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Introduction

Textile wastewater is classified as difficult for treatment. This is due to a significant amount of wastewater produced, its diversified composition, big load of organic and mineral impurities, high toxicity and poor biodegradability. Characteristic features of textile wastewater are its intensive colour as well as high content of surfactants and other organic chemical compounds of different character and structure used in textile finishing processes. Hence, methods to ensure effective textile wastewater treatment are still searched for.

Methods used to decompose impurities in wastewater include oxidation/precipitation in the Fenton process [1 - 4], which is one of the simplest and most economical methods, consisting in nonselective and highly efficient oxidation of organic compounds by means of hydroxyl radicals formed in the chain reaction of hydrogen peroxide decomposition in the presence of bivalent iron salts. The mechanism of the process is represented by the following reaction:

 $\mathrm{H}_{2}\mathrm{O}_{2} + \mathrm{F}\mathrm{e}^{2+} \rightarrow \mathrm{F}\mathrm{e}^{3+} + \mathrm{O}\mathrm{H}^{-} + \mathrm{HO}^{\bullet}(1)$

The process is a radical reaction during which significant quantities of hydroxyl radicals HO[•] are formed. The high reactivity of hydroxyl radicals and their low selectivity enables oxidation of large groups of organic compounds present in textile wastewater, including dyes and detergents. At the same time it makes

Treatment of Textile Wastewater in the Fenton Process in the Presence of Iron and Nickel Nanocompounds

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Abstract

The aim of the studies was to determine the efficiency of wastewater treatment by the Fenton method in the presence of iron and nickel nanocompounds and to compare it with the classical Fenton method. The subject of the studies was textile wastewater generated during the dyeing of cellulose fibres. Wastewater colour and COD were determined in the samples before and after treatment using proper analytical methods. The Fenton process changed by the addition of iron and nickel nanocompounds was optimized taking into consideration the effect of the following parameters: doses of iron and nickel nanocompounds, the hydrogen peroxide dose and the pH of the solution on treatment efficiency. The decomposition of dyes was complete, with the reduction of COD reaching 57%. Optimum doses of iron and nickel oxide nanopowders were 0.02 g/dm³. It was found that the efficiency of pollutant decomposition in the processes of treatment in which iron and nickel oxide nanopowders were applied, was higher than in the classical method.

Key words: textile wastewater, Fenton process, iron nanocompounds, nickel nanocompounds.

the method applicable in wastewater of varying composition and content of pollutants, which is a characteristic feature of textile wastewater. Due to this, textile wastewater can be significantly decolourized and the main load of organic impurities can be removed. The basic advantages of this method include the high efficiency of the oxidation reaction, inexpensiveness and easily availability of substrates, and it is a simple procedure.

The efficiency of oxidation with Fenton's reagent is the highest at pH ranging from 2 to 5 and for an H_2O_2/Fe^{2+} molar ratio of about 1 : 1. The mechanism of this reagent was tested thoroughly for many reactive organic compounds and enzymatic reactions. However, it cannot be considered well explained because of the variety of iron(II) and iron(III) complexes,numerous radical intermediate products, and their consecutive reactions [5, 6]. An important role is also played by Fe³⁺ ions occurring in the process, which decompose H_2O_2 , producing hydroperoxide radicals HO_2 ':

 $Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + H^+ + HO_2^{\cdot}(2)$

Properties similar to those of iron ions in reactions (1) and (2) also have the ions of other metals, such as Cu, Co, Mn and Ti [7].

New trends in researches on pollutant oxidation processes by the Fenton method include the application of iron nanocompounds in the reaction system [8 - 12]. Their presence has an effect on the oxidizing reaction of chemical compounds present in wastewater. Iron nanocompounds exhibit catalytic activity which increases the effectiveness of oxidizing processes. Also other metal nanocompounds, including nickel nanocompounds, are active in supporting pollutant decomposition in the Fenton processes carried out with the use of iron nanocompounds [13, 14].

