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Dissolved and colloidal organic nitrogen removal from WWTP 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 to remove from the SE 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 Fe(III) chloride and calcium hydroxide at the final pH=11.0-11.5. The efficiency of DON removal from thickening waste activated sludge (TWAS) reject water, obtained using coagulation with Fe(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. 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 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%).

Keywords

nitrogen removal; dissolved organic nitrogen (DON); colloidal organic nitrogen (CON); ultrafiltration; adsorption; coagulation; ion exchange

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 (LoT) 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/m^3 with a significant contribution of organic N (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) (Gajewska, 2011; Pagilla et al., 2006; Pehlivanoglu and Sedlak, 2004; WERF, 2008). 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/m^3, 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/m^3. 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 and 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), 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 and McCarty, 1981; Westerhoff and Mash, 2002; Pehlivanoglu-Mantas and Sedlak, 2006).

Reject water, generated in WWTPs, comprises sludge digester liquors (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 their significant effects to main-stream treatment lane. The sludge digester liquors 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 at al. (2006) found that ammonia nitrogen concentrations in digester reject water varied in a wide range, i.e. 450-1700 g NH_4-N/m^3, at flow rates 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 few information about nitrogen concentrations in reject water from waste activated sludge thickeners (WAST).

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 sludge digester liquors and effluents of full-scale BNR activated sludge systems. The examined processes were selected based on the potential of 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 the size exceeding 100,000 PE, and the other plant (Koscierzyna) is a medium-size facility with the 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.

Table 1. Basic characteristics of the studied WWTPs

WWTP	Size	Flow rate	SRT	Configuration of bioreactor	Sludge handling
	PE	m^3/d	d		
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 thickening waste activated sludge

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 eight-hour working time sludge dewatering devices 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 organic N (PON) (>1.2 μm), CON further 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 tests experiments

Laboratory experiments were carried out in a simple experimental apparatus consisting of several glass beakers (V = 1 dm^3) and magnetic stirrers with a controlled rotary speed. Technical pure ferric(III) chloride ($FeCl_3$) and ferric(III) sulphate ($Fe_2(SO_4)_3$) 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. The multiple doses of iron compounds were used, including 100, 200 and 300 g/m^3, which corresponded to the doses of 34.5, 68.9 and 103.4 g Fe/m^3 of ferric(III) chloride or 28, 56 and 84 g Fe/m^3 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 tertiary treatment systemss. After dosing the reagents, a rapid mixing was performed for 60 s, and then followed by a slow mixing for 900 s in order to ensure optimum conditions for flocculation. Finally, the samples were allowed to settle 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, including 50, 200 and 500 g C/m^3. 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/m^3 of cation resin was also used without pH correction (pH ≈ 7.0)and after 5-hour contact time.

Three replicate experiments at 20 ^{0}C were determined for each reagents and doses.

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 a 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 polycarbon filters (Whatman, Kent, UK).

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 (NH_4-N, NO_3-N and NO_2-N) were determined in the filtrate using Xion 500 spectrophotometer (Dr Lange GmbH, Berlin, Germany). The analytical procedures, which were adopted by Dr Lange and SHIMADZU Corporation, followed the Standard Methods for Examination of Water and Wastewater (APHA, 2005). DON concentrations were calculated as a 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 (NH_4-N, NO_3-N and NO_2-N). CON concentrations were calculated as a 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 are presented in Table 2. Results of the DON and CON removal efficiencies from secondary effluents and reject water with the selected physical-chemical processes are summarized in Table 3.

Table 2. The average inorganic N, DON and CON concentrations (± SD) in the sludge digester liquors and secondary effluents in the studied WWTPs

WWTP	NH_4-N	NO_3-N	NO_2-N	DON	CON
	g N/m^3	g N/m^3	g N/m^3	g N/m^3	g N/m^3
Secondary effluents					
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)
Reject water					
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)

Activated carbon adsorption

Adsorption on activated carbon was most efficient and allowed to remove up to 80% of DON in secondary effluents from the Gdansk WWTP (the initial average DON concentration = 2.0±0.07 g N/m^3) and Koscierzyna WWTP (1.0±0.29 g N/m^3), whereas for the Gdynia WWTP (1.5±0.18 g N/m^3) 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/m^3). The DON and CON removal efficiencies were depended on both dose of activated carbon and contact time. The maximum removal efficiency was obtained for the dose of 500 g C/m^3 and the contact time of min. 5 hours (Figure 1).

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 and 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 secondary effluents. Based on the literature data, Pehlivanoglu-Mantas and Sedlak (2006) found that the removal efficiency for effluent DON removal ranged from 56 to 83%. Parkin and McCarty (1981) and later Pehlivanoglu-Mantas and 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 secondary effluents.

