

Dissolved and colloidal organic nitrogen removal from wastewater treatment plants effluents and reject waters using physical–chemical processes

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ABSTRACT

Four physical–chemical processes were compared in terms of the efficiencies of dissolved and colloidal organic nitrogen (DON and CON) removal from the secondary effluents (SE) and reject water from full-scale biological nutrient removal activated sludge systems. Adsorption on activated carbon was most efficient and allowed removal from the SE of up to 80% and 100% of DON and CON, respectively. High efficiencies of DON removal from SE (up to 55%) were also obtained when using coagulation with iron(III) chloride and calcium hydroxide at final pH = 11.0–11.5. The efficiency of DON removal from thickening waste activated sludge (TWAS) reject water, obtained using coagulation with iron(III) chloride, was comparable with the efficiency for the SE. The efficiency of this process with regard to the sludge digester liquors (SDL) was significantly higher, i.e., 65–70% for both DON and CON. The ion exchange process with strongly acidic cation exchange resin (without pH correction) resulted in a relatively small efficiency of DON removal (<15%), and negligible efficiency of CON removal (<10%). Furthermore, ultrafiltration (0.015 μm) of SE and TWAS reject water resulted in a relatively low efficiency of DON removal (10–13% and 10–20% respectively). Ultrafiltration was found to be more effective for DON removal from SDL (41–68%).

Key words | adsorption, coagulation, colloidal organic nitrogen (CON), dissolved organic nitrogen (DON), ion exchange, nitrogen removal, ultrafiltration

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INTRODUCTION

Increasingly stringent regulatory initiatives have been proposed in the USA, European Union, Japan, and other countries, to control N discharges to inland and marine waters to the limit of technology levels. For biological nutrient removal (BNR) systems, designed to maximize nitrification/denitrification and effluent solids removal, the effluent total N concentration may range from 2.0 to 4.0 g N/m³ with a significant contribution of organic nitrogen (ON) (25–50%), mainly as colloidal (CON) and dissolved (DON) forms (WERF 2008). Reported effluent DON contributions vary widely in municipal BNR wastewater treatment plants (WWTPs) – from <2% to as high as 85% of the effluent total N (TN) (Pehlivanoglu & Sedlak 2004; Pagilla *et al.* 2006; WERF 2008; Gajewska 2011). To increase the understanding of the behavior of organic nitrogen in BNR wastewater treatment processes, Czerwionka *et al.* (2012) evaluated the influent N characteristics and fate of N

fractions at eight full-scale BNR activated sludge systems in northern Poland. It was found that the average secondary effluent DON concentrations ranged from 0.5 to 1.3 g N/m³, whereas DON accounted for 12–45% of the effluent TON. The content of the colloidal fraction ranged from 35 to 44% of TON with the average concentrations of 0.7–1.9 g N/m³. Therefore, there is a growing need to investigate specific DON and CON removal technologies to meet stricter effluent TN permit limits.

Biological processes in activated sludge systems have been identified as a potential method of DON removal. Alternatively, physical–chemical processes may be used for this purpose. Arnaldos & Pagilla (2010) reviewed earlier studies and concluded that granular activated carbon and chemical precipitation would be the most efficient processes for DON removal. Other processes, such as ion exchange, membrane processes (microfiltration, reverse osmosis), and

chemical oxidation processes (e.g. with sodium permanganate, hydrogen peroxide, chlorination, ozonation, UV radiation and the process of Fenton) have been found to be less effective (Parkin & McCarty 1981; Westerhoff & Mash 2002; Pehlivanoglu-Mantas & Sedlak 2006).

