

SEWAGE SLUDGE GASIFICATION. DOLOMITE PERFORMANCE UNDER DIFFERENT OPERATING CONDITIONS

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EXECUTIVE SUMMARY

Gasification is a technology that can replace traditional management alternatives used up to date to deal with this waste (landfilling, composting and incineration) and which fulfils the social, environmental and legislative requirements. The main products of sewage sludge gasification are permanent gases (useful to generate energy or to be used as raw material in chemical synthesis processes), liquids (tars) and char. One of the main problems to be solved in gasification is tar production. Tars are organic impurities which can condense at relatively high temperatures making impossible to use the produced gases for most applications.

This work deals with the effect of some primary tar removal processes (performed inside the gasifier) on sewage sludge gasification products. For this purpose, analysis of the gas composition, tar production, cold gas efficiency and carbon conversion were carried out. The tests were performed with air in a laboratory scale plant consisting mainly of a bubbling bed gasifier. No catalyzed and catalyzed (10% wt of dolomite in the bed and in the feeding) tests were carried out at different temperatures (750°C, 800°C and 850°C) in order to know the effect of these parameters in the gasification products. As far as tars were concerned, qualitative and quantitative tar composition was determined. In all tests the Equivalence Ratio (ER) was kept at 0.3.

Temperature is one of the most influential variables in sewage sludge gasification. Higher temperatures favoured hydrogen and CO production while CO₂ content decreased, which might be partially explained by the effect of the cracking, Boudouard and CO₂ reforming reactions. At 850°C, cold gas efficiency and carbon conversion reached 49% and 76%, respectively. The presence of dolomite as catalyst increased the production of H₂ reaching contents of 15.5% by volume at 850 °C. Similar behaviour was found for CO whereas CO₂ and C_nH_m (light hydrocarbons) production decreased. In the presence of dolomite, a tar reduction of up to 51% was reached in comparison with no catalyzed tests, as well as improvements on cold gas efficiency and carbon conversion.

Several assays were developed in order to test catalyst performance under more rough gasification conditions. For this purpose, the throughput value (TR), defined as kg sludge “as received” fed to the gasifier per hour and per m² of cross sectional area of the gasifier, was modified. Specifically, the TR values used were 110 (reference value), 215 and 322 kg/h·m². When TR increased, the H₂, CO and CH₄ production decreased while the CO₂ and the C_nH_m production increased. Tar production increased drastically with TR during no catalysed tests what is related to the lower residence time of the gas inside the reactor. Nevertheless, even at TR=322 kg/h·m², tar production decreased by nearly 50% with in-bed use of dolomite in comparison with no catalyzed assays under the same operating conditions.

Regarding relative tar composition, there was an increase in benzene and naphthalene content when temperature increased while the content of the rest of compounds decreased. The dolomite seemed to be effective all over the range of molecular weight studied showing tar removal efficiencies between 35-55% in most cases. High values of the TR caused a significant increase in tar production but a slight effect on tar composition.

1 INTRODUCTION

The European legislation restricts many traditional management options for sewage sludge such as direct use for cultivation and landfilling. Due to the fact that incineration is subject to strong social opposition in countries like Spain, gasification arises like one of the most attractive alternatives for the management of this waste (Seggiani, 2012).

Sewage sludge gasification is the thermal process by which the carbonaceous content of sewage sludge is converted to combustible gas and a solid waste (char) in a net reducing atmosphere.

Unfortunately, the produced gas contains impurities like dust particles, sulphur, nitrogen and chlorine compounds and tars that must be removed to meet the requirements of end-use applications. In fact, one of the main problems to be solved in gasification is tar production. Tars are organic impurities which can condense at relatively high temperatures making impossible to use the produced gas for most applications. The tar content and the gas composition mainly depend on the gasification conditions (temperature, pressure, and residence time), the gasifying agent, the type of gasifier used and the presence of catalysts for tar destruction/reforming (Šulc, 2012).

