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Research Paper

Anaerobic Membrane Bioreactor Coupled with Nanofiltration Applied to The Treatment of Beverage Industry Wastewater Under Soudano-Sahelian Climatic Conditions

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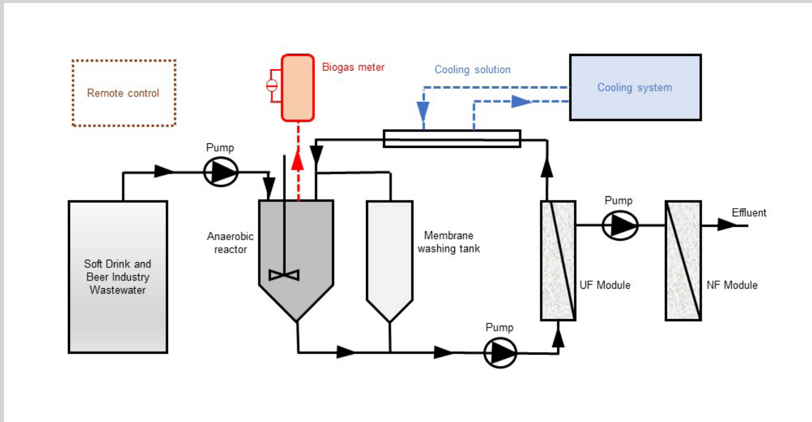
Highlights

- The paper presents the performance of AnMBR coupled with NF for industrial wastewater treatment.
- The operating conditions, pollutant removal, and biogas production of AnMBR coupled with NF have been investigated.
- The characteristics of the final effluent after treatment with the AnMBR and post-treatment with NF show satisfactory pollutant removal efficiency and allow for reuse.

Graphical abstract



Abstract

Beverage production generates large quantities of wastewater with high organic and refractory content due to the material used in the manufacturing or cleaning processes. The present study investigates the treatment of wastewater from a beverage industry with an anaerobic membrane bioreactor (AnMBR) coupled with nanofiltration (NF). Because of the favorable climatic conditions of the Sahelian context, the production of biogas has also been evaluated. The study was conducted with a pilot-scale AnMBR fitted with an external ceramic ultrafiltration membrane. The AnMBR was fed with wastewater produced from the beverage industry. A hydraulic retention time of 1.5 days was employed for the study, whilst a solid retention time (SRT) of 60 days was set. The COD load varied from 0.8 to 5.7 g COD/L/d during 123 days of operation. The effluent from the AnMBR was fed in batch mode to the pilot-scale NF equipped with a composite spiral membrane of polyamide, polysulfone, and polyester. The results obtained showed a faster acclimation of the sludge due to its familiarity with the influent. The significant variations in alkalinity of the industrial wastewater used required buffering for better control of the biomass in the reactor. The AnMBR provided over 99% turbidity removal, whilst COD removal efficiency attained was 94%. NF resulted in almost complete rejection of most ions with removal rates ranging from 90 to 100%. The biogas produced was estimated at 0.21 L biogas/g COD removed.

1. Introduction

The reuse of wastewater is becoming increasingly necessary, especially in regions where water resources are scarce and water supply systems are fragile [1–5]. It, therefore, appears clearly that wastewater, far from its waste status, is today presented as a secondary raw material, a resource and as such

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is increasingly attracting the attention of public authorities, scientists and investors. The industrial sector was the first to look into the valorization of its wastewater, either because the resource was limited, or because it allowed a return on investments.

Industries represent 17% of total freshwater withdrawals for all their activities in the world according to the Food and Agriculture Organization data [6]. Among them, the beer and soft drink industries constitute an important part of this sector in most countries [7]. They use water as one of the main ingredients for the production of beverages [8,9]. The water is used in particular for the production of beverages, packaging, rinsing, cleaning, cooling, and sanitation. The quantities of freshwater consumed are for example estimated between 2.5 and 3.5 liters per liter of carbonated drink produced [10] and between 4 and 11 liters per liter of beer produced [11]. Beverage production, therefore, generates large volumes of wastewater on a daily basis [12,13].

The nature of the pollutants and the volumes of water discharged vary according to the stages of the industrial process. The use of different raw materials, as well as the variation of the operations of rinsing the tanks, and bottles and the cleaning of the production installations lead to the great variability of the effluents generated. This discharged wastewater is highly biodegradable and constitutes various mixtures of chemicals from the raw material and from rinsing or cleaning discharges [14,15].

