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Chemical precipitation and ammonia air stripping as effective pre-treatment methods before membrane filtration of co-digestion effluents

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ABSTRACT

The aim of the research presented was to develop an effective treatment method of highly polluted post-digestion effluents, to a degree allowing the treated waters to be directly released into a natural reservoir. The post-digestion effluents characterized by varied ammonia–nitrogen content underwent high-pressure membrane filtration (nanofiltration [NF], reverse osmosis [RO]) and chemical precipitation (struvite)/ammonia stripping as pre-treatment steps. The application of struvite precipitation and ammonia stripping turned out to be an effective method of binding nitrogen and phosphorus compounds from post-digestion liquors. Waters met all discharge limits after struvite precipitation at stoichiometric ratio of reagents, ammonia stripping of 2–8 h (depending on the initial NH_4^+ concentration) and RO as a post-treatment. Introducing air stripping between struvite precipitation and membrane filtration allowed to reduce the amount of reagents used during struvite precipitation in order to remove the ammonia–nitrogen below discharge limit. The waters purified with NF as a post-treatment did not meet the discharge limits; however, their reuse as technological waters should be taken into consideration.

Keywords: Nanofiltration; Reverse osmosis; Struvite; Ammonia stripping; Post-digestion effluents

1. Introduction

An increased interest in fuels based on vegetable raw materials has been recently observed. The main waste products generated during rape processing into biodiesel are rapeseed cakes and waste glycerin generated at the oil extraction from seeds and oil esterification stage, respectively. Processing of on ton of rape seed generates 650 kg rapeseed cake and 100 kg of glycerol fraction [1]. Oil cakes are usually used as components of animal feed. Nowadays, studies are conducted towards the usage of oil cakes as feedstock in the pyrolysis and biotechnological processes, including production of enzymes, biological

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components [2,3]. Whilst, glycerol fraction, forming the second product of biofuel production, has broad business applications, e.g. in pharmaceutical, cosmetic and plastic industry. For example, 1,3-propanediol is produced from glycerol and the application of micro-organisms. This compound can replace ethylene glycol polyesters [4]. However, in order to obtain a high quality glycerol, depending on the presence of impurities therein, it is necessary to pre-treat the glycerol fraction, which is not always economically justified. In practice, glycerol fraction very often, like cakes, constitutes a component of feed for animals.

Nowadays, more and more frequently, by-products generated during biodiesel production from rapeseed are used as a feedstock for anaerobic digestion (AD). However, these waste products, despite a high content of readily biodegradable components under anaerobic conditions, cannot be used as a single fermentation substrate. In case of rapeseed cakes, the limiting factor is a solid state, while the glycerol fraction is characterized by low pH ~5.5 and small content of nitrogen, which is necessary to preserve the life activity of micro-organisms involved in biochemical transformations [5,6]. However, such by-products can be co-digested together with other substrates, for example, sewage sludge or animal excretes. On the one hand, application of sewage sludge or animal wastes as main feedstock is associated with certain benefits. They provide buffer capacity and the appropriate content of macro- and micro-nutrients. On the other hand, due to the more varied feedstock composition in the co-digestion concept and higher organic loading of the bioreactors, the generated post-digestion liquors contain a higher concentration of organic compounds and nutrients, which have a negative impact on their treatment [7,8]. Post-digestion liquors has a negative impact on the wastewater treatment processes (unfavourable C/N ratio), if directed back to the biological treatment facility. What is more, individual unit processes (e.g. coagulation, ammonia desorption, advanced oxidation, biological methods, immobilized bioprocesses and membrane techniques) do not ensure a complex contaminant removal, mostly due to the high contents of ammonia–nitrogen and organic matter [9–17]. Taking into account the above, it is necessary to apply a multi-step treatment concept so that the treated liquors can be released directly to the natural reservoir. In our previous study, the struvite precipitation targeted at ammonia–nitrogen binding coupled with membrane processes (reverse osmosis [RO] or nanofiltration [NF] + RO) turned out to be an effective treatment method of co-digestion effluents. However, in order to achieve the permeate quality allowing its direct release to the natural receiver, it was necessary to apply an increased molar ratio of reagents ($Mg^{2+}:NH_4^+:PO_4^{3-} = 1.5:1:1.5$) during precipitation (pre-treatment) or two-step membrane process (NF + RO) after struvite precipitation at stoichiometric ratio of reagents [18]. An interesting option of the pre-treatment seems to be the ammonia recovery by air stripping conducted after struvite precipitation, which can minimize the amount of reagents used in the struvite precipitation and/or eliminate the application of the two-step membrane process.

In the present study, an effective treatment method of highly polluted post-digestion effluents was presented. The post-digestion effluents characterized by varied ammonia–nitrogen content underwent high-pressure membrane filtration (NF, RO) and chemical precipitation (struvite)/ammonia stripping as pre-treatment steps. It was assumed that treated waters will be directly released into a natural reservoir. In particular, an attempt was made to establish the influence of pre-treatment methods applied on the effectiveness of subsequent NF and RO.

