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NOVEL TREATMENT FOR DYE DECOLORIZATION USING A MICROREACTOR SYSTEM AND FENTON'S REAGENT

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Wastewater treatments in the textile industry faces many challenges, particularly due to the residues of used dyes, which are pollutants characterized by high chemical stability. One promising technology for addressing the issue is an advanced oxidation process (AOP), often utilizing Fenton's reagent as the oxidizing component. This study focuses on the degradation of the anthraquinone dye Acid Violet 109 using Fenton's reagent in a microfluidic reactor. The microreactor system consists of plunger pump units, a mixer, and a polytetrafluoroethylene (PTFE) tube. The influences of various process parameters have been analyzed, including the concentration of Fe^{2+}, microreactor characteristics, the Fe^{2+}/H_2O_2 molar ratio, and the total flow rate of the reaction mixture. The results show that treatment with Fenton's reagent is successful, with efficiencies between 82 and 99 %.

Keywords: anthraquinone dye; Fenton process; microreactor; AOP; decolorization

НОВ ТРЕТМАН ЗА ОБЕЗБОЈУВАЊЕ НА БОИ СО ПОМОШ НА МИКРОРЕАКТОРСКИ СИСТЕМ И РЕАГЕНС НА ФЕНТОН

Прочистувањето на отпадните води во текстилната индустрија се соочува со многу предизвици, особено поради остатоците од употребените бои, кои претставуваат загадувачи што се карактеризираат со висока хемиска стабилност. Една ветувачка технологија за решавање на проблемот е напреден процес на оксидација (AOP), честопати користејќи го реагенсот на Фентон како оксидациска компонента. Оваа студија се фокусира на разградувањето на антрахинонската боја Acid Violet 109 со користење на реагенсот на Фентон во микрофлуиден реактор. Системот на микрореактор се состои од пумпни единици со клип, миксер и цевка од политетрафлуороетилен (PTFE). Анализирани се влијанијата на различните параметри на процесот, вклучувајќи ја концентрацијата на Fe^{2+}, карактеристиките на микрореакторот, моларниот однос Fe^{2+}/H_2O_2 и вкупната брзина на проток на реакционата смеса. Резултатите покажуваат дека третманот со реагенсот на Фентон е успешен, со ефикасност помеѓу 82 и 99 %.

Клучни зборови: антрахинонски бои; процесот на Фентон; микрореактор; AOP, обезбојување

1. INTRODUCTION

Wastewater from the textile industry is heavily polluted with dyes, metal ions, surfactants, auxiliaries, and other contaminants, and is produced in large quantities. The treatment of such polluted wastewater is not easy due to its complex structure and the high chemical stability of the organic dyes.[1,2] A wide range of methods such as filtration,[3] flocculation,[4,5] adsorption,[6–8] and ozonation[9–11] have been developed for wastewater decolorization. Additionally, biological treatments are also employed in wastewater treatment.[12]

While conventional physicochemical treatments are relatively simple, they often produce sludge as a byproduct. In contrast, biological

treatments are considered cheap and environmentally friendly, but less effective. Among various technologies used in the treatment of wastewater from the textile industry, chemical oxidation has become popular. Advanced oxidation processes (AOPs) are a destructive method increasingly used for industrial wastewater treatment.[13,14] The basic principle of AOP is to generate highly reactive radicals that lead to the mineralization of organic pollutants without producing additional waste.

The Fenton process has been found to be the most suitable process due to its simplicity, cost-effectiveness, and the availability of iron and hydrogen peroxide used in this process.[15] However, a limitation of Fenton-type AOPs is the potential for sludge formation, which requires disposal, thereby increasing operational and disposal costs.[16,17] The Fenton's reagent is a catalytic-oxidizing mixture of iron ions and hydrogen peroxide.

Numerous papers have examined the Fenton reaction with respect to degradation of synthetic dyes, focusing on improvements to the basic Fenton process. For instance, Papić et al.[18] concluded that the combination of homogeneous Fenton processes and UV irradiation can achieve high decolorization efficiency. Their experiments were conducted using three synthetic dyes: C.I. Reactive Yellow 3, C.I. Reactive Blue 2, and C.I. Reactive Violet 2. Some authors have reported that the Fenton process could be improved by developing a different catalyst. Verma et al. successfully used the cobalt(II)/ascorbic acid/hydrogen peroxide system for degradation of six structurally different synthetic dyes.[19] Merouani et al. described an Fe(II)/H_2O_2/H_3NOH^+ system for the removal of persistent textile dyes,[20] which was applied to solutions containing Basic Fuchsin, Rhodamine B, Chlorazol Black, Safranin O, and Light Green SF Yellowish.