Reject water, generated in WWTPs, comprises sludge digester liquors (SDL) (large municipal WWTPs) and liquors from waste activated sludge dewatering (small and medium WWTPs). Handling the reject water has troubled the WWTP operation due to its significant effects on the main-stream treatment lane. The SDL are characterized by high N concentrations (occurring mainly in the form of ammonia nitrogen), greatly exceeding the values found in the 'typical' municipal wastewater. Fux *et al.* (2006) found that ammonia nitrogen concentrations in digester reject water varied in a wide range, i.e., 450–1,700 g NH₄-N/m³, at flow rates of only 0.5–2.0% of the influent flow rates. In practice, this means that the reject water usually constitutes 10–30% of the N load to WWTPs. In contrast, there is only a little information about nitrogen concentrations in reject water from waste activated sludge thickeners.

The aim of this study was to compare, under laboratory conditions, various physical–chemical processes in terms of the efficiencies of DON and CON removal from SDL and effluents of full-scale BNR activated sludge systems. The examined processes were selected based on the potential for practical use in tertiary mainstream treatment systems (ultrafiltration, final coagulation using metal salts or lime), water reuse systems (ion exchange, adsorption on activated carbon) or sidestream treatment systems (ultrafiltration and coagulation using iron salts).

MATERIAL AND METHODS

Description of WWTPs

Samples for the study were collected from three BNR activated sludge systems of different size and configuration,

located in northern Poland. Two of those plants (Gdansk and Gdynia) are the largest facilities in the region with a size exceeding 100,000 population equivalent (PE), and the other plant (Koscierzyna) is a medium-size facility with a size below 50,000 PE. The BNR process configurations are Modified University of Cape Town (MUCT) and Johannesburg (JHB). Sludge management is implemented as anaerobic digestion (AnD) with sludge disintegration or only dewatering thickening waste activated sludge (TWAS). The basic characteristics of the studied plants are presented in Table 1.

The daily average samples of secondary effluent (time-proportioned composite samples) were collected between February and July 2009. The composite samples of reject water were collected in an 8-hour working time sludge dewatering device between April 2011 and June 2013. The samples were filtered through 1.2 µm pore size nitrocellulose filters (Billerica MA, USA). The ON fractions were based on filtration pore-size separation and included particulate ON (>1.2 µm), CON divided into 'high' CON (0.45–1.2 µm) and 'low' CON (0.10–0.45 µm), and DON (<0.1 µm) as defined by Czerwionka *et al.* (2012).

Batch test experiments

Laboratory experiments were carried out in a simple experimental apparatus consisting of several glass beakers ($V = 1 \text{ dm}^3$) and magnetic stirrers with a controlled rotary speed. Technical pure ferric(III) chloride (FeCl₃) and ferric(III) sulphate (Fe₂(SO₄)₃) from Kemipol Company (Poland) and analytical grade calcium hydroxide (Ca(OH)₂) were used in the experiments as a source of ferric and lime, respectively. Multiple doses of iron compounds were used: 100, 200 and 300 g/m³, which corresponded to the doses of 34.5, 68.9 and 103.4 g Fe/m³ of ferric(III) chloride or 28, 56 and 84 g Fe/m³ of ferric(III) sulphate. The lime doses were adjusted to the final pH of less than 9 or 11–11.5. These pH values correspond to the low and high doses of lime used for phosphorus removal in

Table 1 | Basic characteristics of the studied WWTPs

WWTP	Size PE	Flow rate m ³ /d	SRT d	Configuration of bioreactor	Sludge handling
Gdansk	565,000	81,000	21–31	MUCT	AnD (primary and secondary sludge) with secondary sludge disintegration (high pressure homogenizers)
Gdynia	515,500	56,000	11–27	JHB	AnD (primary and secondary sludge) with secondary sludge disintegration (thermal oxidation)
Koscierzyna	36,600	3,200	12–29	JHB	Composting of dewatering TWAS

tertiary treatment systems. After dosing the reagents, a rapid mixing was performed for 60 s, followed by a slow mixing for 900 s in order to ensure optimum conditions for flocculation. Finally, the samples were left to settling of flocs for a period of 60 min.

