibility with continuous roll-to-roll fabric production. Corona discharge plasma is commonly applied for rapid surface activation of textile and polymer films, particularly where improved wettability and coating spreadability are required. Glow discharge plasma, typically operated under low-

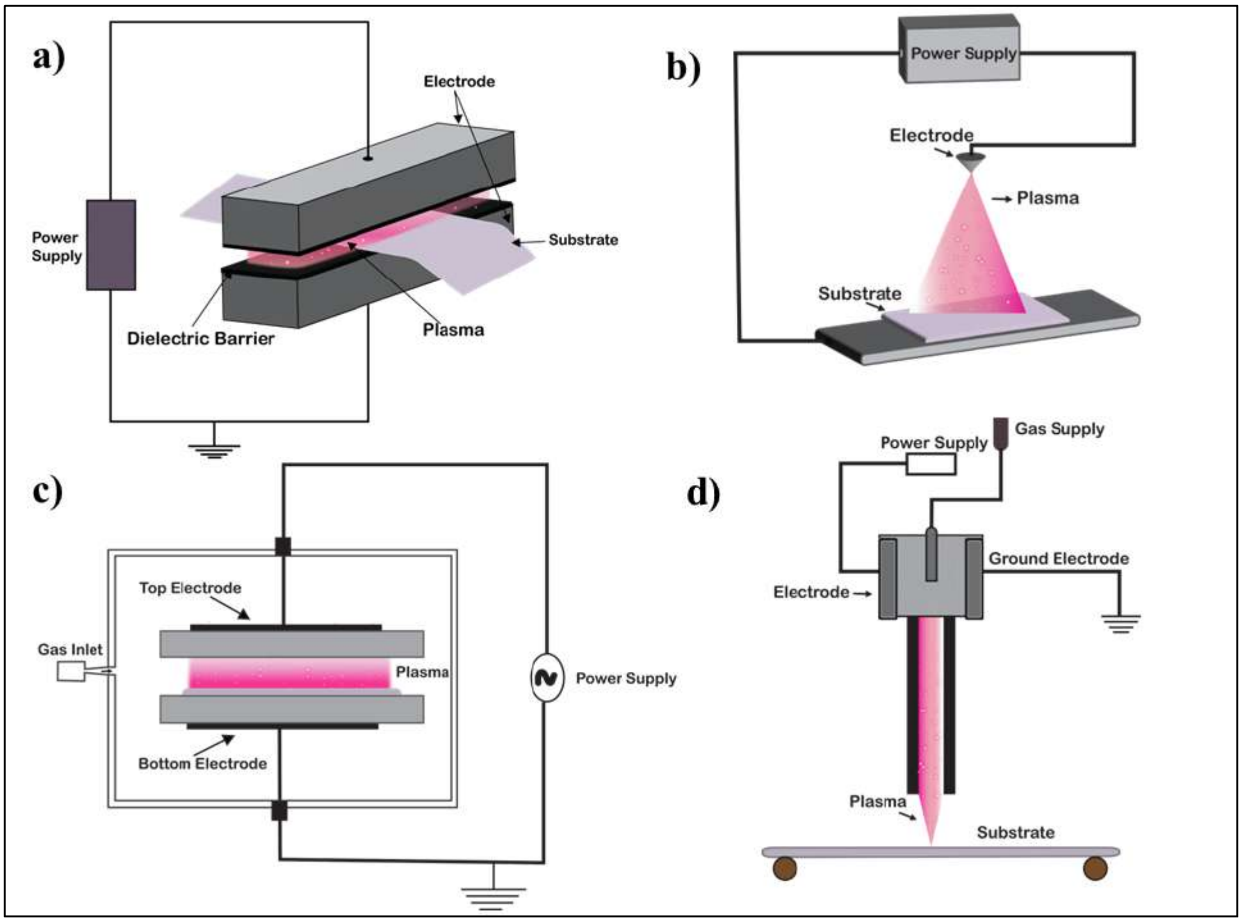

Fig 2.Types of Non-Thermal Plasma a) Dielectric Barrier Discharge b) Corona Discharge Plasma, c) Glow Discharge Plasma, d) Plasma Jet Technology

pressure conditions, offers precise and uniform surface functionalization and is well suited for research-scale studies and high-value textile applications requiring controlled modification. Plasma jet technology, on the other hand, enables localized and selective treatment of textiles and is increasingly explored for patterned functionalization and integration with hybrid flame-retardant finishing systems.

The following subsections therefore discuss DBD, corona discharge, glow discharge, and plasma jet technologies in detail, with particular emphasis on their operational principles, advantages, and relevance to flame-retardant treatments in textile applications.

2.2.1. Dielectric Barrier Discharge (DBD)

Among atmospheric-pressure plasma sources used for flame-retardant textile finishing, dielectric barrier discharge (DBD) plasma is particularly valued for its uniform treatment capability and compatibility with continuous fabric processing. The presence of the dielectric barrier plays a critical role in limiting the discharge current and distributing numerous short-lived micro-discharges uniformly across the electrode surface, thereby suppressing arc formation and preventing excessive thermal loading of the substrate [19]. Consequently, DBD plasma can be operated in a stable manner at atmospheric pressure while maintaining a low gas temperature, making it particularly applicable for the treatment of thermally responsive materials.

Owing to these characteristics, DBD plasma systems are widely employed for continuous surface modification of textiles, polymer films, and nonwoven substrates. In the context of flame-retardant finishing, DBD plasma treatment increases surface energy and promotes the formation of oxygen- and nitrogen-containing functional groups, which enhance wettability and interfacial adhesion. These surface modifications facilitate improved anchoring and long-term durability of flame-retardant coatings and additives on textile fibers. Furthermore, the commercial availability of roll-to-roll DBD plasma systems enables seamless integration into existing textile processing lines, underscoring their industrial relevance and scalability for large-area flame-retardant textile applications.

