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Anaerobic Treatment of Semiconductor Industry Wastewater and Biomass Acclimation

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This work investigated the feasibility of an anaerobic biomass from a municipal wastewater treatment plant (MWTP) to acclimate to an effluent from a semiconductor industry for treating it over a 132 days period in semi-continuous assays. The study consisted of four phases: the Stimulation period, where the inoculum was fed with sucrose; the Acclimation period, involving a continuous increase in effluent concentration; Stabilization 1, with feeding exclusively from effluent; and Stabilization 2, where a mixture of effluents and simulated wastewater (SW) was introduced. Methanisation and chemical oxygen demand removal efficiency were on average 59% and 79% respectively. Average methane production was 0.1 mL of CH_4 per mg of COD. The continuous production of methane, total volatile acids consumption, no accumulation of organic compounds and decrease in alkalinity confirmed a stable operation of the anaerobic system. An overall lower efficiency in methane production in comparison to other studies may indicate other reactions for organic degradation besides methanogenesis, as also be explained by the complex composition of the wastewater (WW) that might have caused inhibition alongside accumulation of degradation intermediates. Therefore, it is important to consider pre- and post-treatments in future studies. Results presented indicate that anaerobic inocula collect from a MWTP can acclimate and effectively treat WW from the semiconductor industry.

1. Introduction

Currently, the use of electronics is ubiquitous to human activity, and semiconductors are key components for their production, with a market size of 543 billion dollars (SIA, 2022). They are materials with specific conductivity with properties varying according to the addition of doping, thus determining their charge transport conditions (Seeger, 1973). Silica is the main material used for its manufacture, followed by other metalloids. The production of semiconductors becomes the main basis for the manufacture of electronics and microelectronics, such as LED TVs or smartphones.

Within the semiconductor manufacturing process, the degree of purity must be high to guarantee their expected properties. To achieve this purity, several stages of chemical treatments and cleaning are carried out. The main process in its manufacturing includes wafer fabrication, oxidation, photolithography, etching, ion deposition and implantation, metallization, electrical matrix sorting and packaging (Tsai et al., 2002). In these processes, a wide range of chemicals are used, including metals, solvents and acids. The remaining solutions and suspended solids are removed by washing and cleaning using vast amounts of ultrapure water. The worldwide consumption of ultrapure water in semiconductors industries was estimated to be about 5.51 $\times 10^8$ m^3 in 2022 (Wang et al., 2023).

Such use of ultrapure water for washing of wafers generates a high volume of complex WW, with different concentrations of pollutants, such as tetramethylammonium hydroxide (TMAH), phosphoric acid, ammonia, surfactants, organic solvents and heavy metals (Sim et al., 2023). Although many of these pollutants are recalcitrant and harmful to the environment, they often present organic character. Therefore, there is room for their treatment by biological processes, which are simple and cost-effective. Anaerobic digestion has become an interesting option for treatment of semiconductor WW with several studies demonstrating its capabilities for

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degrading different chemicals found in these effluents such as dimethyl sulfoxide (DMSO) (Cheng et al., 2019) and TMAH (Syutsubo et al., 2021). Some of the advantages in using anaerobic treatment are the low excess sludge production, energy recovery through methane production, possibility of compact designs and lower capital and operating costs (Zieliński et al., 2022). Although anaerobic microorganisms are known to thrive in complex and recalcitrant chemicals there is a threshold for their degradation efficiency, therefore the concentration of those chemicals will determine the efficiency of the digestion. This study sought to conduct a preliminary analysis to assess the potential of anaerobic biomass for the treatment of recalcitrant effluents produced by the semiconductor industry.

2. Materials and methods

2.1 Materials

Two types of effluent were obtained from a semiconductor industry located in Portugal, one with chemicals and diluted acid mixture (E1) and the other with a mixture of E1 and strong acids (E2). More specifically, it was also confirmed through the processes carried out by the industry that TMAH, isopropanol, a non-ionic surfactant, sodium persulfate, copper sulfate, citric acid, acetic acid, sulfuric acid, hydrofluoric acid and phosphoric acid were utilized in varying concentrations depending on the needs of production. The tanks from where the effluents were collected contained a mixture resulting from different processes, mainly lithography, packaging, plating, dicing, grinding and laser grooving and the many steps of washing and cleaning. To avoid any setbacks with the strong acidic content in the effluent E2, it was collected after the pH control step. Anaerobic inoculum was obtained from a MWTP located in north-central Portugal treating both domestic and industrial WW.

