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ORIGINAL PAPER

Producing Biohythane from Urban Organic Wastes

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Abstract

This study investigated the advantages of the anaerobic codigestion process of two urban organic waste: the organic fraction of municipal solid wastes and the waste activated sludge produced during biological wastewater treatment. In particular, a comparison between mono and double stage anaerobic digestion for biogas and biohythane (hydrogen and methane) production, respectively, was conducted at thermophilic conditions in a pilot scale rig with a hydraulic retention time of 20 days. Considering yields, the specific gas productions for the single stage process was 490 l biogas per kg TVS fed to the system while in the two stage process hydrogen and methane productions reached average values of 24 LH_2 and 570 LCH_4 per kg VS fed to the system, respectively. Obtained biohythane, after upgrading, is particularly valuable for the automotive sector contributing to improve the combustion engine performance and to reduce the contaminants emissions in the atmosphere.

Keywords Waste activated sludge · Organic municipal solid waste · Thermophilic anaerobic digestion · Hydrogen · Methane

Statement of Novelty

At the best of authors' knowledge, few research works covered the topic of two stage anaerobic digestion for the concurrent methane and hydrogen production at pilot scale. This work offers a comparison between the performances of mono and double stage AD for methane and biohythane production from the codigestion of organic municipal solid wastes and waste activated sludge. This research remarks also how codigestion of these substrates allows for a greater biogas production than the case in which they are treated separately. Lastly, a literature comparison of the main contaminants emissions from automotive engines was conducted between the traditional used fuels (compressed natural gas, diesel and gasoline) and biohythane.

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Introduction

Europe and other Western Countries produce 1.3 billion tons of food wastes (FWs) each year. This production corresponds to about one-third of the food production and it is composed for the 45% by fruit and vegetables, for the 30% by cereals, and for another 20% by meat [1, 2]. It was estimated that this amount corresponds to some 300 g of household FW per capita per day, equivalent to some 50–60 g dry matter per person every day. Moreover, at urban scale, also waste activated sludge (WAS) is produced: considering a total COD production of 120 g per person and per day, a sludge production of 50–60 g dry matter per capita per day are expected [3]. As a consequence, more than 100 g dry matter per person per day, from FW and sludge, are produced every day in our urban areas. This amount can be conveniently treated in order to recovery both energy and nutrients, instead to be simply disposed of in landfills. In fact this waste management practice is coherent with the waste hierarchy promoted by the European Union. According to this approach, the first level of attention is directed toward the need to prevent the formation of waste. The following steps concern the reuse, the recovery and the recycling of suitable materials and afterwards the energy recover through a thermochemical or biological process. Only at the end, when there are no more alternatives, the wastes disposal in landfill is allowed [4]. In fact, landfilling should be always avoided as it is responsible for the production of both landfill leachate and greenhouse gas emissions.

One of the best option to achieve the energy recovery from organic waste is the anaerobic digestion approach. Co-digestion, in particular, is the simultaneous anaerobic decomposition of two or more organic substrates to produce methane. Several studies showed the benefits of the co-digestion approach, e.g. dilution of potential toxic compounds, nutrients balance improvement, synergistic effects of microorganisms, increased load of biodegradable organic matter and better biogas yield [5]. As reported, among the different organic substrates, WAS and the organic fraction of municipal solid waste (OFMSW) are the most abundant ones [6]. OFMSW and WAS can be codigested so to obtain a better C/N ratio within the reaction medium to guarantee optimal conditions for the microorganisms' metabolism and, consequentially, to achieve a better biogas yield. In particular, a nitrogen lack has negative effects on the formation of intracellular microorganisms' essential proteins. Conversely, a high nitrogen concentration can imply an excess in the ammonia formation which, as previously seen, is able to inhibit or stop the fermentation. The optimal reported value for the C/N ratio is considered 30 [7]. Typically, WAS presents a low C/N ratio, between 4 and 9; on contrary, OFMSW is characterised by higher values, between 20 and 40 [8].