The use of membrane processes for the treatment of industrial wastewater has shown interesting results for the elimination of dissolved pollutants and the retention of suspended particles [16–19]. Reverse osmosis (RO), nanofiltration (NF), and electrodialysis (ED) are used in particular for the removal of ions and micropollutants in industrial wastewater [20–24] But to limit the impact of brewery wastewater on the environment, the development of compact and efficient treatment systems such as the membrane bioreactor (MBR) seems to be an appropriate alternative [9,23,25–29] The MBR offers several advantages over conventional activated sludge systems, namely the stability of the quality of the treated effluents, the ease of operation, the small footprint and the absolute elimination of bacteria and certain viruses [30]. In addition, MBR allows water to be clarified and disinfected simultaneously without the risk of the formation of halogenated organic compounds, thus allowing the reuse of treated effluents. On the other hand, on this emerging technology, very little data relating to its implementation in the Sahelian climatic and environmental context, where the beverage industries produce both beer and soft drinks, are available. With strong sunshine and average temperatures above 35 ° C for most periods of the year, of the time, anaerobic treatments in this part of Africa clearly appear to be the option of choice for degrading organic matter present in wastewater for biogas production [24,31]. If Industrial wastewater must therefore be recycled onsite for reuse, then the treatment at the outlet of the plant must make it possible to attain water quality that corresponds to the uses.

2. Material and methods

2.1. The anaerobic membrane bioreactor pilot

The AnMBR used has an external membrane configuration. It consists of a reaction part (anaerobic bioreactor), 20 liters in volume, and a liquid/solid separation part (membrane module). To maintain constant liquid volume within the reactor, 2-level sensors (rod) regulate the feed flow between a higher level and a lower level within the tank. A peristaltic pump controlled by its levels supplies the system with wastewater from a common tank and continuously provides an additional substrate. The transmembrane pressure, pH, temperature, dissolved oxygen concentration, and redox potential are recorded every 20 seconds (adjustable sampling frequency) simultaneously on a computer using the software.

The membrane allowing the separation of the treated effluent and the purifying biomass is placed outside the bioreactor. The mixed liquor recirculation loop is provided by a positive displacement pump. This system allows operation in tangential filtration. The suspension is filtered from the inside of the membrane to the outside. The characteristics of the membrane are listed in Table 1. A recirculation pump controls the tangential speed along the membrane. A back-pressure valve placed at the outlet of the membrane casing in the recirculation circuit allows the pressure inside the membrane to be increased if necessary.

In order to monitor the performance of the reactor and its control with respect to clogging, the transmembrane pressure is recorded. Operating at constant flow, an increase in clogging is associated with the corresponding increase in transmembrane pressure. The pressure is measured through pressure sensors and manometers placed at the outlet of the recirculation pump just before the entry of the membrane module, at the outlet of the membrane module, and in the circuit collecting the permeate. Depending on the measurement of the temperature of the anaerobic reactor, a cooling fluid can be injected into the jacket of the heat exchanger of the retentate circuit (recycling). The pilot used is shown in the picture in Fig. 1.

Table 1
Characteristics of membranes used for membrane bioreactor and nanofiltration pilots.

Characteristics	UF (AnMBR)	NF
Type of module	Tubular type P10	Spiral NF270-2540
Membrane materials	Ceramic	Composite (polyamide, polysulfone, polyester)
Filtering surface (m²)	0.45	2.6
Cut-off threshold	15,000 D	200 D
Membrane length (mm)	1,178	1,016
Channel diameter	6 mm	-
Provider	Pall Exekia (France)	Dow Filmtec (China)

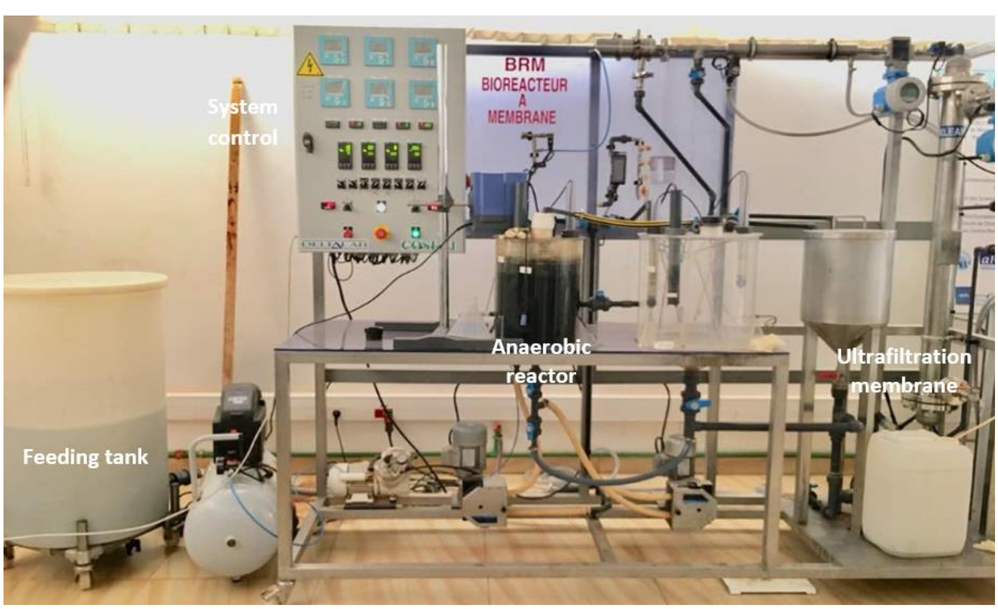

Fig. 1. The AnMBR pilot using for the beverage wastewater treatment.

