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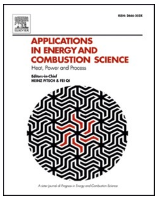

Thermal pre-treatment of waste municipal sewage sludge for thermochemical valorizations: An experimental study on moisture reduction and sludge modifications

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ABSTRACT

SS valorization by thermochemical conversions (e.g., gasification) for power, heat or fuels production has an interesting development outlook. The literature suggests that SS moisture must be lowered below 20 wt% for conventional thermochemical conversions such as gasification. This work focuses on the experimental investigation of the effects of a thermal pre-treatment of SS with air, addressed to adjusting SS moisture. Semi-batch tests were performed on SS at a laboratory scale (40 g of SS, 2 NL/min air, 2 h, 75–250 °C). The temperature of thermal pre-treatment significantly influenced the elemental composition of solid residue, its moisture content, and the properties of liquid condensate, characterized by unprecedented analyses. Temperatures of 120 °C or higher ensured adequate moisture removal; however, a significant depletion of SS dry solids (C, H, N, S) was quantified above 200 °C. Condensed liquids were beyond Italian legislative limits for direct discharge into the environment. Finally, estimates of the heat contributions to the pre-treatment were performed. The collected results are a first step between fundamental understanding and process design, suggesting that the investigated pre-treatment may fit seamlessly into the schemes of traditional and innovative thermochemical processing plants.

1. Introduction

Sewage Sludge (SS) is the main byproduct of wastewater treatment [1,2]. SS contains water, organic molecules (some toxic [3]), microbial nutrients, pathogen microorganisms [4], heavy metals (zinc, copper, nickel, cadmium, lead, mercury and chromium) [2,5]. Estimation of global SS production in 2022 amounted to ~53 million tons of dry solids per year (potentially rising to ~160 million tons if global wastewater would have been treated as in the 27 EU countries/UK) [6]. This production is expected to increase due to growing population, industrialization, and urbanization [7].

SS circular management shall contribute to the sustainability of Countries and communities: e.g., SS is a potential renewable fuel for thermochemical conversions, such as combustion or gasification, to obtain energy as sensible heat in flue gases or heating value of syngas.

The most usual methods to manage SS are landfilling after possible incineration (without energy recovery), landfilling, and improper dumping. Nowadays, more sustainable techniques have been introduced: biological stabilization (anaerobic digestion, composting); land spreading for agricultural utilization as fertilizer; landscaping; conversion into secondary added-value products; thermochemical conversions (e.g., combustion or gasification) to produce power, heat or fuels [2,8–10].

SS can be an asset in circular economies, as a solid renewable fuel for thermochemical conversions. In addition to energy and fuel production, thermochemical conversions generally introduces advantages such as reduction of SS disposable volumes and control of SS-related emissions [11]. From an overall processing point of view, these advantages compensate for the fact that SS has lower heating values (7–15 MJ/kg, Table 1) than those of other conventional biomasses (12–19 MJ/kg [12]).

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Acronyms and Symbols

Acronyms

BOD_5	Biochemical Oxygen Demand at 5 days
COD	Chemical Oxygen Demand
EU	European Union
HHV	Higher Heating Value
HPLC	High-Performance Liquid Chromatography
LHV	Lower Heating Value
SS	Sewage Sludge
TC	Total Carbon
TIC	Total Inorganic Carbon
TOC	Total Organic Carbon

Symbols

$c_{p,k}$	specific heat capacity of k
F_i	mass flow rate of i for energy estimations
m_i	mass of i
M_i	moisture percentage of SS in state i
Q_k	energy contribution of k in the thermal pre-treatment of SS
q_k	specific heat duty of k
r_i	percentage mass shares of i after SS thermal pre-treatment
T	temperature of experimental test of thermal pre-treatments
$X_{j,\ i}$	mass percentage of element j in SS in state i, on dry basis
ΔH_j	heat of decomposition of element j from dry-ash-free fraction of SS
$\Delta j(T)$	variation of element j in SS dry fraction due to thermal pre-treatment at T
$\Delta M(T)$	mass percentage of moisture removed by thermal pre-treatment of SS at T
ΔM_{st}	mass percentage of moisture removed by sterilization
λ	latent heat of water evaporation

