Integrated microwave drying, pyrolysis and gasification for valorisation of organic wastes to syngas

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Abstract

The development of integrated platforms is desirable in order to improve the efficiency of biorefineries. This study deals with an innovative *all-in-one* thermochemical process (drying, pyrolysis and gasification) based on the use of microwave energy, which could eliminate the need to pre-dry municipal solid waste in order to produce a high quantity of synthesis gas. Depending on the initial moisture content and pyrolysis time, it is possible to obtain different syngas compositions but only during the first 20 min. Beyond this time the syngas composition remains almost constant. In addition, the moisture content was found to improve the volumetric gas production by almost 50%. The energy consumption of this *all-in-one* process is discussed by comparing it with the scarce information available in the literature concerning the scaling-up of microwave-assisted processes, which may be more cost-effective at a higher scale.

Keywords

Microwave pyrolysis; Drying; Municipal Solid Waste; Syngas; Energy Efficiency

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1. Introduction

The concept "biorefinery" refers to a processing plant where biomass feedstock is converted into valuable products by means of a combination of biological, thermal and chemical processes. The resulting high-value products, such as chemicals and new materials, derived-fuel and the additional power and heat production may well help to lower energy costs and reduce greenhouse gas emissions produced by traditional power facilities.

Nonetheless, there remain a number of barriers related to the implementation and commercialization of the biorefinery that need to be overcome. Apart from non-technical barriers, such as restrictions on use of land or the environmental effects of large areas of feedstock monoculture, reliable equipment designs for the thermochemical conversion of organic feedstock need to be extensively demonstrated on a larger scale [1]. To this end, integrated process platforms appear the most promising way forward.

In general, the drying of biomass materials before their introduction into the conversion units is desirable, since high moisture contents are known to produce fuel gas of lower effective heating value and lead to higher transportation costs, although a large amount of energy is consumed to remove the moisture. Dryers are designed taking into consideration that the wet material must be heated from the inlet temperature up to the wet bulb temperature in order to produce a driving force for surface water to be removed from the material. Afterwards, the material must be heated to drive water from the inside of the biomass to the surface so that it can evaporate. Nevertheless, the

benefits provided by the drying of organic feedstock must be balanced against the increased operating and capital cost [2].

Pyrolysis and gasification are the main thermochemical conversion processes that have been developed within the biorefinery concept to produce synthesis gas (or syngas). Both technologies need an external source of heating to yield gaseous and oily products, whose characteristics depend on feedstock composition, temperature, reactor configuration and gasifying agent, etc. Pyrolysis may be defined as the regime in which the organic matter is degraded by thermal reactions in the absence of oxidizing agents. A gasification process, however, begins with a pyrolysis step followed by the gasification of char and reforming reactions of tar and light hydrocarbons through the addition of a gasifying component such as steam. As a result, gasification usually yields higher amounts of gas fraction than pyrolysis [3]. In order to develop an integrated platform of these processes in a biorefinery, microwave heating should also be considered as an attractive alternative. This method is very tolerant of water compared to conventional pyrolysis and is suitable for most biomass types without pre-drying [4]. Owing to its volumetric nature and high heating rates, a large quantity of steam is generated from the moisture content at the temperatures at which gasification and reforming reactions take place. As a consequence, drying, pyrolysis and gasification processes of wet organic feedstock could all be integrated together in a single microwave induced process (MIP). This method has already been demonstrated by our research group to be a feasible option [5]. In that particular case, it was used to demonstrate a new alternative for wet sewage sludge management which resulted in a gas fraction rich in CO and H_2, making this approach interesting for further production of valuable chemicals [6]. In the previous study, conventional and microwave heating

techniques were compared, it being concluded that higher gas yields could be obtained with conventional heating, though the gas from microwave heating was richer in syngas. It should be pointed out however, that the gas yields were expressed in terms of weight percentage of the initial sewage sludge mass, which could be misleading for the evaluation of gas production. For instance, a gas fraction richer in hydrogen would result in a lower mass of gas due to the low molecular weight of hydrogen, which does not mean a lower volumetric gas production. An alternative more accurate way to calculate gas production might be based on the volume of gas produced per unit of feedstock mass. Results from the literature show that the production of syngas from biowaste microwave pyrolysis by means of this alternative approach is far higher than that from conventional heating [7].