Microreactor systems are new technologies that can provide, in some cases, much better results than conventional technologies. Microreactors operate continuously, with reactor channel diameters ranging from a few micrometers to hundreds of micrometers. Examples include chip, capillary (channel), microstructured, mesh, catalyst trap, micro-packed bed, falling film, and industrial microreactors.[21] These systems offer numerous benefits over traditional methods, such as precise temperature control, minimal reactant consumption, short reaction times, minimal environmental impact, and high safety levels.[22] In fact, microreactors have often proved to be more efficient than batch treatment systems.[23] They are particularly well-suited for highly endothermic or exothermic reactions, as well as reactions that require the use of hazardous chemicals.[24] Another significant advantage is the simplicity of the scaling-up process, as it is easy to connect thousands of devices while maintaining lab-level performance.[21] Ramos et al. demonstrated that wastewater polluted with textile dye can be treated in a microreactor using photodegradation.[24]

The aim of this study was to analyze the possibility of using a new, sustainable, and environmentally friendly technology for wastewater decolorization. Specifically, this study determined the efficiency of degrading the dye C.I. Acid Violet 109 by Fenton's reagent in a microreactor, focusing on optimizing the parameters that influence the decolorization efficiency.

2. MATERIALS AND METHODS

The anthraquinone dye Acid Violet 109 (AV 109, technical grade) used in this study was obtained from Bazema, Germany. The chemical structure of C.I. Acid Violet 109 is shown in Figure 1. Sulfuric acid (H_2SO_4, Zorka, Šabac) was used to adjust the pH value using a Wastewater Treatment Photometer HI83314, Hanna, USA. Hydrogen peroxide (H_2O_2, 3 %) was purchased from Galena Lab (Serbia), while ferrous sulphate ($FeSO_4 \cdot 7H_2O$), an analytical grade reagent, was obtained from Centrohem (Serbia).

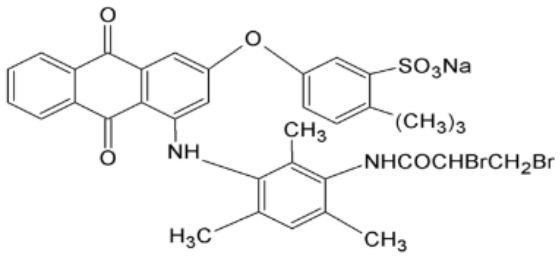

Fig. 1. Chemical structure of Acid Violet 109

The concentration of AV109 dye was maintained at 100 mg dm^{-3} in all experiments. To reduce clean water consumption, $FeSO_4 \cdot 7H_2O$ was added directly to dye solution. After the addition of ferrous salt, the pH was adjusted to the optimal value for the Fenton reaction, which is 3.0, as indicated by literature and previous studies.[25] Hydrogen peroxide was additionally added to achieve the selected Fe^{2+}/H_2O_2 molar ratios. The efficiency of decolorization was measured spectrophotometrically in the visible part of the spectrum at a wavelength of 590 nm, using a UV-Vis spectrophotome-

ter (Shimatzu 1800, Japan), with the monochromator slit aperture fixed at 1 nm.
Efficiency was calculated using equation 1:

$$Efficiency\ [\%] = 100 \times \left(\frac{Ao - A_1}{Ao}\right), \qquad (1)$$

where A_0 is absorbance before treatment and A_1 is absorbance after treatment.

2.1. *Microreactor system*

The experiment was carried out using a microreactor system comprised of two plunger pump units (LC-20AD XR, Shimadzu), one T-mixer, and a PTFE tube. Figure 2 shows the experimental set-up. The microreactor system delivered reactants via the pumps: the first pump (stream 1) supplied a dye solution containing dissolved ferrous salt, while the second pump (stream 2) provided a hydrogen peroxide solution. These two streams combined in the T-mixer before entering the microreactor tube. The microreactor was constructed from chemically resistant high-performance PTFE. Samples were collected in vials for spectrophotometric analysis.

