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In-house-prepared carbon-based Fe-doped catalysts for electro-Fenton degradation of azo dyes

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Abstract: Compounds used in the fashion industry effect the water bodies in the vicinity of textile factories, resulting in the visible coloration of surface water. Fe-doped graphite-based in house prepared electrodes were used in the Fenton-like degradation of Reactive Blue 52 (RB52). The electrodes consisting of high-density graphite in three granulation sizes and three levels of Fe content were characterized using scanning electron microscopy (SEM). The amount of Fe in the electrodes and H_2O_2 concentration in synthetic textile wastewater were optimized. Additionally, the size of graphite grains was varied to investigate whether it effects the degradation rate. Under only 10 min of electro-Fenton degradation, a system with 10 mmol dm^{-3} of H_2O_2 and an electrode made of 7 % of Fe and 70 μm of granulation size of graphite, degraded over 75 % of RB52, and over 99 % after 40 min of treatment. The obtained results indicate that the proposed approach could be beneficial in the field of novel materials for environmental application and that in house prepared carbon could be an excellent replacement for commercially available supports.

Keywords: Reactive Blue 52; granulation; decolorization; hydrogen peroxide; graphite; advanced oxidation processes.

INTRODUCTION

There have been many improvements of the classic Fenton reaction, a homogenous system with Fe^{2+} and H_2O_2 in the acid environment, but the electro-Fenton reactions contribute to *in situ* generation of reactive species in water due to

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the effect of the electric current.[1,2] Electro-Fenton oxidation processes occur at ambient pressure and ambient temperature and therefore belong to the electrochemical advanced oxidation processes (electro AOP).[3,4] In an acidic environment, Fe^{2+} oxidizes to Fe^{3+}, as shown in Eq. (1), but in pH neutral and aerobic conditions, Fenton-like reactions are possible that could lead to a respectable amount of generated oxidative species, as shown in Eqs. (2) and (3):[5,6]

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO^{\bullet} + OH^{-} \quad (1)$$

$$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + HOO^{\bullet} + H^{+} \quad (2)$$

$$Fe^{3+} + HOO^{\bullet} \rightarrow Fe^{2+} + O_2 + H^{+} \quad (3)$$

To prevent Fe deposition and to increase the efficacy of Fenton reactions in pH neutral environment, a stable iron support is often needed.[7,8] Carbonaceous materials are commonly considered as readily available, cost-effective, and durable support materials for numerous electrochemical applications,[9–12] and especially graphite can be attractive in terms of different electrode preparation possibilities, due to its stability.[13–15] The effects of the ratio of iron and hydrogen peroxide on Fenton and Fenton-like reactions are well studied,[16] but novel electrode formulations open questions never asked before, such as granulation of the starting carbonaceous material. Techniques for electrodes production can strongly affect the efficiency, stability and reusability of electrodes.

Rapid fashion changes and mass production of clothes lead to enormous amounts of textile waste, compounds used in the fashion industry mostly effect the water bodies around textile factories, resulting in visible coloration of surface water. Therefore, effective, stable, and low-cost approaches are needed to combat this growing problem.[17,18]

This study was based on electro-Fenton oxidation of Reactive Blue 52 (RB52), a textile azo dye, where up to 10 ppm of H_2O_2 was externally added to the electrochemical system. Fixed amounts of high-density graphite and phenol–formaldehyde resin were used to prepare nine electrodes with three ranges of graphite granulations and Fe contents. The electrodes were applied in the form of pellets to find the optimal combination of graphite granulation, iron content, and H_2O_2 concentration to achieve the fastest removal of RB52 without pH adjustment.

