Excellent performance of anaerobic membrane bioreactor in treatment of distillery wastewater at pilot scale

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1 **Excellent performance of anaerobic membrane bioreactor in treatment of**
2 **distillery wastewater at pilot scale**

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Abstract

A pilot-scale anaerobic membrane bioreactor (AnMBR) was designed and optimized for the treatment of real distillery wastewater. A low hydraulic retention time of 3.5 days was reached after only 3 weeks. The AnMBR could treat up to 3.97 g_{COD}/L/day with high biogas production at 1.36 NL_{biogas}/L_{bioreactor}/day. The performances of an AnMBR and an anaerobic packed-bed bioreactor for treating the same wastewater were compared. The AnMBR had a shorter start-up period (21 days), a higher COD removal efficiency (96.9%), and higher stability and methane production (0.26 L_{CH₄}/g_{CODinput}), indicating the interest of investigating AnMBR industrialization. The membrane performance was also studied, demonstrating a long cleaning cycle interval of at least 44 days. The transmembrane pressure and Food-to-Microorganism ratio were defined to minimize membrane fouling without affecting the anaerobic digestion performance.

Keywords

Membrane bioreactor; Wastewater treatment; Anaerobic digestion; Packed-bed bioreactor; Biogas production

Abbreviations

AnMBR: Anaerobic membrane bioreactor

APBR: Anaerobic packed-bed reactor

COD: Chemical oxygen demand (g/L)

CSTR: Continuously stirred tank reactor

EPS: Extracellular polymeric substances

F/M: Food to microorganism ratio (g_{COD}/g_{MLVSS}/day)

FOS/TAC: Volatile fatty acids content / Buffer capacity

HRT: Hydraulic retention time (day)

MLSS: Mixed liquor suspended solids (g/L)

MLVSS: Mixed liquor volatile suspended solids (g/L)

NPOC: Non-purgeable organic carbon

OLR: Organic loading rate (g_{COD}/L/day)

39	SMP: Soluble microbial particles
40	SRT: Sludge retention time (day)
41	TMP: Transmembrane pressure (bar _g)
42	VFA: Volatile fatty acids (mg/L)

1 Introduction

The interest in and need for renewable energy are increasing with global warming. Wastewater treatment processes can become net producers of renewable energy by converting the organic pollutants of wastewater to biogas via anaerobic digestion [1]. However, researching and selecting appropriate technology to treat wastewater and recover energy remain major challenges. Different types of bioreactors have already been used on an industrial scale for anaerobic digestion for wastewater treatment. The reactor design must consider the slow growth rate of microorganisms involved in anaerobic digestion. This must exceed the dilution rate to prevent biomass washout [2].

The most commonly used bioreactor type was the continuous stirred tank reactor (CSTR) until the 1960s [3]. However, the main drawback of this type of bioprocess relates to combining the sludge retention time (SRT) and hydraulic retention time (HRT). Thus, the retention time is limited by the growth rate of microorganisms. The HRT is usually 20–40 days, leading to an important bioreactor volume [2]. Few technologies have been developed to decouple the SRT and HRT [3]. The main biomass retention technologies used on an industrial scale can be separated into two classes: biofilm-based bioreactors, such as the anaerobic packed-bed reactor (APBR), and self-immobilized microorganism bioreactors, such as the upflow anaerobic sludge blanket (UASB) [2,3].

In packed-bed technology, the reactor is filled with inert and stationary material whose characteristics (e.g., size, shape, porosity, specific surface area) must be considered during bioreactor design. In the APBR, microorganisms are fixed on the surface of packing material, allowing a high SRT compared to the HRT. Sludge particles are also trapped in the interstices of the packing material, increasing the biomass retention [3].

The main advantages of this type of reactor are its simplicity of construction and easy operation and start-up. This technology also has low construction and operating costs, and no agitation is required [2], while a recirculation loop can sometimes be used. However, due to possible clogging of the fixed bed, this technology cannot be used to treat wastewater with a high concentration of suspended solids [3]. This technology can be used with a relatively high loading rate of up to 10 g_{COD}/L/day [3].

New technologies based on membrane filtration for biomass retention are currently being studied for anaerobic digestion. The membrane bioreactor is an attractive technology that couples anaerobic wastewater treatment with membrane filtration. The anaerobic membrane bioreactor (AnMBR) exists in three different configurations: external cross-flow, internal submerged, and external submerged membrane. In the external cross-flow configuration, the membrane is separated from the reactor. Bioreactor sludge is pumped through the membrane, creating a positive pressure leading to permeate production, and the retentate returns to the bioreactor [2].

The main advantages of the AnMBR are its high treatment capacity, up to 30 g_{COD}/L/day, and high effluent quality without suspended solids [2]. This high treatment capacity can be reached due to the high biomass concentrations and low HRT obtained with this technology. Membrane filtration allows complete decoupling of the HRT and SRT. Moreover, the permeate can be reused for some purposes without additional treatment [4]. The AnMBR has been shown to have high biological stability and carbon removal efficiency [5,6].