Granular activated carbon Organosorb 10 from Desotec Company (Poland) and strongly acidic cation exchange resin C100 from PuroLite Company (USA) were used for organic nitrogen removal. Three activated carbon doses were used: 50, 200 and 500 g C/m³. For each dose, five samples were prepared to determine the efficiency of organic nitrogen removal with the contact times of 1, 2, 3, 5 and 10 h. A dose of 15 g/m³ of cation resin was also used without pH correction (pH ≈ 7.0) and after 5 h contact time.

Three replicate experiments at 20 °C were determined for each reagent and dose.

Ultrafiltration experiments

To evaluate the effectiveness of ultrafiltration on DON and CON removal from secondary effluent and reject water, the samples were filtered through 0.1 and 0.015 µm pore-size filters. Five experiments (Gdansk and Gdynia WWTPs) or three experiments (Koscierzyna WWTP) for the daily average samples of secondary effluent and 16–23 experiments for the grab reject water samples were carried out.

Analytical methods

Before the analysis, the samples were filtered under vacuum pressure through 1.2, 0.45 and 0.1 µm pore size nitrocellulose filters (Millipore, Billerica MA, USA). The effect of

ultrafiltration was investigated with 0.015 µm pore-size poly-carbon filters (Whatman, Kent, UK).

Total organic carbon (TOC) and TN concentrations were determined using a TOC analyzer (TOC-V_{CSH}) coupled with a TN module (TNM-1) (Shimadzu Corporation, Kyoto, Japan). The concentrations of inorganic N forms (NH₄-N, NO₃-N and NO₂-N) were determined in the filtrate using a Xion 500 spectrophotometer (Dr Lange GmbH, Berlin, Germany). The analytical procedures, which were adopted by Dr Lange and Shimadzu Corporation, followed the standard methods (APHA 2005). DON concentrations were calculated as the difference between TN after filtration on the appropriate pore-size filter (i.e. 0.1 µm or 0.015 µm) and the sum of inorganic N fractions (NH₄-N, NO₃-N and NO₂-N). CON concentrations were calculated as the difference between TN after filtration on the 1.2 µm pore-size filter and the sum of DON and inorganic N fractions.

RESULTS AND DISCUSSION

The average concentrations of N forms in the examined reject water and secondary effluents (SE) are presented in Table 2. Results of the DON and CON removal efficiencies from SE and reject water with the selected physical-chemical processes are summarized in Table 3.

Activated carbon adsorption

Adsorption on activated carbon was most efficient and allowed the removal of up to 80% of DON in SE from the Gdansk WWTP (the initial average DON concentration = 2.0 ± 0.07 g N/m³) and Koscierzyna WWTP (1.0 ± 0.29 g N/m³), whereas for the Gdynia WWTP (1.5 ± 0.18 g N/m³)

Table 2 | The average inorganic N, DON and CON concentrations (±SD) in the SDL and SE in the studied WWTPs

WWTP	NH ₄ -N g N/m ³	NO ₃ -N g N/m ³	NO ₂ -N g N/m ³	DON g N/m ³	CON g N/m ³
<i>Secondary effluents</i>					
Gdansk (9 samples)	0.69 (±0.45)	6.7 (±0.2)	0.14 (±0.04)	1.96 (±0.45)	0.55 (±0.24)
Gdynia (8 samples)	0.79 (±0.95)	5.8 (±0.3)	0.11 (±0.10)	1.38 (±0.24)	0.76 (±0.25)
Koscierzyna (5 samples)	0.05 (±0.02)	6.1 (±1.9)	0.02 (±0.01)	1.03 (±0.29)	1.35 (±0.46)
<i>Reject water</i>					
Gdansk (29 samples)	888.8 (±95.9)	1,46 (±0.37)	0.25 (±0.71)	29.5 (±10.1)	38.5 (±12.8)
Gdynia (22 samples)	647.0 (±51.0)	1.42 (±0.23)	0.05 (±0.03)	24.6 (±7.4)	33.7 (±9.2)
Koscierzyna (21 samples)	6.7 (±2.9)	0.27 (±0.13)	0.06 (±0.04)	3.4 (±1.2)	2.0 (±1.1)