The influence of the moisture content, and the need for feedstock pre-drying, on the gas characteristics from pyrolysis and gasification has been evaluated in several studies, even though further research is still needed. Xiong *et al*. performed the pyrolysis of different sewage sludges by conventional heating [8]. The steam-rich atmosphere generated by the moisture content of the sludge was found to favour the steam reforming reactions of volatile compounds as well as the gasification of solid char, which in turn improved the gas weight yields and the H_2 content. Xie *et al*. reported that an increase in the moisture content in sewage sludge improved the contents of CO_2, CH_4 and H_2 and reduced the tar yield under air gasification, but they provided no results on gas production [9]. Niu *et al*. simulated the gasification of municipal solid waste with different moisture contents. A higher heat input to the gasifier for turning liquid water into superheated steam and a lower gasification efficiency were the effects of the high moisture contents, suggesting that the input feedstock stream should be predried to

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ensure an optimal gasification performance [10]. Moreover, the H_2 concentration increased slightly in spite of a noticeable drop in CO content. Domínguez *et al.* observed that the influence of sewage sludge moisture on the gas products from microwave pyrolysis was negligible, but only a wet and a dried samples were used [11]. However, they attributed this to the fact that water was eliminated very quickly from the reactor and did not have to time to participate in gasification reactions. This problem could have been resolved by using a better reactor design, to enhance the extent of these reactions. Since microwave heating is driven volumetrically, heterogeneous reactions between the water released and the organic waste are much more favored than under conventional heating, which may lead to an increase in the yield of valuable products. Wang *et al.* recently conducted sewage sludge microwave pyrolysis at 850 ºC in a single mode reactor and observed an increase in the H_2 and CO concentrations at higher moisture contents while the CO_2 and CH_4 concentrations decreased [12]. Interestingly, CO production seemed to increase with moisture if microwave heating was applied instead of conventional heating. The results obtained were substantially higher than under conventional pyrolysis.

Syngas production by means of microwave heating in the presence moisture has been widely discussed. The question that still remains to be answered is whether the presence of moisture improves gas production from the microwave pyrolysis of organic residues. If so, this introduces the possibility of creating a new efficient integrated platform for the green disposal of municipal solid wastes through the integration of the drying step into the pyrolysis reactor. The aim of this work is to demonstrate the feasibility of this alternative.

2. Methods

2.1 Materials

The sample selected for this study was a wet organic fraction from a municipal solid waste, supplied by BEFESA *Gestión de Residuos Industriales* S. L. (Seville, Spain) with a moisture content of 45 wt.%. This fraction was deposited in three vessels and subjected to partial drying in a stove at 60 ºC over different drying times (10 min, 20 min and overnight), resulting in three fractions with moisture contents of 34, 12 and 0 wt.% . The samples were immediately stored in a freezer to preserve their moisture content. The samples will be labelled as MSW-*X*, where *X* is the moisture. To corroborate the effect of moisture on the pyrolysis gas from other residues, two fractions (dry and 60 wt.% of moisture) from a sewage sludge, SS-0 and SS-60, and two other fractions from straw residues (dry and 11 wt.% of moisture), ST-0 and ST-11, were also subjected to MIP. The moisture, ash content and volatile matter data (from a LECO TGA-601) are summarised in Table 1 together with the ultimate analysis results (a LECO-CHNS-932 micro-analyser and LECO-TF-900 furnace were used). The metallic content of the ashes was determined by means of atomic absorption spectroscopy.

Table 1. Proximate and ultimate analyses of the organic wastes and metal content of the ashes.

Feedstock	Proximate analysis (wt.%)			Ultimate analysis (wt.%)[a]				
	Moisture	Ash[a]	Volatile matter[a]	C	H	N	S	O
MSW	46.3	22.6	68.0	36.0	5.8	1.6	0.2	33.8
SS	60.0	15.4	74.5	43.8	6.1	9.7	0.1	24.9
ST	11.5	6.4	76.2	45.0	5.0	1.6	0.1	41.9
	Metal content of ashes (mg$_{metal}$ / kg)							
	Fe	Zn	Mn	Cr	Cu	Pb	Ni	Cd
MSW	8896.3	182.9	82.9	58.7	44.8	30.8	17.6	<0.1
SS	2984.6	459.8	40.4	11.7	153.4	1.6	2.8	1.9
ST	286.9	6.6	18.3	0.0	0.0	<0.1	<0.1	0.4

[a] Dry basis

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2.2 Methods

The pyrolysis was carried out in a microwave oven which consisted of a microwave magnetron with a maximum output power of 2 kW operating at 2450 MHz and a single mode cavity where the sample was irradiated using 150 W of emitted microwave power (representing approximately 225 W of consumed power). The reflected power was regulated until reduced to zero by means of stub tuners. About 3 g of sample (on a dry basis) was placed on an inert bed inside a quartz reactor. The reactor was purged with N_2 for 30 minutes at a flow rate of 50 mL_{STP} min^{-1}. The N_2 flow rate was then set to 10 mL_{STP} min^{-1} for the pyrolysis experiments in order to ensure an oxygen-free atmosphere. The sample was mixed with an appropriate microwave absorbent to induce the thermochemical process, as organic residues are transparent to microwaves. The char obtained from the pyrolysis of previous performed in the same microwave reactor was used as the microwave absorbent in an absorbent-to-sample ratio of 1:1, since a previous study had shown that in the case of the wet fraction from the same organic residue, a higher amount of microwave absorbent would enhance the gasification of the char and, in turn the production of gas [13]. Each experiment lasted 1 hour and was repeated twice. The results from the experiments were calculated as the average of the repetitions. Details of the experimental set-up used to collect the oils and gas can be found elsewhere [13].