EXPERIMENTAL

Chemicals

$Fe(NO_3)_3 \cdot 9H_2O$ (ACS reagent, ≥98 %, Sigma Aldrich, CAS 7782-61-8) was used as a doping material for the in house prepared carbon-based catalysts. A high-density graphite (trademark VPG-4) prepared in three granulation sizes was used as a precursor for the electrodes, while novolac phenol–formaldehyde resin SFPR-054 (Wego Chemical Group, CAS 9003-35-4, hereinafter SFPR binder), light-yellow powder with 26 % of hexamethylenetetramine (with 60 % of carbon content in SFPR binder heated at 950 °C) by weight served as a binder. Ethanol (95 vol. %, Sigma Aldrich) was needed for the preparation of precursors. Reactive Blue 52, a textile azo dye, a model compound (Clariant, CAS 12225-63-7, hereinafter RB52), Na_2SO_4 (≥99 %, Sigma Aldrich, CAS 7757-82-6) a supporting electrolyte, and H_2O_2 (30 vol. %, Sigma Aldrich, CAS 7722-84-1) were involved in the electrochemical degradation tests.

Electrodes preparation

The Fe-doped porous graphite electrodes were produced following a procedure published elsewhere.[19,20] Compared to work of Manojlović and colleagues,[19] this study was expanded by using three median granulation sizes of high-density graphite fractions cut on lathes. The grain size was controlled by adequate sieve openings, e.g. sieves with dimensions between 50 and 90 μm resulted in a median grain size of 70 μm, between 90 and 160 μm gave 125 μm, and those between 160 and 250 μm gave a median grain size of 205 μm (see more in Table I). A fixed amount of SFPR binder (0.9 g) and different amounts of $Fe(NO_3)_3\cdot 9H_2O$ were powdered and mixed. The content of Fe was proportional to the carbon content in the SFPR binder, hence 0, 0.078 and 0.273 g of $Fe(NO_3)_3\cdot 9H_2O$ was weighed to produce electrodes labelled as 0, 2 and 7 % of Fe. Three grams of graphite grains of appropriate size (*i.e.*, 70, 125 or 205 μm of medium size) was combined with the previous ingredients and again combined to make precursors for nine electrodes. These mixtures were then wetted, pelleted, and heated according to the procedure published by Petković *et al.*,[20] resulting in nine Fe-doped porous graphite electrodes, as listed in Table I. The electrodes were labeled according to the median size of graphite grains and Fe content, for example, electrode 70-0 container graphite with roughly 70 μm of grain size and 0 % of Fe. The morphology of the SFPR binder and the prepared electrodes were examined on a scanning electron microscope, Jeol JSM-7001F (SEM).

TABLE I. Properties of the studied Fe-doped porous graphite electrodes

Electrode label	Content of Fe in relation to SFPR binder, %	Graphite grain size μm	Median grain size μm
70-0	0	50–90	70
70-2	2	50–90	70
70-7	7	50–90	70
125-0	0	90–160	125
125-2	2	90–160	125
125-7	7	90–160	125
205-0	0	160–250	205
205-2	2	160–250	205
205-7	7	160–250	205

Electro-Fenton decolorization experiments

RB52 (30 mg dm^{-3}) was used as a model compound for the comparison of electrode effectiveness, while 0.1 mol dm^{-3} Na_2SO_4 was chosen for the supporting electrolyte. The original divided electrochemical cell (Fig. 1) contained 50 cm^3 of azo dye with Na_2SO_4 in the anodic part, with an applied anodic potential of 4.2 V and a current of 30 mA. Additionally, the cathodic part, separated by Nafion 117 perfluorinated membrane (178 μm thick) from the anodic part, was loaded only with supporting electrolyte solution. Fenton-like electrochemical degradation experiments involved all nine electrodes (see Table I) and four H_2O_2 concentrations (namely, 0, 1, 5 and 10 mmol dm^{-3}), including experiments at native pH value, without hydrogen peroxide. The effect of the optimal combination of graphite granulation, Fe content, and H_2O_2 concentration on RB52 decolorization was monitored on UV–Vis spectrophotometer (Evolution 200 Series, Thermo Fisher Scientific) at the λ_{max} = 615 nm. Each electro-Fenton degradation experiment lasted 50 min and samples were withdrawn after every 10 min. Graphs were generated using OriginPro® 8. The preparation of the electrodes was performed at the South Ural State University and the electro-Fenton degradation of RB52 at the University of Belgrade during 2018–2019.

