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Effect of steam flow rate on biological sludge gasification

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Abstract: One of the major environmental issues in industrialized countries is the sustainable disposal of the biological sludge that is daily produced in wastewater treatment plants. This sludge contains a significant amount of non-biodegradable carbon which cannot be utilized in anaerobic digestion. This work explored the feasibility of the thermal decomposition of biological sludge in a steam rich environment and investigated the effect of the steam flow rate on the efficiency of the process. The sludge moisture can be used to replace the water added by the steam reducing the energy consumption of the process. A series of experiments were carried out for the same amount of dried biological sludge at 800 °C under three steam flow rates 4, 9, 16 mL h⁻¹ in a bench scale semi-batch unit. The gas produced under 9 mL h⁻¹ steam flow rate by sludge gasification had the highest H₂ concentration (63.4%), while the highest concentrations of CH₄ and CO were observed in the gas product of sludge gasification under 4 mL h⁻¹. According to carbon mass balances, the sludge carbon was effectively converted into gas products. It was also proved that the water gas shift reaction was promoted at lower temperatures as the steam flow rate increased. In conclusion, the findings highlight the significant influence of steam flow rate on the quality and quantity of gas obtained from biological sludge gasification. This study suggested a relative simple experimental procedure which can be utilized to determine the best suitable moisture content of the raw material.

Keywords: biological sludge; gasification; steam flow rate; syngas

1. Introduction

It is widely recognized that environmental problems have attracted growing interest in recent years. At the same time, these issues require management technologies that promote the coexistence of environmental sustainability and profitability. Among the challenging tasks in wastewater management, special attention is paid to the treatment of sewage sludge due to its high quantity in comparison with the other constituents removed from the plant [1]. Sludge is a residual by-product generated during the primary, secondary and tertiary treatment [2]. Apart from the organic matter, sewage sludge usually contains, nutrients, heavy metals,



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pathogens and toxic compounds [3]. The characteristics of sludge in the wastewater treatment plant depend on the type of plant and its operation method [2]. It has been reported that the quality of sewage sludge can be affected by several processes during the treatment such as screening, comminuting, thickening, anaerobic or aerobic digestion, chemical or thermal conditioning, dewatering and final treatment [4]. A secondary municipal sewage sludge generally contains higher protein and phosphorus content (32%–41% of total solids (TS) and 2.8%–11% of TS, respectively) than that of a digested primary sludge. On the other hand, the latter contains higher amount of TS (6%–12%) [2,4,5].

In the last few years, the disposal of sewage sludge has been changing. Previously, sludge was disposed of in the sea or applied directly as agricultural fertilizer [6], while alternative methods were burial in landfills or and it was incinerated. Since 1998, the disposal of sewage sludge in the sea is considered environmentally hazardous and as a result is forbidden by European legislation (UWWTD), landfilling is being phased out [2]. Although agricultural use remains the primary method of sludge disposal accounting for 42% of sludge utilization, followed by incineration (27%) and landfilling (14%), there has been a growing interest in recent years in utilizing sludge in various industrial sectors, including energy recovery and adsorbent preparations, which make up 17% of sludge utilization [7].

The new strategies that promote sustainability in the field of sludge management include: hydrothermal carbonization, pyrolysis, wet oxidation, gasification and combustion of sewage sludge for further use as an energy source [8,9]. Among these strategies, pyrolysis and gasification processes have gained significant attention since they greatly decrease the amount of sludge, reduce requirements for off-site transportation and offer the possibility of removing or even eliminating organic contaminants [10]. By introducing controlled streams of a gasifying medium such as air, oxygen or steam into the pyrolysis process, the products can undergo further refinement resulting in the production of syngas which can then be either combusted on-site or utilized as a source of energy [10].

Gasification is a process that converts low-value hydrocarbon materials, into a synthesis gas (syngas), that is suitable for use in the production of: electricity, transportation fuels, chemicals, and hydrogen [11–13]. The decomposition of organic matter to a gaseous fuel occurs at high temperatures and adding oxidizing reactants. During the gasification process, usable chemical energy is transferred from the feedstock to the gaseous fraction and a high yield of combustible gas is obtained [12].