2.2. The pilot-scale nanofiltration membrane system

The nanofiltration system is equipped with a high-pressure multicellular centrifugal electric pump which ensures the circulation of the feed fluid. The system is equipped with a pre-filtration device consisting of two cartridge filters: a 25 µm filter and an activated carbon filter. To protect the installation against overpressures, it is fitted with two safety valves calibrated at 5 and 10 bars respectively. A set of sensors and flowmeters allow monitoring of transmembrane pressure, flow rates, electrical conductivity, and temperature (Fig. 2). The spiral nanofiltration membrane module (NF270-2540 from Dow Filmtec) is a composite polymer composed of three layers: a polyester support layer (120 µm), a microporous polysulfone interlayer (40 µm), and a barrier layer (active layer) ultra-thin polyamide on the top surface (0.2 µm). The main characteristics of the nanofiltration membrane used are summarized in Table 1.

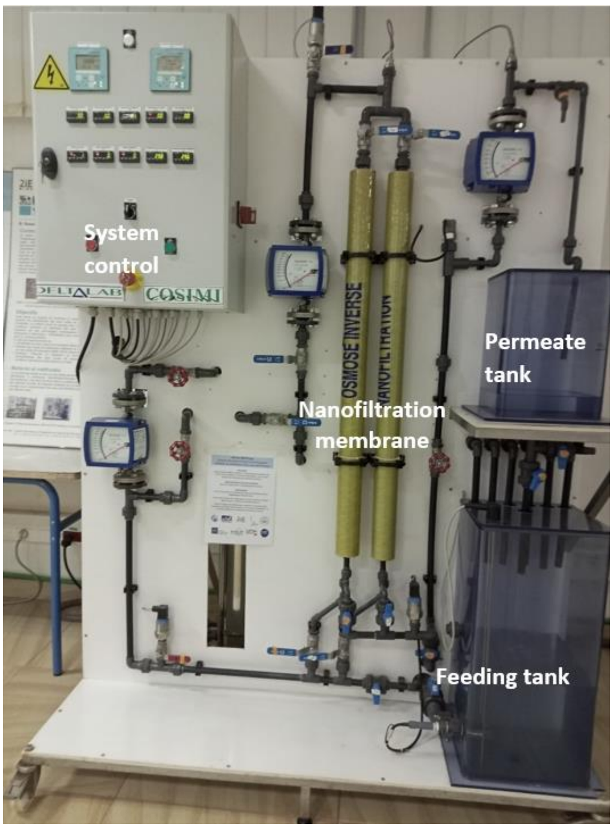

Fig. 2. Pilot-scale NF used as post-treatment for the AnMBR effluent.

2.3 The operating conditions

The sludge used for the inoculation of AnMBR comes from the anaerobic basin of a wastewater treatment plant. This station treats domestic and industrial wastewater in the city of Ouagadougou in Burkina Faso. It is a microphyte lagoon process with an estimated treatment capacity of 140,000 Inhabitant Equivalent and a total volume of approximately 5,460 m^3/d. More than 70% of the wastewater arriving at this station is from the brewery industry, which influenced the choice of inoculum. Acclimatization lasted for about 20 days and the 123-day campaign continues. Since the feed solution is the actual effluent from the brewery, samples were taken at the brewery's pre-treatment station at a frequency of 3 times per week to ensure continuous feeding. In order to validate the operating conditions in the Sahelian climatic context, the pilot operated at ambient temperature, and the recorded operating temperature values were within a range of 28 and 46 °C.

Regarding the pilot-scale NF, the permeate of the AnMBR is recovered and then introduced into the feed tank in order to remove organic and inorganic salts. The NF did not need any special operating conditions: the filtration pressure was set at 5 bars and the tests were carried out at room temperature. The operating conditions used for the experiment are given in Table 2.

Table 2
Operating conditions set for tests with the AnMBR.