Ambashta and Sillanpaa [15] studied the decomposition of a mixture of organic compounds present in wastewater and found that the use of nickel nanocompounds increased the efficiency of treatment, in which the factors leading to pollutant decomposition were nanoiron, the magnetic field and ultrasounds. The use of a bimetallic mixture of ironnickel nanocompounds enabled the initiation and efficient course of the Fenton reaction.

Good results of decomposition with the use of the nanoNi/nanoFe system were also obtained in the case of azo dyes [16]. The degradation of mono-azo dye Orange G in water solutions was investigated. The mechanism of dye particle decomposition was explained. It was found that the degradation of dye was assisted by hydroxyl radicals formed in the system. A decisive role for initiating the reaction had the mutual surface interaction of Fe and Ni. As a result of the transfer of hydroxides from nickel to iron particles the $Fe^{+2}NiO$ system was formed. By interacting with water particles, it led to the generation of hydroxyl radicals. The hydroxyl radicals attacked dye particles, leading to their decomposition in the oxidation processes.

Using the bimetallic nanoFe/nanoNi system, the decomposition of trichloroethylene in aqueous solutions was also investigated [17]. Studies showed that nanoparticles quickly degraded the molecules of trichloroethylene and the main product of degradation was ethane. The rate constant of trichloroethylene decomposition in the Ni/Fe system was twice as high as when using nano-iron only, hence the use of nickel nanoparticles was very favourable from the point of view of TCE decomposition efficiency. Nickel nanoparticles clearly catalyzed the decomposition of TCE carried out with the use of nanoiron, which was confirmed by studies performed for various compositions of the bimetallic Ni/Fe system containing nickel and iron in different proportions in the bimetallic system. The best results of decomposition were obtained at a nickel content in the mixture ranging from 2% to 25% by weight.

A scientific aspect of the present study was to check how the addition of nickel nanocompounds could affect the efficiency of pollutant oxidation in textile wastewater during treatment by the Fenton method. As follows from the literature data, such research has not been carried out so far.

The aim of the studies was to determine the efficiency of wastewater treatment by the Fenton method in the presence of iron and nickel nanocompounds and to compare it with the classical Fenton method.

Materials and methods

Subject of studies

The tested material were three types of model textile wastewater made in laboratory in Textile Research Institute contained three kinds of surfactants generated during dyeing of cellulose fibres. The wastewater contained azo dyes, nonionic, anionic or cationic surfactant, acetic acid, sodium carbonate and sodium chloride. The wastewater had an intensive red colour, its COD ranged from 246 to 288 mg O_2/dm^3 and the initial pH was 10.6.

Experimental procedure

Analytically pure ferrous sulphate FeSO₄×7 H₂O and 30% hydrogen peroxide H₂O₂ (Chempur, Piekary Śląskie, Poland), iron (II, III) oxide nanopowder <50 nm and nickel (II) oxide nanopowder <50 nm (Sigma Aldrich, USA) as well as H₂SO₄ and analytically pure NaOH (POCh, Gliwice, Poland) were used in the experiments. pH values of the wastewater were reduced to 4, 3.5, 3, 2.5 and 2 by means of 5 N sulfuric acid solution. Next samples were completed either exclusively with ferrous sulphate or jointly with ferrous sulphate and iron (II, III) oxide nanopowder, ferrous sulphate and nickel (II) oxide nanopowder, ferrous sulphate and iron (II, III) oxide nanopowder with nickel (II) oxide nanopowder in asolid state, and the solution was stirred until complete dissolution. Then a 30% solution of hydrogen peroxide was added drop-wise to the wastewater. Once H₂O₂ had been added, the wastewater was stirred vigorously for 2 minutes, and then slowly for the next 10 minutes. The wastewater was left for 24 hours. After that the samples were neutralized with 10% solution of NaOH to a pH of about 11. After 24 hours, the wastewater was decanted and filtered.