Another advantage of the OFMSW–WAS co-digestion is the possible exploitation of existing infrastructures [9]. Currently, a total of 8 million tons of OFMSW are digested within the EU countries, while 28% of the AD plants in the industrial sector use WAS as substrate [10–12]. An interesting option is the co-treatment of OFMSW and WAS in the anaerobic digester in waste water treatment plants (WWTPs): in this way, a better exploitation of the WWTPs structures can be achieved. In fact, because of over-sizing design or the treatment of very diluted streams, these reactors are very often operating at low organic loading rate (OLR); large spare volumes are therefore available for the co-treatment of sludge and other organic waste in WWTPs [12, 13]. Anaerobic codigestion allows for the recovery of renewable energy: each ton of OFMSW sent to the anaerobic treatment, in fact, can produce up to 130–180 m^3 of biogas, depending from the quality of the treated substrate (mainly linked to the collection strategy). The biogas can be conveniently converted into useful energy forms: heat, electricity and the combined production of electricity and heat (cogeneration). The actual tendency, at European level, is to move towards an additional approach like upgrading, considering the anaerobic digestion (AD) as the base to produce a more performant biofuel to be used not only in situ (cogeneration), but also in the automotive sector [14, 15]. In this sense, biohythane, a gaseous fuel composed by 10–30% v/v of hydrogen and 70–90% v/v of methane, has received great attention in the last decades [16]. This combination presents two important advantages: the reduction of greenhouse gas emission (CO_2 and NO_x) and the improvement of the combustion efficacy [17]. Biohythane can be obtained from Anaerobic Digestion conducted in two separate stage: the dark fermentation which brings to hydrogen synthesis, followed by the methanogenic stage with the consequent methane formation [18, 19]. At the moment, the main biohythane application is the automotive for its abilities to improve the flame speed propagation in internal combustion engine [20].

The aim of the work is the comparison of the OFMSW–WAS codigestion performances between the single and the double stage AD for methane and biohythane production, respectively. The environmental advantages in the biohythane use in automotive sectors will be also exposed through its comparison with the most fuels, currently adopted in automotive sector.

Materials and Methods

Experimental Set-Up

Two experimental campaigns were conducted to compare the performances of single stage AD for biomethane production and two stage AD for biohythane production, represented in Fig. 1.

Three continuously stirred reactors (CSTR) with working volumes of 230 l, 200 l, and 760 l, respectively, were used in the experimentation. The one stage AD was realized in the 230 l reactor, while the 200 l reactor was used for the dark fermentation (first phase of two stage AD) and the 760 l one for the methanogenic step (second phase of two stage AD). The reactors were heated by a hot water recirculation system and maintained at 55 °C using electrical heater controlled by a PT100-based thermostatic probe. The feeding system was semi-continuous, arranged once per day.

Before feeding, the organic waste was reduced in size using a 2 mm grinder, mixed with WAS and then fed to the single stage reactor (230 l) or to the first stage reactor (200 l fermenter) of the two stage system. The tests had a duration of 365 days. The OLR and the Hydraulic Retention Time (HRT) was of 17 kg TVS/m^3d and 3 days, respectively, for the first phase of the two-stage AD and of 3.5 kg TVS/m^3d and 17 days, respectively, for the second phase of the two-stage AD. Thus, the overall HRT for the two stage AD was of 20 days. With specific reference to the single stage process, the OLR and the HRT were 3.5 kg TVS/m^3d and 20 days, respectively. The HRTs of the two systems were therefore equivalent and obtained results are comparable. HRT

