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The Use of Flat Ceramic Membranes for Purification of the Liquid Fraction of the Digestate from Municipal Waste Biogas Plants

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Abstract: Due to the rising water deficit in agriculture, digestate is increasingly being considered not only as an alternative fertiliser but also as a potential source of water. The use of recycled water for crop irrigation requires that it be treated in such a way that contaminants from the fermented biomass are not returned to the environment. Membrane processes can provide promising results in this regard. This study seeks to achieve membrane filtration using flat ceramic membranes for effective digestate liquid fraction treatment from a municipal waste biogas plant. Membranes of 1, 5, 15, and 50 kDa, and 0.14 and 0.45 µm are examined. The results obtained show that the application of a sedimentation process, as a preliminary step in the purification of the digestate, allows for a significant reduction in the content of contaminants in the solution. By analysing the effectiveness of the liquid fraction of the digestate purification in the sedimentation-membrane filtration process using flat ceramic membranes, it can be stated that all the membranes tested can be applied in the digestate purification. With an increase in the cut-off value, a deterioration in the quality of the digestate can be observed. The use of the sedimentation process before the membrane process not only improves the final quality of the digestate but also reduces the intensity of membrane fouling.

Keywords: digestate; biogas plant; pressure-driven membrane processes; ceramic flat membranes

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1. Introduction

The reasons for the change in environmental protection strategies towards so-called "clean production" are both the constantly increasing pollution of water, soil, and air and a reduction in the availability of non-renewable raw material resources. This can be implemented, for example, by pollution prevention to reduce the amount of generated waste or by producing reusable products. "Clean production" is in line with the concept of a circular economy, and according to its principles, the generated waste should be new, full-value products [1,2]. In 2015, the European Commission published a circular economy package, which includes the "Roadmap to a Resource Efficient Europe" [3] and the announcement "Towards a circular economy: A zero waste programme for Europe" [4]. According to this, a circular economy is an economic model involving growth without increasing resource consumption by changing the structure of production chains and transforming industrial systems.

In both municipal and agricultural biogas plants, the principles of such operation include the use of the biological fermentation process to produce biogas. This technology is seen as one of the forward-looking solutions in the waste management and renewable energy production sector [5–7].

Due to their long activity and intensive research in renewable energy production and alternative waste management, the European leaders in the biogas production sector are countries such as Germany, the United Kingdom, Italy, the Czech Republic, and France [8–10].

According to the Energy Regulatory Office, there were more than 320 biogas plants operating in Poland in 2020 [11,12]. The dominant type of biogas plant (more than 100 installations) are biogas plants that generate energy from biogas produced in sewage treatment plants. The second type, in terms of numbers, are biogas plants generating energy from biogas coming from landfills (also over 100 installations). Only 1% is biogas plants that generate energy from biogas produced from mixed sources. Currently, the largest increase is noted for agricultural biogas plants (processing mainly agricultural waste) with 114 installations, which constitutes 31% of all biogas plants in Poland. The capacity of all of Poland's agricultural biogas plants is ~490 million m^3/year with a total installed capacity of 120.4 MW (as of 27 May 2021) [12].

An inseparable element of the operation of any type of biogas plant is the formation of the digestate. Its characteristic features include the varied physical and chemical properties, depending on the type of raw materials used in the production of biogas, their sources and the fermentation technology used. The digestate consists mainly of undecomposed organic compounds, minerals, and biomass of methane bacteria [13]. The digestate is highly hydrated (2–5% dry matter) [14]. It is also common to have weak alkaline properties, usually with a pH of between 7.5 and 9.0 [15,16].

When starting a biogas plant, the possibility of managing the digestate becomes an extremely important aspect, which depends on the type of waste used. In accordance with current regulations [17], the processes of municipal waste biological conversion can be classified as recovery (e.g., R3 and R10) or neutralisation (D1, D2, and D8).

The management of the resulting digestate is cumbersome, both logistically and in terms of investment and legality. For this reason, there is a steadily growing interest not only in improving waste treatment technologies in biogas plants, but also in developing methods to manage post-fermentation waste [18]. A solution to this problem can be digestate separation into solid and liquid fractions using centrifuges, screw presses, and sieves [19]. The liquid fraction can be used to irrigate fields and the digester feedstock [20]. In contrast, the solid fraction, in addition to fertilising fields in spring and summer, and composting in winter [21,22], can be used to produce fertiliser in granular form [23]. Therefore, the concept of digestate management could be an opportunity for many small and medium-sized enterprises that could be involved in the implementation of municipal biogas plants at this stage.

