Original Scientific Paper https://doi.org/10.24867/GRID-2026-p65

Optimization of Fenton-like Oxidation for Cyan Printing Dye Removal in the Presence of Polyethylene Terephthalate Microplastics

ABSTRACT

The simultaneous presence of synthetic dyes and microplastics in industrial wastewaters represents an emerging environmental challenge that may significantly influence the performance of advanced oxidation processes. In this study, the efficiency of Fenton-like oxidation for the removal of Cyan printing dye in the presence of polyethylene terephthalate (PET) microplastics was investigated. Process optimization was performed using a three-level Definitive Screening Design (DSD) by evaluating the effects of five operating parameters, namely initial dye concentration, green-synthesized nano zero-valent iron (nZVI) dosage, PET concentration, solution pH and hydrogen peroxide concentration. The developed statistical model exhibited excellent predictive capability with a coefficient of determination (R^2) of 0.977 and an adjusted coefficient of determination (R^2adj) of 0.967. Analysis of variance confirmed the statistical significance of the model ($p < 0.0001$). Statistical analysis revealed that solution pH was the most influential factor affecting Cyan dye removal, followed by nZVI dosage, while H_2O_2 concentration, initial dye concentration and PET microplastic concentration also showed statisticaly significant effects. In addition, a significant interaction between nZVI dosage and H_2O_2 concentration confirmed the importance of their balanced ratio, wheras increasing PET concentration slightly reduced decolorization efficiency. The maximum experimental decolorization efficiency reached 85%, while model optimization predicted a maximum efficiency of 96% under the following operating conditions: 108.9 mgL^{-1} initial dye concentration, 48.7 $mgFeL^{-1}$ nZVI dose, 5.3 gL^{-1} PET concentration, pH of 5.82 and 4.78 mM hydrogen peroxide. The obtained results demonstrate that efficient treatment of Cyan printing wastewater in the presence of PET microplastics requires simultaneous optimization of all process variables and provide valuable insight into the combined effects of adsorption and oxidation occurring during the Fenton process.

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KEY WORDS
Fenton oxidation, PET microplastics, cyan printing dye, advanced oxidation processes, wastewater treatment, process optimization, definitive screening design

1. Introduction

Industrial wastewater containing synthetic dyes represents one of the most challenging environmental issues due to its high color intensity, complex chemical composition, and resistance to conventional biological treatment. Large volumes of dye-containing effluents are generated by numerous industrial sectors, including textile, paper, leather, food, cosmetic, and printing industries. Even at low concentrations, synthetic dyes impart

intense coloration to receiving water bodies, reduce light penetration, inhibit photosynthetic activity, and may produce toxic, mutagenic, or carcinogenic degradation products, thereby posing significant risks to aquatic ecosystems and human health. Recent estimates indicate that a substantial fraction of industrial dye-containing wastewaters is still discharged without adequate treatment, emphasizing the need for more efficient remediation technologies (Song et al., 2024; Rathi et al., 2021).

Among printing dyes, Cyan pigments based on copper phthalocyanine derivatives exhibit exceptional chemical, thermal, and photochemical stability owing to their highly conjugated aromatic structure. These characteristics provide excellent printing performance but simultaneously make their removal from wastewater particularly difficult. Conventional physicochemical and biological treatment technologies often fail to achieve satisfactory degradation because of the low biodegradability and structural stability of phthalocyanine compounds. Consequently, the development of efficient treatment strategies for Cyan printing wastewater remains an important environmental challenge (Xiao et al., 2024; Rathi et al., 2021).

Besides dissolved organic contaminants, industrial effluents increasingly contain microplastic particles originating from polymer processing, packaging materials, synthetic coatings, printing substrates, and equipment cleaning operations. Plastic production has increased continuously over recent decades, resulting in the extensive accumulation of plastic waste in terrestrial and aquatic environments (Tumwesigye et al., 2023; Fu et al., 2021; Chamas et al., 2020).

