

# Comparison of Chemical and Physical-chemical Wastewater Discoloring Methods

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Today's chemical and physical-chemical wastewater discoloration methods do not completely meet demands regarding degree of discoloration. In this paper discoloration was performed using Fenton ( $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O} + \text{H}_2\text{O}_2 + \text{H}_2\text{SO}_4$ ) and Fenton-like ( $\text{FeCl}_3 \cdot 6 \text{H}_2\text{O} + \text{H}_2\text{O}_2 + \text{HCOOH}$ ) chemical methods and physical-chemical method of coagulation/flocculation (using poly-electrolyte (POEL) combining anion active coagulant (modified poly-acrylamides) and cationic flocculant (product of nitrogen compounds) in combination with adsorption on activated carbon. Suitability of aforementioned methods was investigated on reactive and acid dyes, regarding their most common use in the textile industry. Also, investigations on dyes of different chromogen (anthraquinone, phthalocyanine, azo and xanthene) were carried out in order to determine the importance of molecular spatial structure. Oxidative effect of Fenton and Fenton-like reagents resulted in decomposition of colored chromogen and high degree of discoloration. However, the problem is the inability of adding POEL in stoichiometrical ratio (also present in physical-chemical methods), when the phenomenon of overdosing coagulants occurs in order to obtain a higher degree of discoloration, creating a potential danger of burdening water with POEL. Input and output water quality was controlled through spectrophotometric measurements and standard biological parameters. In addition, part of the investigations concerned industrial wastewaters obtained from dyeing cotton materials using reactive dye (C. I. Reactive Blue 19), a process that demands the use of vast amounts of electrolytes. Also, investigations of industrial wastewaters was labeled as a crucial step carried out in order to avoid serious misassumptions and false conclusions, which may arise if dyeing processes are only simulated in the laboratory.

Key words: *Discoloration, reactive dyes, Fenton reagent, coagulation/flocculation, poly-electrolyte, water quality*

## Introduction

The 21<sup>st</sup> century has been labeled the century of environmental preservation, emphasizing the importance of water and its role in the global ecological system. Aquatic eco-systems are substantially endangered by industrial wastewaters. Used chemical reagents, usually significantly diverse in chemical composition, range from inorganic compounds, polymers to organic products. The presence of low concentrations of dyes in effluents causes brilliantly colored wastewaters, which indirectly affects aquatic life. Most of the dyes used today are difficult to discolor due to their complex chemical structure and synthetic origin. Structurally different dyes can be sorted into either cationic, anionic or non-ionic groups of dyestuff. Besides dyestuff, there is a wide variety of auxiliaries such as salts, enzymes, surfactants, scouring agents, oils, oxidizing and reducing agents, which after being used in textile wet processes eventually produce harmful contaminants.<sup>1–5</sup>

Accordingly, processes of wastewater purification and recycling gain more importance each day. Literature divides wastewater purifying into two main categories. The first category refers to a survey of procedures and processes of wastewater purifying. Each of the procedures within the category is to produce a higher degree of water purity, removing undesirable substances and making water suitable for recycling or its release into watercourses. Primary purifying procedures consider removing unsettled matter by the processes of settling, emerging and percolation on micro-screens. Secondary purifying procedures consider various biological, chemical and physical-chemical processes, which remove the biodegradable matter. Tertiary purifying procedures, often called "advanced technology", refer to removal of nitrogen, phosphorus, heavy metals and dissolved inorganic matter. The second main category determines the goals of certain purifying degrees in order to distinguish more accurately, what is expected out of each purifying degree.<sup>1–5</sup>

This paper compares certain chemical and physical-chemical wastewater discoloring procedures, showing both their advantages and disadvantages. Further elaboration suggests

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solution of the problem by combining certain methods in order to improve the purity degree. The paper presents the problems which may occur when Fenton and Fenton-like chemical methods are used, referring mainly to the inability of calculating accurately their amount, which leads to undesirable residual amounts of ferric ions. The paper also presents purification by coagulation/flocculation (using poly-electrolyte POEL), which combines anion active coagulant and cationic flocculant. The result is a high discoloration degree, often achieved by overdosing coagulants and unwanted wastewaters burdening with POEL. Solution of the problem presents itself in an additional purification process of adsorption on activated carbon.<sup>6–15</sup>

