Study of temperature profiles and weight loss during sludge smouldering with a macro TGA

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Abstract

We investigated the autoignition and self-heating of three different sludges in air. These are dried, waste water treatment sludges of different origin and share of municipal and industrial wastes. We used a custom testing apparatus to study representative (approx. 20g) sample size, simulating real accidental environment. We tested different heating rates (between 0.1 and 6.0 °C/min) and particle size distribution. We proved that: i) ignition can occur in more than one step; ii) the same solid behaves very differently by changing its particle size distribution and the applied heating rate; iii) the controlling factors are physical, rather than chemical; iv) the hazard is determined (and can be controlled) by modifying the particle size and its density, as well as the bed structure. The main conclusion is that the hazard of self-ignition and heating is largely controlled (thus manageable) by the physical properties of the solids, given that its reactivity in air is always high, once appropriate environmental conditions are met. Consistently, the self-heating hazard assessment requires investigation at a representative scale, where physical processes are relevant.

Keywords: self-heating, solids ignition, TGA, oxidation, smouldering, sewage sludge

1. Introduction

Sewage sludge is a by-product of biological wastewater treatment processes. It is a complex mixture of biomass, moisture, chemicals that are refractory to biological degradation and inorganic materials. Sewage sludge is being considered as a raw renewable energy source with zero-CO_2-emission [1]. The properties of sewage sludge are extremely variable, because each sewage sludge has specific origin and unique production conditions ([2], [3]). Previously studies proved that the sewage sludge can be used efficiently as fuel [4]. However, the handing and transportation of these waste requires the assessments of hazards associated with the self-ignition process. Thus, the properties of these material, together with the presence of fine size particles (due to wearing and tearing) and the presence of oxygen can trigger the reaction and generate enough heat resulting fire of them [5]. Díaz et al., [2], define several criteria for the risk assessment of sewage sludge self-ignition, creating characteristic risk plots and defining risk zones (low, medium, high and very high risk) in which to classify the sludges. Here we aim to compare the hazard of self-ignition onset in beds of dry sewage sludge, exposed to air. In previous contributions ([6], [7]), we reported about the self-heating of a specific, dried wastewater sludge, also at industrial scale. Here we elaborate on the hazard evaluation procedure and criteria, extending the comparison to other sewage sludges. We already showed [3] the gap between analytical techniques and measurements on larger samples. The standard TGA-based studies aim at identifying the intrinsic, chemical kinetics of biomass degradation ([8], [9], [10]). It has already reported that kinetics appears affected by the operation conditions [11], so that a large gap between TGA and real furnace behavior is observed, as reported both in purely experimental works ([2], [12]) and in experimental studies associated with modeling ([13], [14], [15]).

In this study, we question the need to finely characterize the chemical reactivity of the solids in air, struggling to minimize heat and mass transfer limitations, by minimizing the sample size, as in standard TGA. Rather, we suggest to properly investigate the ignition on larger samples (the so-called 'thick biomass') with a modified operating mode.

2. Materials and Methods

2.1 Sewage Sludges

Three different sewage sludges are used in this study: sewage sludge A (SS-A, 10% industrial-90% municipal); sewage sludge B (SS-B, 90% industrial-10% municipal); sewage sludge C (SS-C, 100% municipal). All sludges were granulated particles, from drying process at the wastewater treatment plants: the main properties of the sludges are in Table 1.

Table 1: Characterization of three raw sewage sludges

Parameters		SS-A	SS-B	SS-C
Proximate analysis	Moisture, %	23.46	13.62	12.57
	Volatile matter, %	65.64	66.37	64.55
	Fixed carbon, %	12.62	7.86	7.51
	Ashes, %	21.74	25.77	27.94
Atomic ratio	H/C	1.79	1.73	1.87
	O/C	0.90	0.50	0.70
	O/H	0.50	0.29	0.37
S content, %		1.60	8.20	2.00
Mean Diameter, mm		0.986	0.462	1.699
Intrinsic Density, kg/m^3		2577	2874	2750
Bulk or Apparent Density, kg/m^3		504.1	667.7	635.7
HHV, KJ/Kg		13340	18810	17280
RI*, min^{-1}		33.3	2.5	1.3
IT**, °C		225	204	210
MLT**, °C		247	229	246
Diaz risk classification		high	very high	high

* The Reactivity Index (RI= 100/t) is evaluated modifying the procedure reported in [16] for the Maciejasz index, carrying out the measure in a Dewar flask, on 10 g of sample, added with 15 mL of H_2O_2 (at 30%). The time (t, in minutes) necessary for the sample to increase in temperature by 15K from the initial temperature is measured.
** The IT (Induction Temperature) and MLT (Maximum Weight Loss Temperature) are measured as explained in [2], in our thermogravimetric tests with a heating rate of 2°C/min, from ambient temperature up to 250°C.