Since the digestate is treated not only as an alternative fertiliser [24,25], but also as a source of water, it needs to be treated in such a way that the contaminants from the fermented biomass are not returned to the environment. Pressure-driven membrane processes that produce highly purified water streams can provide promising results in this regard [26]. In the purification of digestate, among others, an ultrafiltration process is applied [27,28] that retains fine suspensions, colloids, bacteria, and viruses. The transport mechanism is sieve-like, which means that particles larger than a pore diameter do not pass through the membrane. The applied transmembrane pressure is in the range of 0.1 to 1.0 MPa.

Both ceramic and polymeric materials are used to manufacture membranes [29]. Membranes manufactured from organic materials, such as polyethersulphone and polysulphone, or cellulosic materials are most commonly used in water or wastewater treatment membrane systems operating worldwide. These membranes are characterised by easy material processing, relatively low costs, and a wide variety of properties. However, because these membranes have varying resistance to strong acids, bases, and chemical oxidants, and may be susceptible to biodegradation, the possibilities for their use are sometimes limited [30].

An alternative to polymeric membranes may be ceramic membranes characterised by high mechanical, chemical, biological, and thermal resistance, steam sterilisability, long lifetimes, and the possibility of using used membranes as ceramic materials in other fields of economy [31]. Their wide use contributes to the saving of raw materials and energy [32]. Ceramic membranes have an asymmetric structure, consisting of a macroporous support and a thin skin layer, which determines the separation properties of the membrane [33].

The pore size of commercially available ceramic membranes ranges from 0.005 to 1 µm. In typical membranes, the thickness of the support layer is 1–3 mm. The skin layer of inorganic ultrafiltration membranes is a few µm thick and is usually formed from zirconium oxide (ZrO_2), aluminium oxide (Al_2O_3), titanium oxide (TiO_2), or cerium oxide (CeO_2) [31,34].

While numerous studies have been conducted on the liquid fraction of digestate from agricultural biogas plant purification (e.g., [14,35,36]), there are very few reports on municipal waste biogas plant digestate liquid fraction treatment. Moreover, despite there being reports present in the literature [14,37] on the use of polymeric membranes in the purification of the liquid fraction of digestate, to our knowledge, there are no literature reports on the application of flat ceramic membranes in this scenario.

Our previous study [38] determined the effectiveness of different polymeric membranes in the treatment of the digestate liquid fraction from municipal waste biogas plants. Since polymeric membranes have not shown satisfactory purification efficiency and exhibit many disadvantages, as discussed above, we decided to conduct a study to determine the suitability of ceramic membranes for this purpose with a special focus on organic matter separation.

2. Materials and Methods

2.1. Materials

Flat ceramic membranes from Tami Industries with different cut-offs were used in this study. The characteristics of the membranes tested are shown in Table 1.

Table 1. Characteristics of flat ceramic membranes used for membrane filtration (compiled on the basis of [39] and our own research).

MEMBRANE TYPE	Cut-Off	Active Layer	Contact Angle, °	Filtration Area, cm^2	Nominal Thickness, mm	pH Range	Max Pressure, MPa	Max Temp., °C
Fine UF	1 kDa	TiO_2	59.6	56	2.5	2–14	0.4	350
Fine UF	5 kDa	TiO_2	57.6			2–14		
UF	15 kDa	ZrO_2	43.8			0–14		
UF	50 kDa	ZrO_2	42.4			0–14		
MF	0.14 µm	ZrO_2-TiO_2	36.6			0–14		
MF	0.45 µm	ZrO_2-TiO_2	36.7			0–14		

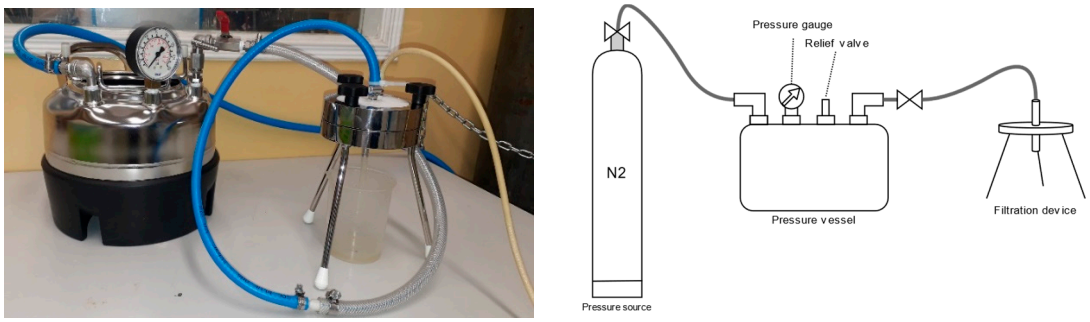
The tests were carried out using the liquid fraction of digestate from a waste biogas plant processing an organic fraction of municipal waste located in a Polish waste management plant (50°53′15.5″ N 17°23′28.0″ E). Separation of the digestate liquid fraction was carried out using sedimentation centrifuges. The characteristics of the experimental solution are presented in Table 2.