Subscripts

0	initial SS in experimental tests of thermal pre-treatments
air	air flow used in thermal pre-treatment of SS
ar	as-received SS
c	condensate
daf	dry-ash-free fraction of SS
dec	decomposition of C, H, N, S of dry-ash-free fraction of SS
dry	dry solids fraction of SS
ext	external
g	gas
$H_2O_{(l)}$	liquid water
$H_2O_{(v)}$	steam
i	generic state of SS or its products after thermal pre-treatments
j	generic element of dry-ash-free fraction of SS, i.e., solid C, H, N, S or O
k	generic energy contributions in thermal pre-treatments of SS
M	moisture of SS
res	solid residue of SS after experimental tests of thermal pre-treatments
st	sterilized SS
tot	total

Thermochemical conversions can give a significant contribution to the Italian situation with SS. In 2023, Italy produced 3.2 million tons of SS from municipal wastewaters: 47.6 % was managed by disposal, 51.3 % by recovery [13]. Italy is currently under four infringement procedures charged by EU *"for failure or inadequate compliance with the directive on urban wastewater treatment"*, concerning the covering of a potential additional load of 27 million equivalent people [14]. Moreover, the demand for depurated water could grow as unconventional supply to cope with occasional droughts [15]. Currently, SS is commonly managed in Italy by biological stabilization [16] followed by dispersion of SS in agricultural land [17]. However, some concerns recently emerged about contaminations associated to SS management, which can introduce persistent organic pollutants, endocrine disruptors, drugs, and heavy metals into the soil [17]. Many European countries do not allow or strongly limit agricultural uses of SS [18,19]. Furthermore, the tightening of legal thresholds for SS pollutants can increase the disposal costs, as occurred in Lombardy region in the period 2017–2019 [19]. Only 1.9 % of the Italian SS production of 2022 was used for energetic valorization [20] . For all these reasons, Italy should strengthen those SS recovery routes different from dispersion in lands, such as SS thermochemical conversions.

The typical moisture content of SS – as delivered by wastewater treatment plants after dewatering [19] – amounts to approximately 80 wt % [21], which constitutes a significant technical issue with thermochemical conversions: the vaporization latent heat of moisture is an extensive endothermic load which decreases the net energy provided to

Table 1
Properties of sewage sludge (SS) investigated for thermochemical conversions in literature.

Moisture [wt %]	Proximate analysis [wt % dry basis]			Ultimate analysis [wt % dry basis]					LHV [MJ/kg dry basis]	HHV [MJ/kg dry basis]	Reference
	Volatile matter	Fixed carbon	Ash	C	H	N	S	O			
n.d.	n.d.	n.d.	57**	16.9	3.3	2.4	0,7	19.8	7–10	n.d.	[23]
n.d.	n.d.	n.d.	39.5**	30	4.4	4.2	1.1	20.8	11–13	n.d.	[23]
11.75	63.60	13.4	23.51	39.48	6.19	3.93	1.45	25.46	n.d.	17.14	[24]
10.0	60.33	5.67	34	32.47	5.61	4.00	0.78	23.13	10.6	n.d.	[25]
10.0	67.67	5.33	27	40.03	6.12	5.18	1.06	20.61	14.8	n.d.	[25]
5.3	53.85	n.d.	38.54	31.79	4.36	4.88	1.67	20.57	12.96	14.05	[26]
5.3	51.74	n.d.	49.31	24.72	3.81	3.59	1.81	18.84	10.75	11.71	[27]
7.3	n.d.	54.58	42.1	29.27	3.83	4.12	0.70	20.00	10	n.d.	[28]
3.53	66.59	2.67	30.73	41.2	5.22	3.21	n.d.	20.72	n.d.	14.1	[29]
6.4–7.5	n.d.	n.d.	41.3–46.7	25.9–28.7	4.8	3.7–4.2	0.9	17.5–23.4	n.d.	n.d.	[30]
7.9	55.1	7.1	37.9	36.2	4.5	5.6	1.1	14.7	n.d.	15.4	[31]
20	55.00	6.38	38.63	31.42	5.03	4.36	1.04	19.52	12	15	[32]
1.74	45.08	21.8	33.91	45.79	2.99	1.49	1.11	14.7	n.d.	16.34	[33]
84*	61.63*	9.41 *	28.96 *	38.18 *	3.40 *	4.67 *	1.05 *	23.74 *	n.d	14.63 *	[34]
80.58 *	50.97 *	7.05 *	41.98*	30.13 *	5.70 *	2.53 *	1.40 *	14.56*	n.d	14.56 *	[35]

