Waste and Biomass Valorization

A mini review of the techno-environmental sustainability of biological processes for the treatment of high organic content industrial wastewater streams --Manuscript Draft--

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| Abstract: | Industrial wastewater contains complex and slowly biodegradable compounds often ineffectively treated by conventional activated sludge (CAS) systems. Alternatively, advanced anaerobic technologies are implemented. The current study reviews different potential anaerobic schemes, factors influencing their final performance and optimum combinations of operational/design parameters. Anaerobic membrane bioreactors, upflow anaerobic sludge blanket reactors, expanded granular sludge beds, anaerobic hybrid reactors and inverse fluidized bed reactors are discussed. Their major advantages include: low energy requirements, energy recovery through biogas generation and high organic load removal. pH~7, operation in a mesophilic environment and a hydraulic retention time long enough to enable anaerobic digestion in economically accepted reactor volumes are conditions that optimize the performance of anaerobic configurations. The evaluation additionally considers environmental aspects. The life cycle assessment of anaerobic industrial wastewater treatment reveals its positive environmental effect in terms of greenhouse gases emissions. Methane (a greenhouse gas) primarily contained in the biogas, despite being produced during anaerobic digestion, is utilized for energy production (heating, electricity) instead of being emitted to the atmosphere. Finally, anaerobic wastewater treatment is analyzed as part of the European Commission Innovation Deal that aims at converting conventional wastewater treatment plants to water reuse. | | | | | |
| Response to Reviewers: | Reviewer #1: The topic and the results of the current study are generally very | | | | | |
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interesting and satisfactorily explained. I recommend the publication after the following minor revisions:

1.'An example for that is the lab-scale study of Jiang et al. [22] who implemented a SBR for the treatment of aniline-rich industry wastewater and observed a COD removal of 95.80%.': Could the authors briefly present 1-2 more examples?

Following the reviewer's comment, we included the following extra examples in section 2 of the revised manuscript: "Similarly, Rajab et al. [81] achieved an average total COD removal of 97±2% in a lab-scale integrated anaerobic/aerobic SBR treating poultry slaughterhouse wastewater. COD removal higher than 95% was obtained by Xiao et al. [82] for the treatment of wastewater generated from silicon solar cell manufacture containing isopropyl alcohol operating a lab-scale achieve SBR. Anaerobic treatment is another process that has been widely used for industrial influents treatment [5]." 2.'In a suspended-growth configuration, a greater in a suspended-growth configuration, a greater number of microorganisms can be retained compared to the attached-growth systems; therefore, a smaller tank volume is required [25].': Which means that the

operational cost is reduced? Please, briefly comment on that.

Following the reviewer's comment, we revised and rewrote this part of section 2 of the revised manuscript as follows: "The main advantages of attached-growth over suspended-growth configurations include lower energy requirements, absence of sludge bulking problems and bigger resistance to system shocks [24-25]."

3.'They optimized the operation by investigating in advance the response of the active UASB microbial community to different OLRs [57].': Please, clarify the 'in advance'. Did they do any preliminary experiments?

Following the reviewer's comment, we clarified this point in section 3.1 of the revised manuscript by adding the following: "Microbial network analysis enabled monitoring the response of the active UASB microbial community to different OLRs and, thus, optimizing the process operation."

4.'Bialek et al. [10] applied psychrophilic conditions in order to investigate the efficiency of the anaerobic treatment in northern countries where the yearly average temperature is below 15°C.': Please, make a small comment on the efficiency of the process under the specific conditions of the study.

Following the reviewer's comment, we clarified this point in section 3.4 of the revised manuscript by adding the following: "At 10°C, the system presented a low average removal efficiency (~69%) and unstable operation with hydrolysis being the rate-limiting step. Biofilm overgrowth resulted in a decrease in the population of hydrogenotrophic methanogens; the latter limited methane production."

Reviewer #2: The main goal of the paper "Benchmarking of low environmental footprint biological processes for the treatment of industrial waste streams" is to analyze different potential anaerobic technologies available to date for the treatment of industrial wastewater. To this aim, not only the technical parameters but also the environmental performance of the different configurations are compared each other in order to find the optimal alternative. It is reflected the work that has been performed in the preparation of this manuscript. However, I consider that there are some minor aspects that should be improved before its publication, which are described below: 1. Title. As mentioned, the authors aim to review the potential alternatives for the management of industrial wastewater based on anaerobic technologies. However, the title of the manuscript may lead to confusion, since readers could interpret that primary data from own configurations will be evaluated and compared with related studies available in literature. In this sense, I encourage authors to modify the title of the manuscript to make clear that a review process is reported in this study focusing on the techno-environmental sustainability of different technologies for the treatment of wastewater from industrial framework. A proposal: "A review of the technoenvironmental sustainability of biological processes for the treatment of industrial waste streams".

Following the reviewer's comment, we rephrased the title of the revised manuscript as follows: "A mini review of the techno-environmental sustainability of biological processes for the treatment of high organic content industrial wastewater streams". 2.Keywords. In my opinion, some of the keywords could be improved. For example, since methane is the main component of biogas, which is used for energy production, I would change "methane" by "methane valorization", "biogas generation" or "energy production". In my opinion, these terms make more sense in accordance with the purpose of the paper. On the other hand, the authors propose two keywords that are

redundant: "advanced anaerobic processes" and "anaerobic digestion". From my perspective, the latter could be removed from the list.

Following the reviewer's comment as well as in an effort to better describe the content of the revised manuscript, we revised the keywords as follows: "Industrial Wastewater, Advanced Anaerobic Technologies, Biogas Production, Life Cycle Assessment, Circular Economy".

3.Introduction. In the last lines of the Introduction section, the authors explain that they will also discuss "the integration of an anaerobic step in systems including a sequence of different processes for industrial wastewater treatment". However, it is a bit difficult to identify in which part of the manuscript this task is carried out. I assume that the authors make reference to the last part of the Environmental Assessment section where they conclude that "the implementation of integrated systems combining multiple steps is a significantly sustainable option for industrial wastewater treatment". However, this issue is not entirely clear. I would suggest to rewrite this part of the Introduction to better clarify that the biological processes previously analyzed will be also evaluated in combination with other treatment technologies which are not necessarily anaerobic or CAS-type.

