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A Brief Analysis of the Efficiency of Biogas Purification Methods for a More Ecological Biomethane: A Review

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Abstract

The injection of biogas into the natural gas distribution network and/or its use as biofuel requires additional treatment. This treatment is commonly called purification and/or enrichment. The idea of biogas purification arises from the fact that, in its raw state, the gas obtained after anaerobic fermentation contains several impurities in varying proportions, such as (25% - 50%) carbon dioxide, traces of hydrogen sulphide (0.005% - 2%), oxygen (0% - 1%), nitrogen (0% - 2%), siloxanes (0% - 0.02%), ammonia (<1%) and halogenated hydrocarbons, (VOC < 0.6%) and water vapor fractions. Once this treatment has been carried out, biomethane (CH_4) is obtained which is approximately 99.99% pure. Thus, several technologies are used nowadays for a more rational use of biogas according to the fields of application. These biogas treatment processes differ from each other from a technological point of view but also in terms of efficiency. Therefore, we have set ourselves the goal of summarizing the existing purification methods in order to compare them in terms of efficiency, environmental friendliness, and the cost of maintaining the installations. Given that environmental considerations are central to our thinking, the use of technologies that employ natural filters such as microalgae seems to be the most appropriate approach if we are particularly concerned with preserving our ecosystem.

Keywords

Impurities, Purification, Biogas, Biomethane, Yield

1. Introduction

The biogas sector has been the subject of particular interest in recent decades in most countries of the world, aware of the major challenge of climate change caused by the use of fossil fuels. Biogases are the result of methanization, which is a natural process of biological degradation of organic matter in an oxygen-free environment due to the action of multiple microorganisms (bacteria). In fact, biogas production is the only area of biomass that makes it possible to create a link between four related areas of the modern economy: household waste management, animal husbandry, agriculture, and energy. In addition, biogas from the anaerobic digestion of organic matter constitutes a promising renewable energy vector for heat production and power in households and industry (Herrmann *et al.*, 2016; Marín *et al.*, 2019) [1] [2]. But this product in its raw state presents some impurities due to its composition (40% - 75% methane (CH_4), 25% - 50% carbon dioxide (CO_2), traces of hydrogen sulphide (0.005% - 2%), oxygen (0% - 1%), nitrogen (0% - 2%), siloxanes (0% - 0.02%), ammonia (<1%) and halogenated hydrocarbons (VOC < 0.6%) and water vapour fractions) (M.S. Shin *et al.*, 2019; D. Marín *et al.*, 2018; Allen *et al.*, 1997; Schweigkofler and Niessner, 1999; Rasi *et al.*, 2007; Chottier, 2011; Harasimowicz *et al.*, 2007; Abatzoglou and Boivin, 2009) [2]-[9].

Carbon dioxide (CO_2) is a gas that reduces the density and lowers the calorific value of biogas, but it is not toxic or corrosive like hydrogen sulfide (H_2S). The latter is harmful to the environment, human health, and is very corrosive to metal parts of motors, pumps, compressors, gas storage tanks, and valves, and reduces the service life of process equipment (ASABE, 1992; ASABE, 2010; MTO, 2009; MTO, 2011; Patni and Clarke, 2003) [10]-[14]. Biogas pollutants must be removed before any possible use. Hence, there is a need to purify the biogas to obtain biomethane, which is composed of CH_4 99%.

Basically, there are two steps involved in the biogas treatment process: cleaning (elimination of harmful and toxic compounds such as H_2S, N_2, O_2, Si, H, VOC, CO, and NH_3), and upgrading (adjustment of the CO_2 content to increase the calorific value of the biogas to an optimal level).

In order to overcome these impurities linked to the composition of the raw biogas, several treatment (purification) methods are used today in industrial and semi-industrial installations and in renowned laboratories. A purification system can take two forms, depending on the nature of the biogas and the elements to be eliminated or reduced:

- A global treatment technology (purification and enrichment) and/or a system

composed of different treatment stages (purification then enrichment). The purification technologies used are often specific to, or sensitive to, specific components. Among others, we can mention the following:

- Condensation, adsorption, and permeation for the removal of water and vapor.
- Permeation, catalytic deoxidation for the removal of oxygen.
- Upstream treatment in the digester (biological oxidation, physico-chemical desulphurization, etc.) and downstream treatment after the digester: activated carbon, biological washing, soda washing, permeation for the elimination of sulphur compounds, etc.
- Activated carbon and combination cooling/activated carbon for Volatile Organic Compounds (VOCs) and VOCSi (including siloxane).
- Permeation (membrane), adsorption (PSA: Pressure Swing Adsorption), physical absorption (washing with water), physical-chemical (washing with solvents, amines...), and cryogenics (crystallization or liquefaction of CO_2) for methane enrichment (separation of CO_2) (IEA, 2009) [15].

The elimination of these pollutants from biogas by these different types of technologies is mandatory in order to meet the required technical specifications for the injection of biogas into natural gas networks ($CH_4 > 95\%$, $CO_2 < 2.5\%$ -4%, $O_2 < 0.001\%$ - 1% and $H_2S + COS < 5$ mg/Nm^3) or use as vehicle fuel (D. Marín, *et al.*, 2019) [2]. These standards relating to biomethane purity seem to be virtually non-existent in Africa in general, and in sub-Saharan Africa in particular. Indeed, industrial-scale mechanization technologies are less widespread. There are several domestic-scale technologies that do not necessarily require the purification of the biogas produced. These projects are often managed by national domestic biogas programs, such as the PNB-SN in Senegal. Therefore, for a better technological transition to large-scale technologies, it is imperative to focus on the concept of technology transfer. For the efficient use and successful topicalization of these existing technologies, it is urgent to understand the different types that exist and their specifications. Furthermore, advanced physical/chemical or biological technologies for CO_2 removal often require prior H_2S clean-up, while few technologies are able to remove CO_2 simultaneously. CO_2 and H_2S removal from biogas (*i.e.*, water/chemical cleaning and membrane separation) is very energy and chemical-intensive, which limits their economic and environmental viability for biogas upgrading (D. Marín *et al.*, 2019) [2]. In this context, algal-bacterial symbiosis represents a cost-effective and environmentally friendly platform for the simultaneous removal of CO_2 and H_2S from raw biogas in a single step (Bahr *et al.*, 2014; D. Marín, *et al.*, 2019) [2] [16]. This paper reviews the currently available technologies for the removal of biogas contaminants, with special focus on H_2S, CO_2, H_2O, O_2, N_2, and siloxanes removal. The potentials and limitations of these technologies are also highlighted in order to choose the most ecologically and efficiently possible methods for a biogas sector that is more respectful of the environmental balance.

2. Biogas Purification's Methods

There are several methods of biogas purification. These purification methods can be divided into two main categories:

- Physico-chemical phenomena (reactive absorption or non-reactive adsorption).
- Biological processes (contamination of consumption by living organisms and conversion into harmful forms).