2. Materials and methods

2.1. Research material

All post-digestion liquors were obtained during digestion processes performed in laboratory conditions. The experiments were conducted for effluents after sewage sludge mesophilic digestion as well as co-digestion of (w/w): 3% of waste glycerol/97% of sewage sludge and 2% of rapeseed cake/98% of sewage sludge. By-products after biodiesel production used as a co-substrates, i.e. waste glycerol and rapeseed cake, were taken from a local biorefinery. Secondary sludge used as a main feedstock during mesophilic AD originated from a full-scale municipal treatment plant—based on Enhanced Biological Nutrients Removal. The proportions of waste glycerol and rapeseed cake in the co-digestion mixtures were established in a separate studies; taking into account the effectiveness of the methane production and overall stability of the AD [19]. Effluents after the co-digestion were dewatered mechanically and filtered to remove solid particles present after AD processes. Table 1 presents characteristics of the liquors used in the present study.

2.2. Ammonium magnesium phosphate (struvite) precipitation

Struvite precipitation was conducted in a 10-dm^3 tank, working in a batch mode. The process was carried out at constant temperature of 20˚C and pH of 9.5

Table 1
Characteristics of the post-digestion effluents

	Post-digestion liquors		
Parameter	Sewage sludge	Waste glycerine + sludge	Rapeseed cake + sludge
pH	7.8 (0.2)*	7.2 (0.1)	7.3 (0.1)
NH_4^+, $mgNH_4/dm^3$	1,062 (31)	1,532 (83)	1960 (88)
TN, mg/dm^3	1,150 (21)	1,655 (76)	2080 (86)
PO_4^{3-}, $mgPO_4^{3-}/dm^3$	369 (23)	359 (25)	456 (34)
TP, mg/dm^3	123 (11)	120 (10)	168 (12)
Mg^{2+}, mg/dm^3	39.5 (1.9)	41.8 (3.2)	49.5 (4.5)
Ca^{2+}, mg/dm^3	14.9 (1.9)	15.5 (2.3)	17.8 (4.2)
COD, mgO_2/dm^3	1,182 (60)	1,475 (84)	2,848 (175)
BZT_5, mgO_2/dm^3	774 (35)	943 (45)	1867 (95)
TS, mg/dm^3	105 (8)	120 (17)	132 (15)

*()—standard deviation.

[20–22]. The pH value of post-digestion liquors was adjusted by means of 1 M NaOH. Retention time of post-digestion liquors in a crystallizer amounted to 24 h. The process was conducted for the stoichiometric ratio of Mg^{2+}, NH_4^+ and PO_4^{3-} as well as 20, 40 and 60% excess amounts of magnesium and phosphorus (Mg^{2+}: NH_4^+: PO_4^{3-} = 1.2:1:1.2; Mg^{2+}: NH_4^+: PO_4^{3-} = 1.4:1:1.4; Mg^{2+}: NH_4^+: PO_4^{3-} = 1.6:1:1.6). The phosphorus and magnesium were augmented by H_3PO_4 and MgO, respectively. The effectiveness of struvite precipitation was based on the degree of NH_4^+ removal compared to its initial content in the post-digestion liquors. The experimental unit used for struvite precipitation is presented in Fig. 1.

2.3. Ammonia stripping

For ammonia stripping, two identical reactors with working volume of 10 dm^3 were used. The process was conducted in a batch mode, at constant temperature of 20°C and pH of 11.5. The free-ammonia content (NH_3) in samples before ammonia stripping was calculated based on the standard formula presented in [23]. Post-digestion liquors after struvite precipitation

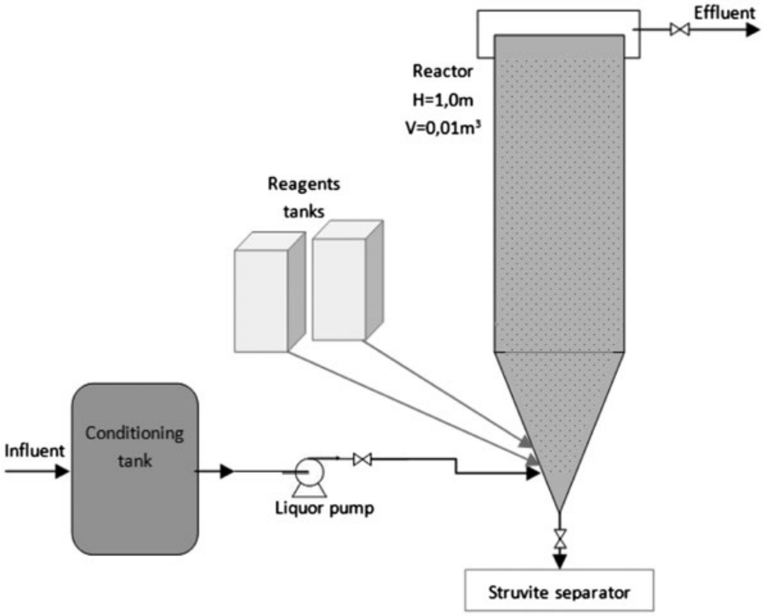

Fig. 1. Experimental unit used for struvite precipitation.