Furthermore, it was necessary to carry out investigations considering different chemical constitutions referring to associated chromophore, as follows: azo, anthraquinone, xanthene and phthalocyanine.<sup>2–9, 11–12</sup>

The obtained results are represented through spectrophotometric measurements with calculated discoloration degrees as well as measurements of standard biological quantities, such as pH, electrical conductivity, BOD<sub>5</sub>/COD and TOC. Also, the amount of residual ferric ions for Fenton-like purifying process was measured.

## Methodology

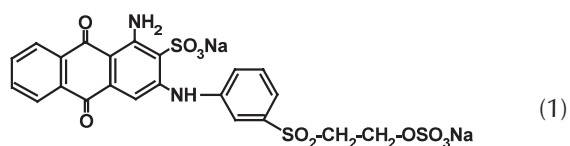
Suitability of selected chemical and physical-chemical methods has been investigated for dyestuff of different chemical constitution. Elaborated purifying methods were, as follows: Fenton and Fenton-like processes as the representatives of chemical discoloration methods and coagulation followed by the flocculation discoloration method as the representative of physical-chemical methods.

Purifying of simulated dye-baths was done as a preliminary investigation step, which was followed by physical and physical-chemical discoloration of industrial dye-bath obtained under real dyeing conditions.

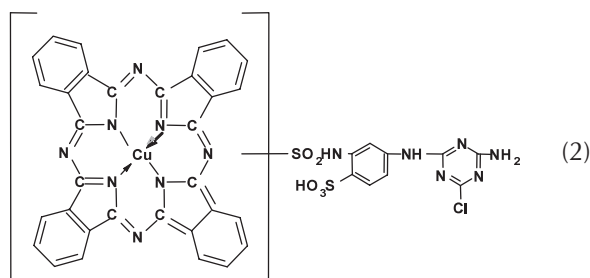
All spectrophotometric values were obtained by measurements carried out on Varian's Cary 50 spectrophotometer, while standard biological quantities were measured on Macherey-Nagel's Nanocolor<sup>®</sup> 500 D spectrophotometer. Electrical conductivity was measured on Metrel's MA 5950 conductivity meter and pH on Metrel's MA 5736 pH meter.

## Dyestuff

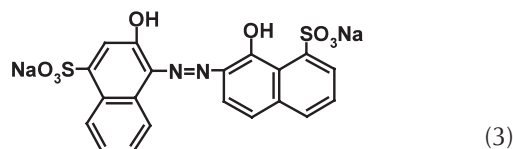
Investigations were performed on commercial dyestuff of different chemical constitution, shown structural (1) through (4).



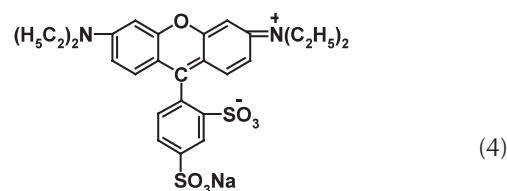
C.I. Reactive Blue 19 – anthraquinone (antrakinon)



C.I. Reactive Blue 116 – phthalocyanine (ftalocijanin)



C.I. Acid Red 158 – azo (azo) (3)



C.I. Acid Red 52 – xanthene (ksanten)

## Methods used

Simulated dye-bath water solutions (for each investigated dyestuff) of  $\gamma = 1 \text{ g l}^{-1}$  mass concentration were discolored by preliminary optimized chemical and physical-chemical methods.