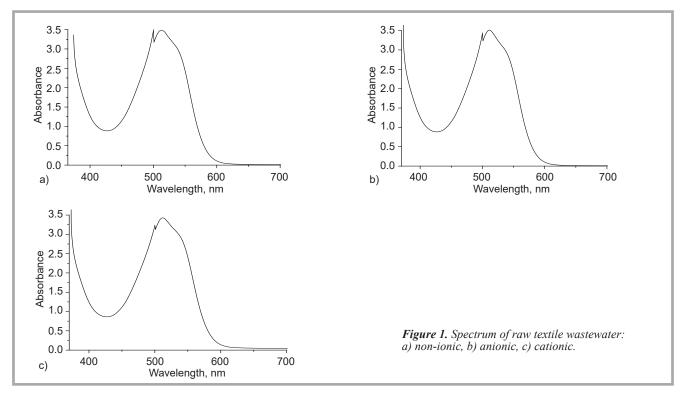
Wastewater quality control

Raw textile wastewater streams and wastewater after treatment were analyzed in terms of the following parameters:

colour,COD.

Analytical methods

The colour of wastewater was determined by the DFZ method. A spectral absorption coefficient (DFZ, German: *Durchsichtsfarbzahl*) was determined on the basis of absorbance measurements by



the spectrophotometric method at three wavelengths (= 436, 525 and 620 nm).

The COD was determined by the Hach-Lange test.

Results of the spectral analysis of raw textile wastewater streams are shown in *Figure 1*.

Since the absorbance of wastewater at a wavelength of 525 nm was much higher than at other wavelengths (see *Figure I*), this wavelength was recommended as the most representative in assessing the changes in wastewater colour.

Results and discussion

The Fenton process was optimized due to studies of the effect of the compound used in the treatment, doses of iron, nano-iron, nano-nickel, hydrogen peroxide and pH of the solution on the decolourization and efficiency of pollutant decomposition.

The effect of doses of iron and nickel oxide nanopowders

Experiments started with determination of the effect of doses of iron and nickel oxide nanopowders on the efficiency of pollutant decomposition in wastewater

Table 1. Changes in colour of textile wastewater: non-ionic (initial absorbance: 436 nm -0.923; 525 nm -3.281; 620 nm -0.033), anionic (initial absorbance: 436 nm -0.923; 525 nm -3.242; 620 nm -0.029) and cationic (initial absorbance: 436 nm -1.006; 525 nm -3.591; 620 nm -0.046) depending on the doses of iron and nickel oxide nanopowder; ferrous sulfate dose -0.5 g/dm³, hydrogen peroxide dose -5 cm³/dm³; pH of the solution = 3.5.

	non-ionic			anionic			cationic			
Parameters of treatment	iron (II.III) oxide nanopowder dose, g/dm ³ nickel (II) oxide nanopowder dose, g/dm ³									
	0.03 0.01	0.02 0.02	0.01 0.03	0.03 0.01	0.02 0.02	0.01 0.03	0.03 0.01	0.02 0.02	0.01 0.03	
colour reduction, %										
436 nm	95.7	89.1	97.0	96.5	96.6	96.6	95.1	93.3	94.1	
525 nm	98.9	99.3	99.3	99.2	99.2	99.3	98.8	98.3	98.6	
620 nm	89.2	93.1	95.8	100	100	100	95.5	91.0	92 5	

in the Fenton process. The wastewater was treated with the use of a total dose of nanocompounds equal to 0.04 g/dm^3 and changing proportions of the iron oxide nanopowder and nickel oxide nanopowder at constant doses of ferrous sulphate (0.5 g/dm³) and hydrogen peroxide (5 cm³/dm³). Experiments were carried out at pH=3.5. The wastewater decolourization is illustrated in *Table 1* and *Figure 2*.

