

PARTICLE COMMINUTION PHENOMENA OF A WET SEWAGE SLUDGE DURING FLUIDIZED BED PYROLYSIS AND COMBUSTION

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Abstract

Thermo-conversion of sewage sludge in fluidized beds is a feasible and attractive solution to reduce the amount of waste directed to landfilling and, at the same time, to mitigate the environmental issues related to the disposal of sewage sludge. The aim of this work was focused on the particle comminution phenomena of a wet sewage sludge particle occurring during fluidized bed pyrolysis and combustion. The experimental investigation was carried out with the aid of different and complementary experimental protocols. The phenomenology of the devolatilization and char burn-out has been studied in a quartz bench-scale fluidized bed by visual observation: self-segregation of fuel particle to the top of the bed during devolatilization and flameless combustion of the volatile matter were observed. SEM/EDX and image analysis have been carried out on char and ash particles generated during the experimental campaign highlighting the formation of a porous outer layer and cavities inside the particles. The physical and chemical features of ash and char particle have been characterized. The effect of particle comminution phenomena on devolatilization stage (air as fluidizing gas) and pyrolysis (nitrogen as fluidizing gas) has been investigated by means of the time series of gaseous species measured at the exhaust varying the initial size of fuel particle. Particle comminution phenomena have been characterized varying the nature oxidative and non-oxidative of the environment, the bed material size, the fluidization velocity excess and the initial size of fuel particle. Particle fragmentation occurs mainly during drying and pyrolysis or devolatilization and scarcely during char burn-out when the initial size of fuel particle exceeds a critical value depending on the bed material size and fluidization velocity excess. Particle comminution phenomena are strongly and moderately emphasized by bed material size and fluidization velocity excess, respectively.

Introduction

Sewage sludge management is one of the most important environmental problems. The necessity of construction of new biological municipal wastewater treatment plants determines a continuous increase in the volume of sewage sludge generated, even if a feasible solution to the disposal of the expected enormous quantities of sludge must be still found. The restrictive environmental legislation that limits more and more the landfilling of this biodegradable waste and the progressive decrease of the use of sludge in agriculture, that can only be considered under very well controlled conditions due to the presence of heavy metals and pathogens, indicates an ever more increasing interest in sludge thermal processes, in particular, in sludge incineration. Combustion and co-combustion are the mostly viable strategies to dispose sewage sludge [1] and, in addition, sewage sludge, due to its biogenic nature, is a promising substitute of fossil fuels in the light of the increasing concern to CO₂ emissions. Fluidized bed combustion, gasification and pyrolysis have been indicated as one of the best option, due to operation flexibility and to high efficiency and low pollutant emissions

achieved with different biogenic fuels, used either alone or in combination with fossil fuels [2-6]. The success of fluidized bed combustion technology for sewage sludge can be attributed, apart from minor specific advantages, by considering the great volume reduction of the produced ash and the destruction of organic micro-pollutants and pathogens [1,7]. However even if several bed units are in operation for sewage sludge combustion, fundamental work on the comprehension of basic mechanisms (e.g. sludge drying, release and combustion of volatiles, combustion of the high-ash content char) taking place during fuel conversion has received considerably less attention and requires additional investigations [8-15]. In particular, the particle comminution phenomena play a key role in the operation of industrial units as regards both environmental issues (particulate emissions) and the stabilization of bed inventory constituted by fuel ash. In this context, sewage sludge has twofold nature: a biogenic component derived by production process nature, and, typically, inorganic one due to the stages of stabilization and conditioning of the wastewater treatment. As a consequence, attrition and fragmentation of sewage sludge particles during thermo-chemical processes could significantly influence the operation of fluidized bed in terms of ash management. On basis of present knowledge, it can be speculated that, as other biogenic fuels, characterized by a high moisture and volatile content, a fragile structure, often anisotropic and a high intrinsic reactivity with respect fossil fuels (coal): i) it will be subject to extensive attrition and fragmentation; ii) it will give rise, upon devolatilization, either to highly porous, friable chars or even to a multitude of fragments of very small size [16]; iii) it will be subjected to self-segregation phenomena during devolatilization [17-18]: coarse fuel particles rapidly rise to the bed surface under the action of the volatiles bubbles and remain there until devolatilization ends; iv) extensive post-combustion of volatiles occurs in the splashing region and/or in the freeboard [19].

The present paper aims at contributing to a better understanding of particle comminution phenomena of a wet sewage sludge during fluidized bed pyrolysis and combustion with the aid of different and complementary experimental protocols. Devolatilization/pyrolysis and char burn-out processes have been analyzed by visual observation and by the time series of gaseous species measured at the exhaust. Particle fragmentation has been characterized in terms of number and dimension of the mm-sized generated fragments as a function of the initial size of the fuel particle for different operating conditions.