### Chemical discoloration methods

- Fenton ( $\text{FeSO}_4 \cdot 7 \text{ H}_2\text{O} + \text{H}_2\text{O}_2 + \text{H}_2\text{SO}_4$ )
- Fenton-like ( $\text{FeCl}_3 \cdot 6 \text{ H}_2\text{O} + \text{H}_2\text{O}_2 + \text{HCOOH}$ )

Purifying conditions were set, as follows: pH 3 set by an appropriate acid ( $\tau = 0.05 \text{ ml l}^{-1}$  of sulfuric or  $\tau = 0.2 \text{ ml l}^{-1}$  formic), succeeded by the addition of  $\tau = 50 \text{ ml l}^{-1}$  of  $\text{H}_2\text{O}_2$  ( $\varphi = 36\%$ ) and  $\gamma = 5 \text{ g l}^{-1}$  of ferric salt. Colored wastewater was then mixed using magnetic mixer at 700 rpm for 15 minutes of reaction time succeeded by the addition of NaOH ( $\varphi = 32\%$ ) setting pH to 8.

All of the processes were carried out with and without ultrasound at temperatures of  $\theta = 25$  and  $60 \text{ }^\circ\text{C}$ . Ultrasound treatment was performed with an ultrasound device Sonics & Materials VibraCell VCX 750 using a constant  $\rho = 20 \text{ kHz}$  frequency of variable amplitude and maximum power output  $P = 750 \text{ W}$  in continuous operation mode.

The discoloration degree for selected dyestuffs was calculated based on obtained spectrophotometric values, the results of which are shown in Fig. 1. The amount of residual ferric ions for C. I. Reactive Blue 19 dyestuff (concentration of 1 and  $\gamma = 2 \text{ g l}^{-1}$ ) was spectrophotometrically determined for the Fenton-like method, with results shown in Fig.

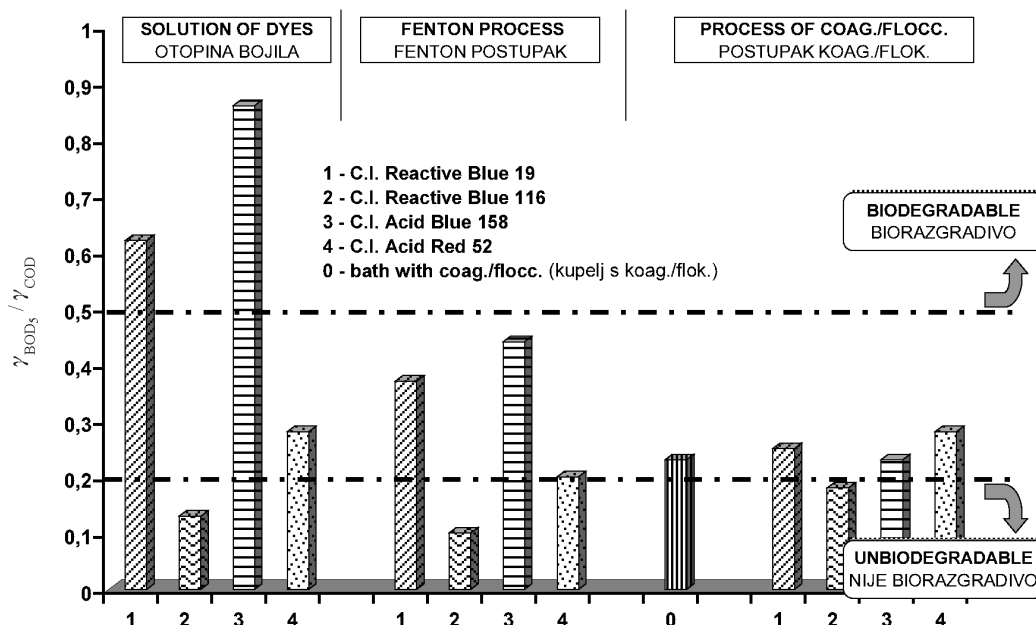


Fig. 1 – Discoloration degree after Fenton treatment and after coagulation/flocculation treatment  
 Slika 1 – Stupanj obezbojavanja nakon obrade Fentonom i nakon postupka koagulacije/flokulacije

