

BEET VINASSE TREATMENT USING ANAEROBIC BIORECTOR WITH EXTERNAL ULTRAFILTRATION MEMBRANE

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ABSTRACT

Vinasse is a byproduct generated during ethanol production, and it is produced in large quantities. Although it contains valuable nutrients like calcium, magnesium, and potassium, untreated vinasse presents challenges due to its high solids, organic matter, acidity, and sulfate content, which can negatively affect soil quality. Implementing appropriate technologies could present new opportunities for vinasse utilization, including industrial water reuse, acquisition of high-quality nutrients, and bioenergy generation. Anaerobic Membrane Bioreactors (AnMBR) have emerged as a promising solution for vinasse treatment, eliminating the need for sludge granulation and ensuring complete retention of biomass. AnMBR systems with ultrafiltration configuration produce a final effluent with distinctly reduced organic load, free of solids, yet rich in nutrients. This study evaluated the anaerobic treatment of beet vinasse using AnMBR with external ultrafiltration membrane for beet vinasse treatment. AnMBR showed high potential for beet vinasse treatment operating with volumetric organic loading rates of up to 10 kg COD.m⁻³.d⁻¹ and a membrane flux of 14 LMH. Achieved a maximum flux of 10 LMH while maintaining a COD removal efficiency of 95 ± 3%. Efficiency decreased to 77 ± 11% at 12 LMH flux and further decreased to 73 ± 8% at 14 LMH flux.

Keywords: Beet vinasse. Anaerobic digestion. Membrane. Ultrafiltration. Nutrients recovery.

INTRODUCTION

Vinasse, a byproduct of ethanol production, is generated at different flow rates in distilleries, ranging from 250 to 500 m³.h⁻¹, equivalent to approximately 13 m³ vinasse.m⁻³ ethanol². Rich in nutrients like calcium, magnesium, and mainly potassium, vinasse holds promise as a fertilizer source. However, its high concentration of solids, organic matter, acidity, and sulfate content can harm soil quality when directly disposed of without prior treatment³. Adopting appropriate technologies could offer perspectives of interest on vinasse utilization, including industrial water reuse, nutrient acquisition for fertilization, and energy generation. Anaerobic digestion presents an option for vinasse treatment, reducing its organic load and simultaneously yielding bioenergy through biogas production. However, traditional high-rate reactors used in anaerobic processing require sludge granulation for successful treatment, with risks of biomass wash-out due to various factors like the presence of recalcitrant compounds, high solids, acidity, alkalinity, salinity, and temperature fluctuations that hinder granule formation⁴ (van Lier et al., 2015). Considering these challenges, anaerobic membrane bioreactors (AnMBR) emerge as a compelling solution for vinasse treatment. Unlike traditional methods, AnMBR eliminates the need for sludge granulation and ensures complete biomass retention. Utilizing an ultrafiltration configuration, AnMBR can polish the effluent from anaerobic treatment, yielding a final effluent with reduced organic load and free of solids⁵ (Ozgun et al., 2013) but nutrient rich. This approach holds significant potential for sustainable vinasse management and resource recovery. In this study, beet vinasse anaerobic treatment was evaluated using an AnMBR equipped with an external ultrafiltration membrane. The pressurized external membrane modules offer advantages over submerged membranes modules, including better hydrodynamic control and higher permeate fluxes. External modules also simplify membrane cleaning and replacement, allowing anaerobic conditions to be maintained in the main reactor during maintenance. This makes it easier to address membrane fouling in an external membrane reactor⁶. This study aimed to assess AnMBR with an external ultrafiltration membrane for treating beet vinasse, aiming to produce high-quality effluent with reduced organic load and no solids. The findings will contribute to future research focusing on vinasse-based fertilizer production through separation processes.