Gasifying agents such as oxygen, steam and air, can affect the composition of syngas [14]. It has been reported that the gas produced during the air gasification of sewage sludge, contained a significant amount of nitrogen thereby reducing the energy content of syngas [15]. A recent research conducted by Quan *et al.* [16], provided insights into the technical aspects involved in gasification and combustion processing of sewage sludge. The most efficient method, with the maximum energy efficiency of 37.1%, was found to be the combination of air gasification and external fired gas turbines (EFGT) without carbon capture. Some studies suggest the steam gasification of sludge, since it improves the quality of syngas. Nipattummakul *et al.* [17] who examined the thermochemical conversion of sewage sludge through steam gasification, observed a 40% increase in the mole percentage of hydrogen

compared to air gasification. Moreover, except for gasifying agent, temperature and gasifying agent flow rate have a great impact on the syngas production. According to Chen *et al.* [18] who studied the gasification of sewage sludge in supercritical water, the gasification efficiency, the carbon gasification efficiency and the hydrogen yield potential under a temperature of 750 °C could reach the respective values of 73.49%, 61.16% and 41.34%.

Although sewage sludge can be used in the gasification process for producing energy with low emissions, more research is needed to improve the technical reliability [19]. The high moisture and ash content of sewage sludge can result in operational challenges, such as slagging in gasification systems [20]. In addition, the evaporation of sludge moisture increases the energy consumption during gasification [21]. However, by applying high temperature, splitting of the water associated with the sludge can occur resulting in an increase in hydrogen production [22]. Therefore, gasification process requires careful control of process parameters such as temperature, gas flow rate and retention time. Achieving optimal conditions for sewage sludge gasification can be challenging, and deviations from ideal operating conditions can contribute to process instability. Since the quantity of steam has a crucial role in gasification, the effect of steam to carbon ratio on simulation gasification yields have to be investigated. The moisture content of the sludge contributes to the overall steam content during the gasification process. As the sludge is heated, the moisture within it turns into steam, which reacts with the carbon in the sludge, promoting the gasification reaction. The presence of moisture can enhance the gasification process by increasing the availability of steam, which aids in the conversion of the carbonaceous material into syngas. Once the flow rate and efficiencies have been determined, the appropriate moisture content of the initial raw material can be calculated. This will facilitate the integration of the wastewater treatment plant and the gasification plant.

The present research investigated the feasibility of the thermal decomposition of biological sludge in a steam rich environment. Samples were pyrolysed and the intermediate products were gasified, in a single stage, under different flow rates of steam. The effects of steam flow rates on the efficiency of sludge thermal degradation either as syngas quantity and quality or as energy utilization were investigated. The moisture content of wet sludge was defined by the experimental results, as the necessary steam flow rate during gasification, for the targeted conversion efficiency or hydrogen production. This study suggested a relative simple experimental procedure which can be utilized to determine the best suitable moisture content of the raw material.

2. Methods

2.1 Apparatus

A bench scale semi-batch unit was used (Figure 1) in order to study the influence of the steam flow rate on the gasification yields. The unit can be divided into three sections: a) carrier gas/steam input, b) reactor and c) tar collection unit. Steam was generated with the introduction of water into a heated coil of stainless steel tube (ANSI 316) with an internal diameter of 6 mm. The flow of steam was regulated by the water supply of a peristaltic pump.

Nitrogen was used as the carrier gas, which was passed through the heated coil. The reactor used was a cylindrical stainless steel (ANSI 316) vessel with a capacity of 502 mL, an internal diameter of 8 cm and height of 11 cm, that contained a thermocouple. The inlet of the gases was at the flange bottom and the outlet was mounted on the lid. The tar was collected using a heated quartz filter and a battery of impinger bottles with isopropanol that were cooled in an ice bath. After each experiment, the tar that collected was analyzed gravimetrically, according to Maniatis [23].

The non-condensable gas product was continuously recorded using a wet gas meter (Ritter Gas meter Drum type TG01). Samples of the gas were frequently collected using 2 L tedlar bags. The composition of the gas was analyzed by gas chromatography (HP Agilent 5890) using a packed column (Hayesep 100/120 mesh, 9 m, 1/8a) suitable for syngas and a thermal conductivity detector (TCD). The carrier gas (Argon) flow rate was constant at 30 mL min⁻¹.