Parameters	Value
Suspended solids (SS) content in the reactor	9 g/L
Volatile suspended solids (VSS) content in the reactor	6 g/L
VSS/SS ratio	67%
Solid retention time	60 days
Hydraulic retention time	1.5 days
Temperature in the reactor	28-46 °C
pH	6.2-7.8
Operating time	123 days
Flux	1.24 LMH
Organic loading rate	0.8-5.7 g COD/L/d

2.3. Analytical techniques

For the study, several parameters were followed. The choice of parameters depends on their relevance in the analysis of the evolution of biomass in the reactors and the examination of the efficiency of the treatment. For all of the operating conditions tested, grab samples of the feed, the biological suspension within the reactor, and the effluent were carried out regularly to assess the performance of the combined system under the imposed working conditions. The quantities measured relate to suspended solids (SS), volatile suspended solids (VSS) the concentrations of ionic species, organic fraction measured through the chemical oxygen demand (COD), biological oxygen demand (BOD5), kinetics biological and clogging dynamics. Analyses were performed in accordance with Standard Methods for the Examination of Water and Wastewater (APHA, 2012). Suspended Solids (SS) and Volatile Suspended Solids (VSS) were measured using standard methods AFNOR NFT 90-105 and 90-029. Chemical Oxygen Demand (COD) was measured using standard methods AFNOR NFT 90-101. Parameters including COD, N-NH_4^+, and P-PO_4^{-3} were measured using Hach kits with a UV visible spectrophotometer DR3900. Bioreactor operating data including pH, electrical conductivity, temperature, redox potential, and transmembrane pressure were monitored during system operation using sensors installed in the experimental setup.

2.4. Characterization of brewery wastewater

An intensive characterization campaign was carried out within the industrial unit during a period of high production. This sampling period was spread over a week during the holiday season corresponding to a time when production takes place continuously 24 hours a day and during 7 days of the week, with an influent flow of 3,500 m^3 per day. An automatic sampler collecting one liter of sample per hour generated 168 individual samples. The analysis focused on the parameters of organic and mineral pollution (organic matter, concentration of ions) and the physical characteristics (pH, electrical conductivity, temperature, turbidity, Suspended Solid) of the wastewater. In an industrial unit, the characteristics of the wastewater produced generally depend on the activities carried out. Production operations in particular are accompanied by moderate variations compared to cleaning and washing operations. This finding is justified by the differences in trends recorded at the level of the individual samples collected. Rinsing, cleaning, and washing activities are characterized by larger volumes of wastewater and larger variations in parameters. For an industry producing both beer and soft drinks, the alternate use of equipment for the production of different drinks requires more stringent washing conditions. These operations involve soda, phosphoric acid, trisodium phosphate, and sodium hypochlorite as appropriate. The production of beverages also involves several raw materials such as corn, malt, and hops for beer, sugars, and extracts for soft drinks. The residues of these products carried away by the washing water are found in the wastewater and are responsible for the high COD values recorded. During the production of beer, there are yeasts, Kieselguhr (diatomaceous powder used as filter media), and spent grain (residues from the brewing of cereals), as the main components of suspended matter. The real beverage industry wastewater had a COD of 4,590±2,210 mg/L, pH of 10.6±1.2, electrical conductivity (EC) of 4,280±2,416 µS/cm, total dissolved solids (TDS) of 1,335±728 mg/L, turbidity of 568±86 NTU, calcium (Ca^{2+}) of 24±4 mg/L, magnesium (Mg^{2+}) of 8±1 mg/L, total Kjeldahl nitrogen (TKN) of 10±1 mg/L, nitrate (N-NO_3^-) of 92±12, phosphate (P-PO_4^{3-}) of 270±15 mg/L, sulfate (SO_4^{2-}) of 18±5 mg/L, sodium (Na^+) of 550±48 mg/L, potassium (K^+) of 67±9 and chloride (Cl^-) of 108±26 mg/L.

Results

3.1 Dynamics of the purification community in the reactor

The performance of biological reactors is linked to the activity of the purifying biomass, in particular its ability to oxidize organic and mineral matter in the presence of various pollutants. However, the passage of microorganisms from one environment to another can affect their activity and therefore their ability to degrade pollution [32,33]. This is why monitoring the growth and activity of biomass is of particular interest for biological degradation processes [34].

Raw wastewater is taken regularly (every two days) on the site of the industrial unit where strong variations in the COD composition have been demonstrated, which leads to significant fluctuations in the incoming volume load between 1235 and 8560 mgO_2/L. The AnMBR was seeded with sludge from an anaerobic reactor from an aerated lagoon treatment plant. Regarding biomass, the evolution of SS and VSS over time is represented by the curves in Fig. 3.

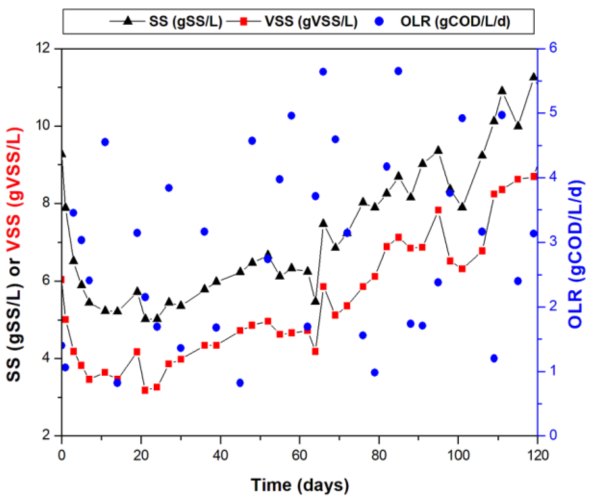

Fig. 3. Evolution of SS and VSS in the reactor.