Regardless of the purification method used (physico-chemical or biological), biogas purification follows certain steps:

- The biogas produced is first analyzed to determine its raw composition.
- Then the biogas is passed through a first filter to reduce impurities.
- Then the biogas is passed through a second filter to purify it again or upgrade it to obtain pure biomethane of approximately 99%.

Since the biogas is free of impurities, the final analysis is carried out to determine its biomethane content. This biomethane thus obtained can be injected into the natural gas network, used as biofuel, or transformed into electricity as shown in Figure 1(a).

The biogas can also be purified or washed successively in two, three, or more filters.

These filters may contain different chemical solutions or different technologies depending on the sensitivity of the biogas component to be eliminated (M. Fernández *et al.*, 2013) [17]. In this case, the biomethane obtained is purer and more enriched and may have a higher calorific value. However, these types of technologies require much more investment from a material and financial point of view, as shown in Figure 1(b).

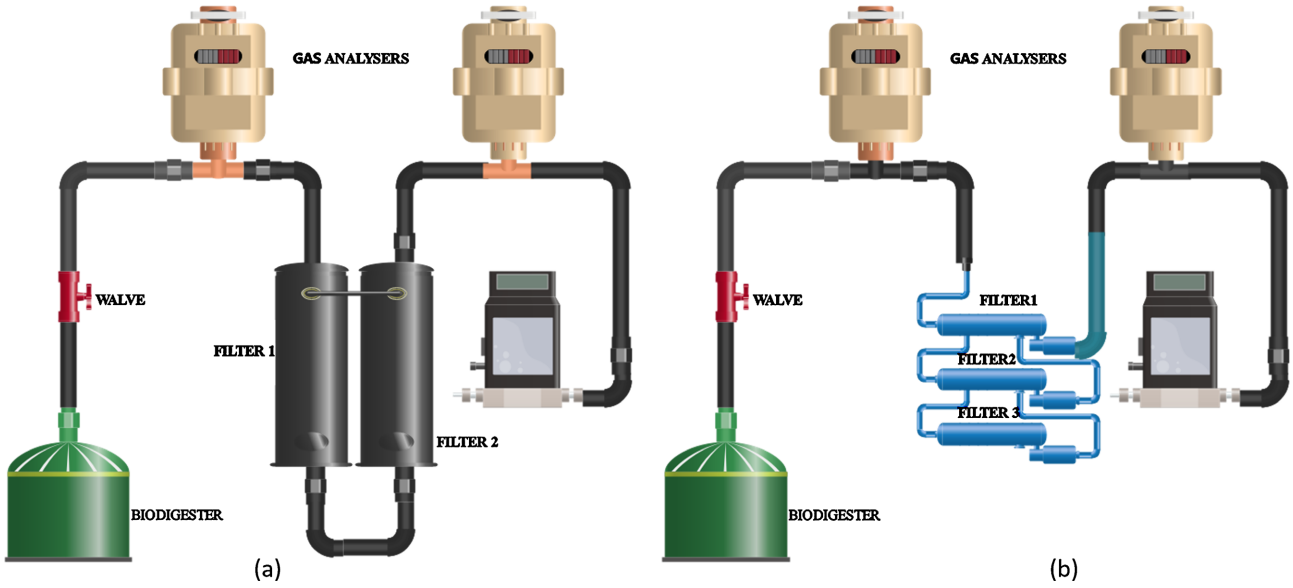


Figure 1. Summary diagrams of a successive biogas purification system (a) and (b).

For example, Lopez *et al.*, 2017 [18], used a laboratory-scale biotrickling filter, which included a cylindrical PVC column (height, 0.45 m; inner diameter, 0.08 m) and was packed with Kaldnes K1 rings (Evolution Aqua Ltd., Wigan,

UK) to a working volume of 1 L. The packing material was characterized by a ring diameter of 1 cm, a density (as received) of 0.17 g·mL^{-1}, a void fraction of 83%, and a water holding capacity (by volume) of 11% (Lebrero *et al.*, 2012) [19]. Gas and liquid flows were operated in countercurrent. MSM (1.0 ± 0.1 L) was recycled continuously at a rate of 0.01 m^3·h^{-1} from a 1.2 L external tank, which was magnetically agitated at 150 rpm (Agimatic-S, Selecta, Barcelona, Spain) (Lopez *et al.*, 2017) [18]. NO_3 was supplied from the $NaNO_3$ stock solution in the storage tank by means of a 120-U peristaltic pump (Watson Marlow, Wilmington, MA). Accordingly, a 120-U peristaltic pump was also used to purge the system and maintain the working volume of the STR. The pH of the recirculating MSM was maintained at 6.9 ± 0.1 by a 120-U peristaltic pump at 6.9 ± 0.1 by periodic addition of 5 M NaOH. The BTF was operated at 25.8˚C ± 1.3˚C in a temperature-controlled room. Double-concentrated MSM was also added periodically to compensate for sampling losses and to provide sufficient nutrients for microbial growth. The BTF was operated under abiotic conditions for 7 days to exclude any potential CH_4 removal due to adsorption or photolysis. Finally, 200 ml of enriched inoculum was introduced into the storage tank to a final volume of 1 L. Biomass attachment to the packing material was observed after 24 hours.

H_2S-free synthetic biogas (70% CH_4, 30% CO_2), obtained by mixing pure CH_4 and CO_2 streams using mass flow controllers (Aalborg TM), was introduced into the BTF at 0.001 m^3.h^{-1} in experimental phase 1 (days 0 - 114). This resulted in an empty bed residence time (EBRT) of 1 h and a methane input load (1 L CH_4) of 495 12 g·m^{-3}·h^{-1}. To evaluate the influence of NO_3 concentration on CH_4 removal and N_2O production, different NO_3 stock solution dosage rates (F) were tested, which required operation of the process at liquid dilution rates (D = F/V) ranging from 0.03 to 0.16 d1. Thus, the NO_3 concentration of the recycle medium was gradually increased as follows: 353 g·m^{-3} (days 0 - 5), 99 16 g·m^{-3} (days 6 - 18), 171 21 g·m^{-3} (days 19 - 31), 211 15 g·m^{-3} (days 32 - 54).