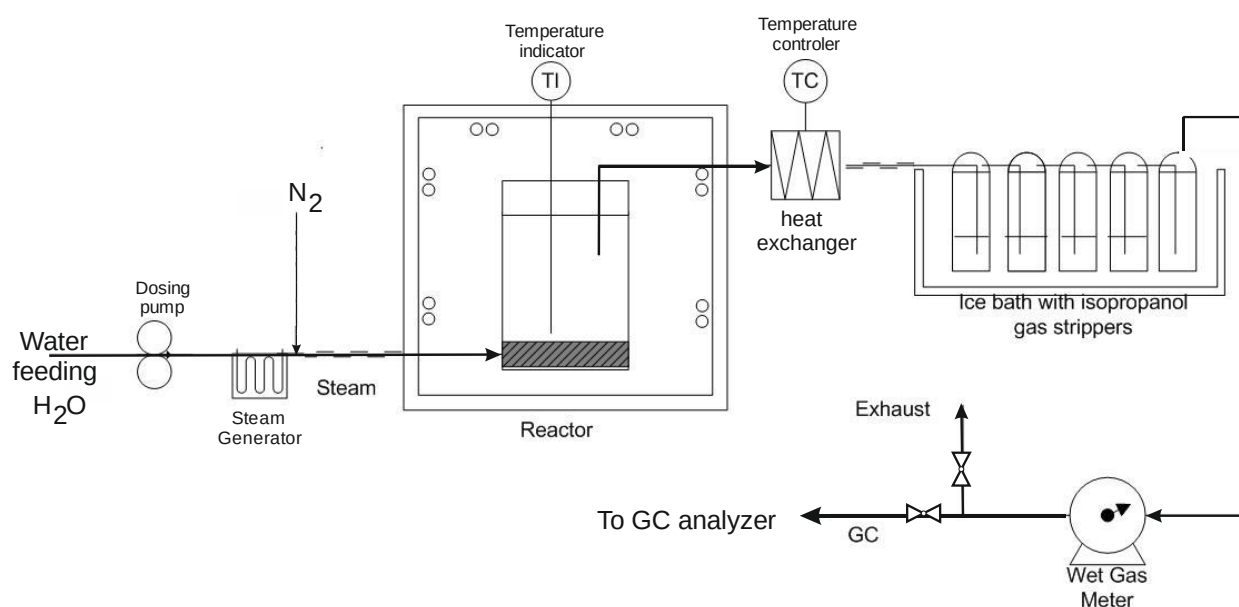


Figure 1. Bench scale gasification reactor.

2.2 Raw material

The biological sludge used in all experiments was collected from a laboratory pilot scale aerobic bioreactor. The latter was operated under a constant hydraulic retention time of 20 d, daily organic load of 20 gC L⁻¹ d⁻¹ and was fed with synthetic milk wastewater prepared by diluting fat-free fresh milk. The main chemical characteristics and inorganic elements of the examined dried sludge are presented in Table 1 and Table 2 respectively.

Table 1. Chemical characteristics of sludge.

Moisture (%wt)	Ash ^a (%wt)	VM ^a (%wt)	LHV kcal kg ⁻¹	Organic Fraction ^b (%wt)			
				C	H	N	O ^c
6.2 ± 0.18	35.6 ± 0.76	68.2 ± 1.98	2900 ± 93.09	47.9 ± 1.12	8.2 ± 0.23	13.2 ± 0.42	30.7 ± 1.32

VM, (Volatile matter); LHV (Lower Heating Value); ^aDry basis; ^bDry ash free basis; ^cCalculated by difference.

Table 2. Inorganic element composition (%wt as received).

Na	Mg	P	K	Ca	Fe	Zn
1.09 ± 0.03	2.49 ± 0.08	18.07 ± 0.70	5.6 ± 0.24	18.84 ± 0.74	2.32 ± 0.06	1.66 ± 0.04

2.3 Experimental procedure

Experiments were performed for the same amount of dried activated sludge at 800 °C with three steam flow rates 4, 9, 16 mL h⁻¹ and every set of experiments was performed in triplicate. Ten grams of dried sludge in volatile matter equivalent were placed in the reactor at room temperature. After a purge of nitrogen, in order to ensure an oxygen free atmosphere, the oven was set to the temperature of the experiment (800 °C). The heating rate was kept constant at 14 °C min⁻¹. The steam flow started when the temperature inside the reactor exceeded 110 °C. The volatiles evolved, passed through the tar collection unit. The sludge was left at the maximum temperature with constant steam flow until all the organic fractions were converted to gas. The end of the experiment was marked by the reduction of outlet gas flow rate and by the elimination of carbonaceous components in it. The temperature inside the reactor and the volume of the outlet gas were constantly monitored. Gas chromatography was used to analyze 12 gas samples collected during each experiment.