Table 1, underlines a number of differences among the three sludges, that can affect the onset and the propagation of biomass smouldering. The water content is much higher for the SS-A: moisture content is expected to delay the reactivity. SS-A has the highest FC and the lowest ashes content: it results in a lowest HHV. The ashes also varies significantly among these sludges: it should be minimized (ideally below 20%), to avoid frequent maintenance of the furnace to remove the ash and to avoid sensible heat losses in the ash formed, thereby lowering the overall furnace efficiency [17]. The mean particle size varies by a factor of 3: the lower the particle size, the higher the expected volumetric reaction rate ([18], [19], [20]). However, a smaller particle size (and specifically fines in the distribution) decreases the permeability of the bed to the co-reactant (oxygen in the air), so that the final influence on the reactivity must be evaluated. The main differences in metals content of sludges are related to their origin (municipal or industrial). The special case of B sludge is due to the industrial wastewater, from local tanneries [7]. The relation between S content and O/H ratio measure

70 the self-ignition risk, according to Díaz et al., 2019 [2]: following their criterion, SS-A and SS-C are classified
71 at high risk of self-ignition, whereas the SS-B is at very high risk, due to the high S content and the low O/H
72 ratio.

73 **2.2 Experimental procedure**
74 Figure 1 sketches the configuration of the experimental set-up used.
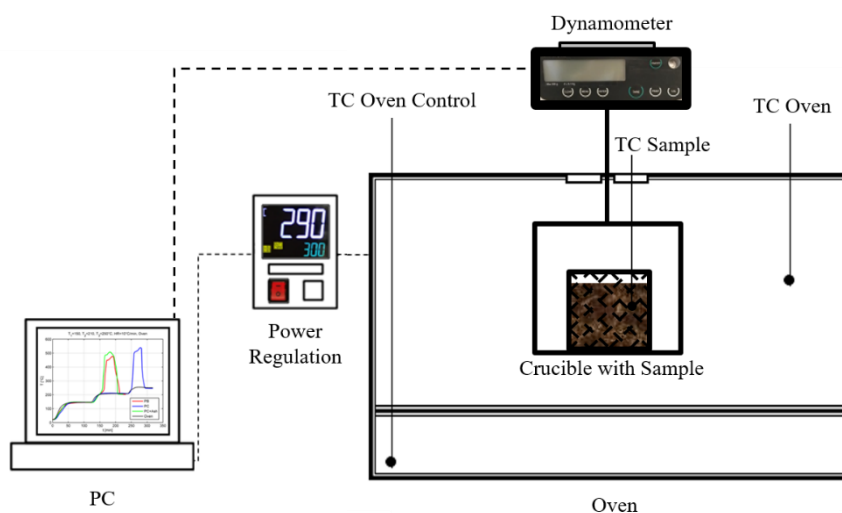
75
76 *Figure 1: Schematic representation of the macro TGA*

77 The smouldering reaction occurs in a gas-permeable cubic container made of a stainless steel aluminum net
78 (mesh size=0.5 mm) of 3.7 cm of length. The crucible is hanging from a scale and it is placed inside a static
79 oven, to evaluate the weight loss during biomass heating and ignition. The heating policy was a unique
80 temperature ramp from ambient temperature up to 250 ºC, with four different heating rates (HR): 0.1, 0.2, 2
81 and 6 °C/min.
82 The temperature of the sample, the oven and the weight of the sample are continuously recorded. When the
83 temperature of the oven reaches 250°C, it is kept constant until the sample weight settles to the final value.
84 This apparatus resembles a standard TGA on large samples, but contrary to the aim of the analytical
85 instruments, where the sample size is purposely minimized to limit the effect of heat and mass transfer
86 resistances, we deliberately investigated larger sample: in real applications the intrinsic reactivity of the
87 material is hindered by overwhelming physical processes, such as gas permeation in the bed and heat
88 dissipation across the bed.

89 **3. Results**
90 Table 2 shows all the experiments used in the following discussion, with the corresponding operating
91 conditions and main results, in term of loss on ignition (LOI, [%]), or total weight loss.
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93
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95
96

Table 2: Table of experiments

Run	Sample	HR [°C/min]	W_0 [g]	LOI [%]	Notes
#1	SS-A1	0.1	19.80	46.7	
#2	SS-A2	0.2	19.70	78.9	
#2Bis	SS-A3	0.2	20.80	76.6	Reproducibility
#3	SS-A4	0.2	20.58	41.0	Pellet
#4	SS-A5	2.0	18.50	81.5	
#5	SS-B1	0.1	24.00	75.5	
#6	SS-B2	0.2	24.30	75.7	
#6Bis	SS-B3	0.2	24.00	73.8	Reproducibility
#7	SS-B4	0.2	20.63	73.3	Fine fraction
#8	SS-B5	0.2	20.59	20.6	Pellet
#9	SS-B9	2.0	23.67	75.3	
#10	SS-C1	0.2	21.56	34.8	
#11	SS-C2	0.2	22.28	45.0	Milled
#12	SS-C3	2.0	20.80	64.8	
#13	SS-C4	6.0	25.95	54.2	