Regarding gasifying agents, the most widely used are air, oxygen, steam or mixtures thereof (Campoy, 2010; Meng, 2011). As far as the type of gasifier is concerned, most of the gasification plants consist of fixed bed, fluidized bed and entrained flow gasifiers (Hernández, 2010; Seggiani, 2012; Šulc, 2012). The gasification conditions may be very different depending on the system chosen. For instance, temperatures between 800 and 900 °C are typical in atmospheric air-blown bubbling fluidized bed gasifiers whereas temperatures of up to 1100°C or even 1500°C are typical for fixed bed and entrained flow gasifiers, respectively (Gómez-Barea, 2012; van der Drift, 2004). Finally, regardless of their chemical composition, the catalysts used in gasification processes can be classified into primary (when located inside the gasifier) or secondary (when located downstream the gasifier). Although the efficiency of secondary methods for tar removal has been extensively demonstrated (Asadullah, 2004; Huang, 2012) major ongoing research is focused on primary methods because they are less complex and expensive than secondary ones (Devi, 2003). Typical in-bed catalysts are dolomite, olivine and alumina (Corella, 2004; de Andrés, 2011). More effective nickel-based catalysts improve tar reduction in the bed, but the rapid degradation of the catalyst makes this option unfeasible so far (Gómez-Barea, 2011). Thus, taking into account both effectiveness in tar removal and the resistance to deactivation, the dolomite seems to be the most attractive primary catalyst for sewage sludge gasification, according to the present knowledge.

This work deals with the effect of the temperature and the use of dolomite as primary catalyst on sewage sludge gasification products, with special attention to tar production. For this purpose, analysis of the gas composition, tar production (Y_{tar}), cold gas efficiency (CGE) and carbon conversion (X_C) are carried out. Regarding tar production, catalysts performance can be greatly influenced by the throughput (TR, hereinafter), defined as the kilograms of sewage sludge as received fed to the gasifier per hour and per- m^2 of cross sectional area of the gasifier. According to Corella (2008), some studies developed at small scale use very low TR (soft conditions, TR close to 100-150 $kg/h \cdot m^2$). As a result of that, tar removal efficiencies found may be very different from those obtained at commercial scale, with TR around 750 $kg/h \cdot m^2$. For this reason, several assays were developed in order to test dolomite performance under TR of 125, 250 and 375 $kg/h \cdot m^2$.

It is widely accepted that the final use of the produced gas defines the need for tar conversion. However, according to Gómez-Barea (2012), the key parameter for the assessment of the suitability of the gas for a given application is the nature of the tar, not only the tar concentration. This can be explained by the fact that the tar composition determines the dew point of the gas. For this reason, qualitative determinations of tar composition were carried out by gas chromatography-mass spectrometry (GC/MS) to know how the different gasification conditions and the presence of dolomite affect the tar composition.

2 MATERIALS AND METHODS

2.1 Materials

The dried sludge samples were received from a sewage sludge drying plant of Madrid, Spain. The elemental analyses of the dried sewage sludge are shown in Table 1. These data were used to estimate the low heating value (LHV) of the sludge (13.1 MJ/kg) by means of the modified Dulong's formula (de Andrés, 2011). Silica sand (and catalyst, if necessary) was used as the bed material. The sludge was crushed and sieved to particle size between 300 and 500 μm . The dolomite was supplied by Dolomitas del Norte S.A., Spain.

TABLE 1 Proximate and elemental analysis of sewage sludge (dry basis) from a sewage sludge drying plant

Parameter		Sludge	Analytical method
Moisture (%)		7.9	UNE-EN 12880-2001
Organic Mat. (%)		55.4	UNE-EN 12879-2001
Ash (%)		44.6	UNE-EN 12879-2001
Carbon (%)		30.7	Elemental micro analyser LECO CHNS-932
Nitrogen (%)		4.3	
Hydrogen (%)		5.0	
Sulphur (%)		1.6	
Oxygen (%)	by difference	13.8	

2.2 Laboratory scale plant

Experiments were carried out in a laboratory scale plant. The reactor used was a stainless steel fluidised bed gasifier with a total height of 700 mm and an inner diameter of 32 mm followed by a freeboard. Both the reactor and the freeboard were heated by an electrical furnace. Inside the gasifier, the bed was held by a distributor plate (0.1- mm pore size). The gasifying agent (air) entering the reactor was electrically preheated.