Then the concentration was gradually reduced to 166 ± 18 g·m^{-3} (day 55 - 72), 87 ± 17 g·m^{-3} (day 73 - 83), and finally to 48 ± 11 g·m^{-3} (day 84 - 114). Nitrate consumption rates (rNO_3) were estimated by mass balance calculations in the STR, considering NO_3 stock solution inputs from the stock solution and outputs through the blowdown. The influence of H_2S on the overall performance of the BTF was evaluated during Step 2 (day 115 - 180) by feeding a synthetic biogas mixture consisting of 29.5% CO_2, 70% CH_4, and 0.5% H_2S. This biogas mixture was also fed by an L/S peristaltic pump (Watson Marlow) from filled 25 L-Tedlar bags (Sigma-Aldrich1, St. Louis, MO) at a flow rate of 0.001 m^3·h^{-1}, resulting in an EBRT of 1 h, and 1 L CH_4 of 477 ± 20 g·m^{-3}·h^{-1}, and 1 L H_2S of 7.9 ± 0.5 g·m^{-3}·h^{-1}. In Step 2, D varied from 0.03 to 0.07 d^{-1} and was increased to 0.12 d-1 to avoid inhibition by SO_4^{2-} accumulation from day 156.

Gas samples were collected periodically from the sampling ports located at the inlet and outlet of the BTF to monitor CH_4, O_2, CO_2, H_2O, N_2, and N_2O by GC-

DCT and ECD. Liquid samples (10 ml) were also collected periodically from the storage tank to determine the concentration of NO_3 in NO_2 gas and SO_4^{2-}.

The aim of this study is to revisit the most commonly used technologies in biogas purification and/or enrichment in order to compare their results with a view to choosing the most efficient and least expensive technology for our next deadlines.

3. Biogas Upgrading Technologies

The results presented in this document are taken from the literature review. The aim of this study is to compare the performance of the technologies used in terms of the efficiency and maintenance cost of the systems in certain specific cases.

3.1. Technologies Using Aqueous Solutions

Most technologies using chemical solutions are very effective. Not only do they make it possible to remove impurities from the biogas (CO_2, H_2S, and H_2O fraction), but also, and above all, to enrich the biogas with a CH_4 composition of around 99% (Tippayawong and Thanompongchart, 2010; Al Mamun *et al.*, 2019) [20] [21]. These performances can be observed in Figure 2, where two (2) solutions were used.

- It is a solution T1: $(Ca(CO)_2)$ + activated carbon + silicate gel T2:
- $(Ca(CO)_2)$ + (Fe_0) + Na_2SO_4.
- These T1 and T2 solutions have different efficiencies with regard to the removal of impurities from the biogas, but also with regard to the CH_4 yield obtained. However, the technology using the T1 solution is more efficient than

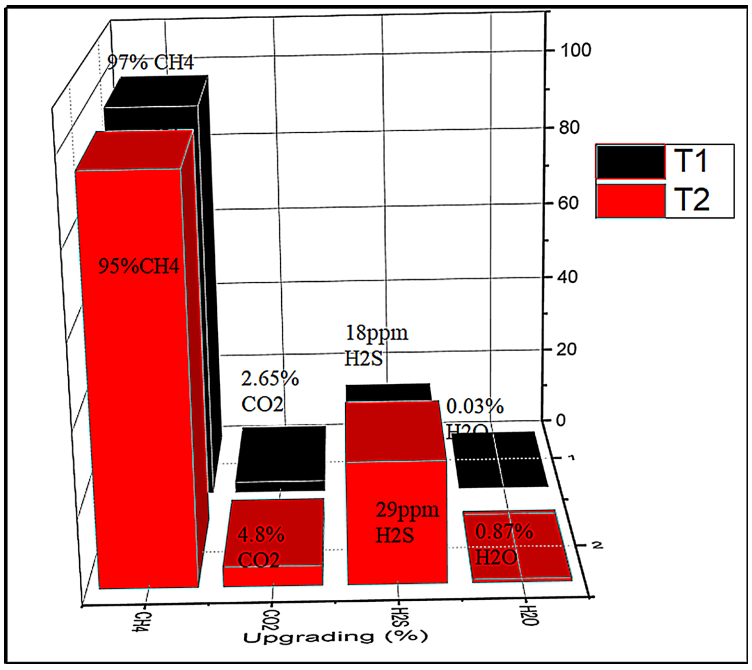


Figure 2. Efficiency of T1 and T2 solutions in biogas purification.

T2, with a CH_4 content of up to 97% (Al Mamun *et al.*, 2019) [21]. This differential behavior in terms of efficiency of the T1 and T2 solutions is often due to the saturation of the reaction media, as the solutions do not have the same sensitivity to the elements to be removed. In addition, a study by Tippayawong and Thanompongchart, 2010 [20], has shown a CH_4 efficiency between 95 and 98% using three (03) types of solvents: NaOH, $Ca(OH)_2$, and MEA (Monoethanolamine). In this experiment, the composition of the biogas used was: 53.1% CH_4, 46.8% CO_2, and 2150 ppm H_2S. At the end of the purification, the proportions obtained according to each solution used were:

- For NaOH: 95% CH_4 and 3.2% CO_2
- For Ca(OH)2: 95% CH_4 and 4% CO_2
- • For MEA: 98% CH_4 and 1.3% CO_2
- It is also important to point out that these solvents were able to eliminate all the fraction of H_2S present in the biogas with one of 2150 ppm, but the efficiency with regard to CO_2 differs. This is what makes MEA more efficient than the other two solvents, as it was able to reduce CO_2 by up to 1.3%. Malla *et al.*, 2016 [22] also conducted similar studies but used several other solvents such as NH_2OH, $2NH_2OH$, KOH, NaOH, and 2RNH2.

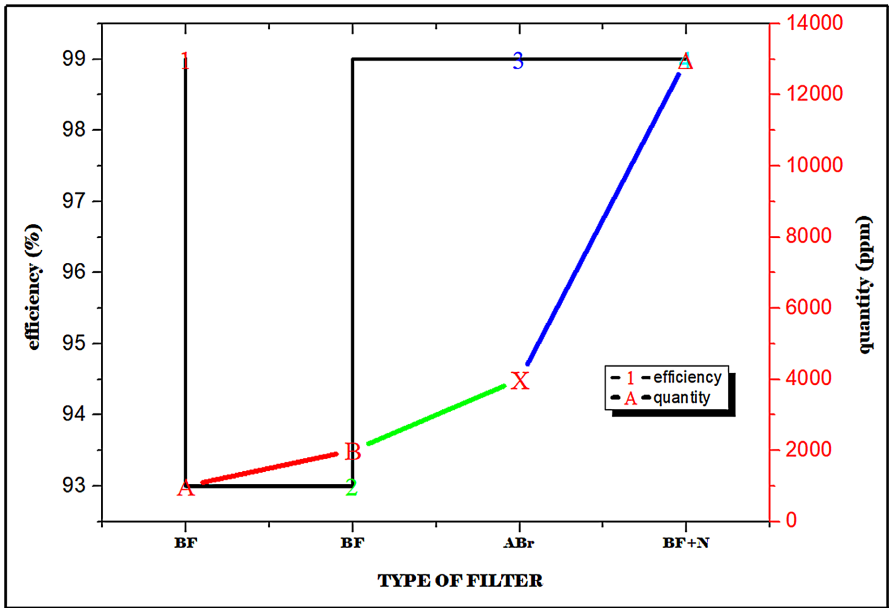
3.2. Technology Using Filters

As far as filters are concerned, three types have been used (BF: Biotrickling Filter; ABr: Anoxic Bioreactor; BF + N: Biotrickling Filter + Nitrate) with different H_2S flow rates are shown.