2.2.2. Corona Discharge

Corona discharge plasma is a form of non-thermal (cold) plasma generated by applying a high voltage across two asymmetrical electrodes, typically consisting of a sharply pointed wire or needle electrode and a grounded planar electrode, in a gaseous environment, most commonly air. This arrangement createsa non-uniform electric field, with intense field strength concentrated at the tip of the sharp electrode. As a result, gas molecules in the surrounding atmosphere become ionised, forming a localised plasma region identified by high electron density and relatively low gas temperature.

Corona discharge plasma is generally produced using sinusoidal voltages in the range of 10–15 kV at frequencies of several tens of kilohertz under atmospheric pressure (≈1 atm) [20]. The discharge current exhibits significant fluctuations and is primarily localised near regions of high electrode curvature, where the electric field intensity is greatest [21]. In polymer and textile surface modification, corona plasma is widely employed due to its ability to increase surface energy, enhance wettability, and introduce polar functional groups without changing the overallcharacteristics of the treated material. Key advantages of corona discharge include low power consumption, atmospheric-pressure operation, and straightforward integration into continuous industrial processing lines, making it particularly suitable for large-scale textile finishing applications.

2.2.3. Glow Discharge

Glow discharge plasma is typically generated under low-pressure conditions between two electrodes using direct current (DC) or radio-frequency (RF) power sources. Under these conditions, energetic species such as electrons, ions, and radicals are produced and interact with the surface of textile substrates, inducing both chemical and physical modifications. Glow discharge plasma is frequently applied for the surface treatment of natural fibres, such as cotton, where precise and uniform modification is required.

Low-pressure DC glow discharge treatment has been shown to significantly increase the flame-retardant performance of cotton fabrics through increased surface hydrophilicity and the introduction of oxygen-containing functional groups, including C–O, C=O, O–C–O, and O–C=O moieties. These functional groups improve surface wettability and capillary wicking behavior, which are critical for the effective uptake, dispersion, and fixation of flame-retardant formulations applied in subsequent finishing processes [22]. Although glow discharge plasma offers excellent control and treatment uniformity, its application is generally limited to batch processing due to the requirement for vacuum conditions.

2.2.4. Plasma Jet Technology

Plasma jet technology is a non-thermal plasma method in which plasma is generated between two concentric electrodes through which a continuous flow of process gassuch as helium, argon, oxygen, or their mixturesis introduced. The plasma is typically excited by radio-frequency or pulsed power sources, which energize free electrons. These electrons collide with the feed gas molecules, producing excited atoms, radicals, and additional ion–electron pairs, thereby sustaining the plasma state [23].

A major advantage of plasma jet systems is their ability to operate under atmospheric pressure without the need for a vacuum chamber, which significantly simplifies system design and enhances industrial applicability. Plasma jets enable localized and directional surface treatment, making them particularly suitable for selective functionalization, patterned modification, and treatment of complex or three-dimensional textile structures. In flame-retardant textile applications, plasma jet technology is increasingly explored as a pretreatment or activation step to enhance coating adhesion and to integrate hybrid flame-retardant systems, such as sol–gel or nanoparticle-based finishes.

3.0 Mechanism of Plasma

In flame-retardant surface engineering, the importance of plasma treatment lies less in plasma chemistry itself and more in the functional surface transformations induced on textile fibres. [24,25]. In plasma-assisted surface engineering, the interaction between these reactive species and the substrate is predominantly confined to the near-

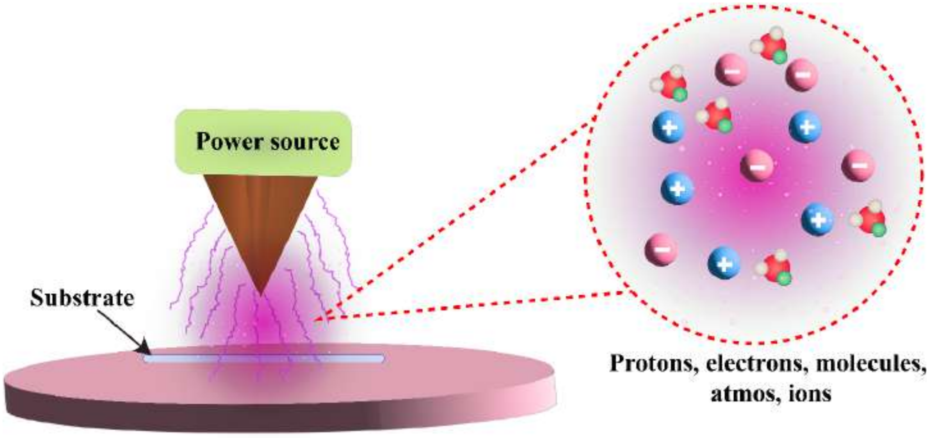

Fig 3.Graphical representation of plasma state

surface region, resulting in chemical and physical modifications without affecting the bulk structure of the material. This surface-selective nature distinguishes plasma treatment from conventional chemical methods, which may penetrate deeper into the substrate and compromise its structural integrity [24,25,26].

In the context of flame-retardant modification, plasma exposure creates reactive surface functionalities and nanoscale features that facilitate stronger interfacial bonding and more uniform distribution of flame-retardant formulations. [24,25]. These processes lead to the formation of new functional groups and increased surface roughness at the nanoscale, both of which enhance surface energy and chemical reactivity. As a result, the affinity of the substrate for flame-retardant agents is significantly improved, facilitating stronger interfacial bonding and more uniform distribution of the applied flame-retardant formulations [24,25,26,27].

Importantly, plasma treatment operates at low gas temperatures in non-thermal plasma systems, ensuring that the intrinsic mechanical and morphological properties of heat-sensitive substrates, such as textile fibers, remain unaffected. This makes plasma particularly suitable for fabrics and polymeric materials that are susceptible to degradation under aggressive chemical or thermal treatments. The plasma-modified surface either acts as an effective anchoring layer for flame-retardant coatings or promotes the formation of protective surface barriers that inhibit flame ignition and suppress flame propagation [24,26].