Table 3 | Summary of CON and DON removal efficiencies from SE and reject water using the selected physical-chemical methods

Process	Description	DON removal, %	CON removal, %
<i>Secondary effluents</i>			
Adsorption using activated carbon	Dose: 50 g C/m ³ , contact time: 1 h	2–26	2–31
		4–36	2–49
		9–54	27–70
		13–66	20–82
		17–63	20–79
	Dose: 200 g C/m ³ , contact time: 1 h	2–34	2–38
		6–46	8–69
		8–60	33–80
		19–71	56–83
		24–69	56–87
	Dose: 500 g C/m ³ , contact time: 1 h	10–53	26–46
		13–63	40–70
		19–75	20–85
	contact time: 2 h	32–80	55–100
	contact time: 3 h	30–82	60–100
	contact time: 10 h		
Ultrafiltration	0.015 µm pore-size filter	3–20%	–
Ion exchange	Strongly acidic cation exchange resin dose: 15 g/m ³ , pH ≈ 7.0 contact time: 5 h	6–15%	3–8%
Coagulation using Fe ₂ (SO ₄) ₃	Rapid mixing: 60 s; flocculation: 900 s No pH correction Dose: 100 g/m ³ 200 g/m ³ 300 g/m ³	5–20	16–23
		13–24	23–47
		22–40	41–53
Coagulation using FeCl ₃	Rapid mixing: 60 s; flocculation: 900 s No pH correction Dose: 100 g/m ³ 200 g/m ³ 300 g/m ³	13–27	22–30
		23–41	31–50
		47–55	54–63
Coagulation using Ca(OH) ₂	Rapid mixing: 60 s; flocculation: 900 s Final pH <9.0 (100–150 g/m ³) or pH = 11.0–11.5 (450–500 g/m ³)	22–39%	13–39%
		37–56%	37–67%
<i>Reject waters</i>			
Ultrafiltration	0.015 µm pore-size filter	41–68% (AnD) 10–20% (TWAS)	–
Coagulation using FeCl ₃	Rapid mixing: 60 s; flocculation: 900 s No pH correction Dose: 100 g/m ³ 200 g/m ³ 300 g/m ³	21–28 (AnD)	17–28 (AnD)
		11–14 (TWAS)	23–28 (TWAS)
		34–45 (AnD)	40–53 (AnD)
		22–34 (TWAS)	28–51 (TWAS)
		62–67 (AnD)	63–71 (AnD)
		31–42 (TWAS)	44–51 (TWAS)

the efficiency was lower and reached approximately 45%. A colloidal fraction of organic nitrogen was removed with a higher efficiency than DON and reached nearly 100% for the secondary effluent from Koscierzyna WWTP. Evaluation of the effectiveness of CON removal in the Gdansk WWTP was impossible due to

very low concentrations (average 0.3 ± 0.2 g N/m³). The DON and CON removal efficiencies depended on both dose of activated carbon and contact time. The maximum removal efficiency was obtained for the dose of 500 g C/m³ and the contact time of a minimum of 5 h (Figure 1).

