Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2021.26661

214 (2021) 214–223
February

Treatment of municipal waste biogas plant digestate using physico-chemical and membrane processes

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Received 15 April 2020; Accepted 26 May 2020

ABSTRACT

Membrane processes preceded by pre-treatment using physical-chemical processes can be useful in the liquid fraction of municipal biogas plants digestate treatment. The effectiveness of purification in an integrated process that combines chemical coagulation/precipitation (using PIX 112, $FeCl_3·6H_2O$, or CaO) with ultrafiltration (using 10 and 30 kDa molecular weight cut-off (MWCO) polymeric membranes made of polyethersulfone (PES) or regenerated cellulose (C)) was determined. The changes in zeta potential and particle size that occur in the solution as a result of the applied purification processes were also analyzed. The results obtained show that the improvement of the efficiency of the digestate treatment is possible with the tested integrated process. The best results were observed with a combination of 20 g/dm^3 $FeCl_3·6H_2O$ coagulation with ultrafiltration on the 10 kDa PES membrane. Moreover, it was observed that the use of PIX 112 coagulant prior to ultrafiltration reduced the fouling in the tested membranes to the greatest extent.

Keywords: Digestate; Biogas plant; Coagulation; Ultrafiltration; Integrated process

1. Introduction

Proper waste management has become an extremely important issue of local government units in the field of environmental protection. Local governments face various problems, including the emerging biodegradable fraction of municipal waste. The (Polish) National Waste Management Plan 2022 [1] assumes the limiting of the storage of these waste fractions to 75% (short-term) and 35% (long-term) by weight in relation to the quantity produced in 1995. Therefore, the introduction of segregation at the so-called "source" becomes an important matter. The fraction obtained in this way becomes an ideal resource, which, after appropriate processing, allows the production of biogas [2]. The energy obtained from it is treated as so-called renewable energy [3]. This is extremely important in view of the challenges facing the polish energy sector, because the development of communal biogas installations using easily accessible raw materials – waste from the nearby area – may become a solution to the problem of meeting Poland's obligations in the field of "green energy" production. There are currently 108 biogas plants operating in Poland on municipal waste landfills [4]. In European countries, there are up to about 15 biogas plants per 1 million capita (e.g., 15 in Norway, 5 in Germany, and 2 in Poland) [5]. The essence of biomethane production is a complex, four-stage methane fermentation process consisting of hydrolysis, acidogenesis, acetate, and methanogenesis [6]. It is carried

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out under anaerobic conditions, with acidifying, acetate, and methanogenic bacteria. The condition for the proper course of the process is to ensure appropriate values of temperature, pH, humidity, salinity, and nutrient content. This allows the process rate to be increased and affects the composition and quality of the biogas produced [7].

The operation of a biogas plant involves, apart from biogas production, the formation of a large amount of digestate [8]. Depending on the size of the biogas plant, the amount of digestate can be up to several thousand tons per year and is approximately similar to the mass of the substrates used in the fermentation process [9]. It can be difficult, in terms of both logistics and legal issues, to manage this amount of hydrated digestate [10]. When the amount of digestate produced exceeds the possibilities of its recovery using the R10 method of pouring waste into fields for fertilizing purposes, other alternative methods of management become necessary [11,12]. These primarily include separation into solid and liquid fractions using centrifuges, screw presses or various types of screens [13]. Each fraction has different physical and chemical properties. A small amount of solid fraction (up to 10% of the total weight of the digestate [14,15]), which is rich in organic matter and plant nutrients, is a valuable source of fertilizer [16], positively affecting the water and sorption capacity of soils [17]. The liquid fraction, on the other hand, can be reused in the methane fermentation process [18]. This is because it is a valuable material that initiates a new process due to the presence of a specific bacterial microflora. Furthermore, the liquid fraction can be used as a full-fledged fertilizer or to dilute the fermentation mixture [19]. It can also be an alternative source of water, which, among other things, can be used in agriculture or other sectors. However, this requires advanced techniques for its purification.

The most common way of further processing the digestate is membrane filtration [20]. However, the application of these techniques encounters many operational problems, especially when treating water or wastewater that is of poor quality and which is characterized by high color, turbidity, and a high concentration of organic and inorganic compounds that are present in dissolved or suspended form [21]. In view of the low efficiency of stand-alone membrane processes in the case of eliminating contaminants, as well as the high fouling intensity of membranes, membrane techniques are increasingly combined with other physical, chemical, or biological processes to form so-called integrated membrane processes [22–24]. Taking into account the above considerations, work has been undertaken to determine the effectiveness of the liquid fraction of the digestate from municipal waste biogas plant treatment in an integrated process that combines chemical coagulation/precipitation with ultrafiltration using polymeric membranes. The influence of the properties and membranes of the tested reagents on the efficiency of reducing the content of organic compounds in the liquid fraction of the digestate was determined. Moreover, the influence of the integrated process variant on membrane fouling susceptibility was also determined. The changes in zeta potential and particle size that occur in the solution as a result of the applied iron-based coagulant were also analyzed.

2. Materials

The research was carried out using the liquid fraction of the digestate coming from a biogas plant that processes the organic fraction of municipal waste. The characteristics of the liquid fraction of the digestate are presented in Table 1.