If an H_2S flow rate between 0 and 1000 ppm (A) is used as input, the efficiency is 99% (1) for BF. However, if the H_2S flow rate is increased up to 2000 ppm (A-B), the efficiency decreases to 93% due to the saturation of the filters (2). Thus, for this drop in performance due to filter saturation, it is necessary to add nitrate (BF + N). This allows a flow rate of up to 13,000 pp (X-D) with an efficiency of 99% (4).

As for ABr, they are 99% efficient (3) with flow rates varying between 0 and 4000 ppm (A-X). The efficiency limits in terms of filter flows are often due to the phenomena of oversaturation caused by excess H_2S, relative to the threshold admitted by these filters, as shown in **Figure 3** (Lopez *et al.*, 2013; M. Fernández *et al.*, 2013) [23] [24].

It should be remembered that this limit due to H_2S saturation can be bypassed to improve filter performance by modulating the flow velocity between 4.4 and 18.9 m/h. Compared to operation without flow liquid velocity control, velocity control resulted in a 10% improvement in removal capacity and, more importantly, a 9% increase in product selectivity over sulphate at a loading rate of 283.8 g S-H_2S $m^{-3}{\cdot}h^{-1}$. Concentration profiles along the height of the biotrickling filter highlighted that the regulation of the liquid runoff velocity gradually led to an improvement in dissolved water quality and to a better distribution of dissolved oxygen and, consequently, to an improvement in the overall performance of the biotrickling filter (LR Lopez *et al.*, 2015; Jin *et al.*, 2005) [25] [26].



BF: Biotrickling Filter; ABr: Anoxic Bioreactor; BF + N: Biotrickling Filter Nitrate (Lopez *et al.*, 2013) [23].

Figure 3. Filter efficiency as a function of the amount of H_2S.

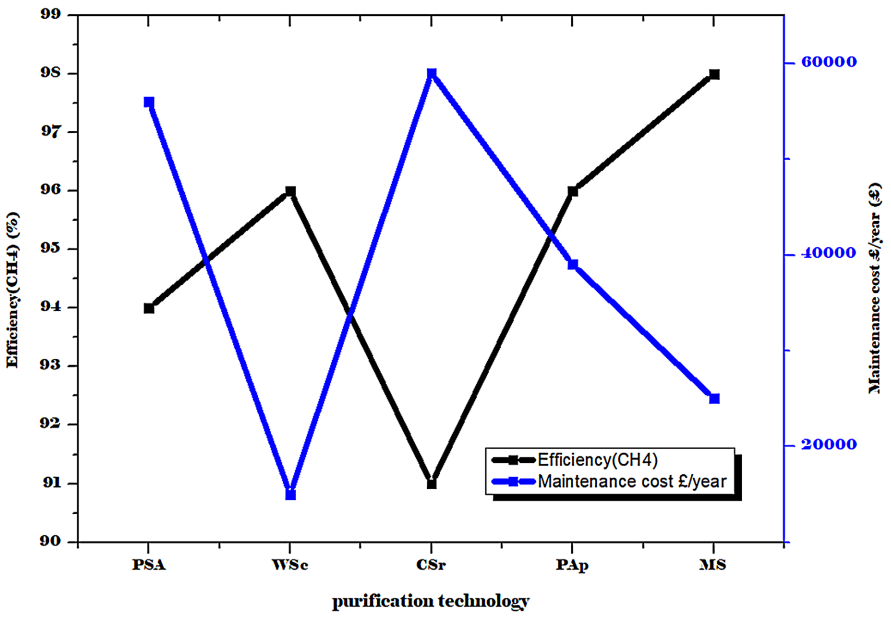
Several studies have also focused on BTFs, concentrating on the predominant microorganisms in these types of reactors and also on their pollutant gas removal performance. Thus, Prachuabmorn and Panich 2010 [27] show us that the performance of BTFs is linked to the capacity for biodegradation of pollutants by microorganisms. Depending on the particularity of the pollutant to be eliminated, we find different types of bacterial consortia. With biochemical analyses of the bacterial communities of the biofilm, it is noted that it is composed of a large number of bacteria and a reduced number of fungi (Zhao *et al.*, 2014) [28]. In addition, several bacterial species were identified, including Pseudomonas, Bacillus, Staphylococcus, and Rhodococcus, but for most bioreactors used for nitrogen, H_2S, and VOC removal, Pseudomonas were identified as the predominant species (Giri *et al.*, 2014; Li *et al.*, 2014; Zheng *et al.*, 2016, H. Wu *et al.*, 2018) [29]-[32]. It is also noted that the simultaneous presence of Bacillus under aerobic nitrification-denitrification conditions, where Staphylococcus can reduce nitrate to nitrite (Cheng *et al.*, 2010) [33]. Rhodococci are involved in the metabolism of harmful environmental pollutants, toluene, naphthalene, ethylene, herbicides, and other compounds (Baltrenas *et al.*, 2015) [34]. Some studies, such as those by Cox and Deshusses, 1998; Kraakman *et al.*, 2011, and Giri *et al.*, 2014 [29] [35] [36], have shown that fungi have a higher efficiency than bacteria in the removal of VOCs, as they allow for easy uptake of many VOCs from the bulk gas phase due to their filamentous structures with aerial mycelia and large surface area. Indeed, fungi seem to have a better capacity to resist low humidity and high acidity environments than bacteria (Prenafetaboldú *et al.*, 2008, Gabriel and Deshusses, 2003; Sakuma, Hattori, and Deshusses, 2006; M. Fortuny *et al.*, 2008) [37]-[40]. Thus, such

potential of fungi as degradation microorganisms in BTFs could be better exploited.