Experimental

A stainless steel atmospheric bubbling fluidized bed combustor 41 mm ID and 1 m high was used for pyrolysis/combustion and fragmentation experiments (Fig. 1A). A 2 mm thick perforated plate with 55 holes (ID 0.5 mm) disposed in a triangular pitch was used as gas distributor. A 0.6 m high stainless steel column for gas preheating and mixing was placed under the distributor. Two semi-cylindrical 2.2 kW electric furnaces were used for heating the fluidization column and the preheating section. Bed temperature, measured by means of a chromel-alumel thermocouple placed 4 mm above the distributor, was regulated by a PID controller. A part of freeboard (0.4 m high) was kept unlagged in order to minimize fines post-combustion in this section. Gases were fed to the column via high-precision digital mass flow-meters. The fluidization column top section was left open to the atmosphere. A stainless steel circular basket could be inserted from the top in order to retrieve fragmented and unfragmented particles from the bed. The tolerance between the column walls and the basket was limited to reduce as much as possible the amount of particles left in the bed when pulling out the basket. A basket mesh of 0.8mm was used, so that the bed material could easily pass through the net openings. Fragments smaller than about 0.8mm were lost through the basket net openings. A stainless steel probe located along the freeboard of the fluidization column has been used in order to convey a fraction of the exhausted gases to gas analyzers. A high

Table 1. Properties of the wet sewage sludge.

LHV (as received), kJ/kg	1245	Ash analysis (as received), %_w	
Proximate analysis (as received), %_w		<i>Na</i>	0.019
<i>Moisture</i>	77.8	<i>Mg</i>	0.081
<i>Volatiles</i>	14.5	<i>Al</i>	1.002
<i>Fixed carbon</i>	1.0	<i>P</i>	0.496
<i>Ash</i>	6.7	<i>K</i>	0.085
Ultimate analysis (dry basis), %_w		<i>Ca</i>	0.547
<i>Carbon</i>	34.7	<i>Ti</i>	0.009
<i>Hydrogen</i>	5.4	<i>Cr</i>	0.019
<i>Nitrogen</i>	5.1	<i>Mn</i>	0.006
<i>Sulfur</i>	0.5	<i>Fe</i>	0.328
<i>Chlorine</i>	1.2	<i>Ni</i>	0.062
<i>Ash</i>	30	<i>Cu</i>	0.025
<i>Oxygen (by diff.)</i>	24	<i>Zn</i>	0.023

complete, the resulting char or ash was retrieved by means of the basket in order to find out the number of the produced fragments and to evaluate their mean particle size. The experiments have been characterized in terms of initial fuel particle size. The influence of bed material size has been investigated adopting quartz in the size range both 150-300 μm and 400-600 μm and maintaining the same gas velocity excess with respect the minimum fluidization velocity ($U-U_{mf}=0.38\text{m/s}$). The influence of fluidization velocity on particle comminution phenomena has been investigated for bed material sieved in the size range 400-600 μm conducting experiments at a fluidization velocity of about 0.8m/s. During all the duration of the tests, the time series of the concentrations of gaseous species measured at the exhaust were acquired and stored for a further post processing. The experiments were repeated to collect a statistically significant number of fragments.

The experiments, carried out in the experimental apparatus equipped with the quartz fluidization column, consisted of the injection, from the top of the column, of a single wet sludge particle into a bed (quartz, 150-300 μm) gently fluidized by air ($U=0.25\text{m/s}$) and kept at constant temperature of about 850°C. Video recordings of the upper part of bed and of freeboard have been performed during the combustion of fuel particles characterized by different initial size.

Selected ash and char particles were observed under a scanning electron microscope (SEM; Philips XL30 with LaB6 filament) and subjected to energy dispersive X-ray (EDX; Edax DX-4) elemental analysis. Few samples were embedded in epoxy resin and then cut and polished for SEM observation and EDX analysis of particle cross-section.

Results and discussion

Figure 2 reports some snapshots captured during the combustion of a 2cm wet sewage sludge particle (4.24g) in the experimental apparatus equipped with quartz fluidization column. Bed material was quartz sieved in the size range 150-300 μm and the fluidizing gas velocity was 0.25m/s. It is worth to note that this low fluidization velocity has been set in order to, on one hand, better visualize the fuel particle motion during combustion process and, on the other, to reduce the influence of bed material and of the operating conditions on the particle comminution phenomena. The upper part of the bed and the first part of the freeboard are dark red and brighter red, respectively, in the images. The origin of time scale corresponds to the time at which the fuel particle approaches the bed surface.

