



Bio-recalcitrant pollutants removal from wastewater with combination of the Fenton treatment and biological oxidation



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ABSTRACT

The implementation of the Fenton treatment as an intermediate step in combined technological scheme for the removal of bio-recalcitrant organic pollutants from industrial effluents is proposed in this study. The treatment schemes for the purification of three heavily polluted wastewaters originated from a municipal non-hazardous waste landfill, industrial hazardous waste landfill and plywood manufacturing factory were developed. Accordingly, the wastewaters were subjected to aerobic biological treatment (BIO) combined with the Fenton process with the final aim of establishing universally applicable treatment scheme.

The study demonstrated that the Fenton oxidation can be successfully included in the high-strength wastewater treatment scheme as an intermediate treatment step for the removal of bio-recalcitrant organics fraction. The elaborated BIO-Fenton-BIO approach was found to be cost-effective for the total organic load reduction and for the selected specific pollutants removal from the studied high-strength wastewaters. Consequently, it could be considered suitable for broader practical implementation.

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1. Introduction

The situation with the pollution load has been improving substantially during the last decade in the Baltic Sea region. However, it has been achieved mainly due to comprehensive improvement of domestic wastewater purification. The further decrease in the pollution load cannot be attained without considerable improvement of the purification of industrial wastewaters. Similar situation is observed in the so-called new EU members as well as developing countries. The latter are especially sensitive to the cost of implemented treatment.

The biological treatment which removes only highly biodegradable contaminants is widely used as a conventional approach in wastewater treatment installations nowadays. However, some wastewater flows contain compounds that are toxic and/or bio-recalcitrant to biological community, degrading neither during the biological treatment nor subsequently by microorganisms present in the receiving water bodies. As a result, bio-recalcitrant compounds that accumulate in water and bottom sediments affect

aquatic biota and the overall ecological equilibrium. The biodegradability of wastewater could be enhanced by the application of chemical oxidation or other chemical-physical processes as a pre- or main treatment step in wastewater treatment schemes. The synergetic effect of combined chemical and subsequent biological treatment has been demonstrated by earlier publications for textile and dyeing wastewater [1–4], pulp mill wastewater [5], coal gasification wastewater [6], toluene, benzene, naphthalene and xylene containing wastewater [7], wastewater generated from pyridine and cyanopyridine manufacturing plants [8], cheese whey wastewater [9], etc.

Among various advanced oxidation technologies (AOTs), the Fenton process and its modifications proved effective in the degradation of wide range of organics present in industrial effluents resulting in a high degree of elimination of bio-recalcitrant compounds [10,11], their partial mineralisation and noteworthy improvement of biodegradation in subsequent biological treatment stage. Therefore, the application of the Fenton pre-treatment step in combined schemes primarily aims at improvement of the biodegradability and the reduction of toxicity to the level beyond which biological treatment can be implemented [12–15]. Indeed, the application of the Fenton pre-treatment for raw wastewater containing high amount of organics could be effective, however,

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Table 1
Main characteristics of raw wastewater samples^a.

Parameter	MLL	PWW	SCL
pH ^b	7.3–7.6	4.75–5.05	7.9–9.3
COD, mg/L ^b	5900–6790	5100–6830	851–2100
BOD ₇ , mg/L ^b	3100–4700	2600–3530	175–330
TOC, mg/L ^b	2000–2500	1570–3240	367–505
DOC, mg/L ^b	1550	585–975	243–463
Total solids (TS), mg/L ^b	6780	2550	4540–8650
Total fixed solids, mg/L ^b	3010	520	4030–7640
Total suspended solids, mg/L ^b	1900	1780–1800	21–24
Recalcitrant COD ^c , %	15	7	15
Inhibition of oxygen consumption, I ₅₀ , %	no ^d	25.5	no->20
Total nitrogen (N _{total}), mg/L ^b	710–840	11–69	10–42
Inhibition of nitrification rate, I ₅₀ , %	5.8–5.9	13–17	16–18
<i>Daphnia magna</i> acute toxicity, EC ₅₀ , % ^e	3.5–7.9	9.3–15.3	19.5
Total phosphorus, mg/L ^b	19	33–60	40–42
Total phenols, mg/L ^b	33–148	51–94	6–18
Lignin and tannins, mg/L ^b	380–730	560–870	n.d. ^f

^a The highest and lowest values are given as the characteristics varied substantially for the samples taken in different time.

^b According to Ref. [16].

^c According to Ref. [17].

^d No inhibition observed.

^e According to Ref. [20].

^f Not determined.

even partial pollutants degradation requires a high load of oxidant that is reflected in substantially increased total expenditure on such treatment. This drawback could be alleviated by the implementation of multi-step treatment utilising the Fenton process as an intermediate step after conventional biological treatment.