were used as feedstock. The retention time of feedstock in the stripping reactor amounted between 1 and 8 h, which corresponded with the stripping tower loading of waters treated between 1.25 and 10.0 $dm^3/dm^3/h$. The aeration rate of 50 $dm^3/h/dm^3$ was applied during the whole experiment. The pH of liquors after ammonia stripping was readjusted to about 6.8 using 2 M HCl. The liquors were stored at 4°C prior to membrane filtration processes. The experimental unit used for ammonia stripping is presented in Fig. 2.

2.4. Membrane filtration

Membrane filtration (NF and RO) was conducted in the device (type GH-100-400) produced by a US-based company, Osmonics. The device worked in the dead-end mode on flat-sheet membranes. The processes were conducted under transmembrane pressure of 2 MPa. The rotary velocity of the stirrer was maintained at the level of 200 rpm. The RO and NF processes were conducted with the application of composite polyamide membrane (type ADF) and cellulose membrane (type SF 10), respectively. Detail characteristics of the membranes used is presented in our previous study [18].

The efficiency of the membrane processes was determined according to the following formula (1):

$$J_v = \frac{V}{F \cdot t}, \quad m^3/m^2 \cdot s \qquad (1)$$

where J_v is the volumetric permeate flux, $m^3/m^2\,s$; V is the the volume of permeate collected after the particular period of time (t), m^3; F is the active surface of membrane, m^2; t is the filtration time, s.

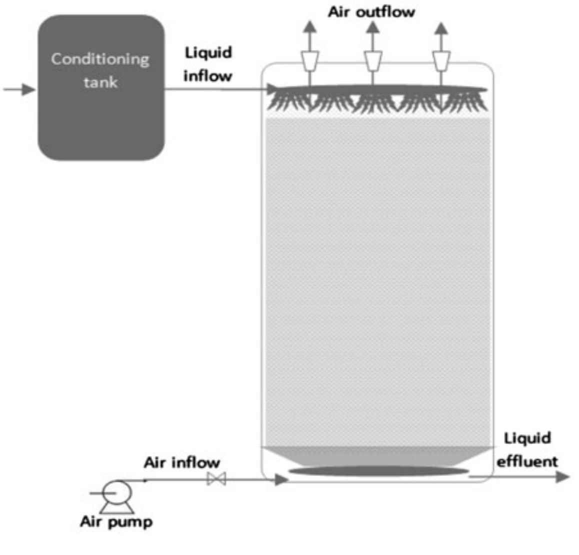

Fig. 2. Experimental unit used for ammonia stripping from waters after struvite precipitation.

2.5. Analytical methods

The scope of the analyses conducted encompassed: pH measurement and determinations of total suspension (TS), chemical oxygen demand (COD), biochemical oxygen demand (BOD_5), ammonia–nitrogen (NH_4^+) and total nitrogen (TN, Kjeldahl nitrogen), phosphates (PO_4^{3-}), total phosphorus (TP), as well as calcium and magnesium [24,25]. The images of struvite crystals were taken by means of a Nikon Alphaphot microscope (×100), coupled with a digital camera. Heavy metals content in the solid fraction after struvite precipitation was determined by inductively coupled plasma-atomic emission spectrometry. Results were presented as averages values based on four replicates. Standard deviations are presented in parentheses.

2.6. Statistical analysis

The ammonia concentration achieved during different conditions of struvite precipitation (varied ratio of reagents) and ammonia stripping (varied time of stripping) were compared statistically. One-way ANOVA test followed by Tukey's HSD test was used for multiple comparisons, with the level of significance set at <0.05. Normal distribution and homogeneity of variances were tested by Shapiro-Wilk and Levene test, respectively. Data significantly equivalent were indicated by the same letters.

3. Results and discussion

3.1. Characteristics of post-digestion liquors

Anaerobic digestion of organic wastes leads to the release of nutrients and organic substances to the liquid fraction. Due to the more varied feedstock, the liquors after co-digestion of sewage sludge with other wastes usually contain more such substances in the liquid fraction compared to liquor after the only sewage sludge digestion [7,8]. Concentration of NH_4^+ in the digestate increased by 48 and 87% after waste glycerol and rapeseed addition as co-substrates, respectively. The liquor after sewage sludge digestion and its co-digestion with waste glycerol contained comparable amounts of phosphorus compounds. In both of them, the soluble phosphorus (PO_4^{3-}) accounted for about 98% of TP. Whilst, liquors originated from rapeseed digestion as a co-substrate contained 24–27% more soluble phosphorus. What is more, the latter compounds accounted for only 87% and the remaining part consisted of organic phosphorus, which can be accounted for specific nature of the co-substrate. The COD concentration increased from 25% (after glycerol addition) to more than doubled concentration (after rapeseed addition) (Table 1). Such liquors require complex treatment solutions, as even the application of highly effective RO did not allow to reduce the level of contaminants so that the treated liquors can be released directly to a natural reservoir [12,18,26].