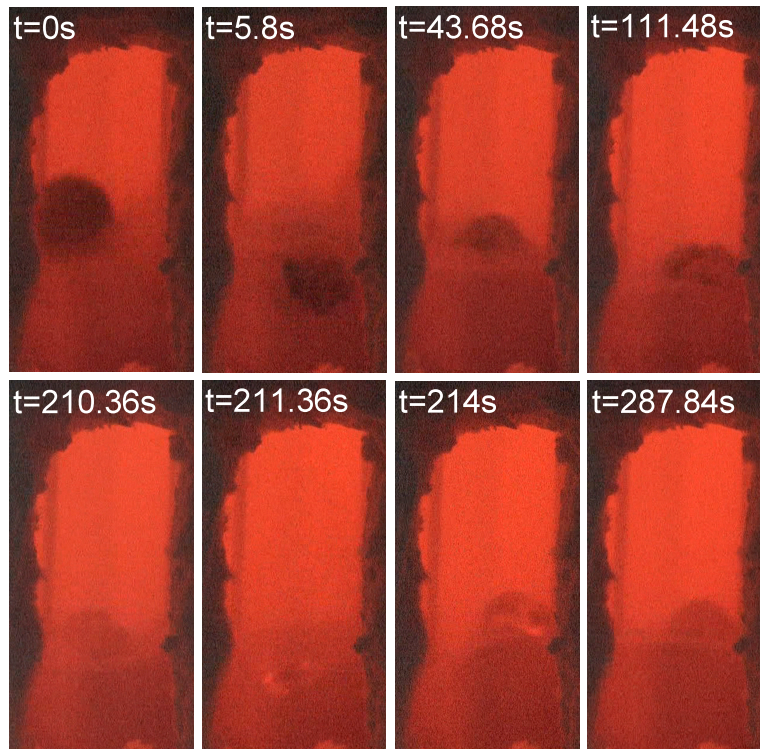


Figure 2. Snapshots captured during devolatilization and char burn-out of a particle of wet sewage sludge. Fluidizing gas: air; fluidization velocity: 0.25m/s; fuel particle size: 2cm; frame rate 25fps.

On the basis of the analysis of the captured images, it can be recognized that the fuel particle appears darker than bed material for $0 < t < 210$ s, then it becomes brighter for $210 < t < 288$ s and, finally, it assumes more or less the same colour of bed material for $t > 288$ s. The different colour observed during combustion is directly related to the fuel particle temperature: during drying and devolatilization fuel particle temperature is smaller than bed temperature, then it overcomes bed temperature during char burn-out and, finally it decreases toward bed temperature when the combustion process is complete. As a consequence, the recognition of the time intervals corresponding to different fuel particle colours can provide an estimate of both devolatilization and char burn-out time.

Taking into account these considerations, the analysis of the acquired images highlights that: i) the fuel particle segregates to the top of the bed during all the drying and devolatilization processes, however, bed material partially covers fuel particle also when it is on the top of the bed ($t=43.68$ s, $t=111.48$ s); ii) volatile matter combustion takes place in flameless conditions; iii) fuel particle segregation also takes place during char burn-out even if bed material often covers fuel particle; iv) particle comminution phenomena seem to be negligible both during devolatilization and char burn-out.

Considering the fuel particle properties are such that devolatilization and drying mainly occur in parallel, the absence of volatiles flames can be mainly due to the large amount of moisture present in the fuel particle which reduces both the particle temperature during devolatilization and the heating value of the released volatile matter. In these conditions, the volatile matter combustion takes place homogeneously mainly in the freeboard without hot spots (flameless combustion). It is also worth to noting that the diagnostic techniques - the flame period and flame extinction time methods - based on the detection of volatile flames around the devolatilizing fuel particles by visual observation, cannot be applied to this kind of solid fuel. Instead, the techniques consisting of the analysis of the time-resolved gas

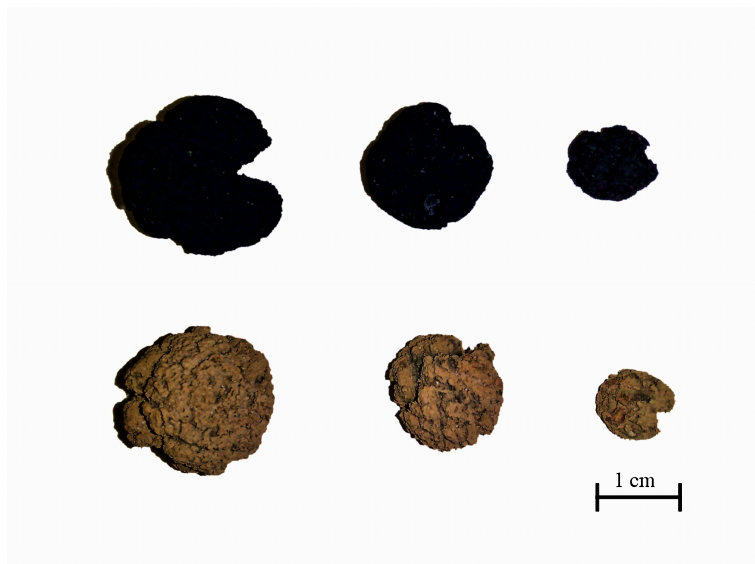


Figure 3. Images of three sludge particles of about 3, 2 and 1cm in size respectively after tests of pyrolysis (black particles) and devolatilization and char burn-out (brown particles).

fluidized bed was operated with quartzite in the size range of 150-300 μ m and with a fluidization velocity of 0.25m/s (i.e. in the same operating conditions of figure 2). The major observation is that particles after either pyrolysis or combustion tests undergo a negligible primary fragmentation: only one fragment was retrieved by means of basket at the end of the thermo-conversion process. It is necessary to recall that fragments smaller than about 0.8mm were lost through the basket net openings.