The Suspended Solid content was 9.3 g/L and the Volatile Suspended Solid was 6.0 g/L. The curve shows a decay phase linked to the change of substrate and operating conditions (days 0-8) [35], then a lag phase (constant concentration from day 8 to 21), and then a phase of almost continuous growth over the remaining 100 days. The sludge adapted very quickly to the new conditions and experienced continuous growth during the experimentation period: the adaptation phase, therefore, lasted 21 days. However, a critical evaluation of the biological performances (Fig. 4) showed it was expedient to prolong this phase to 60 days, which yielded 95% removal efficiency on COD. The sudden variations in the incoming load do not seem to have an impact on the evolution of Suspended Solid or the purification efficiency. They slightly influence the output COD values (Fig. 4). Finally, there is an increase in the VSS/SS ratio. This fell from 63% at the start of the experiment to 86% at the end. It can therefore be concluded that the applied operating conditions led to greater production of biomass (organic matter) to the detriment of mineral matter.

In order to quantify the sludge production and assess the observed conversion rate, the evolution of the cumulative sludge production within the bioreactor was calculated over time. This calculation considers (i) the mass of sludge extracted to reach the solid retention time (age of the sludge) and (ii) the accumulation of sludge in the reactor. The daily sludge production and the cumulative sludge production during the study were therefore evaluated from equations 1 and 2:

$$P_X = Qw \times VSSR + VR \times \Delta VSSR / \Delta t \quad (1)$$

$$P_X\ cumulative = \Sigma P_{Xi} \quad (2)$$

Where P_X is the daily sludge production (g/d), P_X cumulative is the cumulative sludge production (g/d), Qw is the purge flow (L/d), VR is the volume of the reactor (L), VSSR is the volatile suspended solids in suspension in the bioreactor (g/L), ΔVSSR is the variation of volatile suspended solids in suspension in the bioreactor (g/L) and Δt is the time variation (d).

Fig. 5 which represents the evolution of the cumulative production of sludge over time shows fairly rapid stability of the system. It is indeed accepted that a process is considered stable when the linearity of the cumulative P_X is obtained [36]. Thus after 21 days of operation (adaptation phase), the reactor seems to have reached first stability but the slope increases after day 64. This increase in slope is linked to the efficiency of the system. In fact, Fig. 5 shows that the latter evolves linearly within a range from 30 to 95%, between days 21 and 64 to stabilize around 95% for the rest of the tests (days 64-123). The average sludge production rate beyond day 64 is estimated to be 0.129 ± 0.026 gVSS/L/d. Taking an average volume load of 3.8 gCOD/L/d, and a purification efficiency of 95%, the calculation gives an observed conversion rate of 0.045 kgVSS/kgCOD. This rate falls within the range commonly accepted for anaerobic treatments [37,38]

To complete the biomass monitoring, Fig. 6 shows the images of the microscopic observations at the start (a) and the end of the study (b). It appears that from a sparse and isolated microbial community in the inoculum, the purifying biomass has developed and the result is a dense and clustered flora. The increase in the density of microorganisms that appears there confirms the previous observations.

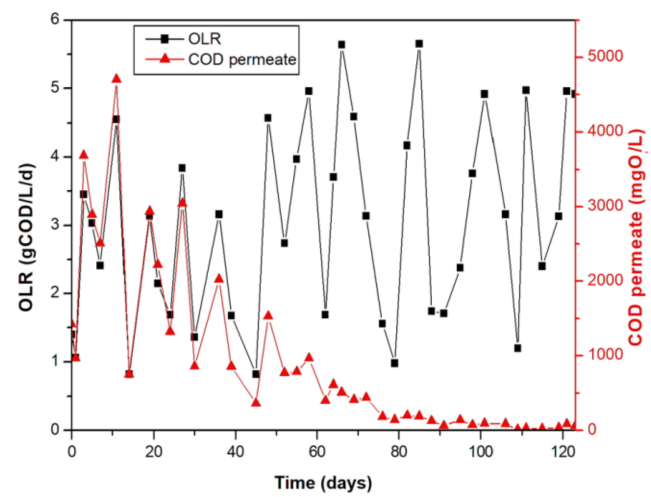

Fig. 4. Evolution of organic load and COD of AnMBR permeate during treatment.