As a result of treatment the nonionic wastewater was very well decolourised. Colour reduction was the highest at wavelength 525 nm and ranged from 98.9 to 99.3%; it was smaller at wavelength 620 nm and ranged from 89.2 to 95.8%, and at wavelength 436 nm itranged from

89.1 to 97.0%. The highest degree of colour reduction was obtained at a dose of iron oxide nanopowder of 0.01 g/dm³ and nickel oxide nanopowder of 0.03 g/dm³. The degree of COD reduction, depending on the doses of iron oxide and nickel oxide nanopowders, ranged from 51.6 to 57.2%. The highest degree of COD reduction was obtained at a dose of iron oxide nanopowder of 0.02 g/dm³ and nickel oxide nanopowder of 0.02 g/dm³.

In the anionic wastewater after treatment, colour reduction at wavelengths 436 nm and 525 nm was from 96.5 to 96.6% and from 99.2 to 99.3%, respectively. Hence, the wastewater was almost completely decolourized irrespective of the doses of

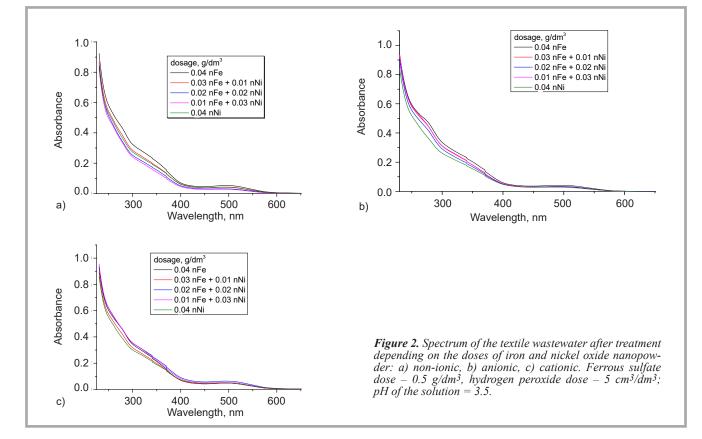


Table 2. Changes in COD of the textile wastewater: non-ionic (initial $COD = 283 \text{ mg } O_2/dm^3$), anionic (initial $COD = 288 \text{ mg } O_2/dm^3$) and cationic (initial $COD = 246 \text{ mg } O_2/dm^3$) depending on the doses of iron and nickel oxide nanopowder.

Parameters of treatment	non-ionic			anionic			cationic			
	iron (II.III) oxide nanopowder dose, g/dm ³ nickel (II) oxide nanopowder dose, g/dm ³									
	0.03 0.01	0.02 0.02	0.01 0.03	0.03 0.01	0.02 0.02	0.01 0.03	0.03 0.01	0.02 0.02	0.01 0.03	
COD, mg O ₂ /dm ³	137	121	133	144	144	136	144	124	154	
COD reduction, %	51.6	57.2	53.0	50.0	50.0	52.8	41.5	49.6	37.4	

nickel and iron oxide nanopowders used. In the case of COD, while increasing the dose of nickel oxide nanopowder in relation to the amount of iron oxide nanopowder used, the degree of COD reduction increased from approximately 50 to 53%. However, differences in the levels of COD reduction were not significant.

As a result of the treatment, the cationic wastewater was very well decolourized. Irrespective of the doses of iron and nickel oxide nanopowders applied, the reduction of colour was very high and at wavelengths 436 nm, 525 nm and 620 nm it ranged from 93.3 to 95.1%, from 98.3 to 98.8% and from 91 to 95.5%, respectively. Depending on the doses of iron and nickel oxide nanopowders, the de-

