

Lech Kos,
Jadwiga Sójka-Ledakowicz,
Karina Michalska,
*Jan Perkowski

Textile Research Institute,
ul. Brzezińska 5/15, 92-103 Łódź, Poland,
E-mail: lkos@iw.lodz.pl

**Institute of Applied Radiation Chemistry,
Lodz University of Technology,
ul. Wróblewskiego 15, 93-590 Łódź, Poland,
E-mail: japerepi@mitr.p.lodz.pl

Decomposition of Azo Dye C.I. Direct Yellow 86 by the Fenton Process in the Presence of Nanoparticles of Iron Oxides

Abstract

The aim of the study was to determine the efficiency of decomposition of azo dye C.I. Direct Yellow 86 by the Fenton method in the presence of nanoparticles of iron oxides and to compare it with the classical Fenton method. Water solutions of the dye were subjected to the classical purification method with the application of ferrous sulfate and – for comparison – to a process in which iron (II,III) oxide nanopowder was added to the ferrous sulfate. Analysis of the effect of the ferrous sulfate, iron (II,III) oxide nanopowder, hydrogen peroxide and the pH of the solution on the treatment efficiency showed that the process was optimised. The use of iron oxide nanopowder increased the efficiency of dye decomposition.

Key words: azo dye, Fenton reaction, nanoparticles, iron oxides, dye decomposition.

Introduction

Decolourisation is a process important for wastewater treatment. Coloured dyes discharged to surface water hinder the access of sunlight, which distorts the conditions in which aquatic ecosystems exist. Additionally the aesthetic quality of reservoirs and their recreational role deteriorate.

A major source of coloured dyes is the textile industry. Their content in textile wastewater depends on the type of raw material being processed, the dyeing technology, wastewater treatment methods as well as on changing fashions and the demand for a given kind of textile.

Hence efficient methods are sought to ensure effective decolourisation of textile wastewater. One of them is the Fenton process, which belongs to the group of so called advanced oxidation processes (AOPs) using hydroxyl radicals formed in a chain process of hydrogen peroxide decomposition in the presence of bivalent iron salts. The hydroxyl radicals are characterised by a high oxidising potential and non-selective activity and are able to decompose even the most resistant pollutants appearing in the wastewater. Dyes, including azo varieties, decompose well in aqueous solutions in all types of advanced oxidation processes. In the Fenton process, besides oxidation, there is also coagulation connected with the precipitation of iron III hydroxide deposits, due to which textile wastewater can be decolourised significantly [1-6]. Advantages of the Fenton process include high efficiency of pollutant decomposition, simple technology and low cost of the method.

A disadvantage of the process is the sludge formed during coagulation, which requires separation, storage in proper conditions and utilisation. Thus it is very important to minimise the amount of iron added to the treated solution so as to reduce the sludge quantity. Therefore it is necessary to make an optimal choice which would take into account the effi-

ciency and rate of oxidation on the one hand and the amount of iron hydroxide deposits on the other.

New trends in researches on the oxidation of pollutants by the Fenton method comprise the use of nanoparticles of iron compounds in reaction systems [7 - 9]. The presence of nanoparticles affects the

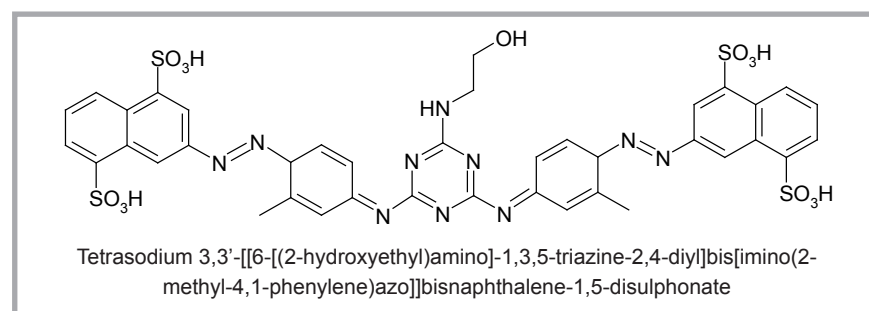


Figure 1. Molecular structure of C.I. Direct Yellow 86.

