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Degradation of nitroaromatics with the Fenton reagent

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Abstract. The feasibility of the Fenton reagent treatment for the degradation and detoxification of nitroaromatic compounds (NAC) such as *p*-nitrotoluene, nitrobenzene, and *m*-dinitrobenzene was studied. The degradation rate of NAC with the Fenton treatment was strongly dependent on the molar ratio NAC/hydrogen peroxide/catalyst (Fe^{2+}). The 90% conversion times of NAC in the Fenton treatment followed the order *p*-nitrotoluene ≈ nitrobenzene < *m*-dinitrobenzene. The degree of nitrogen conversion to nitrate with the Fenton treatment varied from 25% to 100%, depending on the treatment conditions. Total organic carbon removal of 45–47% was obtained when the Fenton reagent treatment with the concentration of hydrogen peroxide 10 mM and catalyst 1 mM was applied. According to the *Daphnia magna* toxicity test the Fenton reagent treatment enabled to reduce the toxicity of NAC.

Key words: nitroaromatic compounds, Fenton reagent, degradation, toxicity assay, by-products.

INTRODUCTION

Nitroaromatic compounds (NAC) are widely used in chemical industries (synthesis of dyes, pesticides, explosives, etc.) and have been associated with groundwater contamination [1]. The large-scale manufacture and use of NAC has led to significant contamination of soils and groundwater. Biological treatment of aqueous solutions of NAC is a complicated problem. NAC are not well biodegraded [2]. Their biological treatment is limited by their toxicity at high concentrations to microorganisms and sometimes produces recalcitrant or toxic by-products [3]. This is why there is a need to develop effective methods for the degradation of these pollutants, either to less harmful compounds or their complete mineralization.

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Some chemical oxidation methods have been recommended as a pre-treatment step for the purification of NAC-containing wastewater. They include treatment with ozone [2] and with an ozone and hydrogen peroxide mixture (known as “peroxone” oxidation) [4]; photocatalytic oxidation with TiO_2 [5, 6]; various advanced oxidation processes (AOPs) including ozone, UV radiation, and hydrogen peroxide [7, 8]; elemental iron (Fe^0)/ultrasound [9]; etc.

The Fenton reagent, an old classical reactive system, is nowadays successfully applied to wastewater treatment as a very simple and relatively cheap way of producing hydroxyl radicals. The use of the Fenton reagent to oxidize toxic organics began only in the late 1960s, although Fenton reported it already a hundred years ago for maleic acid oxidation [10].

The chemistry of the Fenton reagent is presented by a number of equations [11]. The most important of them is:

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

The rate constant for the reaction of ferrous ion with hydrogen peroxide is high and Fe^{2+} oxidizes to Fe^{3+} in a few seconds to minutes in the presence of excess amounts of hydrogen peroxide.

Hydroxyl radicals may be scavenged by the reaction with another Fe^{2+} ion:

$${}^{\bullet}OH + Fe^{2+} \rightarrow OH^{-} + Fe^{3+}. \qquad (2)$$

Catalytic decomposition of hydrogen peroxide by Fe^{3+} follows a radical mechanism that involves hydroxyl and hydroperoxyl radicals, including (1) and (2):

$$Fe^{3+} + H_2O_2 \leftrightarrow H^{+} + Fe\text{–}OOH^{2+}, \qquad (3)$$

$$Fe\text{–}OOH^{2+} \rightarrow HO_2^{\bullet} + Fe^{2+}, \qquad (4)$$

$$Fe^{2+} + HO_2^{\bullet} \rightarrow Fe^{3+} + HO_2^{-}, \qquad (5)$$

$$Fe^{3+} + HO_2^{\bullet} \rightarrow Fe^{2+} + H^{+} + O_2, \qquad (6)$$

$${}^{\bullet}OH + H_2O_2 \rightarrow H_2O + HO_2^{\bullet}. \qquad (7)$$

The Fenton reagent has been widely suggested for the application in the treatment of hazardous organics [12–19]. The treatment eliminates the toxic substances and increases the biodegradability of treated water. The use of $Fe(II)/H_2O_2$ as an oxidant for wastewater treatment is attractive because: (1) both iron and H_2O_2 are cheap and non-toxic; (2) there is no mass transfer limitation as the system has a homogeneous catalytic nature; and (3) the reactor design is much easier than for other advanced oxidation systems.