Several experimental conditions were varied, including total flow rate, Fe^{2+}/H_2O_2 molar ratios, initial Fe^{2+} concentration, and microreactor length and diameter. All data are summarized in Table 1.

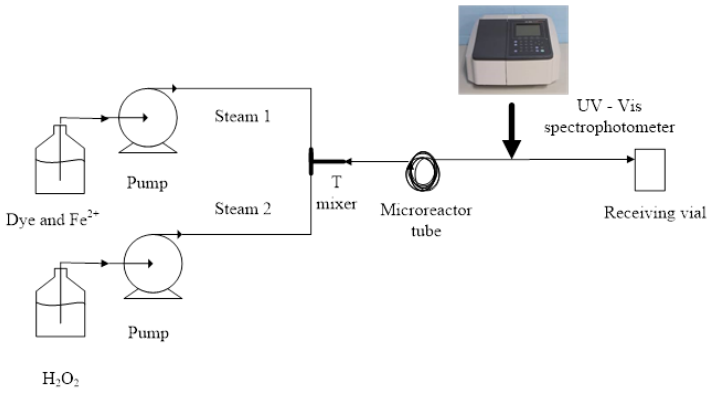

Fig. 2. Experimental setup

T a b l e 1

Reaction conditions

	Value	Unit
Pressure	101.3	kPa
Temperature	25	ºC
pH	3	
Initial dye concentration	100	mg dm^{-3}
Microreactor diameter	0.3 0.5 0.8	mm
Microreactor length	1 5 15	m
Fe^{2+}/H_2O_2 molar ratios	5 10 20 40 100	

The flow regime is highly dependent on the flow rate of the reaction mixture in the microreactor system. To determine the type of flow through which the reaction mixture moves in the system, the Reynolds number was calculated using the data on the viscosity of the mixture, which was measured using a viscometer (LE0089, EU Instruments). The calculated Reynolds values were between 8 and 20. The results indicate that the flow of the reaction mixture was laminar in all case with any of the applied flow rates.

The residence time of the reaction mixture in a microreactor is determined by reactor's length and diameter, as well as the flow rate of the mixture. Data are shown in Table 2.

T a b l e 2

Residence time, s

Flow rate, µl min^{-1}	Microreactor length, m	Microreactor diameter, mm		
		0.3	0.5	0.8
		Residence time, s		
1	1	4	12	30
	5	21	59	151
	15	64	177	452
5	1	0.8	2.3	6.0
	5	4	12	30
	15	13	35	90

3. RESULTS AND DISCUSSION

Few studies have investigated the treatment of anthraquinone dyes with Fenton's reagent. Stupar et al. investigated oxidative degradation of Acid Blue 111 by an electro-assisted Fenton process and compared it to the original Fenton process.[26] The Fe^{2+} concentration ranged from 143.8 to 431.6 μmol dm^{-3}. The Fenton process achieved a degradation efficiency of 87.9 % after 60 min, while the electro-assisted process showed better results, with a decolorization efficiency of 97.5 % after 30 min. Basturk et al. investigated decolorization of Reactive Blue 181 solution using both Fenton and sono-Fenton process.[27] Under optimal conditions, the Fenton and sono-Fenton processes achieved dye removal of 88 % and 93.5 % for 30 minutes, respectively. Shen et al. investigated the combined effects of ozonation and the Fenton process on the degradation of C.I. Acid Blue 80 dye.[28] Their results showed that this combination is highly effective for removing color from wastewater, achieving a 99.58 % decolorization rate in 6 minutes. Laib et al. used a catalyst derived from hydroxide sludge in a heterogeneous Fenton process for degradation of Reactive Blue 19.[29] Under optimal conditions, this process achieved a decolorization efficiency of 92 %.

Research on Acid Violet 109 is quite limited. Dajić et al.[30] conducted experiments where AV109 was treated with Fenton's reagent in a batch system. The results showed that process efficiencies of 90.6 %, 90.5 %, and 98.2 % can be achieved after 60 minutes for Fe^{2+} concentrations of 0.2, 0.5, and 1.0 mM, respectively. In another study, Stupar et al. combined adsorption with the Fenton process to decolorize dye AV109.[31] Under optimal conditions, 99.1 % decolorization was achieved, with 55.4 % resulting from adsorption and 43.7 % from the Fenton process. To our knowledge, only one study addresses the treatment of AV109 in a microreactor system. Svetozarević et al.[32] investigated the application of enzyme technologies to degrade AV109 in a microfluidic reactor. The initial concentration of AV109 was 10 mg dm^{-3}, and the results showed that 95 % efficiency was achieved using soya bean peroxidase, and 76 % with potato peroxidase, after just 3 minutes.