Among the different polymer types, polyethylene terephthalate (PET) is one of the most extensively manufactured engineering plastics owing to its excellent mechanical properties, chemical resistance, and widespread application in food packaging, beverage bottles, and industrial materials. Mechanical abrasion, weathering, and fragmentation generate PET microplastics that are now frequently detected in industrial and municipal wastewaters. Their occurrence has been reported in freshwater systems, marine environments, sediments, soils, atmospheric deposition, drinking water, and living organisms, highlighting their global environmental significance (Du et al., 2021; Chamas et al., 2020).

Among the available treatment technologies, Advanced Oxidation Processes (AOPs) have emerged as one of the most promising approaches for the degradation of refractory organic pollutants. Their effectiveness relies on the in-situ generation of highly reactive oxygen species, particularly hydroxyl radicals (•OH), which possess extremely high oxidation potential and react non-selectively with a wide range of organic compounds. Hydroxyl radicals promote cleavage of chromophoric groups, aromatic rings, and other recalcitrant molecular structures, leading to discoloration, oxidation, and in favourable cases complete mineralization into carbon dioxide and water (Ma et al., 2021; Nidheesh et al., 2018). Owing to these characteristics, AOPs have been successfully applied for the treatment of wastewaters containing dyes, pharmaceuticals, pesticides, antibiotics, and other persistent contaminants (Xiao et al., 2024; Lai et al., 2021; Miklos et al., 2018).

Among AOPs, the Fenton process remains one of the most extensively investigated oxidation technologies because of its operational simplicity, relatively low cost, and high oxidation efficiency. The process is based on catalytic decomposition of hydrogen peroxide in the presence of ferrous ions, producing hydroxyl radicals capable of rapidly degrading complex organic molecules. Numerous studies have demonstrated that Fenton and Fenton-like oxidation processes can achieve decolorization efficiencies frequently exceeding 90% for azo, anthraquinone, triphenylmethane, and phthalocyanine dyes under optimized reaction conditions, confirming their applicability for the treatment of highly colored industrial wastewaters (Heryanto & Tahir, 2025; Xiao et al., 2024; Deng & Brillas, 2023). However, process performance strongly depends on operational parameters such as pH, hydrogen peroxide dosage, iron concentration, reaction time, and wastewater composition (Song et al., 2024).

Despite extensive research on Fenton oxidation of synthetic dyes, considerably less attention has been devoted to complex wastewater systems containing suspended microplastics (Xiao et al., 2024). PET particles may adsorb dye molecules before oxidation, compete for reactive oxygen species, or undergo oxidative surface modifications that alter their adsorption behaviour during treatment (Kovačić et al., 2024). Consequently, the apparent dye removal efficiency may represent the combined contribution of adsorption onto PET surfaces and oxidative degradation by hydroxyl radicals.

Despite the increasing interest in the interaction between advanced oxidation processes and microplastics, systematic investigations on the Fenton oxidation of phthalocyanine-based printing dyes in the presence of PET microplastics remain scarce (Song et al., 2024). Therefore, this study aimed to evaluate the efficiency of the Fenton process for the removal of Cyan printing dye in the presence of PET microplastics.
Process optimization was performed using the Definitive Screening Design (DSD) methodology by systematically evaluating the effects of five key operational parameters: initial dye concentration, nZVI dosage - iron concentration, solution pH, PET concentration and hydrogen peroxide concentration. Optimization was initially conducted in a model aqueous system to establish the optimal operating conditions prior to their application to real printing wastewater.

2. Materials and Methods

2.1. Materials and chemicals

Cyan water-based printing dye (Flint Group, Germany) was selected as the target pollutant in this study. The dye belongs to the phthalocyanine class (Color Index PB15:3; CAS No. 147-14-8) and has a molecular formula of $C_{32}H_{16}CuN_8$ with a molar mass of 576.07 g mol^{-1}. The maximum absorption wavelength (λmax) of the dye was determined at 636 nm and was used for spectrophotometric monitoring throughout the experiments.

Commercial polyethylene terephthalate (PET) microplastic particles (Sigma-Aldrich, USA) were used as representative microplastics because of their widespread occurrence in industrial wastewaters and their environmental relevance.

Hydrogen peroxide (30%, NRK Engineering, Serbia), sodium hydroxide (>98.8%, POCH, Poland), sulfuric acid (>96%, J.T. Baker, USA), and iron(III) chloride hexahydrate (Centrohem, Serbia) were of analytical grade and used without further purification. All aqueous solutions were prepared using deionized water.

