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Introduction to the purification of biogas

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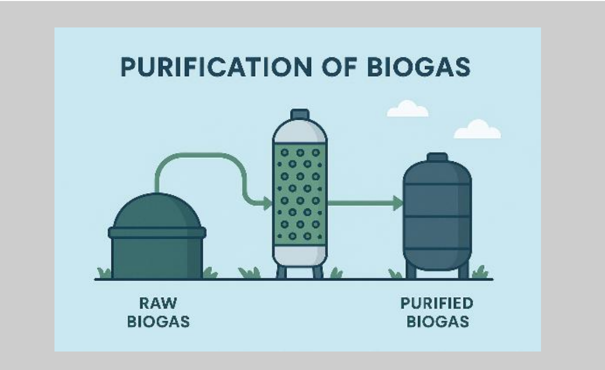
ABSTRACT.

Raw biogas, produced by the anaerobic digestion of various organic wastes, is a renewable source of energy composed of methane (CH_4, 55-65%) and carbon dioxide (CO_2, 35-45%) as well as various impurities that are generated during this biological process. Some of these impurities can degrade equipment, be toxic to people or hazardous for the environment, so biogas has to be purified prior to use.

This technical information note introduces the different concepts and techniques that are used for the purification of biogas, outlining their benefits and drawbacks. Hydrogen sulfide, a particularly problematic impurity, will be used as a case study to illustrate the different systems that can be implemented to purify biogas.



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1. Introduction to biogas

1.1. About biogas

According to the European Environmental Agency, a biogas consists of a gas, rich in methane, which is produced by the anaerobic fermentation of animal dung, human sewage or crop residues in an air-tight container.[1] Many recent research studies have proven that its development will play a critical role in reducing global warming, as mentioned for instance in the European Union's methane strategy published in 2020 and that targets a production of 35 billion cubic meters of biomethane – notably coming from biogas – for 2030. [2,3] In 2018, 35 Mtoe (million-ton oil equivalent) were produced, mainly in Europe, from agricultural resources (animal and vegetal) as represented on the graph below, and the biogas potential was estimated to 570 Mtoe.[4]

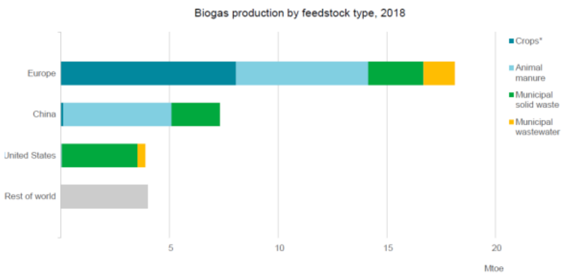

Figure 1. Origin of biogas, per region, in 2018

In 2022, there were about 90 anaerobic co-digestion installations producing biogas in Belgium, around 40 in Flanders and 50 in Wallonia, representing about 2% of the total Belgian gas consumption while 1869 installations producing biogas for energy purposes are listed in France [5]. In Wallonia, about 700 MTo – mainly coming from agricultural wastes – were converted in biogas in 2021.[6]

Once produced, the biogas can be used in three different ways, namely in a decentralised combined heat and power plant (CHP), directly in the close vicinity of the biogas plant or after its upgrade as a fuel or being injected in the natural gas grid. For CHP units, the biogas is dried, desulfurised and used in a gas engine that will deliver power and heat. Generally, the energy produced is used externally and fed into the grid while the heat is used to warm the digester or for other applications (heating building or crop drying for example). This is the most common use of biogas, both globally and in Belgium. Biogas can also be used directly on-site to reduce its energy procurement cost, directly in households, with heat boilers or as fuel for the equipment in the farm. Finally, biogas can be upgraded into biomethane by separating methane and CO_2 and by removing its impurities.

1.2. What are the main impurities in biogas?

Biogas is mainly composed of methane (CH_4) and carbon dioxide (CO_2) in a range of 55-65% and 35-45% respectively. Despite being even more impactful on global warming than CO_2 for an equivalent volume, biomethane is considered the higher-value part of the mixture thanks to its gross heating value of 55 MJ/kg, making it a very interesting fuel gas. Actually, by increasing the amount of CO_2 – a non-burning gas – in the mixture, the overall heat value will decrease for example from 28MJ/m^3 for 75% of biomethane to 16MJ/m^3 for a biogas containing only 45% of CH_4. [7] In some cases, this discrepancy justifies the upgrade of biogas into biomethane but, according to the International Energy Agency (IEA), the extraction of biomethane represent today only 10% of the biogas produced and biomethane represent 0.1% of the total consumption of natural gas. [4] In addition, raw biogas contains different types of impurities, the main ones from agricultural wastes being reported the Table below [8].