The sludge was fed into the reactor a few millimetres above the distributor plate by a dosing system consisting of a hopper and two screw feeders (the dosing and launch screw feeders). The bed height was kept at 100 mm by a concentric pipe inserted through the distributor plate.

Downstream of the freeboard, a cyclone and a micron filter were placed inside a hot box (250 °C) to prevent condensation of the tars. Tar collection was done following the tar protocol and tar production was determined by weighting after distillation.

Gas production was measured by a mass flow meter. The dry gas composition (N₂, O₂, H₂, CO, CO₂, CH₄, C₂H₆ and C₂H₄) was determined by means of a micro gas chromatograph and the tar composition by gas chromatography/mass spectrometry (GC/MS). More detailed information and a diagram of the plant used can be found in de Andrés (2011).

2.3 Experimental conditions

The tests carried out to know the influence of different parameters on the gasification products can be divided into three main groups:

- Influence of the temperature: a set of tests was conducted at temperatures of 750°C, 800°C and 850°C. The equivalence ratio, ER (defined as the ratio between the flow rate of the air introduced into the reactor and the stoichiometric flow rate of the air required for a complete combustion of the sludge), was set at 0.3. It was decided to set the ER to this value because, according to previous experiences, under these conditions tar production was relatively low (6 g/Nm³) and the LHV of the gases remained within acceptable levels (around 4 MJ/Nm³).
- Influence of in-bed use of dolomite: different tests were carried out at 750°C, 800°C and 850°C with a constant ER of 0.3 and with a dolomite content in the bed and in the feeding of 10% by weight.
- Influence of the throughput, TR: the performance of the dolomite under different TR (110, 225 and 322 kg/h•m²) was tested by modifying the flow rate of sludge fed to the gasifier (Table 2). The ER was fixed at 0.3 to be consistent with the before mentioned experiments. For this purpose, the air flow rate introduced in the gasifier was modified and therefore the fluidizing velocity (4, 8 and 12 times the minimum fluidization velocity, u_{mf} (850°C) = 3.1 cm s⁻¹, respectively).

The conditions and results of the tests carried out are shown in Table 2 and Table 3. Prior to each test 80 g of silica sand (or sand/dolomite mixture in tests with catalyst) were placed in the gasifier. Once the temperature of the test had been reached, the gasifier was continuously fed with sludge and a specific sand-catalyst mixture (20% of the mass rate of fed sludge). The sand-catalyst proportion of the mixture in each test is shown in Table 2 and Table 3 (20-0% or 10-10% depending on the test). The sand introduced in the feeding was used to improve fluidization. The total duration of the tests was 60 min.

To validate each test, it was decided that the experiment closure mass balance should be between 95% and 105%.

3 RESULTS AND DISCUSSION

A summary of the results of the gasification tests carried out is presented in Table 2 and Table 3.

TABLE 2 Results of gasification experiment without catalyst. Effect of temperature and throughput (TR)