Nano zero-valent iron (nZVI) particles were synthesized using a green synthesis procedure based on green tea (Camellia sinensis) leaf extract, following the method originally proposed by Machado et al. (2013) and subsequently applied in our previous study (Prica et al., 2020).

2.2. Fenton oxidation experiments

Fenton oxidation experiments were conducted in batch mode using a six-position jar-test apparatus (FC6S, Velp Scientifica, Italy). The experiments were performed in 500 mL glass beakers containing 250 mL of aqueous Cyan printing dye solution. The initial dye concentration was varied between 20 and 180 mgL^{-1} according to the experimental design. PET microplastic particles were added at concentrations ranging from 1 to 10 gL^{-1}, while the concentration of green-synthesized nZVI catalyst varied between 0.75 and 60 $mgFeL^{-1}$. The initial pH of the reaction mixture was adjusted within the range of 2–10 using 0.1 M H_2SO_4 or 0.1 M NaOH solutions. Subsequently, hydrogen peroxide was added at concentrations between 1 and 11 mM to initiate the Fenton oxidation process. All experiments were carried out at room temperature (23 ± 1 °C) under continuous stirring at 120 rpm for 60 min.

Following the reaction, the residual concentration of Cyan dye was determined spectrophotometrically by measuring the absorbance at its maximum absorption wavelength (636 nm) using a UV–Vis spectrophotometer (Genesys 10S, Thermo Scientific, USA). The decolorization efficiency (E, %) was calculated according to Eq. (1):

$$E\ (\%) = \frac{A_0 - A_t}{A_0} * 100 \quad (1)$$

where: A_0 is the initial dye absorbance of Cyan dye aqueous solution; A_t is absorbance of Cyan dye aqueous solution after Fenton-like process.

2.3. Statistical analysis

The experimental matrix was established using a three-level Definitive Screening Design (DSD) to optimize the Fenton oxidation process. Five operating parameters were selected as independent variables: initial Cyan dye concentration (20–180 mgL^{-1}), nZVI dosage (0.75–60 $mgFeL^{-1}$), PET microplastic concentration (1–10 gL^{-1}), solution pH (2–10), and hydrogen peroxide concentration (1–11 mM). Each parameter was evaluated at three levels (–1, 0, and +1), representing the lower, central, and upper experimental values (Prica et al., 2020). Experimental design generation, regression analysis, and process optimization were carried out using JMP® 13 software (SAS Institute Inc., Cary, NC, USA). The DSD methodology was selected because it enables efficient screening of significant factors while minimizing the number of experimental runs and allowing independent estimation of the main effects without confounding with second-order interactions (Pereira et al., 2018).

3. Results and Discussions

3.1. Fenton oxidation process efficiency

The efficiency of the Fenton oxidation process for the removal of Cyan printing dye in the presence of PET microplastics was evaluated using a Definitive Screening Design (DSD). The experimental matrix comprised 28 experimental runs in which five operating variables were simultaneously investigated. The corresponding decolorization efficiencies obtained under different operating conditions are summarized in Table 1.
The obtained results revealed a pronounced dependence of the Fenton oxidation efficiency on the selected operating conditions. Dye removal efficiencies ranged from only 2.0% to 85%, confirming that the process performance was strongly governed by the interaction of the investigated variables.

The lowest removal efficiencies were generally observed under alkaline conditions combined with low catalyst dosage, whereas considerably higher decolorization efficiencies were achieved under acidic conditions and increased nZVI concentrations. The highest experimental removal efficiency (85%) was obtained under intermediate operating conditions, indicating that simultaneous optimization of all process variables is required to maximize the oxidation performance.

The broad variation in removal efficiency demonstrates the complexity of the investigated system, in which dye oxidation and adsorption onto PET microplastics may occur simultaneously. Unlike conventional aqueous systems, the presence of PET particles introduces additional interactions that may influence hydroxyl radical availability as well as the apparent decolorization efficiency. Consequently, optimization based on statistical experimental design represents an appropriate approach for identifying the combined effects of multiple operating variables.