98

99 3.1 The ignition pattern

100 The slow heating of a large sample produces a typical pattern, summarized in Figure 2 for SS-B, which shows

101 the most interesting behavior. The figure also proves the reproducibility of the test, following our procedure.
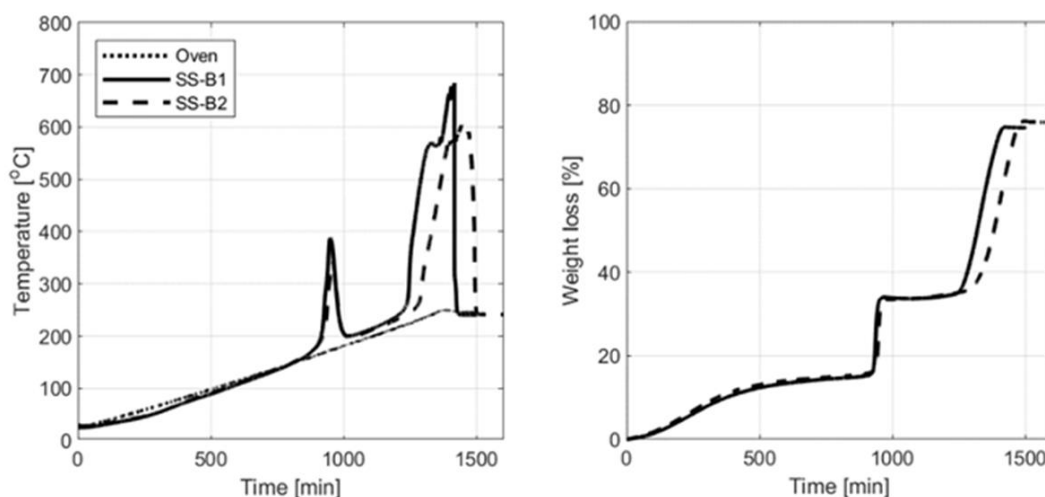

102

103 **Figure 2:** Ignition steps, and reproducibility analysis, in SS-B, at HR=0.2 °C

104 From Figure 2 we see that the sample temperature lags below the oven temperature during drying, because of

105 the endothermic evaporation: a slow heating effectively extract all the moisture even at low temperature.

106 Following desiccation, we observe a clear, 2-steps ignition in SS-B, at two temperatures. Approx. 20% of the

107 initial weight is lost in the first ignition, which is unable to self-support. A further increase in temperature

108 produces a second ignition where an additional 40% of weight is lost, up to the inorganic residues identified

109 by the proximate analysis (see Table 1). In term of oven temperature we see that the onset of reactivity is

110 sharply located at about 180°C for the first step and 230°C for the second. The difference in temperature

111 between sample (approximatively its center) and the oven (thus the boundary of the sample) is the driving force

112 for heat dissipation, which leads to extinction. Accordingly, the sample size is crucial to sustain the exothermic

process. That is well known [20] and motivates the investigation where: i) samples are large enough to clearly observe the exothermic process; ii) the temperature surrounding the sample is kept sufficiently high to limit the dissipation of heat from the sample.

3.2 Effect of the sludge origin

The three waste water treatment sludges considered come from different WWT plants. The gravimetric and thermal evolution according to our testing procedure, at 0.2ºC/min, is shown in Figure 3. A very different reactivity has been identified by this procedure among supposedly similar materials. The SS-A ignited quite gradually, starting from approx. 170°C, through a single, auto-accelerating ignition event that reached 570ºC. The SS-A lost 78% of the initial mass, consistently with its ashes content. The SS-B reacts in 2 steps: the first ignition is very vigorous, at 170ºC, peaking close to 400ºC and then extinguishes, causing a 20% weight loss. The second ignition, at 230ºC, was much more exothermic, reaching almost 700ºC, and completely depleting the remaining 46% of organics, up to a total loss of 74% of the initial mass. The first ignition occurs a very low temperature. It does not survive at this sample scale, but at industrial scale it can continue seamlessly to the second step. This extreme reactivity of SS-B is consistent with the Díaz et al. [2] classification in the 'very high risk' zone, rather than in the 'high risk' one, like SS-A and SS-C. Finally, the SS-C did not show any clear ignition. Still, it slowly reacts from 170 to 250°C, losing 35% of its initial mass, quite far from the total consumption of the organics content.
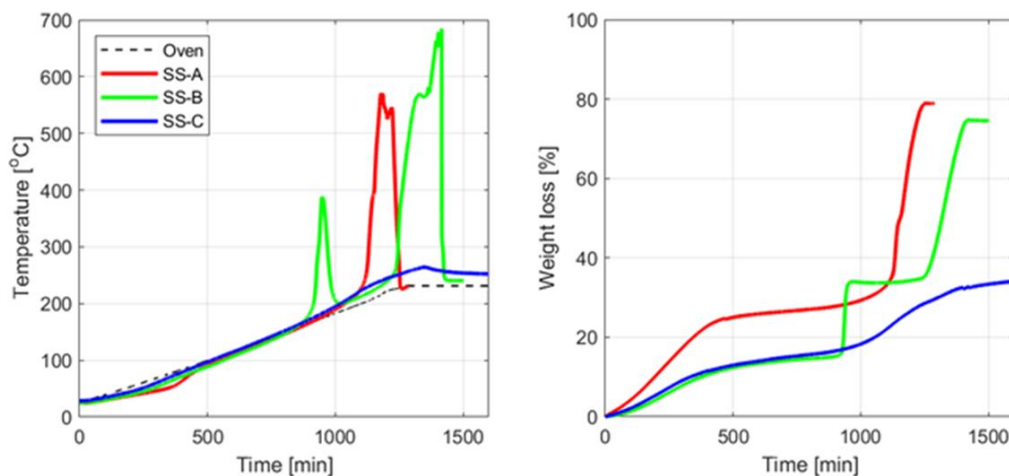
Figure 3: *Onset and progress of reactivity in sludges A, B, C, at HR=0.2 °C/min*