Parameter	Units	T_1	T_2	T_3	T_4	T_5	T_6	T_7	T_8	T_9
Temperature	°C	750	800	850	750	800	850	750	800	850
Sludge	g/min	1.2	1.2	1.2	2.5	2.5	2.5	3.7	3.7	3.7
Sand	% fed sludge	20	20	20	20	20	20	20	20	20
TR	kg/h•m ²	110	110	110	215	215	215	322	322	322
u/u _{mf}		3.6	3.8	4.0	7.3	7.6	8.0	10.9	11.5	12.0
Residence time	s	7.8	7.5	7.1	3.9	3.7	3.6	2.6	2.5	2.4
H ₂		9.4	11.9	13.4	7.6	9.5	11.2	5.3	8.5	10.6
N ₂		63.2	61.0	58.7	65.6	63.6	59.9	67.7	63.3	60.7
CH ₄		3.0	2.8	3.0	2.7	2.6	3.2	2.6	3.0	2.9
CO		6.9	7.9	9.28	6.7	7.5	9.2	7.0	8.2	9.4
CO ₂		13.4	13.2	12.59	13.6	13.2	12.5	13.5	13.4	12.8
C ₂ H ₆		0.11	0.06	0.03	0.17	0.07	0.03	0.14	0.04	0.04
C ₂ H ₄		1.8	1.8	1.6	2.0	2.0	1.8	2.1	2.1	2.1
LHV gas	MJ/Nm ³	3.1	3.4	3.8	2.7	3.0	3.6	2.5	3.1	3.5
Gas production	Nm ³ /kg sludge, daf	2.7	2.8	3.0	2.7	2.8	2.9	2.6	2.7	2.8
Tar concentration	g/Nm ³	7.4	4.1	2.4	12.2	7.7	4.2	15.6	9.8	5.8
Y _{tar}	mg/g sludge, daf	20.3	11.8	7.1	32.4	21.4	12.1	40.8	26.5	16.1
Xc	%	66.9	70.6	76.4	65.0	68.1	75.1	64.1	69.9	73.8
CGE	%	36.8	42.0	49.4	31.8	32.7	46.0	28.3	36.8	42.5
Char	g/kg daf	58.1	42.3	21.1	51.5	43.6	30.4	56.4	48.5	22.0

TABLE 3 Results of gasification experiment with dolomite. Effect of temperature and throughput (TR)

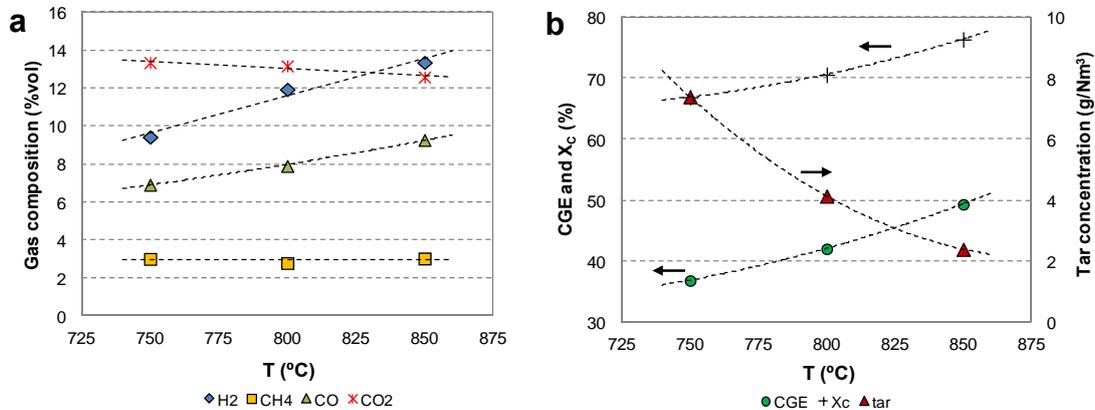
Parameter	Units	T_10	T_11	T_12	T_13	T_14	T_15	T_16	T_17	T_18
Temperature	°C	750	800	850	750	800	850	750	800	850
Sludge	g/min	1.2	1.2	1.2	2.5	2.5	2.5	3.7	3.7	3.7
Sand	% fed sludge	10	10	10	10	10	10	10	10	10
Dolomite	% fed sludge	10	10	10	10	10	10	10	10	10
TR	kg/h•m ²	110	110	110	215	215	215	322	322	322
u/u _{mf}		3.6	3.8	4.0	7.3	7.6	8.0	10.9	11.5	12.0
Residence time	s	7.8	7.5	7.1	3.9	3.7	3.6	2.6	2.5	2.4
H ₂		10.8	12.7	15.5	8.5	10.7	13.1	6.5	9.6	12.1
N ₂		63.1	58.5	56.7	65.0	62.5	58.8	68.7	63.3	60.1
CH ₄		2.8	3.2	2.8	2.8	2.6	2.6	1.9	2.5	2.4
CO		5.3	9.7	11.6	5.7	6.8	10.4	5.4	6.8	9.0
CO ₂		14.4	12.6	10.9	13.7	13.9	12.1	14.2	14.2	13.1
C ₂ H ₆		0.11	0.04	0.02	0.18	0.08	0.03	0.12	0.09	0.04
C ₂ H ₄		1.8	1.7	1.0	2.0	1.8	1.5	1.5	1.9	1.8
LHV gas	MJ/Nm ³	2.9	3.9	4.3	2.7	3.1	3.8	2.1	2.9	3.4
Gas production	Nm ³ /kg sludge, daf	2.8	2.9	3.1	2.7	2.797	2.914	2.6	2.781	2.8
Tar concentration	g/Nm ³	3.5	2.1	1.3	7.5	4.7	2.4	9.8	6.3	3.3
Y _{tar}	mg/g sludge, daf	9.8	6.0	4.0	19.8	13.1	6.9	25.8	17.4	9.3
Xc	%	66.6	76.7	78.7	62.5	68.5	75.1	58.8	68.7	72.4
CGE	%	36.0	48.8	56.9	31.6	37.1	47.7	24.3	35.0	42.2
Char	g/kg daf	68.7	47.6	23.8	79.3	35.7	21.1	49.3	30.0	15.9

