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# Two-stage anaerobic membrane bioreactor for the treatment of sugarcane vinasse: Assessment on biological activity and filtration performance

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## HIGHLIGHTS

- A two-stage AnMBR was designed for the treatment of sugarcane vinasse.
- Intermittent feeding was found to be effective to acclimate the microorganisms.
- COD and DOC removals efficiencies were 96.9 ± 0.7% and 95.0 ± 1.1%, respectively.
- Membrane filtration resistance was found to be predominantly removable.
- SMP protein and EPS protein were correlated to membrane filtration resistance.

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## ABSTRACT

A two-stage submerged anaerobic membrane bioreactor (2-SAnMBR) was designed for the treatment of sugarcane vinasse. For start-up, the flow rate was reduced whenever VFA levels reached critical levels in the methanogenic reactor. After acclimation, the system was operated under a continuous flow. Separation of the stages was observed during the entire period of operation. VFA, COD and DOC levels of raw effluent, acidified effluent and permeate averaged 2141, 3525 and 61 mg VFA $L^{-1}$ (as acetic acid), 15727, 11512 and 488 mg COD $L^{-1}$, and, 3544, 3533 and 178 mg DOC $L^{-1}$, respectively. Overall COD and DOC removal efficiencies of 96.9 ± 0.7% and 95.0 ± 1.1%, respectively, were reached. Methane content of the biogas from the acidogenic and methanogenic reactors ranged 0.1–4.6% and 60.1–70.1%, respectively. Removable fouling strongly affected filtration performance and cake layer formation accounted for most of filtration resistance. Membrane resistance was related to presence of protein-like substances and carbohydrates.



## 1. Introduction

Distillery wastewater is one of the major concerns in alcohol production. For each 1 L of ethanol, approximately 15 L of vinasse are produced (van Haandel, 2005). Important characteristics of sugarcane vinasse include low pH, high levels of BOD, COD, potassium, sulfate and color (Wilkie et al., 2000). The large volumes produced, associated with its high content of biodegradable organic matter, mean that vinasse is a potential source of energy through anaerobic digestion and biogas recovery. In addition to the intrinsic advantages of anaerobic digestion such as low nutrient requirement, small production of excess sludge, lower energy input and generation of methane-rich biogas (Lettinga et al., 1980), anaerobic digestion has been recognized as the most attractive method for vinasse treatment (de Bazúa et al., 1991; Wilkie et al., 2000; van Haandel, 2005). However, anaerobic digestion applied to vinasse treatment is not a well-established technology, and it is put at risk given the fluctuations in the quantity and quality of the vinasse to be processed and the presence of inhibitory compounds.

The instability of anaerobic reactors treating high strength wastewaters is usually associated with an uneven production and consumption of volatile fatty acids. Acidogenic bacteria have the highest growth rates among the microbial consortium and are generally more resistant to environmental stress conditions than syntrophic acetogenic bacteria and methanogenic archea (Ke et al., 2005). Two-stage anaerobic digestion allows the maintenance of optimal conditions for each group of microorganisms involved in each phase of anaerobic digestion, providing improvement in the treatment efficiency, reduction of inhibitory effects of toxic compounds on methanogens (Beccari et al., 1996), higher

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methane content in biogas (Lun et al., 1995; Yeoh, 1997), tolerance to greater organic load (Ghosh et al., 1985), reduction in the accumulation of propionic acid and more stability when undergoing shock loading (Cohen et al., 1982).

Additionally, the application of membrane retention to anaerobic digestion in the anaerobic membrane bioreactors (AnMBRs) ensures complete retention of biomass and total suspended solids (TSS). The system greatly increases effluent quality in terms of COD and TSS, and favors the maintenance of slow growth microorganisms such as methanogens. Despite the advantages of AnMBRs over conventional anaerobic reactors, this technology is limited mainly by membrane fouling, which affects membrane area requirements and operational costs. While probably very similar to aerobic MBRs, a lot less is known about fouling in anaerobic ones, requiring research to understand how reactor operation influences fouling and release of soluble microbial products (SMP), which accounted for much of the soluble COD (Stuckey, 2012).

Many studies have been performed in order to improve the operational conditions and to identify the factors causing flux reduction in AnMBRs. Jeison and van Lier (2007b) and Jeison et al. (2009) verified that the degree of wastewater acidification strongly affects filtration performance. Submerged and side-stream thermophilic AnMBRs treating acidified wastewater exhibited much better performance than those treating partially or non-acidified wastewater. The authors revealed that acidogenic bacteria, induced by the feed with partially or non-acidified wastewater, grow mostly as individual cells rather than as flocs. This increases the relative amount of smaller particles and sludge viscosity and affects the degree of cake layer deposition, reducing filtration performance.

Based on the results discussed above, it is inferred that in a two-stage AnMBR (2-AnMBR), in which the methanogenic reactor is coupled to a membrane module, the occurrence of acidogenesis in an earlier step could prevent acidogenic biomass growth in the methanogenic reactor, enhancing sludge properties and filtration performance. Studies found in the literature comprise both side-stream 2-AnMBR at 37 °C (Saddoud et al., 2007; Saddoud and Sayadi, 2007) and at 55 °C (Wijekoon et al., 2011), as well as submerged 2-AnMBR at 35 °C (Jeong et al., 2010). In general, they presented good performance for the treatment of high strength wastewaters.