MATERIAL & METHODS

Mesophilic anaerobic suspended sludge (39 g VSS. L⁻¹), which was already adapted to beet vinasse was used as seed sludge. The reactor was inoculated with 3.4 L of the seed sludge resulting in an initial concentration of around 22 gVSS.L⁻¹. The beet vinasse was supplied by beet biorefinery located in France and stored at -19°C degrees to maintain the characteristics of the material until the moment of using. The beet vinasse was characterized by chemical oxygen demand (COD) content between 20-26 g.L⁻¹, 2,000 mg sulfate. L⁻¹, 1,059 mg acetic acid. L⁻¹, 4.8 g VSS.L⁻¹, 104.2 mg Na. L⁻¹, 1,395.8 mg K. L⁻¹, 112 mg Ca. L⁻¹ and 115.5 mg Mg. L⁻¹. Centrifugation (3,500 rpm for 7 minutes) was carried out to simulate the yeast removal step that vinasse goes through during the process on industrial scale. After the centrifugation around 70% of the vinasse total suspended solids were removed. As the raw vinasse pH = 3.5, during the reactor start-up the ratio of 0.7 g NaHCO₃.g vinasse COD⁻¹ was supplied to provide a substrate with pH=7. After performance stabilization, the ratio was decreased to 0.3 g bicarbonate. g⁻¹COD and finally

completely removed. The pH inside the reactor was kept around 7 during the whole operation. The temperature was maintained at 35 ± 1 °C.

The AnMBR consisted of a CSTR reactor with a volume of 7.0 L and working volume of 6.5 L, connected to an external ultra-filtration (UF) polymeric membrane module with 30 nm nominal pore size (Pentair X-flow, the Netherlands). The AnMBR was fully automated and controlled. The operational parameters were collected by LabVIEW software (version 15.0.1f1, National Instruments, USA) developed by Carya (Carya, NL). The transmembrane pressure (TMP) was calculated based on the pressure at the membrane module inlet, outlet and permeate exit, automatically carried out by the LabVIEW software. The biogas production was measured with a gas meter (Ritter MGC-10 PMMA R, DE). The AnMBR operation was divided into five different phases in which different conditions regarding the organic load rate (OLR), hydraulic retention time (HRT) and membrane flux (J) were applied (Table 1). Initially, a crossflow velocity (CFV) of 1m/s was applied.

Table 1 Operational conditions

| Phases | Days | HRT (d) | Flow rate (L.d ⁻¹) | OLR (gCOD.L ⁻¹ .d ⁻¹) | J (LMH) |
|--------|-----------|---------|--------------------------------|--|---------|
| I | 0 – 14 | 4.3 | 1.5 | 4.2 | 6 |
| II | 15 – 32 | 3.25 | 2.0 | 5.5 | 8 |
| III | 33 – 97 | 2.6 | 2.5 | 8 | 10 |
| IV | 98 – 107 | 2.2 | 3.0 | 10 | 12 |
| V | 108 – 119 | 1.9 | 3.5 | 10 | 14 |

1 RESULTS & DISCUSSION

The COD removal efficiency remained consistently above 95% throughout most of the reactor operational periods (Fig. 1a). However, a decline occurred from day 97 onward when the OLR was increased to 10 kg COD. m⁻³.d⁻¹ and the flux was raised to 12 LMH and then to 14 LMH. The efficiency hit its lowest point, 62%, on day 110, but subsequently began to recover, reaching 80% by the end of the operation. It couldn't be assured that the reactor would surpass 90% efficiency again, but the trend towards recovery was promising. Compared to other sugarcane vinasse treatment configurations^{7,8}, this study achieved either equal or higher removal efficiencies at the same OLR. However, there haven't been any studies on vinasse treatment in AnMBR with higher OLR published yet^{9,10}.