The gases were analysed in a Varian-CP3800 gas-chromatograph equipped with a TCD detector and two columns connected in series. The first column was 80/100 Hayesep Q (2 m x 1/8 in. x 2mm) and the second column was a 80/100 Molesieve 13X (1.5 m x 1/8 in. x 2 mm). The second column was bypassed by a six-port valve for the analysis of the CO_2 and hydrocarbons. The carrier gas flow (He) was 30 mL/min. The initial oven

temperature was set at 40 ºC, which was maintained for 1.2 min. It was then programmed to rise from 40 to 65 ºC at 50 ºC/min with an isothermic step of 3.3 min. The temperature was then lowered from 65 to 55 ºC at 20 ºC/min and held for 2.6 min. The injector and detector temperatures were fixed at 150 ºC. The TCD was calibrated using a standard gas mixture.

3. Results and discussion

3.1 Gas production

The gas productions from the wet fractions from the MSW (calculated on a dry basis) have been normalized to the gas production from the dry fraction (MSW-0), as shown in Figure 1. Two stages can be clearly identified during the gas production: the first from the initial time to ca. 20 min when production increases, and the second stage from ca. 20 min until the end of the pyrolysis experiment. There is a very large peak of volatiles production at ca. 15 min, during the first stage. From this point, the differences between the gas productions for different moisture contents become more pronounced. Afterwards, the production rate slows down and the differences diminish. Since the relative production is higher than 1 for all the wet samples studied after 5 min, a higher gas production than that corresponding to the dry sample is bound to occur. Furthermore, it can be seen that the higher the moisture content is, the greater the gas production. Figure 2(a) depicts the absolute values of gas production (L g^{-1}_{MSW} on a dry basis). The main reason for this is the gasification reaction of the microwave absorbent and the char formed during the pyrolysis as a result of which syngas and CO_2 are produced:
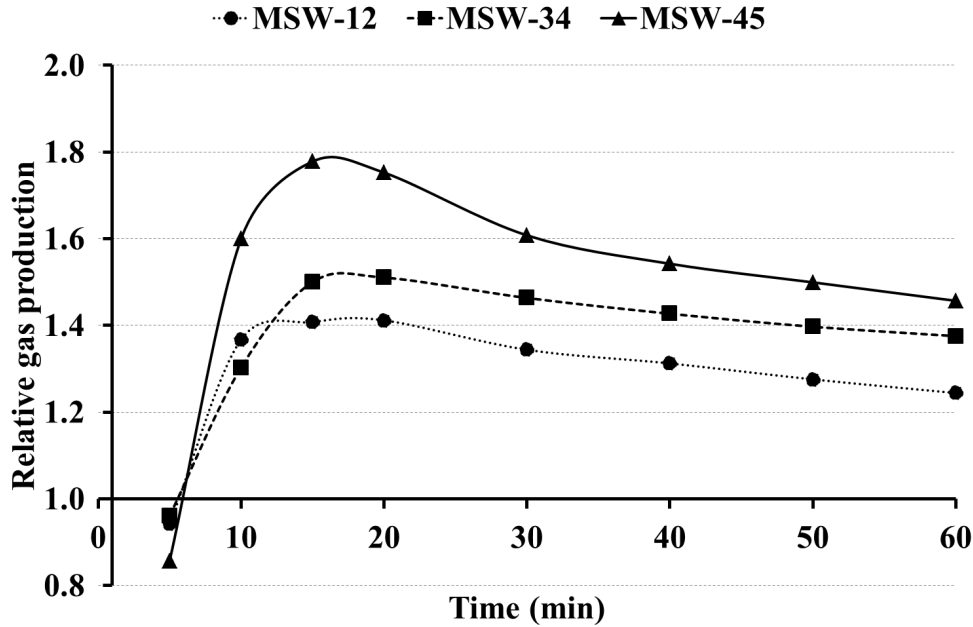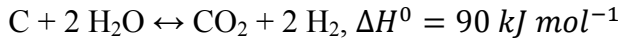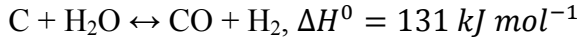
$$C + H_2O \leftrightarrow CO + H_2, \Delta H^0 = 131 \; kJ \; mol^{-1}$$

$$C + 2\,H_2O \leftrightarrow CO_2 + 2\,H_2, \Delta H^0 = 90 \; kJ \; mol^{-1}$$

Figure 1. Relative gas production (L g$^{-1}_{MSW}$ on a dry basis) at different moisture contents in MSW relative to dry MSW gas production

Table 2. The effect of moisture content on the solid MSW pyrolysed

Moisture content (wt.%)	Solid residue yield (wt.%)[a]
0	68
12	61
34	58
45	48

[a] Solid yield is expressed as the ratio of final solid residue mass to the initial load mass (MSW + Microwave absorbent).