Following the reviewer's comment as well as in an effort to better describe the content of the revised manuscript, we revised this part of the introduction as follows: "Moreover, life cycle assessment (LCA) of anaerobic industrial wastewater treatment was presented as a method for holistically evaluating the environmental impact resulting from the application of such technologies. Finally, the analysis considered the contribution of anaerobic wastewater treatment technologies to the concept of circular economy by considering the European Commission Innovation Deal that is based on combining sustainable anaerobic membrane wastewater treatment and water reuse." 4.Environmental assessment. In line with the above, if this clarification is described in the Introduction, the last paragraph of the Environmental assessment section focused on other technologies apart from biological alternatives acquire force and sense. By contrast, it seems that the last paragraph based on physicochemical processes are outside the boundaries of the study.

Following the reviewer's comment as well as in an effort to better describe the content of the revised manuscript, we revised this part of section 4 as follows: "The way in which the operating strategy and design affect the environmental performance of anaerobic technology is still relatively unclear [80]. In this section though, it has been indicated that anaerobic reactors can have a positive environmental assessment especially in terms of GHG emissions. In any case, anaerobic wastewater treatment should be designed considering both economic (e.g. construction, operation, maintenance costs, etc.) and environmental aspects (e.g. eutrophication, GHG emissions, marine ecotoxicity, etc.) to achieve a positive environmental performance. Marine and freshwater eutrophication can only be decreased if the anaerobic treatment is coupled with suitable anaerobic/anoxic post-treatment."

5.English. The English style and grammar are adequate. I only would suggest a revision of the manuscript in order to correct some typographic mistakes. Following the reviewer's comment, we went through the whole manuscript to correct any typos.

6.References. In general, the manuscript is adequately documented. However, any reference from Waste and Biomass Valorization can be found in the text, so that I suggest performing a review on similar articles published in this journal to be included in the references list. Moreover, more attention should be paid on the criteria used in the reference list, since the names of the journals appear indistinctly in either complete or abbreviated format, as well as both upper and lower case. This is the case of references 2 and 12, as an example. Please, review to standardize the format in all cases.

Following the reviewer's comment, we went through the reference list to make sure that the referencing style is homogeneous and in accordance with the journal's guidelines. We also added references from the journal.

Reviewer #3: Well presented paper offering a review of different anaerobic streams and at the same time considering environmental impacts via LCA. It's well-structured and provides a good analysis and evaluation of the available options

taking into account the main parameters in question.

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Title:

A mini review of the techno-environmental sustainability of biological processes for the treatment of high organic content industrial wastewater streams

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Abstract Industrial wastewater contains complex and slowly biodegradable compounds often ineffectively treated by conventional activated sludge (CAS) systems. Alternatively, advanced anaerobic technologies are implemented. The current study reviews different potential anaerobic schemes, factors influencing their final performance and optimum combinations of operational/design parameters. Anaerobic membrane bioreactors, upflow anaerobic sludge blanket reactors, expanded granular sludge beds, anaerobic hybrid reactors and inverse fluidized bed reactors are discussed. Their major advantages include: low energy requirements, energy recovery through biogas generation and high organic load removal. pH~7, operation in a mesophilic environment and a hydraulic retention time long enough to enable anaerobic digestion in economically accepted reactor volumes are conditions that optimize the performance of anaerobic industrial wastewater treatment reveals its positive environmental effect in terms of greenhouse gases emissions. Methane (a greenhouse gas) primarily contained in the biogas, despite being produced during anaerobic digestion, is utilized for energy production (heating, electricity) instead of being emitted to the atmosphere. Finally, anaerobic wastewater treatment is analyzed as part of the European Commission Innovation Deal that aims at converting conventional wastewater treatment plants to water resource recovery facilities able to combine sustainable wastewater treatment and water reuse.

Keywords Industrial Wastewater, Advanced Anaerobic Technologies, Biogas Production, Life Cycle Assessment, Circular Economy

Nomenclature

| Nomenclature | |
|--------------|-------------------------------|
| ABR | Anaerobic Baffled Reactor |
| AH | Anaerobic Hybrid |
| AnMBR | Anaerobic Membrane Bioreactor |
| AS | Activated Sludge |
| CAS | Conventional Activated Sludge |
| COD | Chemical Oxygen Demand |
| d | Days |
| h | Hours |
| EGSB | Expanded Granular Sludge Bed |
| GHG | Greenhouse gas |

| HRT | Hydraulic Retention Time |
|------|---|
| IFBR | Inverse Fluidized Bed Reactor |
| LCA | Life Cycle Assessment |
| OLR | Organic Loading Rate |
| SBR | Sequencing Batch Reactor |
| SRT | Sludge Retention Time |
| UASB | Upflow Anaerobic Sludge Blanket Reactor |

1. Introduction

Wastewater originating from industrial activities contains complex and slowly biodegradable organic compounds which are not easy to treat [1]. Thus, appropriate treatment of industrial wastewater is important in order to avoid phenomena, such as eutrophication of surface waters, hypoxia and algal bloom, which cause pollution of the scarce clean water resources [2-5]. The design of industrial wastewater treatment is challenging due to various factors that are related to the characteristics of industrial streams, such as high chemical oxygen demand (COD) load, different pH values depending on the wastewater origin and salinity levels [6-7]. Anaerobic treatment has been implemented for various industrial influents (e.g. aqueous extractions of winery wastes, biodiesel industry wastewater, soluble fraction of food industry wastes etc.) by the use of configurations such as the anaerobic membrane bioreactors (AnMBRs), the upflow anaerobic sludge blanket reactors (UASB), the expanded granular sludge bed reactors (EGSB), the anaerobic hybrid (AH) reactor, the inverse fluidized bed reactor (IFBR). Moreover, this process offers the potential to produce biogas, which is afterwards utilized for electricity and energy production [4-5, 7-11, 77-79]. With a view to achieving a sustainable performance, wastewater treatment plants are expected to: (i) produce high-quality effluents satisfying the increasingly strict discharge legislation, (ii) expand their wastewater reuse and energy recovery potential in accordance with the concept of circular economy, (iii) have the capacity for upgrading and retrofitting energy-efficient and cost-effective technologies, (iv) decrease the investment costs and, generally, (v) have a low overall environmental impact [2-3, 9, 12-13]. In terms of industrial wastewater treatment, the implementation of anaerobic technology (e.g. AnMBR, UASB etc.) increases the system's efficiency, so that it can meet the standards for the treated effluent reuse or discharge. Nevertheless, the latter does not guarantee the attainment of the desired low environmental footprint since the total energy requirements are not always outweighed by the biogas production [14-16]. The decision-making upon the most appropriate process/configuration depends on several parameters including the specific origin of each wastewater stream. This is due to the fact that several operational parameters (e.g. addition of chemicals, energy requirements etc.) are selected upon the influent origin; thus, it is important to make the most sustainable choice