It is worth comparing the same materials in the same testing apparatus at a higher heating rate, of 2ºC/min. Surprisingly, the results change significantly, as shown in Figure 4. At higher HR the three sludges show a similar behavior.
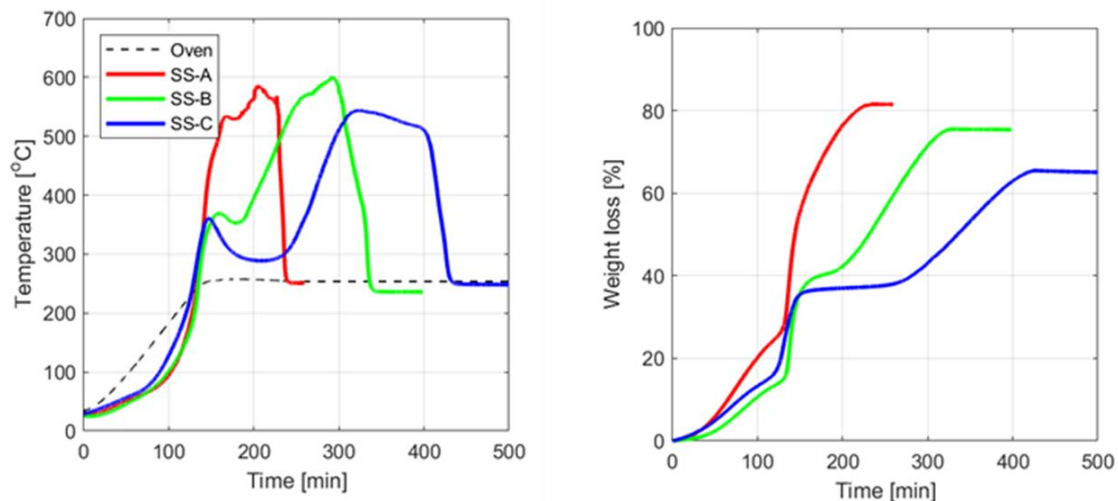
135
136 ***Figure 4:** Onset and progress of reactivity in sludges A, B, C, at HR=2 °C/min*

137 They all ignite, apparently well above 200°C, including SS-C, burning all the organic available. A 2-steps
138 process for SS-C is well evident, as well as for SS-B, better isolated at 0.2 °C/min. All the sludges reach a
139 similar maximum temperature, in the 550-600°C range, the highest for SS-B and the lowest for SS-C. Still,
140 SS-B reaches approx. 100°C less than the lower heating rate (0.1 °C/min). The total time required to burn all
141 the organics (220, 320, 420 min for A, B, C) varies markedly, suggesting a reactivity ranking as SS-A > SS-B
142 > SS-C. We recall that the ranking at 0.2 °C/min appeared SS-B > SS-A > SS-C, while Diaz et al.'s criterion
143 suggests SS-B > SS-A ≈ SS-C. The reactivity also appears proportional to the initial availability of organics.
144 Interestingly, the most reactive sludge, SS-A, has also the highest moisture content, although moisture left the
145 sample when the oxidation reaction start. Still, it could play a role in low-temperature reforming reactions that
146 produce partially oxidized gas species, such as CO and H_2, acting as supporting fuels in the following
147 combustion. However, we believe that the chemical arguments above are questionable, due to the low
148 temperature, unlikely to activate uncatalyzed reactions. On the contrary, a key property distinguishes SS-A,
149 which is its density, the lowest of three samples. A higher bed permeability is a determining factor to support
150 a higher rate of reaction, allowing a higher flux of oxygen inside the bed, as also demonstrated by the Reactivity
151 Index (RI) in Table 1.