As can be seen, the solid fraction yields decrease with the moisture content (Table 2), confirming that the initial water present in the samples has a positive effect on the gasification of char.
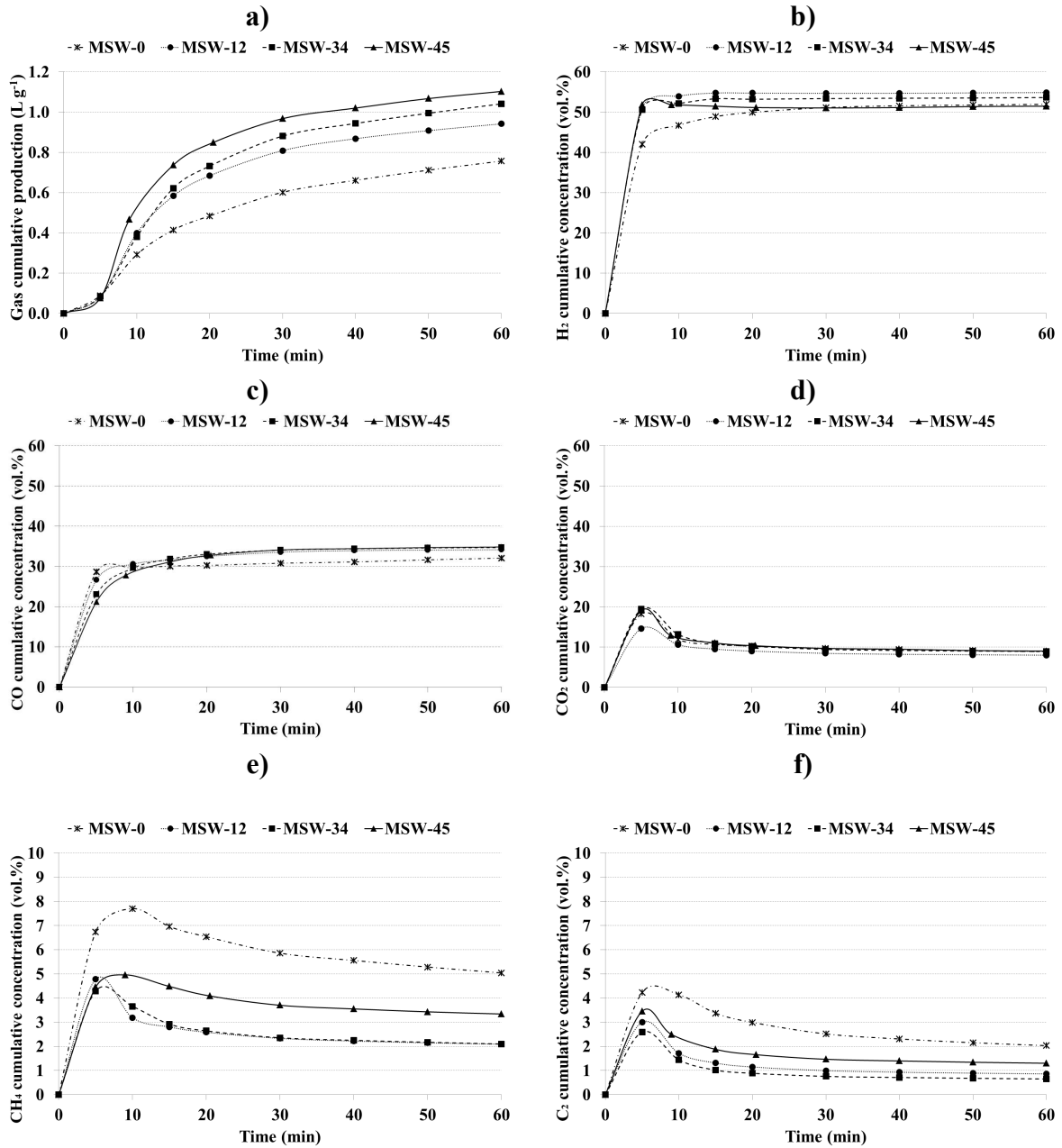
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Figure 2. (a) Total cumulative gas production (on a dry basis) and cumulative

concentration of (b) H_2, (c) CO, (d) CO_2, (e) CH_4 and (f) C_2 hydrocarbons at different

moisture contents in MSW

If the trends in Figure 2(a) are compared, a noticeable gap can be observed between

MSW-0 and MSW-12 (MSW-12 is a 24% higher). However, the differences between

MSW-12, MSW-34 and MSW-45 are smaller, which suggests that a small amount of

water favours the production of gas, although a large quantity does not significantly improve it.