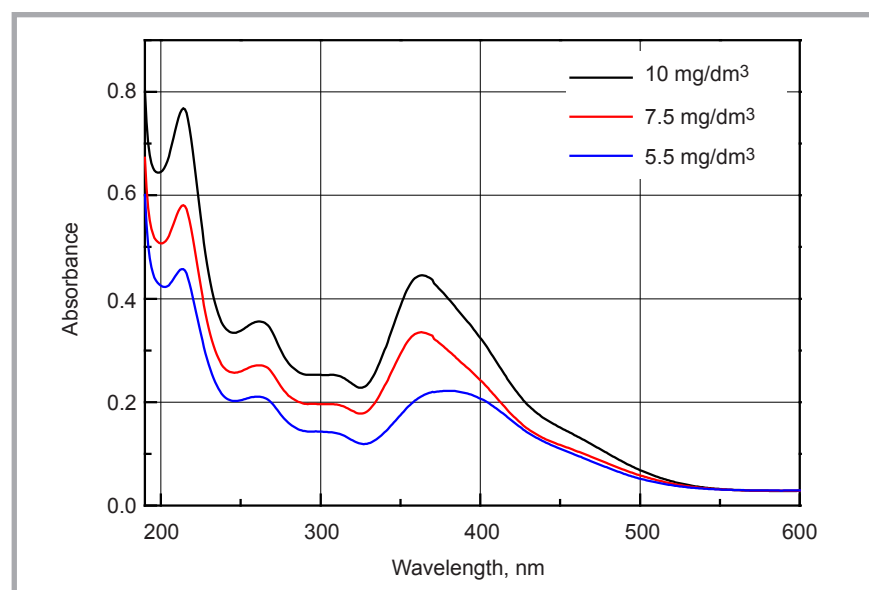


Figure 2. Spectrum of C.I. Direct Yellow 86 dye in water solution for different initial concentrations.

oxidation of many chemical compounds occurring in water and wastewater. In many cases the application of nanoparticles of iron compounds has an influence on increasing the oxidation efficiency, owing to which hazardous chemical compounds present in water decompose faster and more effectively. This refers to many different chemical compounds, including water solutions of trichloroethylene, phenol, dyes, humic acids, antibiotics, metals and hormones [10 - 15]. Certain nanoparticles of metal compounds assist pollutant decomposition in Fenton processes carried out with the use of nanoparticles of iron compounds [16 - 18].

The aim of the studies presented in this paper was to determine the efficiency of decomposition of azo dye C.I. Direct Yellow 86 in the Fenton process in the presence of nanoparticles of iron oxides in comparison with the classical Fenton process. Azo dyes belong to the group of hardly biodegradable xenobiotics, whose efficient removal from wastewater is a necessity.

Methods

Subject of studies. The subject of our studies was water solutions of azo dye C.I. Direct Yellow 86 (laboratory sample) at a concentration of 0.1 g/dm³. Molecular formula – C₃₉H₃₀N₁₀Na₄O₁₃S₄, molecular weight 1066.93 g/mol. The molecular structure is shown in **Figure 1**.

Experimental procedure. Analytically pure ferrous sulfate FeSO₄·7H₂O and 30% hydrogen peroxide H₂O₂ (Chem-pur, Piekary Śląskie), iron (II, III) oxide nanopowder <50 nm (Sigma Aldrich) as well as H₂SO₄ and analytically pure NaOH (POCh, Gliwice) were used in the experiments.

The pH values of the water dye solutions were reduced to 4, 3.5, 3, 2.5 or 2 by means of 2.5 M solution of sulfuric acid. Next the samples were completed either exclusively with ferrous sulfate or jointly with ferrous sulfate and iron (II, III) oxide nanopowder in a solid state, and the solution was stirred until complete dissolution. Then 30% solution of hydrogen peroxide was added drop-wise to the solutions. Once H₂O₂ had been added, the wastewater was stirred vigorously for 2 minutes and then slowly for the next 10 minutes. The solution was left for 24 hours. Next the samples were neu-

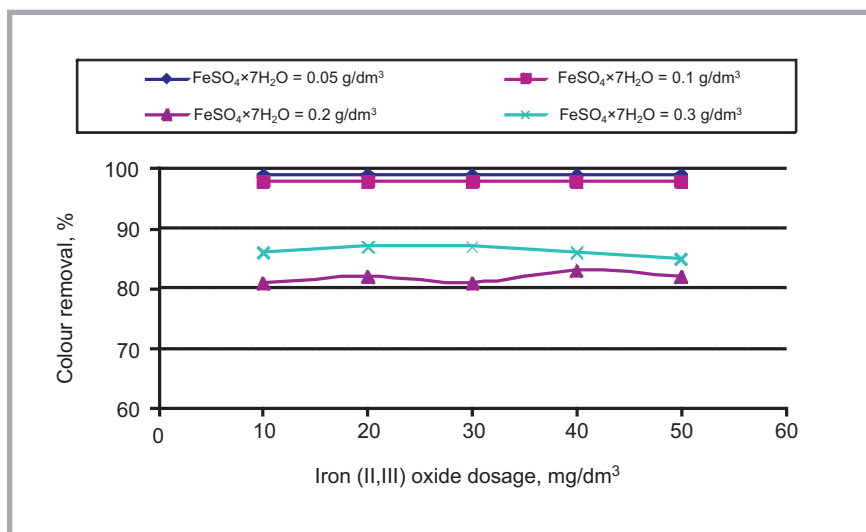


Figure 3. Effect of ferrous sulfate and iron oxide nanopowder on colour removal in C.I. Direct Yellow 86 dye solution in the nanoFenton process. Hydrogen peroxide dosage – 1 cm³/dm³, initial absorbance – 4.45 (wavelength 364 nm), pH 3.5.