Furthermore, the effects of plasma treatment can be precisely controlled by adjusting process parameters such as gas composition, discharge power, and treatment duration. For instance, oxygen- or nitrogen-containing plasmas favor the introduction of polar functional groups, while inert gases primarily induce surface activation through physical etching. This high degree of tunability allows plasma treatments to be tailored to specific material systems and flame-retardant chemistries, establishing plasma technology as a versatile and effective tool for the development of advanced fire-resistant materials [24,25,26,28].By carefully controlling process parameters such as gas composition, discharge power, and treatment duration, plasma treatment can be tailored to maximize flame-retardant efficiency while minimizing fibre damage.

3.1. Pre- and Post-Plasma Treatment on Flame Retardancy

In plasma-assisted surface engineering, plasma treatment can be strategically applied either before or after the deposition of flame-retardant agents. The timing of plasma exposure plays a critical role in determining the effectiveness, durability, and interfacial bonding of flame-retardant systems with the substrate. Pre-plasma and post-plasma treatments serve distinct yet complementary functions, and the selection of either approach depends on the substrate chemistry, the nature of the flame-retardant formulation, and the targeted fire-performance requirements.

3.1.1. Pre-Plasma Treatment

Pre-plasma treatment is performed prior to the application of flame-retardant coatings or additives, with the primary objective of tailoring the surface chemistry and morphology of the substrate to enhance its receptivity toward subsequent finishing processes [2]. Plasma activation modifies the outermost surface layers of textile fibers by inducing bond cleavage and generating reactive functional groups, such as hydroxyl, carbonyl, carboxyl, amino, and amine moieties. The incorporation of these polar functionalities significantly increases surface energy and wettability, facilitating improved spreading, penetration, and uniform deposition of flame-retardant chemicals or nanostructured additives [18].

In addition to chemical activation, pre-plasma treatment may induce controlled surface roughening at the micro- or nanoscale, which further enhances mechanical interlocking between the substrate and the applied flame-retardant layer.

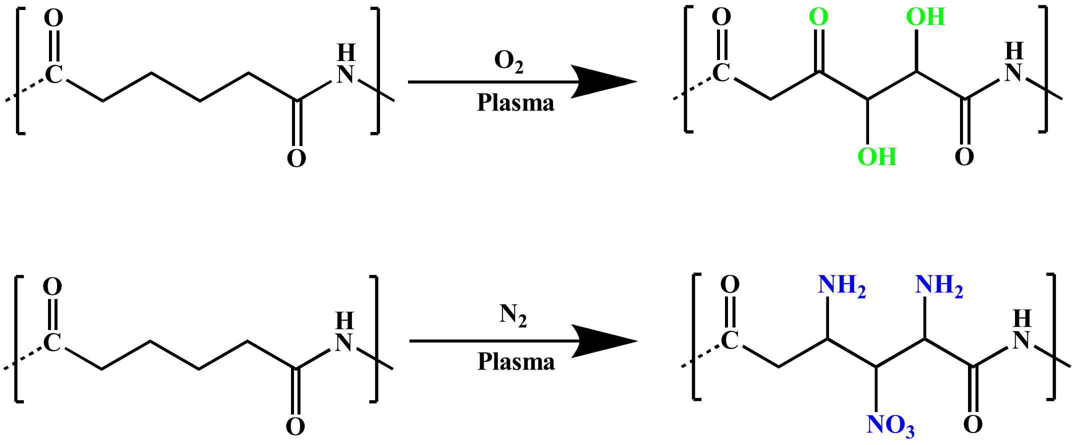

Fig 4. Surface functionalizationby Plasma treatment

Consequently, pre-plasma treatment improves coating adhesion, reduces leaching during laundering or use, and enhances the long-term durability of flame-retardant finishes, particularly for polymeric and textile substrates with inherently low surface reactivity.

3.1.2. Post-Plasma Treatment

Post-plasma treatment refers to plasma exposure applied after the flame-retardant agent or coating has been deposited onto the substrate. This approach is commonly employed to promote curing, crosslinking, and additional surface functionalization of the applied flame-retardant layer. The interaction of plasma-generated reactive species with the coated surface can induce surface crosslinking reactions, improve coating cohesion, and enhance film integrity.

Moreover, post-plasma treatment can activate residual monomers, binders, or reactive groups within the flame-retardant formulation, thereby strengthening interfacial bonding between the coating and the substrate. In some systems, plasma exposure facilitates deeper immobilization of flame-retardant elements or promotes the formation of protective surface barriers that improve resistance to thermal degradation and flame propagation. As a result, post-plasma treatment contributes to improved durability, reduced migration of active components, and enhanced flame-retardant performance without compromising the bulk properties of the underlying material.

To summarise, the principal differences between pre- and post-plasma treatment strategies are presented in Table 1.