Table 3. Summary of CON and DON removal efficiencies from secondary effluents and reject water using the selected physical-chemical methods

Process	Description	DON removal, %	CON removal, %
	Secondary effluents		
Adsorption using activated carbon	dose: 50 gC/m^3, contact time: 1h	2-26	2-31
	contact time: 2h	4-36	2-49
	contact time: 3h	9-54	27-70
	contact time: 5h	13-66	20-82
	contact time: 10h	17-63	20-79
	dose: 200 gC/m^3, contact time: 1h	2-34	2-38
	contact time: 2h	6-46	8-69
	contact time: 3h	8-60	33-80
	contact time: 5h	19-71	56-83
	contact time: 10h	24-69	56-87
	dose: 500 gC/m^3, contact time: 1h	10-53	26-46
	contact time: 2h	13-63	40-70
	contact time: 3h	19-75	20-85
	contact time: 5h	32-80	55-100
	contact time: 10h	30-82	60-100
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	5-20	16-23
	200 g/m^3	13-24	23-47
	300 g/m^3	22-40	41-53
Coagulation using $FeCl_3$	rapid mixing: 60 s; flocculation: 900 s no pH correction		
	dose: 100 g/m^3	13-27	22-30
	200 g/m^3	23-41	31-50
	300 g/m^3	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)	22-39 %	13-39 %
	or pH = 11.0-11.5 (450-500 g/m^3)	37-56 %	37-67 %
	Reject waters		
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	21-28 (AnD) 11-14 (TWAS)	17-28 (AnD) 23-28 (TWAS)
	200 g/m^3	34-45 (AnD) 22-34 (TWAS)	40-53 (AnD) 28-51 (TWAS)
	300 g/m^3	62-67 (AnD) 31-42 (TWAS)	63-71 (AnD) 44-51 (TWAS)