The coagulation/chemical precipitation process was carried out using three different chemical reagents: ferric coagulant PIX 112, $FeCl_3·6H_2O$, and CaO, with their characteristics being presented in Table 2.

In the study, microdyn nadir ultrafiltration membranes made of polyetherosulfone (PES) and regenerated cellulose (C) were used. Their characterization is included in Table 3.

3. Experimental methods

Pre-treatment of the digestate liquid fraction was carried out in 1 dm^3 reactors on a test stand equipped with the Velp Scientifica JLT4 (Italy) mechanical stirrer. A suitable reagent in a dose of 10 or 20 g/dm^3 was added into the reactors (dose selection based on our previous study [29]). The samples were stirred rapidly (150 rpm) for 2 min, followed by 20 min of slow mixing (20 rpm). After that time, the stirrers were turned off and the digestate samples were sedimented for 30 min. The supernatant from each reactor, prior to its analyses, was filtered using medium paper filters.

The membrane filtration tests were carried out on a stand equipped with Millipore's Amicon 8400 cell. The Amicon cell is designed for use with flat membranes and allows for dead-end filtration. The volume of the cell is 400 cm^3 and the diameter of the membrane is 76 mm (active filtration surface of 38.5 cm^2). The cell is placed on a magnetic stirrer to ensure that the concentrations in the entire volume of the solution are balanced. The transmembrane pressure used in the tests was in the range of 0.2–0.5 MPa. To estimate the transport and separation properties of the membrane under study, the following two parameters were used: volume flux of the permeate (J) and organic substances retention coefficient (R). The relative permeability (J/J_0) of the membranes, expressed as the quotient of the permeate flux (J) to the redistilled water flux (J_0), was also calculated. Using the calculated relative permeability value, the membrane fouling susceptibility was determined.

Studies on the purification of the digestate using an integrated process were carried out by combining the chemical coagulation/precipitation process with physical purification on ultrafiltration membranes. In an integrated process, a decanted sample, after being coagulated and left for 30 mins of sedimentation, was ultrafiltered at a pressure of 0.2 MPa.

Digestate treatment experimental setup has been presented in Fig. 1.

The efficiency of the process was determined by measuring the concentration of organic compounds expressed as chemical oxygen demand (COD), 5 d biochemical oxygen demand (BOD_5), and dissolved organic carbon (DOC). The determination of COD and BOD_5 was performed using standard methods: dichromate and dilution, respectively [30]. The concentration of dissolved organic

Table 1
Properties of the liquid digestate fraction from the municipal waste biogas plant

Index	Value
pH	7.2
Conductivity, mS/cm	22
Total suspended solids, mg/dm^3	2,860
Chemical oxygen demand (COD), mg O_2/dm^3	13,060
Biochemical oxygen demand (BOD), mg O_2/dm^3	8,362
Dissolved organic carbon (DOC), mg C/dm^3	6,086
NH_4^+–N, mg N/dm^3	1,742
NO_2^-–N, mg N/dm^3	6.25
NO_3^-–N, mg N/dm^3	Below the limit of detection
PO_4^{3-}, mg/dm^3	18.9
Mesophilic bacteria, CFU/cm^3	111 × 10^6
Thermophilic bacteria, CFU/cm^3	163 × 10^2

Table 2
Reagents used in the research [25–27]

Reagent/coagulant	Calcium oxide	Iron(III) chloride (hexahydrate)	PIX 112
Property			
Producer	Merck (Poland)	Avantor (Poland)	Kemipol (Germany)
Chemical formula	CaO	$FeCl_3 \cdot 6H_2O$	$Fe_2(SO_4)_3$
Molecular weight, g/mol	56.08	270.32	399.9
Physical form	Solid (powder)	Solid	Liquid
Color	White	Yellow–brown	Dark brown
Odor	Odorless	Chlorine	Odorless
pH	12.6 (20°C)	1.8 (1%)	<1
Density, g/cm^3	3.37	1.82	1.50–1.56

Table 3
Major parameters of the experimental membranes [28]

Membrane symbol	Membrane material	MWCO, kDa	Mean pore radius, nm	Contact angle, °	Polarity, %	J_{H_2O}*, m^3/m^{2}d
PES 10	Polyetherosulphone	10	2.04	50.01	44.27	5.1
PES 30		30	8.38			7.6
C 10	Regenerated cellulose	10	5.01	54.76	49.92	1.3
C 30		30	12.55			10.1

*0.2 MPa (self-measurement).

carbon was measured using the HACH IL550 total organic carbon with total nitrogen (TOC-TN) analyzer.

Particle size distribution was carried out using a Mastersizer 2000 laser diffractometer (Malvern), equipped with a HydroMu dispersion unit (Malvern). The measuring range of the apparatus is a particle size from 0.1 to 2,000 µm. In the measuring procedure, depending on the concentration of solid particles, around 3 cm^3 of suspension were poured into 700 cm^3 beaker fulfilled with water cross-flowing through the measuring cell. The particle size measurements were carried out without and afterward under the operation of ultrasounds in the dispersion unit, so the possible agglomerates could have been broken.

Particle size distribution was also performed using a Nicomp 380 DLS apparatus (Nicomp particle sizing systems). This submicron particle size analyzer uses the dynamic light scattering (DLS) method to obtain particle size distributions for samples with particles ranging from 1 nm to 5 µm. The measurement was carried out by placing around 3.5 cm^3 of diluted suspension in a measuring cell. Through the use of Nicomp analysis algorithm, the 380 is able to analyze complex multi-modal distributions with the highest resolution and reproducibility.