Table 1. Common impurities in biogas.

Impurity	Unit	Value
Water	%	< 6
H_2S	ppm	50-20000
NH_3	ppm	50-144
N_2	%	< 1
O_2	%	< 1
CO	%	< 1
Siloxanes	ppm	Traces

Each of these pollutants may raise issues due to their intrinsic toxicity, their impact on the quality of the biogas or on the sustainability of the process. Hydrogen sulfide (H_2S) for instance, is not only an extremely odorous chemical even at low concentration but also highly toxic, inducing from headache as low as 0.008 ppm to neurological effects at 20 ppm in the air, this chemical being labeled as a nerve poison. It can also generate pollutants acidic species when in contact with water or humid air, that can lead to environmental hazardous acidic rains or to corrosion of the equipment during the process. Water, if obviously not toxic, also raises issues as it reacts with the aforementioned chemicals to generate the corrosive species. It also tends to accumulate in the process and can freeze with the effect of temperature or pressure for instance. It also decreases the

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heat value of the biogas, like CO_2 or N_2, therefore reducing its quality. If biogas is directly used as a fuel, siloxanes can irreversibly decompose into silica that can cause great damage to equipment. In addition, particles are present in biogas and need to be removed to prevent clogging or plugging of the pores of an adsorbent/catalyst.

The nature and amount of the pollutants are highly dependent on the origin and properties of the feedstock as well as on the conditions of anaerobic digestion. For instance, siloxanes are more likely to be present in biogas coming from sewage plants. It is therefore mandatory to analyse precisely the biogas produced on a specific site, and with a specific feedstock, to build up its purification process accordingly.

2. Purification of biogas

2.1. Purification by physical means

For the purification of gases, the main physical processes (thus excluding chemical reactions) available include adsorption, absorption (physisorption), membrane separation, precipitation, refrigeration/condensation as well as different methods for the removal of particles.

Adsorption consists in a process in which different molecules adhere to the surface of a specific solid – called the adsorbent – and/or into its pores. These adsorbents can be of different nature such as activated charcoal, zeolites, silica gels, … and their properties shall be selected based on the composition of the gas to be purified. For instance, activated alumina are interesting to remove cyclic volatile siloxanes as they are not noticeably affected by the potential polymerization of these materials that would decrease its efficiency.[9] In terms of process, the selective adsorption is often performed in a pressurised system while, once the adsorbent is saturated, it can be regenerated by reducing the pressure to remove the different pollutants, such system being usually referred to as a pressure swing adsorption process (PSA). Noteworthy, this methodology is often used to upgrade biogas into biomethane, *i.e.* removing the CO_2 from the biogas. These processes are often used to dry the biogas, remove water from the mixture as well as carbonaceous compounds such as volatile organic compounds or halogenated hydrocarbons.

Physical absorption processes consist in flushing a mixture of gas into a liquid phase in which the contaminants or impurities are largely more soluble than the gas to purify. By doing so, a clean gas will leave the column while the impurities will remain, as solutes, in the sorbent. These systems are often completed by a second column for the regeneration of the sorbent, where impurities are removed, for example by heating, flushing another gas or reducing pressure. For instance, water is often used to capture highly soluble compounds such as CO_2, ammonia or – as explained later – hydrogen sulfide. Such process is often referred to as water scrubbing

Biogas upgrading by membrane is a technique based on the difference of permeabilities of different gases or species in a liquid phase through a specific material. Membranes can schematically be viewed as a porous material that would let some gas flow through its pores (for instance CO_2 and impurities) while retaining others (such as CH_4). Such processes are generally compact and enable good recovery and purification. They can, however, be subjected to fouling (*i.e.* the formation of a solid material on or within the pores that clog the whole system) or require a higher capital expenditure (CAPEX) than some other techniques. They also often require a pressurisation of the system. Different commercial membrane solutions are presently available, focusing on the upgrade of biogas into biomethane, with suppliers like Bright[9] or Suez.[11] For the latter, their Valopur® system can treat a large range of volumes ranging from a flow of 40 up to 3500 Nm³/h..[11] Gas such as O_2 or N_2, that are generally difficult to remove, can also be stripped off, taking advantage of their relatively small size.[13]

Other more traditional methods, such as condensation or filtration, can also be used for biogas filtration. For example, water can be condensed by increasing the pressure or decreasing the temperature, while particles, that are often present in the flow of gas, particles can be removed using pocket or cyclone filters.