2.2 Experimental setup and operation

The experiments were performed in four separate phases, lasting for a total of 132 days, as follows. In the Stimulation phase (36 days) the biomass was only fed with sucrose to enhance the metabolic activity and establish a baseline. In the Acclimation phase (30 days) the biomass was fed with a continuous step-increase (10%) in effluent concentration, summing up 10 moments (10%, 13.3%, 17.7%, 23.6%, 31.4%, 41.8%, 55.6%, 74%, 98.5% and 100%). Stabilization 1 phase (15 days) consisted in feeding on 100% effluent. Finally, in Stabilization 2 phase (51 days) the biomass was fed with a mixture of effluents collected from different periods of the industrial operation to evaluate seasonality and complexity of the effluent. In this phase, the COD was also increased with the addition of simulated wastewater (SW) solution made with sodium acetate (representing dissolved acetic acid, a common acid heavily used by this industry) and TMAH with a chemical oxygen demand (COD) of 145 g L^{-1}.

The anaerobic assays were carried out in four glass reactors, two with a working volume of 5 L and two with working volume of 2 L. A fifth 2 L reactor was used as a control for growing inoculum fed only with sucrose. All assays started with a biomass concentration of 7 g VSS L^{-1}. Neutralized effluent was fed, and samples were collected every three days, with reactors maintained at 35°C. At each phase, nutrients and sodium bicarbonate were added to support digestion (Rebac et al., 1997). Biomass sludge samples were taken every six days to assess biomass concentration.

2.3 Analytical methods

Effluent characterization before and after anaerobic treatment followed Standard Methods (APHA, 1999), assessing COD, BOD (5 days, Oxitop®), pH, electrical conductivity (EC), alkalinity, and total volatile acids (TVA). Anaerobic biomass concentration and activity were evaluated through volatile suspended solids (VSS) and specific methanogenic activity (SMA). Methane was purified by NaOH (20% w/w) gas washing (adapted from Tahir et al., 2015) and quantified using a syringe (adapted from Nganyira et al., 2023).

3. Results and discussion

The properties of semiconductor WW are known to be complex and highly variable due to the differences in techniques of production, mixing of effluents from different processes and mainly due to dilution from cleaning water, therefore the often-provided examples of WW with broad ranges for each relevant parameter (Noman et al., 2024). The WW samples used in this study are not different, and although they are from the same tank, the difference on collection day is enough to demonstrate high variability in all parameters, as can be observed in Table 1. Despite the variations, the characteristics are still within the WW profiles found for this industry (Noman et al., 2024).

Table 1: Average values for the characteristics of wastewater collected from the semiconductor industry

Parameters	Diluted Acids tank (E1)	Lowest -Highest values	Effluent Mixture tank (E2)	Lowest - Highest values
pH	6.06	4.74 - 9.10	9.47	5.48 - 11.50
EC (mS cm^{-1})	3.93	0.27 - 11.60	4.43	3.49 - 6.20
Alkalinity (mg L^{-1} of $CaCO_3$)	98.18	68.75 – 125.00	283.33	150.00 – 425.00
TVA (mg L^{-1})	245.84	100.00 - 365.63	233.34	159.38 – 375.00
Kjeldahl Nitrogen (mg L^{-1})	13.16	11.20 - 14.84	13.72	10.36 - 17.08
Total Phosphorus (mg L^{-1})	1.03	0.27 - 1.64	0.42	0.29 - 0.50
COD (mgO_2 L^{-1})	749.91	270.40 - 1,245.40	757.19	504.10 - 1,114.10
BOD (mg L^{-1})	259.94	162 - 411.10	281.43	184.89 - 353.60
Biodegradable COD fraction (%)	34.66	19.83 - 82.90	37.17	25.39 - 66.30
Total Solids (g L^{-1})	5.63	1.85 - 11.76	3.55	1.93 - 4.39
Dissolved Solids (g L^{-1})	5.35	1.76 - 11.32	3.29	1.63 - 4.32
Suspended Solids (g L^{-1})	0.21	0.03 - 0.54	0.26	0.06 - 0.34
Volatile Solids (g L^{-1})	0.11	0.01 - 0.18	0.13	0.05 - 0.18

The overarching goal of this study was to conduct a preliminary assessment and demonstrate the effectiveness of anaerobic processes for treatment of this WW with high variability in its composition, as it has been proposed in previous studies (Syutsubo et al., 2021; Lv et al., 2023). An analysis of the wastewater (WW) characteristics reveals significant biodegradable content, along with macronutrients such as nitrogen (N) and phosphorus (P), which support microbial metabolism. Additionally, most of the solids and organic compounds are dissolved. Considering that these favorable conditions for anaerobic processes are not always found for this type of WW (Zieliński et al., 2022), it is essential to emphasize the advantages of the application of a cost-effective method capable of WW treatment that enables energy and water recovery. However, the presence of highly recalcitrant and inhibitory compounds may pose challenges for microbial activity such as fluoride (Ochoa-Herrera et al., 2009), copper (Otero-González et al., 2014), surfactants (Hu et al., 2012) and TMAH itself (Lv et al., 2023), potentially leading to metabolism disruption but adaptation of the anaerobic microbiota is expected. Table 2 depicts the results of the treated effluent after 132 days of operation.