The timing of plasma treatment, whether applied before or after flame-retardant deposition, has a substantial impact on the resulting fire-retardant performance. Pre-plasma treatment primarily ensures optimal surface activation, promoting uniform coating distribution and strong interfacial adhesion, particularly on hydrophobic or chemically inert substrates. In contrast, post-plasma treatment reinforces the applied coating, facilitates crosslinking and integration with the underlying substrate,

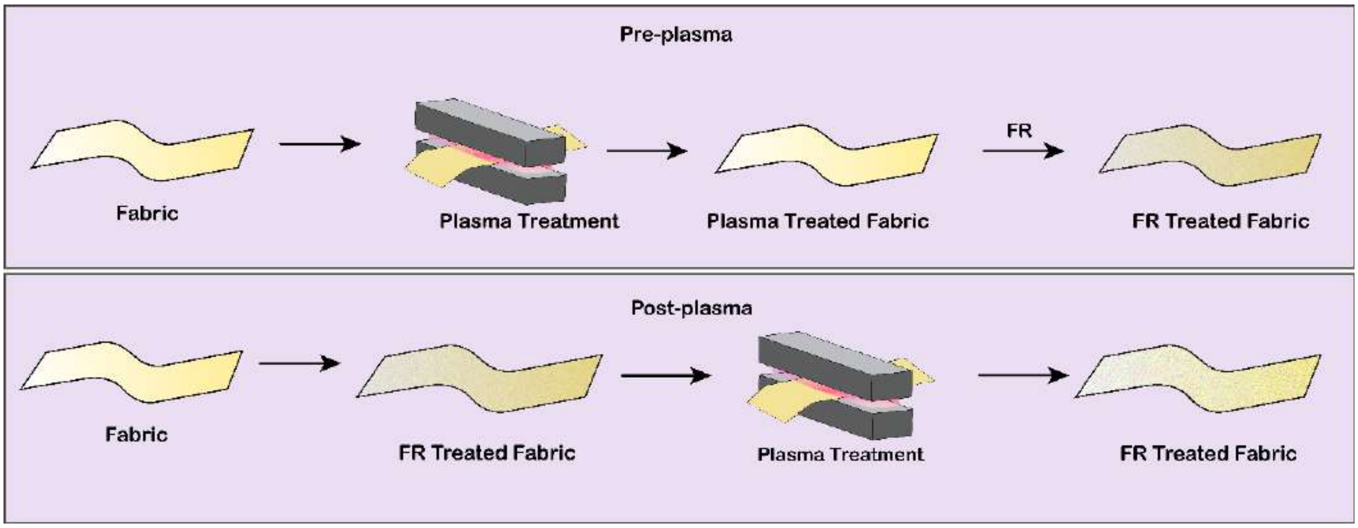

Fig 5. Graphical representation of Pre and Post-plasma treatment

Table 1.Comparison of pre- and post-plasma treatment on flame-retardant textiles

Aspect	Pre-Plasma Treatment	Post-Plasma Treatment
Treatment stage	Applied prior to flame-retardant deposition	Applied after flame-retardant application
Primary function	Surface activation and enhancement of interfacial adhesion	Coating fixation, crosslinking, and curing
Typical objective	Improve substrate wettability and bonding of flame-retardant systems	Enhance coating durability and flame-retardant performance
Suitable applications	Inert or low-surface-energy substrates	Biopolymer-based coatings and post-deposition enhancement
Processing gases	O2, Ar, N2	Air, N2, or mixed reactive gases
Representative systems	Polyester, polypropylene, nylon	Cotton–chitosan, epoxy–intumescent coatings
Resulting enhancement	Uniform coating, stronger interfacial bonding	Improved flame resistance, durability, and retention of active components

and improves the long-term durability of flame-retardant finishes.[24,25,26,29,30,31]

State-of-the-art plasma-assisted flame-retardant processes often combine both pre- and post-plasma treatments to maximize performance. This integrated approach leverages the advantages of surface activation and coating consolidation simultaneously, highlighting the versatility and effectiveness of plasma technology for advanced flame-retardant engineering.

4.0 Applications of Plasma Treatment in Flame Retardancy

Plasma treatment, once regarded largely as a laboratory-scale surface modification technique, has progressed into a mature and environmentally sustainable surface engineering approach with considerable relevance to textile flame retardancy. A key advantage of plasma technology is its ability to selectively alter only the outermost nanometric layers of textile fibres, thereby preserving the intrinsic bulk properties, including mechanical strength, flexibility, and aesthetic appearance. This highly surface-specific modification enables precise tailoring of interfacial chemistry, resulting in enhanced surface energy and the introduction of reactive functional groups that improve the adhesion, fixation, and durability of flame-retardant (FR) systems.

Owing to these characteristics, plasma treatment has emerged as an effective pre- or post-treatment strategy for functionalizing a broad range of textile substrates without excessive chemical consumption or harsh processing conditions. In the following sections, the contribution of plasma-assisted surface modification to improved flame retardancy is critically discussed with respect to four widely used textile materials: polyester, cotton, polypropylene, and polyamide.

4.1. Polyester

Polyester is widely used in apparel, home furnishings, and technical textiles owing to its high strength, dimensional stability, and chemical resistance. However, its inherent flammability, melt-dripping behavior, and chemically inert surface pose significant challenges for effective and durable flame-retardant finishing. Conventional flame-retardant treatments for polyester often rely on physically deposited coatings or additives, which frequently suffer from poor adhesion and limited durability. Consequently, plasma-assisted surface modification has been increasingly explored as a strategy to enhance fibre reactivity and improve the fixation of flame-retardant agents on polyester substrates.

Ayesh et al. [12] systematically investigated the role of atmospheric-pressure plasma treatment in enhancing the performance and durability of organophosphorus flame retardants on polyester fabrics. In their study, polyester fabrics were treated with commercially available flame retardants, including PE-CONC, PCO 900, and resorcinol diphenyl phosphate (RDP), followed by plasma exposure applied either before or after flame-retardant treatment. Plasma treatment, conducted with or without concurrent ultraviolet (UV, 308 nm) laser irradiation, generated reactive functional groups on the otherwise inert polyester surface, thereby enabling stronger chemical interactions between the flame-retardant molecules and the fibre. As a result, plasma-treated fabrics exhibited significantly improved retention of flame retardants, even after rigorous solvent resistance tests involving prolonged water and methanol soaking. Importantly, both plasma pre- and post-treatments markedly enhanced the durability of RDP-based finishes, demonstrating that plasma activationrather than UV irradiationwas the dominant factor governing improvement in the polyester–flame-retardant interfacial strength.