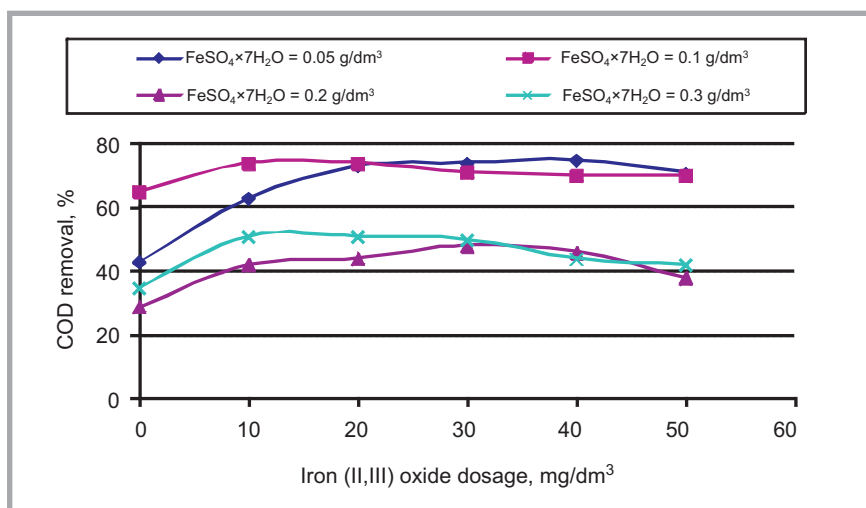


Figure 4. Effect of ferrous sulfate and iron oxide nanopowder on COD removal in C.I. Direct Yellow 86 dye solution in the nanoFenton process. Hydrogen peroxide dosage – 2 cm³/dm³, initial COD = 84 mg O₂/dm³, pH 3.5.

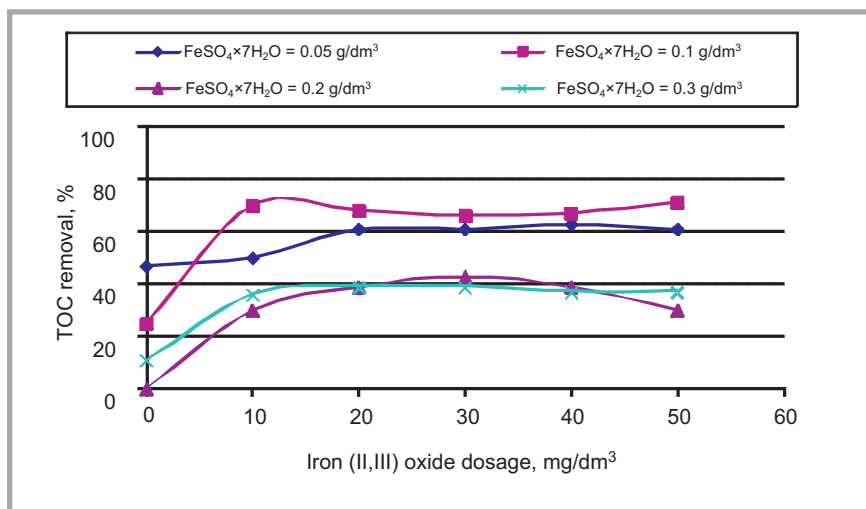


Figure 5. Effect of ferrous sulfate and iron oxide nanopowder on TOC removal in C.I. Direct Yellow 86 dye solution in the nanoFenton process. Hydrogen peroxide dosage – 2 cm³/dm³, initial TOC = 27.9 mg/dm³, pH 3.5.

Table 1. Effect of iron oxide nanopowder dosages on colour removal in C.I. Direct Yellow 86 dye solution in the nanoFenton process depending on the amount of hydrogen peroxide supplied. Ferrous sulfate dosage – 0.1 g/dm³, initial absorbance – 4.45 (wavelength 364 nm), pH 3.5.

H ₂ O ₂ dosage, cm ³ /dm ³	Colour reduction, %					
	Dosages of iron oxide nanopowder, g/dm ³					
	0.00	0.01	0.02	0.03	0.04	0.05
1	99	98	98	98	98	98
2	99	99	99	98	98	98
3	99	98	98	95	97	98
5	99	99	98	98	98	98

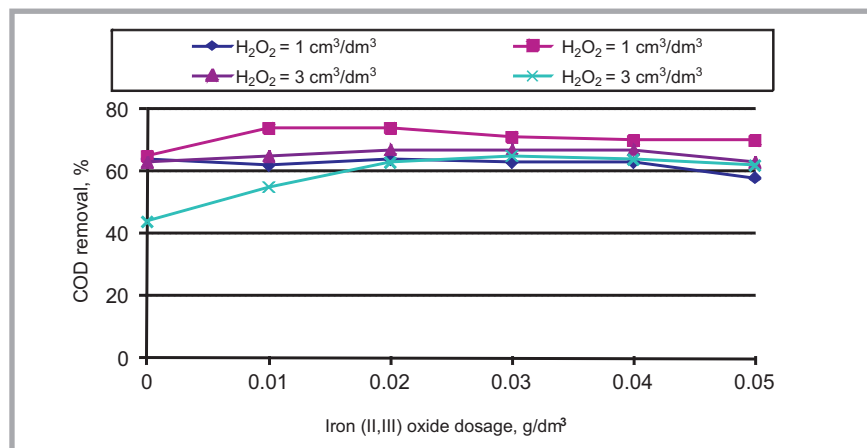


Figure 6. Effect of iron oxide nanopowder dosages on COD removal in C.I. Direct Yellow 86 dye solution in the nanoFenton process at different dosages of hydrogen peroxide introduced to the system. Ferrous sulfate dosage – 0.1 g/dm³, initial COD = 84 mg O₂/dm³, pH 3.5.