Table 2. Properties of the liquid digestate fraction from the municipal waste biogas plant.

pH	7.2
Conductivity, mS/cm	22
Total solids, mg/dm^3	18,090
Chemical oxygen demand (COD), mg O_2/dm^3	6190
Biochemical oxygen demand (BOD), mg O_2/dm^3	2170
Dissolved organic carbon (DOC), mg C/dm^3	3050
NH^{4+}-N, mg N/dm^3	1742
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	$111 \cdot 10^6$
thermophilic bacteria, CFU/cm^3	$163 \cdot 10^2$

2.2. Methods

Tests to determine the transport and separation properties of ceramic membranes were carried out at a Sterlitech laboratory installation with a 316 SS pressure chamber with a volume of 3.8 dm^3. This chamber is designed to work with flat ceramic membranes (Figure 1). The process was run in a dead-end mode at transmembrane pressures ranging from 0.1 to 0.3 MPa, with 3.5 dm^3 of digestate used for each experiment. Each experiment was duplicated.



(**a**) (**b**)

Figure 1. Sterlitech pressure filtration vessel: (**a**) photograph; (**b**) diagram.

The effectiveness of the process was determined by measuring the organic substance contents, expressed by COD, BOD_5, and DOC present in the solution before and after treatment. Changes in the total solid content were also analysed. COD and BOD_5 were determined with the use of standard bichromate and dilution methods, respectively. The DOC concentration was measured using a HACH IL 550 TOC-TN carbon analyser. The total solid content of the samples was determined by a weight method.

The separation efficiency was measured by the value of the retention factor (R) determined from:

$$R = \left(1 - \frac{c_p}{c_f}\right) \cdot 100, \ \% \quad (1)$$

where:

c_p—concentration of contaminants in permeate, g/m^3;

c_f—contaminant concentration in the feed, g/m^3.

R > 90% values were determined with an error of less than 1%. The retention factor was calculated for COD (R_{COD}), BOD_5 (R_{BOD5}) and DOC (R_{DOC}).

Transport properties of the membranes were evaluated by determining the permeate flux (J). It is defined as the volume of permeate obtained from a unit area of the membrane per unit time:

$$J = \frac{V}{A \cdot t}, \quad \frac{m^3}{m^2 \cdot d} \quad (2)$$

where:

V—volume of permeate, m^3;

A—membrane surface area, m^2;

t—filtration time, d.

The estimation of membrane fouling intensity was performed by calculating the value of membrane relative permeability J/J_0, expressed as a quotient of permeate flux J to redistilled water flux J_0 of a new membrane. Additionally, the membrane resistance values were specified to determine the susceptibility of membranes to fouling. When redistilled water was filtered, the membrane resistance value R_{mem} was determined from the Hagen-Poiseuille equation:

$$J = \frac{\Delta p}{\mu \cdot R_{mem}}, \quad \frac{m^3}{m^2 \cdot d} \quad (3)$$

where:

Δp—transmembrane pressure, Pa;

μ—dynamic viscosity coefficient, Pa·s;

R_{mem}—membrane resistance, m^{-1}.

However, in the case of digestate liquid fraction filtration, the Hagen-Poiseuille equation takes the form:

$$J = \frac{\Delta p}{\mu \cdot \left(R_{mem} + R_f + R_{pol}\right)}, \quad \frac{m^3}{m^2 \cdot d} \tag{4}$$

where:

R_f—membrane resistance resulting from blocking membrane's surface and pores by substances in solution, m^{-1};

R_{pol}—resistance of polarisation layer, m^{-1}.

The sedimentation process, which was aimed at preliminary purification of the solution before filtration on the membranes, was carried out for 1–120 h.

3. Results

Preliminary studies were conducted to determine the effect of sedimentation time on the effectiveness of reducing the COD values and total solid concentration of the liquid fraction of the digestate (Figure 2). It was observed that the use of sedimentation, as a pre-treatment method, allows for a reduction in the contaminant content of the solution. Analysis of the kinetic curves revealed that the optimal sedimentation time was 72 h. After exceeding this sedimentation time, an increase in the efficiency of the digestate purification was small and over time, the efficiency of the process stabilised at a constant level. After 72 h of sedimentation, the COD value was reduced by 9.2% and the total solid content by 2.4%. Sedimentation allowed the solution to partially clarify, which resulted not only in a visual change in its quality, but also reduced the content of larger particles that could block the membrane during digestate filtration.