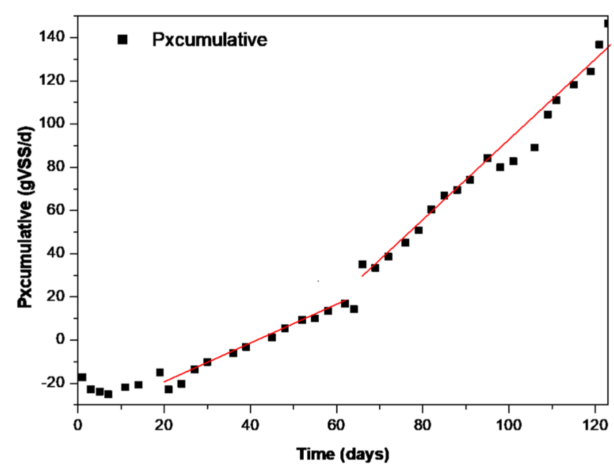

Fig. 5. Evolution of cumulative sludge production.

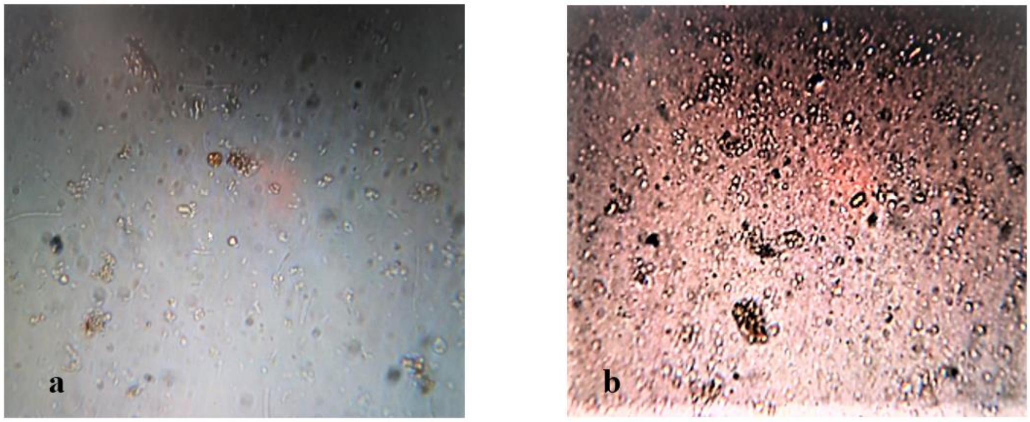

Fig. 6. Microscopic observation of the density of microorganisms. a) density of microorganisms in the inoculum on an x20 magnification. b) density of microorganisms in the reactor on an x20 magnification.

3.2 Purification performance of AnMBR

The purification performance of the treatment systems studied will be analyzed by monitoring the COD in feeds and permeates over time.

Fig. 7 shows the COD concentrations as well as the COD removal efficiencies in the feed and permeate. The COD values ranged from 1,235 to 8,560 mgO_2 / L leading to an average value of 4,590 ± 2,210 mgO_2 / L. Other parameters analyzed recorded mean values of 4,280 ± 2,416 µS/cm for electrical conductivity, 10.6 ± 1.2 for pH, 568 ± 86 NTU for turbidity, 550 ± 48 mg/L for sodium and 110 ± 26 mg / L for chlorides (Table 3). Low COD removal percentages (30 to 40%) were recorded at the initial start of the experiment, this corresponds to the period of acclimatization period of the biomass. Beyond day 24, the rate of elimination of organic pollution increases linearly. From day 64, the reactor appears to have reached peak performance with COD removal percentages greater than 90%. In addition, and despite still strong fluctuations in the wastewater feed, the variations in concentrations in the permeate are smoothed out, which reinforces the value of biological processes to attenuate load fluctuations. The average COD content in the reclaimed water during this period is estimated at 290 ± 60 mg / L, which corresponds to an average COD removal percentage of 94 ± 2%. The residual COD concentration in the permeate varied from 4,102 to 112 mgO_2 / L. The maximum value was obtained at the start of the experiment during the biomass acclimatization phase. During the stability phase (from day 64) the COD content in the permeate remained below 400 mgO_2/L despite the strong variations in the feed. The results obtained are consistent with those reported by [39–41] who noted that COD reductions vary from 70 to more than 95% depending on the nature of the membranes and the type of suspension that can be obtained. In contrast, higher rates of abatement have been obtained by other authors. Indeed, [42] reported a reduction of 99% of COD and 100% of organic matter at a temperature of 30 °C and a pH of 6.9 during the treatment of brewery wastewater with an anaerobic MBR using a ceramic membrane. This is also the case with [43] who obtained a reduction of 98% during the treatment of synthetic brewery effluents with an anaerobic membrane bioreactor operating at a COD volume load of 10 gCOD/L/d. Moving further, it would then be appropriate to investigate the organic matter that makes up the output COD: substrate, hard COD, and metabolite. Also, the results obtained make it possible to note that with a reduced acclimatization phase of 60 days (> 30% for 21 days then the yield goes from 30 to 94% in 39 days), the AnMBR tested led to performance remarkable for the treatment of industrial wastewater with large variations in organic load [44–46].

