



Facilitating the removal of Reactive Blue 194 dye from wastewater under optimized conditions

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ABSTRACT

The paper emphasizes on the removal of Reactive Blue 194 dye (RB-194) using different process variables alone and in combination as well. A Cs-137 γ -radiation source was used to treat RB-194 at a dose rate of 669 Gy/h (Gray/h) with 1–10 kGy irradiation dose in the presence of (0.2–0.6 mL) hydrogen peroxide. The optimization of various process variables such as pH, γ -radiations absorbed dose, initial concentration of RB-194 and concentration of H₂O₂ were done to achieve thoroughgoing degradation. The dye was considerably degraded (79%) by γ -radiation but the addition of 0.6 mL H₂O₂ increased the degradation to 97% at a 10 kGy irradiation dose in acidic pH. The Fourier-transform infrared spectroscopy technique confirmed the presence of various useful groups. The fragmented by-products of RB-194 produced by irradiation at a 10 kGy dose were identified by liquid chromatography-mass spectrometry. The results revealed that dye molecules are degraded after decolorization to lower molecular weight substances, primarily organic acids, and carbon dioxide. The findings demonstrated that RB-194 could be efficiently removed from wastewater using γ -radiations along H₂O₂.

Keywords: Toxic pollutant; Dye removal; Water remediation; Environmental pollution

1. Introduction

Dyes are colored substances that are often used in the rubber, plastics, textiles, and cosmetics industries to color their products. Reactive dyes are suitable for dyeing cellulose and cotton fibers due to their high binding ability. The effluents released by these sectors are said to contain massive

amounts of dyes [1–3]. The dyes are complex organic substances that pollute water owing to their high stability in industrial waste even at extremely low concentrations [4–8]. The release of colored effluents into water is a significant cause of pollution and has an undesirable influence on the marine setting [9–11]. Different procedures like adsorption, membrane filtration, oxidation–ozonation, and coagulation,

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are often used to treat dye effluents. However, these conventional methods only function by transferring the phase of chemicals from their aqueous phase to a non-aqueous phase, which results in complicated pollution issues, making them ineffective in the treatment of wastewater [12–15].

However, other technologies, such as progressive oxidation developments have arose as effective options for treating industrial effluents. These procedures involve the generation of reactive species, for example, OH radicals that have specific benefits, such as a high ability to oxidize organic molecules, resulting in their destruction. These OH radicals are the most efficient oxidizing agent because they initiate a chain reaction that breaks down the large molecules into small and non-toxic compounds. As a result, the organic compounds are effectively mineralized into CO₂ and H₂O. The photocatalytic process [16,17], electro-Fenton, electrochemical process [18–21], sonolysis [22], photo-Fenton [23], ozonation [24], and ionizing radiation [25–27] are just a few of the approaches employed in the advanced oxidation processes (AOP) [28]. Recent studies suggest that gamma radiations may serve as a highly appealing option to treat industrial effluents by degrading dyes when exposed to highly energetic ionizing radiations. The interaction of gamma radiations with water results in the radiolysis of water, forming products such as hydrogen ion (H⁺), hydroxyl radical (*OH), hydrogen peroxide (H₂O₂), hydrated electron (e_{aq}⁻), H₂, and a hydrogen atom (H^{*}). The most reactive species among these are the hydrated electron, hydrogen atom, and hydroxyl radicals; they all react with contaminants in a solution and degrade them into simple compounds [9].

The main objective is to investigate the degradation of Reactive Blue 194 (RB-194) by a γ /H₂O₂-based AOPs. The influence of various factors such as absorbed dose of γ -rays, dye concentration, pH, and concentration of H₂O₂ on the removal of RB-194 was also scrutinized. The efficiency of removal was investigated by analytical techniques and liquid chromatography mass spectrometry (LCMS) was used to analyze the removal by-products.

2. Material and methods

Reactive Blue 194 was supplied by Crystal Chemistry Corporation (Faisalabad) and its molecular structure is shown in Fig. 1. NaOH, H₂O₂, and HCl were obtained from Merck (Darmstadt, Germany). 0.1 M HCl and NaOH were used to adjust the pH of the solutions. All the solutions were prepared using distilled water. The solution was filtered after being subjected to radiation. The features of RB-194 are given in Table 1.