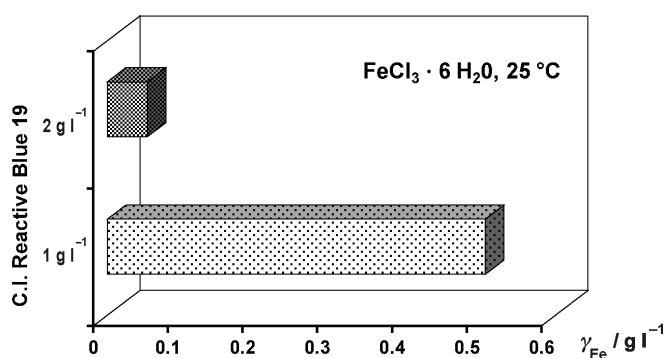
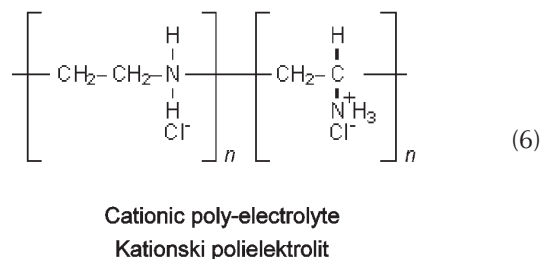


Fig. 2 – Quantity of residual ferric ions for C. I. Reactive Blue 19 dyestuff concentrations of 1 g l<sup>-1</sup> and 2 g l<sup>-1</sup>  
 Slika 2 – Količina zaostalih željezovih(III) iona za bojilo C. I. Reactive Blue 19 u koncentraciji 1 g l<sup>-1</sup> i 2 g l<sup>-1</sup>



Discoloration was performed using the preliminary optimized method, which was to produce an easily removable firm floccule.<sup>6-8</sup> As previously mentioned, the pH of solutions of selected dyestuff was set to 7. Method optimization implies jar test mixing, neutralization of wastewater and varying mixing speeds, from  $n = 100$  rpm (30 min) in the first phase (addition of  $\gamma = 100\ g\ l^{-1}$  of 5% anionic coagulant) to  $n = 50$  rpm (10 min) in the second phase (addition of  $\tau = 7.5\ ml\ l^{-1}$  cationic flocculant). In order to completely eliminate turbidity after the removal of firm floccules, an additional purifying step of adsorption on activated carbon had to be performed. This enabled spectrophotometric determination of discoloration degree, the results of which are shown in Fig. 1, while biodegradability results are shown in Fig. 3.

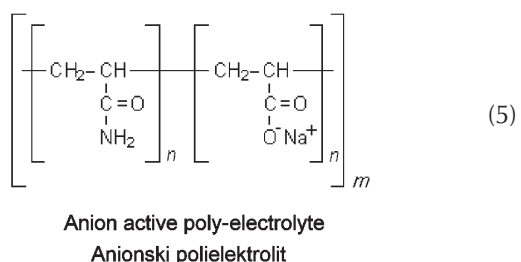
**Discoloration of industrial dye-bath wastewater**