3.3 Evolution of transmembrane pressure and evaluation of clogging

The AnMBR is equipped with a ceramic membrane housed in the housing. Pressure sensors installed on the experimental setup allowed continuous recording of the transmembrane pressure (TMP) during the operation of the system. The evolution of the TMP is presented in Fig. 8. The curve representing the evolution of the transmembrane pressure **(**TMP) over time in Fig. 8 shows that it increases regularly during the experiment. The inlet pressure was set at 2.5 bar and the TMP varied from 0.007 to 1.689 bar during testing. This change is more significant from day 83. Since the inlet pressure is kept constant, the increase in the SS concentration in the reactor could be the cause of this change in the TMP. Also, the variations in organic load in the feed solution seem to be felt at the level of the TMP, the variation of which has lost its regularity over time.

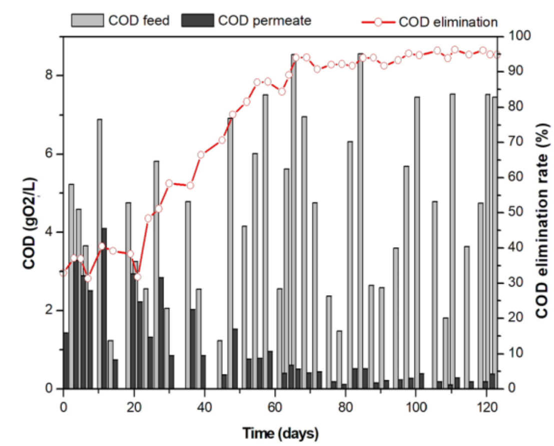

Fig. 7. Feed and AnMBR permeate COD evolution.

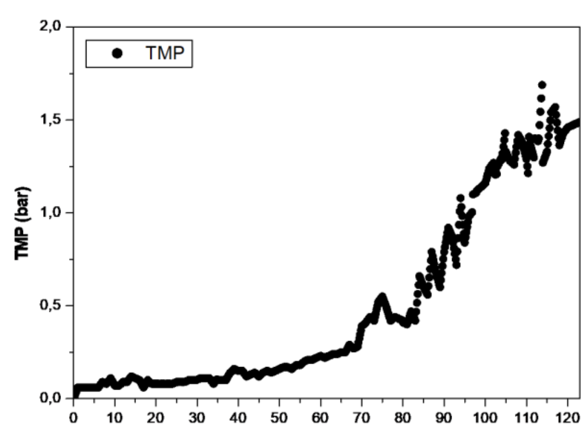

Fig. 8. Evolution of TMP during filtration with AnMBR.

Table 3
Pollution parameter and elimination rate.

Parameters	Raw wastewater	AnMBR permeate	NF permeate	Reduction rate (%)		
				AnMBR	NF	AnMBR+NF
COD (mg O_2/L)	4590	290	10	94	97	100
Conductivity (µS/cm)	4280	3220	206	25	94	95
TDS (ppm)	1335	1289	71	3	94	95
Turbidity (NTU)	568	9	3	98	66	99
Ca^{2+} (mg/L)	24	21	2	13	92	93
Mg^{2+} (mg/L)	8	8	1	0	91	91
TKN (mg/L)	34	30	14	12	53	59
N-NH_4^+ (mg/L)	92	73	19	31	89	92
P-PO_4^{3-} (mg/L)	270	232	9	14	96	97
Na^+ (mg/L)	550	527	41	4	92	93
K^+ (mg/L)	67	58	6	13	89	91
Cl^- (mg/L)	108	81	18	25	77	83

3.4 Post-treatment of AnMBR effluents with nanofiltration

The use of AnMBR resulted in mean COD values of 290 ± 60 mg/L and sodium of 527 ± 64 mg/L in the permeate. With regard to the regulations, even if these levels allow direct discharge into the sewer network, they do not allow the reuse of treated wastewater. This is why the AnMBR permeate was subjected to a post-treatment by nanofiltration. The tests were carried out batch-wise with an inlet pressure of 5 bar at room temperature (28-42 ° C during the study period). The results show that the reduction rates obtained with nanofiltration are between 53 and 96%. The NF, therefore, allowed a good reduction in the concentration of the main pollutants (Fig. 9). The highest percentage of elimination is obtained with orthophosphate ions (trivalent), the concentration of which has dropped from 270 mg/L to 9 mg/L in the final permeate. Sodium was removed at 92% with a final concentration of 41 mg/L on average. The average COD concentration in the permeate is 10 mgO_2/L leading to a total removal yield of 99.8% for the AnMBR-NF coupling. Comparable elimination percentages have been reported by [47] for COD and sodium during the treatment of brewery wastewater with NF. As expected, NF is a good tertiary treatment for the removal of electrical conductivity (EC) and therefore total dissolved salts (TDS) [48]. Coupling the NF with the AnMBR, therefore, makes it possible to have an effluent offering both a risk-free discharge into the sewer network [49] and above all the possibility of recovering the treated wastewater as washing, watering green spaces, and for agriculture irrigation [50] It was also observed that it is unfortunately not possible to be selected according to the potential interest of the ions. Indeed, the retention of sodium and chlorides (undesirable) are of the same order of magnitude as those of salts of agronomic interest (N, P, K, Mg).