The standard ASTM D 1102-84 method was followed for the measurement of moisture and ash percentage (Reapproved 1990). For the measurement of volatile matter, the standard ASTM method D 3175-20 was used. Phosphorus was measured according to Std Methods 424e. The EUROVECTOR EA 3000 elemental analyzer was used for the elemental analysis. The standard ASTM method D 5865-07a was used for the calorific value measurement.

3. Results

The influence of steam flow rate on the yield of the fractions and on the gas composition during gasification of biological sludge are shown in Table 3. The experiments were named SX where X is the steam flow rate in mL h⁻¹. From the table's data, it can be observed that the ash and tar production were independent of the steam flow rate and they were characteristics of the raw material, which is in good agreement with the literature [12,24]. The gas production was 12.04, 12.77 and 14.91 L for the sludge gasification under 4, 9 and 16 mL h⁻¹ steam flow rate respectively. The higher the steam flow rate, the higher the gas production as far as its volume is concerned. According to the percentages of the gas

components (Table 3), the gas produced by sludge gasification under 9 mL h^{-1} steam flow rate had higher H_2 concentration, while the highest CO_2 production was measured in the syngas of sludge gasification under 16 mL h^{-1} . It has been proved that the steam gasification enhances the hydrogen production. Nipattummakul *et al.* [25] have reported that the increase in steam flow rate from 3.10 to 7.75 g/min during steam gasification of palm trunk waste, reduced the duration of gasification and led to an increased hydrogen yield. In addition, Wang *et al.* [26] investigated the hydrogen yield obtained from sewage sludge after alkaline pyrolysis in combination with catalytic gasification under different steam flow rates and temperatures. Their research revealed that the addition of steam improved the hydrogen production and a steam flow rate of 5 mL h^{-1} led to the highest H_2 production ($34.23 \text{ mmol g}^{-1}$ with a hydrogen purity of 81.5%). However, a higher steam flow rate of 10 mL h^{-1} decreased the production of hydrogen due to the endothermic catalytic reaction that is affected by temperature. Moreover, in agreement with Wang *et al.*, the highest concentrations of CO and CH_4 in present study, were observed in the gas product of sludge gasification under 4 mL h^{-1} . The variations in the gas composition can be attributed to various chemical reactions that take place during the experimental time course and during the temperature rise. As it has been mentioned, within a specific range of flow an elevation in the flow rate changes the equilibrium of the water gas shift reaction in the direction of hydrogen production promoting the steam reforming reaction of alkanes [26].

Table 3. Fraction yields (%wt.) and gas composition (%vol) from gasification of sludge as a function of steam flow rate.

Experiments	S4	S9	S16
Steam flow rate (mL h^{-1})	4	9	16
Ash (%wt.)	36.4 ± 1.29	35.1 ± 0.95	35.1 ± 1.34
Tar ^a (%wt.)	13 ± 0.43	13.6 ± 0.38	13.6 ± 0.53
Gas ^a (%wt.)	50.6 ± 1.75	51.3 ± 1.49	51.3 ± 1.94
Gas Composition ^b			
H_2 (%vol)	59.4 ± 2.45	63.2 ± 2.51	61.4 ± 2.60
CO (%vol)	22.9 ± 0.90	19.3 ± 0.73	15.8 ± 0.77
CH_4 (%vol)	1.7 ± 0.06	1.4 ± 0.06	1.4 ± 0.06

^adry basis; ^bNormal state of volume (m^3).

In order to clarify these reactions, Figures 2–4 present the flow rate of gas components during S4, S9, S16 experiments respectively. From these Figures, it is clear that the maximum production rate of CO was observed at different times and consequently different temperatures. For experiment S4 the maximum CO production rate was 0.65 L h^{-1} at 793°C and 1.8011 h, while for S9 it was 0.57 L h^{-1} at 783°C and 1.4341 h and for S16 it was 0.46 L h^{-1} at 770°C and 1.088 h. Thus, it can be concluded that the higher the steam flow rate, the lower the temperature of the maximum CO production rate is. The latter can be attributed to the water gas shift reaction (equation 1). In experiments with a higher steam flow rate, vapor

(one of the reaction reactants) was available at a higher concentration in the reaction vessel and thus the water gas shift reaction occurred at lower temperatures. In other words, the occurrence of steam promotes the water gas shift reaction, resulting in higher levels of H_2 and lower concentrations of CO as the temperature increases [27]. Furthermore, it has been mentioned that if there is an abundance of water during steam gasification, the oxidation of CO is promoted, leading to a higher CO_2 production [22].