However, membrane bioreactors are not usually used on an industrial scale for anaerobic treatment. Control of membrane fouling by liquid/gas recirculation and chemicals makes

this process more complex to design than other bioreactor types and leads to higher operating costs [2]. Membrane fouling in the bioreactor is usually caused by organic matter attached to the membrane surface or trapped in the membrane pores. This organic matter is composed of microorganisms, soluble microbial particles (SMP), and extracellular polymeric substances (EPS). Generally, SMP and EPS are proteins or hydrocarbon molecules secreted by microorganisms during growth and methane production. Meng et al. [7] showed that SMP and EPS are the main foulants in AnMBR. The concentration, composition, and size of SMP and EPS were impacted by controlling parameters such as the organic loading rate (OLR, expressed as $\text{g}_{\text{COD}}/\text{L}/\text{day}$), SRT, and food-to-microorganism ratio (F/M, expressed as $\text{g}_{\text{COD}}/\text{g}_{\text{MLVSS}}/\text{day}$) [8,9,10]. Controlling the SRT for a given OLR allows control of the biomass concentration in the bioreactor. Thus, controlling the SRT enables control of the F/M ratio. The amount of SMP and EPS produced by microorganisms depends on the F/M ratio.

This study investigates the start-up phase and overall performance of an AnMBR treating distillery wastewater at pilot-scale. The anaerobic digestion performance of AnMBR technology was studied together with an APBR process treating the same wastewater. APBR technology was used as a reference for this wastewater, as this technology is currently used for industrial-scale treatment in the distillery. Membrane fouling was also investigated.

2 Materials and methods

2.1 AnMBR

2.1.1 Plant description

Fig. 1 shows the outline of the pilot-scale AnMBR. The total reactor volume was 170 L with a working volume of 150 L and an internal diameter of 0.4 m. The reactor was connected to an external membrane module with a liquid recirculation pump (IWAKI MDT15). Two membranes were tested in this study. The first was a spiral-wound membrane made of hydrophilic polysulfone, with a surface area of 0.5 m² and pore size of 10 kD (Alfa Laval). The second membrane was a multichannel tube made of ceramic with 19 channels. The surface area was 0.25 m², pore size 0.1 µm (Orelis Environnement – KLEANSEP™ BW), and channel diameter 3.5 mm. The reactor was equipped with a pH probe and temperature sensor (JUMO 201020) in a second external recirculation loop with a sampling port.

Figure 1: Schematic of the pilot-scale anaerobic membrane bioreactor used in this study.

The bioreactor was fed continuously with substrate in the reserve, which was kept cold by recirculation through the cold storage (4 °C) with a peristaltic pump (Watson Marlow Series 300). Overflow substrate was returned to the cold storage by gravity. The substrate

was kept cold to prevent degradation. The permeate flow rate was controlled with a peristaltic pump (MasterFlex – 7518-10) on the permeate side of the membrane, which controlled the feed flow rate by natural level adjustment. No sludge was discarded during the experiment except for sampling (SRT > 100 days).

Bioreactor mixing was ensured by continuous gas and liquid recirculation. The liquid recirculation flow rate in the membrane was 700 L/h. The liquid recirculation flow rate for the pH probe (JUMO 201020) and sludge sampling was 600 L/h. Gas recirculation occurred by cycles of 10 minutes' recirculation at 480 NL/h and 10 minutes without recirculation. The temperature was set at 37 °C with a water bath and coil in the bioreactor. No pH adjustment was performed in the bioreactor.

2.1.2 Analytical procedure

The following parameters were recorded regularly during all operations: FOS/TAC was recorded daily by pH titration using 0.1 N H₂SO₄, performed according to the Nordmann method [11]. The buffer capacity (TAC) of the system was determined through titration of 20 mL of sample up to pH 5. The volatile organic acids (FOS) were then obtained after a second titration step between pH 5.0 and pH 4.4. The biogas composition was recorded daily using a Micro-GC 490 instrument from Agilent Technologies. The sample was introduced through a heated line at 100 °C. Two columns were used: a molecular sieve 5 Å (MS-5A, 10 m) to separate O₂, N₂, and CH₄ and a PoraPLOT U (PPU, 10 m) to quantify CO₂. Separation was achieved at 100 °C and 60 °C with backflushes of 4.7 and 16 s, respectively. Columns pressure was 29 psi. The carrier gas was helium, and the detector was a thermal conductivity detector (TCD). The total run took 80 s. Calibration was performed with standard gas from Air Liquide, diluted using flowmeters from

Bronkhorst. Quantification was achieved using the peak areas in external calibration; the concentrations ranged from 0.05% to 100%. The permeate weight was recorded daily with a scale (OHAUS-type DEFENDER 5000). The total volume of biogas was recorded daily using a RITTER drum-type gas meter (TG 0.5). Volatile fatty acids (VFA) were recorded 3 times a week by HPLC (Ultimate 3000 Thermo Fisher) equipped with an Aminex HPX-87H (7.8×300 mm, $9 \mu\text{m}$) column at 50°C with UV detection. The mobile phase was 8 mM H_2SO_4 with a flow rate of 0.8 mL/min, and 20 μL of sample was injected for analysis. Acetic, butyric, and propionic acids were quantified. The chemical oxygen demand (COD) was recorded 3 times a week using Spectroquant® cell tests (1.14541) with a Spectroquant® Multy photometer. Mixed liquor suspended solids (MLSS) and mixed liquor volatile suspended solids (MLVSS) were recorded weekly. Two centrifugations of 50 mL of sample (4,500 g, 15 min) were performed, followed by sequential drying at 105°C and 550°C .