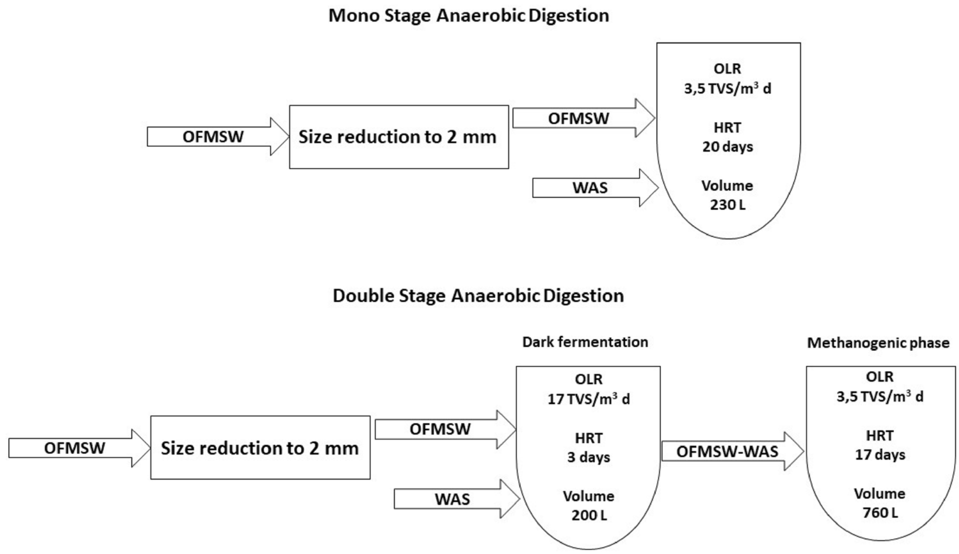

Fig. 1 Scheme of the mono and two stage anaerobic digestion, conducted in pilot scale with OFMSW and waste activated sludge

and OLR, together with pH, are very important parameters for the hydrogen and methane productions in continuous mode. Very low HRT comports the wash out of the reactor, which means that all the active microorganisms escape out from the reactor. On contrary, through high OLR it is possible to cause a decrease of pH as consequence of the overloading of the reactor. These conditions favour an overproduction of organic acids and of hydrogen [21, 22].

The OFMSW/WAS ratio in the feedstock was determined considering the typical productions of 250 g per person per day for biowaste at 25% dry matter, therefore equivalent to some 60 g per person per day dry matter, and 60 g dry matter per person per day for excess sludge. The OFMSW/WAS ratio adopted in this study was therefore 1/1 on a TVS basis. Taking into account this ratio, the TS and TVS concentrations in the influent mix were about 155 g/kg and 130 g/kg, respectively.

Substrates and Inoculum

The inoculum for the reactors was collected in the WWTP located in Treviso (northern Italy) from a 2000 m^3 mesophilic anaerobic digester, treating the WAS used in this research work. The inoculum was then acclimatized in the three reactors for two months to the thermophilic temperature (55 °C) [11]. The substrates for the codigestion were OFMSW from the municipality of Treviso and WAS from Treviso WWTP, adequately mixed to obtain the OLR reported in the previous paragraph.

Table 1 shows the main characteristics of these two substrates.

WAS (Table 1) showed an average concentration of 47 g/kg and a VS content of 69%. N and P contents were 3.2 and 0.9 g/kg, respectively. These characteristics can be considered typical for this substrate.

Table 1 Waste activated sludge and OFMSW characteristics

Parameters	Waste activated sludge	OFMSW
TS (g/kg)	47.86 ± 14.28	259.9 ± 38.80
TVS (g/kg)	33.06 ± 10.10	226.1 ± 41.30
TVS/TS	69.06 ± 4.10	90.7 ± 2.58
COD (g/kg)	51.30 ± 11.50	241.3 ± 48.90
TKN (g/kg)	3.29 ± 0.75	6.7 ± 1.30
P_{TOT} (g/kg)	0.91 ± 0.33	1.5 ± 0.70

The OFMSW used in the experimental trials was characterized by an average content of total solids approximately at 25% and a fraction of volatile solids of 90% over TS (Table 1). These values indicate the high content of biodegradable organic matter, which presents a balanced ratio of macronutrients (COD:TKN:P), making OFMSW a very suitable substrate for biological treatment processes. In particular, the COD/TKN ratio for the mix of OFMSW and WAS turned out to be on average equal to 36, more than twice that of the WAS considered and within the optimal range for microorganisms metabolism [9].