[17-18]. In this study, the emphasis is put on the use of anaerobic configurations for the treatment of industrial wastewater streams. Our goal was firstly to investigate how this is correlated with factors such as COD removal, organic loading rate (OLR), pH, temperature, hydraulic retention time (HRT) etc. and, secondly, how these are optimally combined towards a sustainable performance. Biogas production was also used as performance indicator of the examined anaerobic processes. Moreover, life cycle assessment (LCA) of anaerobic industrial wastewater treatment was presented as a method for holistically evaluating the environmental impact resulting from the application of such technologies. Finally, the analysis considered the contribution of anaerobic wastewater treatment technologies to the concept of circular economy by considering the European Commission Innovation Deal that is based on combining sustainable anaerobic membrane wastewater treatment and water reuse.

2. Technologies for the Biological Treatment of Industrial Wastewater

This section is dedicated to technologies for the industrial wastewater treatment and reuse; the emphasis is put on schemes, which stand as an alternative to the CAS systems. The activated sludge process (AS), although widely applied, requires the use of chemicals and involves high capital, operational and maintenance costs [5]. The sequencing batch reactors (SBRs) are used for municipal, as well as, industrial wastewater treatment as a more flexible version of the CAS systems. They operate under a sequence of phases (filling, reaction, settling and decantation) within a single tank, which functions both as an equalization tank and as a clarifier. In terms of industrial wastewater treatment, SBRs can be implemented and produce effluents respecting the discharge limits [19-21]. Jiang et al. [22] applied a SBR at lab scale for the treatment of aniline-rich industry wastewater and observed a COD removal of 95.8%. Similarly, Rajab et al. [81] achieved an average total COD removal of 97±2% in a lab-scale integrated anaerobic/aerobic SBR treating poultry slaughterhouse wastewater. COD removal higher than 95% was obtained by Xiao et al. [82] for the treatment of wastewater generated from silicon solar cell manufacture containing isopropyl alcohol operating a lab-scale achieve SBR. Anaerobic treatment is another process that has been widely used for industrial influents treatment [5]. In this process, anaerobic microorganisms convert organic material into usable energy (in the form of biogas) and an amount of biosolids [23]. It occurs either through attached-growth or suspended-growth processes. The main advantages of attached-growth over suspended-growth configurations include lower energy requirements, absence of sludge bulking problems and bigger resistance to system shocks [24-25]. The main configurations for suspended-growth anaerobic wastewater treatment include: the anaerobic membrane bioreactor (AnMBR), the upflow anaerobic sludge blanket reactor

(UASB), the expanded granular sludge bed (EGSB), the anaerobic hybrid (AH) reactor, the inverse fluidized bed reactor (IFBR) [26-31].

The AnMBR couples the anaerobic suspended-growth bacteria biological process with membranes for solid-liquid separation, thus allowing biomass immobilization. In some configurations, the membrane is placed on the side stream (external, cross-flow configuration) and a recirculation pump provides the required transmembrane pressure within the membrane module. Thus, the cross-flow velocity constantly interrupts the development of a filtration cake onto the membrane. Alternatively, the membrane is submerged either directly in the AnMBR or in a separate chamber. These two configurations require no recirculation pump which reduces the energy consumption and microorganism stress because of lower shear forces due to the absence of the cross-flow effect. However, the anaerobic conditions are less favorable for filtration and more prone to fouling; the latter restricts the full-scale adoption of the process since significantly lower permeate fluxes can be sustained compared to the aerobic MBRs [7, 32]. Nevertheless, successful full-scale AnMBR implementations for the treatment of industrial wastewater exist. The first full-scale AnMBR was installed in North America for the treatment of food industry wastewater with a design influent COD of 39,000 mg L⁻¹. The average effluent COD was constantly very low (<210 mg L⁻¹). Furthermore, the operating expenses were gradually reduced because the system was capable of progressively developing higher mixed liquor concentrations; thus, there was less need to dewater and dispose the solids [33]. In smaller scale, Van Zyl et al. [34] found a COD removal of 96.8% in a lab-scale AnMBR treating coal industrial wastewater (influent COD: 18,000 mg L⁻¹) and Zayen et al. [35] COD removal of 90.7% in a pilotscale AnMBR for landfill wastewater (feed solution COD increasing from 15,000 to 30,000 and, finally, to 41,000 mg L⁻¹). Successful commercial AnMBR applications have also been developed. For instance, Memthane-type AnMBRs have been engineered to produce high-quality effluent through the implementation of ultrafiltration membranes. This system has been successfully applied for the treatment of various industrial streams of high organic strength. Specifically, Memthane technology enabled 95% COD removal from the wastewater of one of the largest dairy manufactures in South Africa (Woodlands Dairy; influent COD: 10,000 mg L⁻¹). Furthermore, 99% COD removal became possible for the Paulaner brewery wastewater (Munich, Germany; influent COD: $8,393 \text{ mg } \text{L}^{-1}$) by the use of this system [84].