3.3. Technologies Using Physico-Chemical Processes

The physico-chemical processes for biogas purification are many and varied, ranging from the simplest to the most complex in terms of technology. These processes are reputed to be very effective because they can simultaneously remove impurities from the biogas (CO_2, H_2S, H_2O, NH_3, O_2, N_2, and siloxane...) by enriching it with more than 99% CH_4 (W.-C. Lin *et al.*, 2013; Panwar and Kadam, 2017; Cea-Barcia *et al.*, 2018; Lopez *et al.*, 2015; Lopez *et al.*, 2013) [23] [25] [41]-[43]. However, these types of processes require a lot of maintenance. This has allowed us to look more closely at the work of Patterson *et al.*, 2011, [44] which combines the performance of the processes with the cost of maintenance in euros per year, as shown in **Figure 4**. Thus, the processes studied are: PSA Absorption, WSc: Water Scrubbing, CSc: Chemical (amine) Scrubbing, PAp: Physical Absorption, and MS: Membrane Separation. The conclusion we were able to draw is that there are technologies such as CSc which have a high cost of maintenance per year (€59,000/year) but are less efficient, 91% CH_4. Others such as WSc have a low maintenance cost (€15,000/year) and are more efficient. But the best technology in terms of maintenance cost per year and efficiency is the MS with a cost of €25,000/year and an efficiency of 98% CH_4 [45] **Figure 4**. Membrane separation offers two advantages:

Firstly, maintenance costs are lower than for all other technologies using



PSA Absorption, WSc: Water Scrubbing, CSc: Chemical (amine) Scrubbing, PAp: Physical Absorption, and MS: Membrane Separation (Patterson *et al.*, 2011) [44].

Figure 4. Efficiency of technologies correlated with the cost of maintenance in Euros per year.

physicochemical processes. This significantly reduces additional expenses for users, thereby streamlining cash flow.

Beyond the low maintenance costs, membrane separation is more efficient than other technologies in the same family. The combination of these two major criteria allows users to achieve a faster overall return on investment from on-farm anaerobic digestion facilities.

These cost and efficiency ratios can be explained by the fact that some technologies need to be renewed frequently, while others can operate for periods before reaching their saturation point.

3.4. Technologies Using Microorganisms as Filters

In addition to technologies that eliminate H_2S, there are nowadays processes that allow the biogas to be enriched by eliminating (reducing) CO_2. These processes are known as biological processes. To enrich the biogas, microalgae cultures are often used, which can vary from one experiment to another.

Microalgae are ubiquitous photosynthetic microorganisms that occupy a variety of habitats. Oxygenic photosynthesis by microalgae is thought to be responsible for the formation of the Earth's current oxygen-rich and life-supporting atmosphere. According to (http://www.algaebase.org/) [41], there are currently about 150,000 identified species of microalgae, which explains the large diversity of the microalgal population available for research. Microalgae can be prokaryotic, such as photosynthetic bacteria (often called blue-green algae), or eukaryotic in nature. The cell structure of microalgae is simple, and they grow by fixing atmospheric CO_2 into organic biomass under the influence of light energy, making them the primary producers of an ecosystem. Carbon fixation by microalgae occurs in two stages: the light reaction and the dark reaction. In the light reaction, the light energy in the photosynthetically active region involves several mechanisms, ranging from the collection of carbon through the transfer to the formation of cytochrome complexes and reception by the enzyme ferredoxin. The light reactions of photosynthesis that occur in the presence of chlorophyll are summarized as follows (Masojidek *et al.*, 2013) [46].

Rubisco has a very low affinity for CO_2, and O_2, being a bifunctional enzyme, represents a very competitive substrate. Microalgae overcome the inefficiency of Rubisco by carbon concentration mechanisms and by packaging Rubisco at high concentrations in specific organelles (Wang *et al.*, 2015) [47]. Similarly, highly efficient energy-dependent inorganic carbon uptake mechanisms can accumulate up to 100 times atmospheric CO_2 as intracellular carbonate via enhanced carbonic anhydrase activity at low CO_2 concentrations (Spalding, 2008) [48]. Although well studied in model organisms (e.g., Chlamydomonas reinhardtii), these results generally apply to all microalgae. According to Brennan and Owende, 2010 [49], microalgae have a very high photosynthetic efficiency, ranging from 1% to 20%, compared to the photosynthetic efficiency achievable by land plants, which is on the order of 1% to 2%. For example, the oil productivity of oleaginous microalgae

could be as high as 51,927 kg biodiesel/ha per year with low-oil microalgae, while for a typical oil crop such as jatropha it is only 656 kg biodiesel/ha per year, with the photosynthetic efficiency of terrestrial plants being 1% - 2% (Mata *et al.*, 2010) [50]. Some microalgae have the ability to use organic carbon as a source of energy and carbon in the absence of light, and this is known as the heterotrophic culture mode. The simultaneous use of organic and inorganic carbon in the presence of low light intensities is called a mixotrophic culture mode [51]. Mixotrophic cultivation is of great interest for the simultaneous removal of organic carbon from the anaerobic digestate and CO_2 from the biogas, for biogas upgrading and bioremediation of the digestate. Regarding the CO_2 tolerance of microalgae, several studies have been carried out to understand and control these mechanisms. According to (Yew *et al.*, 2019) [52], due to their high CO_2 fixation capacity and photosynthetic efficiency, in addition to technologies that eliminate H_2S, there are nowadays processes that allow the biogas to be enriched by eliminating (reducing) CO_2. These processes are known as biological processes. To enrich the biogas, microalgae cultures are often used, which can vary from one experiment to another.

The integration of anaerobic digestion and microalgae cultivation for carbon capture from biogas by microalgae has many advantages: an environmentally friendly method to improve the methane content of biogas, efficient capture and use of carbon on site, and generation of energy-rich microalgal biomass with value-added products such as antioxidants, lipids, and polyunsaturated fatty acids. The biogas is a residual gas from fermentation and therefore available at room temperature, which reduces the need for additional energy consumption during cultivation, which is carried out at room temperature. However, most studies have confirmed that photoautotrophic/mixotrophic growth of microalgae is possible using CO_2 from biogas as a carbon source. The concentration of CO_2 in biogas varies from 30% to 45%, and some microalgae are tolerant of higher CO_2 concentrations.

Indeed, according to studies by Solovchenko *et al.*, 2014 [53], a new Desmodesmus sp. 3Dp86E-1 was able to achieve high biomass of up to 1.1 g/L under high light conditions, and the biomass content was similar to that of cultures grown under 5% and 20% CO_2 conditions. However, cultures grown under 100% CO_2 had a lag phase of 3 days, and the total carbon fixation rate was 1.5 L CO_2 $day^{-1} \cdot L^{-1}$ culture volume. Scenedesmus obliquus SJTU-3 and Chlorella pyrenoidosa SJTU-2 were found to be tolerant of 50% CO_2 with a biomass production of 0.69 $g \cdot L^{-1}$, and higher biomass accumulation (>1.22 $g \cdot L^{-1}$) occurred with increasing CO_2 concentration up to 20% (Tang *et al.*, 2011) [54].