3.2 Gas composition

The gas composition of each MSW fraction is depicted in Figures 2(b)-(f). As pointed out by Domínguez *et al*. [11], the gas composition remains essentially the same regardless of the moisture content, once the second stage (> 20 min) has been reached. In fact, there appears to be a value of moisture content value at which the steam generated reaches a saturation point preventing further gasification reactions. This possibility has also been suggested in another study based on the conventional pyrolysis of sewage sludge [8], in which a moisture content of ca. 50 wt.% is proposed as being the critical value in the production of hydrogen. Nevertheless, with microwave heating this saturation point seems to be reached at lower moisture contents, at least in the case of the organic MSW used in the present study, since at the end of the experiments the difference in hydrogen concentrations among the wet fractions was less than 7 vol.%, whereas in the study of *Xiong*, it reached ca. 40 vol.%. Besides, both the hydrogen (Figure 2(b)) and syngas concentrations (Figure 3) are greater in the case of MSW-12, hence 12 wt.% can be assumed to be the critical moisture content in these cases. Nevertheless, during the first 5 min, MSW-45 is the fraction which gives off more hydrogen. In the case of CO (Figure 2(c)), although the differences are also very small, it can be seen that the presence of moisture slightly improves the CO produced, but only after 10 min, perhaps due to the prolongation of char gasification. However, at the beginning of the process, the CO concentration is 50% greater in the case of the dry fraction MSW-0. In the case of CO_2, the profiles depicted in Figure 2(d) show a common trend. Thus, the water gas shift reaction, as illustrated below, which should

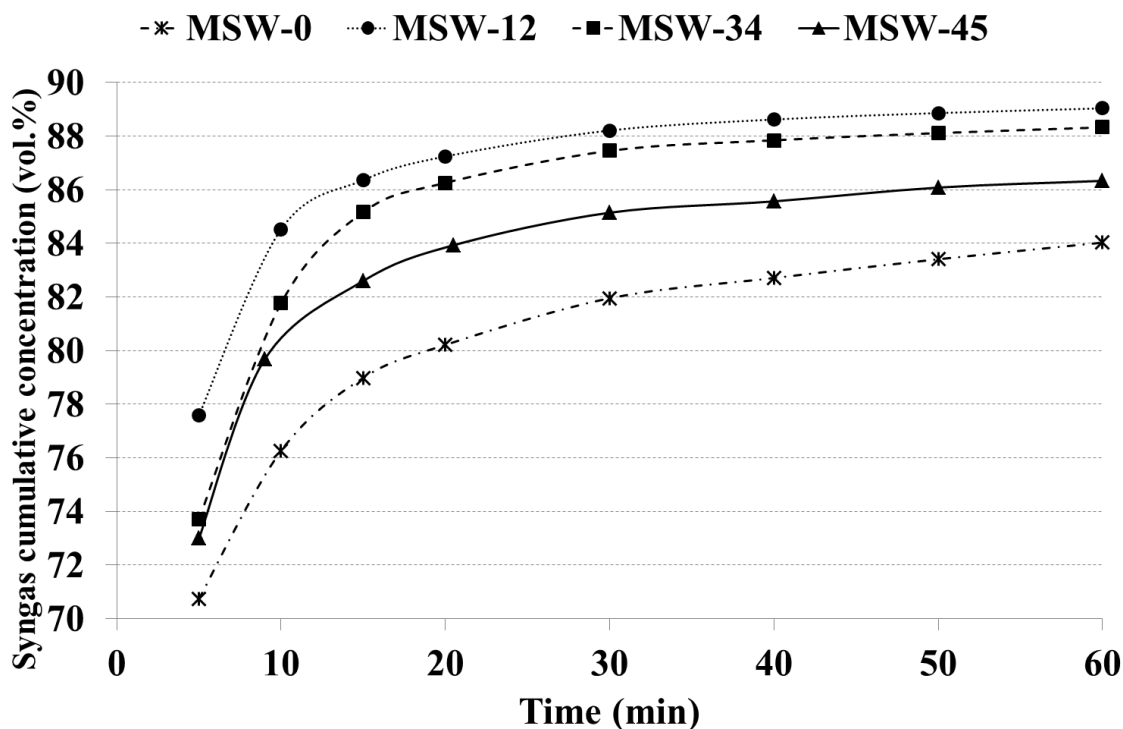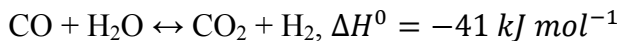lead to a higher CO_2 concentration with moisture, cannot be taking place to any significant degree:

$$CO + H_2O \leftrightarrow CO_2 + H_2, \Delta H^0 = -41 \; kJ \; mol^{-1}$$

Figure 3. Syngas concentration (vol.%) from the MIP of the MSW as a function of the moisture content and the pyrolysis time

As for the CH_4 concentration, the MSW-0 sample generates more methane and the differences between the wet fractions are more pronounced. For instance, MSW-0 produces ca. 130% more than MSW-12 and MSW-34. This marked difference could be due to a mixed effect of the dilution of methane at a higher moisture content and the reforming reactions of hydrocarbons. However it has not been possible so far to elucidate which explanation is more plausible.