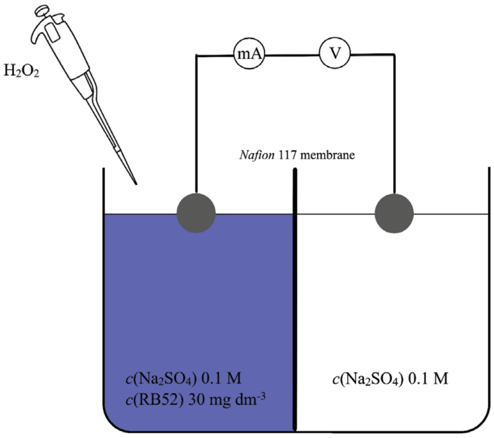


Fig. 1. Experimental setup.

RESULTS AND DISCUSSION

In this study, three levels of Fe content in Fe-doped graphite electrodes were combined with three ranges of grain size of graphite used to obtain electrodes for optimal Reactive Blue 52 (RB52) degradation. Additionally, H_2O_2 was added in four concentration levels to optimize electro-Fenton decolorization without pH value adjustment. The morphology of the SFPR binder at 5000× and 100× of magnification on SEM is shown Fig. 2.

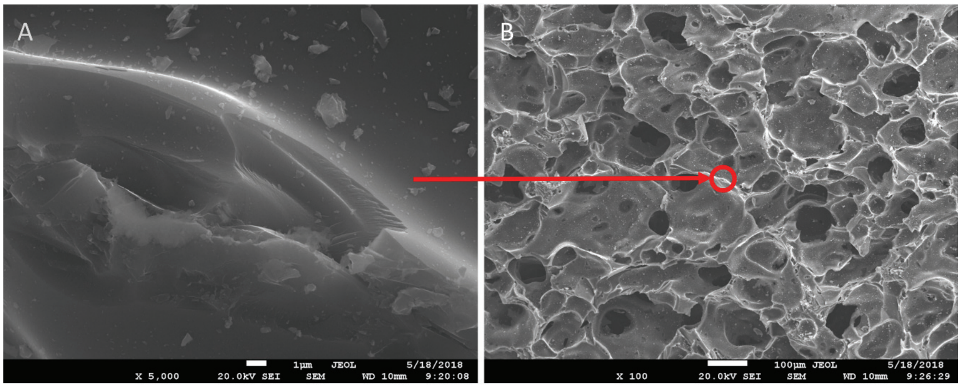


Fig.2. SEM images of SFPR binder at: A – 5000 and B – 100× of magnification using the secondary electron imaging (SEI) technique.

It could be concluded from Fig. 3 that each material was covered with large pores, formed in a heavily branched structure, and filled with spherical vacancies. By comparing the same magnification of the SEM images, it could be concluded that pore sizes were proportional to the grain size, indicating that the proposed procedure could serve as a controllable method for the preparation of the electrodes listed in Table I. Electrodes without iron are represented in Fig. 3A, D and G. The other electrodes shown in Fig. 3 represent the morphology of electrodes with 2 and 7 % of iron, where each material was covered with a thin layer of Fe_3O_4 of high crystallinity and octahedral in shape, forming a high surface area, which is preferable for environmental use.