In a related but more integrated strategy, Zhou et al. [32] employed plasma technology not merely as a surface activation step, but as a key enabler for constructing a highly durable flame-retardant architecture on poly(ethylene terephthalate) (PET) fabrics. Owing to their smooth surface morphology and chemical inertness, PET textiles present considerable challenges for conventional finishing processes. To overcome this limitation, low-temperature oxygen plasma was used to induce surface etching, generate free radicals, and introduce polar functional groups on the PET surface, thereby enhancing its chemical reactivity. The plasma-activated PET was subsequently treated with maleic acid (MA), which covalently grafted onto the modified surface and acted as a reactive intermediary layer. This MA-functionalized interface then enabled the effective incorporation of pentaerythritol phosphate urea salt (PEPAS), a phosphorus–nitrogen-rich flame retardant. The resulting system formed a compact and chemically integrated polymer network that exhibited strong resistance to ignition and promoted the formation of a dense, intumescent char layer during combustion, effectively suppressing flame spread and melt dripping. The treated fabrics displayed excellent flame-retardant performance, achieving a limiting oxygen index (LOI) of 29.3 and retaining a V-0 rating in UL-94 vertical burning tests even after 20 laundering cycles, while maintaining tensile properties close to those of untreated PET. These findings clearly illustrate the potential of plasma technology to transcend conventional surface modification roles, functioning instead as a powerful platform for covalent flame-retardant engineering in advanced textile applications.

Further evidence of the effectiveness of plasma-assisted flame-retardant finishing of polyester has been reported by Nandy et al. employing 3-hydroxyphenyl phosphinyl propanoic acid (3HPP) as a phosphorus-based flame-retardant agent [5]. Atmospheric-pressure plasma was used to activate the polyester surface prior to flame-retardant application, resulting in a substantial increase in flame-retardant uptake and retention. Consequently, the limiting oxygen index (LOI) of untreated polyester, initially measured at 20.8 %, increased to approximately 30 % following treatment with a 4 % 3HPP formulation. Notably, the enhanced flame-retardant performance was highly durable, with the LOI remaining at ~28 % even after 20 laundering cycles, indicating strong fibre–flame-retardant interactions facilitated by plasma activation. In addition to improved fire resistance, the treated polyester fabrics retained their mechanical strength and visual appearance, underscoring the suitability of this approach for practical applications in protective clothing and industrial textiles.These findings, when viewed alongside related plasma-assisted studies, underscore a broader trend rather than an isolated material-specific outcome.

Overall, these studies demonstrate that plasma-assisted surface modification provides an effective route to overcome the inherent chemical inertness of polyester fibres. By enhancing surface reactivity and promoting durable bonding of phosphorus-based flame retardants, plasma treatment enables significant improvements in flame resistance and wash durability without compromising mechanical or aesthetic properties. As such, plasma technology represents a promising and scalable strategy for advanced flame-retardant finishing of polyester textiles.

4.2. Cotton

Chemical finishing has long been the dominant approach for imparting flame retardancy to cotton textiles, typically involving the application of phosphorus-, nitrogen-, or halogen-containing compounds through padding, coating, and curing processes. Although these treatments can provide effective fire resistance, they generally require high chemical add-on levels and elevated curing temperatures, resulting in increased energy consumption, wastewater generation, and residual chemical discharge. In addition, aggressive chemical processing may adversely affect fabric handle, tensile strength, and long-term durability. These drawbacks have stimulated growing interest in alternative surface-engineering strategies that can enhance flame retardancy while reducing chemical usage and preserving the intrinsic properties of cotton fabrics.

In this context, plasma-assisted surface modification has emerged as a promising, environmentally benign, and resource-efficient technique. Plasma treatment selectively alters only the outermost nanometric layers of cotton fibres, enabling targeted surface functionalization without disturbing the bulk cellulose structure. Such surface-specific modification is particularly advantageous for flame-retardant applications, as it enhances fibre surface energy and reactivity, thereby promoting stronger interfacial bonding between cotton and flame-retardant agents while avoiding the extensive fibre degradation often associated with conventional wet chemical treatments.

Edwards et al. [33] demonstrated the feasibility of plasma-assisted chemical grafting by successfully immobilizing phosphoramidate-based flame-retardant monomers onto cotton fabrics using post-treatment dielectric barrier discharge (DBD) plasma. Plasma exposure generated reactive oxygen-containing functional groups on the cotton surface, facilitating covalent bonding of the flame-retardant monomers and resulting in enhanced flame-retardant performance and durability. These findings highlighted the effectiveness of DBD plasma as a platform for chemical fixation of flame-retardant species.

Related studies by Tsafack et al. [34,35] investigated vacuum plasma-enabled grafting of phosphorus-based flame-retardant monomers onto cotton. While low-pressure plasma effectively activated cellulose surfaces and promoted stable fibre–flame-retardant interfaces, limitations related to scalability and processing cost constrained its industrial applicability. To overcome these challenges, atmospheric-pressure plasma was subsequently explored as a pre-treatment prior to conventional flame-retardant finishing. This surface activation allowed a reduction in curing temperature from 180 °C to 160 °C and shorter processing times while still achieving satisfactory flame retardancy, with limiting oxygen index (LOI) values $\geq$ 25.

However, these studies also revealed important trade-offs associated with plasma exposure. Excessive plasma treatment resulted in surface etching and partial fibre degradation, leading to measurable reductions in tensile strength, as confirmed by scanning electron microscopy (SEM) and X-ray photoelectron spectroscopy (XPS). Despite this, plasma-treated cotton surfaces exhibited increased concentrations of oxygen-rich functional groups, including hydroxyl, carbonyl, and carboxyl moieties, which significantly enhanced the adhesion and retention of flame-retardant chemicals. These findings emphasize the critical need for careful optimization of plasma parameterssuch as power, treatment duration, and gas compositionto balance surface activation with mechanical integrity [2].