The major drivers for the wider adoption of the AnMBR process include: low energy requirements, energy recovery in the form of methane, capacity for removing high organic loads and low sludge production. On the other hand, the main barriers for the extensive AnMBR application are related to the operational cost for the membrane cleaning and replacement due to fouling which can occur by both organic material as well as inorganic

precipitations (e.g. calcium, nitrogen, phosphorus, magnesium, struvite). Other disadvantages are the energy required for the gas recirculation, the need for nutrient supplementation, the slow growth of the microorganisms involved and the dissolved methane escape in the treated effluent [14, 16, 26, 36-38, 93-95]. The benefits from biogas recovery can counterbalance the operating cost. One example is the world's largest chocolate factory (Mars factory, Veghel, Netherlands; influent COD: 10,000 mg L⁻¹) where wastewater is treated through a Memthanetype AnMBR that achieves almost complete COD removal (99%). The system can provide 1,000,000 m³ biogas for home boilers and cover 10% of the plant's total energy requirements [83-84]. Moreover, the energy requirements of a semi-industrial AnMBR plant treating wastewater with high sulfate concentration (105 mg SO₄-S L⁻¹) were minimized to 0.07 kWh m⁻³ at ambient temperature (17-33°C) after the sludge retention time (SRT) optimization [39]. Pretel et al. [39] applied the AnMBR technology for the treatment of high-sulfate influents in warm/hot climates resulting in energy production up to 0.11 kWh m⁻³. At temperatures higher than 25° C, labscale AnMBRs have been successfully implemented not only for the treatment of low-strength [14, 87-88] but also for high-strength wastewater [85-86] achieving more than 80% COD removal. Satisfying AnMBR performance under psychrophilic conditions (<20° C) with approximately 90% COD removal has been observed for pilot-scale low-strength wastewater treatment [89-90]; the latter supporting the conclusions of past review papers according to which anaerobic low-strength wastewater treatment can be efficiently performed at low temperatures if long SRTs are applied to ensure adequate solids degradation [91-92]. However, proof of successful operation for high-strength wastewater at temperatures around 25° C is required to consider the AnMBR as a significantly cost-effective industrial wastewater treatment technology. Finally, the integration of an AnMBR step within a broader treatment system is frequently observed because it is unsure whether the AnMBR technology per se can always meet the strict discharge limits imposed by the current legislative framework. Thus, the AnMBR wide implementation and market penetration are still hindered. In this frame, sustainable wastewater treatment using innovative AnMBR technology has been chosen as one of the two Innovation Deals of the European Commission. The goal is to overcome legislative obstacles and promote the shift from conventional wastewater treatment plants to resource recovery facilities. Following the concept of circular economy, end users are not regarded as simple buyers but as active contributors to a sustainable wastewater treatment that ensures full use of the wastewater value [96-99].

In the UASB process, the sludge granules grow in a tubular reactor [40]. It is applied for anaerobic treatment of domestic wastewater mainly in warm climates; high temperatures offer the appropriate conditions for anaerobic degradation. The latter along with simple operation, limited land requirements and the ability to

treat at high rate (i.e. at low HRTs) justify the wide UASB application in developing tropical countries [41-43]. The UASB technology has also been widely used for high-strength wastewater achieving stable operation due to high anaerobic reaction rates combined with limited sensitivity to fluctuating parameters (e.g., acidity, HRT) [100]. For example, Djalma Nunes Ferraz Junior et al. [44] developed a lab-scale UASB for the treatment of wastewater coming from ethanol production achieving a COD removal of 96.1 \pm 1.7% (influent COD=35,200 mg L⁻¹). High organic matter removal efficiencies (COD removal=94.7%). were obtained with the application of a lab-scale UASB by Sivakumar and Sekaran [45] treating dairy wastewater (influent COD=3,456 mg L⁻¹). However, there are also disadvantages related to the UASB operation. For instance, external additives (e.g. natural polymer, aluminium chloride, powdered bamboo-charcoal etc.) are likely to be needed to enhance sludge granulation and, subsequently, ensure high biomass retention times during high-strength wastewater treatment [100-102]. Moreover, the different kinetics between hydrolysis and methanogenesis can require changes in the reactor design and operation at two discrete stages [103-104]. More importantly, the UASB effluent often fails to comply with strict discharge standards and demands post-treatment through alternative technologies (e.g. SBRs, membrane bioreactors etc.), to remove nutrients and/or increase COD and total suspended solids removal [105-106].

EGSB is a modified UASB version, developed to attain higher upflow velocities and accommodate more variable loading rates under a lower footprint [46, 48]. The higher upflow velocity increases the granular sludge bed fluidization, which, subsequently, improves the contact between wastewater and sludge [47]. Full-scale EGSB applications with significant COD removal have been noted. Petropoulos et al. [48] treated winery wastewater with a lab-scale EGSB (COD=1,256 mg L⁻¹) and achieved COD removal~96% at 37° C. Ince et al. [107] observed an average COD removal of $86\pm8.2\%$ during the treatment of maize processing wastewater. In addition, Warmenhoven and Spanjers [108] achieved 82% COD removal while treating fruit juice packaging factory wastewater. AH is an efficient process that combines anaerobic filtration with the UASB process, achieving stable and economic operation through high SRT but low HRT [49]. It has been effectively applied for industrial wastewaters, such as wine industry wastewater (e.g. lab-scale study by Wahab et al. [49]: influent COD ranging from 500 to 24,000 mg L⁻¹; COD removal=94%) and brewery wastewater (e.g. lab-scale study by Li et al. [50]: influent COD=108,900-136,700 mg L⁻¹; COD removal=92%), as well as for leachate treatment (e.g. full-scale study by Mokhtarani et al. [109]: influent COD=81,000 mg L⁻¹; COD removal=91%) [29, 51]. Fluidized beds have been placed within reactors in order to achieve shorter HRTs than the respective ones in the UASB systems. IFBRs can treat higher wastewater volumes in less space, since they provide an increased specific surface area for

the biomass; thus, shorter HRTs can be applied. The floatable particles have a lower specific density than the liquid; thus, they are fluidized downwards. The produced biogas flows in the opposite direction than the liquid and this enhances the bed expansion. Thus, the fluidization velocities in this inverse system are lower than in the upflow ones, which leads to lower energy consumption [10, 27, 52]. Under lab-scale IFBR implementation, COD removal efficiency higher than 90% was observed in the studies by Arnaiz et al. [52] for dairy wastewater (influent COD=30,000 mg L⁻¹) and Alvarado-Lassman et al. [53] for brewery wastewater (influent COD=2,083 mg L⁻¹).