152 **3.3 Effect of the different Heating Rate**
153 For this purpose, each sample was tested at different HRs. Results are shown in Figure 5 to Figure 7. Note that
154 HR between 0.1 and 6 ºC/min have been considered. Accordingly, the time scale of the experiments varies
155 widely, between hours and days. We point out once more that the faster heating rate (6°/min) is still quite low
156 compared to the common values (10 to 20°/min) used in standard TGA analysis.
157 The results for Sludge A, Figure 5, shows that a larger HR produces a sharper ignition, both in terms of rate of
158 temperature rise and rate of weight loss (i.e. the rate of reaction). An extremely low heating rate (0.1°/min)
159 may even cause a significant sludge degradation (almost 50%) without a noticeable temperature rise, above the
160 oven temperature.
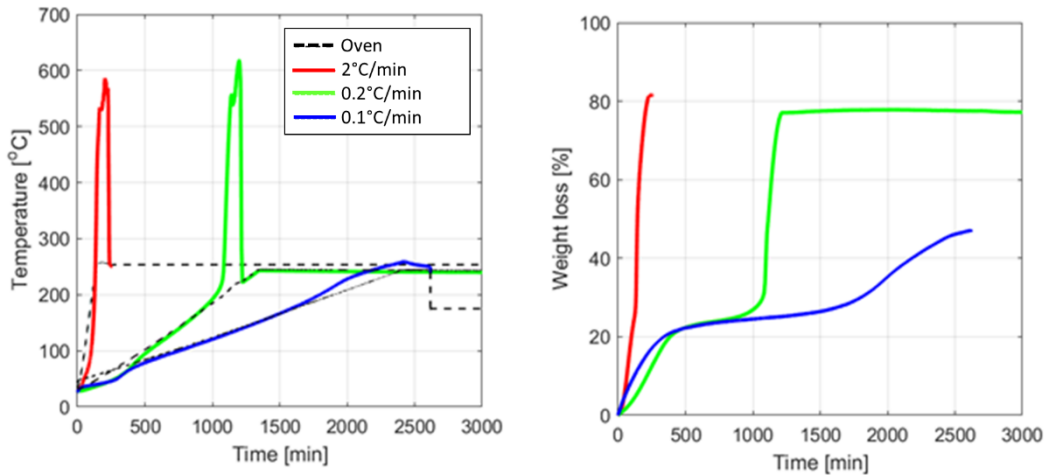
Figure 5: Onset and progress of reactivity in SS-A at 0.1, 0.2, and 2°C/min

That takes almost 2 days to occur. In this case, the rate of heat dispersion from the material to the environment matches the generation of heat, preventing a hot spot to develop into a vigorous ignition. That has already been reported [21], similarly suggesting that heat dispersion prevents self-ignition. We would rather conclude that explosive evolution is prevented by controlling the heating rate, while smouldering takes place also at very low heating rate, with a rate of progress controlled by the heat dispersion across the sample. Once more, we conclude that the degradation progress is ruled by the heat and mass transfer rates, rather than the intrinsic, chemical reactivity of the solid, once the latter is significant enough (and that is quite common in air).

In the case of sludge B, Figure 6, the highest (2°/min) heating rate somehow hides the two-steps ignition. The first ignition is still noticeable, but the sample has no time to extinguish the first process that the second one begins. The final degradation degree is perfectly reproducible, but the degradation history appears very different in terms of sample or oven temperature. While it is very reproducible when analyzed in terms of temperature of the sample, it can be very misleading if the environment temperature is considered. This double-step ignition has been reported by others, as [13], [22] and [23].
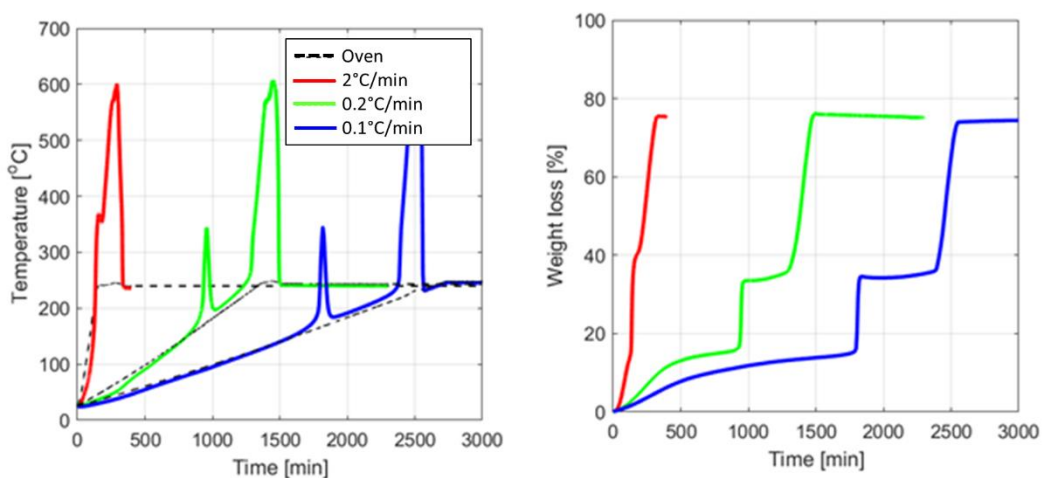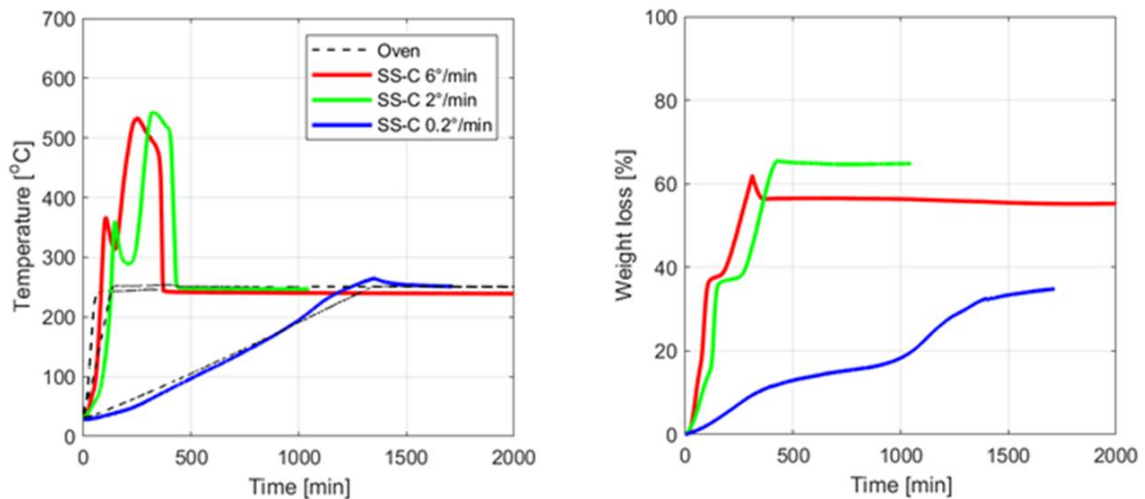