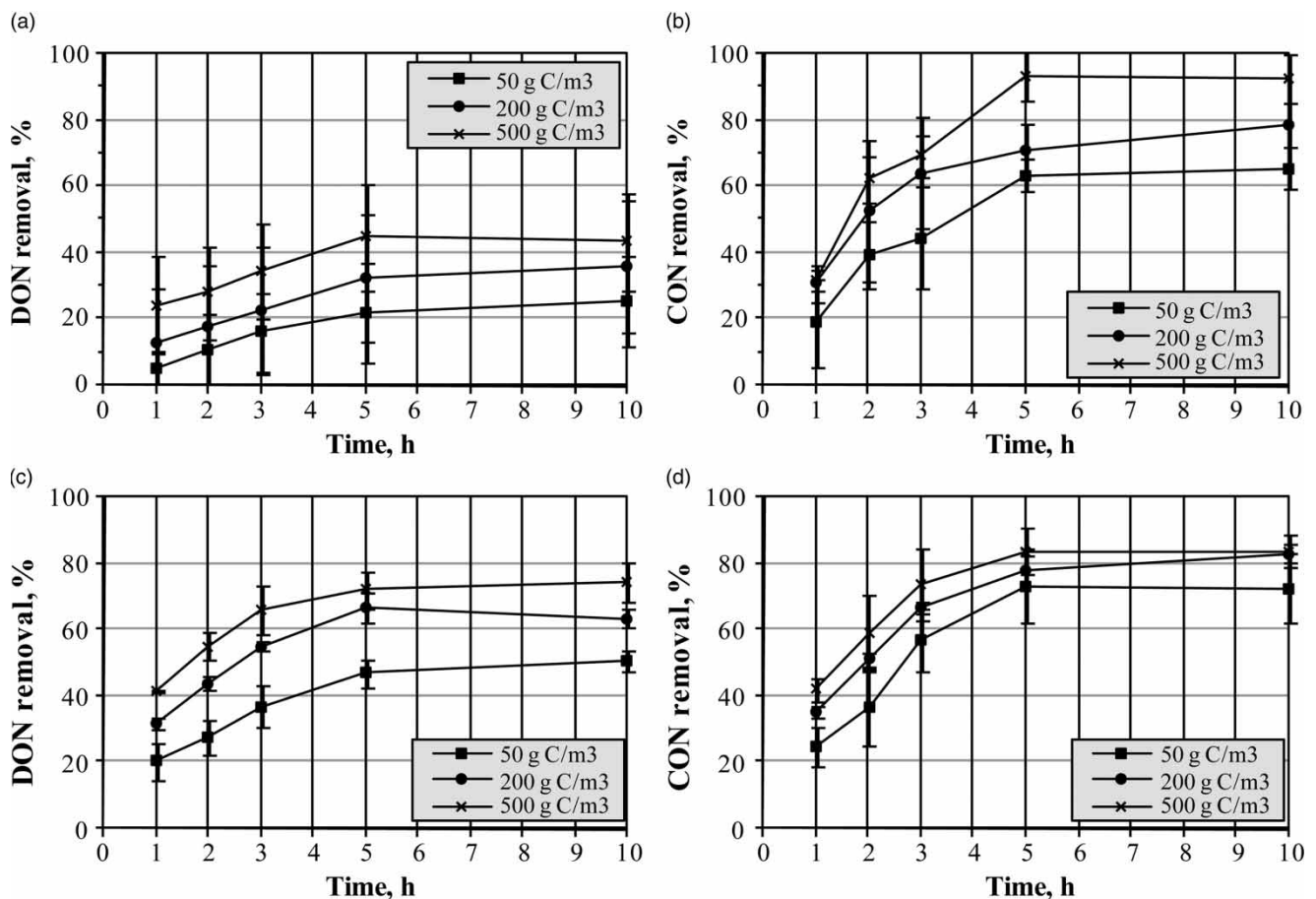


Figure 1 | Efficiency of DON and CON removal from SE by adsorption on activated carbon: (a), (b) Gdynia WWTP, (c), (d) Koscierzyna WWTP.

The removal efficiency of the dissolved solids (DS) by adsorption on activated carbon is significantly affected by non-polar properties and hydrophobic behavior of the DS relative to the solvent (Parkin & McCarty 1981). The biodegradation process tends to favor the removal of hydrophilic substances, which results in higher concentrations of hydrophobic, non-polar organic substances in SE. Based on the literature data, Pehlivanoglu-Mantas & Sedlak (2006) found that the removal efficiency for effluent DON removal ranged from 56 to 83%. Parkin & McCarty (1981) and later Pehlivanoglu-Mantas & Sedlak (2008) suggested that the activated sludge process produced hydrophilic DON. This results in a low efficiency of DON removal compared to other organic matter present in SE.