3.2. Statistical evaluation of the developed model

The adequacy of the developed DSD model was evaluated using regression analysis and analysis of variance (ANOVA). As presented in Table 2, the proposed model exhibited an excellent agreement with the experimental data, with a coefficient of determination (R^2 = 0.98) and an adjusted coefficient of determination (R^2adj = 0.97), indicating that approximately 98% of the variability in Cyan dye removal could be explained by the selected operating variables.
Furthermore, the relatively low root mean square error (RMSE = 5.16) confirms the satisfactory predictive capability of the developed model.

The ANOVA results further confirmed the statistical significance of the regression model. As shown in Table 3, the calculated F-value of 101.136 with a probability value below 0.0001 indicates that the model adequately describes the relationship between the investigated variables and the response. Therefore, the developed statistical model can be considered reliable for both interpretation of individual process parameters and optimization of the Fenton oxidation process.

Table 1

Decolorization efficiency (%) of the Fenton process for a synthetic Cyan printing dye solution

Number	Dye concentration (mgL^{-1})	nZVI dosage (mgL^{-1})	PET concentration (mgL^{-1})	pH	H_2O_2 concentration (mM)	Fenton process efficiency (%)
1	100	60	10	10	11	45.15
2	100	0.75	1	2	1	58.63
3	180	30.4	1	10	11	21.66
4	20	30.4	10	2	1	47.72
5	180	0.75	5.5	2	11	72.16
6	20	60	5.5	10	1	74.28
7	180	60	1	6	1	82.95
8	20	0.75	10	6	11	12.6
9	180	60	10	2	6	69.44
10	20	0.75	1	10	6	2.01
11	180	0.75	10	10	1	2.75
12	20	60	1	2	11	62.94
13	100	30.4	5.5	6	6	79.71
14	100	60	10	10	11	40.41
15	100	0.75	1	2	1	60.31
16	180	30.4	1	10	11	15.36
17	20	30.4	10	2	1	65.99
18	180	0.75	5.5	2	11	71.21
19	20	60	5.5	10	1	72.55
20	180	60	1	6	1	70.75
21	20	0.75	10	6	11	10.14
22	180	60	10	2	6	65.63
23	20	0.75	1	10	6	6.53
24	180	0.75	10	10	1	6.86
25	20	60	1	2	11	58.88
26	100	30.4	5.5	6	6	84.98
27	100	30.4	5.5	6	6	81.69
28	100	30.4	5.5	6	6	76.15

Table 2
Summary of fit

Source	Value
R2	0.98
R2 Adj	0.97
Root Mean Square Error	5.16

Table 3
ANOVA results for the proposed model

Source	Degrees of freedom	Sum of Squares	Mean Square	F parameter
Model	8	21533.035	2691.630	101.136
Error	19	505.665	26.610	Prob>F
C. Total	27	22038.700	-	<0.0001

The estimated regression coefficients are presented in Table 4. Among the investigated factors, solution pH exhibited the greatest influence on the process efficiency, followed by nZVI dosage. This observation is consistent with the well-established mechanism of the Fenton reaction, which is known to achieve maximum efficiency under acidic conditions. At low pH values, iron remains predominantly in its soluble form, enabling continuous catalytic decomposition of hydrogen peroxide and efficient generation of hydroxyl radicals.
As the pH increases, ferric hydroxides are progressively formed, reducing the concentration of catalytically active iron species and consequently decreasing the oxidation efficiency. Similar behaviour has been widely reported for homogeneous and heterogeneous Fenton systems treating dye-containing wastewaters (Xiao et al., 2024; Nidheesh et al., 2018).

Furthermore, increasing the catalyst concentration enhanced hydroxyl radical production by providing a greater number of active iron sites available for hydrogen peroxide activation. Consequently, higher nZVI dosages promoted faster degradation of the chromophoric structure of the phthalocyanine dye. However, excessively high iron concentrations may also lead to non-productive radical consumption through scavenging reactions, indicating that an optimum catalyst dosage is required to maximize process performance.