Analytical Methods

The effluents of the reactors were monitored 2/3 times per week in terms of total and volatile solids content, chemical oxygen demand, Total Kjeldahl Nitrogen (TKN) and total phosphorus. The process stability parameters, namely pH, Volatile Fatty Acid (VFA) content and speciation, total and partial alkalinity and ammonia, were checked daily. All the analyses, except for volatile fatty acids (VFAs), were carried out in accordance with the standard methods [22]. VFAs content was monitored using a gas chromatograph (Carlo Erba instruments) with hydrogen as gas carrier, equipped with a Fused Silica Capillary Column (Supelco NUKOL, 15 × 0.53 × 0.5 µm film thickness) and with a flame ionization detector (200 °C). The temperature during the analysis started from 80 °C and reaches 200 °C through two other steps at 140 and 160 °C, with a rate of 10 °C/min. The analysed samples were centrifuged and filtrated on a 0.45 µm membrane. Gas productions were monitored continuously by a gas flow meter (Ritter Company, drum-type wet-test volumetric gas meters), while the biogas composition was daily recollected in a 0.5 l bag and measured by a gas-chromatograph (GC Agilent Technology 6890 N) equipped with the column HP-PLOT MOLESIEVE, 30 × 0.53 mm ID × 25 µm film, using a thermal conductivity detector and argon as gas carrier.

Results and Discussion

Digestate Characterizations

The characteristics of the final digestate were very similar in the two studied processes (Table 2): the total and volatile solids were about 25 and 15 g/kg, respectively. These values corresponded to a TS and TVS reduction of about 84% and 88%, respectively, both for the single stage AD and for the two stage AD processes. These values demonstrated the good efficiency of the AD process in the conversion of the organic material into biogas. The residual organic material in the digestate is probably due to the heterogeneous nature of the OFMSWs composed both by easily degradable (fruits, pasta, rice) and by more recalcitrant compounds, contained in vegetables, mainly in paper wastes. These last ones are characterized by high content of lignocellulosic materials which are only partially degraded by microorganisms and require longer retention times [23, 24].

N and P contents were 35 and 10 g per kg dry matter, respectively. These values are coherent with N and P digestate values available in literature, making it interesting for agricultural application as fertilizer, after opportune upgrading processes [25]. In particular, the liquid phase of digestate was rich in ammonia, which can be evaporated and treated with sulfuric acid to produce ammonia sulfate, recognized to be a fertilizer. On the contrary, struvite fertilizer can be recovered from phosphorous compounds in solid phases by magnesium salts additions [26].

Single and Two-Stage AD Comparison

The differences between the single and the two-stage AD processes were more evident considering their overall performances (Fig. 2, Table 2). Figure 2 shows the average values of Specific Methane Production (SMP) which were 0.49 Nm^3 CH_4/kg TVS for the single stage AD, and 0.57 Nm^3 CH_4/kg TVS for the methanogenic reactor (second stage of double stage AD). In addition, 0.024 Nm^3 H_2/kg TVS are also produced along the first stage of double stage AD. More details are provided in Fig. 3. It shows the volumetric biogas composition for the last part of the double stage AD test (days 250–365), when a quasi-steady state condition was reached. Hydrogen concentration is refereed to the first stage of double stage AD, while methane and carbon dioxide content are referred to the second stage, the methanogenic one. From Fig. 3 it was evident the good performance of the two stages, with a hydrogen content between 10–15% v/v, and a