Coagulation

High efficiencies of DON removal from SE (up to 55%) were obtained by using coagulation with Fe(III) chloride (FeCl_3) and calcium hydroxide ($\text{Ca}(\text{OH})_2$) (at the final pH

of 11.0–11.5). Chemical precipitation with other coagulants, such as Fe(III) sulphate ($\text{Fe}(\text{SO}_4)_3$) and calcium hydroxide (final pH < 9.0), removed DON less efficiently (maximum 40%). In all the cases, the efficiency of coagulation with respect to DON removal was highly dependent on the coagulant doses.

The pH of the solution is very important for coagulation with iron and aluminum salts. The results presented by Bratby & Parker (2011) indicated that the effect of pH correction is small at doses up to $300 \text{ g FeCl}_3/\text{m}^3$. At higher doses, the authors observed a significant difference in the final concentration of DON in the experiments without correction of pH (increased concentration) and after correction to 5.5 (fixed concentration). The maximum removal efficiency for ferric(III) chloride coagulation was obtained in this study (51–55%) and by Bratby *et al.* (2008) (approximately 31%) for the same dose of $300 \text{ g FeCl}_3/\text{m}^3$ (Figure 2(a)). The use of iron sulphate, at the same doses, resulted in a significantly lower nitrogen removal efficiency (27–28% for the Gdynia and Koscierzyna WWTPs and 40% for the Gdansk