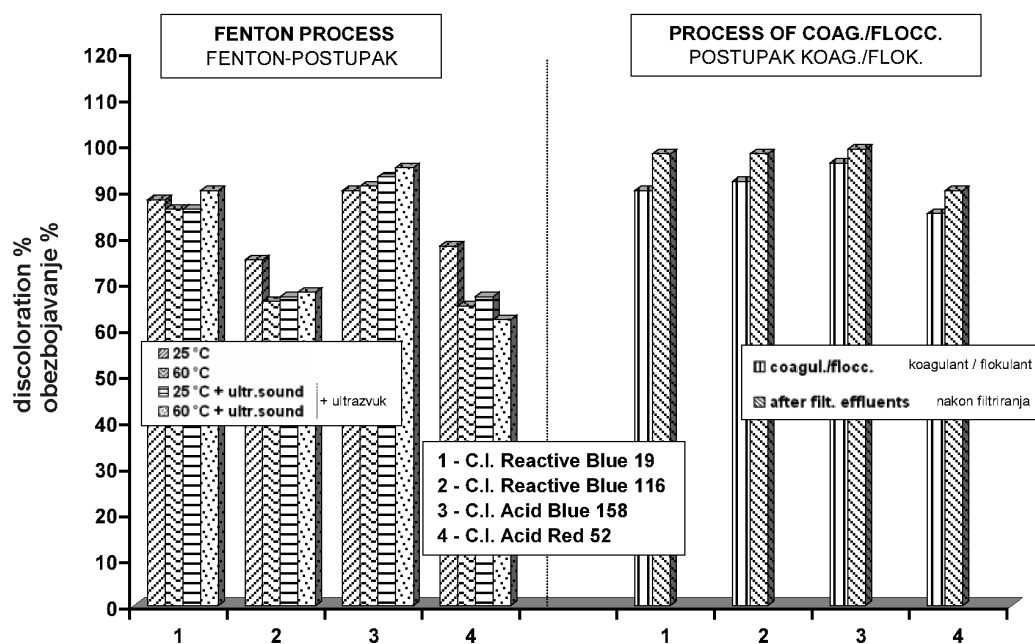
After the investigations and analyses performed on simulated dye-bath wastewaters, dyeing under real industrial conditions had to be carried out. Cotton material was dyed using 3% C.I. Reactive Blue 19 dye-bath with the addition of  $\gamma = 60\ g\ l^{-1}$  NaCl,  $5\ g\ l^{-1}$  Na<sub>2</sub>CO<sub>3</sub> and  $\tau = 2\ ml\ l^{-1}$  NaOH  $\varphi = 32\ \%$ . After-treatment processes included material rinsing and neutralization of obtained wastewaters. Discoloration methods were Fenton-like (FeCl<sub>3</sub> · 6H<sub>2</sub>O + H<sub>2</sub>O<sub>2</sub> + HCOOH process) and coagulation/flocculation followed by adsorption on activated carbon.

2. Biodegradability was established based on BOD<sub>5</sub>/COD values shown in Fig. 3.

*Physical-chemical methods*

- coagulation/flocculation (using poly-electrolyte (POEL) combining anion active coagulant (modified poly-acrylamides), eq. 5, and cationic flocculant (product of nitrogen compounds, eq. 6));
- adsorption on activated carbon.<sup>8, 9</sup>



Fig. 3 – Biodegradability of dyes: BOD<sub>5</sub>/COD valuesSlika 3 – biorazgradivost bojila: vrijednost BPK<sub>5</sub>/KPK

### Water quality analyses

Analyses of wastewaters included measurements of pH, electrical conductivity, discoloration degree, TOC and BOD<sub>5</sub>/COD. Obtained results are shown in Table 1.

### Conductivity measurement

Conductivity ( $\kappa$  (mS cm<sup>-1</sup>)) as an ecological control parameter was measured after each process phase (conduct meter MA 5950, Metrel). In addition, before dyeing, conductivity of different NaCl concentrations was measured and func-

tional dependence between conductivity and electrolyte concentration,  $\kappa = f(c)$ , was defined (Fig. 4). The range of measured electrolyte referred to the amount used in practical use in reactive dyeing. The letter  $f$  symbolizes the law of correspondence between the independent variable and the function, and in the case of an analytical representation of a function it also indicates a mathematical operation which should be performed on "x" to obtain "y", according to correlation index  $R^2 = 0.9983$ . The NaCl concentration for dyeing from recycling bath was calculated using a mathematical model derived from the performed measuring  $\gamma_{\text{NaCl}} = f(\kappa)$ .

Table 1 – Ecological quantities of simulated dye-bath wastewater obtained before and after purifying processes (3 % C. I. Reactive Blue 19)

Tabela 1 – Ekološka veličina simulirane otpadne vode bojodisaone dobivene prije i nakon procesa pročišćavanja (3 % C. I. Reactive Blue 19)