Table 2 Digestate characteristics and hydrogen and methane specific yields

Parameters	Units	First phase	Second phase	Single stage
Total solids	g/kg	48 ± 5	25 ± 4	26 ± 2
Total volatile solids	g/kg	37 ± 4	16 ± 2	17 ± 1
COD	g/kg TS	40 ± 3	19 ± 2	18 ± 2
TKN	g/kg TS	34 ± 1	35 ± 1	34 ± 1
P tot	g/kg TS	11 ± 0	12 ± 1	10 ± 1
pH		5.3 ± 0.01	8.2 ± 0.5	8.0 ± 0.26
VFA	mgCOD/l	10,631 ± 1628	258 ± 114	301 ± 102
Yields				
Hydrogen	%	36 ± 8	–	–
Methane	%	–	64 ± 2	60 ± 9
Specific hydrogen production (SHP) First phase of double stage AD	Nm^3 H_2/kg TVS	0.024 ± 0.05	–	–
Specific methane production (SMP) Second phase of double stage AD	Nm^3 CH_4/kg TVS	0.009 ± 0.005	0.57 ± 0.01	
SMP Mono stage AD	Nm^3 CH_4/kg TVS			0.49 ± 0.04

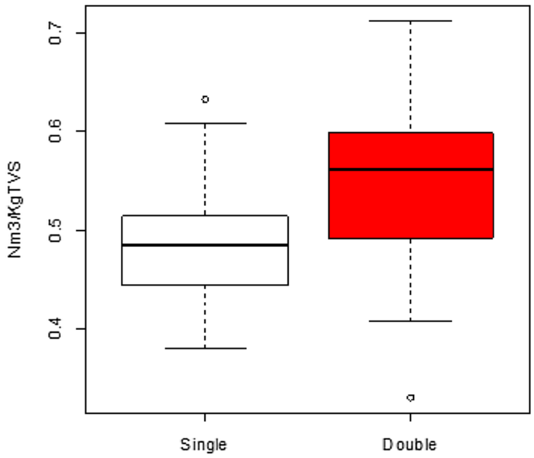

Fig. 2 SMP obtained in single and two stage anaerobic digestion

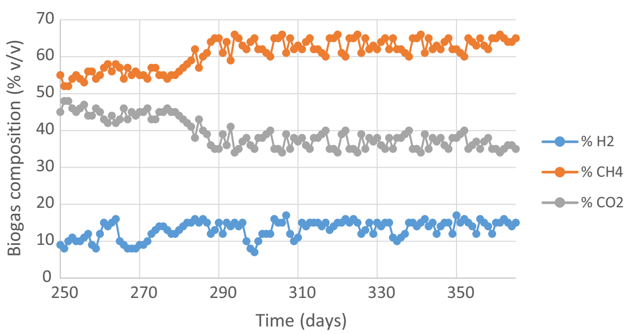

Fig. 3 Biogas composition of the two stage AD (days 250–365)

methane which reached high level (60–65% v/v), mainly in the last 80 days of the test.

Instead, Table 2 recorded the average values of all the most relevant parameters, referred to all the duration of the single and double AD tests. Table 2 shows a low methane production (0.009 Nm^3/kg TVS) along the first stage of double stage codigestion, when hydrogen is mainly produced. Low methane production along first stage of double stage codigestion is not usual and it is due to the presence of some methanogens, which survived to high OLR and short HRT. The performances in terms of biomethane production were better for the two stage AD, demonstrating that dark fermentation had a crucial role to increase the OFMSW conversion into biogas and biomethane. This was due to a better degradation of the organic matter [27] promoted by the physical separation of the two distinctly different groups of bacteria (acidogens and methanogens) along the double stage AD: the separation allows to maximize their growth by maintaining the optimum conditions in each tank. The first group, acidogenic bacteria, is grown in an acidogenic reactor where the pH is naturally low and the residence time is between 1 and 4 days. The second group, methanogenic bacteria, need for a higher pH and larger residence time (15–20 days) [28].