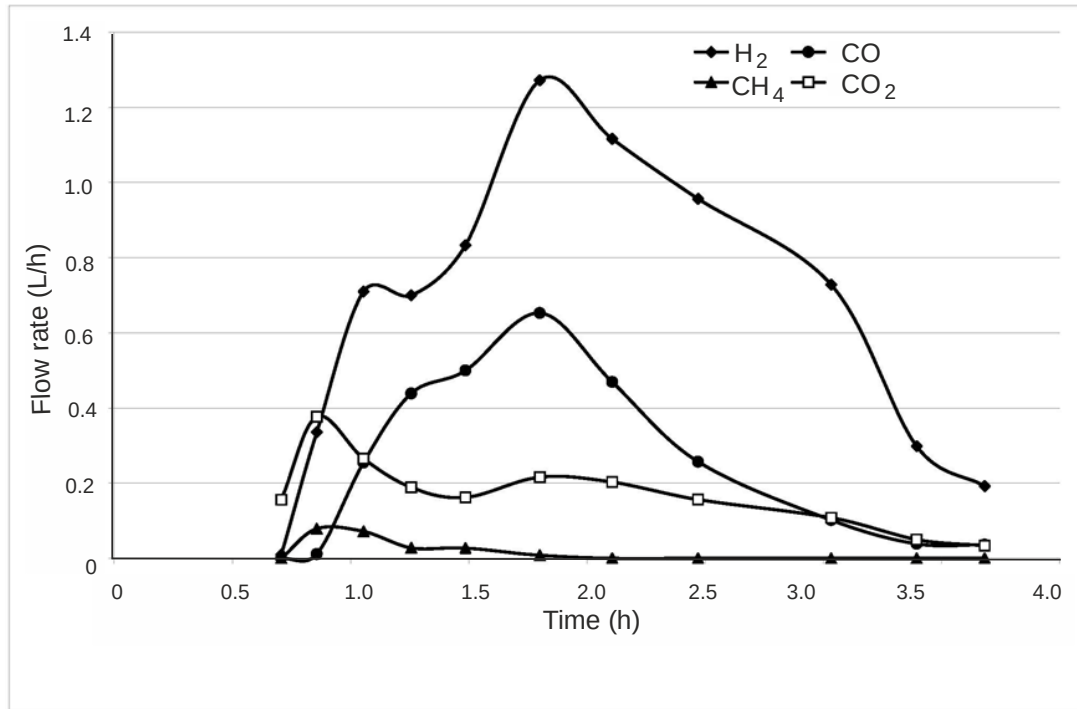
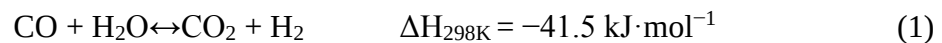


Figure 2. Flow rate of gas components during biological sludge gasification under steam flow rate 4 mL h^{-1} .

Water-gas shift reaction:



From Figures 2–4, it is clear that CH_4 production was maximized for all experiments very early. According to the literature, methane production indicates that the first pyrolysis stage took place and methane release indicates that the gasification process started [28]. It has been proved that an increased steam flow rate promotes the formation of CH_4 and the composition of gas can be improved (low hydrogen per carbon monoxide ratio) [29]. However, the excess steam during gasification can reduce the temperature leading to reduced gas production and formation of more tar [30]. In this context, every experiment was separated into two stages, the pyrolysis stage, where methane was detected and the gasification stage. The carbon mass balances for each experiment revealed the efficiency of each stage in mass carbon terms (Table 4). The efficiency of each stage was calculated as the percentage of initial carbon in the sludge that had been incorporated into carbonaceous components either in the tar or in the gaseous products.

It is noteworthy that in terms of industrial applications, gasification is also of practical significance. If wet sludge could be used to produce syngas, the amount of energy consumed by sludge dewatering would be reduced. There is a theoretical basis for simulating the pyrolysis process of wet sludge using dry sludge with a steam flow rate [26]. Peng *et al.* [31] conducted co-gasification of wet sewage sludge and forestry waste, utilizing the moisture present in the sewage sludge as the gasification agent. The findings indicated that the highest levels of H_2 and CO content were achieved when the wet sewage sludge content was at 50%.