3.1 Effect of the temperature on the gasification products

Fig. 1a shows the effect of the temperature on the gas composition under a TR of 110 kg/h•m² (reference TR in this work). The H₂ and CO content increased with the temperature while the CO₂ concentration decreased. It can be explained by effect of the CO₂ reforming reactions and the Boudouard reaction, especially at the highest temperature (850°C). As a result of that, the CO/CO₂ ratio increased from 0.51 at 750°C to 0.74 at 850°C. Slight differences were found for the CH₄ content whereas the concentration of C_nH_m (C₂H₆ and C₂H₄) decreased as the temperature increased (Table 2). Regarding tar production, reductions of up to 67% were found by increasing the temperature from 750°C to 850°C (Fig. 1b). Although not shown, both the LHV of the produced gas and the gas production (Y_{gas}) increased with

the temperature. Higher temperatures favoured the production of combustible gases as well as a more intense volatilisation of the sludge, the decomposition of the tars and the conversion of the char (Campoy, 2009). The cold gas efficiency (CGE) varied between 37% (at 750 °C) and 49% (at 850 °C) while carbon conversion (X_C) varied between 67% (at 750 °C) and 76% (at 850 °C).

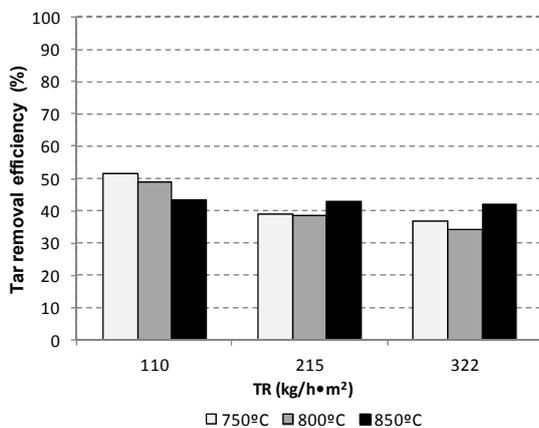
FIGURE 1 Effect of the temperature on gasification products (TR=110 kg/h·m²). (a) effect on gas composition; (b) effect on cold gas efficiency (CGE), carbon conversion (X_C) and tar concentration (tar)



3.2 Influence of in-bed use of dolomite

The presence of dolomite has a clear effect on the gasification products. As far as gas composition was concerned, the dolomite increased the H₂ and CO content in comparison to tests without catalyst. However, the production of CO₂ and C_nH_m decreased. These results can be explained by the prevalence of the cracking and the CO₂ reforming reactions. No relevant differences were found regarding CH₄ content. Fig. 2 shows the tar removal efficiency obtained with the dolomite. As it can be seen, the reductions found slightly varied depending on the temperature. Specifically, under the reference TR of 110 kg/h·m², the tar removal efficiency ranged between 51%_± at 750°C_± and 44%_± at 850°C.