3.1.1. Chemical method of struvite precipitation

3.1.1.1. Effectiveness of ammonia–nitrogen removal. Firstly, the post-digestion waters were treated by the chemical method of struvite precipitation. Under stoichiometric molar ratio of Mg^{2+}, NH_4^+ and PO_4^{3-}, the degree of NH_4^+ removal reached the value of 85–89%, while the residual NH_4^+ amounted to between 120 mg/l (liquor after sewage sludge digestion) and 214–300 mg/l (liquors after co-digestion processes) (Table 2, Fig. 3). As the higher dosage of $Mg^{2+}$and PO_4^{3-} was added, the degree of NH_4^+ removal significantly increased. This is in accordance with previous studies [27]. In case of liquors after sewage sludge digestion, the highest degree of NH_4^+ removal was in the range of 98% and was achieved after application of 40% increased amounts of $Mg^{2+}$and PO_4^3. Further increase of magnesium and phosphorus did not have a positive influence on the NH_4^+ removal (data equivalent statistically, Table 2). Whilst, the highest NH_4^+ removals for co-digestion liquors were in the range of 94–96% (Mg^{2+}:NH_4^+:PO_4^{3-} = 1.4:1:1.4) and 96–97% (Mg^{2+}:NH_4^+:PO_4^{3-} = 1.6:1:1.6) (Fig. 3). Significantly lower NH_4^+ residual content was recorded in case of 60% excess dosage of Mg^{2+} and PO_4^{3-} compared to 40% excess dosage of Mg^{2+} and PO_4^{3-} (statistically significant difference). In case of 60% increased Mg^{2+} and PO_4^{3-} ratio, the weight ratio of (PO_4^{3-}/NH_4^+) reached the value of 7.5–16.4, i.e. above the value resulted from struvite formula (5.27), so further increase of phosphates turned out to be unjustifiable.

As the MgO was used in the form of Mg^{2+}, the struvite precipitation caused an additional decrease in COD concentration (Table 2). The effect was ascribed to MgO coagulative properties. It was assumed that a part of organic substances might have been absorbed by generated $Mg(OH)_2$ and precipitated together with struvite. A simple relationship was observed—the higher the dosage of MgO used, the higher the degree of COD removal was (Fig. 3). The MgO properties allow for a pH value increase towards the level needed for effective course of struvite precipitation. Lack of post-reaction chlorides or sulphates in the solution after precipitation makes MgO an attractive Mg^{2+} source. The main weakness of MgO is its slow solubility and thus longer reaction times compared to $MgCl_2$ or $MgSO_4$ [28,29].

3.1.1.2. Chemical composition, quality and structure of the struvite precipitated. Regardless of the origin of the post-digestion liquors, chemical composition of struvite precipitated did not differ significantly from the theoretical proportion of particular ingredients in the struvite formula (Table 3). Slightly higher contents of phosphorus and magnesium might have been the result of other compounds precipitated together with struvite. Moreover, phosphates can react with calcium, which is a major interfering ion affecting the deposit composition and decreasing struvite purity [30]. Since magnesium was added in order to comply with struvite chemical formula, it was assumed that interactions between calcium and phosphorus will not lead to a significant phosphate decrease in the solution. Moreover, the struvite contained negligible amounts of heavy metals (Table 3), which can be

Table 2
Characteristics of liquor after struvite precipitation (the same letters showing that the values are not significantly different $p > 0.05$)

	Post-digestion waters								
	Sewage sludge			Waste glycerine + sludge			Rapeseed cake + sludge		
Molar ratio Mg^{2+}:NH_4^+:PO_4^{3-}	NH_4^+ (mg/dm^3)	PO_4^{3-} (mg/dm^3)	COD (mg/dm^3)	NH_4^+ (mg/dm^3)	PO_4^{3-} (mg/dm^3)	COD (mg/dm^3)	NH_4^+ (mg/dm^3)	PO_4^{3-} (mg/dm^3)	COD (mg/dm^3)
1:1:1	120d (13)*	52.1 (6.1)	978 (75)	214b (15)	54.1 (4.0)	1,225 (90)	307a (13)	68.2 (3.1)	2,345 (145)
1.2:1:1.2	64.1f (10)	75.2 (6.0)	894 (58)	114d (19)	95.2 (7.5)	1,116 (55)	173c (15)	123 (9.1)	2,185 (170)
1.4:1:1.4	22.2h (2.1)	123 (13)	798 (46)	66.4f (5.2)	131 (5)	979 (77)	109d (7)	170 (14)	1979 (113)
1.6:1:1.6	23.1h (3.5)	377 (22)	705 (40)	50.1g (4.3)	491 (11)	912 (39)	83.2e (5.2)	620 (27)	1829 (126)

*()—standard deviation.