Table 3 shows how HSP and MSP depend essentially on the feedstock nature. It is evident that reported HSP and MSP are usually lower than those found in our study, where WAS and OFMSW have been treated in codigestion mode. WAS, when used as single substrate, showed the lowest biogas productions due to the low biodegradability and C/N ratio with consequent high ammonia formation during fermentation [5]. Ammonia, in fact, can cause inhibition of the AD, in particular of the methanogens during the second stage of AD when its concentration is greater than 3 g/l. On the contrary, it was demonstrated that small additions of OFMSW in WAS improved methane production to 40% for the abundance of simple sugars and carbohydrates, such as lignocellulose. In addition, OFMSW which is characterised by low proteins contents determines low ammonia concentrations and can prevent the fermentation inhibition [31]. Some studies concluded that there is no indication of methanogenic failure in the co-digestion of OFMSW and WAS at any ratio [34].

Table 3 Hydrogen specific production (HSP) and methane specific production (MSP) from OFMSW and waste activated sludge (WAS) as single substrates

Test description	HSP (Nm^3/kg TVS)	MSP (Nm^3/kg TVS)	References
OFMSW treated at thermophilic conditions in both phases. Variable OLR during test	0.27	0.29	[29]
OFMSW treated at thermophilic condition with a HRT of 3 days for dark fermentation and 12.5 days for the methanogenic phase	0.05	0.41	[30]
OFMSW treated at thermophilic condition with a HRT of 3 days for dark fermentation and 12.5 days for the methanogenic phase with recirculation to first stage	0.22	0.71	[31]
WAS treated at thermophilic condition (60 °C) with HRT of 6 days for dark fermentation and 18 days for methanogenic phase	0.08	0.31	[32]
WAS treated at mesophilic conditions	0.75	0.19	[33]
OFMSW—WAS codigestion	0.024	0.57	This work

Lastly, the beneficial performances of biohythane production was also demonstrated from the environmental point of view. Different LCA studies showed that biohythane can guarantee lower CO_2 emissions than the mere methane production since hydrogen presence contributes to reduce by 10% CO_2 emissions. However, no particular improvements were observed in the case of OFMSW and WAS co-treatment with respect to the treatment of OFMSW and WAS singularly. These cases, in fact, exhibited similar reductions of CO_{2eq}, amounting to about 40 tons for the entire life cycle of the reactors [5].

Comparison Between Biohythane from Two Stage AD and Traditional Fuels Used in the Automotive Sector

Vehicles are important contributors of the air pollution, mainly in the major urban areas. The most important parameters used to determine the air quality are concentrations of CO_2 (g equivalent), CO (%), NOx (ppm), SO_2 (ppm) and Hydro-Carbons (HC) (ppm) emissions from vehicles. The contaminants emissions from the automotive sector depend on several factors: (i) the fuel adopted for the engine combustion, (ii) the vehicle typology, (iii) the presence of catalyst able to capture contaminants and (iv) operative temperature of the engine (usually cold engines have higher emissions) [35].

In this paragraph we compare the emissions from biohythane produced in this study and the emissions of the most used fuels in the automotive sector, namely, gasoline, diesel and compressed natural gas (CNG). Table 4 shows typical emissions from the three previously reported fuels. The values are an average of contaminants emission calculated from different vehicles typologies (cars, buses, trucks, and motorcycles).

The worst CO emissions derive from gasoline and CNG which have comparable emissions. On the contrary, diesel release the lowest CO amount in the atmosphere. This is explicable considering that diesel contain more energy per litre and, in addition, diesel engines are more efficient than petrol ones [35]. However, the diesel engine combustion contributes to large amount releasing of noxious substances, such as nitrogen oxide (NOx), SO_2, HC and particulate matter (PM). Kang et al. [36] found that diesel combination with propane helped to improve the engine performance and to reduce the NOx and PM concentrations. It is important to underline that gasoline engines provided with a catalyst have much lower CO, HC and NOx emission [37].

Table 4 Main vehicles emissions from the most used conventional fuels

	CO (%)	NOx (ppm)	SO_2 (ppm)	HC (ppm)
CNG	1.63	40	25	1150
Gasoline	2.1	45	30	3500
Diesel	0.19	85	120	19,500

CO and NOx emissions from CNG are of the same order as those emitted from gasoline vehicles. HC emissions are also lower than gasoline and diesel engines because of the lower carbon content than other fuels: carbon fraction in methane is 0.75, while for diesel and gasoline is 0.85 and 0.90, respectively [38]. CNG reduces also SOx releasing in air due to absence of sulphur compound in methane.