Table 2: Mean values of anaerobic digestion treatment

Parameters	Diluted Acids tank (E1)	Lowest - Highest values	Effluent Mixture tank (E2)	Lowest - Highest values
Feed COD (mgO_2 L^{-1})[a]	1,113.90	837.60 - 1,390.70	1,211.70	901.30 - 1,433.20
Final COD (mgO_2 L^{-1})[a]	230.40	130.1 - 393.80	238.40	163.70 - 370.60
COD Removed (%)	79.31	71.68 – 84.46	80.32	74.14 – 81.83
Final pH[a]	8.04	7.45 - 8.54	8.07	7.53 - 8.66
Final Alkalinity[a]	1,262.90	543.75 - 2,262.50	1,356.78	612.50 - 2,337.50
Final EC (mS cm^{-1})[a]	3.97	2.77 - 4.60	4.42	3.24 - 5.36
Final Total Volatile Acids (mg L^{-1})[a]	58.77	27.50 - 140.62	62.53	33.75 - 126.06
CH_4 Produced (mL)[a]	131.68	12.00 – 412.00	111.50	2.00 – 442.00
Methanisation Efficiency (%)[a]	65.60	45.67 – 83.34	52.52	26.55 – 68.56
SMA (g CH_4-COD g^{-1} VSS d^{-1})[a]	0.009	0.002 - 0.028	0.011	0.003 - 0.038

[a]. Global average for each feeding run

It is worth mentioning that methane production remained constant despite the different stages. COD removal was on average 79% for both types of WW, and although the values are lower when compared with other investigations, such amount of degradation is in accordance with other studies such as Cheng et al. (2019) who reported COD removal ranging 70-90% with a influent COD of 1,800 mg/L, but when the organic load was increased to 8,000 mg/L the microbial culture was inhibited and COD removal dropped to values below 70%. Danshita et al. (2018) reported a COD removal rate of 50% for a WW containing 1500 mg/L of COD before the acclimation of biomass to TMAH degradation and achieving COD removals of 90% on average after acclimation despite being operated in psychrophilic temperatures. Following studies in such conditions, Syutsubo et al. (2021) reported a removal of 96% of COD. It is important to emphasize that in the studies cited, synthetic or diluted WW were used, thus reducing the impact of other contaminants in a real WW.

The methanisation efficiency obtained values ranged between 65% and 52% based on the input gCOD and produced gCOD.CH_4. However, SMA had low values when compared to other works, resembling SMA from reactors with a notable presence of sulfidogenic microbiota (Gámez et al., 2008). A low SMA and high conversion of COD to CH_4 may indicate that there is little substrate available for all microorganisms, indicating that there is the possibility of generating even more methane when at higher loads. Even considering the potential presence of sulfidogenic microbiota competing for resources, there was no significant impact on methane production, as a high consumption of TVA confirms that there was no accumulation of volatile acids, demonstrating stability in the system. The constant production of volatile acids from acidogenesis was confirmed through the alkalinity reduction throughout the experiment. It is also possible to verify from Figure 1 that there was no accumulation of organic compounds over the different phases, confirming its degradation even when higher loads were added with different mixtures of WW.

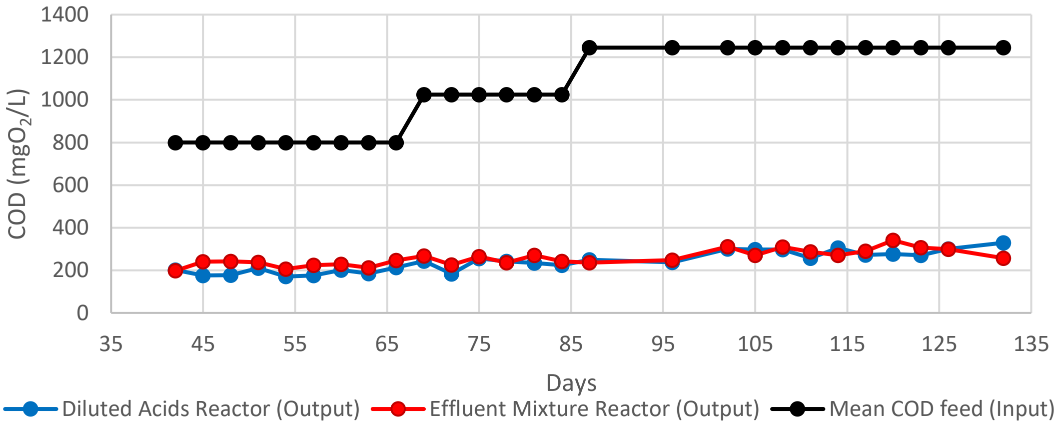

Figure 1: Mean COD concentration for feed and treated effluents, from Acclimation to Stabilization 2 phases.