Figure 6: Onset and progress of reactivity in SS-B at 0.1, 0.2 and 2°C/min

7

178 In the case of sludge C, Figure 7, we applied the largest HRs, up to 6°/min, because of its apparent difficulty

179 of igniting. Indeed, SS-C did not ignite at 0.2°C/min (see Figure 3) like the other sludges (Figure 5 and Figure

180 6). The behavior of SS-C at the highest HR appears similar to SS-A, with an almost single ignition step. It

181 suggests that the rate of heat dissipation across the sample is larger in SS-C, perhaps because of a larger particle

182 size. That is consistent with the observation that the minimum HR required to observe the onset of a self-

183 sustained degradation, with distinguished local overheating compared with the environment, follows the

184 sequence of the average particle size:

185   SS-B: D_{mean}= 0.462 mm HR_{min} = 0.1°C/min

186   SS-A: D_{mean}= 0.986 mm HR_{min} = 0.2°C/min

187   SS-C: D_{mean}= 1.699 mm HR_{min} = 2°C/min

188 It is concluded that a self-sustained degradation in a sample of significant size is more affected by the physical

189 properties of the solids and its assembly, that rule the heat and mass transfer rate (such as the particle size),

190 than the chemical composition of the material ([2], [3], [23]).

191

192
193 *Figure 7: Onset and progress of reactivity in SS-C at 0.2, 2 and 6°C/min*

194 **3.4 Effect of the particle size**

195 The particle size distribution of the materials, as received and after milling or pelletization, is shown in

196 Figure *8*. It has been obtained by sieving. It is observed that sludges differ markedly by PSD. The average

197 diameter (see Table 3) suggests an order SS-B<SS-A<<SS-C.

198   *Table 3: Mean Diameter, Lenght and Specific Area of raw and modified sewage sludges*

Parameters	Units	SS-A	SS-B	SS-C	B Fine	C Milled	Pellet A	Pellet B
D_{Mean}	mm	0.986	0.462	1.699	0.283	0.285	5.000	5.800
L_{Mean}	mm	-	-	-	-	-	11.870	14.890
$A_{Specific}$	mm^{-1}	6.085	12.987	3.531	21.201	21.053	0.968	0.824

199

However, the shape of the PSD is also different. The SS-C is not only coarser, but also narrowly distributed; there are no fines, i.e. particles smaller than 675 μm, not even particles larger than 1.9 mm. Fines, quite abundant in SS-A and SS-B, dramatically affect the bed porosity. In SS-B, 98% are particles with a diameter < 1 mm, better described as powder than granules. However, SS-A and SS-C look like granular material because of a significant fraction at larger size, approaching 2 mm. Following the previous observation and speculations on the role of particle size in supporting (or preventing) the sludge self-ignition, we investigated variations on the original particle size distributions (PSD) of SS-A, SS-B and SS-C.

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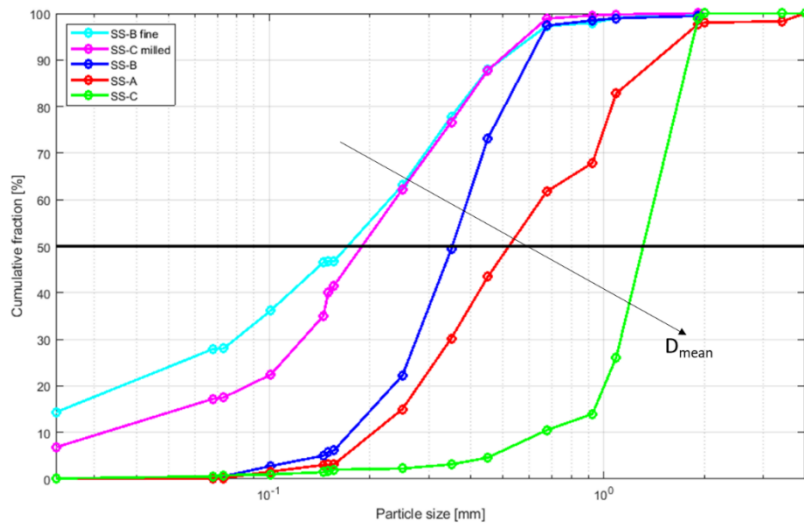