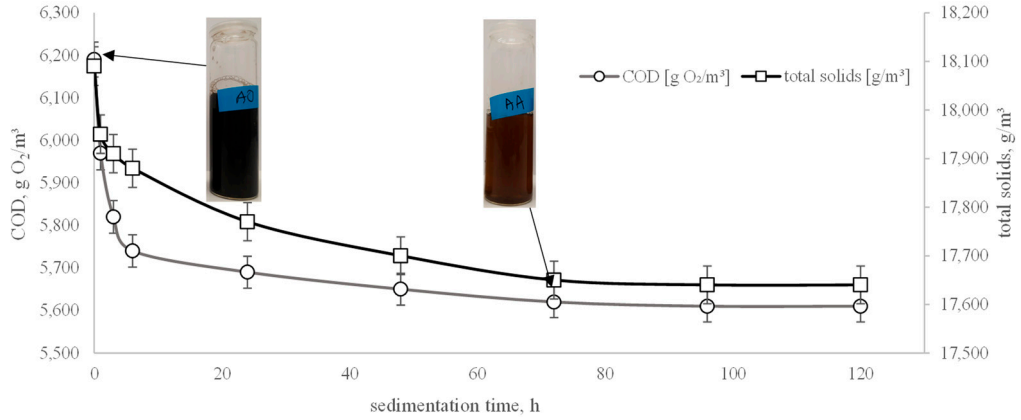


Figure 2. Influence of sedimentation time on the effectiveness of COD and total solid reduction in the liquid fraction of digestate.

Based on the analysis of the obtained measurement results, it was determined that all samples of the digestate liquid fraction, subjected to further filtration on membranes, would be purified using 72 h of sedimentation (except for the examination of preliminary sedimentation influence on the membrane relative permeability).

The effectiveness of the treatment of the liquid fraction of digestate on flat ceramic membranes was evaluated by analysing the influence of the membrane cut-offs and the process parameters (transmembrane pressure and process duration) on the change of the COD, BOD_5, and DOC values. The results of the digestate purification by the sedimentation-membrane filtration using flat ceramic membranes, presented in Figure 3, show that the membranes tested can be used for digestate purification, although a deterioration in

permeate quality could be observed as the cut-off value increased. Since, for all tested membranes, the separation of contaminants was mainly determined by a sieve mechanism, the separation efficiency was significantly influenced by the ratio between the size of the contaminants and the pore diameter of the membrane, which is, for example, ~1 nm for a 1 kDa membrane, ~2 nm for a 5 kDa membrane and ~6 nm for a 50 kDa membrane [40].

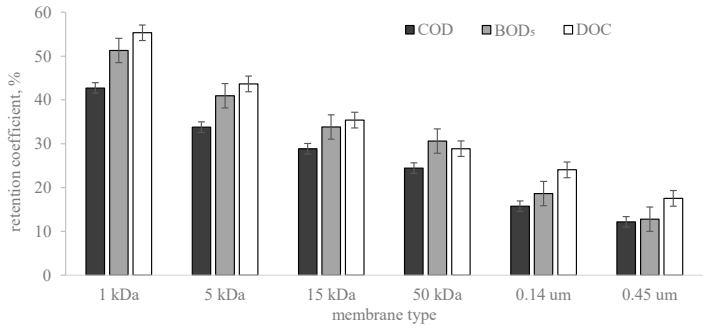


Figure 3. Effect of membrane type on the COD, BOD_5 and DOC reduction in the digestate liquid fraction using sedimentation-membrane filtration (TMP 0.3 MPa).

The highest organic substance content in the permeate, which is connected with its poorest quality, was obtained when microfiltration membranes were used (pore diameters of 0.14 and 0.45 µm). It was observed that the use of more compact membranes resulted in an improvement in the purification efficiency of the digestate. The smaller the membrane cut-off value, the lower the amount of contaminants that pass into the permeate and these are substances with a smaller molecular weight. The best separation was observed when a 1 kDa cut-off membrane was used. For example, at a transmembrane pressure of 0.3 MPa, the values of R_{COD}, R_{BOD5}, and R_{DOC} were 43%, 51%, and 55%, respectively. Comparing the results obtained for ceramic membranes with our previous studies [28,38] involving polymeric membranes with similar pore size and liquid fraction of digestate from a municipal waste biogas plant and an agricultural biogas plant, a higher removal efficiency of organic pollutants was found for ceramic membranes. For example, the 10 kDa PES polymeric membrane allowed for R_{COD}, R_{BOD5}, and R_{DOC} up to 39%, 43%, and 27%, respectively (TMP 0.3 MPa, liquid fraction of the digestate from a municipal waste biogas plant) [28].