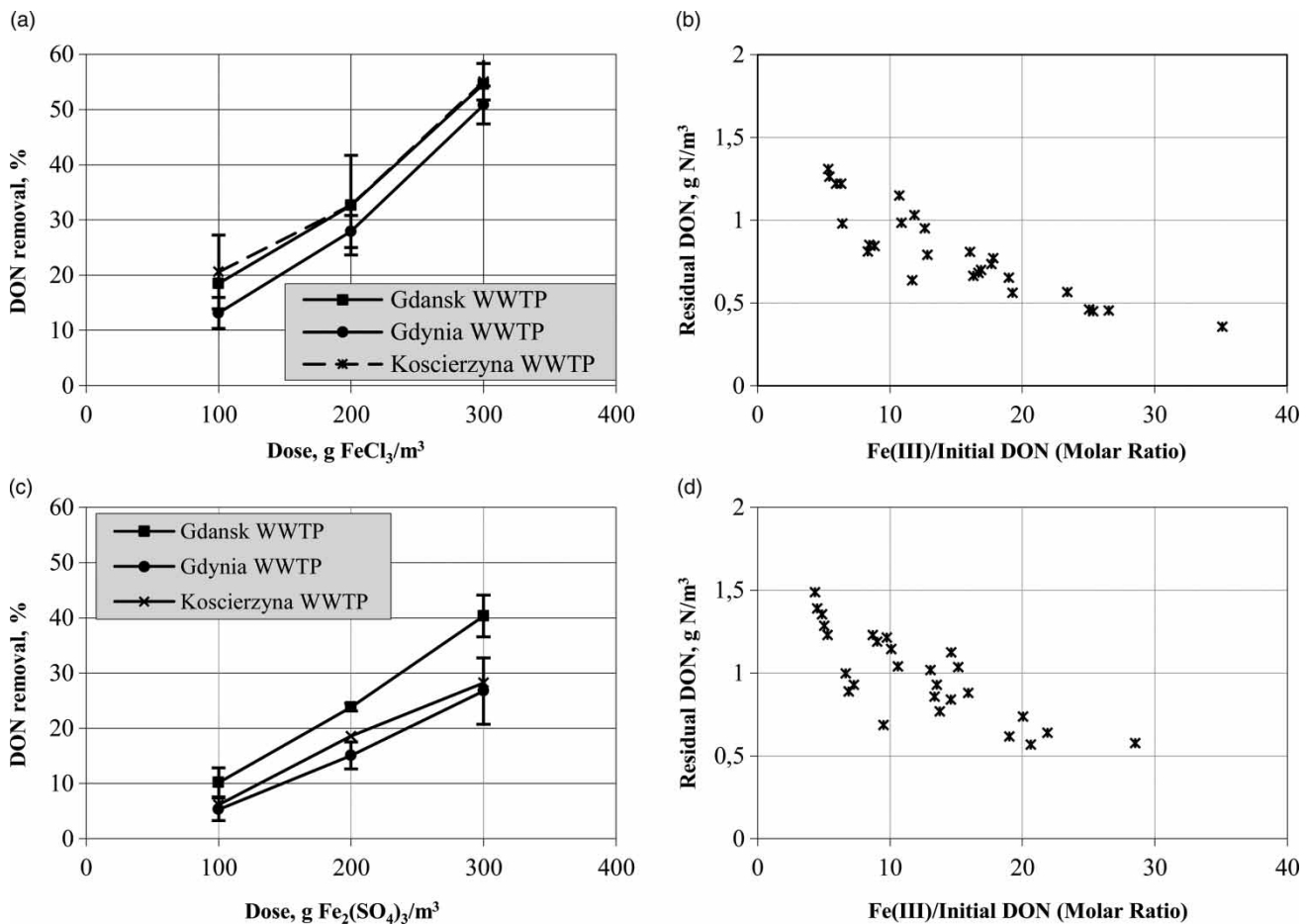


Figure 2 | Efficiency of DON removal from SE and effect of Fe(III)/Initial DON molar ratio on residual DON concentrations by coagulation: (a), (b) ferric(III) chloride, (c), (d) ferric(III) sulphate.

Table 4 | DON after 0.10 μm filtration ($\text{DON}_{0.1\mu\text{m}}$) and ultrafiltration (DON_{UF}) of the SE and reject water at the studied WWTPs (average \pm standard deviation)

WWTP	Number of tests	$\text{DON}_{0.1\mu\text{m}}$ g N/m ³	DON_{UF} g N/m ³	DON removal %
<i>Secondary effluents</i>				
Gdansk	5	1.74 \pm 0.40	1.56 \pm 0.39	10.0 \pm 6.4
Gdynia	5	1.41 \pm 0.22	1.21 \pm 0.14	13.1 \pm 4.5
Koscierzyna	3	1.03 \pm 0.30	0.92 \pm 0.26	10.4 \pm 2.6
<i>Reject water</i>				
Gdansk	23	28.59 \pm 12.27	12.21 \pm 4.58	56.7 \pm 9.2
Gdynia	18	24.85 \pm 7.79	11.51 \pm 3.34	52.8 \pm 7.1
Koscierzyna	16	3.42 \pm 1.08	2.85 \pm 0.86	16.4 \pm 3.3

WWTP) (Figure 2(c)). Such results could be affected by the lower dose (approximately 20%) of iron. In order to compare the results for the examined iron salts, correlations between the molar ratios of Fe(III)/initial DON

concentrations in SE and the residual DON concentration after coagulation are presented in Figure 2(b) (FeCl_3) and Figure 2(d) ($\text{Fe}_2(\text{SO}_4)_3$). In both cases, to achieve the final DON concentration of 0.5 g N/m³, the use of the molar

ratio of 20–30 Fe(III)/initial DON was required. In order to obtain such concentrations of the final DON by coagulation with aluminum, a significantly lower molar ratio of Al(III)/initial DON (=0.8) was required with the DON removal efficiencies of approximately 70% (Arnaldos & Pagilla 2010). Different results were presented by Bratby & Parker (2011). The authors noted that the molar ratio of Al(III)/removed DON of approximately 100 would be required in such a case.

The efficiency of DON removal from TWAS reject water from the Koscierzyzna WWTP, when using coagulation with Fe(III) chloride, was comparable with the efficiency for SE. The efficiency of this process with regard to the anaerobic digestion reject water was significantly higher and reached 65–70% for both DON and CON.