3.1. *Effect of initial Fe^{2+} concentration in reaction mixture*

The goal of the first set of experiments was to establish the optimal amount of Fe^{2+} in the reaction mixture. Fe^{2+} ions play a significant role in the reaction as a catalyst. The experiments were conducted in a microreactor with a diameter of 0.3 mm and a length of 1 m, using three different Fe^{2+}/H_2O_2 molar ratios. The results are presented in Figure 3. The results showed that change in molar ratio did not affect the efficiency. However, increasing the concentration of Fe^{2+} ions significantly improved efficiency. Figure 3 revealed that for an initial Fe^{2+}concentration of 1.0 mM dm^{-3}, maximum efficiency (approximately 94 %) was achieved. It can be concluded that increasing the Fe^{2+}concentration from 0.5 to 1.0 mM dm^{-3} resulted in a 7 % increase in efficiency.

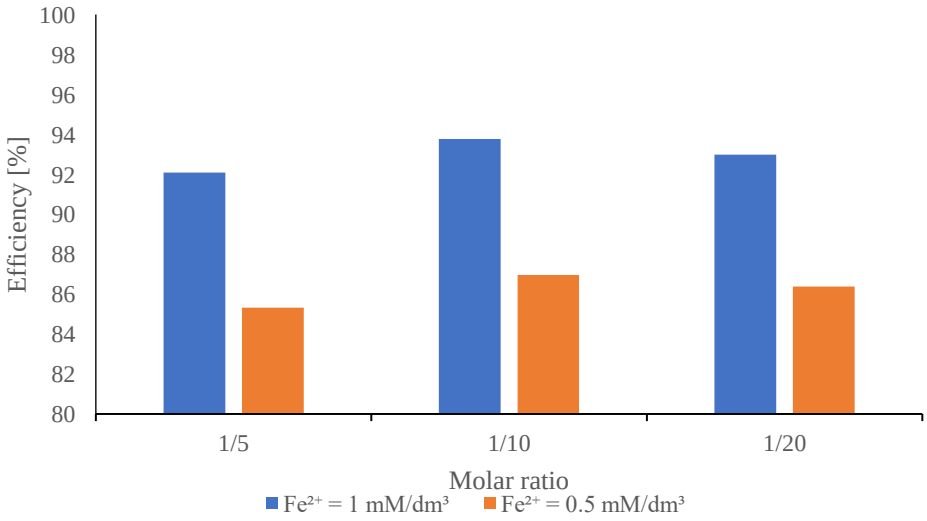

Fig. 3. Influence of Fe^{2+} concentration in reaction mixture on efficiency of dye degradation

3.2. *Effect of microreactor characteristics*

It is known that the diameter and length of a microreactor determine key reaction conditions, such as mixing efficiency and residence time. The influence of microreactor length and diameter at a flow rate of 1 µl min^{-1}, along with different molar ratios of Fe^{2+} ions and hydrogen peroxide (Fe^{2+}/H_2O_2), is shown in Figure 4.

The results (Figure 4) show that the achieved efficiency of decolorization ranged from 86 to 99%. In general, the highest efficiency was achieved in microreactors with a diameter of 0.3 mm, regardless of length. This is likely because the smaller diameter can provide better mixing of the reaction mixture. It was also observed that the efficiency for microreactor lengths of 5 m and 15 m were similar. The lower efficiency observed with the 1 m microreactor may be due to the shorter reaction time. This implies that maximum efficiency is nearly achieved after 5 m. For the 1 m microreactor, efficiency ranged from 89 to 94 %, whereas for the 5 m to 15 m microreactors, the average efficiency exceeded 95 %.

The influence of microreactor length and diameter at a flow rate of 5 µl min^{-1}, along with different molar ratios of ferrous salt and hydrogen peroxide (Fe^{2+}/H_2O_2), are shown in Figure 5.