Table 4
Parameter estimates sorted by statistical significance

Term	Estimate	Std Error	t ratio	Prob >|t|
pH	-17.268	1.154	-14.970	<0.0001*
nZVI dosage	16.989	1.154	14.730	<0.0001*
H_2O_2 concentration	-6.614	1.154	-5.730	<0.0001*
PET concentration	-3.667	1.154	-3.180	0.0049*
nZVI dosage * H_2O_2 conc.	-5.626	1.824	-3080	0.0061*
Dye concentration	3.257	1.154	2.820	0.0109*

Hydrogen peroxide concentration and PET concentration also significantly affected dye removal, although to a lesser extent. In addition, the interaction between nZVI dosage and H_2O_2 concentration was found to be statistically significant ($p = 0.0061$), suggesting that the oxidation efficiency depends not only on the individual reagent concentrations but also on their mutual balance during hydroxyl radical generation. Initial dye concentration showed a statistically significant positive effect, indicating that the selected concentration range was suitable for efficient oxidation under optimized operating conditions.

Although hydrogen peroxide is the primary source of hydroxyl radicals, its excessive addition may reduce oxidation efficiency because H_2O_2 competes with dye molecules for hydroxyl radicals, producing less reactive hydroperoxyl radicals ($HO_2•$). This phenomenon has been frequently reported in Fenton-based oxidation processes and explains why increasing the oxidant concentration beyond the optimum value does not necessarily improve pollutant removal (Deng & Brillas, 2023).

PET microplastic concentration was likewise identified as a statistically significant factor. Increasing the concentration of PET particles resulted in a slight decrease in the overall Fenton oxidation efficiency. This behaviour may be attributed to the dual role of PET microplastics within the reaction system. On one hand, PET particles can adsorb Cyan dye molecules onto their surface, thereby reducing the dissolved dye concentration. On the other hand, adsorption may limit the accessibility of dye molecules to hydroxyl radicals, while PET surfaces may additionally interfere with radical reactions occurring in the aqueous phase. Consequently, the experimentally observed decolorization efficiency represents the combined outcome of adsorption and oxidative degradation, rather than oxidation alone (Song et al., 2024).

Overall, these findings demonstrate that the efficiency of the Fenton oxidation process is governed by a combination of operational parameters rather than by a single factor, highlighting the importance of multivariable optimization for complex wastewater systems containing both dissolved dyes and suspended PET microplastics.

3.3. Optimization of the Fenton oxidation process

The optimal operating conditions for the Fenton oxidation process were determined using the prediction profiler generated by the Definitive Screening Design (DSD) model (Figure 1). The profiler simultaneously illustrates

the influence of each process variable on the predicted decolorization efficiency while maintaining the remaining variables at their optimum levels. This approach enables visualization of both the direction and magnitude of individual factor effects and provides a practical tool for selecting the most favorable operating conditions.

According to the developed model, the maximum predicted decolorization efficiency of 96% was achieved at an initial Cyan dye concentration of approximately 108.9 mgL^{-1}, with 48.7 mgL^{-1} of nZVI, 5.3 gL^{-1} of PET microplastics, a pH of 5.82 and 4.78 mM hydrogen peroxide. These results demonstrate that complete optimization of the process does not necessarily correspond to the highest or lowest values of all investigated variables, but rather to an optimal balance between catalyst dosage, oxidant concentration, solution pH, and PET content.

The prediction profiler clearly indicates that the concentration of nZVI exerts a positive effect on the process efficiency within the investigated range, whereas increasing PET concentration beyond its optimal value leads to a gradual decrease in dye removal. The latter observation is consistent with the well-known scavenging effect of excess hydrogen peroxide, which consumes hydroxyl radicals and decreases their availability for oxidation of the target pollutant. Similarly, increasing PET concentration may reduce the apparent oxidation efficiency due to enhanced adsorption of dye molecules onto the polymer surface, thereby altering the balance between adsorption and oxidative degradation.

The optimum pH predicted by the statistical model was approximately 5.8, which is higher than the optimum commonly reported for conventional homogeneous Fenton systems. This behavior may be attributed to the use of green-synthesized nZVI as a heterogeneous iron source, whose catalytic activity can be maintained over a wider pH range compared with dissolved ferrous ions. Furthermore, thesimultaneous presence of PET microplastics may modify the reaction environment through adsorption phenomena occurring at the solid–liquid interface, resulting in a shift of the optimum operating conditions.