The wastewater treatment sector can majorly contribute to a quicker swift towards circular economy via the wide implementation of breakthrough technologies (e.g. anaerobic reactors) that minimize energy consumption but ensure resource recovery [61]. Suspended-growth systems for anaerobic treatment (AnMBR, UASB, EGSB, AH, IFBR etc.) have been effectively applied for the treatment of industrial influents resulting in high COD removal and energy recovery. The crucial point is to carefully select the design parameters and ensure their optimal combination so that the energy production through anaerobic treatment outweighs any potential cost related to oversizing and membrane cleaning.

3. Factors Affecting the Performance of Anaerobic Technologies in Industrial Wastewater Treatment

This section discusses the main factors that affect the performance of the examined anaerobic technologies for the treatment of high-strength industrial wastewater. Table 1 provides an overview of existing studies on the treatment of industrial streams with the implementation of the technologies discussed in section 2. The goal is to identify how target parameters (e.g. OLR, pH, temperature, HRT) influence the performance of the system in terms of contaminants removal.

| Reference | Scale/ Configuration | Industrial Wastewate r Source | Temp. (°C) | рН | HRT or Cycle duration | OLR (kg COD m ⁻³ d ⁻ ¹) | Influent COD (mg L ⁻¹) | COD Removal | Biogas production or other observation |
|-----------|--|-------------------------------------|---------------|---------|--|--|---------------------------------------|--|--|
| [30] | Lab-scale External- submerged AnMBR | Bamboo industry | 28-30 | 7.4-8 | HRTs (d): • 2 (OLR=11kg COD m ⁻³) • 5 (OLR=4.4 kg COD m ⁻³) • 10 (OLR=2.2 kg COD m ⁻³) | 11 (HRT=2d) 4.4 (HRT=5d) 2.2 (HRT=10d) | 22,000 | HRT from 5d to 10d: from 91% to 93% HRT from 10d to 2d: from 93% to 80% | HRT≥5d: membrane fouling effectively controlled |
| [55] | Lab-scale External- Crossflow UF AnMBR | Distillery | 53-55 | 7.5-8.5 | HRT=15d | 2.06 | 22,600 | 97% | Biogas production rate steadily increased & stabilized at≈2.8L d⁻¹ Methane content of biogas≈ 55% |
| [54] | Pilot-scale External- Crossflow UF AnMBR | Brewery | 36±1 | 6.9-7.2 | HRT=2.5-4.2d | 28.5 | 80,000- 90,000 | 97% | Methane content of biogas decreased from 80% to 65% towards the end of operation (probably due to microbial community changes caused by the high OLR) Methane yield: 0.28 m³ CH₄ (kg COD removed) ⁻¹ |
| [56] | Lab-scale UASB (hollow centered packed bed) | Palm oil mill | 55 | 6.8-8 | HRT=2d (for the optimized set of operating parameters) | 27.65 (for the optimized set of operating parameters) | 32,580±9,500 | 91.8% (for the optimized set of operating parameters) | Methane content of biogas≈ 60% Max COD removal (=97.5%) at OLR=6.66 kg COD m⁻³d⁻¹, HRT=5d & biogas with 65.6% of methane |
| [57] | Pilot-scale UASB | Sugar- processing | 35 | 6-7 | HRT=1d | 13.8 | 128,400 | 87%-95% | • Methane content=68.5% • at OLR=13.8 kg COD m ⁻³ d ⁻¹ |

Table 1 Effect of target parameters on the efficiency of alternative anaerobic systems for the treatment of various industrial streams.

| [58] | Lab-scale EGSB | Brewery | 20 & 15 | 6.8-7.2 | HRT=18h | • 20°C: 9.7 • 15°C: 5.5 | • 20°C: 7,300 • 15°C: 5,200 | • 20°C: >85% • 15°C: 73% | From 20 to 15°C: proportion of <i>Methanosaeta</i> (acetate-utilizing methanogens) decreased from 60% to 49.3% Reactor performance strongly influenced by temperature under psychrophilic conditions |
|------|----------------------------|-------------------|------------|---------------|---|--|--------------------------------|--|---|
| [59] | Pilot-scale EGSB | Coal gasification | 35 | 7-7.5 | HRT=4d | 0.63 | 2,340-2500 | 65% | • Methane production rate= 227.23 mL CH ₄ L ⁻¹ d ⁻¹ at OLR=0.63 kg COD m ⁻³ d ⁻¹ |
| [20] | Lab-scale Anaerobic SBR | Palm oil mill | 26-30 | 8.33- 9.14 | Cycle duration=22h (fill, react, settle, decant) | 1.8-4.2 | 13,950-17,050 | 95.1%-95.7% | Anaerobically digested palm oil mill effluent requiring aerobic SBR post-treatment to meet discharge limits |
| [29] | Lab-scale AH & SBR | Fruit-juice | 26 | 7.5 | Single-stage AH: HRT=10.2h Two AH reactors: HRT=20h Two-stage AH followed by SBR: HRT=31h | Single-stage AH: 11.8 Two AH reactors: 5.9 Two-stage AH followed by SBR: 5.3 | 4,980±1,706 | Single-stage AH: 42% Two AH reactors: 67.4% Two-stage AH followed by SBR: 99% | • Integrated system of two-stage AH reactors followed by SBR producing effluent appropriate for reuse in agriculture |
| [27] | Lab-scale IFBR | Pulp and Paper | 36±1 | 7.5 | Continuous operation: HRT=3.5h Batch operation: HRT=8h | 20 | 1,000-8,000 | Continuous operation: 81% Batch operation: 92% (with the progression of batch cycles) | Continuous operation: 0.237 L CH₄ (g COD)⁻¹ Batch operation: 0.283 L CH₄ (g COD)⁻¹ (with the progression of batch cycles) |
| [10] | Lab-scale IFBR | Dairy | 10 | 6.8-7.2 | HRT=2d | 0.5 | 1,000 | 69%±10% | Methane production: 0.241 L CH₄ d⁻¹ Poor mixing in the reactor provoking poor hydrolysis of the substrate and, thus, low COD removal |

3.1 COD removal

The origin of the industrial wastewater stream plays an important role in the efficiency of the examined anaerobic processes in terms of COD removal and energy recovery [4]. Speece [60] underlined the effectiveness of anaerobic wastewater treatment for industrial wastewaters, noting, however, that the degradability rate to methane can be variable; the latter is observed because the industrial influent composition can be detrimental to the methanogenic activity.