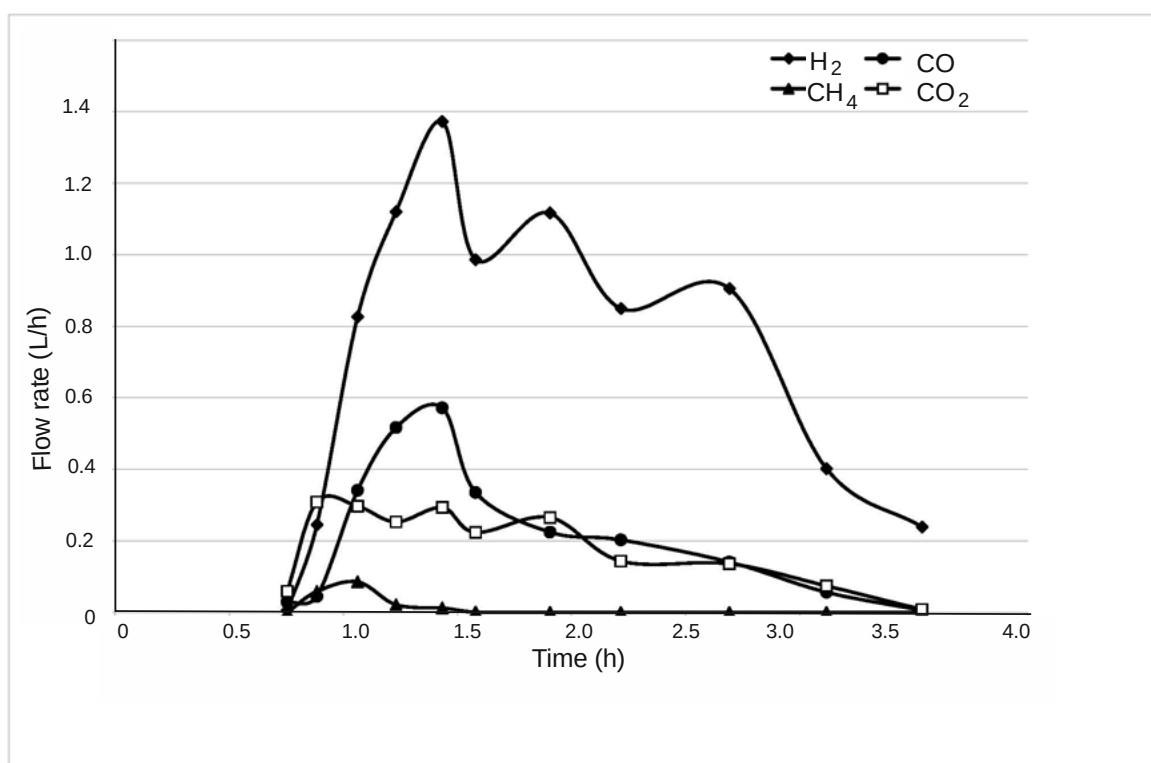


Figure 3. Flow rate of gas components during biological sludge gasification under steam flow rate 9 mL h^{-1} .

Table 4. Efficiency of pyrolysis, gasification and tar production stage in mass carbon terms as a function of steam flow rate.

Experiments	S4	S9	S16
Steam flow rate (mL h^{-1})	4	9	16
%C tar ^a	37.6 ± 1.29	37.3 ± 1.78	32.7 ± 1.35
%C pyrolysed ^a	32.7 ± 0.97	26.8 ± 1.07	30.8 ± 1.06
%C gasified ^a	29.8 ± 1.13	35.9 ± 1.47	36.5 ± 1.45

^adry basis.