2.1.3 Membrane fouling and cleaning

The membrane flux was measured regularly for the ceramic membrane by measuring the flow rate of permeate produced from wastewater after disconnecting the peristaltic pump. Thus, the pressure at the permeate side was assumed to be atmospheric pressure. The pressure at the retentate side was measured using a probe (Brukert 8316).

The membrane was cleaned whenever the flux decreased below 12 L/h/m^2 . First, it was ensured that no clogging occurred in the membrane channels. Chemical cleaning was then conducted using NaOH solution (10 g/L) for 30 minutes at 70°C and rinsing with distilled water. Afterward, HNO_3 solution (5 g/L) was used, and the membrane was submerged

for 20 minutes at 50 °C. The membrane was rinsed with water before being reused in the bioreactor.

2.2 APBR

2.2.1 Plant description

Fig. 2 shows the outline of the APBR. The reactor used was a CSTR-10S model provided by Bioprocess Control and adapted for APBR use. The total reactor volume was 12 L with a working volume of 10.6 L and an internal diameter of 0.2 m.

Figure 2: Schematic of the anaerobic packed-bed reactor used in this study.

The stirrer was removed, and the working volume of the reactor was filled with disordered plastic hollow cylinders with ribs (Flocors™). These had an average height of 30 mm, an internal diameter of 30 mm, and an external diameter of 35 mm. They had a porosity of 95% and a specific area of 230 m²/m³. The reactor was filled with 0.55 L of Flocors™ corresponding to a surface area of 0.127 m² available for the microorganisms. Flocors™ was kept in the bioreactor by using a pipeline of lower diameter than the Flocors™ size. The reactor was equipped with a pH probe, a redox probe, and a temperature sensor.

Substrate feeding, stored at 4 °C, and digestate discharge were performed with a peristaltic pump (Gilson Minipuls Evolution®). Bioreactor mixing was ensured with an external loop with another peristaltic pump (Gilson Minipuls Evolution®) at a flow rate of 0.7 L/h. The temperature was set at 37 °C with a water bath and the bioreactor wall jacket.

2.2.2 Analytical procedure

During all operations, the consumed substrate weights and extracted digestate were recorded daily. The total biogas volume was measured daily using a volumetric gas flowmeter (μFlow, Bioprocess Control). The FOS/TAC was recorded 3 times a week and determined using a TitraLab AT1000 Series titrator from Hach. The non-purgeable organic carbon (NPOC) was recorded 3 times a week using a total organic carbon analyzer, TOC-L CSH/CSN, from Shimadzu after filtration of the sample with a 0.2 μm filter. For each batch of wastewater, the COD and NPOC were determined to correlate both values to compare the AnMBR and APBR. The COD and NPOC are proportional for a given wastewater type [12]. However, the carbon content in the APBR effluent could be underestimated while samples were filtered before NPOC analysis. The low suspended solids with the APBR suggest that underestimation was limited. The biogas composition was recorded 3 times a week using a GEMBIO biogas analyzer (Gruter & Marchand). The concentrations of CH₄ and CO₂ in the biogas were measured with an IR projector (2 Hz pulsations).

2.3 Sludge inoculum

The AnMBR was initially inoculated with sludge obtained from the 10-L APBR. It was composed of 0.38 g/L total suspended solids and 0.26 g/L volatile suspended solids.

The reactor was filled with 300 L inoculum by continuously filtering 150 L of inoculum through the membrane to increase the biomass in the bioreactor, as digestate from the APBR had low volatile suspended solids. Thus, the initial suspended solids in the pilot bioreactor were doubled compared to the sludge.

No inoculum was used for the APBR. Consortia were developed from microorganisms found in the wastewater used as substrate.

2.4 Experimental setup

During all experiments, the feed flow rate was regulated by following the recommendations in Table 1, according to FOS/TAC values for the AnMBR and APBR. The aim was to achieve optimal biogas production by balancing organic acid production (FOS) with the buffer capacity of the system (TAC).

FOS/TAC ratio	Interpretation of FOS/TAC ratio	Feed flow rate control
>0.6	Highly excessive feed flow rate	Stop feeding
0.5–0.6	Excessive feed flow rate	Reduce feed flow rate by 10%
0.4–0.5	Plant is heavily loaded	Monitor the plant more closely
0.3–0.4	Biogas production at a maximum	Keep feed flow rate constant
0.2–0.3	Feed flow rate is too low	Increase feed flow rate by 10%
<0.2	Feed flow rate is far too low	Increase feed flow rate by 20%

Table 1: Followed recommendations for feed flow rate during start-up period

For the AnMBR, the experiment was conducted in three phases. The first was the study of the start-up period. During the second phase, the substrate composition was changed to evaluate the resilience performance of the AnMBR process instability. The third phase was the process performance evaluation of the AnMBR running at a stable stage with a high OLR.