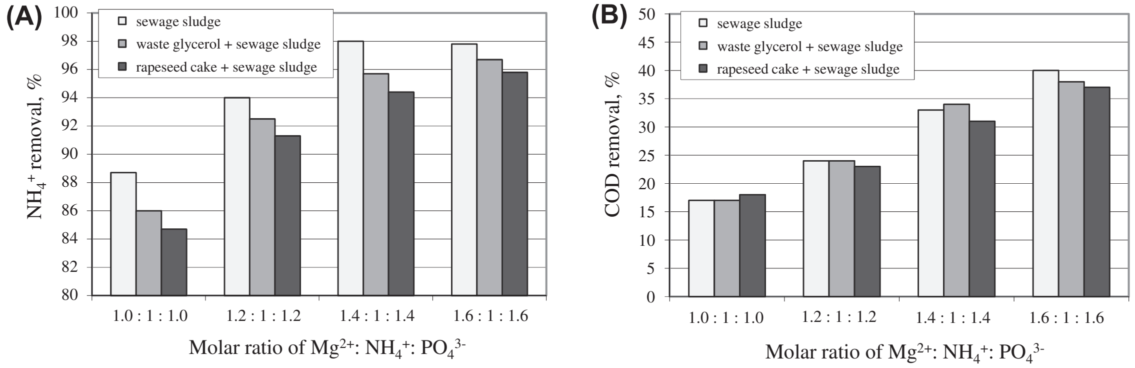

Fig. 3. Influence of struvite precipitation conditions on the NH_4^+ (A) and COD (B) removal.

very important factor while assessing its agricultural application.

The impact of precipitation conditions, including the initial NH_4^+ content and dosages of reagents used during precipitation, on the structure of struvite crystals was also taken into account. In this study, orthorhombic structure of crystals was observed. The origin of post-digestion waters did not influence the structure of struvite precipitated. This is in agreement with our previous results [31]. However, in the conditions of increased molar ratio of reagents, larger forms of struvite were observed (Fig. 4). The influence of potential factors determining the structure of struvite precipitated reported previously in the literature is not clear. Besides, the molar ratio of reagents, one of the most important factors, which has an influence on the structure of generated struvite crystals, is the initial ammonia–nitrogen content in the solution. Our previous studies showed that very high concentration of NH_4^+ (>5,000 mg/l) is privileged for generating "needle like" struvite structure [18].

3.1.2. Ammonia stripping from post-digestion liquors

Secondly, the post-digestion waters after struvite precipitation were treated by the application of ammonia stripping. In the conditions of the ammonia striping applied in this study (pH 11.5, 20°C), NH_3 constituted about 99% of the ammonia–nitrogen. Regardless of the previous step of the pre-treatment, the degree of NH_4^+ removal reached the value of 87–91% (Fig. 5), which is in the range most commonly cited in the literature (88–95%) [32,33]. Liquor after sewage sludge digestion subjected to struvite precipitation at stoichiometric ratio of reagents and subsequent stripping of 8 h allowed

Table 3
Composition and heavy metals content in the struvite crystals precipitated

Parameter	Range of value	Average value	Theoretical values
Struvite composition			
P, %TS	13.48–13.52	13.50 (0.02)*	12.69
N, %TS	5.53–5.65	5.60 (0.02)	5.73
Mg, %TS	11.56–12.55	12.29 (0.32)	9.95
Ca, %TS	1.89–2.17	2.02 (0.13)	–
Heavy metals content			Struvite precipitated
Cd, mg/dm^3	1.02–1.12	1.05 (0.03)	
Cu, mg/dm^3	15.9–16.3	16.2 (0.2)	
Ni, mg/dm^3	5.45–6.15	5.88 (0.33)	
Pb, mg/dm^3	7.17–8.17	7.57 (0.40)	
Zn, mg/dm^3	28.2–35.2	31.2 (2.5)	
Hg, mg/dm^3	< 0,1	< 0,1	
Cr, mg/dm^3	15.4–19.4	17.3 (1.1)	

*()—standard deviation.