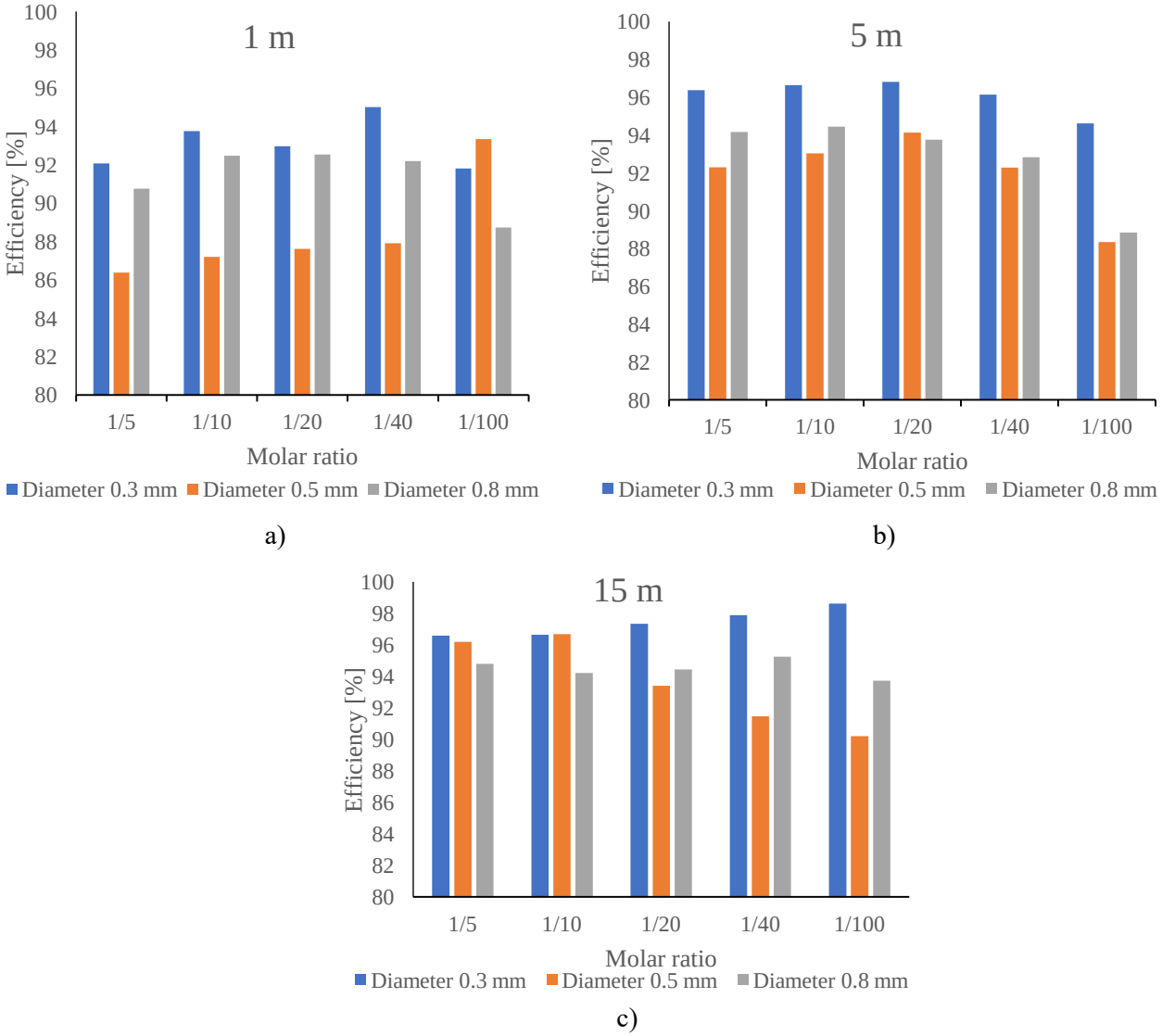

Fig. 4. Effect of microreactor characteristics on efficiency of decolorization for microreactor diameters 0.3, 0.5, and 0.8 mm and flow rates of 1 µl min^{-1} for (**a**) 1 m, (**b**) 5 m, and (**c**) 15 m length