The 10-L APBR ran for a few months at a stable stage with a performance similar to the industrial APBR used by the distillery (Cristanol, France) and was used as a model for evaluating the AnMBR performance.

2.5 Wastewater

The substrate used as nutrient feedstock was wastewater from the industrial distillery (Cristanol, France). It was a blend of evaporation condensate of the distillery from sugar beet and wheat ethanol production and cleaning water from the different distillery tanks. Suspended solids represented 3 ± 1 g/L. A new batch of fresh wastewater was collected every 2 to 3 weeks. Variability was observed between each batch. Especially, higher COD was observed during sugar beet campaign periods. The pH was adjusted to 7 ± 0.2 with potassium hydroxide before feeding. During the first phase, the bioreactor was fed with wastewater only (average COD: 6.9 ± 1.4 g/L). During Phase 2, saccharose was added to the wastewater to evaluate the resilience performance of the AnMBR to instability. Saccharose represented half the COD during this phase (average COD: 20.1 ± 4.1 g/L). Urea was added to the effluent with saccharose to maintain a constant C/N ratio, which was different for every wastewater batch. The third phase was performed during sugar beet campaign periods when wastewater had a high COD content (average COD: 12.2 ± 0.8 g/L); no saccharose was added in Phase 3.

2.6 Calculations

2.6.1 FOS/TAC

The FOS/TAC ratio was used to evaluate the biological stability of the process. The formula used for the FOS/TAC calculation was based on the Nordmann method [11], which empirically estimates FOS and TAC. The FOS value was expressed in mg/L of

acetic acid, and the TAC value was expressed in mg/L of calcium carbonate. Both values were estimated by titration of 20 mL sample with 0.1 N H₂SO₄.

$$FOS = ((B \times 1.66) - 0.15) \times 500 \quad TAC = A \times 250$$

With A: Titration volume at pH 5 (mL) and B: Titration volume from pH 5 to pH 4.4 (mL).

2.6.2 Food-to-microorganism ratio

The MLVSS was measured weekly. This was used as the microorganism concentration for calculating the F/M ratio. The MLVSS was shown to increase exponentially during Phase 1; thus, the MLVSS concentration was estimated for each day using the variation rate estimated for each week and the last MLVSS measurement.

$$Variation\ rate = \frac{Ln(MLVSS\ at\ day\ n) - Ln(MLVSS\ at\ day\ n + 7)}{7}$$

$$MLVSS\ at\ day\ n + i = Exp(Ln((MLVSS\ at\ day\ n) + i \times Variation\ rate))$$

The F/M ratio was calculated by dividing the OLR, expressed as g_{COD}/L/day, and the microorganism concentration, expressed as g_{MLVSS}/L_{reactor}. The OLR was calculated from the feed flow rate and feed COD concentration. The average OLR of the last 2 days was used for the F/M ratio calculation.

The F/M ratio is a crucial parameter that significantly affects the process performance and membrane fouling in the AnMBR system. When this ratio is high, a high biomass yield is obtained, causing a low SRT, contributing to high sludge production [9]. It is also a source of biomass deflocculation and higher SMP and EPS production, which both increase membrane fouling [9]. Liu et al. [9] showed that a high F/M ratio of 3.6

g_{COD}/g_{MLVSS}/day led to higher membrane fouling than 0.1 g_{COD}/g_{MLVSS}/day, even if the MLVSS was higher when a low F/M ratio was applied. Membrane fouling is mainly explained by SMP and EPS concentrations in the AnMBR [7,9]. A high F/M ratio can also affect the balance between acidogenesis and methanogenesis, affecting the digestion performance. One of the main causes of anaerobic digestion failure is acidification due to overloading. The bacterial community of the acidogenesis step has a higher growth rate than the archaeal community of the methanogenesis step. Thus, a high F/M ratio risks VFA accumulation.

Thus, a low F/M ratio is preferred to stabilize the process and achieve a higher removal efficiency. However, if the F/M ratio is low, a higher MLVSS is required for the same OLR, increasing membrane fouling and the start-up time to reach the desired MLVSS.

2.6.3 Membrane flux

The membrane flux was measured in liters per hour per square meter (L/h/m²). The permeate pressure was assumed to be atmospheric pressure. The membrane flux was calculated as follows:

$$\text{Membrane flux} = \text{Measured flowrate (L/h)} / \text{Membrane surface area (m}^2\text{)}$$

3 Results and discussion

3.1 AnMBR process performance

290

291 **Figure 3: Evolution of the OLR and biogas production of the AnMBR. A: Phase 1:**
 292 **Start-up phase; B: Phase 2: Resilience trial by adding saccharose to the wastewater;**
 293 **C: Phase 3: Wastewater during sugar beet campaign.**

3.1.1 Phase 1: Start-up phase

Fig. 3A shows the OLR ($\text{g}_{\text{COD}}/\text{L}/\text{day}$) applied to the AnMBR and the biogas production ($\text{L}_{\text{biogas}}/\text{L}_{\text{bioreactor}}/\text{day}$) during the first phase when the spiral-wound membrane was used. The experiment started with an OLR fixed at $0.40 \text{ g}_{\text{COD}}/\text{L}/\text{day}$. The OLR increased rapidly according to the FOS/TAC value. After 21 days, the process had been stabilized at a short HRT reaching 3.5 days, corresponding to an OLR of $2.48 \text{ g}_{\text{COD}}/\text{L}/\text{day}$, with low volatile fatty acids (VFA) in the permeate ($<50 \text{ mg/L}$).