A further analysis of the particles shows that the size of fuel particles is reduced by a factor of about 0.75-0.8 after both pyrolysis and combustion, whereas the weight loss is in agreement with the data obtained with the proximate analysis: fixed carbon and ash content 1.0 and 6.7%_w, respectively. On the basis of these data, it can be concluded that the particles result to be very porous after thermo-conversion processes.

As matter of fact, it becomes more evident from the analysis of Figure 4 that reports two images of the internal structure

concentration profiles measured during devolatilization/pyrolysis and char burn-out are still adequate [10,15,20-21].

Increasing the initial fuel particle size, the devolatilization and the char burn-out time increases, but the combustion pattern remains that reported in figure 2.

Figure 3 shows a photograph of three sludge particles of about 3, 2 and 1cm in size retrieved from the fluidized bed by means of basket after tests of pyrolysis (black particles) and devolatilization and char burn-out (brown particles) carried out in the experimental apparatus equipped with stainless steel fluidization column. The

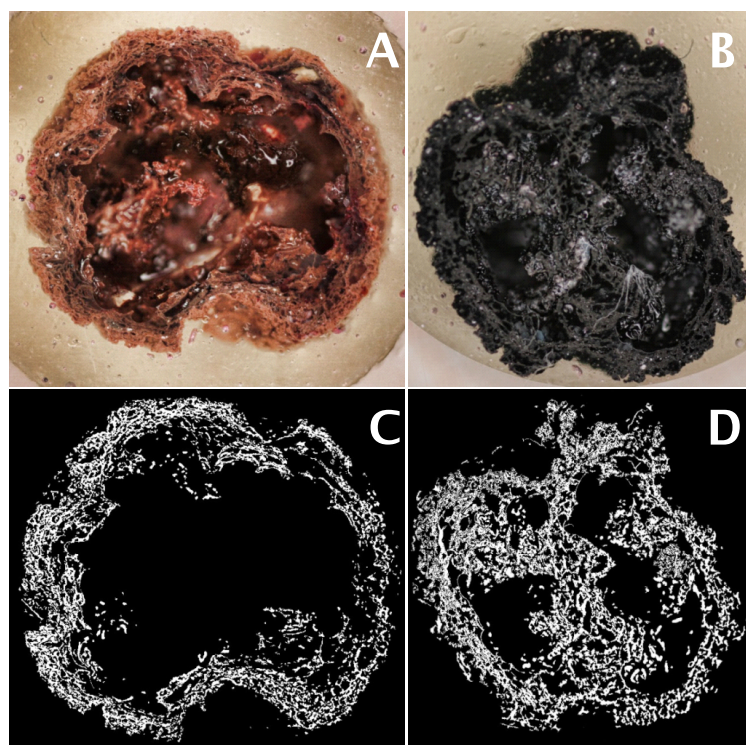


Figure 4. Images of the internal structure of two sludge particles after tests of devolatilization and char burn-out (A) and pyrolysis (B) and the resulting post-processed images (C and D, respectively)

of two fuel particles after pyrolysis and combustion together with the post-processed images: an ash and a char particle having a size of about 1cm were cut in the middle and embodied in a epoxy resin for further SEM/EDX analysis. The images show that, both ash and char particles, exhibit a highly anisotropic pore structure characterized by large pores and cavities and by a coherent outer layer which, probably, preserves the structural integrity of the particles. The major difference between ash and char particle is that the ash internal structure is more porous: the large cavities present in the char particle seem to be collapsed to one when the ash particle is considered. The images reported in figure 4A and B were further analyzed by an post-processing software in order to estimate voidage fraction along the cross section. The post-processed images, reported in figures 4C and D, enable an estimate of voidage fraction of about 0.84 and 0.7 for ash and char particle, respectively.

Figure 5 reports SEM images of ash and char particles of about 1cm already reported in figures 3 (external surface), 4A and 4B (internal structure). The SEM images allowed to observe in more details the physical structure of the sewage sludge particle after pyrolysis and combustion. The analysis of the both cross section and the external surface of the particles highlights that the outer layer of both ash and char particle is made of porous coherent structure, probably, of partially molten matter characterized by a thickness of about 500 μ m. The EDX analysis obtained for these SEM images is reported in table 2 and it is compared with ash analysis (ICP-MS) of the fuel particle reported in table 1. On the whole, it can be observed that the ash chemical composition of the particles obtained by SEM-EDX is very similar to that measured by ICP-MS on the fuel sample as received. In particular, it can be observed: i) the physical structure of char and ash particle is substantially constituted by all the major elements present initially in the sewage sludge with, approximately, the same concentration; ii) Fe and Ca concentration in the char and in the ash particle is slightly lower than that measured in the fuel sample as received. The latter result could be due to the volatilization of CaCl_2 and FeCl_3 during devolatilization or pyrolysis stage.