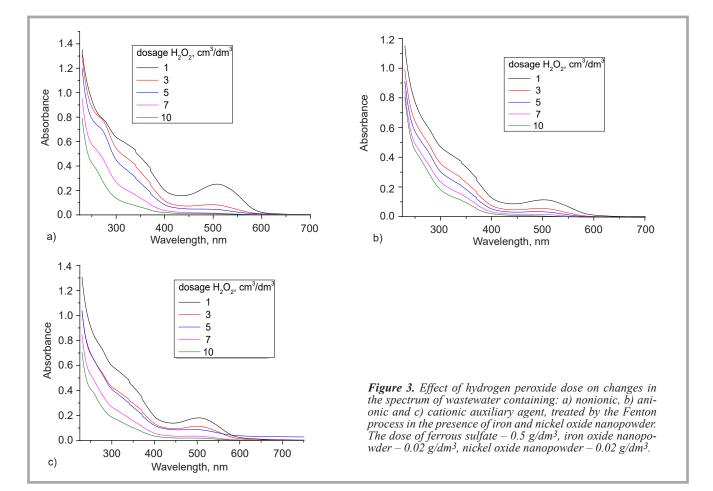
gree of COD reduction was from 37.4 to 49.6%. The highest reduction of COD was obtained when the doses of iron and nickel oxide nanopowders were equal to 0.02 g/dm^3 .

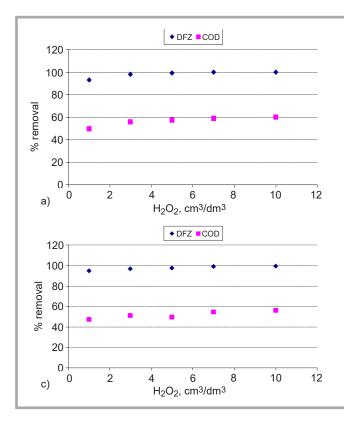
Therefore to sum up, irrespective of the proportions of the amounts of iron oxide nanopowder and nickel oxide nanopowder added and the ionic character, the wastewater streams were very well decolourized, with colour reduction reaching from 98.3 to 99.3% (at wavelengths 525 nm). This resulted probably from the fact that at the treatment parameters used, the decolourization degrees obtained were high in general. The COD reduction is illustrated in *Ta-ble 2*.

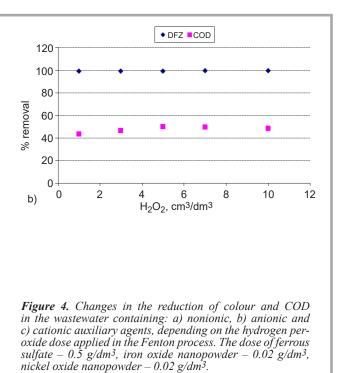
In the anionic wastewater the COD reductionincreased with an increase innickel oxide nanopowder in the total dose. In the cationic and nonionic wastewater the COD reduction was the best with the nickel nanopowder dose of 0.02 g/dm³. It seems that nickel nanocompounds catalyzed the process of pollutant decomposition in the Fenton process better than iron nanocompounds. The decomposition of pollutants was the best in nonionic wastewater (the maximum COD reduction reached 57.2%), while in cationic wastewater it was the least (the maximum COD reduction was 49.6%).

The effect of hydrogen peroxide dose

The next series of experiments covered determination of the effect of hydrogen peroxide concentration on the decomposition of impurities in textile wastewater. From 1 to 10 cm³/dm³ of hydrogen peroxide was added to the wastewater. The dose of iron oxide nanopowder appled was 0.02 g/dm³, the that of nickel oxide nanopowder - 0.02 g/dm³ and ferrous sulphate 0.5 g/dm³, and the pH of







the solution -3.5. *Figures 3* and *4* show the experimental results.

In nonionic wastewater with an increase in the hydrogen peroxide dose there was an increase in pollutant decomposition. The degree of wastewater decolourization increased from 92.9 to 99.8%, and as a consequence the wastewater at the highest doses of H2O2 was almost completely decolourized. The dose of hydrogen peroxide had an even more pronounced effect on the increase in COD reduction, increasing from 49.5% at the smallest applied dose of H₂O₂ (1 cm³/dm³) to 59.7% at the biggest dose ($10 \text{ cm}^3/\text{dm}^3$). The use of hydrogen peroxide doses exceeding 7 cm3/dm3 did not increase the COD reduction significantly.