2.2. Purification by chemical means

As opposed to physical ones, chemical purifications involve chemical reaction(s) that will transform the impurities into other species.

Impurities can thus be adsorbed or incorporated in/on a solid to lead to reactive adsorption. Different examples exist at a large scale for H_2S capture and some will be described in part 2.3 of this technical note. At a more academic level, Guo *et al.* described a protocol to capture ammonia using waste wood shaving sand and biowaste impregnated with sulfuric acid to trap ammonia.[14] NH_3 is here transformed in $[NH_4]_2SO_4$ on the surface and removed from the gas. The material can subsequently be used as fertiliser.

Another technique consists in bubbling the gas into a solution that contains a chemical that can react with one of the pollutants. This technique is widely used in the purification of natural gas where amines – such as monoethanol amine or diethanol amine – are used to react with carbon dioxide.

Finally, it is possible to directly add chemicals in the digester to trap the impurities during the synthesis of the biogas. In this case, it is important not to lower the quality of the digestate as this solid phase from the anaerobic digestion is often used as fertiliser. For example, ammonia can be precipitated under alkaline conditions out of the liquid digestate by forming struvite (*i.e.* magnesium ammonium phosphate) that is known to be a slow release fertiliser.[15]

2.3. Case study – hydrogen sulfide

As mentioned previously, hydrogen sulfide (H_2S) is one of the most problematic impurities present in biogas, due to its toxicity, smell, and potential for corrosion, therefore posing problem to equipment, people, and the environment. It is produced during the reduction process

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of the sulfur compounds (such as amino acids or sulfates for instance) by microbial action during the anaerobic digestion. Awe *et al.* provided an informative table related to the H_2S tolerance of various technologies using biogas that are summarised in Table 2 below.[13]

Table 2 Tolerance to H_2S of various technologies

Technology	**H_2S tolerance (ppm)**
Boiler and Stirling engines	< 1000
Kitchen stoves	< 10
Turbines	< 10000
Fuel cells	<1 to <20
Natural gas upgrade*	< 4

* Variation depending on the countries

Considering that untreated biogas contains 50 to 20000 ppm of H_2S, it is mandatory to work on the purification of this compound and a large panel of technologies have been employed to remove it from biogas.

Absorption processes are largely used for H_2S removal, using either water or organic solvents with or without additives *via* traditional gas-liquid contactors. Water can be used for pure physical absorption as H_2S is about 80 times more soluble in it than methane. Such system, generally used under pressure to increase the amount H_2S solubilised, can be employed with or without a regeneration loop but the latter will lead to a very high consumption of water (150L/Nm^3), while the former will decrease it by a hundred-fold.[15] Figure 2, based on the publication of Hagen *et al.*,[17] represents a process of water scrubbing which includes a regeneration loop.

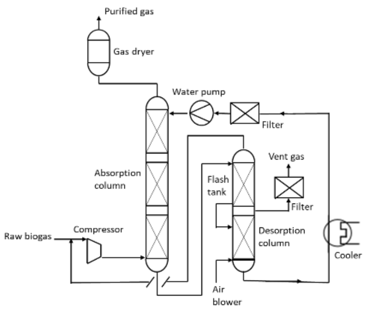

Figure 2 Hydrogen sulfide (water) scrubbing process with regeneration loop

Other solvents than water can also be used for such purifications, the most known consisting of mixtures of polyglygol dimethyl ethers (Selexol® process) or of cold methanol (Rectisol® process) in which both CO_2 and H_2S are more soluble than methane.

Chemically reactive liquids can also be used to react with hydrogen sulfide, they contain for instance Iron (II) or Iron (III) species such as Fe(II)Cl_2, Fe(III)Cl_3 or Fe(III)$(OH)_3$., implying the reactions described in the equations below.