*as obtained from wastewater treatment; **this percentage is referred to dehydrated sludge; n.d. = not determined.

the end user [22]. Table 1 gathers main properties of several SS investigated in the literature within the context of thermochemical conversions: the moisture of fed SS is usually lower than 20 % in weight, a value that can be assumed as a reasonable upper limit.

Thermal pre-treatments are common unit operations to regulate the SS moisture content [29]; moreover, they reduce lousy smell, lower SS volumes, destroy pathogen microorganisms, avoid SS stickiness, and form hard SS granules [29]. With regard to thermochemical conversions of SS, the thermal pre-treatments for moisture removal are generally grouped as [8]: (i) partial drying (dry solids <85 %) or full drying (dry solids >85 %), according to resulting moisture fraction; (ii) contact drying, convection drying, radiation drying, or mixed forms, according to the operated heat transfer.

The convection thermal pre-treatment exploits a gaseous medium (e.g., flue gas, steam, or other hot gases) that flows in direct contact with SS, for heat and mass transfer [8]. It is a mature configuration that integrates easily into traditional thermochemical conversion plants, by realizing heat recoveries from available process gaseous flows [36]. The vapors from SS are conveyed with the drying gas [8], so the load of pollutants released by SS can become a potential issue regarding gaseous effluents, requiring efficient flue gas treatment [37].

Recent literature highlighted that, although energy-demanding, drying is indispensable to enable downstream thermochemical processes [38]. Zhu et al. [39] examined the influence of temperature and sample geometry, reporting that spherical samples exhibited the highest drying efficiency at 200 °C. Sirok et al. [40] highlighted the role of porosity and layer structure in convective drying, showing how these factors affected airflow resistance and drying kinetics. Xu et al. [41] explored thermal drying across five temperature intervals (90–210 °C), demonstrating that calcium oxide addition accelerated moisture removal, with optimal performance at 15 % additive content. Dai et al. [42] reported large-scale sludge drying with flue gas (100–200 °C), achieving 86 ton/day throughput at 160 °C, while preserving 95 % of sludge calorific value and lowering flue gas emissions. Furthermore, reviews on gaseous emissions from thermal drying underline the environmental relevance of NH_3, H_2S, and VOCs release, aimed at optimizing emission control strategies and overall energy efficiency [37].

This work investigates the convection thermal pre-treatment of a real dewatered waste SS (from a municipal wastewater treatment plant in Central Italy). Semi-batch laboratory-scale tests were performed under air flow at different temperatures between 75–250 °C. An unprecedented focus – by means of dedicated physical-chemical characterizations – was performed on the effects of pre-treatment temperature on composition of SS solid residuals and condensates. Beyond the expected moisture removal, temperature-dependent modifications within the solids were measured, pointing to the onset of early thermochemical transformation. By correlating operating temperature with residual solid and condensate compositions, this study provides novel insights into the phase-specific behavior of organics, inorganics, and volatile compounds. The results will inform future industrial development of SS thermochemical conversion, supporting optimization of pre-treatment strategies in terms of quality of biomass and energy.

2. Materials and methods

2.1. SS feedstock

An actual wastewater treatment plant in Central Italy (270,000 equivalent people) supplied a bulk of SS (“as-received SS”, SS_{ar}), after a treatment by anaerobic digestion, post-thickening, and dehydration.