Figure 8: PSD of raw sludges A, B, C and PSD of B fine fraction and C milled

We speculated that the difficulty of SS-C in developing a self-sustained oxidation is due to its coarse and narrowly dispersed size, supporting a convective heat dissipation through the bed. To prove such hypothesis, we milled SS-C. Its smouldering is shown in Figure 9, and compared to SS-A and SS-B as received.
The results confirm the hypothesis: a much finer SS-C shows a 2-steps ignition, quite similar to SS-B. Milled SS-C would now be classified as more reactive than SS-A, according to the results of Figure 9.
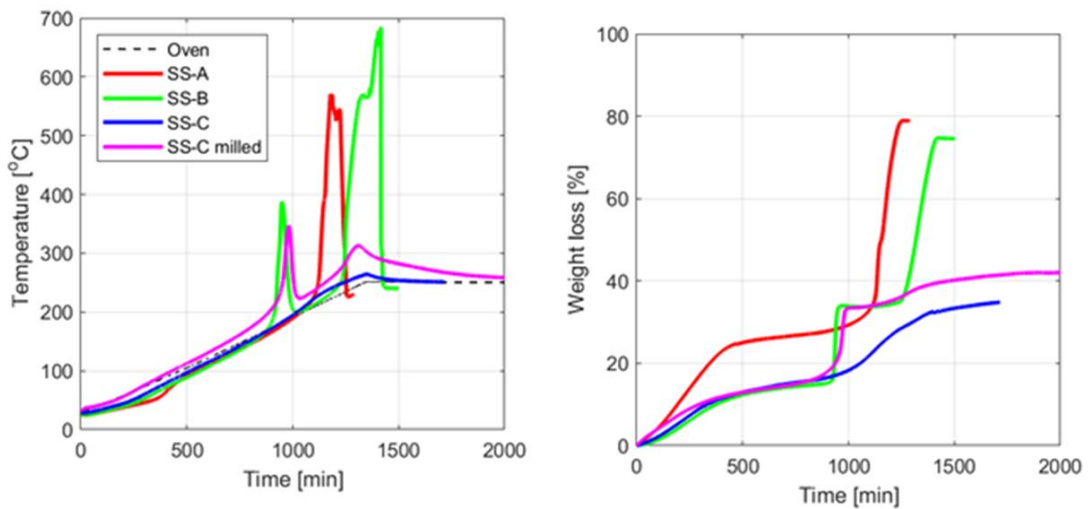
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Figure 9: Onset and progress of reactivity in SS-A, SS-B, SS-C and milled SS-C, at 0.2 °C/min

216 Despite the chemical nature of SS-C being the same, a different PSD causes a considerable difference of
217 apparent reactivity. It means that the reactivity due to the chemical composition is the same, but the
218 environment at the reaction locus is different, and specifically the local temperature and oxygen availability.
219 A finer material reduces the heat and mass- transfer rate through the bed. The first limitation allows a higher
220 local temperature to develop, pushing on the rate of reaction. The second limitation (reduction of permeability
221 to oxygen) is a penalty for the reaction. In addition, the smouldering is a heterogeneous, gas-solid reaction; as
222 such, the greater the surface of solids exposed to the air, the greater the rate of reaction per unit volume.
223 According to this, grinding is expected to increment the hazard of waste storages [24].
224 We further checked such speculations comparing the behavior of SS-B collected in different parts of the drying
225 plant, the regular output and the filter-bags cleaning the exhaust gases. Once again, the chemical nature is the
226 same, but the SS-B from filters is much finer. The comparison is reported in Figure 10.
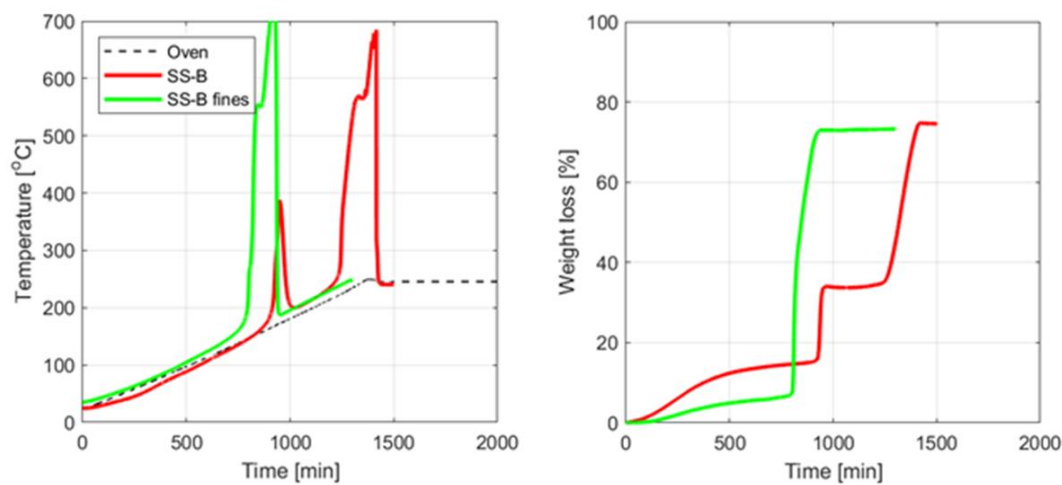
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228 ***Figure 10: Onset and progress of reactivity in SS-B and fines of SS-B, at 0.2 °C/min***