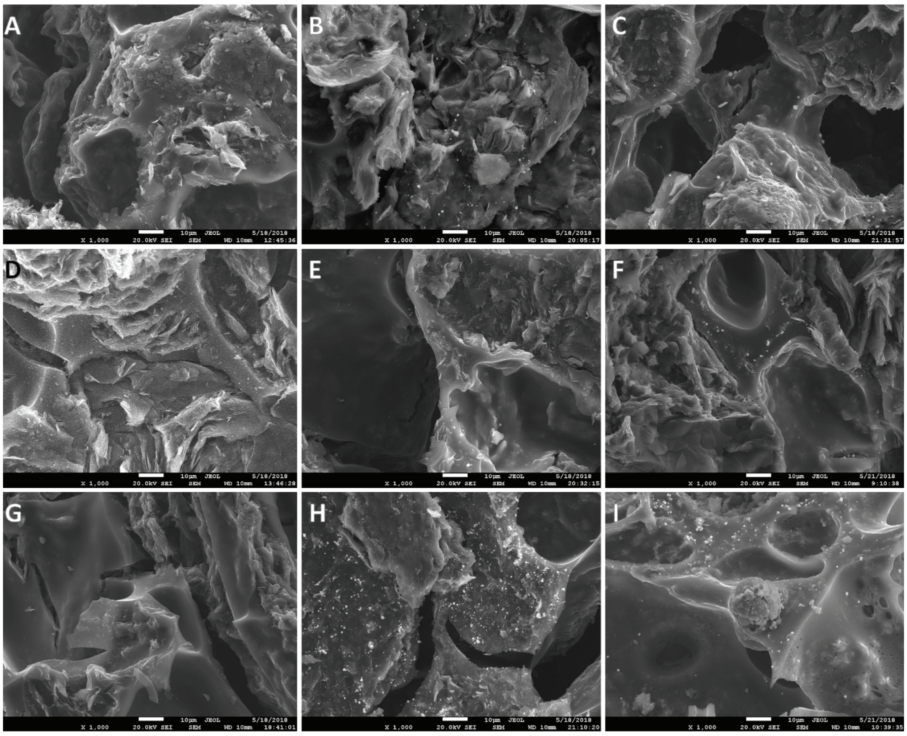


Fig. 3. SEM images of: A – 70-0, B – 70-2, C – 70-7, D – 125-0, E – 125-2, F – 125-7, G – 205-0, H – 205-2, I – 205-7 electrodes under 1000× of magnification using SEI technique. The first number stands for the granulation value and the second number for the amount of Fe (see Table I).

The changes in the decolorization rate of RB52 are shows in Fig. 4A–C as a function of Fe content and graphite grains size, with no added hydrogen peroxide. The highest degradation rate after 50 min of treatment was achieved (over 75 %) when the electrode with 2 % of Fe and 125 μm of graphite granulation (label 125-2, see Table I) was applied, as shown in Fig. 4B. The raw data for

Figs. 4–8 are organized in Tables S-I–S-IX of the Supplementary material to this paper. On the other hand, RB52 was the least degraded (below 45 %) with electrodes of 70 μm grain size of graphite and without iron (Fig. 4A). In all cases (Fig. 4A–C), the middle level (125 μm) of graphite granulation accomplished higher decolorization rates, compared to the other two granulation sizes. Moreover, in the case of electrodes with 205 μm grains size (Fig. 4C), there were no significant differences in RB52 degradation efficiency between the electrodes with the lowest and the highest Fe content (almost 60 % and over 51 % of degraded RB52, respectively). The results showed in Fig. 4 were achieved without the addition of H_2O_2 and could be attributed to the *in situ* electrochemical production of hydroxyl radical and other reactive species, as presented in Eqs. (1)–(3).[5,6]

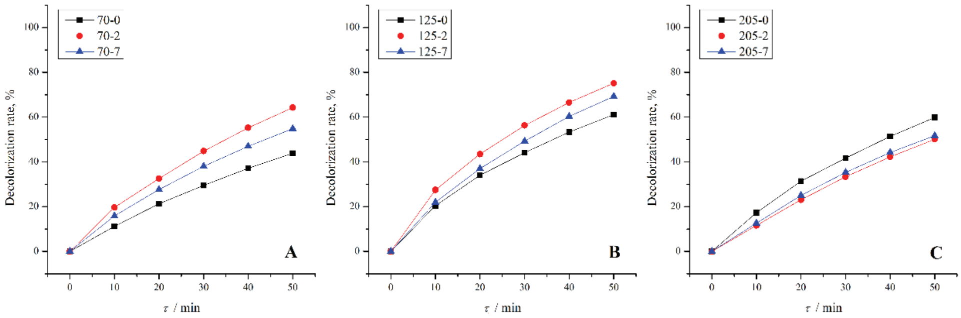


Fig. 4. Change in RB52 decolorization rate for electrodes made of different graphite granulation size; A – electrodes with no Fe, B – electrodes with 2 % of Fe and C – electrodes with 7 % of Fe (starting conditions: 30 mg dm^{-3} RB52 solution in the absence of H_2O_2).