The syngas concentration has been plotted in Figure 3 as a function of the moisture content and the pyrolysis time and ranges from 70 to 88 vol.%. It is clear that the syngas concentration is significantly enhanced with time at the expense of higher energy consumption. This is because more thermal cracking takes place, enhancing the conversion of volatiles to H_2 and CO, as occurs when microwave heating is used [6, 14]. The highest moisture contents (34 and 45 wt.%) seem to disfavour the syngas content compared to MSW-12. Interestingly, the dry fraction MSW-0 produces the smallest syngas concentration at the expense of hydrocarbon (CH_4 and C_2) production. This indicates that MSW-12 is the most appropriate fraction for maximising the syngas concentration.

Figures 4(a)-(b) show the quality of the gas fraction in terms of H_2/CO and CO/CO_2 ratios. At the very beginning of the first pyrolysis stage, the highest H_2/CO ratio corresponds to MSW-45 (2.44), but there is a significant decrease affecting all the wet fractions from that point, especially during the first stage (< 20 min). This is due to the increased CO production as can be seen in Figure 2(c). However, for MSW-0 the opposite tendency was found with H_2 production increasing in the first stage and reaching its highest H_2/CO value (1.62) at 60 min. It can be concluded therefore that the moisture content improves the gas quality towards H_2 if the pyrolysis time is not extended beyond 10 min. The H_2/CO ratio will then remain constant at approximately 1.5-1.6 regardless of the moisture content. In the case of the CO/CO_2 ratio, a rising tendency with time is observed, which may indicate that CO_2 is being converted to CO, as can be observed from Figure 2(d), where there is a progressive fall in the CO_2 concentration pattern from the very beginning of the process. MSW-12 shows the

highest CO/CO_2 ratio over the entire experiment. This sample, which displays the

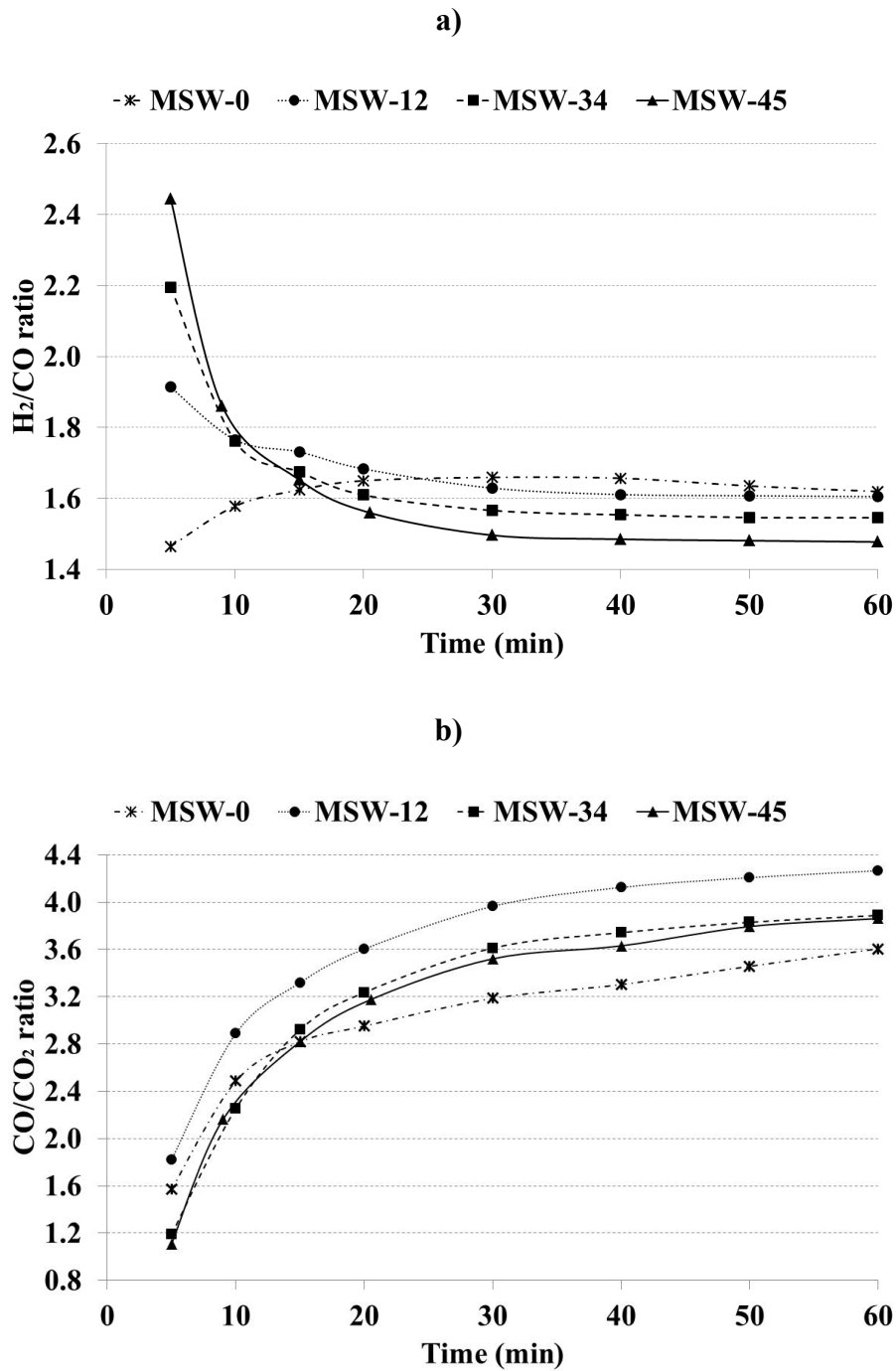critical moisture content, gives rise to the greatest extent of gasification reactions.