Bath Kupelj	pH	$\kappa$ (mS cm <sup>-1</sup> )	Discoloration degree Stupanj obezbojavanja (%)	TOC	BOD <sub>5</sub> /COD BPK <sub>5</sub> /KPK
Industrial dye-bath Industrijska kupelj za bojadisanje	10.30	87.9	–	800	0.64
After rinsing and neutralization Nakon ispiranja i neutralizacije	7.82	16.0	–	250	0.52
Chemical method Fenton-like Modificirani Fenton-postupak Kemijska metoda (FeCl <sub>3</sub> · 6 H <sub>2</sub> O + H <sub>2</sub> O <sub>2</sub> + HCOOH), 25 °C	7.12	16.1	88	70	0.37
Physical-chemical method coagulation/flocculation koagulacija/flokulacija Fizikalno-kemijska metoda	7.42	15.8	90	150	0.26
adsorption on activated carbon adsorpcija na aktivnom ugljenu	7.30	13.3	98	100	0.03



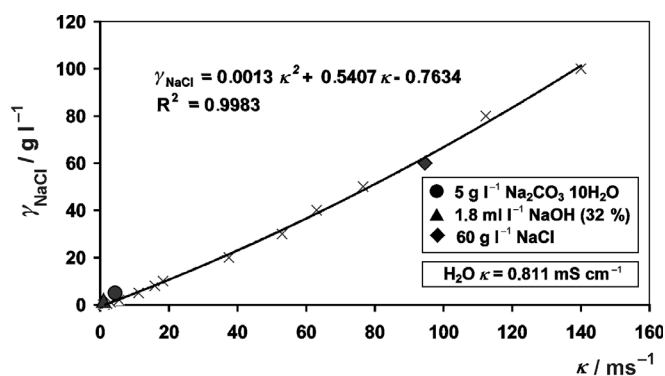
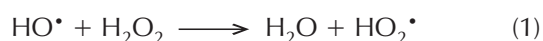


Fig. 4 – Functional dependence of electrical conductivity on electrolyte concentration ( $\vartheta = 25\text{ }^{\circ}\text{C}$ )

Slika 4 – Funkcijska ovisnost vodljivosti o koncentraciji elektrolita ( $\vartheta = 25\text{ }^{\circ}\text{C}$ )

## Results and discussion

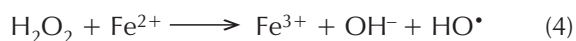
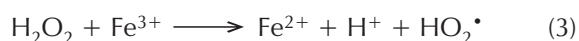
In the field of discoloration and wastewater purifying there are many scientific methods, which are more or less appropriate for application under real conditions. This paper aims to compare the most frequently used, easily applicable methods and to review their advantages and disadvantages. One of the most popular methods of wastewater discoloration is oxidation by  $\text{H}_2\text{O}_2$ ,<sup>6,15</sup> which primarily needs to be activated, due to its relative stability. There are several known ways of hydrogen peroxide activation, like UV irradiation, ferric salts or ozone. According to literature, ultrasound provides additional highly active and non-selective HO radicals.<sup>2,3,6</sup> Furthermore, while acting as a source of free radicals, hydrogen peroxide may act as a scavenger of generated free radicals, as indicated by the chemical equation below:



The results shown in Fig. 1 confirmed positive ultrasound effect causing discoloration, which was recorded only for dyestuff of less stable structure, while more stable dyestuff molecules like phthalocyanine (eq. 2) and xanthene (eq. 4) record a reaction between  $\text{HO}_2^{\bullet}$  radicals and ferric ions, which leads to termination:



The main initiators of  $\text{H}_2\text{O}_2$  catalytic decomposition reaction for both Fenton and Fenton-like method are Fe(III) ions. However, the Fenton-like method ( $\text{FeCl}_3$ ) implies the use of organic formic acid in order to set the appropriate pH 3. Within the acidic medium  $\text{FeCl}_3$  reacts with  $\text{H}_2\text{O}_2$  according to reactions (eq. 3) and (eq. 4) delivering a discoloration degree of over 90 %.



However, both the investigated chemical methods record the negative effect of higher wastewater temperature, which varied depending on dyestuff chemical constitution. Higher temperatures caused increased decomposition of

$\text{H}_2\text{O}_2$ , which resulted in lower discoloration degree. The highest discoloration degree was obtained for C. I. Reactive Blue 19 (over 90 %).