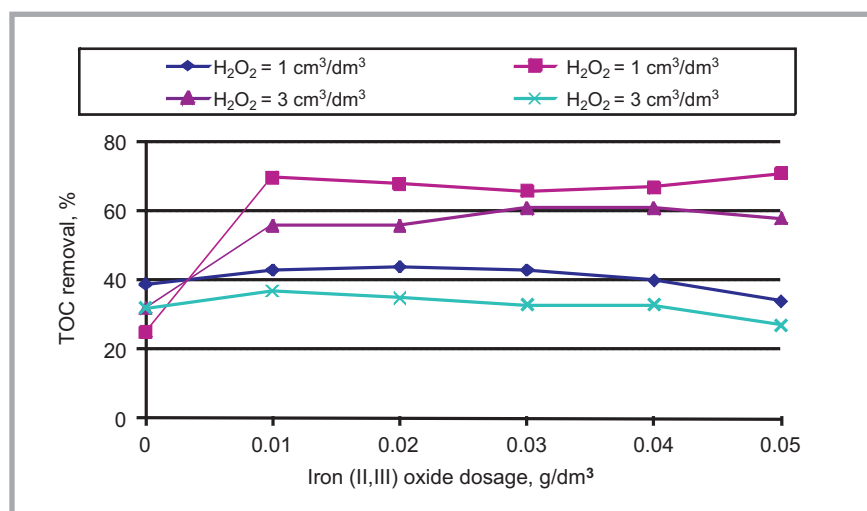


Figure 7. Effect of iron oxide nanopowder dosages on TOC removal in C.I. Direct Yellow 86 dye solution in the nanoFenton process at different quantities of hydrogen peroxide added to the system. Ferrous sulfate dosage – 0.1 g/dm³, initial TOC = 27.9 mg/dm³, pH 3.5.

tralised with 10% solution of NaOH to a pH of about 11. After 24 hours the purified water dye solutions were decanted and filtered.

Analytical control. In samples before and after the treatment, the colour was determined by the spectrophotometric

method. The absorbance was measured at a wavelength of 364 nm. COD (Chemical Oxygen Demand) and TOC (Total Organic Carbon) were also determined by Hach-Lange tests. The initial spectrum of the C.I. Direct Yellow 86 dye for different concentrations is shown in **Figure 2**.

Results and discussion

In the experiments the effect of process conditions (the amount of ferrous sulfate, iron oxide nanopowder, hydrogen peroxide and pH of the solution) on the efficiency of dye decomposition was determined. Checked also was how the addition of iron oxide nanopowder improved the rate and effectiveness of decomposition as compared with the classical Fenton process. The initial dye concentration was 100 mg/dm³. The experiments were carried out with a ferrous sulfate amount ranging from 0.05 to 0.3 g/dm³ at iron oxide nanopowder dosages varying from 0.01 to 0.05 g/dm³. The weight ratio of iron oxide nanopowder to ferrous sulfate ranged from 1:1 to 1:30. The dosages of hydrogen peroxide were from 1 to 5 cm³/dm³. The effect of the pH on dye decomposition was studied at a pH equal to 2, 2.5, 3, 3.5 and 4. Colour reduction, COD and TOC were determined after processing.

The effect of iron oxide nanopowder dosage

The studies began with the determination of the effect of iron oxide nanopowder dosage on the efficiency of C.I. Direct Yellow 86 dye decomposition in the Fenton process.

Irrespective of the amount of iron oxide nanopowder added, the wastewater was very well decolourised, with the colour removal ranging from 81 to 99%. The best decolourisation of 98 - 99% was achieved with the smallest ferrous sulfate doses (0.05 - 0.1 g/dm³). **Figure 3** shows selected results.

The COD removal achieved in the experiment ranged from 38 to a maximum of 74% depending on the dosages of both ferrous sulfate and iron oxide nanopowder applied. The amount of iron oxide nanopowder added at a constant dosage of ferrous sulfate had an influence on the COD removal, increasing the treatment efficiency by several percent. The optimum dosages of iron oxide nanopowder ranged from 0.02 to 0.03 g/dm³. The best COD removal was achieved at the lowest dosages of ferrous sulfate applied (0.05 - 0.1 g/dm³). Examples of the results obtained are illustrated in **Figure 4**.