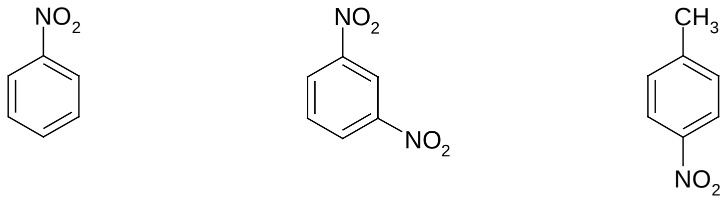
There are only a few studies concerning the degradation of NAC with the Fenton reagent and other AOPs. Beltran et al. [7] and Ghaly et al. [20] used AOPs such as O_3/UV, O_3/H_2O_2, H_2O_2/UV, and $O_3/UV/H_2O_2$ for the degradation of nitrobenzene (NB) and *o*-nitrotoluene, respectively. In both studies it was reported that AOPs enable 10 to 20 times higher NAC removal rates than ozonation alone.

Li et al. [21] applied UV-assisted Fenton oxidation to degrade nitrotoluenes in water. They found that tri- and di-nitrotoluenes are oxidized most effectively at pH 3.0 with UV-mediated Fenton oxidation, whereas mononitrotoluenes are rapidly degraded even without UV. Pseudo first-order constants for Fenton oxidation decrease with the number of nitro substituents and are affected by isomer configuration of the ring.

Lipczynska-Kochany [16] determined quite slow removal of aqueous NB with the Fenton reagent at pH 6.9 and at the ratio of $NB/H_2O_2/Fe^{2+}$ 1 : 80 : 0.35. Under such treatment conditions the half-life of NB was 250 min. The Fenton reagent was found to be among the most effective AOPs for the treatment of trinitrotoluene (TNT) production wastewater leading to 68.5% COD and 97.5% TNT reduction [8].

Thus, there is some evidence that processes based on radical generation are quite effective for the destruction of NAC. Therefore, the Fenton reagent as a simple and cheap AOP was applied in the present study.

The main target of the current research was to evaluate the feasibility of the Fenton reagent for the degradation of NAC such as *p*-nitrotoluene (p-NT), NB, and *m*-dinitrobenzene (m-DNB) (see Fig. 1).



nitrobenzene (NB) *m*-dinitrobenzene (m-DNB) *p*-nitrotoluene (p-NT)

Fig. 1. Nitroaromatic compounds studied.

In addition to ascertaining the conditions for NAC treatment, the degree of nitrogen mineralization, decrease of total organic carbon (TOC) during the treatment, and the toxicity after the Fenton treatment were investigated.

EXPERIMENTAL

The chemicals studied were NB, m-DNB, p-NT. NAC, purchased from Aldrich Co, were of analytical grade. The samples were prepared dissolving NAC in twice-distilled water. The initial concentration of NAC in all experiments was 1.0 mM. The pH value was adjusted using 0.1 N and 1.0 N aqueous sulphuric acid to 3.0, which is considered optimal for the Fenton treatment [11, 22].

The degradation rate of NAC in aqueous solution during the Fenton treatment was examined under batch conditions. A standard procedure was that 700 mL of fresh NAC solution was treated in a cylindrical glass reactor with magnetic stirring. Different concentrations of H_2O_2 (from 2.0 to 20 mM) and the catalyst Fe^{2+} (from 0.2 to 2.0 mM) were used. Thus, the molar ratio of the oxidant to NAC varied from 2 to 20. The reaction was stopped by the addition of 10% aqueous solution of Na_2SO_3. All experiments were carried out at $20 \pm 1\,°C$.