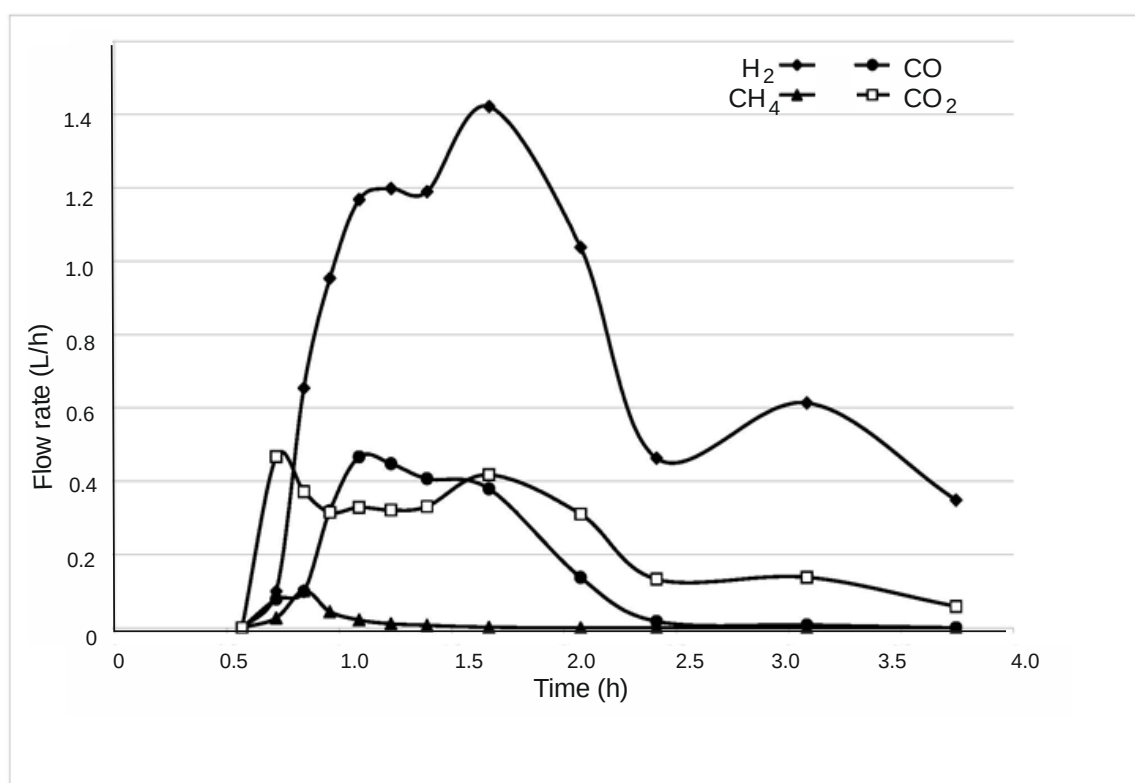


Figure 4. Flow rate of gas components during biological sludge gasification under steam flow rate 16 mL h^{-1} .

From all the experimental data, it was also obvious that the biological sludge is a raw material that is susceptible to gasification process with high efficiencies. The experiment conducted under 16 mL h^{-1} steam flow rate resulted in the highest efficiency 36.5% of conversion of sludge in gasification gas products. This result was within expectations since the higher the vapor concentration, the more efficient the gasification process. The steam flow rate influences not only the final efficiency of the gasification stage but its rate as well. Figure 5 illustrates the time evolution of carbon in gas components during the gasification stage. It is obvious that the carbon production rate varies. It was calculated that the carbon production rate is 1.9 gC h^{-1} for the gasification experiment with 16 mL h^{-1} steam flow rate, while it was 1.1 and 0.9 gC h^{-1} for experiments with 9 and 4 mL h^{-1} steam flow rate respectively. Thus, it can be concluded that the gasification rate is proportional to the steam flow rate. Nguyen *et al.* [32] who studied the steam gasification of biomass in a bubbling fluidized bed reactor, pointed out that the increase in hydrogen and carbon monoxide content is directly related to the steam-to-biomass ratio, while the gas yield and carbon conversion efficiency show significant improvement as the temperature rises. In addition, Singh *et al.* [29] who studied the impact of steam flow rate and temperature on steam gasification of food waste, pointed out that for any steam flow rate that they used the yield of gas produced was high and the tar yield was low when the temperature applied was 800°C . They highlighted that by increasing the temperature value (from 700 to 800°C), the syngas yield would be increased and the steam requirement would be decreased.