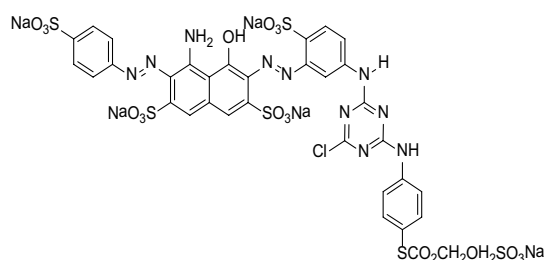


Fig. 1. Molecular structure of dye.

The solution was filtered through filter paper after being subjected to radiation. RB-194 belongs to azo group of dyes and anionic in nature. The RB-194 solutions of different concentrations (50–150 ppm) were prepared by dilution from the stock solution (1,000 ppm). De-ionized water was used to prepare the dye solutions. A Jenway 3010 pH meter (London, United Kingdom) was used to check the pH of solutions before and after treatment. The UV-Visible spectrophotometer (CE Cecil 7200, UK) was used to determine the RB-194 wavelength before and after gamma treatment. RB-194 was analyzed at a wavelength ranging from 200–800 nm.

2.1. Irradiation of samples

The γ -irradiation was conducted at the Nuclear Institute for Agriculture and Biology (NIAB), Faisalabad. The Cs-137 radiation source was standardized by Fricke dosimeter and Eq. (1) was used to determine dose rate [30]. Dose-rate was 669 Gy/h (Gray/h, unit of unit of ionizing radiation dose) at the time of sample irradiation. A series of investigations were carried out to study the effect of H₂O₂ on the degradation of RB-194 induced by gamma radiations. The rate of dose was 669 Gy/h (Gray/h, unit of ionizing radiation dose). RB-194 was irradiated at doses ranging from 1 to 10 kGy using a Cs-137 gamma irradiation source. A 100 mL RB-194 solution was added in 120 mL Pyrex with an initial concentration ranging from 50 to 150 ppm before treatment. Before irradiation, H₂O₂ was introduced to the sample, ranging from 0.2 to 0.6 mL. The pH of the solution was adjusted before treatment. All the procedure was carried out at room temperature [31]. The absorbed dose is calculated using Eq. (1).

$$D = \left[\frac{N \times \Delta A \times 100}{\epsilon \times \rho \times G(\text{Fe(III)})} \right]$$

where ΔA is the absorbance values before and after irradiation of the samples, D is absorbed dose, N is Avogadro's number, ϵ is the molar extinction coefficient of ferric ion, ρ is the density of Fricke solution and $G(\text{Fe(III)})$ indicates the number of Fe ions produced/100 eV of absorbed radiation energy.

2.2. FTIR and LCMS analysis

A Shimadzu IR-408 (Japan) spectrophotometer was used for Fourier-transform infrared spectroscopy (FTIR)

Table 1
Structural information of RB-194 dye [29]

Name of dye	RB-194
Molecular weight (g/mol)	1,205.38
Molecular formula	C ₃₃ H ₂₂ ClN ₁₀ O ₁₉ S ₆ Na ₅
λ_{max} (nm)	600
Chemical nature	Anionic blue
Reactive group	Azo group
Color index name	Blue

analysis. Methanol was used to extract the samples before analysis. FTIR spectroscopy was used to investigate the various functional groups present in RB-194 [32]. In order to determine the deteriorated end products, the degraded samples were subjected to LCMS (Thermo Fisher Scientific, USA) analysis at the NIBGE, Faisalabad. The mass spectra of irradiated samples were studied and monitored at various time intervals. The product-ion scan identified the production of several fragments. The instrument used an electrospray ionization source and was polarized in the positive direction [33].

3. Results and discussion

The absorption spectrum of RB-194 was observed using a Thermo Electron Corporation UNICAM UV 300 UV/Vis spectrometer (Bayern, Germany). Fig. 2 shows the absorption spectrum for RB-194. The maximum wavelength of dye was found to be 600 nm. Samples of RB-194 were analyzed at the λ_{\max} 600 nm for the measurement of absorbance [34].