Therefore, the aim of this study was the development of strategies for the implementation of combined aerobic biological and the Fenton treatment in a multi-step technological scheme for the efficient and cost-effective removal of bio-recalcitrant organic pollutants as well as the overall improvement of the effluent quality. Three high-strength wastewater samples from different sources such as leachate from a landfill specialised on municipal non-hazardous waste management and recycling (MLL), hardwood soaking basin wastewater from plywood industry (PWW), and leachate from oil shale processing semicoke landfill area (SCL) containing significant amount of recalcitrant organics (Table 1) were studied and the treatment schemes for their purification were developed. The results obtained within this study give a valuable knowledge for the subsequent practical application and help to upgrade the classical wastewater treatment scheme introducing the Fenton treatment step for the degradation of bio-recalcitrant organics, the improvement of biodegradability and purification degree of high-strength wastewater, and, thus, lowering the total organic pollution load.

2. Materials and methods

2.1. Analytical methods

The chemical oxygen demand (COD), total suspended solids, total solids, total nitrogen, pH, a 7-d biochemical oxygen demand (BOD₇), were determined according to Ref. [16]. Total fixed solids – solids that remain after firing a sample at 550 °C in muffle furnace determined from total sample Ref. [16] were used to characterise the total inorganic solids present in wastewater.

Total and ferrous iron were determined by the o-phenanthroline method [16]. The concentrations of lignin and tannins were measured spectrophotometrically by the tyrosine method and total

phenols by the sodium nitrite method using a HACH-Lange cuvette test [16]. Total organic carbon (TOC) and dissolved organic carbon (DOC) were measured by a TOC analyser multi N/C[®] 3100 (Analytik Jena) for both unfiltered and filtered (0.45 μm cellulose acetate membrane) samples respectively.

In order to assess the biodegradability of raw and treated wastewater samples the Zahn–Wellens test [17] with duration of 28 d was performed. The Zahn–Wellens test results were used for assessing the amount of recalcitrant organics fraction in wastewaters by determining the lowest achieved value of COD during 28 d of test period. The inhibition tests of oxygen consumption and nitrification rate by activated sludge for raw wastewater were carried out according to Refs. [18] and [19], respectively. Tests were taken during three hours of aerobic conditions, reduction of dissolved oxygen concentration (oxygen consumption test) and production of nitrate (nitrification test) were measured. Activated sludge used in MLL and PWW biodegradability and inhibition tests originated from municipal wastewater treatment plant (WWTP) in Tartu (Estonia) and Kohtla-Järve (Estonia) in SCL tests. The latter is acclimated to phenolic wastewater as it receives effluents from the semicoke landfill.

2.2. Characterisation of the wastewater

The wastewater samples have been taken from March 2012 till September 2014. MLL samples were obtained from landfill, which is in operation since 2003 specialising in municipal non-hazardous waste management and recycling. SCL samples were collected from a semicoke (hazardous waste rich in phenols) landfill area of an oil-shale thermal treatment plant. PWW samples were taken from hardwood soaking basin of a local plywood industry. The collected wastewater samples were stored at 4 °C. The main parameters of the studied wastewaters are presented in Table 1.

2.3. Experimental procedure for the chemical treatment

All the Fenton process trials were performed in a batch mode. Wastewater samples (0.5 L) were treated in a 1-L glass reactor for a period of 1, 2, 4, 6 or 24 h with a constant agitation (400 rpm). The pH of the wastewater samples was not adjusted in the Fenton treatment if not specified otherwise. The catalyst (FeSO₄·7H₂O) was added, and after its complete dissolution, the Fenton reaction was initiated by adding an oxidant (H₂O₂) all at once. The Fenton treatment has been carried out at a H₂O₂/COD weight ratio (w/w) varied in the range of 0.5/1–4/1 and a H₂O₂/Fe²⁺ w/w ratio of 5/1; the latter has been demonstrated to be optimal in our previous study [21]. The oxidation was terminated by the adjustment of pH to ~9 with NaOH (10 M) that resulted in formation of ferric oxyhydroxide precipitate. The residual iron concentration after 24 h sedimentation of ferric-hydroxy complexes in the water phase was very low (<5 mg/L) indicating effective iron precipitation. Finally, the supernatant was collected for further analysis and the post-treatment. The experiments on wastewater 24-h oxidation with H₂O₂ solely were carried out as blank tests under treatment conditions identical for the respective Fenton treatment trials.

The coagulation with ferric sulphate (KEMIRA PIX-322, Fe_{total} 12.5 ± 0.3%) was performed for raw wastewater in a jar test apparatus (Kemira, Finland) for PWW. The coagulant doses varied in the range of 100–1000 mg(Fe_{total})/L and the operating conditions followed the standard procedure as has been described in [22]. Afterwards, the supernatant was collected for further analysis and treatment.

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