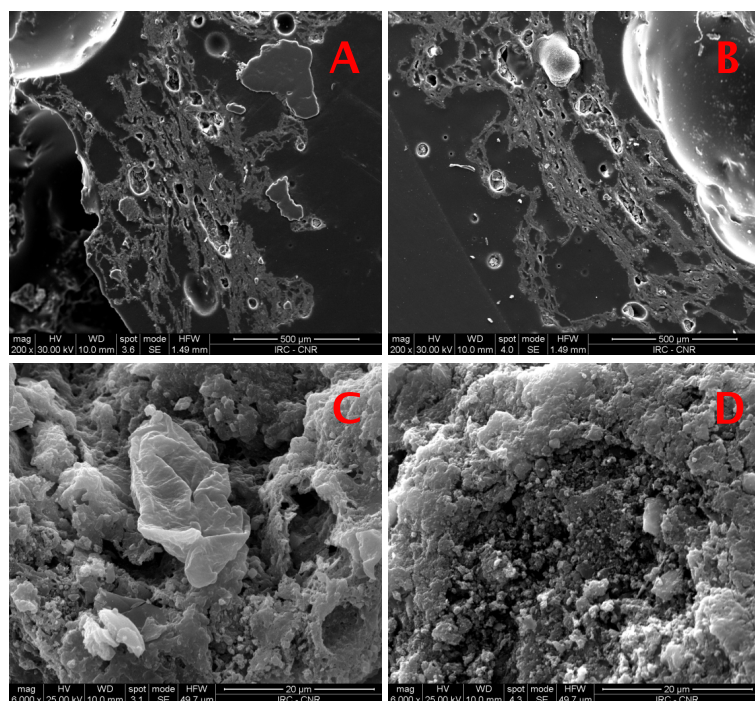


Figure 6. SEM images of the internal structure and external surface of outer layer of two sewage sludge particles after combustion (A and C, respectively) and pyrolysis (B and D, respectively).

The effect of particle comminution phenomena on the fuel devolatilization/pyrolysis and char burn-out patterns of a single particle of sewage sludge at 850 $^{\circ}$ C has been assessed on the basis of the analysis of the time series of gas concentration measured at the exhaust. Reported data referred to tests carried out with quartzite in the size range of 150-300 μ m and at a fluidization velocity of 0.4m/s. Figure 6 reports the time series of gas concentration (CO , CO_2 , SO_2 , NO , H_2 , CH_4) corresponding to three different typical pyrolysis tests carried with a single fuel particle. These three fuel particles with an initial averaged size of about 12, 22 and 28mm retrieved by means of basket after pyrolysis showed a

Table 2. Normalized ash chemical composition.

Analysis	Ash analysis, % _w (on ash basis)				
	ICP-MS	SEM/EDX			
	Sample	Outer layer		External surface	
Material	fuel (a.r.)	ash	char	ash	Char
Na	0.7	1.2	0.9	3.6	1.3
Mg	3.1	3.3	2.0	4.4	2.5
Al	37.8	39.9	43.0	36.9	31.6
P	18.7	23.7	21.8	19.2	18.1
K	3.2	3.3	4.2	3.5	2.9
Ca	20.7	15.5	14.7	15.7	22.1
Ti	0.3	1.1	1.1	1.5	5.7
Cr	0.7	0.5	1.0	1.3	0.8
Fe	12.4	9.2	9.0	10.3	12.3
Ni	2.3	2.2	2.3	3.4	2.7

negligible fuel particle comminution.

The analysis of the time-resolved gas concentration measured at the exhaust highlights that the time-history of release of measured species is different: shorter for NO and CH₄, longer for H₂ and CO, whereas it has an intermediate behavior for CO₂ and SO₂. As a consequence, the time-resolved CO₂ concentration profile has been adopted to estimate the pyrolysis time corresponding to the 95% of the total released amount which, in this case, results in 148, 305 and 340s for 12, 22

and 28mm fuel particles, respectively. As expected the pyrolysis time increases increasing the particle size.

Figures 7 reports the time series of gas concentration (CO, CO₂, NO) corresponding to typical combustion tests of un-fragmented and fragmented fuel particles. In particular, the time-resolved gas concentration profiles during the combustion of 12.7 and 22.8mm un-fragmented fuel particles and of a fragmented 21.8mm fuel particle have been reported in figure 7 in order to compare the combustion pattern when the fragmentation history is