Beyond plasma-only strategies, hybrid approaches combining plasma treatment with photochemical activation have also been reported. The integration of atmospheric-pressure plasma with ultraviolet (UV) laser irradiation has been shown to improve the durability of non-durable flame-retardant finishes on cotton fabrics. Using nitrogen-, phosphorus-, and silicon-based systems, including diammonium phosphate (DAP), urea, and 3-aminopropyltriethoxysilane (APTS), plasma/UV-treated cotton fabrics exhibited enhanced flame resistance even after

30 min water soaking at 40 °C, simulating practical laundering conditions. Surface chemical analyses indicated an increased density of reactive groups, particularly carboxylic acid functionalities, which promoted stronger covalent bonding between flame-retardant species and cellulose chains. Among the tested formulations, the DAP–urea–APTS system demonstrated superior durability, retaining approximately 12 % of the flame-retardant load after soaking and exhibiting reduced burning rates and increased char formation [36].

A notable advancement in durable cotton flame retardancy was reported by Nandy et al.[37] who developed a chemically crosslinked phosphorus-based system reinforced by atmospheric plasma treatment. Cotton fabrics were treated with 3-hydroxyphenyl phosphinyl propanoic acid (3HPP) as the flame-retardant agent, 1,2,3,4-butanetetracarboxylic acid (BTCA) as a crosslinking agent, and dicyandiamide (DCDA) as a catalyst. FTIR and XPS analyses confirmed successful covalent incorporation of the phosphorus moiety into the cellulose matrix. Optimization of processing conditions increased the LOI from 17.4 % for untreated cotton to 29.5 %, which was further enhanced to 31.5 % following atmospheric plasma treatment, clearly demonstrating the role of plasma activation in strengthening fibre–flame-retardant interactions. Thermal analysis revealed improved thermal stability and enhanced char formation, while cone calorimetry showed reduced heat release and delayed ignition. Importantly, excellent wash durability was achieved, with an LOI of 28.1 % retained after 20 laundering cycles, effectively addressing a key limitation of conventional cotton flame-retardant finishes.

Overall, the literature clearly indicates that plasma-assisted approacheswhether applied as pre-treatment, post-treatment, or in combination with chemically crosslinked systemsoffer a viable pathway for enhancing flame retardancy, durability, and sustainability of cotton textiles. When appropriately optimized, plasma technology enables reduced chemical consumption, improved fixation efficiency, and preservation of fabric properties, making it a highly attractive strategy for next-generation flame-retardant cotton applications.

4.3. Polypropylene

Polypropylene (PP) is one of the most widely utilized thermoplastic polymers in technical textiles, automotive interiors, geotextiles, filtration media, packaging materials, and protective applications due to its advantageous combination of low density, high chemical resistance, good mechanical strength, and cost efficiency. However, from a fire safety perspective, PP presents significant challenges. It is intrinsically flammable, exhibits a low melting temperature during combustion, and tends to undergo severe melt dripping, which not only accelerates flame spread but also contributes to secondary ignition hazards. These characteristics render conventional flame-retardant treatments less effective and often necessitate high additive loadings, which can negatively affect mechanical performance, processability, and recyclability.

In recent years, plasma-based surface modification has emerged as a promising alternative strategy to address the inherent flammability of PP without compromising its bulk properties. As comprehensively reviewed by Horrocks [38], plasma treatments offer a non-thermal, solvent-free, and environmentally compatible route for enhancing flame retardancy in polymeric textiles. Rather than relying solely on bulk incorporation of flame-retardant additives, plasma technology enables targeted modification of the polymer surface, creating active sites that promote stronger interaction with flame-retardant agents. This approach is particularly attractive for PP, which is chemically inert and lacks polar functional groups, making it poorly receptive to conventional aqueous or solvent-based finishing formulations.

The effectiveness of plasma-assisted treatments for PP and related polymers has been further elucidated by Morent et al. [39], who systematically investigated the influence of medium-pressure dielectric barrier discharge (DBD) plasma treatments using different working gases, including air, helium, and argon, on nonwoven polyester (PET) and polypropylene (PP) textiles. Their findings revealed that plasma exposure significantly enhanced surface wettability for both polymer systems, although the extent of modification was more pronounced for PET than for PP. This difference was attributed to the aromatic ester structure of PET, which is more susceptible to plasma-induced oxidation and functionalization. Nevertheless, even for PP, plasma treatment introduced oxygen-containing functional groups and increased surface roughness, thereby improving liquid spreading and absorption behavior. Such enhancements in wettability are critical for flame-retardant finishing, as they facilitate uniform deposition, improved penetration, and stronger adhesion of flame-retardant formulations onto the textile surface.

The importance of plasma-induced wettability and surface activation has also been highlighted in the broader literature on plasma-enhanced fire performance. Jama et al. [40] reviewed several alternative plasma-based strategies aimed at improving the fire response of polymeric materials, emphasizing that plasma treatments can act either as a standalone surface modification technique or as a pre-treatment step that significantly boosts the efficiency of subsequent flame-retardant coatings. These studies collectively demonstrate that plasma activation can reduce the reliance on high chemical loadings by maximizing the effectiveness of flame-retardant agents at the material interface.

4.4. Polyamide

Beyond polyolefins, plasma-assisted flame retardancy has also been successfully demonstrated for polyamide-based textiles, which are widely used in protective clothing, upholstery, and industrial fabrics. A particularly illustrative case study involves the surface modification of polyamide 66 (PA66) fabrics using microwave vacuum plasma technology. In this approach, the fabrics were treated with a helium/oxygen (He/O_2) gas mixture under carefully optimized plasma conditions to generate a highly reactive surface enriched with polar functional groups. The plasma treatment significantly increased surface hydrophilicity and created reactive sites capable of forming stronger interactions with thiourea, a nitrogen-based flame-retardant compound.