Zeta potential measurements were conducted at 25°C using a zeta potential analyzer (Malvern Zetasizer 2000). The diluted suspension was conditioned in a beaker for

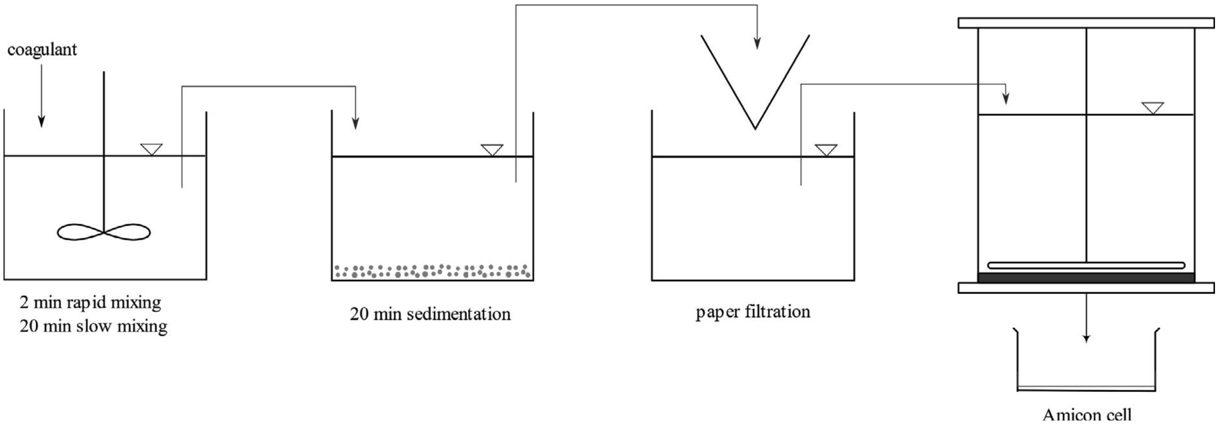

Fig. 1. Experimental setup.

10 min at a given pH. The pH was adjusted using sodium hydroxide or hydrochloric acid. Then, the suspension was placed in the electrophoresis cell using a syringe. The value of zeta potential was determined as an average of five successive measurements.

4. Results and discussion

4.1. Separation and transport properties of membranes in ultrafiltration and in integrated processes

The research conducted on the evaluation of the usefulness of coagulation/chemical precipitation for the removal of organic compounds from the digestate liquid fraction coming from the municipal waste biogas plant allowed the influence of the applied reagent type, and its dose, on the effectiveness of the digestate purification to be determined. Based on the earlier results of our study [29], it was found that the optimum contact time for flocculation is equal to 20 min, and this was used in the study.

When analyzing the results presented in Fig. 2, it can be concluded that an increase in the dose of each of the tested reagents resulted in a decrease in the BOD_5, COD, and DOC values. All of the tested reagents can be effectively used to purify the digestate liquid fraction, however, $FeCl_3{\cdot}6H_2O$ proved to be the most useful in this respect. It allowed the BOD_5, COD, and DOC content to be reduced by 54%, 43%, and 31%, respectively, at a dose of 10 g/dm^3. By analogy, a dose increase up to 20 g/dm^3 resulted in an increase of separation efficiency by 77%, 56%, and 47%, respectively. The highest effectiveness of eliminating organic compounds, obtained when using $FeCl_3$, may result from the formation, during coagulation, of heavier and better sedimentary flocs. This is in comparison to those formed after the application of PIX 112 or CaO.

It was also found that the biodegradable fraction of organic compounds were eliminated to the highest degree during coagulation/chemical precipitation, while in the case of the removal of all organic compounds, the elimination efficiency was much lower and did not exceed 50% (for 20 g/dm^3 of $FeCl_3$).

The analysis of the digestate liquid fraction ultrafiltration effectiveness showed (Fig. 3) that the final quality of the permeate was determined by the membrane cut-off. As the membrane cut-off value increased, and thus as the membrane pore diameter increased, a decrease in the separation efficiency of organic compounds from the digestate was observed. The higher the value of this parameter, the larger the particles that were transferred to the purified solution. The effects of digestate purification on polymeric membranes showed that the driving force value, which induces transport through the membrane, had no significant effect on the quality of the permeate. In the analyzed pressure range (0.2–0.5 MPa), the content of organic compounds in the permeate remained at a comparable level. Moreover, when comparing the suitability of the analyzed membranes for the purification of the digestate, it can be observed that the differences between the separation efficiency of the membranes made of PES and regenerated cellulose are minimal. Comparing the three parameters used for the determination of the organic matter content in the samples, the highest separation efficiency was observed for BOD_5 and COD, and the lowest for DOC. For example, with a transmembrane pressure of 0.2 MPa, and for the examined cut-off 10 kDa membranes, the R_{BOD_5}, R_{COD}, and R_{DOC} equaled 42%, 41%, and 27% for the PES membranes, and 38%, 38%, and 30% for the C membranes, respectively. On the other hand, these values were much lower for the cut-off 30 kDa membranes: 25%, 26%, and 11%, and 22%, 21%, and 12%, respectively.