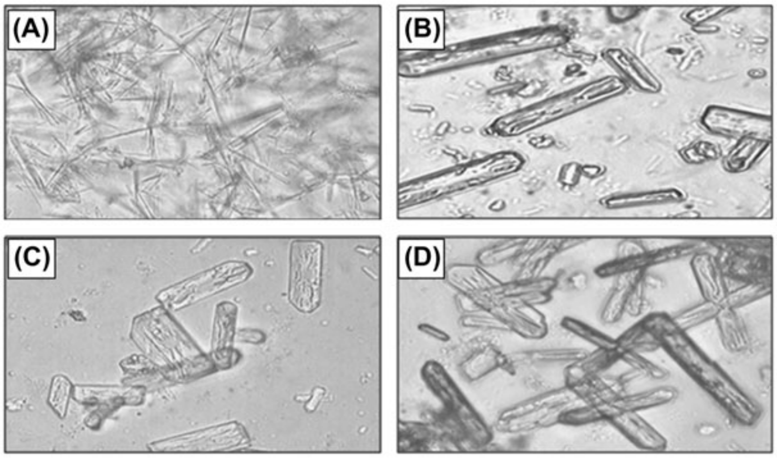

Fig. 4. Influence of precipitation conditions on the structure of struvite crystals (A—Mg^{2+}: NH_4^+: PO_4^{3-} = 1:1:1; B—Mg^{2+}: NH_4^+: PO_4^{3-} = 1.2:1:1.2; C—Mg^{2+}: NH_4^+: PO_4^{3-} = 1.4:1:1.4; D—Mg^{2+}: NH_4^+: PO_4^{3-} = 1.6:1:1.6; liquors after waste glycerine and sewage sludge co-digestion).

to decrease the ammonia concentration to the level of 10–20 mg/dm^3. The co-digestion liquors contained between 10–20 mg NH_4^+/dm^3 met the above-mentioned limit after struvite precipitation at 20% increased dosage of Mg^{2+} and PO_4^{3-} and ammonia stripping for 6–8 h (Fig. 5).

For stripping of ammonia from waters pre-treated previously at Mg^{2+}, NH_4^+ and PO_4^{3-} ratio amounting to 1.4:1:1.4, the stripping longer than 4 h (waters after sewage sludge digestion) and 6 h (co-digestion effluents) did not have a significant influence on the effectiveness of the process. For larger ratio of

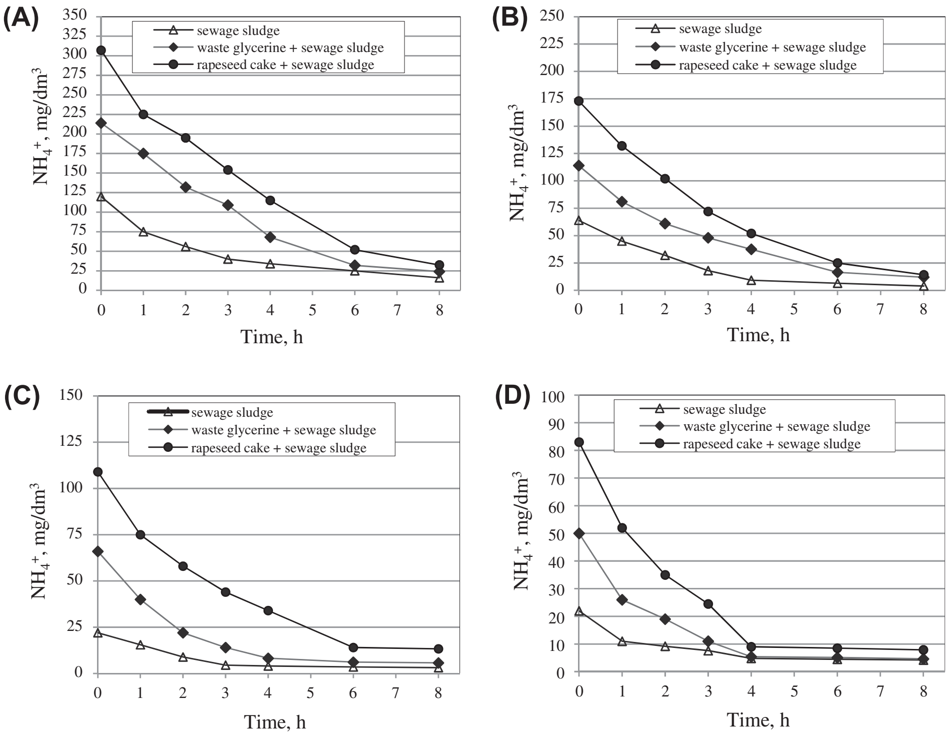

Fig. 5. Ammonia stripping from liquors after struvite precipitation at different molar ratios of reagents (A—Mg^{2+}: NH_4^+: PO_4^{3-} = 1:1:1; B—Mg^{2+}: NH_4^+: PO_4^{3-} = 1.2:1:1.2; C—Mg^{2+}: NH_4^+: PO_4^{3-} = 1.4:1:1.4; D—Mg^{2+}: NH_4^+: PO_4^{3-} = 1.6:1:1.6).

reagents (Mg^{2+}: NH_4^+: PO_4^{3-} = 1.6:1:1.6), the stripping of all analysed liquors above 4 h did not have a significant impact on the process (data equivalent statistically).