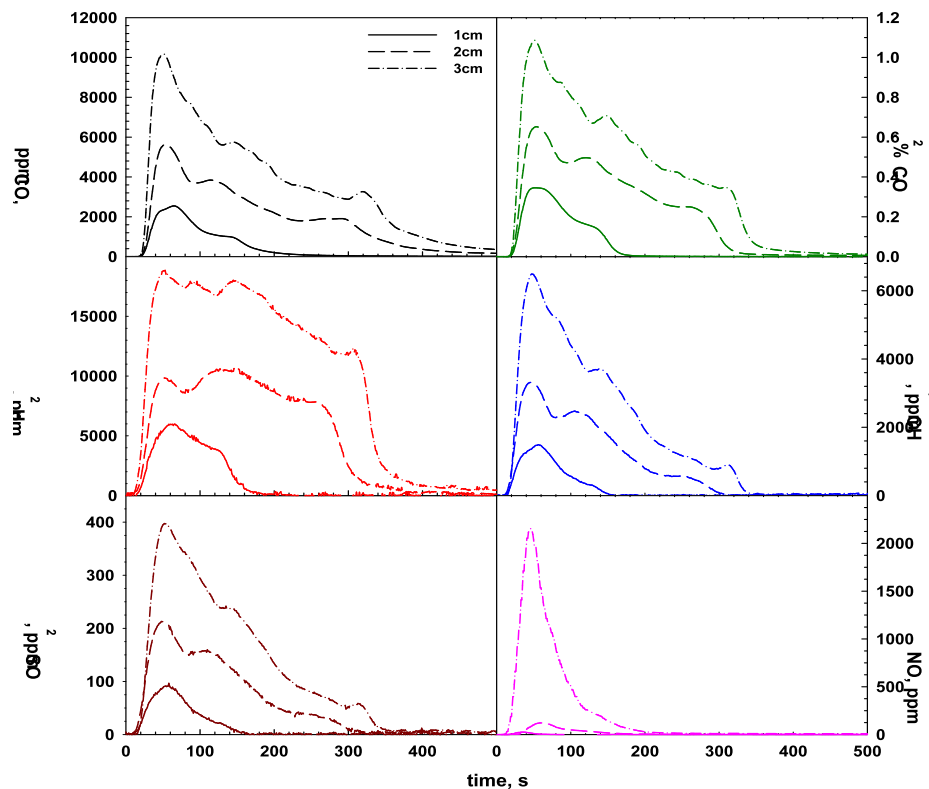


Figure 6. Time-resolved CO, CH₄, H₂, SO₂, NO and CO₂ concentration during pyrolysis experiments carried out with single un-fragmented sewage sludge particles characterized by different initial size. Initial fuel size: 12.4, 22.1 and 27.8mm.

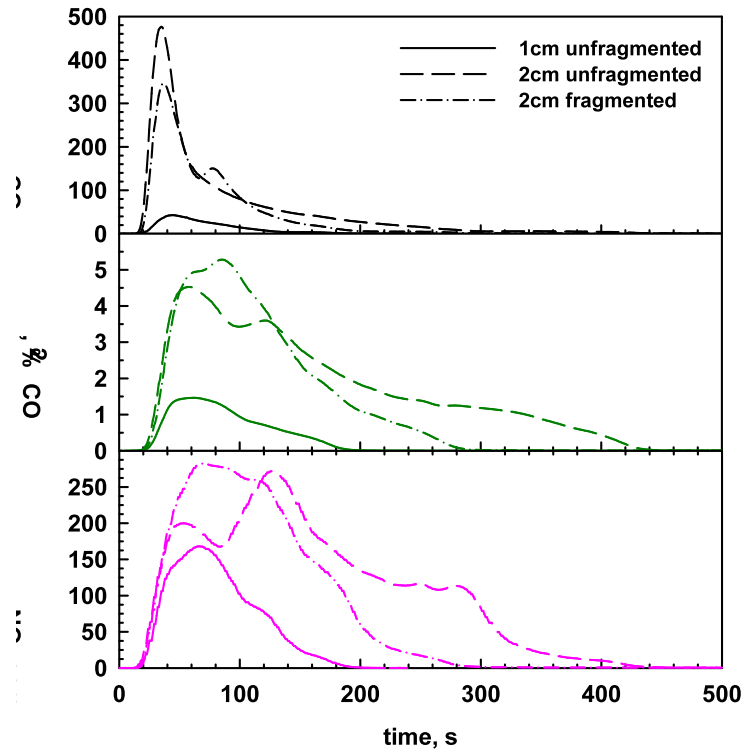


Figure 7. Time-resolved CO, NO and CO₂ concentration during combustion experiments carried out with single un-fragmented sewage sludge particles (initial size: 12.7 and 22.8mm) and with a 21.8mm fragmented sewage sludge particle.

different. On the basis of the analysis of the time series of CO₂ concentration, it has been evaluated both devolatilization and complete burn-out time. For the un-fragmented particles the devolatilization time was of 110 and 250s for 12.7 and 22.8mm fuel particles respectively, while the complete burn-out time was 161 and 358s, respectively. For the fragmented particle the results were of 160 and 230s for the devolatilization and complete burn-out time, respectively.

The comparison between combustion and pyrolysis results (Figures 6 and 7) highlights: i) CO₂ profile presents a shoulder at the end of the process due to char-burn-out; ii) the devolatilization time is smaller than pyrolysis time indicating that also without the presence of flames volatile matter combustion enables faster the devolatilization process; iii) as expected, particle fragmentation reduces both devolatilization and char burn-out time.