In the wastewater containing anionic agents in all samples the wastewater decolourization was nearly 100%. Hence the wastewater was completely decolourized. The reduction of COD increased from 43% at the smallest applied dose of H_2O_2 (1 cm³/dm³) to 50% at the dose of 5 cm³/dm³. At bigger doses of hydrogen peroxide the COD reduction decreased, and at the dose of 10 cm³/dm³ it was 48%. The use of hydrogen peroxide doses bigger than 5 cm³/dm³ was therefore aimless.

In the case of wastewater containing cationic agents with a bigger dose of hydrogen peroxide the pollutant decomposition increased. Decolourization of the wastewater increased from 94.9 to 99.5%, and as a consequence at the biggest H_2O_2 doses the wastewater was almost totally decolourized. The effect of the hydrogen peroxide dose on COD reduction was even more pronounced, increasing from 47% at the smallest H_2O_2 dose (1 cm³/dm³) to 56% at the biggest dose applied (10 cm³/dm³).

The resulting relations are consistent with the data described in the literature with respect to the classical Fenton process [18]. The mechanism of Fenton reaction shows that while using too high doses of hydrogen peroxide beyond a certain limit, the oxidation reaction rate does not increase, but decreases. A too high concentration of hydroxyl radicals favours their recombination. Additionally the likelihood of competitive reactions which do not lead to the oxidation of pollutants increases. As can be seen from the results, a similar mechanism occurs in the Fenton process carried out in the presence of iron and nickel oxide nanopowder.

The effect of pH

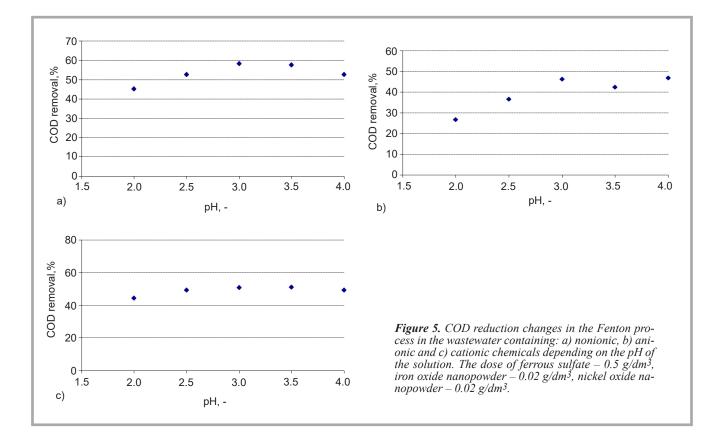
An important parameter affecting the Fenton process is the pH of the solution.

Therefore the effect of pH on the reaction carried out using iron and nickel oxide nanopowders was tested. The dose of ferrous sulphate was 0.5 g/dm³, iron oxide nanopowder 0.02 g/dm³ and hydrogen peroxide 5 cm³/dm³. *Figure 5* shows the results of changes in COD reduction in the wastewater depending on the initial pH.

In the case of wastewater containing nonionic auxiliary agents, with an increase in pH in the range from pH 2 to pH 3 the reduction of COD increased from 45.2% to 58.3%. The optimum pH was 3.0 because its further increase caused a decrease in COD reduction to 52.7% at pH 4.

In the case of wastewater containing anionic chemicals, with an increase pH in the range from pH 2 to pH 3 the reduction of COD increased from 27% to 46%. At a further increase in the pH of the wastewater to pH 4 the reduction of COD remained at a similar level.

In the case of wastewater containing cationic chemicals, with an increase in pH in the range from pH 2 to pH 3.5 the reduction of COD grew gradually from 44.3 to 51.2%. Any further increase in the pH of the wastewater resulted in a decrease in COD reduction.