3.1. Effects of dye concentration

The effect of dye concentration on the degradation of RB-194 was studied by varying the initial concentration of dye from 50 to 150 ppm while keeping H_2O_2 constant at 0.6 mL. The solutions were exposed to various doses of gamma radiation, ranging from 1 to 10 kGy. For a given amount of H_2O_2 , the degradation was not correlated with the amount of dye present at a particular point. At 50 ppm, the degradation (%) was significantly greater than at 150 ppm. The degradation of RB-194 was 69% at 150 ppm, 75% at 100 ppm, and 79% at 50 ppm when exposed to a dose of 10 kGy as shown in Fig. 3. The reason for the lower degradation at the higher initial concentration of RB-194 could be attributed to a lower ratio of OH radicals to the target pollutants, or an increasing competition for OH radicals between the pollutant and its degradation products. These results are in accordance with earlier research [1,35,36].

3.2. Effects of H_2O_2

The effect of H_2O_2 concentration (0.2 to 0.6 mL) on the degradation of RB-194 was investigated. The results are

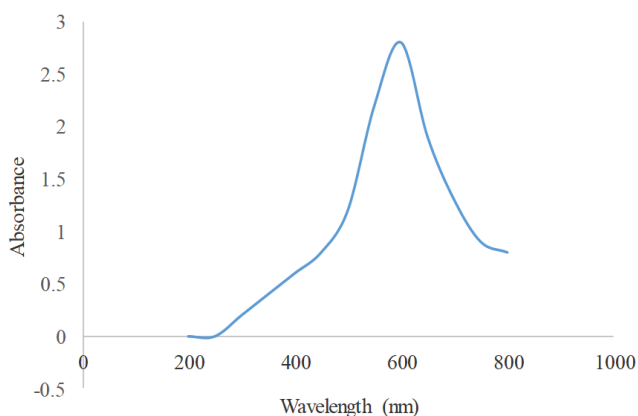


Fig. 2. Absorption spectrum of RB-194 showing λ_{\max} at 600 nm.

shown in Fig. 4. The findings show that the rate of degradation was directly proportional to the concentration of hydrogen peroxide. More the concentration is, more is the degradation. The 97% of the dye was degraded at 0.6 mL H_2O_2 at an absorbed dose of 10 kGy. H_2O_2 interacts rapidly with hydrated electrons produced by the radiolysis of H_2O_2 , resulting in the production of hydroxyl radicals. This rise in the rate of degradation might be due to a rise in hydroxyl radicals produced by the radiolysis process. This observation supports the fact that $\cdot\text{OH}$ radicals may degrade the chromophoric groups more effectively than hydrated electrons. The results revealed that the degree of removal of dye increases as the quantity of H_2O_2 increases until it reaches a certain dose that corresponds to the maximum degradation [9].

3.3. Effects of pH

The chemistry of the targeted pollutant, and the reactivity of OH, and e_{aq}^- are influenced by pH. Hence, the degradation of RB-194 dye by gamma irradiation was examined at various pH levels ranging from 3 to 9. At an absorbed dose of 10 kGy, the current study observed a maximum degradation of 95% at pH 3, followed by 90% at pH 5, and at pH 7 and 9, dye degradation was 84% and 80%, respectively. The results are shown in Fig. 5. Acidic pH has been found to scavenge e_{aq}^- which may influence its reactivity

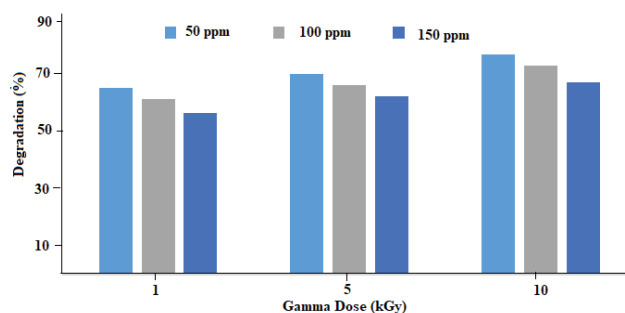


Fig. 3. Effect of initial concentration of dye on RB-194 degradation under gamma irradiation (The gamma radiation dose rate was 669 Gy/h for 1, 5 and 10 kGy, sample was irradiated for 89.69, 448.43 and 896.86 min, respectively).