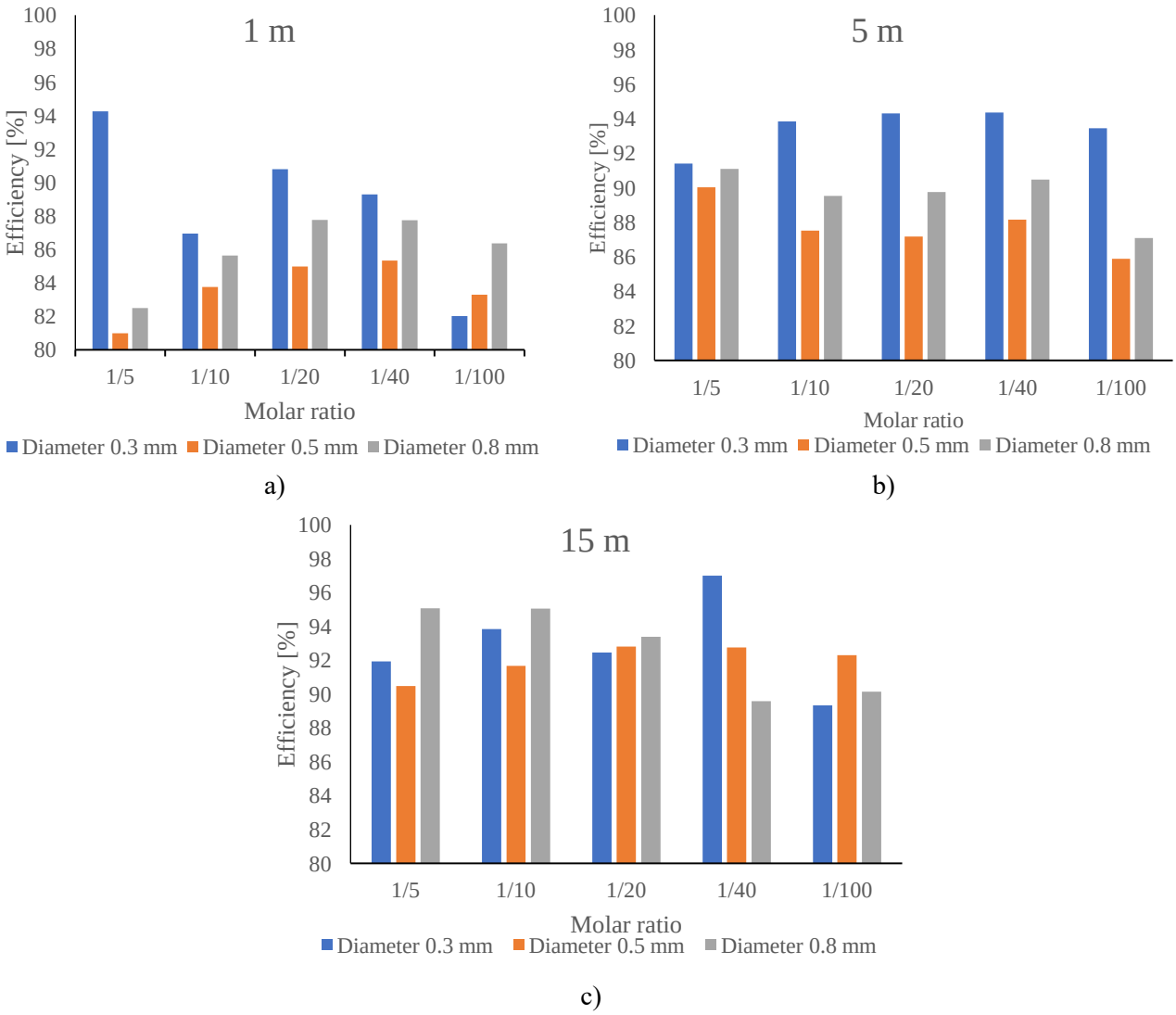

Fig. 5. Effect of microreactor characteristics on efficiency of decolorization for microreactor diameters 0.3, 0.5, and 0.8 mm and flow rates of 5 µl min^{-1} for (**a**) 1 m, (**b**) 5 m, and (**c**) 15 m length

In general, Figure 5 revealed that efficiency in a 1 m long microreactor is significantly lower compared to efficiency achieved in 5 and 15 m microreactors. The high velocity of the reaction mixture and short residence time negatively impacted efficiency. Once again, the highest efficiency was achieved in microreactors with a diameter of 0.3 mm for each reactor length and a 1/5 molar ratio of Fe^{2+} ions. The reaction was most successful in a 15 m long microreactor. The efficiency of decolorization in the 1 m long reactor ranged from 81 % to 94 %, in the 5 m long reactor from 88 % to 94 %, and in the 15 m long reactor from 90 % to 97 %. On average, the efficiencies were 86 %, 90 %, and 92 % for microreactor lengths of 1 m, 5 m, and 15 m, respectively. Additionally, Figure 5 indicates that at a flow rate of 5 µl min^{-1}, the influence of the molar ratio of Fe^{2+} ions is more pronounced than at a flow rate of 1 µl min^{-1}.