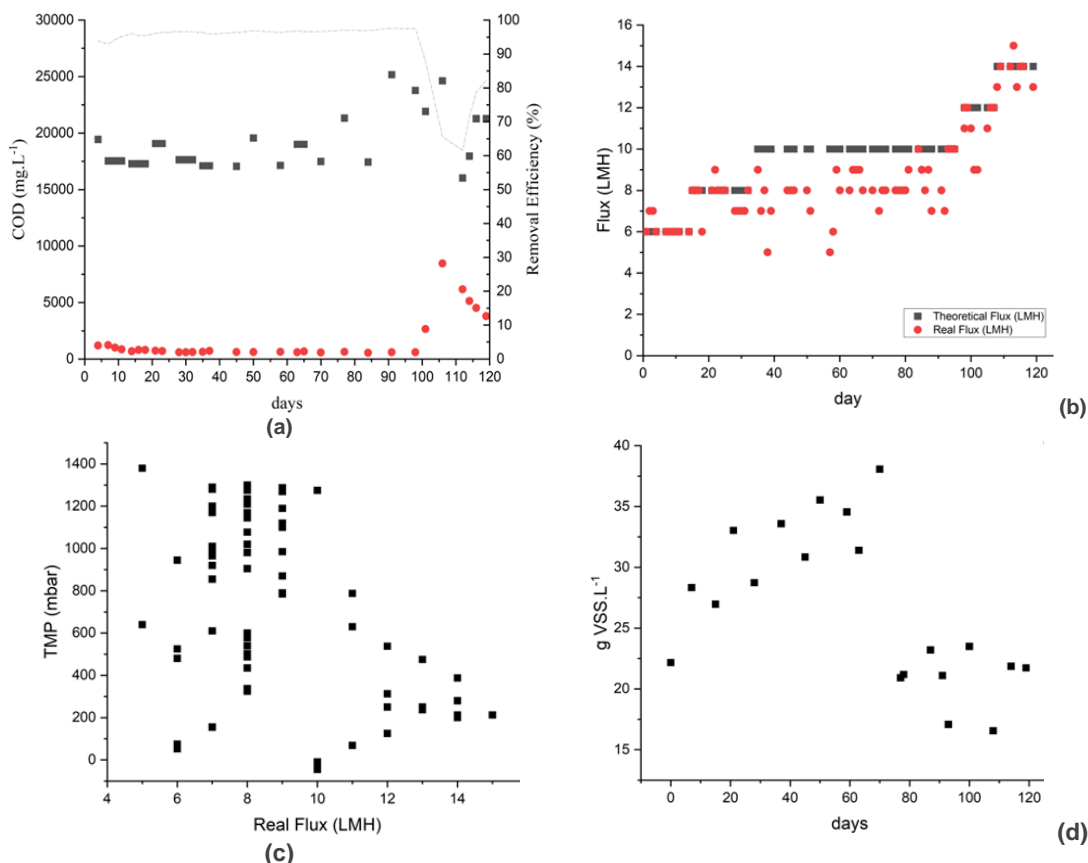


Figure 1 Monitoring AnMBR: (a) COD removal efficiency ■ Input; ● Output; (---) removal efficiency, (b) membrane flux, (c) VSS concentration and (d) TMP monitoring in each flux applied.

Membrane performance is summarized in Table 2. Strategies were tested to enhance flux, including increasing the CFV and decreasing VSS concentration within the reactor. At phases I (day 1 to 14) and II (day 15 to 32), fluxes of 6 and 8 LMH were respectively applied, maintaining the desired flux without any compromising issue (Fig. 1b). Increasing the flux to 10 LMH in phase III resulted in flux maintenance loss and TMP increase (Fig. 1c). Attempting to increase CFV from 1 to 2 m/s on day 39 didn't improve the process. Subsequently, the sludge content in the reactor was reduced on day 72, leading to VSS concentration reduction from 38 to 21 g VSS.L⁻¹, with no flux improvements, despite the TMP decrease. A further reduction to 17 g VSS.L⁻¹ on day 93 didn't affect COD removal efficiency, but a tube issue with the recirculation pump occurred due to the high CFV (2 m/s), necessitating a reduction to 1.5 m/s. Despite these changes, COD removal efficiency and real flux remained stable at 10 LMH. Following the VSS concentration decrease and membrane cleaning, the TMP significantly reduced (Fig. 1d). Flux was then increased to 12 LMH, maintaining real flux at 12 LMH, but with the first observed reduction in COD removal efficiency. To assess membrane efficiency at higher flux, despite biodegradability efficiency loss, flux was increased to 14 LMH after operating for 10 days at 12 LMH. Sludge was purged to maintain a concentration around 16 g VSS.L⁻¹ and to keep the TMP low. Despite flux increase, the OLR didn't change due to the lower vinasse concentration compared to the previous phase (Table 1). Real flux was maintained, but with losses in COD removal efficiency dropping to around 60%. Flux increase was stopped to avoid reactor collapse from the rising OLR.