The ammonia stripped from the post-digestion liquors can be absorbed from the air into a strong H_2SO_4 solution, which can be crystallized in the $(NH_4)_2SO_4$ form and used as a fertilizer [33,34]. Simultaneously, with ammonia stripping, about 12–23% of the soluble organic matter has been removed, depending on the duration of the process (data not shown). This trend was also observed in other studies [34], which suggest that it is necessary to control volatile organic matter emissions during ammonia stripping and additional equipment, e.g. biofilters may be required.

3.2. Treatment of liquors with the application of membrane techniques

3.2.1. Effectiveness of RO

Finally, pre-treated liquors were post-treated with the application of NF or RO. The application of two-step pre-treatment (struvite precipitation/ammonia stripping) influenced the effectiveness and efficiency of the subsequent membrane filtration in a positive way. The removal of ammonia–nitrogen and TN during RO amounted to 80–82 and 83–85%, respectively. The effectiveness of NF as regards nitrogen compounds removal from the pre-treated liquors reached the value of 60–62% (N-NH_4^+) and 70–72%% (TN). Taking into account the above, liquors after sewage sludge digestion pre-treated at least at molar ratio of reagents (struvite precipitation) and stripped for at least 4 h met the discharge limit (<10 mg N/dm^3) [35]. The same waters post-treated by NF required the pre-treatment involving at least struvite precipitation at molar ratio of reagents and 8 h of ammonia stripping. In case of co-digestion liquors, the above-mentioned discharge limit was met after struvite pre-treatment at molar ratio of reagents/stripping of 8 h and subsequent RO. Whilst, NF required at least chemical treatment at increased molar ratio of reagents (20% increase of Mg^{2+} and PO_4^{3-}) and 6–8 h of ammonia stripping in order to meet the discharge limits as regards nitrogen compounds removal.

Regardless of the pre-treatment conditions applied, the removal of phosphorus by means of RO reached the level of >99.5%, and decreased its concentration below discharge level [35]. NF also ensured a high-level phosphorus compounds removal, i.e. 95–96%, which is in agreement with previous studies [36]. However, the discharge level of phosphorus after NF was only reached after application of struvite precipitation at molar ratio of reagents (liquors after sewage sludge digestion) and 20% increased dosages of reagents (co-digestion effluents). After struvite precipitation as a pre-treatment at increased dosages of reagents, the liquors contained high concentration of residual phosphates (Table 2), which subsequently led to a higher concentration of phosphorus in the permeate after NF. Taking into consideration the results of nutrients removal during applied membrane processes, it was concluded that initial concentration of NH_4^+ and PO_4^{3-} as well as the ammonia–nitrogen binding during struvite precipitation/ammonia stripping played the crucial role on their residual content after RO or NF treatment.

The application of RO and NF allowed removing about 99% and 65–67% organic compounds, respectively. Thus, post-treated liquors after RO were characterized by very low COD (4–9 mgO_2/dm^3) and BOD_5 (2–6 mgO_2/dm^3) concentrations below discharge levels. In all cases, the permeate after NF still contained significant concentration of organic compounds (COD: 203–576 mgO_2/dm^3, BOD_5: 278–372 mgO_2/dm^3). Both membrane processes allowed to decrease the concentration of total solids below discharge limits and eliminated the secondary load of Cl^-, which occurred in the treated liquors as a result of pH value adjustment before applying membrane filtrations. The most optimum conditions of the pre-treatment, allowing to decrease the contaminants content below discharge limits, after subsequent membrane filtration (NF or RO) were selected. It was taken into account that conducting struvite precipitation at increased molar ratio of reagents and prolonged time of stripping increase the operating costs (reagents for precipitation and energy for stripping). The characteristics of the treated liquors after the least demanding pre-treatment conditions, which ensured the complex removal of contaminants, below discharge levels, is presented in Table 4.

3.2.2. Efficiency of the RO as a post-treatment

The initial struvite precipitation in all post-digestion liquors influenced the efficiency of the subsequent membrane process in a positive way. The initial volumetric fluxes reached the value of 3.25×10^{-6} and 8.5 $m^3/m^2 s$ for the RO and NF, respectively. In all cases, greater values of the flux occurred in case of the processes conducted for the liquor after initial pre-treatments. The flux value of liquor after sewage sludge digestion, which did not undergo initial stabilized after 3–3.5 h of conducting

Table 4
Characteristics of liquors pre-treated at the most appropriate (the least demanding) conditions and post-treated by membrane filtration