As the efficiency of contaminant separation obtained in the ultrafiltration process, even with the use of a denser 10 kDa membrane, is not at a satisfactory level, the usefulness of the integrated process, being a combination of the coagulation/chemical precipitation process with ultrafiltration, was assessed in the next stage of research. As shown in Fig. 4, the combination of the two analyzed individual processes allowed the efficiency of the digestate purification to be improved. It was also found that the separation efficiency in the integrated process depends on both the dose of reagent added to the digestate before ultrafiltration and on the membrane cut-off. An increase in the dose of the reagent at the pre-treatment stage resulted in an improvement in the final quality of the digestate treated in the integrated process. Contrary to the membrane filtration without pretreatment, the membrane cut-off value

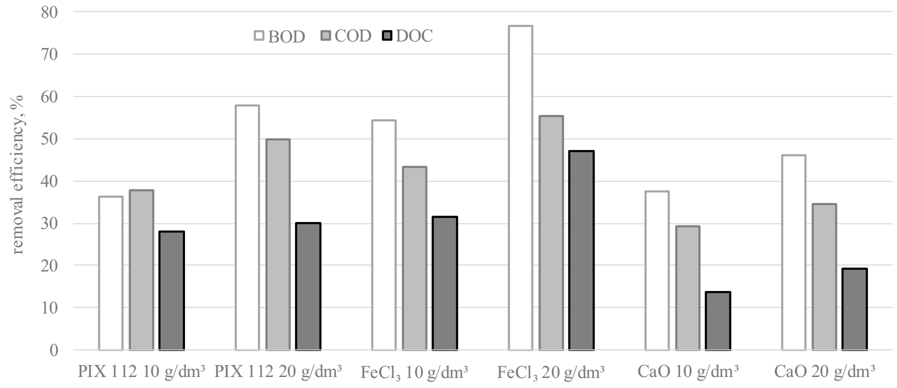

Fig. 2. BOD$_5$, COD, and DOC removal efficiency for CaO, FeCl$_3$·6H$_2$O, and PIX 112.

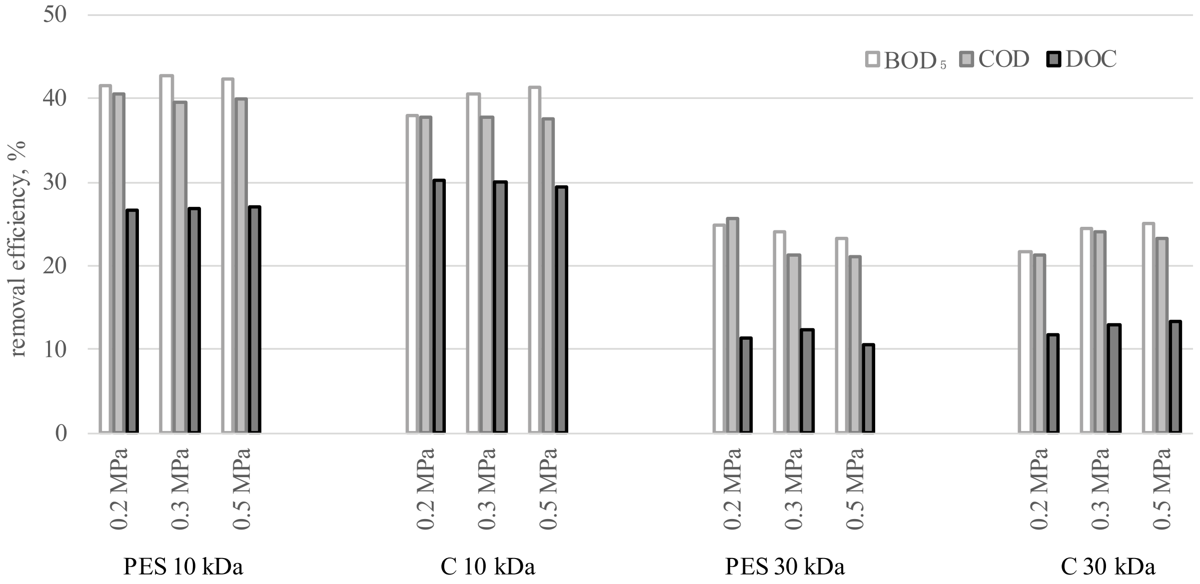

Fig. 3. Influence of membrane properties on the removal efficiency of BOD$_5$, COD, and DOC.

had no significant influence on the separation efficiency of organic compounds. Of the tested combinations of reagents and membranes in the integrated process, the best results of eliminating organic substances were observed for coagulation using FeCl$_3$·6H$_2$O at a dose of 20 g/dm^3 at a pre-treatment stage combined with the 10 kDa PES membrane ultrafiltration. For these conditions, the retention coefficient of R_{BOD_5}, R_{COD}, and R_{DOC} was equal to 78%, 69%, and 61%, respectively. Very similar separation efficiencies of organic matter, at the same coagulant dose, were found for the 30 kDa PES membrane.