With increasing CO_2 concentration, other observations were made, such as increased accumulation of lipids and polyunsaturated fatty acids (Tang *et al.*, 2011) [54]. Furthermore, six microalgae strains (Chlorella vulgaris TISTR 8580, Chlorella protothecoides TISTR 8243, Chlorococcum sp. TISTR 8416, Chlorella sp. TISTR 8263, Scenedesmus armatus TISTR 8653, and a marine Chlorella sp.) were evaluated for their tolerance and growth at 50% CO_2 [55]. According to the results

of this study, Chlorella sp. TISTR 8263 showed the highest specific growth rate (0.469 day^{-1}) at 50% CO_2, with a lipid content of 25% and a lipid productivity of 13.9 $mg{\cdot}L^{-1}{\cdot}d^{-1}$. Chlorella sp. marine and C. protothecoides TISTR 8243 had a specific growth rate of 0.457 day^{-1} and 0.413 day^{-1}, respectively (Tongprawhan *et al.*, 2014) [55]. Chlorella vulgaris FACHB-31, Scenedesmus obliquus FACHB-416, and Neochloris oleoabundans UTEX-1185 were able to grow well in the presence of 45% - 55% CO_2 in the presence of activated sludge, with only a slight decrease in biomass productivity. The biomass productivities of these cultures ranged from 0.138 to 0.153 g/L/d in the presence of 55% CO_2 (Sun *et al.*, 2016) [56]. Chlorella sp. ZY-1 was found to be tolerant up to a CO_2 concentration of 70%. Maximum cell growth and biomass accumulation (6 $g{\cdot}L^{-1}$) was obtained at 10% - 20% CO_2, but the growth rate was not significantly inhibited up to 60% CO_2 (2.8 $g{\cdot}L^{-1}$ biomass), and a biomass of 0.766 g/L was obtained at 70% CO_2 (Yue and Chen, 2005) [57]. A Chlorella sp. KR-1 was also tolerant up to 70% CO_2 with a biomass production of 0.71 $g{\cdot}L^{-1}$, and it could still reach 2.8 $g{\cdot}L^{-1}$ at 50% CO_2 and 3.6 g/L at 30% CO_2 (Sung *et al.*, 1999) [58]. Acutodesmus deserticola was tolerant at 20% CO_2 with a biomass production of 1.65 $g{\cdot}L^{-1}$ (Varshney *et al.*, 2016) [53], while Scenedesmus bajacalifornicus achieved a maximum biomass of 0.68 g/L at 25% CO_2 (maximum biomass of 0.85 $g{\cdot}L^{-1}$ at 15% CO_2) [59].

As a result of these findings, there are now new reports of the isolation of microalgae with high CO_2 tolerance and the evaluation of their effectiveness as candidates for carbon mitigation. Moreira and Pires suggested that bioenergy with carbon capture and storage using microalgae can result in negative emissions, but the real challenge is the successful, sustainable, and economic integration of carbon capture, microalgal biomass production, and bioenergy production [60].

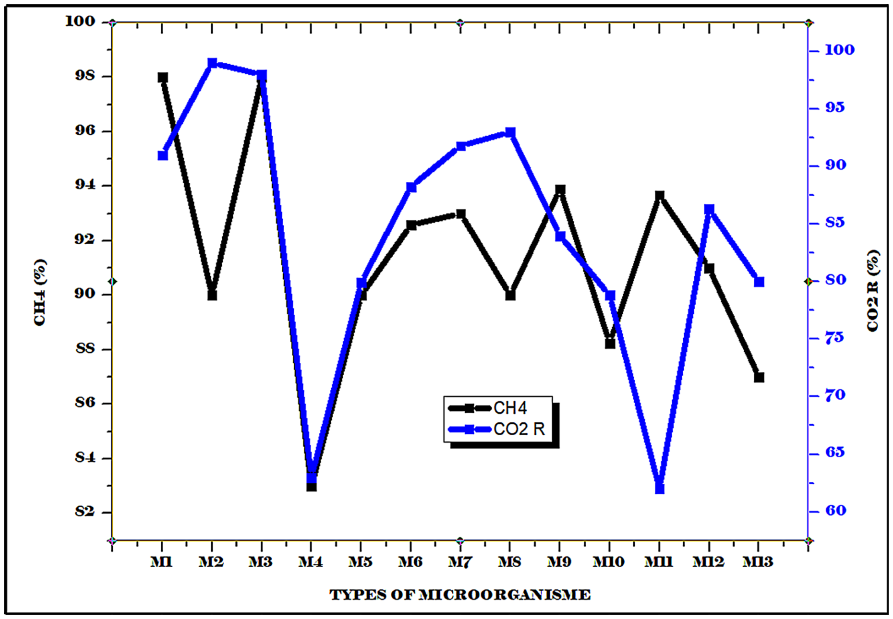
Thus, with regard to the purification and enrichment of biogas, we were also interested in the work of D. Nagarajan *et al.*, 2019 [61].

During this work, they used several families of microalgae. Among these families of microorganisms, we can mention, among others, those shown in **Figure 5**.

To compare their efficiency, we plotted their CO_2 reduction capacity and their biogas enrichment power in terms of CH_4 concentration on the same graph. Thus, as **Figure 6** shows, we can see that the microorganisms with the highest percentage are M3, with a CO_2 removal rate of more than 98% and a CH_4 rate of 98%. However, the microorganisms that reduce CO_2 the least are M11, with a rate of 62%; but they have a higher CH_4 enrichment rate than M13 (93.69% > 87%). This differential efficiency can also be explained by the phenomenon of the admitted threshold, which differs from one technology to another. Others have also used microalgae [ph1].

With yields of up to 99% CH_4, proving the potential of microorganisms in biogas purification.

Other studies have focused on the effect of other parameters on CO_2 removal by microalgae. These include the effects of light intensity, alkalinity (pH), temperature, and seasonal variation.



M1: Microalgal consortium comprised of *Chlorella vulgaris*, *Stigeoclonium temie*, *Nitzchia closterium*, and Navicula amphora, M2: Algal bacterial consortium, M3: *Chlorella sorokiniana*, M4: *Chlorella vulgaris*, *Scenedesmus obliquus*, M5: Scenedesmus obliquus FACHB416 with Ganoderma lucidum 5765, M6: *Chlorella vulgaris* and nitrifying, denitrifying activated sludge, M7: Indigenous microalgae that naturally grew in the reactor, M8: *Chlorella sorokiniana*, M9: *Chlorella vulgaris* FHCB31 and *Ganoderma cucium*, M10: *Scenedesmus obliquus* FACHB31, M11: Chlorella sp., M12: Chlorella sp. MB-9, M13: Chlorella sp. MM-2.

Figure 5. Percentage of CO_2 reduction and biogas enrichment by microorganisms.