It was also observed (Figure 4) that the magnitude of the driving force forcing the transport through the membrane had no significant effect on the permeate quality. In the analysed pressure range (0.1–0.3 MPa), the content of organic compounds remained at a comparable level. For example, for a pressure of 0.1 MPa and a 15 kDa membrane, the values of COD and DOC retention factors were 30% and 36%, respectively. Increasing the transmembrane pressure to 0.3 MPa resulted in values of 29% and 35%, respectively.

It was also verified whether the time of the membrane filtration process affects the efficiency of organic pollutant elimination from the treated solution. The results of changes in the retention values of COD and DOC for each of the membranes tested are shown in Figure 5. These studies show that the effectiveness of the separation of pollutants on the membrane is practically unchanged with time. Longer filtration did not affect separation properties of the membrane. The retention of organic substances did not change. For example, a membrane with a cut-off of 1 kDa allowed the COD retention coefficient to reach ~42% and the DOC reached ~53%, and during the analysed time interval, changes in separation efficiency did not exceed 2%. The constant value of the retention coefficient of pollutants during membrane filtration also indicates that a sieve mechanism was decisive for the elimination of pollutants and not, for example, the phenomenon of sorption on the membrane, the influence of which for some membranes was observed at the beginning of the membrane filtration process.

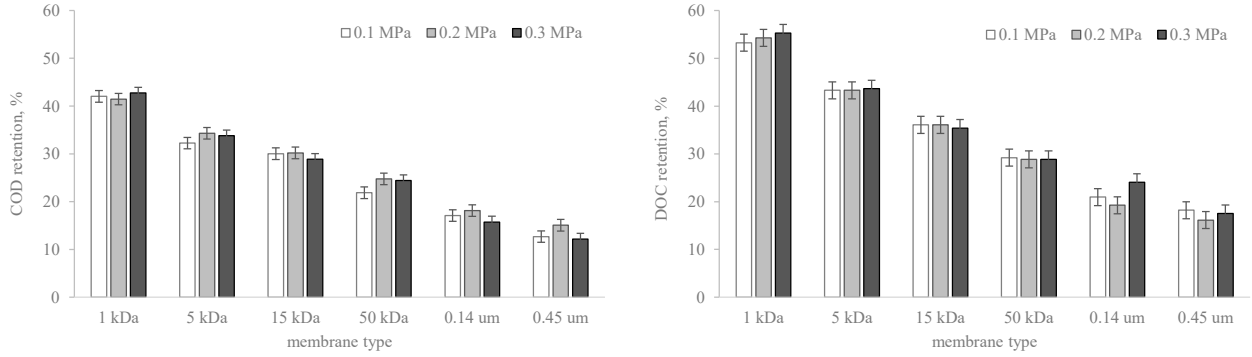


Figure 4. Influence of transmembrane pressure on COD (**a**) and, DOC (**b**) retention.