FIGURE 2 Tar removal efficiency of the dolomite as a function of the throughput (TR) and the temperature



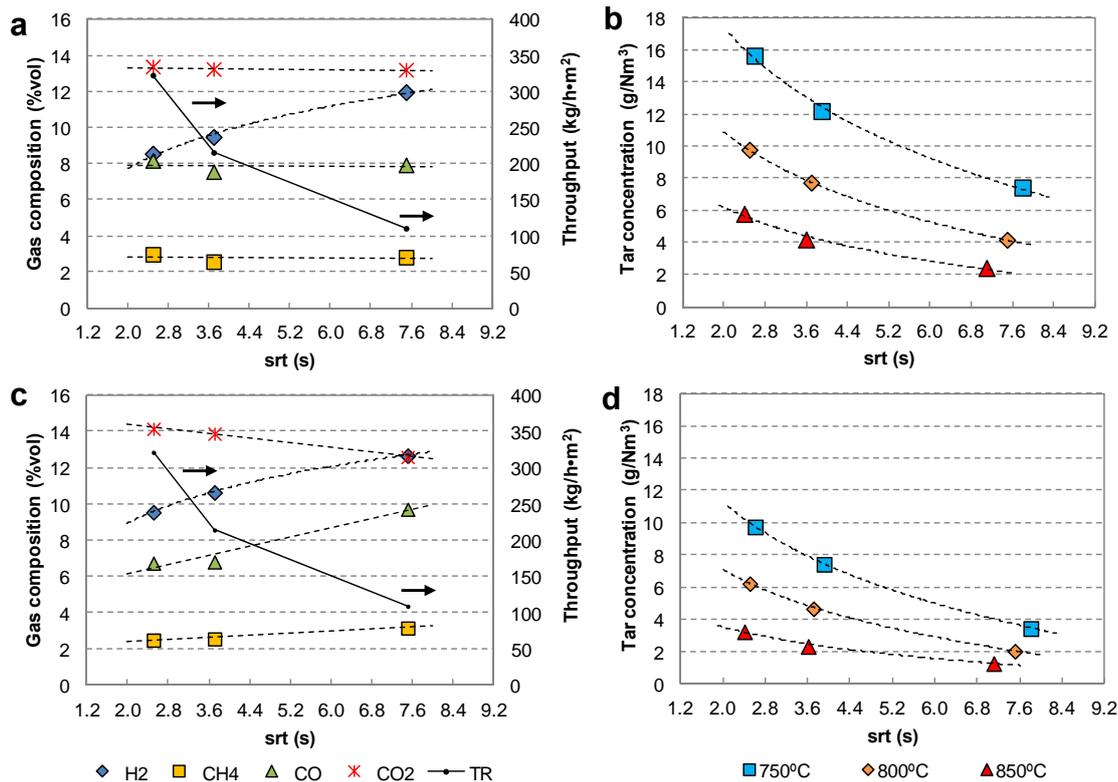
The changes in gas composition (and hence in their LHV) and the slight increase in gas production related to the conversion of tars into permanent gases, modified the carbon conversion and the cold gas efficiency. On average, under the reference TR, the carbon conversion increased 4% and the cold gas efficiency 10% when dolomite was used.