When using membrane techniques for digestate cleaning, in addition to analyzing the separation properties of the membranes, it is also necessary to assess their transport properties. It is particularly important to perform an analysis of the intensity of membrane fouling. Ultrafiltration of the solution, not preceded by chemical pre-treatment, caused a very significant deterioration of the hydraulic performance of the membranes (Fig. 5). It was observed that the decrease in permeate flux was influenced by both the membrane material and its cut-off. Membranes made of a stronger hydrophobic PES were much more prone to fouling when compared to more hydrophilic C membranes. Moreover, a greater decrease in permeate flux, as compared to that measured for redistilled water, was observed for membranes with larger cut-off values, which indicates that in their case, internal fouling, caused by the penetration of impurities into the membrane pores, was dominant.

When considering the possibility of using an integrated coagulation/chemical precipitation – ultrafiltration process for the purification of the digestate liquid fraction, membrane fouling should also be taken into account.

Comparison of the J/J_0 values obtained during the digestate liquid fraction purification in a stand-alone ultrafiltration process with those determined for the coagulation/chemical precipitation – membrane filtration integrated

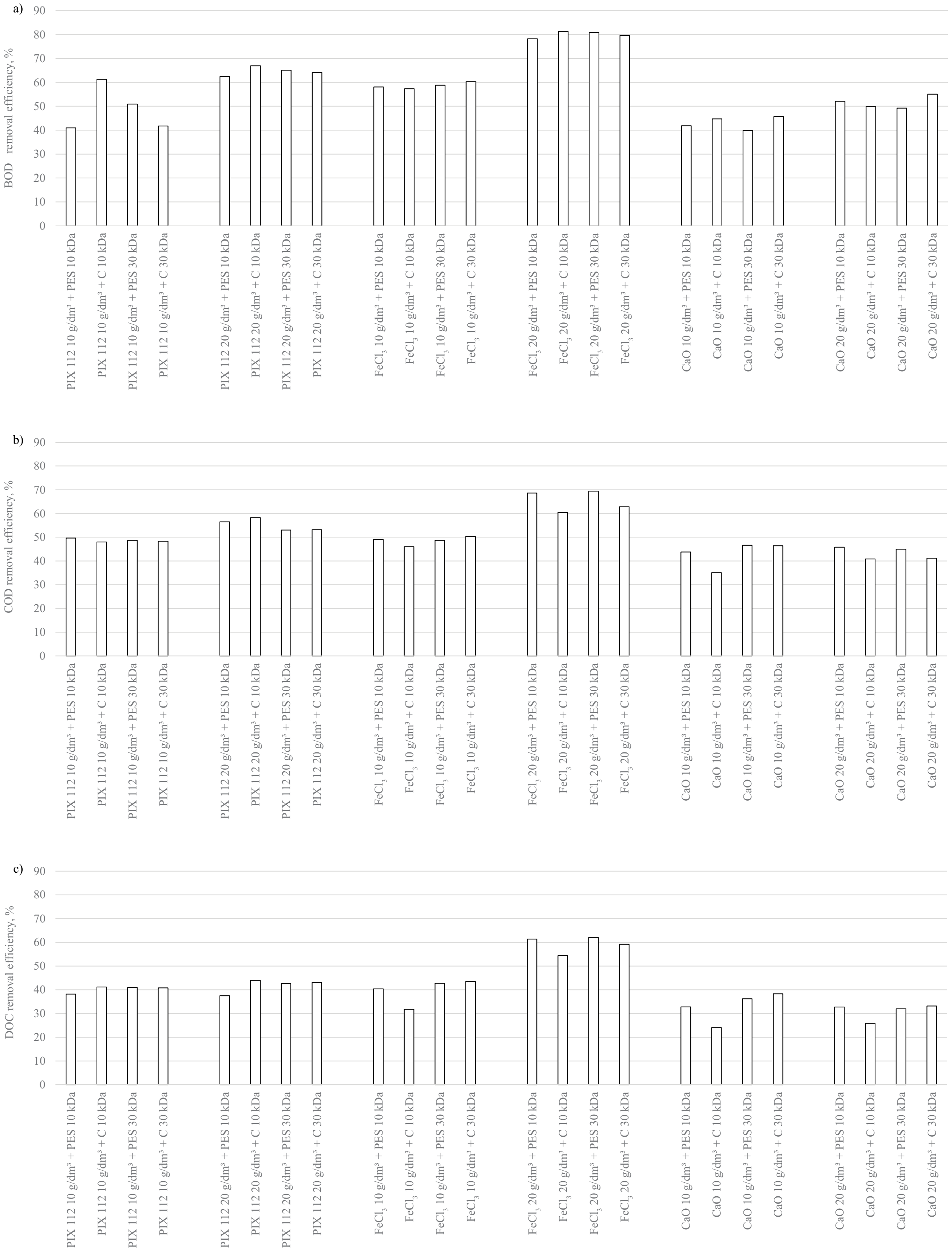

Fig. 4. Efficiency of separating organic substances in integrated processes (a) BOD_5, (b) COD, and (c) DOC (TMP = 0.2 MPa).

process (Fig. 5) shows that the use of each of the tested reagents at the pre-treatment stage enables the transport properties of membranes to be improved. It was found that the use of PIX 112 coagulant prior to ultrafiltration allows the problem of the tested membranes' fouling to be reduced to the greatest extent. Moreover, for PIX 112 and CaO, an increase in the reagent dose resulted in some increase in the J/J_0 ratio, which means that the blockage intensity of the membranes has decreased. The opposite phenomenon could be observed for $FeCl_3{\cdot}6H_2O$. When $FeCl_3$ is used as a coagulant, more iron ions remain in solution after the coagulation/sedimentation process than are found when PIX 112 ($Fe(SO_4)_3$) is dosed. This may result in their concentration in the polarization layer and then precipitation on the membrane surface.