Table 1 summarizes the results of studies dealing with the treatment of different industrial wastewater streams characterized by different COD levels, all higher or equal to 1,000 mg L⁻¹. The COD concentration varied from 1,000 mg L⁻¹ [10] (dairy wastewater treated in a lab-scale IFBR) to 80,000-90,000 mg L⁻¹ [54] (brewery wastewater treated in a pilot-scale AnMBR) and 128,400 mg L^{-1} [57] (sugar-processing wastewater treated in a pilot-scale UASB). The results of the studies reported in Table 1 revealed that the application of suitable anaerobic processes can lead to high COD removal efficiencies and high methane production. Anderson et al. [54] and Kim et al. [57] observed that COD was removed by 97% and 87%-95%, respectively. On the contrary, COD removal was only $69\pm10\%$ in the case of the significantly lower influent concentration in the study by Bialek et al. [10]; the cause was insufficiently intense mixing inducing low substrate hydrolysis. After ensuring stable physical conditions (heating, mixing etc.), Anderson et al. [54] noted that the increase of the mixed liquor volatile suspended solids (8 to 50 kg m⁻³) had no negative effect on the COD removal. Nevertheless, the methanogenic bacteria activity was negatively affected leading to the decrease of the biogas methane content from 80% to 65%. Kim et al. [57] positively correlated the composition of the active bacterial and archaeal communities (84% of Lactococcus and 80% of Methanosaeta, respectively) with the methane production and the OLR (4.01 L CH₄ at 13.8 kg COD m⁻³ d⁻¹). Microbial network analysis enabled monitoring the response of the active UASB microbial community to different OLRs and, thus, optimizing the process operation.

Due to location-specific nutrient removal limitations and effluent quality concerns, more advanced treatment is often required [110]. COD removal (Table 1) was higher than 80% for most of the examined processes and initial COD loads. The high COD removal reported in Table 1 gives a preliminary indication of the efficiency of anaerobic treatment either per se or in combination with CAS technologies, as well as its potential to serve the circular economy concept via the simultaneous attainment of nutrient recycling and energy recovery [96]. Anaerobic treatment in combination with CAS process was applied at pilot-scale in the study by Wu et al. [68] where a three-stage system of a catalytic-ceramic-filter anaerobic reactor followed by a UASB and, by an AS reactor at the last stage achieved 98% COD removal while treating monensin production wastewater. Optimized

operation ensured satisfying methanogenic activity, anaerobic treatment (with or without the use of pre/posttreatment depending on the location-specific desired nutrient removal and effluent quality) can be a sustainable individual/integrated treatment option for industrial effluents characterized by high levels of organic content.

3.2 OLR

In this section, the attention is drawn on the effect that the OLR has on the anaerobic scheme performance. In Table 1, lower OLRs coincided with higher COD removal at a given temperature. The latter was demonstrated in the study by Wang et al. [30] (lab-scale AnMBR treating bamboo industrial wastewater: 80% of COD removal at OLR=11 kg COD m⁻³d⁻¹; 93% COD removal at OLR=2.2 kg COD m⁻³d⁻¹), as well as in the work by Poh and Chong [56] (lab-scale UASB for the treatment of palm oil mill wastewater: 91.8% COD removal at OLR=27.65 kg COD m⁻³d⁻¹; 97.5% COD removal at OLR=6.66 kg COD m⁻³d⁻¹). Although higher OLRs accelerate granulation, they disturb the balance between acidogenic and methanogenic populations causing poor reactor performance [56, 62]. Thus, it is essential to test different OLRs for a given temperature and apply the optimal one, which will not jeopardize the biomass activity.

3.3 pH

Anaerobic digestion strongly depends on the pH [63]. pH higher than 9 in a system applying an anaerobic process coupled with membranes has been reported to result in less biogas production and poor membrane performance, since anaerobic treatment takes place at a pH range of 6.5-8.5, with the optimum pH range being between 7 and 8 [16, 64-66]. The optimal pH for the methanogenic bacteria is 6.8-7.2 (i.e. around 7); if it drops below 7, the acidogenic bacteria prevail over the methanogens. As a consequence, acid zones are formed inside the reactors and methane production is reduced [67]. Moreover, pH shocks lead to dispersion of the sludge flocs. Small-sized particles (e.g. colloids) exist in suspended sludge and provoke increased fouling when AnMBRs are implemented [65]. The pH is maintained around 7 in most of the studies listed in Table 1. However, pH stabilization requires the addition of chemicals, especially in the case of industrial streams characterized by low pH [16]. The need for pH neutralization increases the overall operational cost, as well as the environmental footprint of the applied process. Thus, the use of chemicals for the adjustment of the influent pH at ~7 requires optimization to reduce the environmental impact and cost of an anaerobic process.

3.4 Temperature

Higher temperatures (30-55°C) are favorable for methane production, but disadvantageous in terms of energy requirements. However, temperatures higher than 60°C are detrimental to biomass. Thus, in the case of industrial wastewater influents which have high temperatures (e.g. 90°C), pre-cooling is needed for mesophilic (33-42°C)/thermophilic (50-60°C) anaerobic treatment [16, 23, 47]. Anaerobic treatment under mesophilic conditions has moderate energy requirements of an industrial influent and results in satisfactory biogas production. Most of the studies included in Table 1 apply the anaerobic process in mesophilic environments. The effect of low temperature in the process performance has been investigated in several studies [10, 58]. Bialek et al. [10] applied psychrophilic conditions in a lab-scale IFBR for dairy wastewater treatment in order to investigate the efficiency of anaerobic treatment in northern countries where the yearly average temperature is below 15°C. At 10°C, the system presented a low average removal efficiency (~69%) and unstable operation with hydrolysis being the ratelimiting step. Biofilm overgrowth resulted in a decrease in the population of hydrogenotrophic methanogens; the latter limited methane production. Xing et al. [58] examined the operation of a lab-scale EGSB at 20°C and 15°C for the treatment of brewery wastewater. The proportion of *Methanosaeta* (acetate-utilizing methanogens) decreased from 60% to 49.3% when the temperature dropped from 20°C to 15°C, which subsequently resulted in decreased methane production. COD removal was also affected; at 20°C, COD removal exceeded 85% (for an influent COD of 7,300 mg L⁻¹), whereas at 15^oC COD removal was 73% for a lower influent COD maintained at 5,200 mg L⁻¹. It was observed that relatively satisfactory COD removal at the lower -temperature became possible only after the reduction of the wastewater COD content. The application of mesophilic conditions in anaerobic treatment is recommended for the process stability; the latter being translated into the following: optimal conditions for the occurrence of anaerobic decomposition, sufficient COD removal and satisfying methane production.