The influence of bed material size and fluidization velocity on the particle comminution phenomena has been assessed during both pyrolysis and combustion of sewage sludge varying the initial size of the fuel particle (d_{in}). Figures 8 and 9 report the number (N_{out}) of particle fragments collected (i.e. multiplication factor as each test was carried out with a single particle) and the average size (d_{out}^{max}) of the largest fragment, respectively, at the end of pyrolysis as well as of combustion tests as a function of d_{in} . Reported data referred to experiments carried out with quartz sieved in the size range 150-300 μ m at a fluidization velocity of 0.4m/s and with quartz sieved in the size range 400-600 μ m at a fluidization velocity of 0.47 and 0.8m/s.

Particle comminution results reported in both figures highlight that the number of fragments generated by particle fragmentation and d_{out}^{max} do not significantly depend on the nature oxidative (air, combustion tests) or non-oxidative (nitrogen, pyrolysis tests) of the environment where the thermo-chemical process takes place whatever operating condition is considered. This result indicates that the particle fragmentation occurs mainly during drying

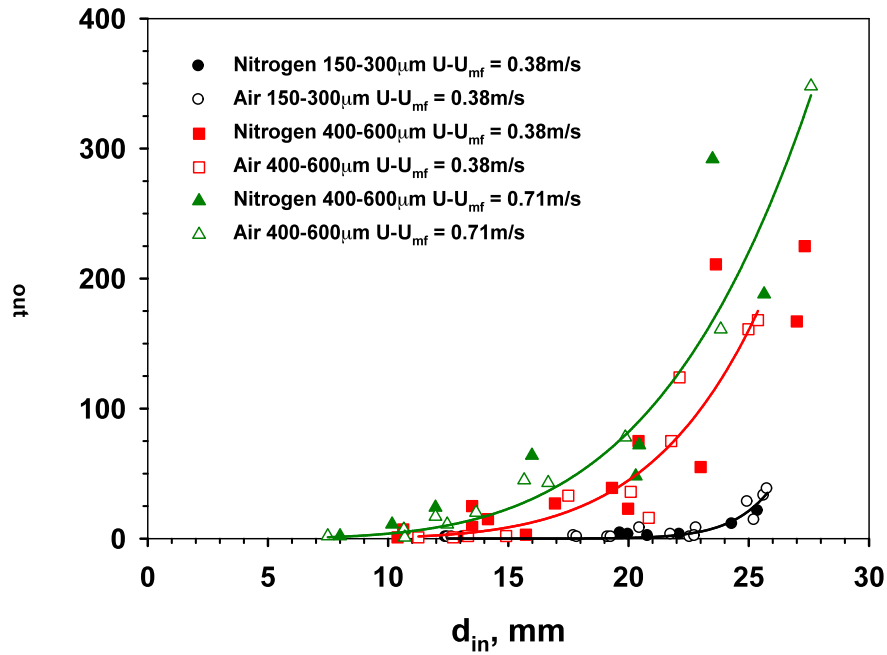


Figure 8. Multiplication factor as a function of average initial size of the fuel particle for different operating conditions.

and pyrolysis or devolatilization and scarcely during char burn-out. It is likely that the poor fixed carbon content of the tested sewage sludge is finely dispersed into fuel matrix and it does not give any contribution to the mechanical structure of the particle in all the stages of the thermo-chemical processes.

The analysis of data of multiplication factor as a function of d_{in} for tests carried out with quartzite in the size range of 150-300 μm and operating the bed with a fluidization velocity of 0.4m/s ($U-U_{mf}=0.38\text{m/s}$) shows that: i) it exists a critical initial (d_{in}^c) fuel particle size, in the order of 18-20mm, below which the sewage sludge particle does not fragment; ii) N_{out} strongly increases with d_{in} once d_{in} exceeds d_{in}^c . Increasing the bed material size (400-600 μm) and keeping constant the fluidization velocity excess ($U=0.47\text{m/s}$ and $U-U_{mf}=0.38\text{m/s}$), N_{out} as a function of d_{in} shows a similar behavior highlighting a smaller value of d_{in}^c , in the order of 11-12mm. The main observed difference is that N_{out} strongly increases with bed material size keeping constant d_{in} . It is worth to note that the collected particles during tests carried out with finer bed material (150-300 μm), that did not undergo fragmentation, were completely filled with bed material particles which could escape from the pores of the char or ash particle by gently shaking. Probably, the fuel particles filled by bed material particles could be less subjected to fragmentation compared to empty particles. This phenomenon was not observed in tests carried out with the coarser bed material (400-600 μm), during which fuel particles undergo a greater particle comminution.

On the other hand, keeping constant bed material size (400-600 μm) but increasing the fluidization velocity excess ($U=0.8\text{m/s}$ and $U-U_{mf}=0.71\text{m/s}$) it can be observed that: i) N_{out} as function of d_{in} has a similar trend; ii) d_{in}^c is smaller in the order of 7-8mm; iii) N_{out} slightly increases with fluidization velocity excess keeping constant d_{in} .