Completely different results were observed when 1 mmol dm^{-3} of H_2O_2 was added to all the prepared electrodes because in the presence of added hydrogen peroxide electro-Fenton reactions occurred to a greater extent (Fig. 5A–C). According to the results, 2 % of iron leads to the fastest RB52 degradation rates, achieving roughly 60 % degradation after only 10 min of treatment for all graphite granulation sizes, which could imply the mutual dependence of the iron content in the electrode and the quantity of H_2O_2 for optimal Fenton reactions. Similar to the previous results, electrodes 70-2 and 125-2 with (Fig. 5) or without H_2O_2 (Fig. 4) culminated in higher degradation rates compared to larger grain sizes (Fig.s 4C and 5C, respectively). Moreover, with 1 mmol dm^{-3} of H_2O_2 in the system, electrodes with no iron and lower grain sizes (70-0 and 125-0) also resulted in slightly higher endpoints (92 and 87 % after 50 min of treatment, respectively), compared to 205-0 with 83 % of RB52 degraded at the same point. The only exception to the observed rule in granulation size effect were electrodes with 7 % of Fe. Namely, 1 mmol dm^{-3} of H_2O_2 led to less than 80 % of degradation rate for the 70-7 electrode, but around 94 % for the other two electrodes (125-7 and 205-7) after 50 min of treatment.

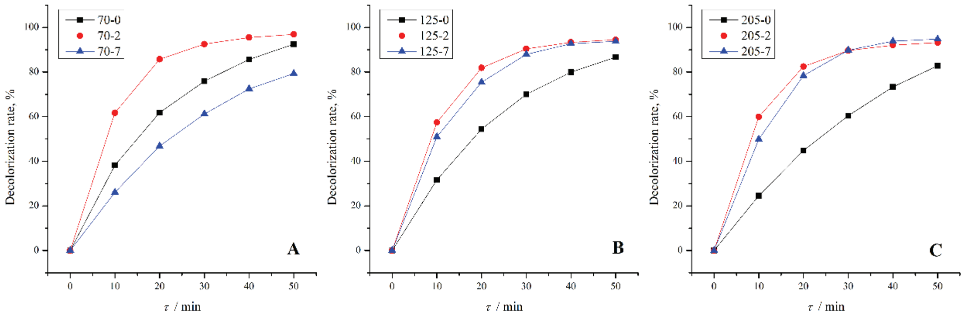


Fig. 5. Change in RB52 decolorization rate when using electrodes with different Fe contents; graphite granulation size: A –70, B –125 and C –205 µm (starting conditions: 30 mg dm^{-3} RB52 solution and 1 mmol dm^{-3} H_2O_2).

High degradation rates were also achieved with five times higher H_2O_2 concentration (Fig. 6A–C). Furthermore, there was no significant difference in the decolorization rate (roughly 95 % at 50 min) between electrodes with 2 and 7 % of iron, for all three granulation levels, while the RB52 degradation was somewhat slower without iron present in the electrodes.