The research also showed that the material from which the membrane is made was a significant factor in determining whether the chemical pre-treatment of the solution will contribute to a decrease in fouling. For more hydrophilic C membranes, the blocking intensity was significantly reduced, while for PES membranes these changes were not so spectacular. Similarly, as it was found for the stand-alone digestate ultrafiltration process, smaller J/J_0 values (and thus stronger membrane fouling) were observed for membranes with higher cut-off values, that is, larger pore diameters. This indicates that the coagulation/chemical precipitation did not sufficiently remove those impurities that penetrate into the membrane structure and cause pore blockage.

4.2. Changes in the size and zeta potential of digestate fractions after coagulation and coagulation/ultrafiltration

The use of chemical digestate purification processes, in this case, coagulation/chemical precipitation, results in chemical reactions in the solution after the addition of the reagents, the consequence of which is, among others, a change in their zeta potential – a measure of the magnitude of the electrostatic or charge repulsion/attraction between particles. When analyzing the electrostatic interactions between the components of the solution and the membrane, changes in the zeta potential of the membranes used must

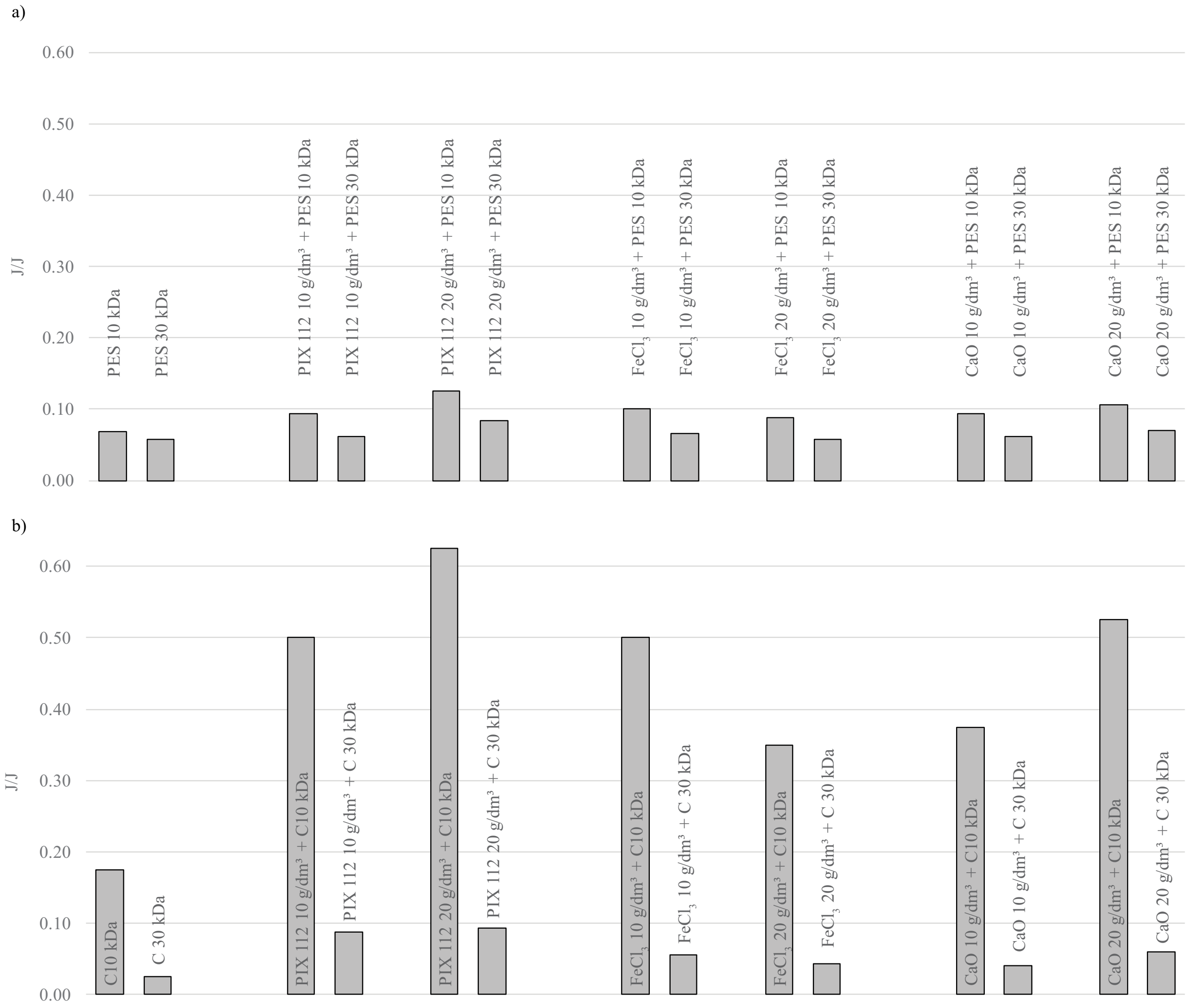

Fig. 5. Relative permeate permeability of membranes for stand-alone UF membranes and for membranes in an integrated process (TMP = 0.2 MPa) (a) PES and (b) C.

also be taken into account. According to Salgin et al. [31], PES membranes have negative zeta potential (ζ potential) values under pH 4–8, which is due, among others, to the ionic strength of the solution and the type of cations and anions in the solution. Salinas-Rodriguez et al. [32] reported that PES is generally more negatively charged than C membranes. For the same material, different MWCO produced different zeta potential values. For PES, the 100 kDa membrane was more negative than the 30 kDa membrane. For C, the 30 kDa membrane was more negative than the 100 kDa membrane. The iso-electric point for the PES and C membranes was found at acidic pH values; for C at pH < 3, and for PES at pH < 4.5. At basic pH values of >10.5, the measured zeta potential values increased in all cases.