The samples (5 mL each) were taken at intervals during the experiment. The progress in the degradation of NAC was followed with high performance liquid chromatograph (HPLC) (Millichrom, Nauchpribor, USSR) equipped with a UV-spectrophotometer (190–360 nm). A reverse phase column, packed with 5 μm Separon C18 (Chemapol, Czechoslovakia), was 64 mm in length and 2 mm in diameter. The isocratic method with a solvent mixture of 55% acetonitrile and 0.2% acetic acid in water with a flow rate of 100 μL min^{-1} was applied. The analytical wavelength and the retention time were 270 nm and 4.1 min for NB, 288 nm and 5.2 min for p-NT, and 270 nm and 3.9 min for m-DNB. The concentration of nitrate ions formed as a result of nitrogen mineralization was measured with ion chromatography [23].

The concentration of TOC of the initial and treated samples was measured with a Shimadzu TOC-5000 analyser equipped with an autosampler (ASI-5000) and platinum based catalyst. The carrier gas was synthetic air at a rate of 150 mL min^{-1}.

After the treatment of the solutions their pH values were adjusted to approximately 9.0 using 40% NaOH. In the basic medium iron ions precipitated, and after that they were filtered through a paper (blue ribbon) filter. The total iron residual concentration was measured in the treated and filtered solutions according to [24].

A specific hydrogen peroxide concentration was adjusted in the NAC solution before the treatment and no further H_2O_2 was added during the degradation. The residual concentration of hydrogen peroxide was measured spectrophotometrically (Specord UV/VIS) at 410 nm as a complex with Ti^{4+} [25].

Acute toxicity of the initial NAC and the toxic effects after the Fenton treatment to *Daphnia magna* (Cladocera, Crustacea) were studied. The *Daphnia* clone used was of Finnish origin (*Daphnia magna EF*) from the North Savo Regional Environmental Centre in Kuopio, Finland. NAC were treated until at least 90% conversion of the initial compound to products was achieved. The initial concentration of NAC was 1.0 mM. The living conditions were kept optimal and the 24-hour toxicity test was carried out according to [26]. A special computer program PROBIT was used for the approximation of median effective concentration values (EC_{50}) and their 95% confidence limits.

RESULTS AND DISCUSSION

The Fenton reagent with the different concentrations of hydrogen peroxide and the catalyst (Fe^{2+}) was applied for the degradation of NAC. The concentration of both hydrogen peroxide and the catalyst (Fe^{2+}) affected the degradation of NAC during the Fenton treatment. As it was ascertained in our previous studies, the optimal molar ratio hydrogen peroxide/catalyst in the Fenton treatment is close to 10 and hydrogen peroxide/contaminant >3 [19], which is in concordance with the data of other authors [13, 17, 27]. Therefore, in the present study the ratios applied were close to the aforementioned ones. Half-lives and 90% conversion times for the degradation of NAC calculated from the degradation curves are presented in Table 1. The effects observed in the treatment of NB are presented in Fig. 2 as a typical example. As can be seen from Fig. 2, the low concentration of the oxidant (in this case 2 mM H_2O_2; H_2O_2/NB molar ratio 2 : 1) did not allow achieving 90% degradation of NB whereas a 2.5-fold increase in the H_2O_2/NB molar ratio to 5 : 1 led to 90% conversion of the target compound within 14 min of treatment. The further increase of H_2O_2/NB molar ratio to 10 : 1 enabled to degrade NB instantly as 90% degradation of NB required less than 3 min of treatment (see Table 1). On the other hand, when the other parameters were kept constant a 2-fold increase of the catalyst concentration also accelerated the initial stage of NAC degradation (see Fig. 2 and Table 1). Nevertheless, a 2-fold increase of the catalyst concentration did not affect the final result as the differences in the 90% degradation times were insignificant. Moreover, the increase in the catalyst concentration resulted in a rise in the amount of sludge produced.