229 The reactivity of finer SS-B is higher; the lack of coarser particles increased the heat insulation of the bed,
230 allowing the smouldering front to propagate since the first ignition, up to the total consumption of the organics.
231 On the other hand, pelletization is a process that increases the particles size, while producing much denser
232 solids. We evaluated the effect on both SS-A and SS-B. Results are shown in Figure 11.
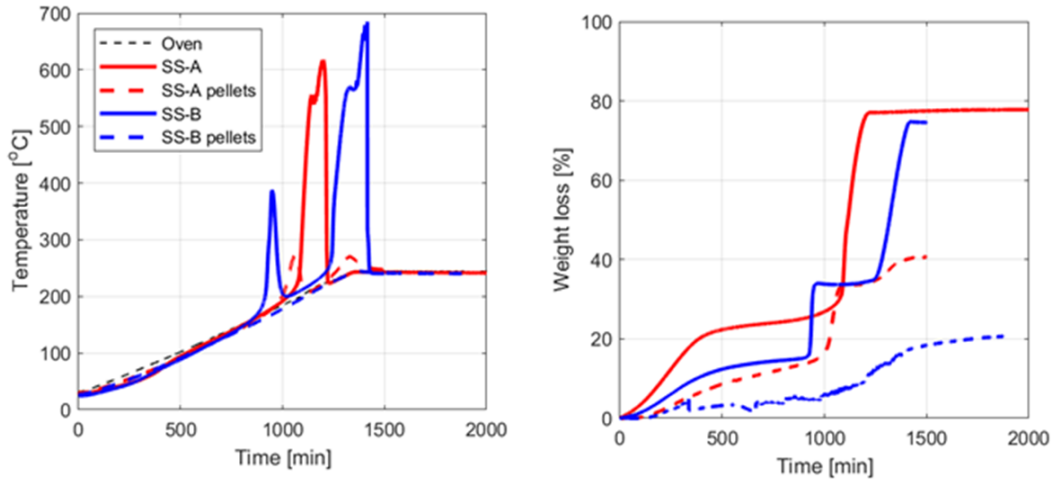
Figure 11: *Onset and progress of reactivity in SS-A and SS-B before and after pelletization, at 0.2 °C/min*

The pelletization of sludge significantly limited the reactivity onset and progress. The local temperature rise above the oven temperature is unperceivable in pelletized SS-B and quite small in SS-A, now showing a 2-steps ignition. The final loss of mass was much lower than for granular samples, suggesting that a large fraction of organic matter did not react. We explain the results in terms of a dramatic increase of intraparticle diffusive limitations, making harder the supply of oxygen to the core of the solids. At the same time, pellets produce coarser bed, where the interparticle heat transfer is facilitated, also by convection, reducing the bed thermal insulation effects. We believe that the difference between pellet of SS-A and SS-B are only partially due to their different chemical nature; more likely, since the self-heating phenomenon is superficial in nature, small differences in the thermal behavior are due to the different value of pellet specific area (A/V) (0.968 mm^{-1} for pellet A and 0.824 mm^{-1} for pellet B), as shown in Table 3. At any rate, results on pellets confirm that compaction is an effective mean to mitigate the sludge intrinsic reactivity ([24], [25]).

4. Conclusions

The study shows that the self-heating processes often observed with oxidable solids in air, such as sewage sludge, at industrially significant scale is controlled by physical rather than chemical solids properties. The same solid can undergo quite different oxidation process, with large overheating and fast progress or very slow degradation and barely detectable overheating, by simply changing its physical or environmental conditions. A determining physical properties of the solids is its particle size distribution that affects bed compaction and specific surface area. Environment influences the course of degradation through the rate heat of supply (or removal). Accordingly, the exothermic degradation of biomass (and any oxidable granular material), characterized by intrinsic non-homogeneity, requires investigation at a representative scale, where physical processes play a role. The set-up presented here proved extremely useful to confirm the above conclusions; that also suggests better investigating the role of physical process, whereas difference in chemical reactivity appears of minor concern. Finally, the study suggests that an effective hazard reduction practice in storage of such materials is increasing the particles size; that prevents heat accumulation in the bed. Pelletization is an additional measure to lower the hazard of self-heating, because it also reduces the intraparticle oxygen permeability.

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