3.5 HRT

Long HRTs are usually applied during anaerobic treatment of industrial effluents to ensure that the substrate hydrolysis and the methanogenesis are given enough time to occur [16]. This is in accordance with the study by Wang et al. [30] who observed a decrease of the COD removal from 93% to 80% with the decrease of HRT from 10 to 2 days. Tawfik and El-Kamah [29] achieved 99% COD removal operating a lab-scale integrated system of a two-stage AH reactor followed by a SBR for the treatment of fruit-juice industry wastewater at a HRT of 1.3 days. The authors also examined shorter HRTs in non-integrated systems including single-stage AH operation

(HRT=10.2 hours; COD removal=42%), as well as two-stage AH operation (HRT=20 hours; COD removal=67.4%), but none of them enabled the production of a treated effluent appropriate for reuse in agriculture.

Shorter HRTs lead to a shorter contact time between the sludge and the substrate and, consequently, to a poorer system performance; a significant amount of biomass does not settle and substrate is washed out without appropriate treatment [30]. Over the last 30 years, anaerobic treatment has been efficiently applied in the domain of wastewater treatment [112]. Nevertheless, its wide application has been hindered by the difficulty in retaining the slowly growing anaerobic microorganisms when operating at short HRTs and low temperatures [111]. On the other hand, the need to decrease the overall cost and operate in smaller reactor volumes pushes towards the application of shorter HRTs [69]. AnMBR tanks in specific, that are characterized by a high concentration of suspended solids, are likely to face accumulation of soluble microbial components and increased membrane fouling because of a potentially insufficiently low HRT [30, 37]. The latter was one of the main drivers in the study by Wang et al. [30] who tested the effect of different HRTs (2, 5 and 10 days) on the membrane fouling in a lab-scale AnMBR treating a bamboo industry stream; a minimum HRT (\geq 5 days) was required for an effective control of membrane fouling. Thus, it is important to identify the optimal HRT for each configuration performing anaerobic industrial wastewater treatment. It should be the one combining adequate substrate degradation and cost optimization of the process in terms of reactor volumes.

3.6 Biogas production

One of the major advantages in anaerobic wastewater treatment is the recovery of biogas which usually has the following composition: 60-65% of methane (CH₄), 35-45% of carbon dioxide (CO₂) and 0-5% of nitrogen (N₂) [16]. Methane can be used in anaerobic digestion to produce energy. Carbon dioxide has multiple potential uses: cooling, acid replacement, calcium carbonate production (later utilized in gypsum or soda ash production), and carbon source for algae growth. Nitrogen ammonia can be stripped out from wastewater and then be utilized together with sulphuric acid to produce ammonium sulphate. Wastewater can constitute a source of energy and valuable nutrients through the shift from conventional to anaerobic wastewater treatment. Hence, anaerobic wastewater treatment plants can function as resource recovery facilities where any potential operating cost is outweighed by the biogas production [61, 96].

Biogas with more than 60% of methane is required before using it for digester heating, electricity generation and fuel production [16, 56]. Lower methane yields can be explained by the high methane solubility especially in low temperatures, such as 15°C [16, 70]; this justifies the decreased methanogenic activity in the

study by Xing et al. [58] (lab-scale EGSB treating brewery wastewater). Higher temperatures benefit methane generation [16]. In addition, pH can act as an inhibitory factor for biogas generation when it is below 6 or above 8.5 with the optimal pH for methanogens being around 7 [16, 67]. The majority of studies in Table 1 apply a pH around 7 in order to eliminate the effect of this parameter in biogas production. Poh and Chong [56] obtained optimum methane production efficiency and purity (biogas with 65.6% of methane) combined with a COD removal of 97.5% by applying OLR=6.66 kg COD m⁻³d⁻¹, HRT=5 days and thermophilic conditions (55°C) at pH~7. However, the application of higher OLR and lower HRT (OLR=27.65 kg COD m⁻³d⁻¹ and HRT=2 days) resulted in satisfactory biogas methane content (57.4%) and COD removal efficiency (91.8%) with smaller reactor volumes. Process optimization should take into consideration technical, cost and environmental indicators, thus allowing the application of a minimal HRT, the achievement of satisfying COD removal and, finally, a biogas production with sufficient methane content.

4. Environmental assessment

Life Cycle Assessment (LCA) is applied for the assessment of the environmental impact associated with a whole process/product/service by considering the environmental load of every single stage during the life cycle of the process/product/service under investigation (Baumann and Tillman, 2004; ISO 14040, 2006; Hospido et al., 2012). LCA has been widely applied in wastewater treatment (Larsen et al., 2007; Corominas et al., 2011; Hospido et al., 2012). However, the LCA of anaerobic processes for industrial wastewater treatment is limited to few studies.