While analysing the TOC values it was found that in this case they depended on the amount of both ferrous sulfate and

iron oxide nanopowder. The quantity of iron oxide nanopowder added at a constant dosage of ferrous sulfate affected the TOC removal by way of improving the treatment efficiency by several percent. The optimum dosages of iron oxide nanopowder were in the range of 0.02 to 0.04 g/dm³. As in the case of COD, the best TOC removal was achieved at the lowest ferrous sulfate dosages (0.05 – 0.1 g/dm³). Selected results are shown in **Figure 5**.

In summary, the oxidation degree obtained in the experimental conditions was high, reaching 71%. However, the solution probably still contained low-molecular products of oxidation such as formic and acetic acid, which were barely susceptible to the Fenton reagent [19]. The value of TOC removal was a few to several percent lower than the COD reduction degree, which confirms the thesis that organic products of dye oxidation remain in the solution.

The effect of hydrogen peroxide dosages

Besides the dosages of ferrous sulfate, the results of dye decomposition achieved in the presence of iron oxide nanopowder were affected also by the amount of hydrogen peroxide present in the system. **Table 1** presents results of decolourisation depending on the amount of iron oxide nanopowder added at different dosages of hydrogen peroxide at a constant amount of ferrous sulfate equal to 0.1 g/dm³. As follows from the data presented, irrespective of the amount of iron oxide nanopowder and hydrogen peroxide the

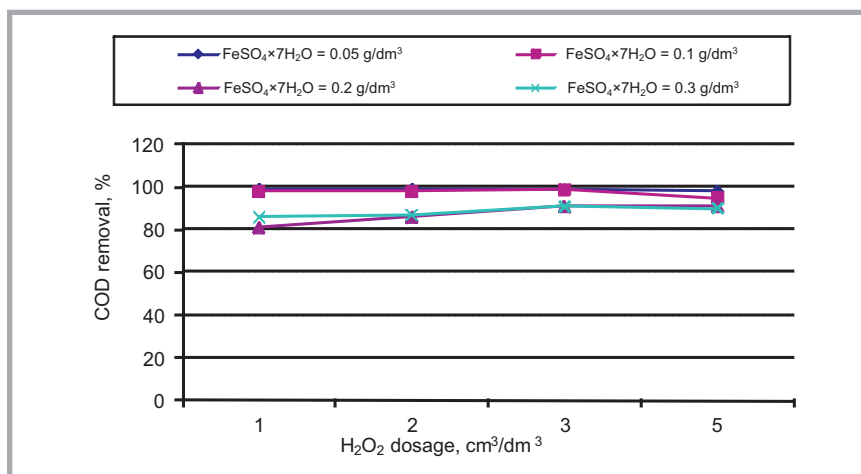


Figure 8. Effect of hydrogen peroxide dosage on colour removal in C.I. Direct Yellow 86 dye solution in the nanoFenton process at different ferrous sulfate dosages. Dosage of iron oxide nanopowder – 0,03 g/dm³, initial absorbance – 4.45 (wavelength 364 nm), pH 3.5.

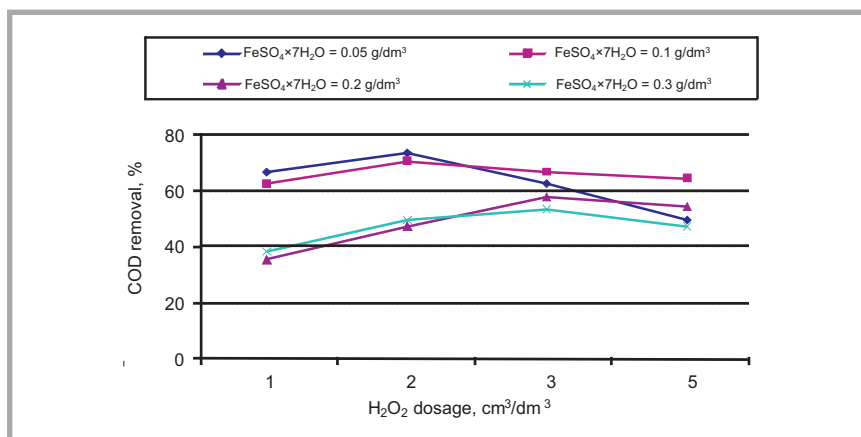


Figure 9. Effect of hydrogen peroxide dosage on COD removal in C.I. Direct Yellow 86 dye solution in the nanoFenton process at different ferrous sulfate dosages. Dosage of iron oxide nanopowder – 0.03 g/dm³, initial COD = 84 mg O₂/dm³, pH 3.5.

wastewater was very well decolorised; the colour removal was from 95 to 99%.