Quantitative analysis revealed that plasma-treated PA66 fabrics exhibited approximately 38% higher thiourea uptake compared to untreated samples, confirming the role of plasma activation in enhancing chemical affinity and adsorption efficiency. This increased uptake directly translated into improved flame-retardant performance, as evidenced by a measurable increase in the limiting oxygen index (LOI) from 40.9% to 42.7% when the same thiourea concentration was applied. More importantly, the study demonstrated that plasma-treated fabrics achieved comparable flame-retardant performance using only half the thiourea concentration required for untreated fabrics. This reduction in chemical loading not only minimizes potential adverse effects on fabric handle and mechanical properties but also aligns with current sustainability goals by reducing chemical consumption and environmental impact [4].

From an industrial and environmental standpoint, these findings collectively underscore the transformative role of plasma technology in flame-retardant textile engineering. Plasma-assisted treatments enable precise control over surface chemistry, enhance the durability and effectiveness of flame-retardant finishes, and offer a viable pathway toward reducing additive usage. For polymers such as PP and PA66, which are traditionally challenging to functionalize, plasma activation provides a powerful tool to overcome interfacial limitations and achieve high-performance fire protection while preserving the intrinsic advantages of the base material. As a result, plasma-based surface engineering is increasingly regarded as a key enabling technology for the next generation of sustainable, high-performance flame-retardant textiles.

5.0 Comparison of Flame-Retardant Strategies for Textile Materials

Several approaches have been reported in the literature to impart flame retardancy to textile materials, including conventional chemical treatments, sol–gel coatings, layer-by-layer (LbL) assemblies, and inherently flame-retardant fibres. These techniques differ significantly in their working mechanisms, chemical requirements, environmental impact, processing conditions, and influence on fabric properties [41,42,43,44,45,46]. In recent years, plasma-assisted surface modification has emerged as an effective and environmentally favourable alternative due to its dry processing nature and minimal alteration of textile characteristics. A comparative summary of these flame-retardant strategies is presented in Table 2.When evaluated specifically for textile applications, plasma-assisted strategies provide a balanced combination of durability, environmental compatibility, and processing flexibility that is difficult to achieve using conventional flame-retardant approaches alone.

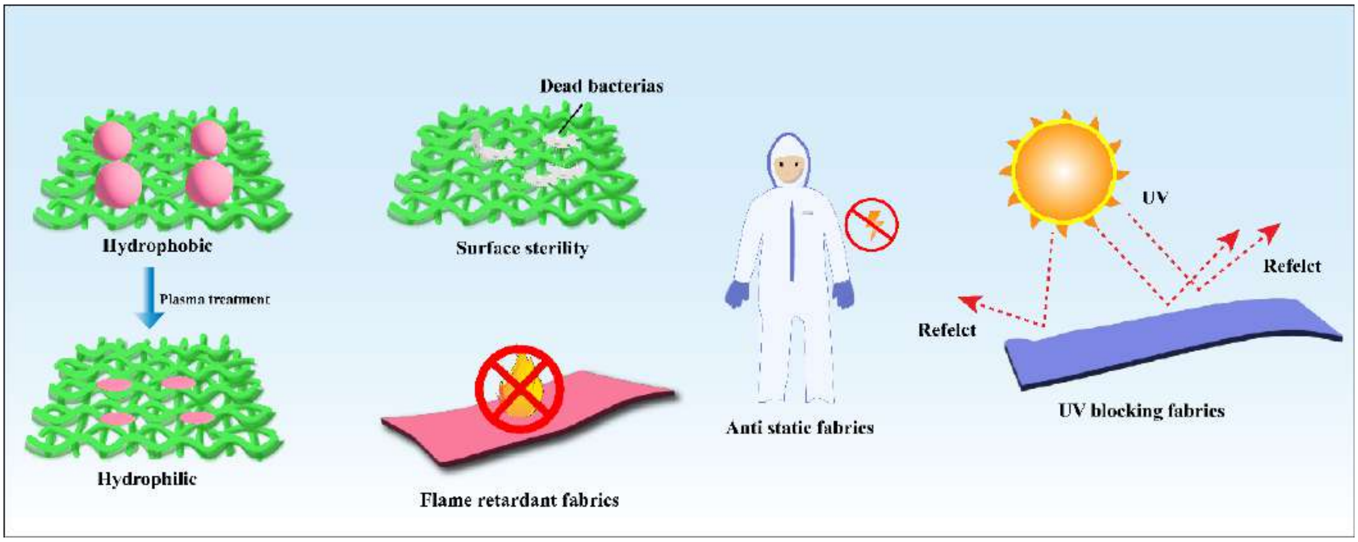

Fig 6. Other application areas of plasma technology

Table 2. Comparative Overview of Plasma-Assisted and Conventional Flame-Retardant Strategies for Textile Materials