The development of a holistic approach for the environmental impact assessment of full-scale anaerobic processes integrating LCA, qualitative indicators and impact categories is required. In the case of anaerobic industrial wastewater treatment, the LCA impact categories can include energy and resource requirements, sources of greenhouse gas (GHG) emissions, toxicological data, technical costs etc. [71-73]. Georgiopoulou et al. [74] conducted LCA to evaluate the potential environmental and economic impact of five different biological wastewater technologies (AS, high-rate and extended aeration, pre-denitrification, aerated lagoon and anaerobic treatment (UASB reactor)) for full-scale dairy wastewater treatment. Amongst all the alternative scenarios, the results showed that anaerobic treatment proved to be the most environmentally friendly process, resulting in less GHG emissions with the added value of biogas production. The latter is significant from a global warming potential perspective, since methane (GHG contained in the biogas) is utilized for energy production instead of being emitted to the atmosphere. The dissolved methane which escapes in the treated effluent is an issue of concern

since it is then released into the atmosphere. However, dissolved methane losses are much more critical in dilute sewage such as domestic/municipal wastewater rather than industrial wastewater [113]. Foley et al. [75] examined the environmental impact of three industrial wastewater treatment options (i.e. full-scale inventory data for highrate anaerobic treatment with biogas generation, pilot-scale inventory data for microbial fuel cell treatment with direct electricity generation and lab-scale inventory data for microbial electrolysis cell with hydrogen peroxide (H₂O₂) production) through a LCA. Negative environmental impacts were principally associated with electricity consumption and transportation/disposal of biosolids in all the examined options. Anaerobic treatment demonstrated a more environmentally friendly performance in terms of resource requirements and GHG generation, though. O'Connor et al. [76] assessed and compared the environmental performance of fourteen processes (constructed from six unit processes: dissolved air flotation, clarification, AS, UASB, ultrafiltration and reverse osmosis) for the full-scale treatment of pulp and paper effluent in terms of various impact categories (i.e. GHG emissions, water recovery, freshwater aquatic ecotoxicity, eutrophication discharge impact reduction). LCA indicated that AS pre-treatment in the UASB resulted in reduction of the freshwater aquatic ecotoxicity, eutrophication and GHG emissions impact categories.

The way in which the operating strategy and design affect the environmental performance of anaerobic technology is still relatively unclear [80]. In this section though, it has been indicated that anaerobic reactors can have a positive environmental assessment especially in terms of GHG emissions. In any case, anaerobic wastewater treatment should be designed considering both economic (e.g. construction, operation, maintenance costs, etc.) and environmental aspects (e.g. eutrophication, GHG emissions, marine ecotoxicity, etc.) to achieve a positive environmental performance. Marine and freshwater eutrophication can only be decreased if the anaerobic treatment is coupled with suitable anaerobic/anoxic post-treatment.

5. Anaerobic wastewater treatment within the concept of circular economy

Wastewater treatment based on the circular economy principles aims at resource recovery and water reuse, reducing energy requirements and chemical consumption as well as at decreasing the environmental impacts. Anaerobic technology for industrial wastewater treatment has the potential to serve the concept of circular economy and has been included in one of the two Innovation Deals of the European Commission. Its wide application can transform wastewater treatment plants into facilities which allow the recovery of water, energy, nutrients and other chemicals along with the reuse of reclaimed water in various sectors (e.g. agriculture, industry,

drinking water, etc.). Under such circumstances, the end-users of the anaerobically treated wastewater become actively engaged in the effort to shift from conventional wastewater treatment to resource recovery plants [96]. Based on this novel vision on wastewater treatment, wastewater treatment plants will move further from treatment of sewage and play multiple roles: resource factories, energy producers, used water refineries, water recycle and reuse facilities. For instance, part of the reused and nutrient-rich water can be used for irrigation/fertigation, thus reducing water and fertilizer costs in agriculture. It can also undergo further treatment to reach higher quality water for alternative uses such as aquifer recharge. Moreover, reclaimed water can be utilized in industries to cover the water needs for several purposes such as evaporative cooling, boiler feed, washing and mixing [61]. Following the Innovation Deal proposal, sustainable wastewater treatment combining anaerobic treatment with membrane technology and water reuse is the target. Nevertheless, actions are still needed to achieve this goal; e.g. review the legal barriers which often hinder and restrict and water reuse, promote the collaboration between entities and water management stakeholders, disseminate the results to the potential end-users and society.

6. Conclusions

In this mini review, anaerobic wastewater treatment (e.g. AnMBRs, UASB, EGSB, AH, IFBR configurations) was presented as an efficient way to treat industrial wastewater streams and produce treated effluents that can meet location-specific discharge limits. More importantly, the implementation of anaerobic technologies was analysed as a means to transform wastewater treatment plants into water reuse and energy recovery facilities in line with the concept of circular economy.

Parameters such as COD removal, OLR, pH, temperature and HRT were examined to see how they are related to the anaerobic configurations performance and how they can be optimally combined. Biogas production was additionally considered as an indicator of the anaerobic plants contribution to energy recovery and, hence, as a way to compensate for part of the operating costs. Moreover, LCA of anaerobic industrial wastewater treatment was presented as a method to examine potential environmental impacts.

Stable operating conditions, intense mixing and, consequently, sufficient substrate degradation are required to achieve satisfying COD removal. Furthermore, the application of an optimal OLR coupled with a suitable temperate is needed to maintain the balance between acidogenic and methanogenic populations without compromising the reactor performance. Moreover, optimal reactor performance calls for pH stabilization around 7, operation in a mesophilic environment and application of a minimal HRT to ensure satisfying substrate degradation and enhanced biogas production (with sufficient methane content) under economically acceptable reactor volumes. In that case, efficient treatment is achieved together with energy recovery without high operational/maintenance costs and significant negative environmental impacts. Especially in terms of GHG emissions as a LCA impact category, anaerobic technologies for industrial wastewater treatment can be positively assessed provided that the dissolved methane escaping with the treated effluent is limited; methane (GHG) contained in the biogas produced through anaerobic treated is converted to energy instead of being emitted to the atmosphere.

According to the principles of circular economy, wastewater can be regarded as a source of energy and nutrients. Anaerobic technology for industrial wastewater treatment has been integrated in the two Innovation Deals of the European Commission that focus on attaining sustainable wastewater treatment along with water reuse. In this frame, the old paradigm of conventional wastewater treatment plants engineered just to perform nutrient removal is expected to be replaced by advanced anaerobic treatment that allows the recovery of water, energy and nutrients, as well as the reuse of water in different domains (e.g. agriculture, industry, drinking water sector, etc.). Within the circular economy concept, end-users are no longer considered as simple consumers but as active participators in an effort to fully exploit wastewater. However, legal barriers concerning water reuse and market exploitation of recovered products, gaps in the cooperation between entities and water management stakeholders as well as limited dissemination still restrict the wide penetration of the anaerobic technology to the market.

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