Figures 6 and **7** show changes in COD and TOC. It follows from the relations

presented that in the case of COD the optimum iron oxide nanopowder dosages were in the range from 0.03 to 0.04 g/dm³ irrespective of the amount of hydrogen peroxide added. TOC removal was the

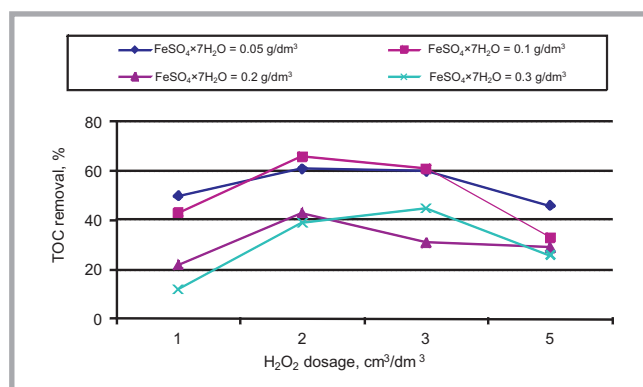


Figure 10. Effect of hydrogen peroxide dosages on TOC removal in C.I. Direct Yellow 86 dye solution in the nanoFenton process at different dosages of ferrous sulfate. Dosage of iron oxide nanopowder – 0.03 g/dm³, initial TOC = 27.9 mg/dm³, pH 3.5.

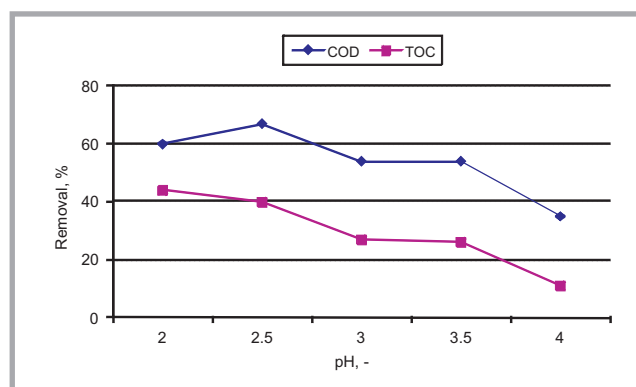


Figure 11. Effect of the pH on COD and TOC removal in C.I. Direct Yellow 86 dye solution in the nanoFenton process. Dosages: ferrous sulfate – 0.3 g/dm³, iron oxide nanopowder – 0.03 g/dm³, hydrogen peroxide – 3 cm³/dm³, initial COD = 84 mg O₂/dm³, TOC = 27.9 mg/dm³.