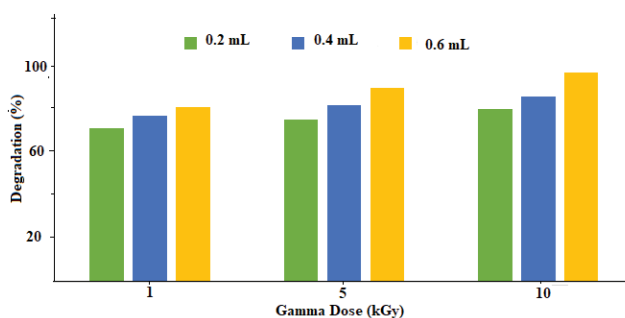


Fig. 4. Effect of different concentration of H_2O_2 (35%) on RB-194 degradation under gamma irradiation (The gamma radiation dose rate was 669 Gy/h for 1, 5 and 10 kGy, sample was irradiated for 89.69, 448.43 and 896.86 min, respectively).

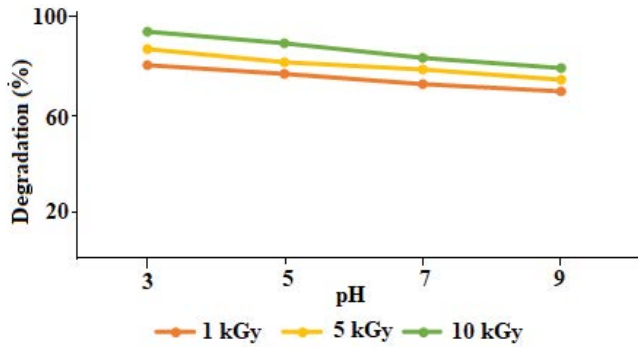


Fig. 5. Effect of pH on RB-194 degradation under gamma irradiation (The gamma radiation dose rate was 669 Gy/h for 1, 5 and 10 kGy, sample was irradiated for 89.69, 448.43 and 896.86 min, respectively; H₂O₂ concentration 35%; 0.6 mL).

with hydrogen peroxide. The rate of production of hydroxyl radicals is affected by the inhibition of the reaction between e_{aq}⁻ and H₂O₂. The reduced redox potential of hydroxyl radicals and the •OH scavenging by anions are the most probable reasons for the decreased degradation at high pH. The increased degradation rate of RB-194 at pH 3 may be due to the lowest scavenging of •OH and e_{aq}⁻ [1,36].

3.4. FTIR analysis

RB-194 was subjected to FTIR analysis to identify the presence and nature of various functional groups. The vibrational spectra of dye before and after γ -irradiation are shown in Fig. 6. The peaks at 3,386 and 1,545 cm⁻¹ were associated with the O–H and N=N stretching vibrations. The CH and C=O bending vibrations were responsible for the appearance of the wide bands at 1,466 and 1,135 cm⁻¹, respectively. The broad peaks at 1,038 and 997 cm⁻¹ are attributed to the S=O and C=C stretching vibrations [37]. The findings show that the functional groups that had been present before gamma irradiations had vanished, and no new functional groups had appeared. This suggests that the RB-194 dye was degraded under the influence of gamma irradiations.

3.5. LCMS analysis

LCMS was used to evaluate the by-products formed during the dye removal by γ -irradiations. The column was made of C18e, and the mixture of 130 mM ammonium acetate and acetonitrile compose the mobile phase. The temperature of the column was kept constant. Each sample was added to a triple quadruple tandem mass spectrometer for detection. The mass spectra of the RB-194 after irradiation and degradation pathway are shown in Figs. 7 and 8, respectively. The peaks of the degradation by-products, 4-hydroxynaphthalene-2-sulfonic acid, and 3-amino-5-hydroxynaphthalene-2,7-disulfonic acid were examined with *m/z* values of 226.33 and 320.25, respectively and degradation by-products with their *m/z* values are presented in Table 2. The results of the LCMS spectrum revealed that RB-194 was converted to low molecular weight substances after gamma irradiation, which are non-toxic end-products. in comparison to other wastewater treatment methods [38–43],