3.3. *Effect of Fe^{2+}/H_2O_2 molar ratio*

The influence of the Fe^{2+}/H_2O_2 molar ratio on degradation efficiency is presented in Figure 6. The results are presented for a microreactor with a diameter of 0.3 mm, as this diameter yielded the highest efficiency. It can be concluded that reaction mixtures with molar ratios of 1/5, 1/10, and 1/20 follow the same trend, starting with a relatively high efficiency that stabilizes after 5 m. The molar ratio of 1/40 exhibits the highest initial efficiency, which increases linearly with reaction time. Conversely, the molar ratio of 1/100 has the lowest starting efficiency, but rises rapidly, achieving 99 % efficiency after 15 m. However, the difference in efficiency of molar ratios of 1/5 and 1/100 was only 2 %, indicating that approximately the same efficiency was attained for all analyzed reaction mixtures. Therefore, it can be concluded that

Fe^{2+}/H_2O_2 molar ratios do not have significant influence on efficiency. From an environmental standpoint, the wastewater treatment process should consider the consumption of chemicals. In this case, since the Fe^{2+}/H_2O_2 molar ratios do not have significant impact on efficiency, the 1/5 molar ratio should have an advantage due to its lower consumption of H_2O_2.

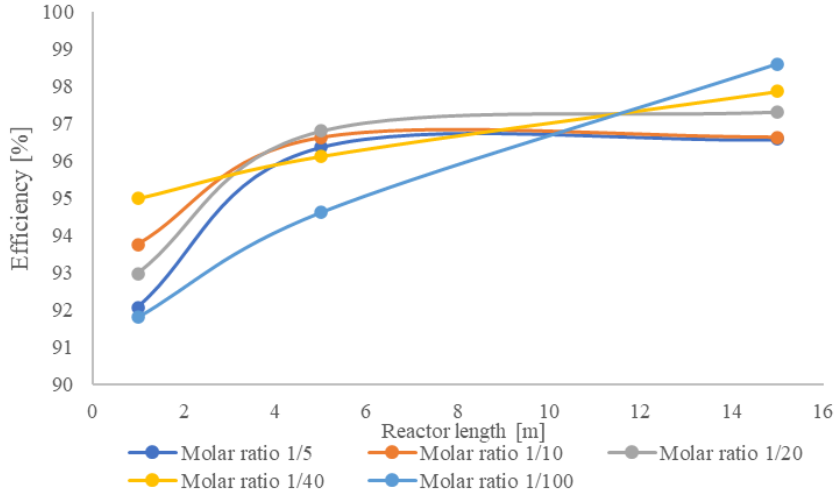

Fig. 6. Effect of molar ratio on the efficiency of dye degradation in reactor with diameter of 0.3 mm

3.4. *Effect of flow rate of the reaction mixture*

Figure 7 presents the influence of flow rate and the molar ratio of Fe^{2+} ions on efficiency. Decolorization efficiency is presented for the microreactor with a diameter of 0.3 mm and a length of 1 m. It should be stressed that for all molar ratios, efficiency is higher at a flow rate of 1 µl min^{-1}. This effect is most pronounced for the molar ratio 1/100, with an approximate difference of around 10 %.

Figure 8 shows the UV spectra of wastewater before and after treatment in the microreactor system for illustration.