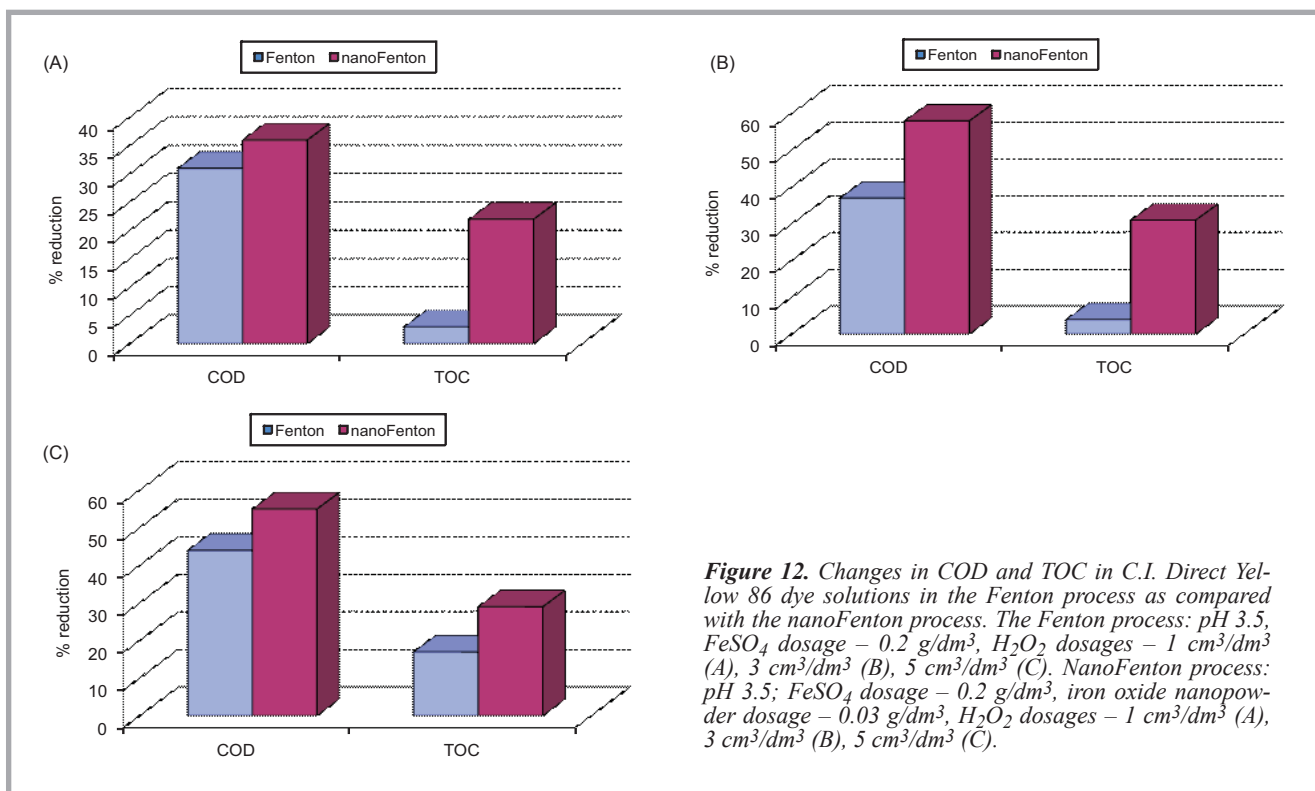


Figure 12. Changes in COD and TOC in C.I. Direct Yellow 86 dye solutions in the Fenton process as compared with the nanoFenton process. The Fenton process: pH 3.5, $FeSO_4$ dosage – 0.2 g/dm³, H_2O_2 dosages – 1 cm³/dm³ (A), 3 cm³/dm³ (B), 5 cm³/dm³ (C). NanoFenton process: pH 3.5; $FeSO_4$ dosage – 0.2 g/dm³, iron oxide nanopowder dosage – 0.03 g/dm³, H_2O_2 dosages – 1 cm³/dm³ (A), 3 cm³/dm³ (B), 5 cm³/dm³ (C).

highest for iron oxide nanopowder dosages of 0.03 - 0.04 g/dm³ at a hydrogen peroxide amount equal to 2 - 3 cm³/dm³. At H_2O_2 dosages of 1 and 5 cm³/dm³, the optimum amount of iron oxide nanopowder was 0.01 to 0.02 g/dm³. When these dosages were increased, the efficiency of dye decomposition decreased.

Figures 8, 9 and 10 (see page 117) show the effect of hydrogen peroxide dosages on the colour and on the COD and TOC removal at a constant iron oxide nanopowder dosage of 0.03 g/dm³ and different dosages of ferrous sulfate ranging from 0.05 to 0.3 g/dm³.

As follows from the data above, the values of COD removal achieved depended both on the amount of hydrogen peroxide introduced to the system and ferrous sulfate dosages. The best effects of COD removal were obtained for the smallest dosages of ferrous sulfate, i.e. 0.05 and 0.1 g/dm³ at the lowest dosages of hydrogen peroxide, ranging from 1 to 3 cm³/dm³. At ferrous sulfate dosages of 0.2 and 0.3 g/dm³ the COD removal was much lower. For instance, at a hydrogen peroxide dosage of 1 cm³/dm³ the removal of COD for a ferrous sulfate dosage of 0.05 - 0.1 g/dm³ was 63 - 67%, while at ferrous sulfate dosages of 0.2 - 0.3 g/dm³ it was only 36 - 39%.

Similar relations were obtained in the case of TOC. The highest values of TOC reduction were reached at the lowest dosages of hydrogen peroxide and ferrous sulfate applied. An increase in ferrous sulfate dosages caused a more significant decrease in TOC removal than in the case of COD. For instance, at a hydrogen peroxide dosage of 1 cm³/dm³ COD removal for a ferrous sulfate dosage of 0.05 g/dm³ was 67%, while at an amount of ferrous sulfate equal to 0.3 g/dm³ it was only 12%, i.e. four times less. Bigger amounts of ferrous sulfate clearly impeded the process of dye mineralisation. Moreover the TOC removal was smaller than the COD removal. Dye oxidation was more effective than its mineralisation. In the solution there were low-molecular organic compounds which had been oxidised to a large extent. The use of hydrogen peroxide dosages exceeding 3 cm³/dm³ was disadvantageous because a significant decrease in dye decomposition was reported.

The relationships presented above are concordant with data described in literature relating to the classical Fenton process [20]. It follows from the mechanism of the Fenton reaction that when too high dosages of hydrogen peroxide are used after exceeding a certain limit the reaction rate of oxidation does not increase; on the contrary – it starts to slow down.

A too high concentration of hydroxyl radicals contributes to their recombination. In addition, it is more probable that concurrent reactions not leading to dye oxidation will occur. As the research shows, a similar mechanism is observed in the Fenton process carried out in the presence of iron oxide nanopowder.

The effect of the pH of the solution

An important parameter affecting the Fenton process is the pH of the solution, which is why the effect of the pH on the reaction carried out with the use of iron oxide nanopowder was studied. The dosage of ferrous sulfate was 0.3 g/dm³, iron oxide nanopowder 0.03 g/dm³, and hydrogen peroxide 3 cm³/dm³. **Figure 11** (see page 117) shows changes in the removal of COD and TOC in the dye solution depending on the initial pH.

In the case of COD, at a pH increasing from 2 to 2.5, the removal of this parameter increased from 60 to 67%, and at a further increase of pH it dropped to 54% (pH range from 3 to 3.5). At a pH above 3.5, a further decrease in COD removal was observed. At pH 4 it was 40%. With an increasing pH, TOC removal decreased stepwise from 44% (pH 2) to 11% (pH 4). Mineralisation of the dye molecules was most efficient at pH from 2 to 2.5.