This result indicates that the AnMBR approach induces a short start-up period limited to 21 days to reach a low HRT, while the start-up phase can take several months in a conventional anaerobic digester. Moreover, other studies showed that a short HRT can be reached in less than 2 weeks in an AnMBR [6]. The ability of the AnMBR to have complete control of the SRT is the main reason for both anaerobic digestion performance improvements.

3.1.2 Phase 2: Resilience trial

During the second phase, the resilience of the AnMBR was investigated by adding saccharose to the wastewater. Phase 2 was started with a similar OLR ($2.30 \text{ g}_{\text{COD}}/\text{L}/\text{day}$) to Phase 1 ($2.48 \pm 0.09 \text{ g}_{\text{COD}}/\text{L}/\text{day}$), which was increased according to the FOS/TAC value. The first batch of wastewater in this phase had a COD concentration of 8.02 g/L , and saccharose was added to double the COD concentration. Fig. 3B shows the OLR applied to the AnMBR and the biogas production during this phase. Until day 9, the FOS/TAC ratio remained low (0.19 ± 0.02) despite the change in organic carbon in the wastewater ($\text{FOS/TAC} = 0.30 \pm 0.02$ in Phase 1). It was feasible to treat up to $3.37 \pm 0.16 \text{ g}_{\text{COD}}/\text{L}/\text{day}$ with high removal efficiency ($98.5\% \pm 0.3\%$).

On day 9, a new batch was used with a higher COD concentration (12.11 g/L), and saccharose was added to reach a COD concentration of 24.22 g/L. Easily fermentable sugar led to a rapid pH decrease in the cold storage, to pH 5 before feeding, inducing bioreactor acidification to pH 6.6. The pH decrease arose from the VFA accumulation, which reached 920 mg/L. This led to a FOS/TAC increase to 1.06 due to decreased methanogenic activity compared to the acetogenesis activity. The bacterial community of the acetogenesis step had a faster growth rate and activity than the archaeal community of the methanogenesis step. Thus, saccharose was quickly converted into acetic acid, which accumulated. Moreover, the archaeal community growth and activity were inhibited when the pH decreased to 6.6, further increasing the VFA accumulation. Thus, the feed pH was corrected, and the feed flow rate was decreased while the VFA concentration remained high (1,311 mg/L) 2 days later. The pH correction of the substrate was insufficient for VFA degradation. Feeding was then stopped for 2 days and slowly restarted. Four days after stopping the feed, no VFA was detected in the permeate (<50 mg/L), and an OLR similar to that before acidification was applied (3.20 g_{COD}/L/day). This result confirms that the AnMBR is highly resilient toward organic carbon variations since the FOS/TAC ratio remained low after adding saccharose for a similar OLR before day 9. Moreover, after the pH shock and VFA accumulation, recovery of the process stability and consumption of VFA was achieved within 4 days by reducing feeding. Thus, the AnMBR appears to be a promising technology to prevent biomass losses during uncontrolled acidification events due to its ability to rapidly resume control. Other literature results showed the high resilience of the AnMBR technology [5,13]. Basset et al. [5] investigated the AnMBR performance under a fluctuating COD input. They showed that increasing the specific OLR to 0.47 g_{COD}/g_{MLVSS}/day led to a decrease

in COD removal from 95.7% to 63.6%. However, stable operation was recovered immediately by decreasing the specific OLR to 0.30 g_{COD}/g_{MLVSS}/day. In CSTR technology, if acidification occurs, biomass growth is stopped and lost, and a longer period is required to recover a proper balance.

3.1.3 Phase 3: Stable stage during sugar beet campaign

During the last period, the bioreactor was supplied with effluent without adding saccharose. Moreover, the bioreactor was equipped with a ceramic membrane. Fig. 3C shows the results for the OLR and biogas production in Phase 3.

After 10 days of operation, fouling caused a decrease in membrane flux. Consequently, an OLR decrease was measured. After membrane cleaning, the feed flow rate could be increased again according to the FOS/TAC value to increase the OLR. After a month, the process reached a stable stage at an HRT of 2.9 days and an OLR of 3.97 g_{COD}/L/day with high COD removal (95.4%) and high digestion stability (FOS/TAC = 0.25).

The AnMBR represents a physical barrier, inducing COD particle accumulation inside the bioreactor and releasing a permeate with a low COD content. The COD value in the AnMBR effluent was 0.53 g/L in Phase 3, while the total COD value was 30-fold higher inside the bioreactor. The high removal efficiency of the AnMBR was also observed in other studies [17,18,19]. In a study by Ozgun et al. [18], the total COD values reached approximately 1 to 2 g/L in the bioreactor, while the permeate COD remained low (0.10 g/L).