When different NAC were degraded under the same treatment conditions, according to 90% conversion times their degradation rate followed the order p-NT ≈ NB > m-DNB (see Fig. 3). The degradation curves for NB and p-NT were quite similar, whereas m-DNB degraded at a significantly lower rate. Under the treatment conditions presented in Fig. 3 only 63% conversion of m-DNB occurred in 1 hour of treatment and no further degradation of m-DNB was observed.

Summarizing the above-discussed part of the study, it can be concluded that the molar ratios of chemicals required to attain >90% conversion of NAC in the Fenton treatment were 10 : 1 : 1 for H_2O_2/m-DNB/catalyst (Fe^{2+}) and 5 : 1 : 0.5 for H_2O_2/p-NT (or NB)/Fe^{2+}.

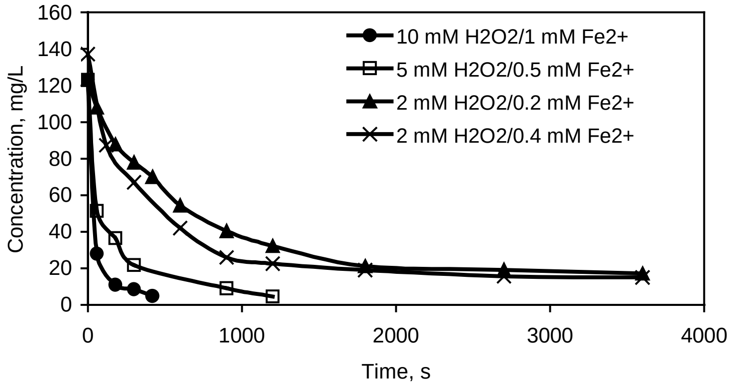


Fig. 2. Decline of nitrobenzene concentration during the Fenton treatment at pH 3.0.

Table 1. Half-lives ($T_{1/2}$) and 90% conversion times ($T_{90\%}$) for the degradation of nitrobenzene (NB), *m*-dinitrobenzene (m-DNB), and *p*-nitrotoluene (p-NT) with the Fenton reagent

Compound	H_2O_2 added, mM	Fe^{2+} added, mM	Fenton		Nitrogen conversion degree, %
			$T_{1/2}$, min	$T_{90\%}$, min	
NB	10	1	<1	2.7	81
NB	5	0.5	<1	14.0	51
NB	2	0.4	6.1	*	25
NB	2	0.2	9.0	*	no data
m-DNB	20	2.0	<1	18.3	100
m-DNB	10	2.0	<1.3	23.3	97
m-DNB	10	1	11.0	52	89
m-DNB	5	0.5	30	*	45
p-NT	10	1	<0.25	3.2	100
p-NT	5	0.5	2.8	13.3	67
p-NT	2	0.4	2.5	44	28
p-NT	2	0.2	23	50	30

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* 90% degradation was not achieved within 2 hours of treatment

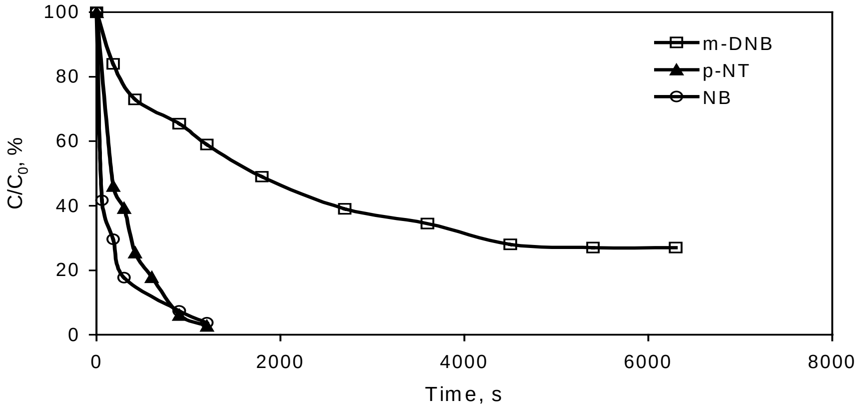

Fig. 3. Degradation of nitrobenzene (NB), *m*-dinitrobenzene (m-DNB), and *p*-nitrotoluene (p-NT) with the Fenton reagent (5 mM H_2O_2/0.5 mM Fe^{2+}) at pH 3.0.