Given the few studies on the application of an acidogenic reactor followed by a methanogenic membrane bioreactor for wastewater treatment, this research aims to contribute to the development of AnMBR technology, raising new concerns about 2-AnMBRs. The present research describes the biological and filtration performance of a two-stage submerged anaerobic membrane bioreactor (2-SAnMBR) treating sugarcane vinasse at room temperature (22 °C). Analyses of the effluent and biogas from both reactors were performed in order to estimate the degree of acidification and organic matter degradation in each phase of anaerobic digestion. The main fouling mechanisms are discussed as well as the influence of soluble microbial products (SMP) and extracellular polymeric substances (EPS) on them.

## 2. Methods

### 2.1. Vinasse samples

Vinasse samples were obtained from a distillery located in the state of São Paulo (Brazil), which produces ethanol from sugarcane juice. The plant has the capacity to grind up to 800 thousand tons of cane per harvest. The samples were kept refrigerated at 4 °C. The physicochemical composition of the samples is shown in Table 1.

### 2.2. Experimental setup

A schematic of the experimental setup designed for this study is shown as Fig. 1. Two anaerobic reactors made of acrylic were placed in series with the purpose of separating acidogenic and methanogenic stages. Acidogenesis was carried out in an upflow anaerobic reactor with 14 cm diameter and 87 cm height. Valves situated in the side outlets enabled the operation in four levels, corresponding to 2.3 L, 3.8 L, 6.7 L and 11.0 L. The acidogenic reactor (AR) effluent fed the methanogenic reactor by gravity. Methanogenesis was carried out in a continuous stirred anaerobic reactor (IKA RW 16 Basic® stirrer, 250 rpm speed) with 24 cm diameter and 67 cm height. The working volume was controlled by four electric level sensors, corresponding to 7.8 L, 9.3 L, 20.1 L and 24.0 L, which were connected to the peristaltic pump and switched it off if the level was above the selected one. The methanogenic reactor (MR) was fitted with a microfiltration (MF) unit. The membrane module was composed of 205 polyetherimide hollow-fibers with nominal pore size of 0.45 μm and 7 cm length, whose total surface area was 0.045 $m^2$ (Pam Membranas Seletivas, Brazil). Transmembrane pressure (TMP) was provided by a vacuum tank connected to a pump and was controlled by a valve to regulate air inlet. The vacuum pump maintained the vacuum tank (VT) negatively pressurized so as to allow the suction of permeate. The vacuum in VT was controlled to maintain the permeate flux constant. The filtration took place until the VT upper level was reached. Permeate was then discharged to the permeate tank (PT), which also functioned as a feed tank for back-flush. When back-flush or relaxation was activated, the vacuum line was interrupted. If relaxation was used instead of back-flush the back-flush pump was kept powered off. The time between filtration and back-flush/relaxation was automatically programmed via a timer. The system was maintained at room temperature (general average 22 °C, minimum average 19 °C, maximum average 27 °C).

### 2.3. Operational conditions

The reactors were inoculated at an initial concentration of 20 g MLVSS $L^{-1}$ with flocculent sludge from a single-stage UASB reactor treating domestic sewage in the Centre for Research and Training on Sanitation UFMG/COPASA – CePTS, located in Belo Horizonte, Brazil. Before starting the 2-SAnMBR operation, an acclimation period was needed in order to select and enrich the microorganism populations associated with acidogenic and methanogenic biochemical reactions in the first and second reactors, respectively. Undiluted vinasse (Sample 1) was used as the feed wastewater. In this period, the treated effluent was collected using a peristaltic pump and transferred to the permeate tank. The settled sludge was recycled daily in the methanogenic reactor. VFA safe levels for anaerobic digestion were maintained in the methanogenic reactor by reducing the flow rate when the VFA reached critical levels (above 1000 mg $L^{-1}$). During the whole period of operation, the pH in the acidogenic reactor was not controlled, as the pH in the methanogenic reactor was kept above 6.5 by supplying sodium bicarbonate when necessary.

Once start-up was finished, the membrane module was fitted to the methanogenic reactor. The working volumes of the acidogenic and methanogenic reactors were kept at 6.7 L and 24.0 L, respectively. Sample 1 fed the system from day 0 to day 3 and Sample 2 fed the system from day 4 to day 57. The 2-SAnMBR operated under an average organic loading rate (OLR) of 2.5 gCOD $L^{-1}$ $d^{-1}$, at an infinite sludge age. Most of the time, 40 s of relaxation for each 8 min of filtration was the only strategy used to avoid membrane fouling; the instantaneous flux was 4.8 L $m^{-2}$ $h^{-1}$, in order to achieve a permeate productivity equivalent to an average flux of 4.4 L $m^{-2}$ $h^{-1}$.