One major negative effect of the presented chemical discoloration processes, is the inability of stoichiometrically adding Fe salts according to the amount of dye in wastewater, the quantity of which varies significantly depending on the very dyeing process being performed, best seen in Fig. 2, where the amount of residual ferric ions decreases with increased dyestuff mass concentration, suggesting how a great portion of ferric ions took part in decomposing the dyestuff. An arising ecological problem are the excessively added ineffective Fe-salts, which end up only additionally burdening wastewaters.

Comparison of both applied chemical methods delivered unsatisfactory results regarding legislation, which was confirmed by low biodegradability values, as indicated in Fig. 3. Both described methods in which ferric salts i.e. inorganic coagulants are used, possess not only oxidative properties, but physical as well, meaning coagulation preceded by precipitation, when applied under alkali conditions.

Applied organic coagulant/flocculant i.e. application of physical-chemical methods provided results showing the discoloration degree to be less dependent of dyestuff chemical constitution, but rather to depend principally on spatial structure, dyestuff molecule size and solubility. This was confirmed through discoloration degree data obtained for highly soluble xanthene C. I. Acid Red 52 dye. The disadvantages of this method regarding discoloration (Fig. 1) and biodegradability (Fig. 3) were eliminated by using activated carbon filter, thus indicating a need to combine certain physical and physical-chemical methods in the aim of obtaining satisfactory results.

The described methods were applied to the simulated dye-bath. However, their applicability was to be confirmed by investigations carried out under real industrial conditions, where e. g. cotton reactive dyeing processes are followed by use of large quantities of salts, thus additionally burdening water. The Fenton-like process of purifying and physical-chemical method in which poly-electrolytes are used, were investigated both from the economic and environmental aspect.

Industrial wastewater obtained after cotton reactive dyeing (C. I. Reactive 19) showed typically high values of ecological quantities (Table 1), which recorded a significant decrease after each purifying method performed. In addition, the advantage of all presented methods is the insignificant influence of dyeing auxiliaries on both the discoloration degree and measured ecological quantities. One of the important disadvantages of physical-chemical methods came to light after  $\text{BOD}_5/\text{COD}$  measurements. The intolerably high  $\text{BOD}_5/\text{COD}$  value presented a problem which had to be solved. This was done by an additional purification phase of adsorption on activated carbon, after which  $\text{BOD}_5/\text{COD}$  values assimilated most acceptable values, indicating water purity rather more than low biodegradability, confirmed by low TOC values.

One of the most important steps carried out was to determine the influence of auxiliary chemicals ( $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ , NaOH (32 %), NaCl) in dye-bath on electrical conductivity

( $\kappa$ ). No auxiliary chemicals, besides NaCl, increase significantly electrical conductivity (Fig. 4). Sodium chloride is the most commonly used electrolyte in cotton reactive dyeing, the amount of which bonds in small quantities with cotton textile fibres, thus ending in wastewaters causing an undesirable additional burden. This was the reason for establishing the correlation between the amount of electrolytes set as an input and electrical conductivity set as an output determining quantity. In addition, this paper suggests electrical conductivity as a wastewater control quantity, which can act as a direct pointer of water impurity caused by industrial dyeing processes.

## Conclusion

The elaborated methods are easy to use and apply, and usable for a wide variety of dyestuffs considering their chemical and dyeing properties. The disadvantage of both chemical and physical-chemical methods is the inability to stoichiometrically add chemicals used for discoloring, which ends up additionally burdening wastewaters. Avoiding the preliminary step of optimizing the process of purification could lead to unnecessary pollution of wastewaters, now caused by excessive amounts of ferric ions or poly-electrolytes. Discoloring issues were proved to depend on overall contents of wastewaters, not only from the aspect of their heterogeneity, but also from the aspect of chemical structure of compounds present in wastewater. Industrial dye-house wastewaters usually consist of mixtures of dyes that are chemically differently grouped, spatially formed, and usually vary in molecular size. Accordingly, the presence of chemically stable xanthene dyestuff resulted in a low discoloration degree. Typical auxiliaries used in cotton reactive dyeing are electrolytes e.g. NaCl and Na<sub>2</sub>SO<sub>4</sub>, which end up in water once the technological process of dyeing has been completed. The results confirmed the necessity of combining several purifying methods in order to achieve acceptable ecological values. All of the applied methods achieved high discoloration degrees, though more thorough purifying demands a process of membrane purifying, which will assure complete elimination of salts from wastewaters.