(1) $FeCl_2 + H_2S \rightarrow FeS + 2HCl$

(2) $2FeCl_3 + 3H_2S \rightarrow FeS + S + 6HCl$

(3) $2\ Fe(OH)_3 + H_2S \rightarrow 2Fe(OH)_2 + S + H_2O$

(3)' $Fe(OH)_2 + H_2S \rightarrow FeS + 2H_2O$

Figure 3 Reactions of hydrogen sulfide with Iron compounds

These chemical scrubbing processes are highly efficient but may lead to problems due to clogging and/or foaming that must be addressed. Industrial processes relying on the redox chemistry of Iron ions include, for instance, the Sulferox process from company Shell treating from 0.1 to 20t/d[18] or the LO-CAT process from Merichem Technologies.[19] Noteworthy, iron salts (such as $FeCl_2$, $FeCl_3$ or $FeSO_4$) can also be added in the anaerobic digester, leading to the precipitation of FeS at a cheap operation cost but such a process is also difficult to control and requires specialised supervision.[13] Aqueous alkaline solutions can also be used, where potassium hydroxide will convert hydrogen sulfide into sodium (hydrogen) sulfide.[20]

In the case of adsorption, traditional systems consist of impregnated activated charcoal as well as metal oxides like Iron (III) oxide (Fe_2O_3), Zn (II) oxide or Iron (III) hydroxide. Noteworthy, metal oxides are often immobilised on a solid support to prevent aggregation from water condensation and to increase surface area.[20] As another example, carbon-based sorbents, such as activated carbons, are widely used in pressure-swing adsorption processes (PSA) due to their low production cost and large specific surface areas. They can be impregnated for example with alkaline materials like K_2CO_3, Na_2CO_3, $NaHCO_3$, KOH, NaOH, … to promote chemisorption.[21] Such systems are generally performed under pressure and higher temperature with the addition of air to support the partial oxidation of sulfur into S(0) as described below.[22]

(1) $2H_2S + O_2 \rightarrow S_2 + 2H_2O$

Oxidants such as KI or $KMnO_4$ can also be used to support this oxidation process.

For such systems, presence of water would be detrimental as it could react with CO_2, forming carbonate and subsequently promoting the formation of sulfuric acid that can deactivate the actives sites of the catalysts. Moreover, the elemental sulfur generated in the process can coat the catalyst, therefore decreasing its activity, and the disabled activated charcoal shall then either be replaced or regenerated.

Industrial adsorbents for H_2S include, amongst others, SulfaTreat (based on Iron oxides) from company Slb, DARCO® BG from company NORIT (based on activated carbon) or the AddSorb™ OX series from Jacobi that use doped activated carbons from coconut shells to convert H_2S into sulfur oxides.

Membrane solutions can also be used for H_2S abatement, with elimination rate up to 98% but remain complex solutions and are rarely implemented.[13]

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The following Table, based on Awe *et al.*,[13] briefly summarises the advantages and drawbacks of these techniques for H_2S scrubbing.

Table 3. Some advantages and drawbacks of the different techniques for H_2S scrubbing.

Techniques	Some advantages	Some drawbacks
Impregnated activated charcoal	High efficiency, compact	High operating expenses (OPEX), sensible water and O_2, costly regeneration
Iron oxides	High removal efficiency, limited capital expenditure (CAPEX)	High OPEX, exothermic regeneration, coating of surface, reagent disposal
Iron chlorides in digesters	Low CAPEX, simple operation and maintenance	Low efficiency, change pH temperature, specialized supervision
Water scrubbing	H_2S <15 cm³/m³, CO_2 removal	High pressure, low T°C, not regenerative
Membranes	High removal, also remove CO_2	High OPEX, maintenance, complex solution

Conclusion

The conversion of organic waste into biogas via anaerobic digestion represents a valuable source of renewable energy. However, the economic and operational viability of these systems will highly depend on its complex and variable composition and, subsequently, on its purification. In this technical note, various methods for treating raw biogas with impurities such as amines, siloxanes or hydrogen sulfide are briefly described. However, the selection of an optimal purification protocol is not an universal choice but clearly a project-specific decision that must be driven by a careful analysis of various data, including the specification required for the application that is targeted, the amount of biogas to purify and the scale of the biogas production facility, the composition/variability of the raw biogas and, ultimately, the overall economic feasibility of the selected process, balancing CAPEX and long term OPEX. Thus, a successful biogas process relies on a carefully engineered purification procedure that integrates the right technologies to meet specific demands.

Acknowledgments

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Certech has also the capabilities for testing purifying process at lab and pilot scale, studying the capacities of sorbent on specific pollutants (H_2S, SO_2, VOC, …).

Certech can develop new adsorbent material such as metal organic framework (MOFs) used for advanced biogas purification.

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