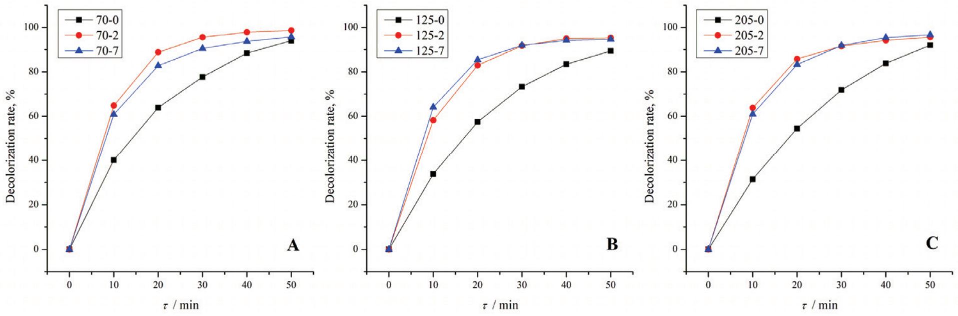
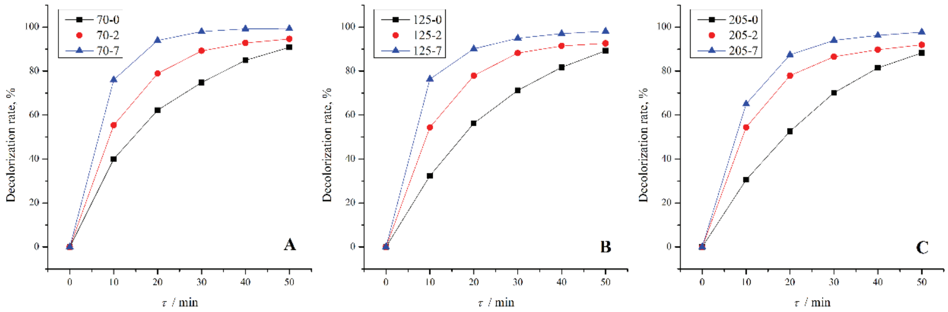


Fig. 6. Change in RB52 decolorization rate when using electrodes with different Fe content; graphite granulation size: A –70, B –125 and C –205 µm (starting conditions: 30 mg dm^{-3} RB52 solution and 5 mmol dm^{-3} H_2O_2).

The highest concentration of H_2O_2 investigated in this study was 10 mmol dm^{-3}, as depicted in Fig. 7. Under these conditions, almost 80 % of dye was degraded in only 10 min by electrodes 70-7 and 125-7 (Fig. 7A and B), the latter being the only system in which 7 % of iron led to the fastest decolorization. Although endpoints in all systems were remarkable and comparable with lower C (H_2O_2), this extent of hydrogen peroxide slightly suppressed the decolorization by electrodes with 2 % of Fe (Fig. 7A–C).

The best results shown in the previous figures, selected for each amount of H_2O_2 combined with various electrodes, are summarized in Fig. 8. Obviously, the highest hydrogen peroxide concentration led to the quickest, but also the most effective RB52 degradation, as already shown in Fig. 7C. All of the systems with added H_2O_2 enable more efficient electro-Fenton reactions, which lead to similar outcomes, in terms of RB52 decolorization in 50 min of treatment. What also stands out, electrodes with the lowest graphite grains size (70 μm) came out to give higher degradation rates, compared to the other two tested sizes. In most cases, iron improved degradation percentage, and the optimal iron content was 2 % (1 and 5 mmol dm^{-3} of H_2O_2), except for the 10 mmol dm^{-3} of H_2O_2, where a higher amount of Fe was needed.



Fig, 7. Change in RB52 decolorization rate when using electrodes with different Fe contents; graphite granulation size: A –70, B – 125 and C –205 μm (starting conditions: 30 mg dm^{-3} RB52 solution and 10 mmol dm^{-3} H_2O_2).

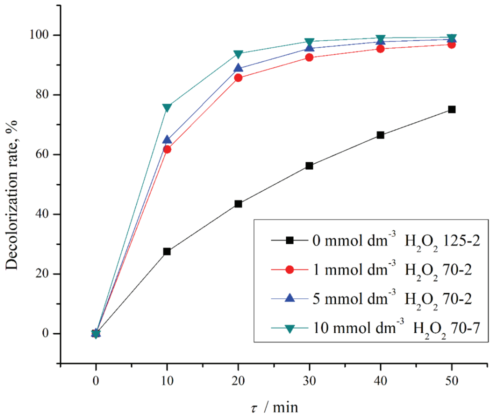


Figure 8. Comparison of the best RB52 decolorization rates for selected electrodes – with different Fe content, H_2O_2 concentration, and graphite granulation size.