## List of symbols

### Popis simbola

$\gamma$	– mass concentration, g l <sup>-1</sup> – masena koncentracija, g l <sup>-1</sup>
COD	– chemical oxygen demand – kemijska potrošnja kisika
BOD <sub>5</sub>	– biological oxygen demand – biološka potrošnja kisika
$f$	– frequency, Hz – frekvencija, Hz

$P$	– power, W – snaga, W
$n$	– stirring speed, min <sup>-1</sup> – brzina miješanja, min <sup>-1</sup>
pH	– indication of liquid acidity or alkalinity – mjera kiselosti ili alkalnosti otopine
$R^2$	– correlation indeks – indeks korelacije
TOC	– total organic carbon – ukupni organski ugljik
$\eta$	– discoloration degree, % – stupanj obezbojanja, %
$\vartheta$	– temperature, °C – temperatura, °C
$\kappa$	– electrical conductivity, mS cm <sup>-1</sup> – električna provodnost, mS cm <sup>-1</sup>
$\theta$	– time, min – vrijeme, min
$\sigma$	– volume concentration, ml l <sup>-1</sup> – obujamska koncentracija, ml l <sup>-1</sup>
$\varphi$	– volume fraction, % – obujmni udjel, %

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## SAŽETAK

## Usporedba kemijskih i fizikalno-kemijskih metoda obezbojavanja otpadnih voda

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Današnje kemijske i fizikalno-kemijske metode obezbojavanja otpadnih voda ne zadovoljavaju u potpunosti zahtjeve vezane uz stupanj obezbojavanja. U ovom radu obezbojavanje se provodilo pomoću Fentonove kemijske metode ( $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O} + \text{H}_2\text{O}_2 + \text{H}_2\text{SO}_4$ ) i modificirane Fentonove kemijske metode ( $\text{FeCl}_3 \cdot 6 \text{H}_2\text{O} + \text{H}_2\text{O}_2 + \text{HCOOH}$ ), te pomoću fizikalno-kemijske metode upotrebom koagulanata/flokulanata (polielektrolita-POEL), koja kombinira anionski koagulant (modificirani poliakrilamid) i kationski flokulant (produkt dušikovih spojeva) uz adsorpciju na aktivnom ugljenu. Prihvatljivost opisanih metoda ispitivana je za reaktivna i kisela bojila, s obzirom na njihovu raširenu primjenu u tekstilnoj industriji. Također, provedena su ispitivanja na bojilima različitih kromogena (antrakinon, ftalocijanin, azo i ksanten) s ciljem određivanja važnosti prostorne strukture molekule. Oksidativno djelovanje Fentonovog i modificiranog Fentonovog postupka dovodi do razgradnje obojenog kromogena i visokog stupnja obezbojavanja. Međutim, ustanovljena je nemogućnost dodavanja POEL u stehiometrijskom odnosu (također prisutno kod modificiranog Fentonovog postupka), odnosno dodavanje koagulanata u suvišku kako bi se postigao viši stupanj obezbojavanja, što u konačnici dovodi do opterećenja voda POEL-ima. Kvaliteta vode na ulazu i izlazu kontrolirana je pomoću spektrofotometrijskih mjerenja i uobičajenih bioloških parametara. U prilog tome, dio ispitivanja bio je vezan za industrijske otpadne vode bojadi-saone, u kojoj se pamučni materijal bojadisao bojom C. I. Reactive Blue 19 i to tehnološkim postupkom koji zahtijeva uporabu velike količine elektrolita. Također, ispitivanje industrijskih otpadnih voda označeno je ključnim korakom izbjegavanja niza krivih pretpostavki i zaključaka, koji bi mogli nastati u slučaju laboratorijskog simuliranja procesa bojadisanja.

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