Figure 9 reports the average size of the largest fragment generated (with and without particle fragmentation) as a function of the initial average size of the fuel particle. It can be observed that, whatever the operating condition is considered,: i) for d_{in} smaller than d_{in}^c , d_{out}^{max} follows the line $0.78d_{in}$ which describes the particle shrinkage in absence of significant

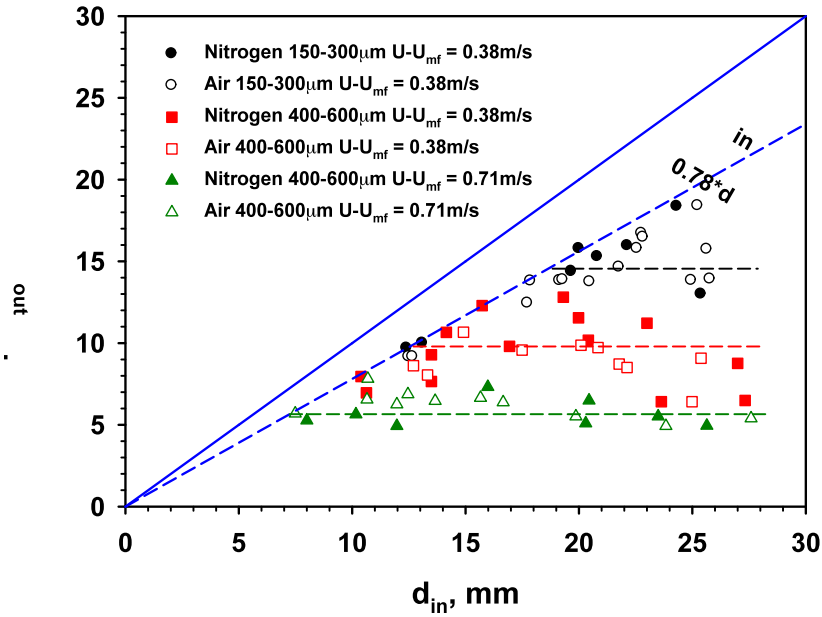


Figure 9. Average size of the largest fragment produced by the particle comminution as a function of average initial size of the fuel particle for different operating conditions.

particle comminution phenomena; ii) for d_{in} larger than d_{in}^c , d_{out}^{max} substantially remains constant increasing the initial size of the fuel particle at a value of about 15, 10 and 5mm for tests carried out with 150-300 μ m quartz at $U-U_{mf}=0.38$ m/s, with 400-600 μ m quartz at $U-U_{mf}=0.38$ m/s and at $U-U_{mf}=0.71$ m/s, respectively. As a consequence, the size of the largest fragment decreases with increasing both bed material size and fluidization velocity excess once d_{in} is larger than d_{in}^c .

Conclusions

The phenomenology of the devolatilization and char burn-out of a wet sewage sludge investigated by visual observation in a quartz bench-scale fluidized bed highlighted that the fuel particle segregates to the top of the bed during devolatilization and volatile matter burns in flameless conditions, probably, due to the large amount of moisture present in the fuel particle. SEM/EDX and image analysis of un-fragmented char and ash particles showed the formation of a porous coherent outer layer and large cavities inside the particles, especially for ash ones. The voidage fraction of the cross section of ash and char particles has been estimated in the order of 0.84 and 0.7, respectively. The inorganic chemical composition of ash and char particle results to be similar to that measured in the fuel sample as received. Particle comminution phenomena reduce the time-scale of devolatilization (air as fluidizing gas), pyrolysis (nitrogen as fluidizing gas) and char burn-out as it results evident from the analysis of the time series of gaseous species measured at the exhaust. Particle comminution phenomena have been evaluated by retrieving fragmented and un-fragmented particles by means of a stainless steel circular basket inserted from the top of the fluidized bed in terms of the number (N_{out}) of particle fragments collected and the size (d_{out}^{max}) of the largest fragment at the end of pyrolysis and combustion as a function of initial size of fuel particle (d_{in}) for different operating conditions. It was observed that: i) particle fragmentation occurs mainly during drying and pyrolysis or devolatilization and scarcely during char burn-out; ii) N_{out} is equal to one for d_{in} smaller than a critical size of fuel particle (d_{in}^c), then it strongly increases with d_{in} ; iii) N_{out} strongly and moderately increases with bed material size and fluidization

velocity excess, respectively; iv) d_{in}^c decreases increasing both bed material size and fluidization velocity excess; v) d_{out}^{max} is equal to $0.78d_{in}$ in absence of particle fragmentation, whereas it remains constant increasing d_{in} (for $d_{in} > d_{in}^c$) at a value of about 15, 10 and 5mm for tests carried out with 150-300 μ m quartz at $U-U_{mf}=0.38$ m/s, with 400-600 μ m quartz at $U-U_{mf}=0.38$ m/s and at $U-U_{mf}=0.71$ m/s, respectively. On the whole, bed material size strongly raises particle comminution phenomena in terms of both number and size of fragments, whereas fluidization velocity excess plays a less significant role

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