The data obtained are in accordance with literature data for the classical Fenton process. For most of the compounds tested the optimum pH value in the classical Fenton process is about 3 [18]. A decrease in the oxidation efficiency at higher pH values is caused by the precipitation of iron in the form of hydroxide. It is not recommended either to use too low pH values since 'OH radicals can react with H⁺ ions, which leads to a decrease in their concentration in the solution and reduction of the oxidation efficiency of organic compounds.

Comparison of the classical Fenton process with that carried out with the use of iron and nickel oxide nanopowders

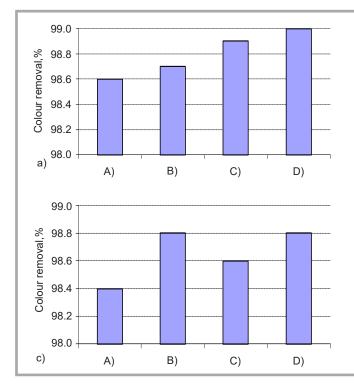
In the next stage of studies the effects of textile wastewater treatment obtained in the classical Fenton process were compared with the results obtained in the process carried out in the presence of iron and nickel nanocompounds. *Figures 6* and 7 show results of the investigations. As can be seen from these data, wastewater decolourization and COD reduction depended on the wastewater type, the amount of iron and nickel oxide nanopowders applied and also on the treatment variant.

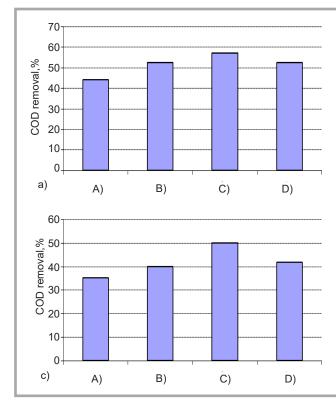
In the case of wastewater containing nonionic agents the highest decolourization was obtained in the Fenton process carried out in the presence of nickel oxide nanopowder, and the lowest one in the classical Fenton process. However, differences in the degree of decolourization, in particular the variants of treatment, were not big. More pronounced differences in reduction degrees, in particular variants of treatment, were observed in the case of COD. The highest COD reduction of 57.2% was obtained while using both iron and nickel oxide nanopowders. This was much bigger than in the case of other treatment variants in which the COD reduction ranged from 44.2% (the classical Fenton process) to 52.7%.

In the case of wastewater containing anionic substances the highest decolourization of 99.2% was obtained in the Fenton process carried out in the presence of iron and nickel oxide nanopowders;however, the difference was very small as compared to other variants of treatment. The wastewater was better decolourized than the one containing nonionic agents. Clearer differences in the reduction in particular variants of treatment were reported in the case of COD. The highest reduction of COD was achieved in the Fenton process in the presence of nickel oxide nanopowder, which was about 56% against 45.1% in the classical Fenton process, ranging from 50 to 51.4% in other variants of treatment.

In the case of wastewater containing cationic substances the decolourization degree ranged from 98.4 to 98.8%, with the highest one being in the variant of treatment which involved the use of nickel oxide nanopowder in the Fenton process. The COD reduction ranged from 35.4% (the classical Fenton process) to 49.6% (the nanoFenton process carried out in the presence of nickel oxide nanopowder).

It follows from the data obtained that iron and nickel nanocompounds catalyzed the Fenton reaction, increasing the efficiency of pollutant decomposition in textile wastewater as compared with the classical process. At the same dose of ferrous sulphate the reduction of colour and COD depended on the presence and amount of iron and nickel nanocompounds. In general, the best results for the reduction of colour and COD were obtained with the use of only nickel nanocompounds and with simultaneous use of iron and nickel nanocompounds. Worse results were obtained while using only iron nanocompounds, and the worst in the classical Fenton process.