	Sewage sludge		Glycerine + sludge		Rapeseed cake + sludge		
	Struvite precipitation (1:1:1) + stripping of 4 h		Struvite precipitation (1:1:1) + stripping of 6 h		Struvite precipitation (1:1:1) + stripping of 8 h		
Parameter	Before RO	After RO	Before RO	After RO	Before RO	After RO	Discharge limits [34]
pH	6.8	7.4 (0.1)*	6.8	7.3 (0.1)	6.8	7.3 (0.1)	6.5–9.0
NH_4^+, mg/dm^3	34.1 (2.6)	6.0 (0.5)	33.2 (2.6)	6.1 (0.6)	32.5 (2.1)	5.8 (0.4)	10[a]
TN, mg/dm^3	42.5 (3.1)	6.4 (0.4)	40.2 (3.2)	6.4 (0.5)	42.5 (3.0)	7.0 (0.3)	
TP, mg/dm^3	19.0 (0.7)	0.2 (0.05)	19.8 (0.6)	0.2 (0.05)	36.3 (1.4)	0.3 (0.08)	1.0[a]
COD, mgO$_2$/dm^3	863 (45)	6.2 (1.5)	1,009 (85)	6.5 (1.2)	1,440 (95)	9.2 (1.6)	125
BZT$_5$, mgO$_2$/dm^3	559 (35)	4.2 (1.0)	637 (52)	4.5 (1.2)	929 (75)	6.2 (1.5)	15
TS, mg/dm^3	125 (10)	< 1.0	120 (9)	< 1.0	140 (15)	< 1.0	35

*()—standard deviations.

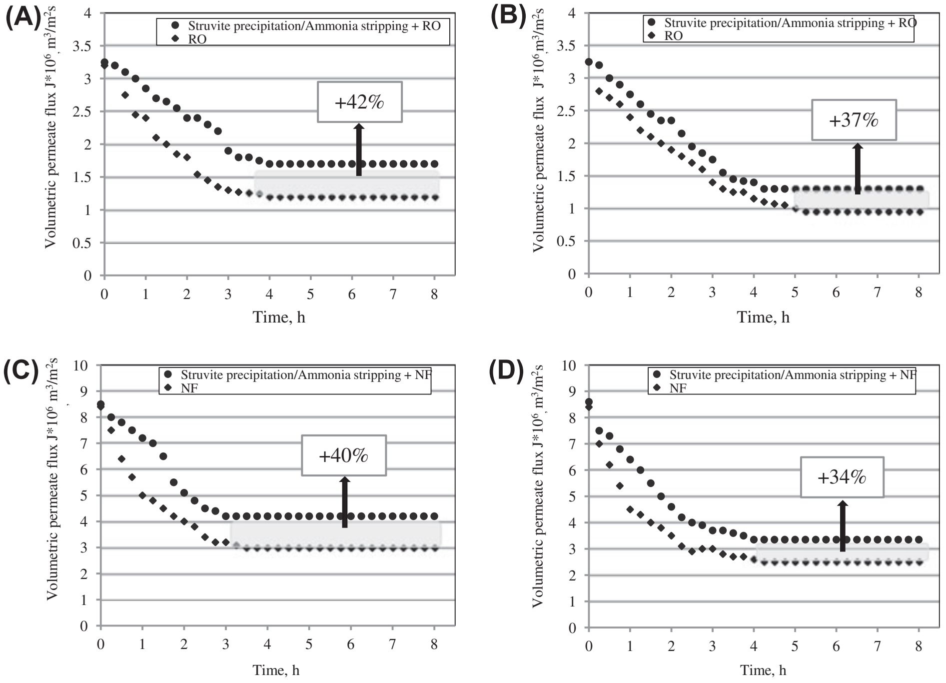

Fig. 6. Influence of pre-treatment on the volumetric fluxes of liquors post-treated by RO and NF (A, C—liquors after sewage sludge digestion, B, D—liquors after co-digestion of rapeseed cake and sewage sludge; selected assays).

the RO and NF at the level of 1.2×10^{-6} and $3.0\,m^3/m^2\,s$, respectively. Whilst, the value of the flux after pre-treatment reached about 37–40% (RO, after 5 h) and 34–40% (NF, after 4 h) higher values. The effect was ascribed to the lower content of contaminants, mostly organic matter, in the liquors undergoing initial pre-treatment. Fig. 6 presents influence of pre-treatment on the volumetric fluxes of liquors post-treated by RO and NF (selected assays).

4. Conclusions

The application of the two-step pre-treatment (struvite precipitation/ammonia stripping) and subsequent RO or NF ensured the removal of nitrogen and phosphorus compounds below discharge limits. However, the complex removal of contaminants, including organic compounds, required the application of RO after the pre-treatment. Thus, waters fulfilled discharge standards after struvite precipitation (Mg^{2+}: NH_4^+:

$PO_4^{3-} = 1{:}1{:}1$), ammonia stripping of between 2–8 h and RO as a post-treatment. The reuse of liquors post-treated by NF, e.g. as technological water (e.g. for cleaning purposes or feedstock dilution) should also be taken into consideration. Introducing air stripping between struvite precipitation and membrane filtration allowed to reduce the amount of reagents used during struvite precipitation in order to remove the ammonia–nitrogen below discharge limit. Moreover, the pre-treatments impacted the efficiency of subsequent membrane processes in a positive way, and thus will likely lengthen lifecycle of membranes used.

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