This result also indicates that Fe and H_2O_2 ratio follows a certain positive correlation trend, and the outcome of electro-Fenton oxidation could be predicted and adjusted to the desired results, mostly based on the Fe/H_2O_2. On the other hand, decolorization of RB52 was still possible, even without H_2O_2 – the 125-2 electrode and no H_2O_2, which resulted in almost 30 % degradation efficiency after 10 minu of treatment, had the endpoint for the same electrode comparable to the result achieved with 70-7 and 10 mmol dm^{-3} H_2O_2 in the first 10 min when roughly 80 % of azo dye was removed. This result could imply that the degradation system without added hydrogen-peroxide could be compensated with longer treatment time, and *vice versa* – the degradation time could be shortened with higher content of oxidation agent.

In the electro-Fenton oxidation of an azo dye Reactive Blue 52 presented here the maximal concentration of H_2O_2 was 50 times less than reported elsewhere.[21] Although higher concentration of H_2O_2 often leads to better degradation rates, the excess of hydrogen peroxide added can have a scavenging effect on hydroxyl radicals.[1] Even though the degradation efficiencies are often tested by the degradation rate of colored substances, their applicability is not limited to the textile industry, but could be expanded to many other organic chemicals with similar functional groups.

CONCLUSIONS

The proposed study contributes to a better understanding of electro-Fenton degradation of Reactive Blue 52, in terms of the role of the graphite granulation size, iron, and H_2O_2 content. Under only 10 min of electro-Fenton degradation, a system with 10 mmol dm^{-3} of H_2O_2 and an electrode made of 7 % of Fe and 70 μm granulation size of graphite, degraded over 75 % of the treated azo dye. Additionally, less than 1 % of dye remained after 40 min of treatment. It should be emphasized that even lower H_2O_2 concentrations lead to a satisfactory degradation rate, when 70-2 electrodes were used. Even though the removal efficiency of the presented system was tested by the degradation rate of colored substance, this study could serve as a basis for similar electrochemical degradations and its applicability could be expanded to many other organic chemicals with analogous functional groups, with further optimization of the experimental parameters.

SUPPLEMENTARY MATERIAL

Additional data and information are available electronically at the pages of journal website: https://www.shd-pub.org.rs/index.php/JSCS/article/view/11130, or from the corresponding author on request.

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ИЗВОД

ИЗРАДА И СВОЈСТВА КАТАЛИЗАТОРА НА БАЗИ УГЉЕНИКА ДОПОВАНИХ ГВОЖЂЕМ ЗА ЕЛЕКТРО-ФЕНТОНСКУ РАЗГРАДЊУ АЗО-БОЈА

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Једињења која се користе у модној индустрији утичу на водна тела у околини текстилних фабрика, што резултира видљивим обојењем површинских вода. Домаће електроде на бази графита допиране гвожђем биле су укључене у деградацију Reactive Blue 52 (RB52) механизма попут Фентонове реакције. Електроде су се састојале од графита велике густине у три величине гранулације и три количине Fe и окарактерисане су помоћу скенирајуће електронске микроскопије (SEM). Оптимизоване су количина Fe у електродама и концентрација H_2O_2 у синтетичкој текстилној отпадној води. Додатно, величина графитних зрна је варирана како би се испитало да ли утиче на брзину разградње. За само 10 min електро-Фентонове деградације систем са 10 mmol dm^{-3} H_2O_2 и електродом од 7 % Fe и 70 μm величине гранулације графита разградило се преко 75 % RB52 и преко 99 % након 40 min третмана. Добијени резултати указују на то да предложени приступ може бити користан у области нових материјала за примену у животној средини и да домаће припремљени угљеник може бити одлична замена за комерцијално доступне носаче.

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