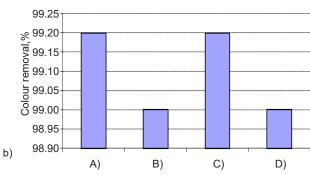


Figure 6. Colour reduction in wastewater (wavelength 525 nm) containing a) nonionic, b) anionic and c) cationic chemicals depending on the variant of treatment.

- A) Fenton; doses: H₂O₂ 5 cm³/dm³, ferrous sulfate -0.5 g/dm³
- B) nanoFenton; doses: H₂O₂ 5 cm³/dm³, ferrous sulfate 0.5 g/dm³, iron oxide nanopowder 0.04 g/dm³,
 C) nanoFenton + nanoNi; doses: H₂O₂ 5 cm³/dm³, ferrous
- C) nanoFenton + nanoNi; doses: H₂O₂ 5 cm³/dm³, ferrous sulfate - 0.5 g/dm³, iron oxide nanopowder - 0.02 g/dm³, nickel oxide nanopowder - 0.02 g/dm³,
- D) Fenton + nanoNi; doses: H₂O₂ 5 cm³/dm³, ferrous sulfate 0.5 g/dm³, nickel oxide nanopowder 0.04 g/dm³

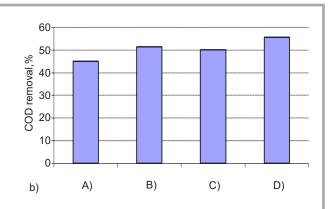


Figure 7. COD reduction in wastewater containing: a) nonionic, b) anionic and c) cationic chemicals depending on the variant of treatment.

- A) Fenton; doses: H₂O₂ 5 cm³/dm³, ferrous sulfate -0.5 g/dm³
- B) nanoFenton; doses: H₂O₂ 5 cm³/dm³, ferrous sulfate -0.5 g/dm³, iron oxide nanopowder - 0.04 g/dm³,
- C) nanoFenton + nanoNi; doses: H₂O₂ 5 cm³/dm³, ferrous sulfate 0.5 g/dm³, iron oxide nanopowder 0.02 g/dm³, nickel oxide nanopowder 0.02 g/dm³,
- D) Fenton + nanoNi; doses: H₂O₂ 5 cm³/dm³, ferrous sulfate 0.5 g/dm³, nickel oxide nanopowder 0.04 g/dm³

Conclusions

The use of iron and nickel oxide nanopowder in the Fenton process resulted in a better decomposition of pollutants in textile wastewater than with the classical Fenton process. At the same time a high level of wastewater decolourization was maintained. The results of treatment depended on the ionic character of wastewater, proportions of the amount of iron and nickel nanocompounds, the doses of reagents, including hydrogen peroxide, and on the pH of the solution.

Iron and nickel oxide nanopowders catalyzed the process of pollutant decomposition, improving its efficiency and increasing the mineralization of pollutants. The decomposition of dyes was complete,with the reduction of COD reaching 57%. The use of iron and nickel oxide nanopowders in the reaction system caused an increase in oxidation efficiency by several percent as compared to the classical Fenton process. The efficiency of pollutant decomposition in the wastewater treated in the Fenton process in the presence of nickel oxide nanopowder depended on the amount of hydrogen peroxide added to the system. An increase in hydrogen peroxide dose ranging from 1 to $10 \text{ cm}^3/\text{dm}^3$ resulted in an increase in COD reduction up to 10%. The oxidation process was most efficient at a pH from 3.0 to 3.5. The optimum doses of iron and nickel oxide nanopowders were 0.02 g/dm^3 .

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- there are headquarters of scientific research institutes
 It has intellectual and production potential for the textile industry (Lodz University of Technology, Institute of Biopolymers and Chemical Fibers, Textile Research Institute, Institute of Security Technologies MORATEX, Polish Textile Association, Institute of Leather Industry)

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