a)

b)

Figure 4. (a) H_2/CO and (b) CO/CO_2 ratios from the MIP of the MSW as a function of

the moisture content and the pyrolysis time

In order to check whether the moisture content affects the gas production in a positive way with different organic feedstocks, two other residues (sewage sludge and agricultural straw residue) were subjected to microwave pyrolysis under the same experimental conditions as the MSW. The results from these experiments are presented in Table 3. In all the cases, a significant reduction in solid yield is observed at the expense of an improvement in the gas yield, as occurred with MSW, especially in the case of the wet sewage sludge, SS-60, which yielded 60 wt.% of gas fraction. With respect to the gas composition (values are reported at 60 min), differences are more appreciable in SS and ST, particularly in the case of hydrocarbons content (HC). This is due to the fact that moisture content favours the gasification of tar and light hydrocarbon gases. The gas fraction from the pyrolysis of the straw residue was clearly the fraction most affected by moisture content. For this reason, feeding wet feedstock to a full-scale pyrolysis reactor would promote syngas production considerably. Anyway, from these results it can be concluded with a fair degree of certainty that regardless of the moisture content which is bound to improve gas production, the type of feedstock is of extreme importance for gas production as previously reported by our research group [15].

Table 3. Characteristics from the MIP of MSW, SS and ST at different moisture contents

Feedstock	Solid yield (wt.%)	Liquid yield (wt.%)[a]	Gas yield (wt.%)	$[H_2]$ (vol.%)	$[CO]$ (vol.%)	$[CO_2]$ (vol.%)	$[CH_4]$ (vol.%)	$[HC]^b$ (vol.%)
MSW-0	68	6	26	52.0	32.1	8.9	5.0	2.0
MSW-45	48	4	48	51.5	34.9	9.0	3.3	1.3
SS-0	64	9	27	35.5	23.0	6.2	3.5	31.8
SS-60	34	6	60	38.8	32.1	4.6	3.5	21.0
ST-0	68	4	28	42.0	27.1	11.4	3.5	16.0
ST-11	59	3	38	53.1	34.9	8.6	2.7	0.7

[a] The liquid yield is calculated on a dry basis
[b] Hydrocarbons concentration (C_2 and higher)

3.3 Energetic considerations

A lower energy consumption is usually stated as one of the main advantages of microwave heating. However, this aspect in relation to the scaling-up of microwave pyrolysis has not been dealt with in depth in the literature. Only one study on low temperature pyrolysis of wheat straw deals with the energy requirements when the feedstock mass to be pyrolysed is increased from a few grams up to 200 g [4]. In that case, it can be observed that there exists a critical mass at which the specific energy consumption needed to achieve the desired temperature falls drastically; i.e., energy consumption in lab-scale experiments can lead to wrong conclusions, since the specific energy needed for a few grams is much higher than that needed for a larger quantity of feedstock. To corroborate whether this also happens in other microwave heating processes, Figure 5 shows the corresponding trends for two other very different processes: the heating of SiC samples up to 600 ºC and of pure water up to 80 ºC. As can be seen, regardless of the type of process and targeted temperature, there is always a critical mass and a hyperbolical trend with increasing mass. For example, the microwave heating of 5 g of pure water requires ca. 85 kJ·g^{-1} to reach 80 ºC and for this temperature to be held for 15 min, whereas only 4.25 kJ·g^{-1} are necessary for 300 g to reach the same temperature, representing a reduction in specific energy of 95%.