However, the OLR reached in our process remained two to three fold lower compared to some studies using an AnMBR. Mnif et al. [14] reached an OLR of 8.0 g_{COD}/L/day without affecting the reactor performance. Hu et al. [16] reached an OLR of 12.6

g_{COD}/L/day with high COD removal efficiency (91.8%). This difference in the OLR can be explained by a higher COD concentration in the wastewater used in their study (up to 22 g_{COD}/L) and a higher surface membrane area: 4.6 m²/m³, 2.7 times larger than in our study. A higher membrane surface area leads to a higher flux and thus a higher feed flow rate.

3.1.4 MLVSS evolution in AnMBR

Fig. 4 shows the evolution of MLVSS. The MLVSS is usually used to estimate the microorganism concentration [15]. The exponential variation in the MLVSS in Phase 1 suggests exponential growth of microorganisms. In this phase, the average variation rate of MLVSS was $0.031 \pm 0.005 \text{ day}^{-1}$. The variation rate of MLVSS at the beginning of Phase 3 was lower: $0.009 \pm 0.004 \text{ day}^{-1}$. At the end of the experiment, the MLVSS was stabilized at 5.6 g/L. These results suggest that no apparent microorganism growth occurred (stationary phase of growth). The difference in the MLVSS variation rate might explain the slightly lower methane yield in Phase 1 ($0.24 \text{ L}_{\text{CH}_4}/\text{g}_{\text{CODinput}}$) compared to Phase 3 ($0.26 \text{ L}_{\text{CH}_4}/\text{g}_{\text{CODinput}}$), as a higher proportion of the COD input was used for microorganism growth in Phase 1, leading to higher accumulation of MLVSS.

Figure 4: Evolution of MLVSS in AnMBR over time to evaluate the biomass growth.
Phase 1: Start-up phase; Phase 2: Resilience trial by adding saccharose to the wastewater; Phase 3: Wastewater during sugar beet campaign.

3.2 Performance comparison

Table 2 shows the AnMBR performance reached in recent studies using this technology to treat real industrial wastewater. It also shows the performance at the stable stage reached in Phase 3 of our study compared with the performance of the APBR treating the same batch of wastewater.

3.2.1 Performance comparison with recent studies using AnMBR

Other recent studies (2020–2021) used AnMBR technology to treat real industrial wastewater. Table 2 shows their performances [20,21,22,23]. The performance achieved in our study based on the OLR is higher than in most studies shown. Only one study [22] showed a higher OLR (7.9 g_{COD}/L/day). However, the OLR mainly depends on the wastewater COD concentration and biodegradability. The relatively high COD

concentration of the distillery wastewater may explain the higher OLR. The higher OLR reached in our study led to higher biogas production (1.36 NL/L/day) than in most other studies. Only the study with a higher OLR showed higher biogas production (2.42 NL/L/day) [22].

The HRT reached in our experiment (2.9 days) is similar to that in other studies (1.5 to 5 days) despite the lower membrane surface area used ($1.7 \text{ m}^2/\text{m}^3$), showing the advantage of the external tubular membrane, which has a higher flux in our study. However, this requires more energy for fouling control than for submerged membranes.

Moreover, the other studies confirm a high COD removal efficiency, which can be reached with the AnMBR (92% and 99%) [20,22]. Only one study [21] reported a lower COD removal efficiency (82%), explained by the complex structure and lower biodegradability of organic compounds in real textile wastewater. Additionally, other studies [20,22] report a high methane production of at least $0.21 \text{ NL CH}_4/\text{gCOD}_{\text{input}}$.

3.2.2 Performance comparison of AnMBR and APBR

3.2.2.1 Start-up phase

In the APBR, the start-up period lasted 121 days to reach an HRT of 3 days, while only 21 days were required for the AnMBR. This result suggests a shorter start-up phase in the AnMBR compared to the APBR. However, no inoculum was used in the APBR experiments, while the AnMBR was inoculated with a culture medium obtained from the APBR. Moreover, the short start-up period of the AnMBR may be due to the inoculum used, which was already adapted to the wastewater used in this study.

3.2.2.2 Stable stage

Table 2 shows the performance of the AnMBR at the stable stage reached in Phase 3 compared with the performance of the APBR when treating the same batch of wastewater.

These results reveal the main differences between the AnMBR and APBR. A higher specific biogas production was obtained in the AnMBR, $0.26 \text{ L}_{\text{CH}_4}/\text{gCOD}_{\text{input}}$, while $0.15 \text{ L}_{\text{CH}_4}/\text{gCOD}_{\text{input}}$ was obtained in the APBR for the same wastewater batch. Furthermore, the COD removal efficiency was higher in the AnMBR than in the APBR, and the VFA (eq CH_3COOH) content was lower in the AnMBR. Moreover, the lower FOS/TAC ratio provided higher stability in the AnMBR. Despite the scale difference of the two bioreactors, the performance differences can mainly be attributed to the technology used since the 10-L APBR is a model of the industrial-scale anaerobic bioreactor (Cristanol, France).