Effect of light intensity

Light is the energy source for photosynthesis in microalgae, and it is essential that light is provided at an optimal intensity for maximum photosynthetic efficiency. In full biogas upgrading with bioremediation of biogas sludge, the cultivation mode is mixotrophic and aims to utilize the organic carbon present in the digester sludge as well as the available nitrogen and phosphorus. In mixotrophic mode, low light intensities are generally preferred. High light intensities are known to inhibit the uptake and assimilation of organic carbon in microalgae (Perez-Garcia *et al.*, 2011) [62]. According to Choix *et al.* 2017b [63], light intensity can significantly affect CO_2 removal from biogas in Leptolyngbya sp. CChF1. Scenedesmus sp. prefers moderate light intensities of 150 - 170 μmol·m^{-2}·s^{-1} to increase the methane content of raw biogas from 61.75% to 94.4% with concomitant removal of nutrients from the slurry. The removal efficiencies of COD, TN, and TP were 93.08%, 84.2%, and 86.76%, respectively (Ouyang *et al.*, 2015) [64]. Works such as Yan and Zheng, 2013; Yan *et al.*, 2016a; Zhao *et al.*, 2013; and Zhang *et al.*, 2017 [65]-[67] focused on Chlorella sp.

However, the optimal light intensity was 400 - 1000 μmol m^{-2}·s^{-1} with a light augmentation strategy, and a final methane content of the biogas of 92.87% was achieved [66].

Effect of the pH of the culture medium

The optimal pH of the medium for microalgae strains varies between species and strains, and most microalgae culture systems optimize the pH requirement. In the context of biogas upgrading, the pH of the medium is of primary importance, as it determines the available form of carbon dioxide. Carbon dioxide dissolves in water and can be available in three different forms: carbonic acid, bicarbonate, and carbonate. At a near-neutral pH of about 5.5 - 7, carbon dioxide is available as both carbonic acid and bicarbonate, but as the pH increases, bicarbonate predominates [61] [68]. Thus, the work of Nolla-Ardevol *et al.*, 2015 [69] demonstrated that anaerobic digestion of spirulina at pH 10 using an alkaline halo anaerobic microbial consortium produced biogas with a methane content of 96%. Under all conditions, CO_2 was present at an average of 6% with no H_2S, and the alkaline medium was known to behave as a CO_2 scavenger. Chlorella sp. showed better CO_2 removal from the biogas when the pH of the medium was increased to 7.8 [55]. The efficiency of CO_2 removal increased from 70% to 89% when parameters such as light intensity, initial cell concentration, and media components for buffering activity were optimized by response surface methodology, and the methane content of the biogas increased to 94.7% [55]. Chlorella sp. co-cultured with aerobic activated sludge showed an increase in CO_2 removal efficiency of the actual biogas when the pH was increased from 7 to 8.1. The CO_2 removal efficiency increased from 23% to 62%, with a CO_2 fixation rate of 285 mg CO_2 $L^{-1}{\cdot}d^{-1}$ [70]. Furthermore, the work of Franco-Morgado *et al.*, 2017 [71] carried out in a high-throughput algal tank with an alkaliphilic microalgae-bacteria consortium (including Picochlorum sp., Halospirulina sp., and a consortium of sulphur-oxidizing bacteria) was used to enhance synthetic biogas at high alkalinity and pH 9.3. A CO_2 removal efficiency of 91.5% was achieved.

But it is important to note that the highest CO_2 removal efficiencies of 99.3% and 97.8% were obtained at an alkaline pH between 9.7 and 11 in a high-flow algal tank treating synthetic biogas [72]. Another similar study showed that CO_2 removal efficiency in a high-flow outdoor algae pond was strongly influenced by alkalinity, and the highest removal efficiency of 95% was obtained at the highest alkalinity [73]. Thus, alkaline conditions are known to be suitable for efficient CO_2 removal by photosynthetic biogas upgrading.

Effect of temperature

Temperature is an important factor affecting the growth of microalgae, and most microalgae prefer an ambient temperature of around 25˚C to 30˚C for maximum biomass production without affecting photosynthetic efficiency [49].

Biogas is a fermentation gas and is therefore available at room temperature without the need to cool it down before using it in the culture. Microalgae that can grow efficiently at ambient temperatures can be used for biogas utilization, while thermophilic and heat-tolerant microalgal strains are needed to use the flue gas as a source of carbon dioxide. Indeed, Meier *et al.* noted that the solubility of CO_2 is temperature dependent and that solubility decreases at higher

temperatures and vice versa. Thus, feeding biogas during the dark periods of the light/dark cycle or feeding at night under outdoor conditions can improve CO_2 solubility and enhance biogas recovery [74]. A similar effect of temperature on CO_2 solubility was observed by Rodero *et al.* (Rodero *et al.*, 2018) [72]. They showed that a higher CO_2 removal efficiency was achieved at 12˚C than at 35˚C. Similar night feeding has also been applied for synthetic biogas valorization by a consortium of microalgae in open algal ponds at high flow rates (Franco-Morgado *et al.*, 2018). Similarly, a lower temperature could lead to a decrease in evaporative water losses in open photobioreactors; otherwise, the water loss has to be compensated by adding fresh water [72].

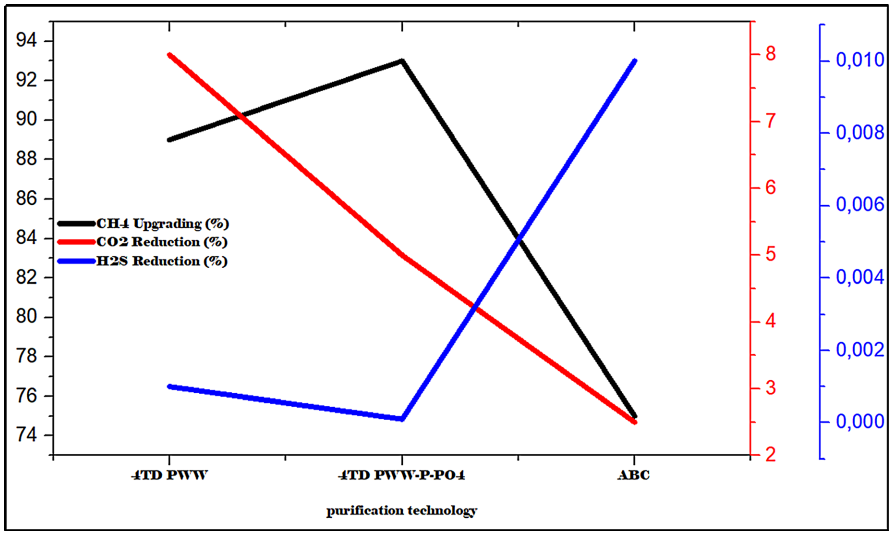
Effect of seasonal variations in outdoor conditions

Outdoor cultivation is an economical option for growing microalgae on a commercial scale, but outdoor cultivation is subject to variations in light intensity and temperature due to seasonal and diurnal variations. In general, outdoor cultivation performs better in sunny summer conditions than in winter, and the winds and rains of the monsoon season severely affect the crop. Thus, the work of Marín *et al.*, 2018a [75] in a high-flow outdoor algae pond, which performs integrated biogas valorization and digestate bioremediation, achieved better performance during the summer months. CO_2 concentrations in the biogas were 0.7% in August and 11.7% in winter (December) (for an initial concentration of 30%), and methane concentrations were 85.2% in December compared to 97.9% in summer (June). A high biomass productivity of 22.5 $g/m^2/d$ was obtained during the summer months. In a similar study treating synthetic biogas in an outdoor high-flow algal tank, methane concentrations in the enhanced biogas were in the order of 99.6% in summer (August) compared to 85.6% in winter (December). CO_2 concentrations were around 0.1% in spring (May), while they were 11.6% in winter (December) for an initial concentration of 30% [75] [76]. A CO_2 capture efficiency of 70% was obtained in cloudy weather with a methane content of 84% in biogas, and the capture efficiency increased to 80% in sunny weather with a methane content of 87%.