● **Heating of SiC**   □ **Torrefaction**   ▲ **Heating of water**

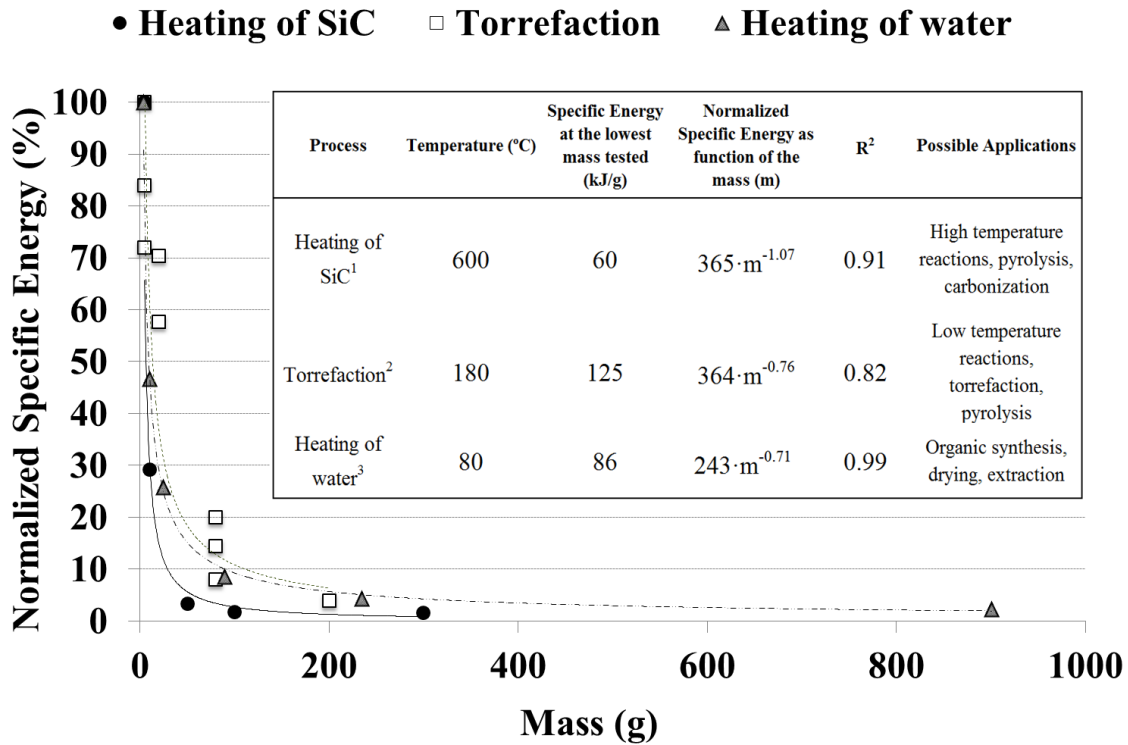| Process	Temperature (ºC)	Specific Energy at the lowest mass tested (kJ/g)	Normalized Specific Energy as function of the mass (m)	R^2	Possible Applications
Heating of SiC[1]	600	60	$365 \cdot m^{-1.07}$	0.91	High temperature reactions, pyrolysis, carbonization
Torrefaction[2]	180	125	$364 \cdot m^{-0.76}$	0.82	Low temperature reactions, torrefaction, pyrolysis
Heating of water[3]	80	86	$243 \cdot m^{-0.71}$	0.99	Organic synthesis, drying, extraction

Figure 5. Normalized specific energy dependence on the mass to achieve the desired temperature (i.e., the energy per unit of mass divided by the energy required for the lowest mass tested) in three different microwave induced processes. References: [1]Electromagnetic simulations in MW and RF processing. AMPERE Short Course given by Dr. Eng. Paolo Veronesi from Department of Engineering "Enzo Ferrari" (Modena, Italy) on 16th September 2013; [2]Reference [4]; [3]Experiments performed in our laboratory.

Presumably, in our microwave pyrolysis-gasification process, a similar behavior can be expected when scaling-up. As 3 g of MSW was used in this study, the energy consumption corresponding to 225 W during a one hour experiment was 270 kJ·g^{-1} (810 kJ). Therefore, a reduction in consumption of more than 90% could be achieved by increasing the residue mass to beyond 200 g. However, it is necessary to take into account the moisture content since the latent heat of evaporation of water (2.257 kJ·g^{-1}) cannot be ignored. At a small scale (as in our MSW pyrolysis experiments), since there

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is a very high specific consumption, the energy required to evaporate the water is relatively small. For example, 2.46 g of water were present in the MSW-45 pyrolysis and only 5.55 kJ in 810 kJ were theoretically necessary to evaporate the moisture, which is less than 1% of the total amount of energy. Nonetheless, at higher scales the situation is completely different. For example, to pyrolyse 200 g of MSW-45 using the same input power as at lab-scale, it would be necessary to evaporate 90 g of water, representing 203 kJ or 25% of the total amount of energy, the remaining energy being left to overcome the pyrolysis. However, this would lead to a poorer syngas quality, since the power density would be lower. Ultimately, although the integration of the drying step into our microwave pyrolysis-gasification process might be feasible, an economic assessment of the consumption of the microwave scaled-up process and the value of the final products would help to decide whether this process could become cost-effective.

4. Conclusions

This paper has dealt with the integration of the drying step of organic waste into a microwave pyrolysis reactor, as an alternative process for syngas production. This would make it unnecessary to install a previous dryer to the pyrolysis reactor and also allows the *in-situ* gasification of the solid waste, improving the gas yield regardless of the organic waste.

This is the first time that the influence of the moisture content on microwave pyrolysis of municipal solid waste has been studied in a systematically way, having found out that there appears to be an optimal moisture content which maximises the syngas. In this

study, 12 wt.% of moisture in MSW was found to be the saturation point (yielding ca. 90 vol.% of syngas) beyond which syngas concentration started to fall.

Nevertheless, despite these advantages, the energy required to evaporate the moisture is high, which could be a limiting factor for the energy balance of a successful prototype design.

Acknowledgments

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