The high COD removal efficiency of the AnMBR resulted from a higher retention time of suspended COD particles, which are trapped in the bioreactor and can thus be consumed by microorganisms. Inside the AnMBR, suspended solid represented 77% of the total COD, which is consistent with the scientific literature [8,18,24]. These suspended particles were not retained in the APBR. This led to a greater methane yield for the AnMBR ($0.26 \text{ L}_{\text{CH}_4}/\text{gCOD}_{\text{input}}$) than that obtained in the APBR ($0.15 \text{ L}_{\text{CH}_4}/\text{gCOD}_{\text{input}}$). Thus, compared to the APBR process, the AnMBR process resulted in a 1.7-fold greater methane yield and biogas production. This result shows better use of the COD in the AnMBR compared to the APBR. This difference is not reflected in the COD removal efficiency since the APBR effluent COD measurement did not consider suspended solids.

The higher soluble COD removal efficiency observed in the AnMBR (95.4%) compared to the APBR (93.0%) can be explained by the better hydraulic conditions in the AnMBR compared to the APBR. A high biomass concentration with free cells in the AnMBR led to better contact between microorganisms and the COD. Conversely, the APBR struggles to maintain intimate and prolonged contact between the microorganisms and COD. Moreover, clogging can occur [3]. Effluents of the anaerobic wastewater treatment plant still require further aerobic treatment to reach the required quality for disposal in a river or spreading in the environment. With a higher COD removal in the AnMBR, less energy will be required for the aerobic treatment.

3.3 Membrane fouling

Although the AnMBR showed much promise for wastewater treatment and biogas production, membrane fouling remains a major limiting factor regarding industrial applications. In this study, the ceramic membrane lifespan was investigated to estimate the cleaning frequency and flux recovery.

Figure 5: Evolution of the ceramic membrane flux in AnMBR to evaluate the filtration performance of the membrane through the experiment. The dotted lines represent membrane cleaning days.

Fig. 5 shows the membrane flux throughout the AnMBR experimental operation using a ceramic membrane. The initial membrane flux was 25 L/h/m². This value quickly decreased to 19 L/h/m² and remained between 15 and 20 L/h/m² for more than 30 days. Beyond this, a faster membrane flux decrease was observed from 15 to 7.7 L/h/m² in less than 10 days, probably caused by cake formation at the membrane surface and clogged pores.

First, membrane chemical cleaning was performed using cold acid and base solutions after 44 days of operation at low transmembrane pressure (TMP), under 0.15 bar_g. This led to flux recovery up to 18 L/h/m² (72% recovery compared to the new membrane). However, after only 4 days, the flux decreased to 12 L/h/m², showing that a cold solution was inefficient for membrane cleaning. A second cleaning strategy was applied. The ceramic membrane was cleaned using base and acid solutions at 70 °C and 50 °C, respectively, leading to 60% recovery compared to the new membrane. The membrane could then be used for 20 days at low TMP (under 0.15 bar_g). However, on day 70, the flux dropped to 6 L/h/m². Thus, the TMP was increased. It was still possible to use the membrane for 36 days at 0.30 bar_g. In total, the ceramic membrane was used for almost 2 months without cleaning. The flux after cleaning was 18 L/h/m², showing a high recovery of the flux. This showed that increasing the pressure did not cause irreversible fouling, suggesting that the pressure can be increased to maintain the flux for months. However, maintaining a moderate TMP for as long as possible is recommended to lower pore clogging.

A proper membrane cleaning procedure and bioreactor monitoring allowed the AnMBR to be run for almost 2 months (56 days) without cleaning. However, this performance was only possible with a high recirculation flow rate applied to the membrane (700 L/h). The

reduction of the recirculation rate to 300 L/h led to a 70% flux decrease within 2 days, leading to inability to maintain the desired HRT. This tremendous flux decrease was expected since this lower recirculation rate corresponded to a liquid speed of 0.5 m/s, which is deemed extremely low for an ultrafiltration membrane. A liquid speed of at least 2 m/s is recommended for an ultrafiltration membrane [25]. Nevertheless, this high recirculation rate would lead to high operation costs on an industrial scale for anaerobic treatment.

In our study, no sludge discharge was applied during 8 months of operation. After the two cleaning cycle intervals described previously, the membrane had been used for 4 months without cleaning (data not shown). During this period, the membrane flux decreased progressively to 10 L/h/m² with a TMP enhanced to 1 bar. Moreover, channel clogging occurred. The soluble COD concentration inside the bioreactor was eightfold higher than the COD of the permeate. This showed the accumulation of organic content in the bioreactor after 8 months of operation without sludge discharge. This organic matter probably comprised SMP and EPS, which are known to cause membrane fouling [7].

Some studies have shown that a long SRT led to serious membrane fouling for long-term use [26]. A high SRT led to accumulation of organic content and increased the SMP production by microorganisms [10]. This suggests faster membrane fouling because of biomass attached to the membrane surface or trapped in pores. Huang et al. [27] almost doubled their cleaning interval by decreasing the SRT from 90 to 60 days; membrane cleaning intervals were changed from 25 to 40 days.