In addition, the use of coagulation of impurities present in the solution causes changes in the proportion of fractions of a certain size, which is very important from the point of view of membrane operation, especially its susceptibility to fouling.

Due to the fact that the combination of coagulation using $FeCl_3·6H_2O$ with ultrafiltration turned out to be the most effective (from the point of view of permeate quality) in the purification of the digestate liquid fraction, the raw digestate and purified samples obtained in the stand-alone coagulation, as well as the coagulation/UF integrated process, were analyzed for the size distribution of suspended solids and for their ζ potential in the pH range of about 2–12 (Fig. 6).

Raw digestate suspension was characterized by a pH of around 7.4, high turbidity, and black color coming from small and stable particles in suspension. Larger particles were settling down to the bottom, but the suspension was turbid due to small suspended particles. The particle size distribution was wide and ranged from 0.4 to 300 μm, and even aggregates up to 900 μm in size were produced without the application of ultrasound. The median diameter was about 12 μm and the lower and upper decile were 1.9 and 76 μm, respectively. After the application of ultrasound, particle diameter median (d_{50}) decreased to 11.5 μm, lower decile (d_{10}) to 1.8 μm, and upper decile (d_{90}) to 62 μm.

Digestate is a mixture of particles with different electrophoretic potential. The suspension of raw digestate was characterized by high negative values of the ζ potential in the pH range of 5–12 (ζ potential from –25 to –35 mV). The isoelectric point was at pH 2.

The use of $FeCl_3·6H_2O$ as a coagulant in the amount of 10 g/dm^3 resulted in a very small number of black-colored digestate particles remaining in the solution, which settled down after some time. On the other hand, small yellow-colored particles appeared, which remained mostly suspended. In slightly alkaline aqueous solution, $FeCl_3·6H_2O$ reacts with hydroxide ion to form iron(III) hydroxide flocs, more precisely $FeO(OH)^-$. In this case, the pH of the suspension slightly decreased to about 7. The size distribution shifted significantly towards finer particles. The median diameter was about 8 and 6 μm, respectively before and after application of ultrasound. d_{10} and d_{90} were equal to 3 and 31 μm, and after ultrasound: 1.6 and 16 μm, respectively. 10 g/dm^3 of $FeCl_3·6H_2O$ reduced the negative value of the ζ potential by about 5–10 mV in the pH range of 2.5–12. However, no isoelectric point shift was observed.

Increasing the dose of $FeCl_3·6H_2O$ to 20 g/dm^3 resulted in the formation of more sediment and a much higher turbidity, and orange coloration, of the suspension. $FeCl_3$ is hydrolyzed in water, as a result of which the solution became reddish-brown and acidic (in this case, the pH decreased to about 4.1). Compared to the raw digestate, the disappearance of the finest and largest fraction is clearly visible on the particle size distribution. d_{50} was about 21.5 μm and decreased to 17.3 μm after the application of ultrasound. The lower and upper decile were equal: 8.2 and 52.7 μm, and 5.8 and 44 μm. 20 g/dm^3 of $FeCl_3·6H_2O$ generates a positive high electrokinetic potential in the pH range of 1.8–5. The isoelectric point appears at pH 8 and, after reaching about –5 mV, it again reaches a positive value at a pH of about 11.

At the most advantageous combination – $FeCl_3·6H_2O$ at a dose of 20 g/dm^3 and the PES 10 kDa membrane, the purest sample was obtained. The sample was transparent and without a yellowish coloring. The pH of this solution was 3.7. This dose of iron chloride was chosen because the lower dose (10 g/dm^3) did not give positive results in the purification of the raw digestate. In the case of using a regenerated cellulose membrane, such good results were not observed. After the purification process, the pH of the solution was 4.1. The color of the solution was yellowish with a small amount of completely dispersible sludge in the solution. The number of particles in the sample allowed only the change of the ζ potential with regards to the pH to be measured. Particle size distribution was performed using the DLS (Nicomp) method. In this case the measurement is carried out in a cell without suspension movement (as in the case of the laser diffraction apparatus), so if there are larger particles in the sample, they settle to the bottom and only smaller ones can be measured. The volume distribution showed one peak of particles of about 875 nm in diameter. The concentration of iron ions caused the particles to show a positive ζ potential in the whole examined pH range. The value of the ζ potential decreased systematically from about +25 mV to about +2 mV in the pH range of 2–12.