Stabilization 1 achieved the highest COD removal and methanisation, with SMA increasing from 0.0061 to 0.0137, indicating microbial adaptation to the effluent's composition and organic load. In contrast, Stabilization 2, with a mixed effluent containing higher contaminant concentrations, caused system destabilization, confirmed by the SMA decrease to 0.0070, suggesting inhibition. While complex effluents with harmful compounds like heavy metals or TMAH can be treated, their effectiveness is reduced. Other works also experienced a drop in overall treatment efficiency due to the presence of heavy metals (Yang et al., 2012), especially copper (Otero-González et al., 2014) and high concentrations of TMAH (Lv et al., 2023). Not only do the compounds in the effluent can cause inhibitions, but also the degradation intermediates of the digestion can also be inhibitory, such as the case with the degradation of TMAH, which the final product is ammonia that can unbalance the anaerobic system at high concentrations (Whang et al., 2014).

It was observed by Li et al. (2023) that not only the methanogens are responsible for the degradation of TMAH in semiconductor WW, corroborating that a reactor with greater diversity of microorganisms is more capable of degrading complex compounds. Although sulfidogenic microorganisms can unbalance the anaerobic system due to competition for resources with methanogens, Sierra-Alvarez et al. (2007) demonstrated that they are capable of mineralizing copper ions, thus being an alternative for reducing this metal in the final effluent and as a form to reduce the impacts generated by toxicity. The presence of sulfidogenic microorganisms can also be effective to degrade other compounds that are not suitable for methanogenic archaea such as surfactants according to Hollingsworth et al. (2005).

The experimental design and execution, characterized by semi-continuous feeding, inherently promotes the accumulation of inorganic compounds and other substances that are non-biodegradable by anaerobic microorganisms, such as copper, fluoride, and ammonia, within the reactor. Over time, as these compounds progressively accumulate, the efficiency of the anaerobic treatment is expected to decline, despite the system's adaptation to the WW. This context is possible to occur even in other types of configurations such as continuous systems due to a greater flow of effluent to the reactors. Therefore, it is necessary to consider further strategies such as pre- and post-treatments for this type of WW. Several studies have already sought the combination of treatments to increase the treatment efficacy of these effluents. Considering the increase of biodegradation to improve the removal of organic compounds, different authors proposed the use of oxidative systems as a pre-treatment to achieve them, either by using ozone (Satyendra Tripathi and Touseef Hussain, 2021), Fenton (Park et al., 2001) or anodic oxidation (Mousset et al., 2018) with varying degrees of efficiency. As a post-treatment for the removal of anaerobic degradation products, such as ammonia, the use of aerobic and anoxic systems was found to reach reaching a maximum removal of nitrogen of 63% along with a TMAH reduction between 70 and 100% (Lin et al., 2015). Sierra-Alvarez et al. (2007) combined a crystallization reactor filled with quartz salt and a sulfate reducing bioreactor to remove copper, reaching 99% and 70% removal of copper and COD respectively. Alternatively, electrochemical processes can complement anaerobic treatment by facilitating the removal of solids, including heavy metals by electro flotation, or promoting the degradation of complex compounds by electrooxidation (Yang et al., 2014).

4. Conclusions

This study demonstrated that semi-continuous bioreactors inoculated with anaerobic microorganisms from a MWTP can effectively treat WW from the semiconductor industry. On average, methanisation and COD removal efficiency were 59% and 79%, respectively, with methane production of 0.1 mL of CH_4 per mg of COD. The continuous production of methane, TVA consumption, alkaline decline and the absence of COD accumulation confirmed system stability. Low SMA and high methanisation suggest limited carbon sources for microorganisms. The potential presence of sulfidogenic biota may help remove inhibitory compounds like copper and surfactants. The increased organic load and effluent complexity reduced treatment efficiency, highlighting the need for pre-treatment to improve biodegradability and post-treatment to remove inhibitory byproducts. Further research at larger scales is needed to validate these findings in industrial settings.

Nomenclature

BOD – biological oxygen demand, $mgO_2\ L^{-1}$
COD – chemical oxygen demand, $mgO_2\ L^{-1}$
EC – electrical conductivity, $mS\ cm^{-1}$
VSS – volatile suspended solids, $g\ L^{-1}$
TVA - total volatile acids, $mg\ L^{-1}$
SMA – specific methanogenic activity, $g\ CH_4\text{-}COD\ g^{-1}\ VSS\ d^{-1}$
TMAH – tetramethylammonium hydroxide
DMSO – dimethyl sulfoxide
WW - wastewater
MWTP – municipal wastewater treatment plant
SW – simulated wastewater

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