3.3 Influence of the throughput, TR

Fig. 3 shows the effect of the throughput (TR) on the produced gas composition and the tar concentration. Actually, the data are presented as a function of the space residence time (srt) of the gas in the gasifier (*srt*, is defined as the gasifier volume divided by the air volumetric flow rate). As previously explained in Sección 2, the TR was modified between 110, 225 and 322 kg/h·m² by increasing the flow rate of sludge fed to the gasifier. In order to keep the ER at 0.3, the air flow rate introduced in the gasifier and thus, the *srt*, had to be modified (Table 2). The trends shown in Fig. 3a for 800°C were similar to those found for the other temperatures in tests without catalyst. There was an increase in H₂

content with the *srt* while the CH₄, CO and CO₂ production slightly changed. According to Chen (2003), the longer is the residence time the higher is the cracking reaction, which would partially explain the increase in H₂ content and the decrease in C_nH_m production obtained by increasing the *srt* (Table 2). Different results were found in tests with dolomite (Fig. 3c). It can be seen that the production of H₂, CO and CH₄ increased by increasing the *srt* while the CO₂ content decreased. These results are in agreement with Hernández (2010), who stated that an increase of the *srt* increases the concentration of the combustible species in the producer gas as a result of the closer approach to equilibrium values.

FIGURE 3 Effect of the throughput (TR) on the gasification products. (a) effect on gas composition at 800°C in tests without catalyst; (b) effect on tar concentration at 750°C, 800°C and 850°C in tests without catalyst; (c) effect on gas composition at 800°C in tests with dolomite; (d) effect on tar concentration at 750°C, 800°C and 850°C in tests with dolomite



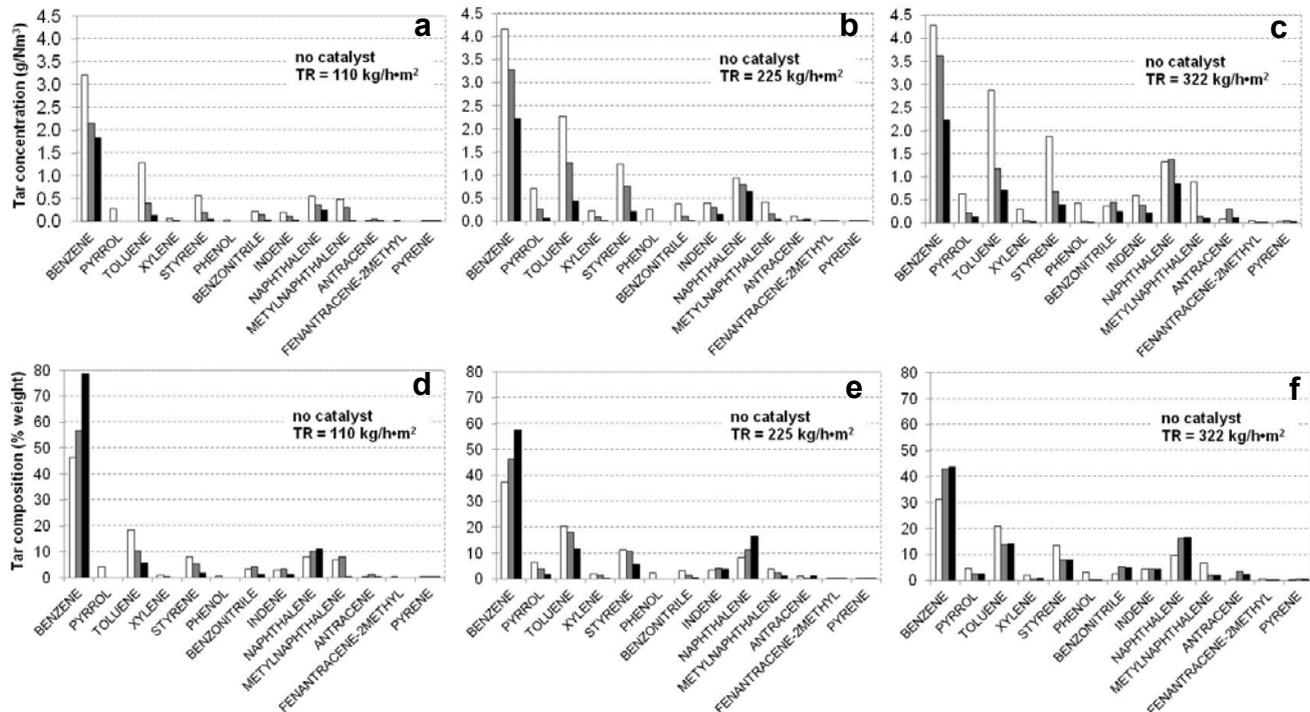
Regarding tar production, there was an increase in tar concentration when *srt* decreased (higher TR) under all the temperatures tested (Fig. 3b and Fig. 3d). Low *srt* reduce the effect of the cracking and reforming reactions on the tar removal. As it can be seen in Fig 2, even under the highest TR, the tar removal efficiency of the dolomite kept almost constant and always above 35%. In fact, at 850°C, no relevant differences in dolomite performance were found in the range of TR studied. As far as CGE and X_C were concerned, both parameters increased with the *srt* as a result of slight increases of the LHV and the Y_{gas} (Table 2).