The residual iron can be removed after the Fenton treatment by increasing the pH of the solution. Iron is precipitated at pH ≥ 9.0 in the form of $Fe(OH)_3$. The residual concentration of total iron after alkalization and subsequent filtration remained mainly at 0.3–1.0% of the initial value when 90% degradation of NAC was achieved.

There are not enough earlier data to compare the degradation rate of NAC. Still, the half-lives of NB in the current study were substantially shorter (from less than 1 to 9 min) than those reported by Lipczynska-Kochany [16] (250 min) due to the more reasonable molar ratio of H_2O_2 to Fe^{2+} applied. The Fenton reagent enabled to achieve ≥90% conversion of all NAC under study within acceptable time and chemicals consumption. Consequently, there is no need to use significantly more expensive photo-Fenton treatment for NAC degradation.

Cleavage of the nitro-group from the aromatic ring and its conversion to nitrate took place during the degradation of NAC. The degree of organically bounded nitrogen conversion to nitrate after the Fenton treatment varied in a wide range from 25% to 100% (see Table 1). The degree of nitrogen mineralization was somewhat higher for p-NT and increased definitely when higher concentrations of chemicals in the Fenton reagent were used (see Table 1). The data indicated also that cleavage of the nitro-group took place at the advanced stages of the treatment. Still, a very high nitrogen mineralization degree can be attained in the Fenton treatment: from 81% to 100% of organically bounded nitrogen was converted to nitrate when the NAC/H_2O_2/catalyst ratio was 1 : 10 : 1 (see Fig. 4).

The degree of carbon mineralization was lower than nitrogen mineralization. Up to at least 90% degradation of NAC led to 45–47% TOC removal when the Fenton reagent with a concentration of hydrogen peroxide 10 mM and catalyst 1 mM was applied. At this stage the organics in the solution are probably carboxylic acids, which are quite resistant to mineralization.

According to the *Daphnia magna* toxicity test the Fenton reagent treatment enabled to reduce or even remove the toxicity of NAC (see Table 2). For example, the treatment of p-NT with the Fenton reagent containing 10 mM of hydrogen peroxide and 1 mM of the catalyst led to ultimate detoxification of the NAC solution. The same results were obtained for m-DNB when treated with 20 mM hydrogen peroxide and 2 mM of the catalyst. The increase in the concentrations of chemicals in the Fenton treatment led to more effective removal of toxicity (see Table 2). Nevertheless, the complete toxicity removal required somehow higher concentrations of the Fenton reagent than those needed for complete degradation of the target NAC.

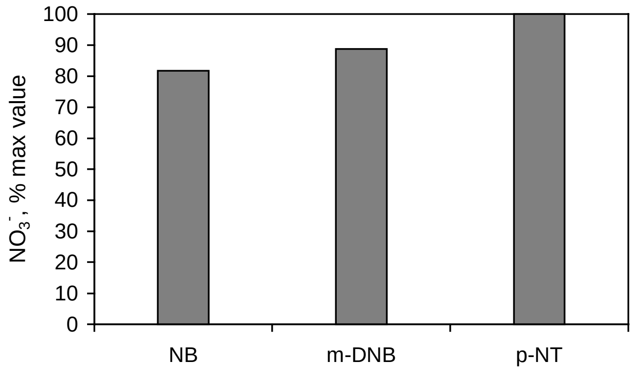

Fig. 4. Nitrogen conversion degree to nitrates after the Fenton treatment (10 mM H_2O_2/1 mM Fe^{2+}) of nitrobenzene (NB), *m*-dinitrobenzene (m-DNB), and *p*-nitrotoluene (p-NT).