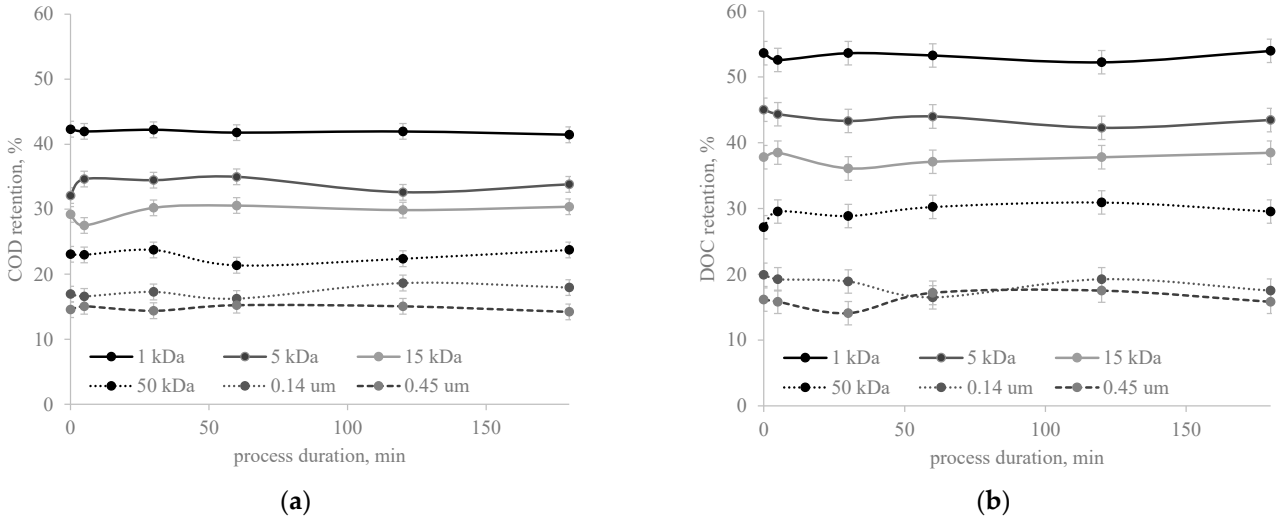


Figure 5. Influence of membrane filtration time on the reduction efficacy of COD (**a**) and DOC (**b**) during digestate liquid fraction treatment (TMP 0.2 MPa).

Summarising the obtained results of the separation of organic pollutants obtained during the purification of the liquid fraction of digestate on flat ceramic membranes, it can be stated that only the use of compact ultrafiltration membranes (1 kDa) allows for a significant reduction in the values of indices characterising the concentration of organic compounds. However, the concentration of organic contaminants in the permeate is still high enough to make direct irrigation of most crops impossible. Hence, the obtained permeate should still be post-treated using other membrane processes, e.g., nanofiltration, or physicochemical processes (e.g., adsorption). However, it can be supposed that, given the pore diameters of dense ultrafiltration membranes of a few nm and the size of microorganisms that may be present in the liquid fraction of the digestate (tens of nm to a few μm), the permeate obtained is microbiologically safe.

From the point of view of the suitability of individual membranes for purification of the studied solution, attention should be paid not only to their separation properties but also to their transport performance. The membranes tested differed not only in absolute hydraulic capacity (Figure 6), which was mainly due to differences in pore diameters, but also in susceptibility to fouling. Figure 7 shows the effect of transmembrane pressure values on the relative permeability values of the tested membranes in order to investigate their blockage intensity. The analysis of the obtained results shows that, for all membrane types, an increase in the value of transmembrane pressure resulted in a decrease in the J/J_0, which indicated a higher intensity of membrane fouling. This effect was most visible

when membranes with 5 and 15 kDa cut-offs were used. Moreover, it was noted that an increase in the cut-off value of the membranes, and thus an increase in the membrane pore radius, resulted in a decrease in the relative permeability of the membranes. This was most evident for the membranes with pore diameters of 0.14 and 0.45 µm, for which J/J_0 was ~0.01 almost regardless of the applied pressure. These microfiltration membranes were the most susceptible to blockage among those tested. This confirms literature reports that membranes with larger pore diameters (in this case,, microfiltration membranes) are much more susceptible to fouling than is the case with more compact membranes, e.g., ultrafiltration ones [41]. The former are dominated by fouling caused by particles from the feed phase, blocking the membrane pores.

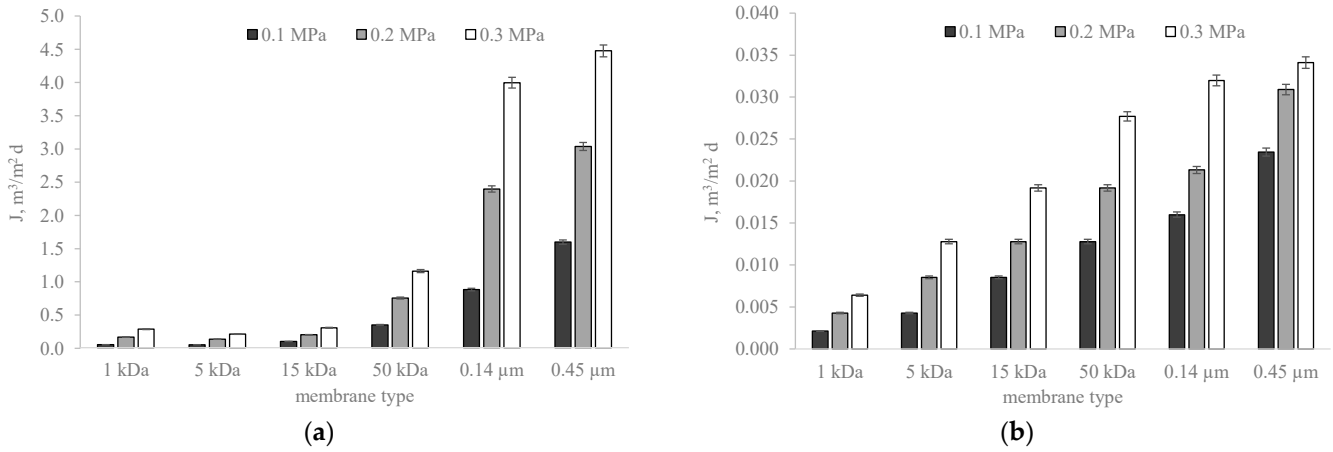


Figure 6. Influence of TMP on membrane permeability of (**a**) redistilled water and (**b**) digestate liquid fraction (after sedimentation pre-treatment).