3.4 Effect of gasification conditions on tar composition

Even though the GC/MS detected a lot of peaks, most of them were considered negligible. From these peaks, 13 compounds (summing up more than 95% of the total tar weight) were selected as major components of the tar in order to simplify the analysis presented (Fig. 4). Benzene, toluene, styrene and naphthalene were the most important components of the tar produced. As shown in Fig. 4a,b,c, the yield of all the components evaluated decreased when the temperature increased over the range of TR studied. This can be explained by the effect of the thermal cracking and the reforming reactions (Phuphuakrat, 2010). Regarding the effect of the temperature in the relative composition of the tar, different trends were found depending on the component observed Fig. 4d,e,f. Even though the relative production of most of tar components decreased, the benzene and naphthalene behaved in a different way and their relative yield increased by increasing the temperature. These findings agree with those reported by Kinoshita (1994). According to these authors, lower temperatures favour the formation of aromatic tar species with diversified substituent groups (such

as phenol, xylene and toluene) while higher temperatures favour the formation of more stable aromatic tar species without substituent groups such as benzene and naphthalene. For example, with rising temperature, phenols are decarbonylated into radicals of the cyclopentadiene type, supposed to be intermediates in the formation of indene and naphthalene (Brage, 1996).

FIGURE 4 Effect of the TR and temperature on the tar yield and composition (□ 750°C; ■ 800°C; ■ 850°C).



The yield of all the components increased with the TR because higher TR are related to lower *srt* and, therefore, the time available for the tar removal reactions is lower. As for changes in tar composition, unlike the rest of components, the relative production of benzene decreased as TR increased.

Regarding the effect of the dolomite in the tar yield and tar composition, the use of the catalyst decreased the yield of all the components studied. Although not shown, no relevant differences were found in the relative compositions of the tar in the presence of dolomite and the bar diagrams were similar to those presented in Fig. 4d,e,f.

4 CONCLUSIONS

This work deals with the effect of the temperature, the throughput (TR) and the use of dolomite as primary catalyst on the sewage sludge gasification products.

- The production of combustible gases (H_2 , CO and CH_4) increased by increasing the temperature, making the produced gases more suitable for thermal applications. Over the range of temperatures studied (750-850°C), reductions of tar yield of up to 67% were found. The changes in gas composition and the increase in gas production detected at higher temperatures increased both the carbon conversion and the cold gas efficiency.
- The use of dolomite as primary catalyst resulted in an additional increase of H_2 and CO content in the gas produced. However, the main advantage of the use of this catalyst is the decrease in tar yield. Under the gasification conditions studied, the tar removal efficiency ranged between 35% and 51%. Even under the highest TR, the tar removal efficiency of the dolomite kept almost constant, especially at 850°C.
- Lower TR (higher gas residence time) resulted in an increase in H_2 , CO and CH_4 content and a decrease in CO_2 production as a result of the closer approach to equilibrium values. On the other hand, higher TR increased tar production because of the reduction of the effect of the cracking and reforming reactions on the tar removal.
- Benzene, toluene, styrene and naphthalene were the most important components of the tar produced. Higher temperatures decreased tar production but increased the relative yield of benzene and naphthalene. The use of dolomite reduced the tar yield but did not produce relevant changes in its relative composition.

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