According to literature data, for the compounds most tested the optimum pH value in the classical Fenton process is about 3 [20]. The reduction in oxidation efficiency at higher pH values is due to the precipitation of part of the iron in the form of hydroxide. The use of too low pH values is not recommended either because $\cdot\text{OH}$ radicals can react with H^+ ions, which leads to a decrease in their concentration in the solution and a reduction in the oxidation efficiency of organic compounds.

Comparison of the classical Fenton reaction with the process carried out with the use of iron oxide nanopowder

An important issue is to compare the results of decomposition of C.I. Direct Yellow 86 dye obtained in the classical Fenton process with those achieved in the process carried out in the presence of nanoparticles of iron oxide. For these reasons experiments with dye decomposition in the classical Fenton process were performed using hydrogen peroxide dosages in the range from 1 to 5 cm^3/dm^3 and $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ from 0.05 to 0.3 g/dm^3 at pH 3.5.

Figure 12 shows changes in COD and TOC removal in C.I. Direct Yellow 86 dye solutions in the Fenton process as compared with the nanoFenton process. The Fenton process was carried out using a ferrous sulfate dosage of 0.2 g/dm^3 and hydrogen peroxide dosages of 1, 3 and 5 cm^3/dm^3 (pH 3.5). The nanoFenton process was carried out using the same process parameters as in the Fenton process and additionally iron oxide nanopowder in the amount of 0.03 g/dm^3 .

As follows from the data presented, in general the presence of iron oxide nanopowder had a beneficial effect on the Fenton process's efficiency. At the same dosages of ferrous sulfate and iron oxide nanopowder the removal of both COD and TOC depended on the amount of hydrogen peroxide introduced to the system. At an H_2O_2 dosage of 1 cm^3/dm^3 in the presence of iron oxide nanopowder, COD removal increased from 31 to 36, and TOC from 3 to 22%. At an H_2O_2 dosage equal to 3 cm^3/dm^3 , COD removal increased from 37 to 58, and TOC from 4 to 31%, while at an H_2O_2 dosage of 5 cm^3/dm^3 COD removal rose from 44 to 55, and TOC from 17 to 29%.

The rise in COD and TOC removal was the lowest at an H_2O_2 dosage of 1 cm^3/dm^3 , but grew significantly at a higher dosage of H_2O_2 (3 cm^3/dm^3). Next at a still higher dosage of H_2O_2 equal to 5 cm^3/dm^3 its slight decrease was reported. In the nanoFenton process a much higher increase in TOC removal was achieved as compared with the growth of COD removal. The iron oxide nanopowder catalysed the dye decomposition, contributing in this way to a significant increase in its mineralisation. The use of too big dosages of hydrogen peroxide after exceeding a certain limit leads to a decrease in the dye oxidation rate, which is in agreement with literature data for the decomposition of organic compounds in the Fenton process [20].

Conclusions

C.I. Direct Yellow 86 dye underwent satisfactory decomposition in the nanoFenton process. The solution decolorisation was complete and COD and TOC removal reached a maximum 74 and 71%, respectively. Iron oxide nanopowder introduced to the reaction system caused an increase in oxidation efficiency, which was revealed by the rising reduction in COD and, especially, TOC in relation to the classical Fenton process. Iron oxide nanopowder catalysed the dye oxidation, thus intensifying the process efficiency and dye mineralisation, which was illustrated by a multiple increase in TOC reduction in comparison with the classical process.

The results of dye decomposition obtained depended on the amounts of iron oxide nanopowder, ferrous sulfate and hydrogen peroxide as well as on the pH of the solution. It was advantageous to use smaller dosages of iron oxide nanopowder and ferrous sulfate at hydrogen peroxide dosage equal to 2 - 3 cm^3/dm^3 . The additional advantage of using smaller ferrous sulfate dosages was the reduction in the amount of iron II applied in the Fenton process, which resulted in the formation of smaller quantities of sludge while not lowering the process efficiency.

Due to the fact that little is known of the theoretical aspects of the nanoFenton process, it is planned to carry out a series of researches to determine the mechanism of the action of iron nanoparticles on the formation of hydroxyl radicals and their reactions. Determination of the

dye decomposition kinetics should make it possible to identify the mechanism of oxidation processes.



Acknowledgements

The study was financed by the Polish National Science Centre. Project no. N N523 751240.

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- crush tester (RCT, CMT, CCT, ECT, FCT), SCT, Taber and Lorentzen&Wettré (bending 2-point method) Lorentzen&Wettré (bending 4-point method and stiffness resonance method), Scott-Bond (internal bond strength), etc.,
- IGT (printing properties) and L&W Elrepho (optical properties), etc.,
- power-driven press, fall apparatus, incline plane tester, vibration table (specialized equipment for testing strength transport packages),
- atomic absorption spectrometer for the determination of trace element content, pH-meter, spectrophotometer UV-Vis.

Contact:

INSTITUTE OF BIOPOLYMERS AND CHEMICAL FIBRES
ul. M. Skłodowskiej-Curie 19/27, 90-570 Łódź, Poland
Elżbieta Baranek Dr eng. mech.,
tel. (+48 42) 638 03 31, e-mail: elabarunek@ibwch.lodz.pl