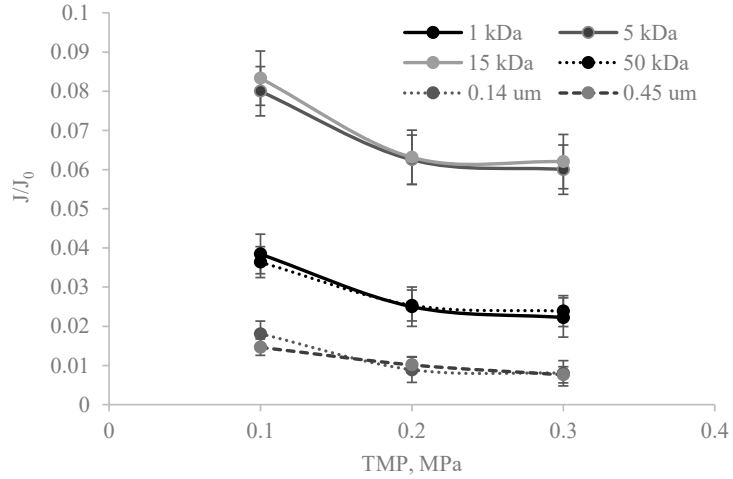


Figure 7. Influence of TMP on membrane relative permeability of digestate liquid fraction.

As shown in Figure 6, the flux values of the liquid fraction of the digestate were much smaller than those measured for redistilled water. This was due to the increase in flow resistance values due to membrane fouling and formation of polarisation layer at the membrane surface (Figure 8). For example, when the redistilled water was filtered, the 50 kDa cut-off membrane resistance values at 0.1 and 0.3 MPa amounted to 2.45×10^{13} and 2.23×10^{13} m^{-1}, respectively. Meanwhile, for ultrafiltration of the liquid fraction of the digestate, the total flow resistances ($R_{mem} + R_f + R_{pol}$) were 6.74×10^{14} and 9.34×10^{14} m^{-1}, respectively. Comparing the results obtained during water and digest filtration for transmembrane pressures of 0.1–0.3 MPa, it was observed that the total resistances of more compact membranes (1 and 5 kDa) did not change significantly with increasing pressure.

For the membrane with larger pore diameters, the resistance slightly increased with the increase in transmembrane pressure, which could be a result of the thickening, due to the higher driving force, of the structure of the filtration cake forming on the membrane surface (in case of filtration of digestate solution). This could also have intensified the phenomenon of particle penetration into the membrane structure, especially the microfiltration one, resulting in an increase in flow resistance.

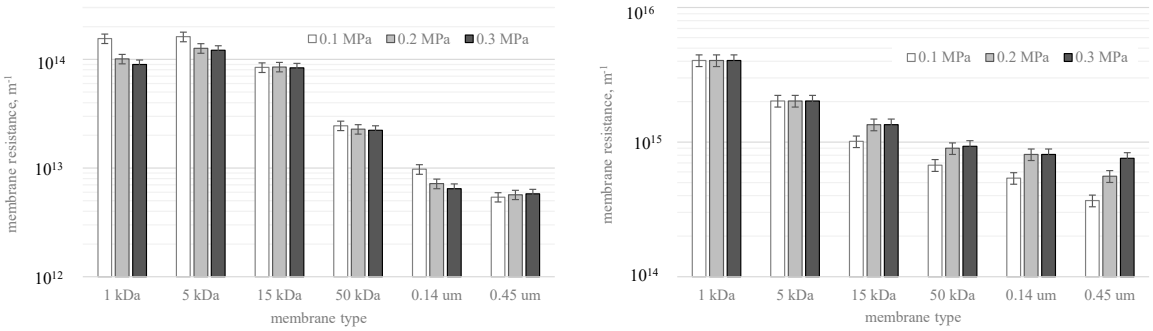


Figure 8. Total flow resistance changes during filtration of redistilled water (**a**) and digestate liquid fraction (**b**) at different TMP.