Aspect	Plasma-Assisted Treatment	Conventional Chemical Treatment	Sol–Gel Technique	Layer-by-Layer (LbL) Assembly	Inherently Flame-Retardant Fibres
Flame-Retardant Mechanism	Activation of the textile surface by energetic species (ions, radicals, electrons, and UV photons), promoting surface functionalization and enhanced adhesion of flame-retardant moieties.	Flame retardancy achieved through bulk or surface application of halogenated, phosphorus-, or nitrogen-containing additives or finishes.	Formation of an inorganic or organic–inorganic hybrid protective barrier via hydrolysis and condensation of metal alkoxide precursors.	Fabrication of nanostructured multilayer assemblies through alternate adsorption of oppositely charged polyelectrolytes or nanoparticles.	Flame-retardant groups are chemically incorporated into the polymer backbone during fibre synthesis, resulting in intrinsic flame resistance.
Chemical Requirement	Enables a substantial reduction in flame-retardant chemical usage due to surface-specific modification.	Requires comparatively high add-on levels to achieve effective flame retardancy.	Involves tailored sol–gel precursors and controlled processing chemistry.	Requires multiple deposition cycles using aqueous solutions.	No post-processing flame-retardant chemicals are required.
Environmental Impact	Considered environmentally favourable as a dry, solvent-free process with minimal waste generation.	Often associated with solvent usage and chemical effluents, leading to environmental and regulatory concerns.	Environmental impact depends on precursor selection; some alkoxides and solvents may pose environmental risks.	Generally regarded as eco-friendly due to water-based processing and low solvent consumption.	Fibre production is energy-intensive, and recycling or disposal at end-of-life can be challenging.
Effect on Fabric Properties	Preserves fabric mechanical integrity, handle, and breathability, as modification is limited to the fibre surface.	May adversely affect tensile strength, flexibility, and fabric hand.	Can increase fabric stiffness; hybrid coatings may partially alleviate this drawback.	Minimal impact on fabric feel and flexibility; breathability is typically maintained.	Retains intrinsic fibre properties but is largely limited to synthetic fibres.
Processing Conditions	Operates at near-ambient temperature and pressure, offering reduced energy consumption and short treatment times.	Commonly requires high-temperature curing and longer processing durations.	Typically conducted at ambient conditions, followed by thermal or UV curing.	Performed at room temperature; however, the multi-step nature can be time-consuming.	Requires specialised polymerisation and spinning equipment and is not applicable to finished textiles.

6.0 Advantages and Limitations of Plasma-Assisted Flame-Retardant Surface Modification

Plasma-assisted surface modification offers several advantages for the development of flame-retardant textile coatings, particularly due to its ability to tailor surface chemistry without altering the bulk characteristics of the substrate. Since the plasma interaction is confined to the outermost fibre layers, the intrinsic mechanical properties of the textile are largely preserved [24,26]. Exposure to plasma generates polar functional groups on the fibre surface, such as hydroxyl (–OH), carbonyl (–C=O), and carboxyl (–COOH) functionalities, which increase surface energy and significantly improve the adhesion and durability of subsequently applied flame-retardant coatings or finishes [29,30,31]. From an environmental standpoint, plasma processing is widely regarded as a sustainable surface-engineering technique, as it is typically solvent-free, operates as a dry process, and substantially reduces chemical consumption compared with conventional wet-chemical methods [25,26]. Furthermore, plasma treatment induces minimal fibre damage and is applicable to a wide range of textile substrates, including cotton, polyester, and blended fabrics [24,30].

Despite these advantages, several limitations associated with plasma-assisted flame-retardant treatment must also be considered. The capital investment and operating costs of plasma equipment can be relatively high, particularly for vacuum-based systems or configurations integrating ultraviolet radiation, which may limit large-scale industrial adoption [24,28]. In addition, textiles composed of thermally sensitive or low-melting-point polymers may be prone to surface degradation if plasma parameters are not carefully controlled [29,47]. The range of flame-retardant chemistries that can be effectively immobilised on plasma-activated surfaces is also restricted, as plasma treatment primarily induces surface functionalisation rather than bulk modification [25,30]. Consequently, plasma treatment alone may be insufficient for applications requiring deep or long-term flame-retardant performance. Moreover, achieving reproducible and application-specific outcomes requires precise optimisation of multiple processing parameters, including gas composition, treatment time, applied power, and the distance between the electrode and the substrate, which increases process complexity [24,28].

7.0 Conclusion

Plasma-assisted surface treatment has emerged as a highly promising and sustainable strategy for imparting flame retardancy to textile and polymeric materials. Unlike conventional chemical finishing approaches, which often rely on large quantities of flame-retardant chemicals and energy-intensive curing processes, plasma treatment operates as a dry, solvent-free technique that selectively modifies only the outermost nanometric layers of the substrate. This highly controlled surface functionalization preserves the intrinsic mechanical strength, morphology, and aesthetic qualities of the material while avoiding bulk degradation or loss of performance.

One of the key advantages of plasma treatment lies in its ability to introduce reactive surface functionalities, such as hydroxyl, carbonyl, and carboxyl groups, which significantly enhance the interfacial bonding and fixation of flame-retardant agents. Improved chemical anchoring at the fibre surface leads to enhanced durability of flame retardancy, reduced migration or leaching of additives, and the possibility of lowering overall chemical loading. As a result, plasma-assisted approaches offer a viable pathway toward environmentally responsible fire-protection systems that align with current regulatory and sustainability demands.

This review critically examines the role of plasma treatments applied either as a pre-treatment or post-treatment step to improve flame-retardant efficacy across a variety of textile substrates, including both natural and synthetic fibres. The discussion encompasses recent advances reported in the literature, highlighting how plasma activation improves flame-retardant uptake, performance, and wash durability under practical conditions relevant to both laboratory studies and industrial applications. Despite these advantages, several challenges remain, such as the high initial investment required for plasma equipment, the limited depth of surface modification, and the sensitivity of treatment outcomes to process parameters including gas composition, power input, and exposure time.

Nevertheless, the collective evidence suggests that these limitations are primarily engineering and optimization challenges rather than fundamental barriers. With continued advances in plasma reactor design and process control, plasma-assisted surface treatments have strong potential to be scaled for industrial textile finishing. In the context of increasing emphasis on sustainability, regulatory compliance, and fire safetyparticularly within the apparel, protective clothing, and technical textile sectorsplasma technology represents a compelling route toward the development of efficient, durable, and environmentally responsible flame-retardant systems.

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Installation Damage of Geosynthetics

The geosynthetics are prone to some amount of damage during their installation. To assess the quantity of the installation damage, a standard method was initially developed by Watts and Brady of the Transport Research Laboratory in the United Kingdom. The procedure has also discussed in the ASTM D 5818 with similar requirements. We are at BTRA doing the test following same ASTM D 5818 method followed by respective tensile strength. For the time being we are using the construction site for the sample preparation. If customer will agree, BTRA will collect the sample from site after standard procedure and provide the report.

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