Due to the energy needed for the recirculation loop in the external configuration, it is necessary to investigate other filtration processes, such as submerged membranes, as they

are considered more economically feasible on an industrial scale. Moreover, it is necessary to investigate the optimal SRT to prevent membrane fouling.

3.4 F/M ratio

In our study, the OLR was applied based on the FOS/TAC ratio. The MLVSS increase was observed over time, showing the microorganism growth. Thus, the OLR was also increased because the higher the microorganism concentration, the higher the COD that can be treated. The OLR and MLVSS values were used to follow the evolution of the F/M ratio over time.

During the two stable stages in Phases 1 and 3, two different F/M ratios were observed, respectively: 1.05 and 0.74 gCOD/gMLVSS/day. In Phase 2, the F/M ratio was 0.96 gCOD/gMLVSS/day. In the first phase, anaerobic digestion performances were similar despite the higher F/M ratio.

At the end of the experiment, the MLVSS was stabilized at 5.6 g/L, showing a halt in microorganism growth, indicating that the F/M ratio of 0.7 gCOD/gMLVSS/day was too low to support the biomass growth. Mnif et al. [14] observed the same stabilization of the biomass concentration at 2.5 g/L of volatile suspended solids under their conditions with a high F/M ratio (2.5 gCOD/gVSS/day).

The F/M ratio decrease over time for the same bioprocess stabilization suggests that the biomass activity with a long SRT (>100 days) tends to decrease, probably because of accumulation of dead microorganisms or suspended organic matter that did not contain microorganisms. This shows that applying a shorter SRT is necessary for process performance and membrane fouling. Working with an F/M ratio as high as 1.05 gCOD/gMLVSS/day in Phase 1 did not affect the process performance. Thus, the SRT must be controlled to maintain this ratio around 1 gCOD/gMLVSS/day for a constant OLR. A ratio

of 1.05 gCOD/gMLVSS/day was relatively high compared to other studies. Basset et al. [5] reported an F/M of 0.30 gCOD/gMLVSS/day and Kunacheva et al. [28] used an F/M of 0.5 gCOD/gMLVSS/day. Our results showed a relatively high biological activity of the consortium compared to other studies. However, the ratio in our study was determined during the start-up phase. Further investigations with a stabilized F/M ratio during multiple cleaning cycle intervals are required to confirm this ratio, which depends on the OLR, HRT, and membrane characteristics and surface.

4 Conclusion

The results obtained in this study confirm that the membrane bioreactor is a promising technology for wastewater treatment by anaerobic digestion. Excellent digestion performances were reached: a high methane production of 0.26 L_{CH4}/gCOD_{input}, a high COD removal efficiency of 96.9%, and a short start-up period (21 days). All these performances exceeded the performances of the APBR, which is the currently used industrial-scale process for this wastewater.

Moreover, a long interval between cleaning of at least 44 days was reached, confirming its interest for industrial-scale development. However, this technology still requires improvement for membrane fouling control, as a high energy demand was observed in our study for membrane filtration. Further study with a submerged membrane could limit the energy demand for this process.

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Wastewater	Configuration	Membrane surface (m ² /m ³)	HRT (days)	Temperature	pH	Wastewater COD content (g _{COD} /L)	OLR (g _{COD} /L/day)	Biogas production (NL/L/day)	Methane production (NL CH ₄ /g _{COD} input)	CH ₄ (%) – CO ₂ (%)	COD Removal efficiency (%)	VFA (eq CH ₃ COOH mg/L)	FOS/TAC	Reference
Malting plant Wastewater	Submerged flat sheet	6 *	1.5	18 °C	7.4	3.2	2.1	0.6 *	0.21	80.95 – 13.51	92	-	-	[20]
Textile effluent	Submerged flat sheet	2.9	2	30 °C–40 °C	-	0.7–1.2	0.20–0.35	-	-	-	82	-	-	[21]
Confectionery wastewater	Submerged flat sheet	4	4.1–2.3	35 °C	7	18–18.9	4.4–7.9	1.45–2.42	0.31–0.26	-	99	-	-	[22]
Brewing wastewater	Submerged flat sheet	7	5	37 °C	7.3–7.7	4.9	0.98	0.213 LCH ₄ /L/day	-	-	-	-	-	[23]
Distillery Wastewater	External tubular ceramic	1.7	2.9 ± 0.1	37 °C	7.36 ± 0.09	12.2	3.97 ± 0.15	1.36 ± 0.05	0.26 ± 0.01	74.7 ± 0.4 – 25.3 ± 0.4	95.4 ± 0.7	476 ± 40	0.25 ± 0.03	This study
Distillery Wastewater	APBR	na	2.8 ± 0.4	37 °C	7.50 ± 0.19	12.2	4.48 ± 0.94	0.94 ± 0.23	0.15 ± 0.04	71.6 ± 1.0 – 28.4 ± 1.0	93.0 ± 2.8	826 ± 40	0.51 ± 0.02	This study

652 Table 2: Performance of AnMBR compared to other recent studies on AnMBR treating real industrial wastewater (2020–2021) and to

653 APBR technology. na: not applicable. *Volume of the reactor was estimated from membrane flux and HRT