As mentioned above, pre-sedimentation of the solution, prior to membrane separation, allowed for the removal of a certain amount of contaminants (Figure 2), contributing to the improvement of final quality of the digestate and to a reduction in the intensity of membrane blockage (Figure 9). The comparison of the values of J/J_0 ratios, obtained during the purification of the liquid fraction of the digestate in the process of stand-alone membrane filtration, with those determined for the sedimentation-membrane filtration system allows us to notice a large increase in the J/J_0 ratio, which means a reduction in the intensity of membrane blockage. This effect was observed for each of the membranes tested, and it was particularly significant for more compact membranes (5 and 15 kDa). The use of sedimentation allows us to remove from the solution compounds that, in the absence of pre-treatment, settle on the membrane surface and penetrate the membrane pores, which deteriorates the hydraulic performance, in case of running the process with a constant TMP value. For example, for a membrane with a cut-off of 15 kDa at TMP equal to 0.3 MPa, the J/J_0 value for stand-alone ultrafiltration was 0.028, while using sedimentation before the ultrafiltration process increased the ratio to a value of 0.062.

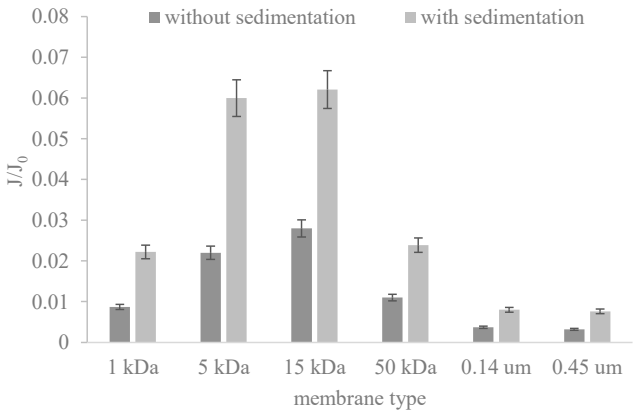


Figure 9. Influence of digestate pre-treatment on membrane relative permeability (TMP 0.3 MPa).

Evaluating the influence of the membrane operation time on the change of membrane relative permeability value (Figure 10), it was found that, due to progressive blocking

of membranes, the value of J/J_0, in the initial time of running the process successively decreases, to stabilise at a certain level afterwards. This effect is particularly evident for ultrafiltration membranes with cut-offs of 5 and 15 kDa.

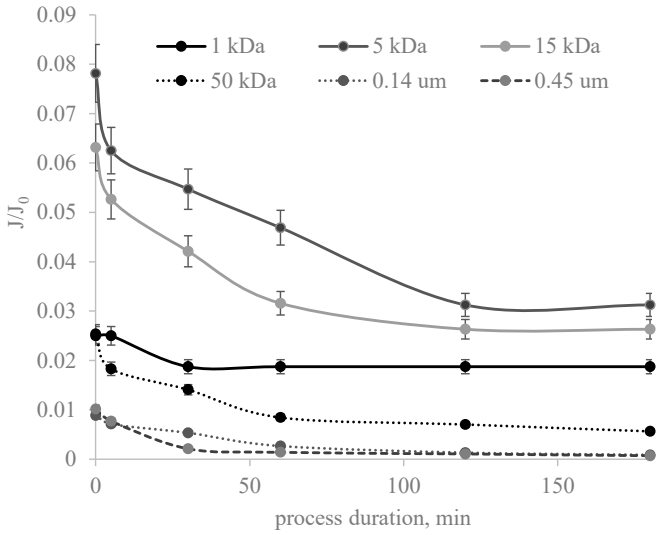


Figure 10. Influence of membrane filtration time on membrane relative permeability (TMP 0.2 MPa).

4. Conclusions

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

- all the ceramic ultrafiltration and microfiltration membranes tested can be used for the purification of the liquid fraction of digestate from municipal waste biogas plants; however, the increase in membrane cut-off or pore size results in a deterioration of the digestate quality;
- the best separation efficiency of organic substances was obtained for the most compact membrane with a cut-off of 1 kDa; the use of 1 kDa membrane allowed to reduce DOC content by up to 55%, BOD_5 up to 51%, and COD up to 43%;
- the use of sedimentation as a pre-treatment of the solution, prior to membrane separation, allows for a reduction in the content of contaminants in the solution;
- the optimum sedimentation time was 72 h, and a further extension of the sedimentation time caused the effectiveness of the process to be set at a virtually constant level; pre-sedimentation, preceding filtration on ceramic membranes, allowed for reduction in membrane fouling intensity, which resulted in the improvement of membrane transport properties;
- comparing the obtained separation efficiency with results of our earlier works [28,38] for polymeric membranes of comparable pore size, it was found that the ceramic membranes were more effective in separation of organic substances from the digestate liquid fraction.

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