Among the alternative fuels for automotive sectors, biodiesel represents the most studied by scientific community nowadays. The absence of sulphur and aromatic contents, renewability and biodegradability and 30–71% lower greenhouse gas emissions represent the most relevant advantages of this biofuels. Moreover, the use of biodiesel fuel can be accomplished by little or no modification on the diesel engine. Anyway, biodiesel technical performances are lower than diesel ones, presenting poor storage stability and cold flow properties, inferior spray characteristics and lower heat content [39].

In the last decades biohythane from two stage AD has received great attention as alternative vehicles fuels for its great potential in combustion engines and the ability to reduce contaminants emissions in atmosphere. In fact, the obtained biogas can be upgraded and after CO_2 removal either biomethane or biohythane can be obtained [31].

Biohythane, being a hydrogen–methane blend obtained from two stage AD of clean organic biomasses, have several environmental benefits contributing to reduce CO_2 equivalent amount and the NOx release in the atmosphere [40, 41]. Conventional diesel fuel presents the following environmental impacts: 139 g/kg CO_2, 0.085 g/kg NO_X, 0.018 PM g/kg. When biomethane is used the emissions are lowered to the following levels: 108.68 g/kg CO_2, 0.045 g/kg NO_X, 0.017 PM g/kg with a clear benefit in terms of the emissions. Finally, when biohythane was used as biofuel it could be calculated that emissions for CO_2 and particulate matter (PM) remained the same found for the use of biomethane while the NOx level was further decreased down to 0.036 g/kg [42].

Hydrogen addition in methane blends can improve the performance of internal combustion engines, usually fed by methane from fossil sources. It reduces the methane number, which is expressed as percentage of methane in the biohythane and it is related to the knock resistance. Furthermore, the hydrogen lower ignition energy in air respect to methane (0.02 mJ vs. 0.29 mJ, at stoichiometric conditions) helps to burn better, but make the mixture subject to pre-ignition by contact with hot spots or residual gases. The turbulent flame speed propagation's increasing in internal combustion engine is achieved when hydrogen is added to methane, in accordance to the stoichiometric laminar speed, which is 1.9 m/s for hydrogen and only 0.3 m/s for methane. Moreover, biohythane offers the possibility to expand the lean burn limit, because of a more stable combustion [20].

Hythane has been tested for the first time in automotive sectors in Montreal in 1995. The project, the Montreal Hythane Bus Project, used hythane having 10% v/v of hydrogen to fed some buses allowing of 45% decreasing of the NOx emission. In 2008 the Italian research center ENEA projected a 8 m long bus, whose performances were evaluated changing the hydrogen concentration, from 5 to 25% v/v. The research experience confirmed the reduction of hydrocarbons and CO emissions decrease [5]. More recently, such automotive companies (Toyota, Fiat) projected the first car adapted to be hythane [42].

The major obstacle to a largely biohythane adoption is represented by the gas distribution system. Although, methane distribution system is already consolidate in different countries, hydrogen presence in biohythane requires some modifications in the pipelines. Thus, national incentives could help in the transition, mainly in Countries where CNG cars constitute a developed market, as in Italy.

Conclusions

In this study WAS and OFMSW were codigested in thermophilic conditions both in single and two stage configuration. Results showed that the single stage process produced on average 0.49 Nm^3 CH_4 per kg TVS fed to the reactor while the two stage process produced 0.57 Nm^3 CH_4 per kg TVS fed in the methanogenic reactor (second stage) and 0.024 Nm^3 H_2 per kg TVS fed in the first reactor. The use of biohythane in the automotive sector clearly decreased the potential impacts on air quality when compared with emissions from the use of conventional diesel, with a reduction of about 45% of the NOx emissions and of 10–20% of the CO emissions, according to the hydrogen concentration present in the biohythane.

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