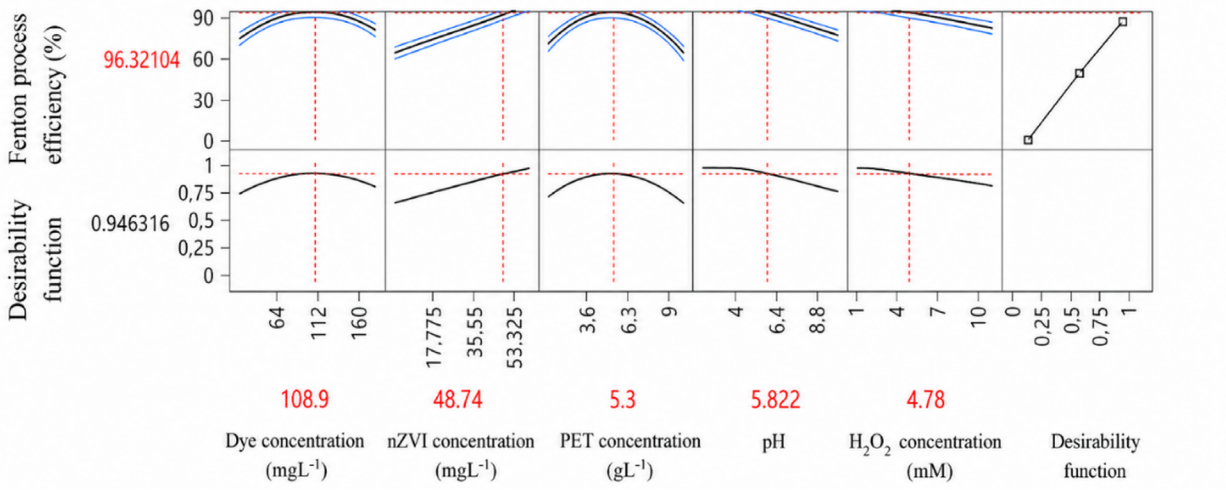
Overall, the prediction profiler confirms that efficient removal of Cyan printing dye can be achieved only through simultaneous optimization of all investigated process variables. The developed DSD model therefore represents a reliable tool for predicting process performance and defining operating conditions suitable for subsequent application to real printing wastewater.

4. Conclusion

The present study demonstrated that the efficiency of Fenton oxidation for the removal of Cyan printing dye is strongly influenced by the simultaneous presence of PET microplastics and by the operating conditions of the oxidation process. Application of the Definitive Screening Design enabled efficient evaluation of five process variables while substantially reducing the number of required experiments.

The developed statistical model showed excellent predictive performance (R^2 = 0.98), confirming the suitability of the DSD approach for optimization of complex oxidation systems. Among the investigated variables, solution pH and nZVI dosage were identified as the dominant factors governing dye removal, whereas excessive PET concentrations negatively affected the process efficiency. The interaction between nZVI dosage and hydrogen peroxide concentration was also found to be statistically significant, emphasizing the importance of maintaining an appropriate catalyst-to-oxidant ratio.

The optimized operating conditions predicted a decolorization efficiency of approximately 96%, demonstrating the high potential of the investigated process for



» **Figure 1:** *Optimization diagram of Fenton oxidation process*

treating Cyan printing wastewater. Furthermore, the results indicate that PET microplastics not only represent an additional contaminant but may also influence the overall treatment efficiency through simultaneous adsorption of dye molecules and modification of oxidation processes occurring in the reaction system.

Overall, the findings contribute to a better understanding of Fenton oxidation in complex wastewater matrices containing both synthetic dyes and microplastics and provide a basis for the future application of optimized Fenton-based technologies for the treatment of real printing industry effluents.

5. Acknowledgements

We gratefully acknowledge the financial support of the Ministry of Science, Technological Development, and Innovation of the Republic of Serbia (Grants No. 451-03-33/2026-03/200125, 451-03-34/2026-03/200125, and 451-03-34/2026-03/200156) and the University of Novi Sad, Faculty of Technical Sciences through the project "Scientific and Artistic Research Work of Researchers in Teaching and Associate Positions at the Faculty of Technical Sciences, University of Novi Sad 2026" (No. 01-3609/1).

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