Table 2 Operational parameters monitoring

| Theoretical Flux (LMH) | Real Flux (LMH)* | TMP (mbar)* | CFV (m/s) | VSS (g/L)* | COD removal efficiency (%) * |
|------------------------|------------------|-------------|-----------|------------|------------------------------|
| 6 | 6 | 70 | 1 | 22.2 | 94 |
| 8 | 8 | 691 | 1 | 28.7 | 96 |
| 10 | 7 | 1215 | 1 | 33.6 | 97 |
| 10 | 8 | 1124 | 2 | 38 | 97 |
| 10 | 8 | 1132 | 2 | 21 | 97 |
| 10 | 10 | 70 | 2 | 17 | 95 |
| 12 | 12 | 572 | 1.5 | 23.5 | 77 |
| 14 | 14 | 301 | 1.5 | 16.6 | 73 |

*Average values

2 CONCLUSION

AnMBR reduced the polluting load of beet vinasse generating an effluent free of solids. The maximum attainable flux was 10 LMH without losing the performance of COD removal efficiency of $95 \pm 3\%$. At a flux of 12 LMH the attained efficiency was $77 \pm 11\%$ and at a flux of 14 LMH this was $73 \pm 8\%$. A VSS concentration below 20 g VSS.L⁻¹ is seemingly essential for reducing the TMP and to maintain the desired flux. Using AnMBR to treat vinasse offers a promising solution for turning vinasse into a valuable resource. This approach not only helps to recycle water for industrial use but also to extract nutrients and recover the biochemical energy in the form of biogas. The researched AnMBR approach is a new perspective in managing ethanol production byproducts.

REFERENCES

- JUNQUEIRA, T.L., CHAGAS, M.F., GOUVEIA, V.L.R., REZENDE, M.C.A.F., WATANABE, M.D.B., JESUS, C.D.F., CAVALETT, O., MILANEZ, A.Y., BONOMI, A. 2017. Techno-economic analysis and climate change impacts of sugarcane biorefineries considering different time horizons. *Biotechnology. Biofuels* 10, 50. <https://doi.org/10.1186/s13068-017-0722-3>
- SANTOS PS, ZAIAT M, NASCIMENTO CAO, FUESS LT. 2019. Does sugarcane vinasse composition variability affect the bioenergy yield in anaerobic systems? A dual kinetic-energetic assessment. *Journal of Cleaner Production*; 240:118005.
- FUESS, L.T., GARCIA, M.L. (2014). Implications of stillage land disposal: A critical review on the impacts of fertigation. *J. Environ. Manage.* 145, 210–229. <https://doi.org/10.1016/j.jenvman.2014.07.003>
- VAN LIER, J.B., VAN DER ZEE, F.P., FRIJTERS, C.T.M.L., ERSAHIN, M.E. 2015. Celebrating 40 years anaerobic sludge bed reactors for industrial wastewater treatment. *Rev. Environ. Sci. Bio/Technology* 14. <https://doi.org/10.1007/s11157-015-9375->
- OZGUN, H., KANAN, R., EVREN, M., KINACI, C., SPANJERS, H., LIER, J.B. VAN. 2013. A review of anaerobic membrane bioreactors for municipal wastewater treatment: Integration options, limitations, and expectations. *Sep. Purif. Technol.* 118, 89–104.
- LIN, H., PENG, W., ZHANG, M., CHEN, J., HONG, H., ZHANG, Y. 2013. A review on anaerobic membrane bioreactors: Applications, membrane fouling and future perspectives. *Desalination* 314, 169–188. <https://doi.org/10.1016/j.desal.2013.01.019>
- AQUINO, S., FUESS, L.T., PIRES, E.C. 2017. Media arrangement impacts cell growth in anaerobic fixed-bed reactors treating sugarcane vinasse: Structured vs. random biomass immobilization. *Bioresour. Technol.* 235, 219–228. <https://doi.org/10.1016/j.biortech.2017.03.120>
- FUESS, L.T., COLLING, B., FERREIRA, M., CRISTINA, M., FERREIRA, A., LOUREIRO, M., BONOMI, A., ZAIAT, M. 2018. Diversifying the technological strategies for recovering bioenergy from the two-phase anaerobic digestion of sugarcane vinasse: An integrated techno-economic and environmental approach. *Renew. Energy* 122, 674–687. <https://doi.org/10.1016/j.renene.2018.02.003>
- MOTA, V.T., SANTOS, F.S., AMARAL, M.C.S. 2013. Two-stage anaerobic membrane bioreactor for the treatment of sugarcane vinasse: Assessment on biological activity and filtration performance. *Bioresour. Technol.* <https://doi.org/10.1016/j.biortech.2013.07.110>
- SANTOS, F.S., RICCI, B.C., FRANÇA NETA, L.S., AMARAL, M.C.S. 2017. Sugarcane vinasse treatment by two-stage anaerobic membrane bioreactor: Effect of hydraulic retention time on changes in efficiency, biogas production and membrane fouling. *Bioresour. Technol.*

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