Using $FeCl_3·6H_2O$ in the amount of 20 g/dm^3 in the initial purification stage, and then the use of 30 kDa cut-off membranes, significantly improved the quality of the tested samples when compared to the use of membranes only. After the process, as a result of the addition of iron(III) chloride, the pH of the solution was lowered to below 4. The solution was slightly yellow in color and a brown-red flocculated precipitate accumulated on the bottom (less for the PES membrane and more for the C membrane), which easily dispersed after mixing, giving a more yellow color to the solutions. After being set aside for a longer time, it appeared again at the bottom. The Fe(III) cation is small and has a large charge, so it easily hydrolyses with water, forming a coordination complex with six water molecules, $Fe(H_2O)_6^{3+}$, which is yellow in color. When dissolved in water, it should precipitate, forming insoluble iron(III) hydroxide. $Fe(OH)_3$, however, forms a colloidal solution of this compound, which has the typical brown color of iron(III) salt solutions. The amount of sediment in the samples allowed the ζ potential to be measured, but due to the sediment density and structure, the measurement of particle size distribution was performed using the DLS method. In the sample

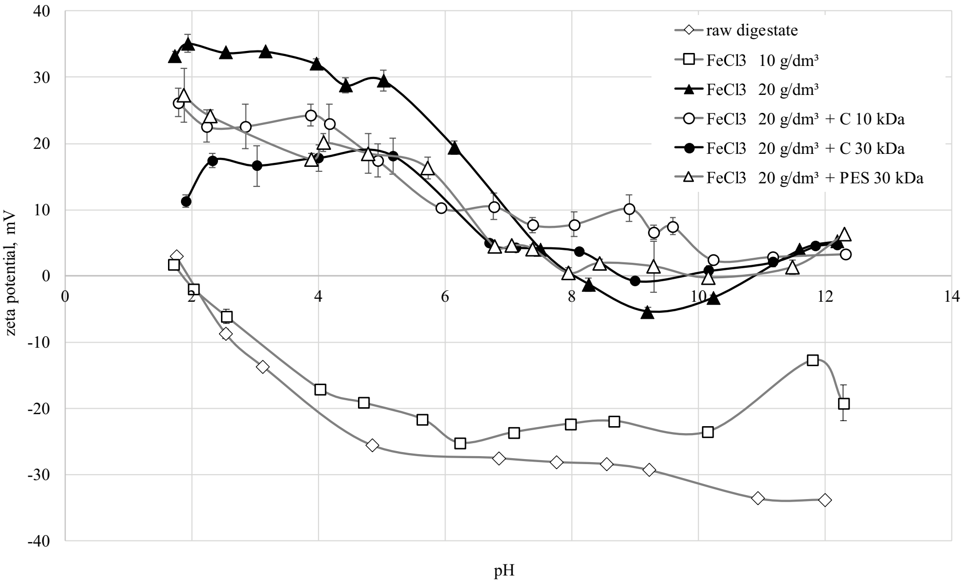

Fig. 6. ζ potential of raw digestate and digestate after treatment using coagulation and the integrated process with the use of $FeCl_3·6H_2O$.

after 30 kDa PES membrane filtration, the average diameter of the particles from the volume distribution was about 680 nm, and for the C membrane – 670 nm. Compared to the sample subjected to only 20 g/dm³ $FeCl_3·6H_2O$ coagulation, the particle positive ζ potential for both membranes was lower in the pH range 2–7. For the PES membrane, the ζ potential varied from about +25 mV, and for the C membrane from +18 to +5 mV. At pH 8.5–11.5, it fluctuated around zero and then reached about +5 mV at a pH above 12.

5. Conclusions

The research presented in this paper allowed for the following conclusions to be drawn:

- the coagulation/chemical precipitation process can be effectively used for pre-treatment of the digestate liquid fraction, and the effectiveness of the process is determined by the type and dose of the reagent used,
- the separation of organic contaminants from the digestate is also possible during the ultrafiltration process, and the purification effect depends on the membrane cut-off and its material,
- the use of an integrated process combining chemical coagulation/precipitation and ultrafiltration for the purification of the digestate allows for the more effective removal of organic compounds than that found for these processes running separately (e.g., upgrade to 59% of DOC removal for $FeCl_3·6H_2O$ with 30 kDa C ultrafiltration vs. 12% for ultrafiltration and 47% for coagulation alone),
- the best digestate purification effects, that is, the lowest concentration of organic substances, were achieved by combining coagulation with 20 g/dm³ $FeCl_3·6H_2O$ with ultrafiltration on a 10 kDa cut-off PES membrane (62% of DOC, 69% of COD, and 78% of BOD_5 removed),
- the use of the PIX 112 coagulant prior to ultrafiltration reduced the problem of blocking the membranes being tested to the greatest extent (e.g., J/J_0 for C10 kDa: 0.18 upgraded to 0.5 with PIX 112 10 g/dm³ pretreatment),
- the application of coagulation with $FeCl_3$ at the pretreatment stage of digitate treatment resulted in an increase in the zeta potential value of the particles remaining in the solution, which may have affected the fouling intensity of the membranes used.

Acknowledgments

Research was conducted as part of the interdisciplinary project sustainable technology for the staged recovery of an agricultural water from high moisture fermentation products RECOWATDIG carried out under the water joint programming initiative co-financed by 20 funding partner organizations from 18 countries and with the support of the European Commission (full list: http://www.waterjpi.eu/images/documents/JC2018/water jpi 2018 joint_call_participating_countries.pdf).

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