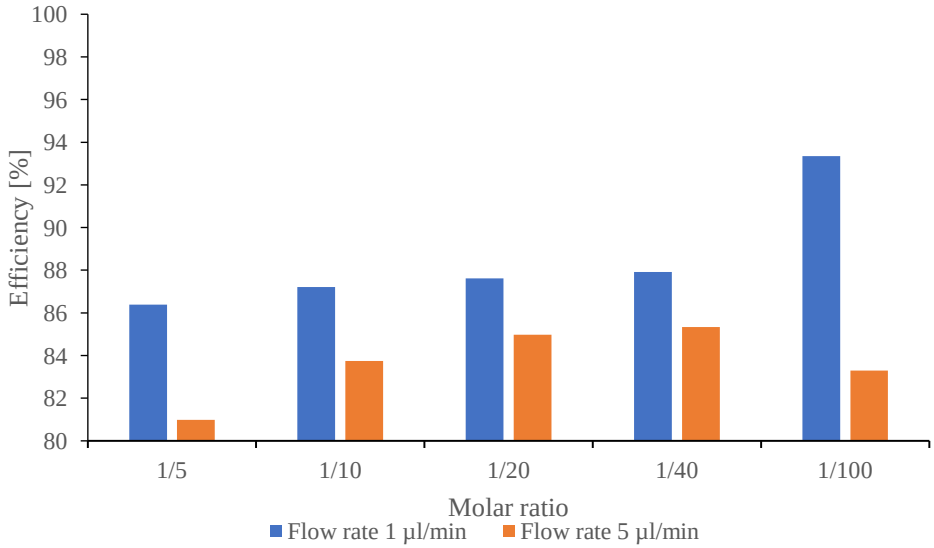

Fig. 7. Influence of reaction mixture flow rate on efficiency of dye degradation in 1 m long reactor with of 0.3 mm diameter

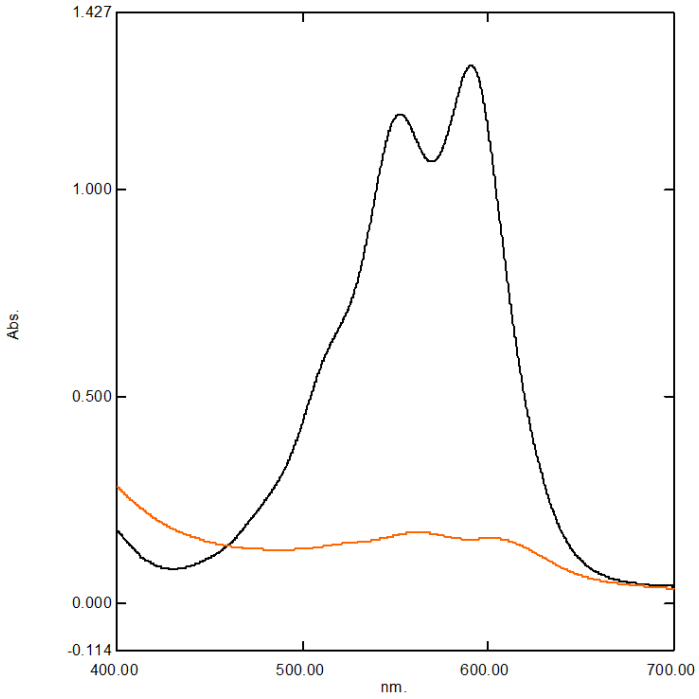

Fig. 8. UV spectra before (—) and after (—) treatment of wastewater in the microreactor

4. CONCLUSIONS

The aim of this study was to determine the feasibility of treating textile wastewater with Fenton's reagent in a microreactor system. The influence of Fe^{2+} concentration, microreactor characteristics, Fe^{2+}/H_2O_2 molar ratio, and total flow rate of the reaction mixture was analyzed. The results showed that better efficiency was achieved with an initial Fe^{2+} ion concentration of 1 mM dm^{-3} compared to 0.5 mM dm^{-3}. Different microreactor lengths showed that after 5 m, the efficiency increased marginally. As expected, the highest efficiency was achieved in microreactors with a smaller diameter, which can be explained by the better mixing achieved in a smaller diameter. The Fe^{2+}/H_2O_2 molar ratio did not significantly affect efficiency; thus, from an environmental perspective, a molar ratio of 1/5 is advantageous due to reduced consumption of H_2O_2. Higher efficiency was achieved at a total flow of 1 µl min^{-1}, compared to 5 µl min^{-1}, particularly for shorter microreactors (1 m). The combination of a high flow rate and short length resulted in a very short residence time, which was insufficient for the reaction to complete.

In general, efficiencies above 95% can be achieved in a 5 m long microreactor with a diameter of 0.3 mm, using a total flow of 1 µl min^{-1} and Fe^{2+} ions concentrations of 1 mM dm^{-3}.

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