Table 2. EC_{50} values for nitrobenzene (NB), *m*-dinitrobenzene (m-DNB), and *p*-nitrotoluene (p-NT) and the toxic effects after the Fenton treatment of aqueous solution containing 1 mM of nitroaromatic compounds

Compound	EC_{50} value for NAC, mg/L	Conditions of treatment		Toxic effects after treatment
		Fe^{2+} added, mM	H_2O_2 added, mM	
NB	41 (33%)	1	10	EC_{50} = 76%
m-DNB	43 (25%)	20	2	Not toxic
m-DNB	43 (25%)	1	10	EC_{50} = 64%
m-DNB	43 (25%)	2	10	EC_{50} = 24%
p-NT	9.7 (7%)	1	10	Not toxic
p-NT	9.7 (7%)	0.2	2	EC_{50} = 28%

The molar ratio of the chemicals in the Fenton treatment recommended from the results of both the kinetics and the toxicity studies could be H_2O_2/m-DNB/catalyst (Fe^{2+}) 20 : 1 : 2 and H_2O_2/p-NT (or NB)/Fe^{2+} 10 : 1 : 1. Thus, the Fenton reagent treatment can be used not only for effective degradation of the studied NAC, but also for the detoxification of their aqueous solutions.

CONCLUSIONS

The degradation rate of nitrobenzene, *m*-dinitrobenzene, and *p*-nitrotoluene with the Fenton treatment was significantly dependent on the concentration of hydrogen peroxide and of the catalyst (Fe^{2+}). A low ratio of hydrogen peroxide to NAC did not allow achieving complete degradation of the studied NAC in the Fenton treatment. Nevertheless, the Fenton reagent enabled to achieve ≥90% conversion of NAC within acceptable time and chemicals consumption. Detoxification and almost complete nitrogen mineralization can be attained. The molar ratio of the chemicals in the Fenton treatment recommended from the results of the present study could be hydrogen peroxide/*m*-dinitrobenzene/catalyst (Fe^{2+}) 20 : 1 : 2 and hydrogen peroxide/*p*-nitrotoluene (or nitrobenzene)/Fe^{2+} 10 : 1 : 1. Therefore, there is no need to use the significantly more expensive photo-Fenton treatment or other AOPs for NAC degradation.

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Aromaatsete nitroühendite lagundamine Fentoni reaktiiviga

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Aromaatsete nitroühendite (NAC) ulatuslik tootmine on viinud selleni, et nende sisaldus pinnases ja põhjavees on märkimisväärne. NAC sisaldava vesilahuse töötlemine bioloogiliste meetoditega on saasteainete suure toksilisuse tõttu keeruline. Käesoleva uurimuse peaeesmärk oli hinnata Fentoni reaktiivi mõju selliste NAC lagundamiseks ja detoksifitseerimiseks nagu *p*-nitrotolueen, nitrobenseen ja *m*-dinitrobenseen. NAC lagunemiskiirus Fentoni reaktiiviga töötlusel sõltus tugevasti vesinikperoksiidi ja katalüsaatori (Fe^{2+}) kontsentratsioonist. 90%-liste konversiooniaegade põhjal langeb uuritud NAC lagunemise kiirus töötlusel järjekorras: *p*-nitrotolueen ≈ nitrobenseen > *m*-dinitrobenseen. NAC lagundamisel toimus eraldunud nitrorühma konversioon nitraadiks. Orgaaniliselt seotud lämmastiku konversiooniaste oli pärast uuritud ühendite 90%-list lagunemist Fentoni reaktiiviga töötlusel 25–100%. 45–47%-line orgaanilise süsiniku eemaldamine saavutati juhul, kui töötlusel oli vesinikperoksiidi kontsentratsioon 10 mM ja katalüsaatori oma 1 mM. *Daphnia magna* toksilisuse testi kohaselt on Fentoni reaktiiviga töötlus võimeline vähendama NAC lahuste toksilisust.