Lime precipitation at the final pH of 11.0–11.5 proved to be a very efficient process for DON and CON removal from SE. The average efficiencies for the three studied WWTPs were $48.1 \pm 5.5\%$ and $54.8 \pm 8.1\%$, for DON and CON, respectively. For comparison, much lower efficiencies for DON removal (<25%) were obtained by Chen *et al.* (2011) with the final pH of 11.3–11.5. In the study of Pehlivanoglu-Mantas & Sedlak (2006), the efficiency of DON removal by lime precipitation was $33 \pm 6\%$ (no information about the final pH was given).

Ion exchange

The ion exchange process with a strongly acidic cation exchange resin (without pH correction) resulted in relatively small efficiencies of DON removal (<15%) and CON removal (<10%) (Table 2). These results are consistent with the values presented by Pehlivanoglu-Mantas & Sedlak (2006) for DON removal by cation exchange with pH in the range of 7–8. These authors noted that a higher efficiency (up to 40%) can be achieved by lowering pH to 2.

Ultrafiltration

Ultrafiltration (0.015 μm) after 0.10 μm pre-filtration of SE resulted in relatively small amounts of DON removed. The average reductions in $\text{DON}_{0.10 \mu\text{m}}$ concentrations were 10–13% (Table 3). The low efficiency of ultrafiltration could result from the fact that DON consisted mainly of low molecular weight (LMW) compounds passed through a 10 kDa filter (0.005 μm) (Pehlivanoglu-Mantas & Sedlak 2008). Keller *et al.* (1978) and Parkin & McCarty (1981) also found that the percentage of LMW fraction (<1.8 kDa) in SE DON amounts to 50–66%.

Low efficiencies of ultrafiltration with respect to DON removal were also obtained for the TWAS reject water from Koscierzyzna WWTP. In contrast, ultrafiltration (0.015 μm) was found to be more effective for DON removal from anaerobic digester reject water (41–68%) (Table 4). This may indicate that DON in the examined reject water contained significant amounts of high molecular weight compounds (>30 kDa).

CONCLUSIONS

This study focused on verification of the results reported in the literature with respect to DON removal from SE by selected physical–chemical processes. A new contribution of the study is the removal effectiveness of those processes with regard to the CON fraction (0.1–1.2 μm) in SE. Novel findings also refer to organic nitrogen removal from reject water including the fractionation and removal potential of organic nitrogen by ultrafiltration and coagulation with iron(III) chloride. Due to the importance of the recovery of nutrients from wastewater, the examined processes certainly become an important area for further research.

Furthermore, based on the results of this study, the following findings may be emphasized.

- Adsorption on granulated activated carbon was most efficient and allowed removal up to 80% and 100% of DON and CON, respectively, from SE.
- High efficiencies of DON removal from SE (up to 55%) were also obtained when using coagulation with Fe(III) chloride and calcium hydroxide at the final pH = 11.0–11.5.
- Chemical precipitation with other coagulants, such as Fe(III) sulphate and calcium hydroxide (at the final pH < 9.0), removed DON less efficiently (<40%).
- The efficiency of DON removal by coagulation with Fe(III) chloride was comparable for TWAS reject water and SE. The efficiency of this process with regard to SDL was significantly higher, i.e., 65–70% for both DON and CON.
- The ion exchange process with a strongly acidic cation exchange resin (without pH correction) resulted in relatively small efficiencies of DON removal (<15%) and CON removal (<10%).
- Ultrafiltration (0.015 μm) after 0.10 μm pre-filtration of SE and TWAS reject water resulted in a relatively low efficiency of DON removal (10–13% and 10–20%,

respectively). In contrast, ultrafiltration was found to be more effective for DON removal from SDL (41–68%).

ACKNOWLEDGEMENTS

This research has been financially supported by the Polish National Science Centre under the grant no. N 523 621 439 and by the Polish Ministry of Science and Higher Education under the grant no. WERF/45/2007 and as a part of collaboration with the Water Environment Research Foundation Program:02-CTS-1a.

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First received 18 January 2014; accepted in revised form 28 May 2014. Available online 11 June 2014