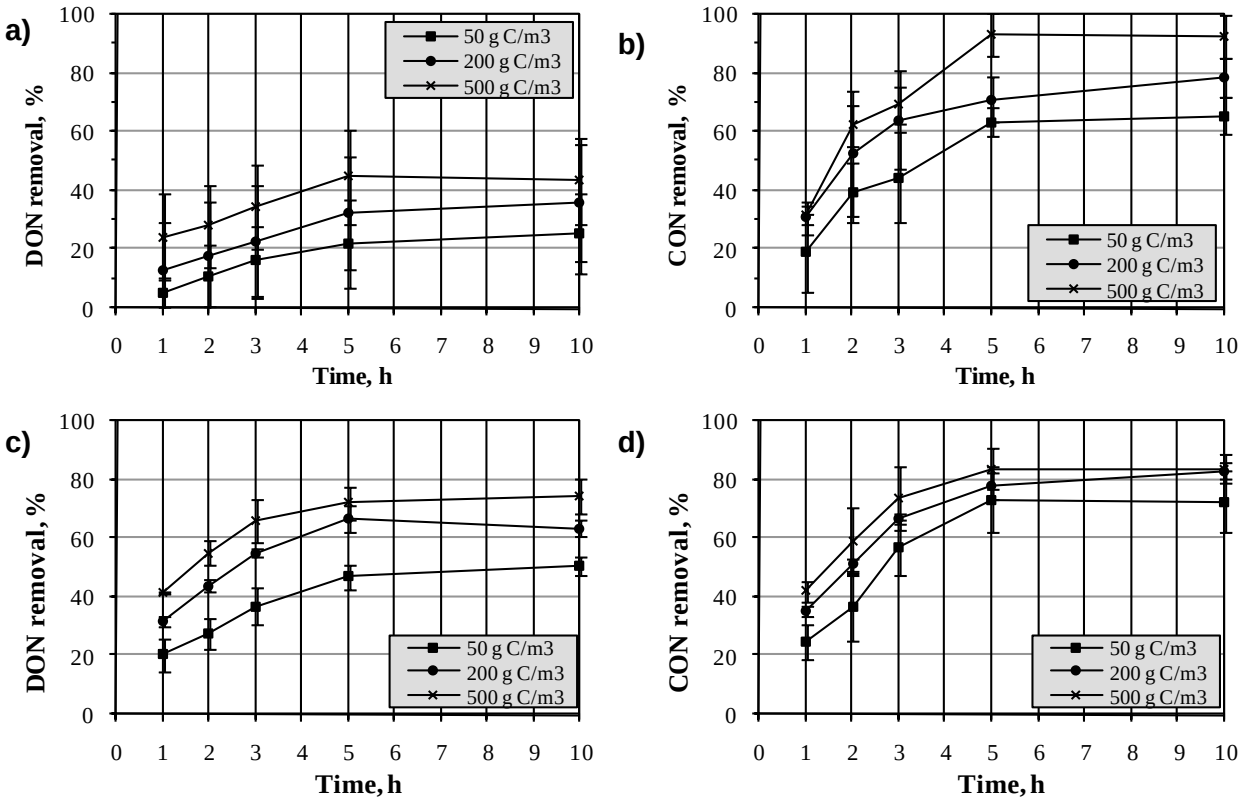

Figure 1. Efficiency of DON and CON removal from secondary effluents by adsorption on activated carbon (a-b) Gdynia WWTP, (c-d) Koscierzyna WWTP

Coagulation

High efficiencies of DON removal from secondary effluents (up to 55%) were obtained by using coagulation with Fe(III) chloride ($FeCl_3$) and calcium hydroxide ($Ca(OH)_2$) (at the final pH ratio of 11.0-11.5). Chemical precipitation with other coagulants, such as Fe(III) sulphate ($Fe(SO_4)_3$) and calcium hydroxide (the final pH<9.0), removed DON less efficiently (max. 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 and Parker (2011) indicated that the effect of pH correction is small at doses up to 300 g $FeCl_3/m^3$. 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 and Parker (2008) (approx. 31%) for the same dose of 300 g $FeCl_3/m^3$ (Figure 2a). 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 WWTP) (Figure 2c). 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 Fe(III)/ initial DON concentrations in secondary effluents to the residual DON concentration after coagulation are presented in Figure 2b ($FeCl_3$) and Figure 2d ($Fe_2(SO_4)_3$). In both cases, to achieve the final DON concentration of 0.5 g N/m^3, 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 aluminium, a significantly lower molar ratio Al(III)/initial DON (= 0.8) was required with the DON removal efficiencies of approximately 70% (Arnaldos and Pagilla, 2010). Different results were presented by Bratby and Parker (2011). The authors noted that

the molar ratio Al(III)/removed DON of approximately 100 would be required in such a case.