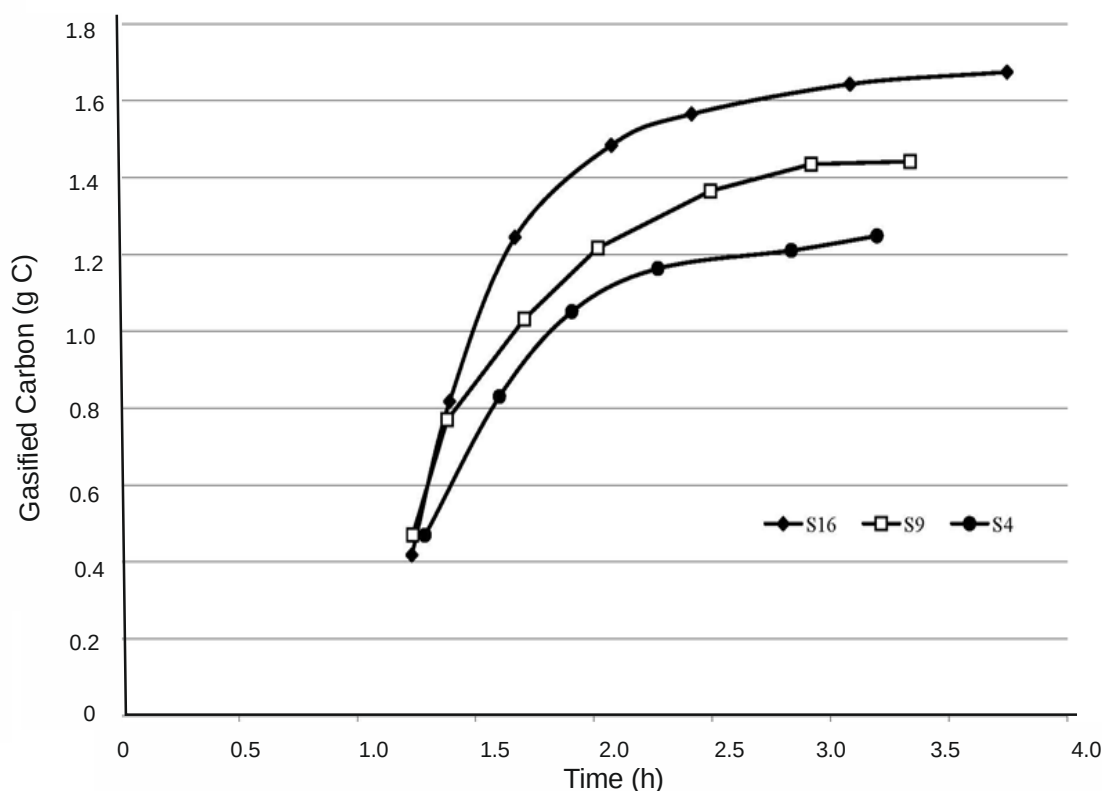


Figure 5. Carbon incorporated in gas products during gasification stage of biological sludge. The experiments were named SX where X is the steam flow rate in mL h^{-1} .

Another important issue that should be addressed for the examination of the efficiency of the sludge gasification process was the recovery of the raw material energy content. In all experiments, the same amount of biological sludge was used, which, expressed in energy terms, was equal to 187 kJ in terms of LHV. The energy content of the gas product in all experiments was estimated by taking into consideration the gas composition, the calorific value of each component and the assumption that the product gas is ideal. [33]. Through these calculations, it was revealed that the energy recovery was 68%, 72% and 79% for biological sludge gasification under steam flows of 4, 9 and 16 mL h^{-1} . Since the moisture content of the sludge could be efficiently used as gasification agent (steam) [34], the energy consumption during the process could be reduced.

4. Conclusion

This research documented that biological sludge is a raw material that can be effectively gasified with high efficiencies both in carbon mass terms and in energy recovery. The ash and tar production, were independent of the steam flow rate and they were characteristics of the raw material. The steam flow rate had a strong influence on the gas quality and quantity. The most favorable conditions for obtaining a fuel gas with high concentration of hydrogen, were sludge gasification under 9 mL h^{-1} steam flow rate. The water gas shift reaction was promoted at lower temperatures as the steam flow rate increased. As far as carbon mass

balances were concerned, it was documented that sludge gasification conducted under the 16 mL h⁻¹ steam flow rate resulted in the highest efficiency 36.5% of sludge conversion in gasification gas products with the maximum rate (1.9 gC h⁻¹).

Therefore, a high steam flow rate during the gasification process is recommended for upscaling the production of hydrogen. In order to reduce the energy consumption of the process, sludge moisture could also be taken into account during the steam flow rate adaptation. This study recommends a simple experimental procedure for steam application during gasification of any biological sludge.

Conflicts of Interests

The authors declare no conflict of interest.

Authors' Contribution

Conceptualization, A.V.; methodology, A.S.; formal analysis, A.S.; investigation, A.S. and D.T.P.; writing—original draft preparation, A.V. and D.T.P.; writing—review and editing, D.T.P.; supervision, A.V. All authors have read and agreed to the published version of the manuscript.

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