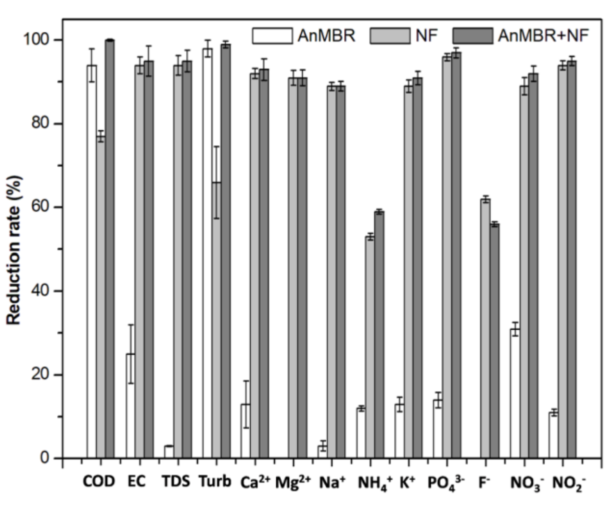

Fig. 9. Purification performance of AnMBR and NF coupling.

3.5 Biogas production

Lower volumes of biogas were recorded at the start of the study, which confirms the implementation of the treatment and therefore of the methanogenic microorganisms. In a stabilized regime, i.e. after 60 days of operation, the corresponding biogas production yields varied between 0.18 and 0.27 L biogas/gCOD eliminated throughout the study with an average yield of 0.21 ± 0.03 L biogas/gCOD eliminated. This yield remains slightly lower than the theoretical yield of 0.5 L CH4/gCOD eliminated [51,52]. It is also lower than that obtained by [43] (0.53 ± 0.015 L biogas/gCOD eliminated). Despite malfunctions when the reactor was started up, the actual wastewater used showed a potential for biogas production. Fig. 10 shows the volumes of biogas measured over time and the corresponding production yields.

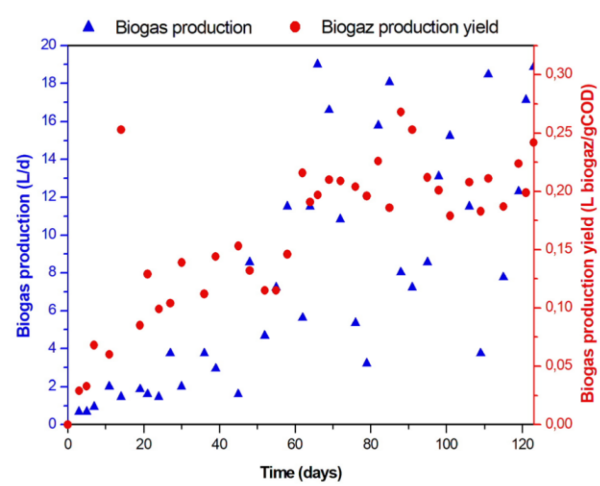

Fig. 10. Biogas production during wastewater treatment in the beverage industry.

Conclusions

The implementation of the membrane bioreactor under anaerobic conditions for the treatment of wastewater from the brewery industry has led to remarkable performance, making this technology suitable for the treatment of industrial sewage with large variations in organic load. The organic matter removal yields reached 94% after 60 days. Thus, the start-up from a local inoculum was fairly rapid and achieved high organic matter removal yields in a relatively short time. Post-treatment with nanofiltration made it possible to have a final effluent of better quality with a significant increase in most of the parameters. The volumes of biogas measured reached 19 L/d leading to an average production yield estimated at 0.21 ± 0.03 L biogas/gCOD eliminated. The treated wastewater has shown a biogas production potential and confirms the advantage of anaerobic digestion of agro-food industry effluents in this climatic environment with characteristics favorable to this type of treatment. The use of NF resulted in a final effluent with COD and sodium contents allowing reuse for irrigation and safe discharge into the sewer system and the environment. Unfortunately, the sodium rejection rate is of the same order of magnitude as that of salts of agronomic interest (N, P, K, Mg).

Acknowledgments

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