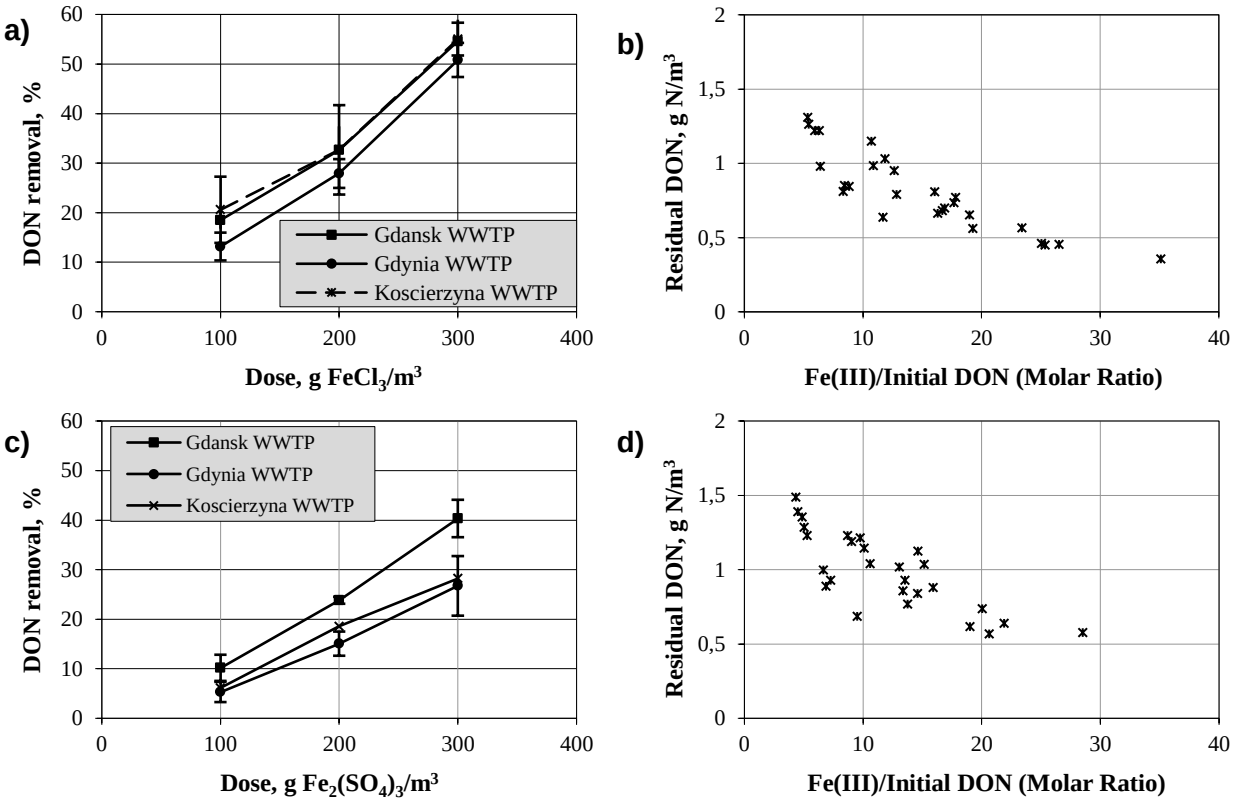

Figure 2. Efficiency of DON removal from secondary effluents and effect of Fe(III)/Initial DON molar ratio on residual DON concentrations by coagulation (a-b) ferric(III) chloride, (c-d) ferric(III) sulphate

The efficiency of DON removal from TWAS reject water from the Koscierzyna WWTP, when using coagulation with Fe(III) chloride ($FeCl_3$), was comparable with the efficiency for secondary effluents. 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 ratio of 11.0-11.5 proved to be a very efficient process of DON and CON removal from secondary effluents. The average efficiencies for the three studied WWTPs were 48.1±5.5 % and 54.8±8.1 %, for DON and CON, respectively. For comparison, much lower efficiencies for DON removal (<25%) were obtained by Cheo at al. (2011) with the final pH of 11.3-11.5. In the study of Pehlivanoglu-Mantas and Sedlak (2006), the efficiency of DON removal by lime precipitation was 33±6% (no information about the final pH was given).

Ion exchange

Ion exchange process with 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 and Sedlak (2006) for DON removal by cation exchange with pH in the range of 7-8. These authorsnoted 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 secondary effluents resulted in relatively small amounts of DON removed. The average reductions in $DON_{0.10\mu m}$ 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 and Sedlak, 2008). Keller et al. (1978) and Parkin and McCarty (1981) also found that the percentage of LMW fraction (<1.8 kDa) in secondary effluents DON amounts to 50-66%.

Low efficiencies of ultrafiltration with respect to DON removal were also obtained for the TWAS reject water from Koscierzyna 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 (HMW) compounds (> 30 kDa).

Table 4. DON after 0.10 µm filtration ($DON_{0.1\mu m}$) and ultrafiltration (DON_{UF}) of the secondary effluents and reject water at the studied WWTPs (average ± standard deviation)

WWTP	number of tests	$DON_{0.1\mu m}$	DON_{UF}	DON removal
		gN/m^3	gN/m^3	%
secondary effluents				
Gdansk	5	1.74±0.40	1.56±0.39	10.0±6.4
Gdynia	5	1.41±0.22	1.21±0.14	13.1±4.5
Koscierzyna	3	1.03±0.30	0.92±0.26	10.4±2.6
reject water				
Gdansk	23	28.59±12.27	12.21±4.58	56.7±9.2
Gdynia	18	24.85±7.79	11.51±3.34	52.8±7.1
Koscierzyna	16	3.42±1.08	2.85±0.86	16.4±3.3

CONCLUSIONS

This study focused on verification of the results reported in the literature with respect to DON removal from secondary effluents by selected physical-chemical processes. A new contribution of the study are the removal effectiveness of those processes with regard to the CON fraction (0.1-1.2 µm) in secondary effluents. 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) chlorine. Due the importance of the recovery of nutrients from wastewater, the examined processes certainly become an important area for further research.

Furthrmore, based on the results of this study, the following findings may be emphasized:

- Adsorption on granulated activated carbon was most efficient and allowed to remove up to 80% and 100% of DON and CON, respectively, from secondary effluents.
- High efficiencies of DON removal from secondary effluents (up to 55%) were also obtained when using coagulation with Fe(III) chloride ($FeCl_3$) and calcium hydroxide ($Ca(OH)_2$) at the final pH=11.0-11.5.
- Chemical precipitation with another 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 andsecondary effluents. The efficiency of this process with regard to sludge

digester liquors was significantly higher, i.e. 65-70% for both DON and CON.

- Ion exchange process with strongly acidic cation exchange resin (without pH correction) resulted in a relatively small efficiencies of DON removal (<15%) and CON removal (<10%).
- Ultrafiltration (0.015 μm) after 0.10 μm pre-filtration of secondary effluents 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 sludge digester liquors (41-68%).

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