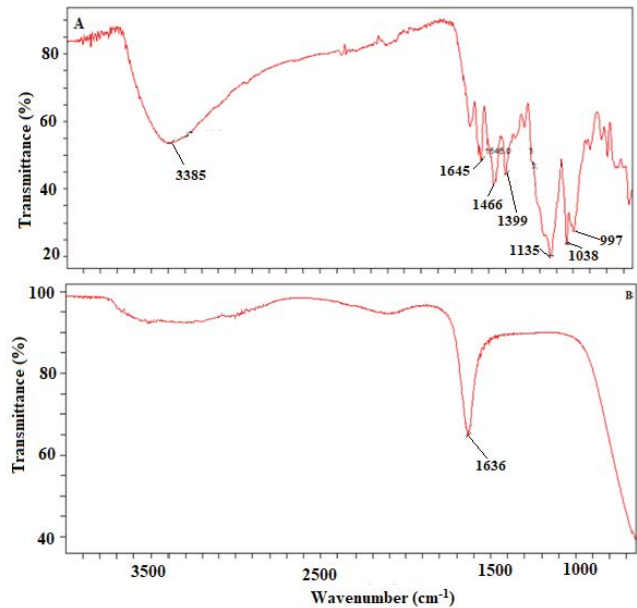


Fig. 6 Fourier-transform infrared spectra of RB-194 (A), before gamma irradiations, (B), after gamma irradiations + H₂O₂.

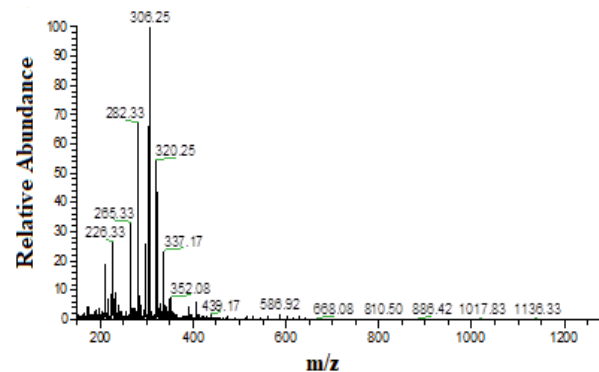


Fig. 7. LCMS spectrum of RB-194 after gamma irradiations.

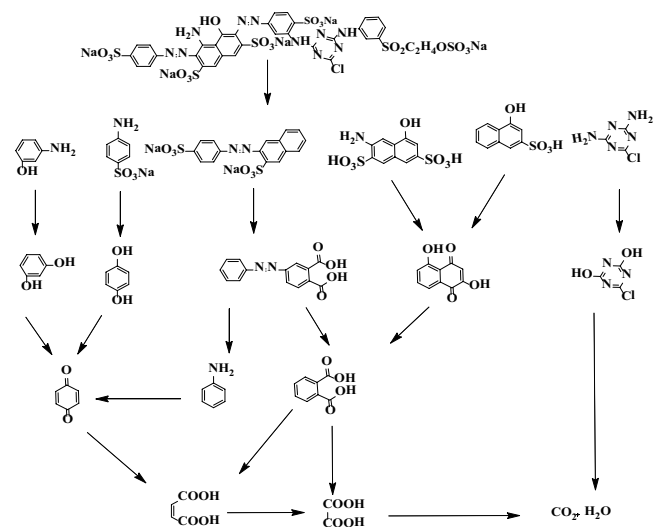
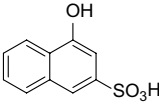
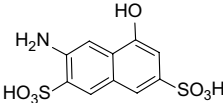


Fig. 8. Proposed degradation pathway of RB-194.

Table 2
Reactive Blue 194 by-products identified by LCMS

S. No.	Name of compound	Molecular weight	Structure	m/z value	Remarks
1	4-hydroxynaphthalene-2-sulfonic acid	224.23		226.33	Detected
2	3-amino-5-hydroxynaphthalene-2,7-disulfonic acid	319.31		320.25	Detected

the AOPs is one of viable techniques to treat the wastewater without producing secondary pollution.

4. Conclusions

The advanced oxidation technique based on $\gamma/\text{H}_2\text{O}_2$ was found to be very efficient for dye degradation. The process variables such as γ -radiations absorbed dose, pH, initial concentration of RB-194, and concentration of H_2O_2 were optimized to achieve thoroughgoing degradation. RB-194 was considerably degraded (79%) by γ -radiation but the addition of 0.6 mL H_2O_2 increased the degradation to 97% at a 10 kGy irradiation dose in acidic pH. The breakdown of azo bonds with their associated functional groups by γ -radiation was identified by FTIR analysis, and the destruction of dye into low molecular weight substances was also demonstrated by liquid chromatography-mass spectrometry analysis. Additionally, the findings demonstrated that under ideal circumstances, dye will be completely degraded. The results demonstrated that the $\gamma/\text{H}_2\text{O}_2$ -based AOPs is very efficient for the removal of contaminants from wastewater.

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