3.5. Technologies Using Physico-Chemical and Biological Processes

The works that have interested us most in these types of processes are those of Marín *et al.*, 2019 [2]. As shown in **Figure 6**, these types of processes can eliminate both CO_2 up to 2.5% (ABC) and H_2S up to 0%, enriching the biogas up to more than 93% (4TD PWW-P-PO_4). The originality of the work of D. Marín *et al.*, 2019 [2] is that it also provides information on the physicochemical composition of the microorganisms used, which is of paramount importance in their use.

The capacity for simultaneous wastewater treatment and biogas valorization by the Phototrophic Purple Bacteria (PPB) and the Algae-Bacteria consortium was comparatively evaluated. According to D. Marin *et al.*, 2019 [2], a limited decrease in CO_2 concentration from 28.6% to 24.1% was observed with the use of the algae-bacteria consortium, while for the use of the PPB inoculum, the CO_2 concentration



4TD PWW: 4 times diluted Piggery wastewater treatment, 4TD PWW-P-PO_4: 4 times diluted Piggery wastewater treatment + 50 mg P-PO_4, ABC: Algal Bacterial Consortium [2].

Figure 6. Physicochemical and biological processes for the purification and enrichment of biogas.

recorded is 3.3%. Indeed, the observation of the acidic pH (5.4 ± 0.7) of the culture broth of the algae-bacteria system imposed by the biogas headspace is probably the cause of the inhibition of the photosynthetic activity of microalgae. Several other parameters were also studied for these two systems and all results are listed in Table 1.

Table 1. Comparative study of the performance of PPB and the Algae-Bacteria Consortium in wastewater treatment and biogas recovery (D. Marin *et al.*, 2019 [2]).

Parameters studied	Algae-Bacteria Consortium	Phototrophic Purple Bacteria (PPB)
CH_4 Concentration	73.6%	93.3%
H_2S Concentration	0.47%	0.10 %.
CO_2 Concentration	28.6% to 24.1%	3.3%
TN concentration	38 ± 7 mg/L	From 2498 ± 0 to 1483 ± 7 mg/L (RE of 41%)
Concentration TN	200 ± 13 mg/L	624 ± 33 mg/L
Concentration of inorganic carbon (IC)	14 ± 4 mg/L	0 to 459 ± 40 mg/L

4. Biogas Upgrading Summary

Biogas purification and/or enrichment has become a challenge for today's industrial biogas plants. To meet this challenge, scientists and anaerobic digestion specialists around the world, concerned about the future of our planet, are striving to find the most efficient and environmentally friendly ways to purify biogas to achieve a CH_4 content of around 99.99%. We therefore set ourselves the goal of

revisiting the existing technologies and methods of purification. These methods are classified into five main families that are linked to each other. These are physical, chemical, biological, physicochemical, and physicochemical and biological methods. It is important to note that a comparative study of the effectiveness of all existing biogas purification methods seems utopian given the wide variety of characteristics and parameters involved. Nevertheless, this study has enabled us to observe the performance and limitations of the different methods used around the world.

This study has enabled us to observe the performance and limitations of the different methods used worldwide.

Most of the methods are often very efficient with a low CO_2 and/or H_2S inlet flow. When the concentration of CO_2 and/or H_2S increases, most of the filters or solutions used become saturated. This leads to additional maintenance costs. However, some filters can remove H_2S up to values of 14,000 ppm and CO_2 up to 100%, but this often results in some damage to the immediate environment of the farms due to the renewal of solutions and the changing of exhausted filters. Thus, the use of microalgae in the purification and enrichment of biogas seems to be a hope for the preservation and restoration of our common heritage, the ecosystem. To achieve this, we need to multiply research synergies for a brighter future of anaerobic digestion. The multiplication of synergies requires technology transfer from one region of the world to another. Indeed, most of the biogas purification systems or methods used in industrialized countries are almost absent and unknown in many African countries. For example, in Senegal, the only filters used by the PNB-SN are iron filings, which only remove H_2S. It is important to remember that there are almost no studies on the effectiveness of these filters. This partly hinders the diffusion of anaerobic digestion technology in these countries despite the existence of an immeasurable potential biomass resource. The development of the anaerobic digestion sector in sub-Saharan Africa requires the appropriation, mastery, and development of technologies that can eliminate all impurities from the biogas to produce biomethane with a CH_4 content of around 99.9%.

5. Conclusions

Biomethane is of great interest and relevance in light of developments in the global energy system, with specific reference to the price volatility and environmental issues of fossil fuels. This paper presents the scientific and technological advances in the field of biogas treatment and valorization.

Biogas purification and enrichment have become a necessity for the production of biomethane in accordance with established conventions and standards. These standards vary from one country to another and often from one use to another, depending on whether it is used for electricity and heat production by cogeneration, used as a green fuel, or injected into the gas network. However, in order to obtain the final product, which is biomethane, biogas must undergo several treatments according to the existing technologies. These technologies are classified

according to whether the purification method is physical, chemical, physicochemical, or biological.

Thus, the analytical study of these methods of purification of biogas allows us to understand the existing differences between them from the point of view of effectiveness and the impurities reduced or eliminated during these processes. The analysis of the results has shown that the performances of the methods differ based on their basic principles, but are also sensitive to some external parameters. Also, these methods generate additional costs in terms of maintenance. Therefore, performance or cost minimization is not the only criterion for selecting biogas upgrading technology. It is also essential that the specific technology can satisfy the specific requirement. This has led us to establish technological eligibility criteria that depend on performance, efficiency, and especially maintenance cost and the quality of the biomethane produced. One of the methods that seems to be the most popular is membrane separation (MS). However, one of the parameters that escapes us at this level is the cost of acquisition and installation of these technologies.

This review has established that there are still blind spots in biogas treatment and purification, namely, the new technologies that are being implemented even though some of them are on the laboratory scale. Therefore, further efforts are needed to bridge the knowledge gap between these new methods and large-scale operations.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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