Microbial activity can significantly change the SS composition in the order of days [43]. Some residual activity was noted in the bulk of SS_{ar}, indeed. Therefore, the SS_{ar} bulk was sterilized all at once in an oven at 121 °C for 30 min, then frozen at −24 °C [25] (“sterilized SS”, SS_{st}). This avoided inhomogeneity of SS samples during the experimental campaign.

The percentage mass variation due to sterilization was ascribed only to moisture loss (ΔM_{st}) and calculated by Eq. (1), measuring the initial sample mass of SS_{ar} (m_{ar}) and the final sample mass of obtained SS_{st} (m_{st}).

$$\Delta M_{st} = \frac{m_{ar} - m_{st}}{m_{ar}} 100 \tag{1}$$

2.2. Thermal pre-treatment tests and measurements

Experimental tests of convective thermal pre-treatment were performed by a Parr-pressure reactor MODEL 4563 M. An initial mass of SS_{st} (about 40 g, m_0) was loaded into the reactor and 2 NL/min of air (5.0 grade) were fed continuously to ensure a sufficiently high space velocity. Each test lasted 2 h at the chosen set-point temperature (75 °C, 120 °C, 150 °C, 200 °C, or 250 °C). Downstream, the outlet gas flow passed through a double-pipe glass condenser, cooled by ethylenic glycol at 4 °C to separate condensable species. In the end, the cooled gas was vented. At the end of each test, a solid phase remained in the reactor vessel (“SS solid residue”, SS_{res}), and a liquid phase was accumulated downstream by the condenser (“condensate”). Three test replications were performed per each set-point temperature. Further details on the test rig and procedure are in Supplementary Material, Section A.2.

The final masses of SS_{res} (m_{res}) and condensate (m_c) were measured to calculate the respective percentages r_{res} (Eq. (2)) and r_c (Eq. (3)), referred to m_0. The missing part of m_0 was attributed to vented gases and possible losses (r_g, Eq. (4)).

$$r_{res} = \frac{m_{res}}{m_0} 100 \tag{2}$$

$$r_c = \frac{m_c}{m_0} 100 \tag{3}$$

$$r_g = 100 - r_{res} - r_c = \frac{m_0 - m_{res} - m_c}{m_0} 100 \tag{4}$$

2.3. Characterizations

2.3.1. Characterizations of solid phases

The moisture content of SS_{st} and SS_{res} was measured according to the standard BS EN ISO 18,134:2022 [44]: the initial solid sample was dried at 105 ± 2 °C under stagnant air in an oven (Heraeus VT6130), until solid mass was constant (deviation ≤ 0.2 %). Five replications of the procedure were carried out and moisture content was calculated by Eq. (5) (M_i = moisture content, m_i= initial mass of i, m_{dry} = final mass, $i = st$ for SS_{st}, res for SS_{res}). The moisture content of SS_{ar} (M_{ar}) was estimated indirectly, by Eq. (6). The moisture removal due to thermal pre-treatment at temperature T ($\Delta M(T)$) was calculated by Eq. (7).

$$M_i = \frac{m_i - m_{dry}}{m_i} 100 \quad \text{with } i = st, res \tag{5}$$

$$M_{ar} = \Delta M_{st} + M_{st} \left(\frac{100 - \Delta M_{st}}{100} \right) \tag{6}$$

$$\Delta M(T) = \frac{m_0 (M_{st}/100) - m_{res} (M_{res}(T)/100)}{m_0 (M_{st}/100)} \tag{7}$$

The ash content of SS_{ar} and SS_{st} was determined on completely dried SS samples according to standard BS ISO 1171:2010, by incineration at 825 °C for 120 min in a muffle furnace under stagnant air. Three replications were performed. Ash content measured on dry basis was then rescaled on SS_{ar} and SS_{st} by means of moisture percentages M_i with $i = ar, st$, respectively.

The proximate analysis of SS_{st} and SS_{res} (volatile matter, fixed carbon, ash content) was carried out according to ASTM D5142/02 [45], by

HITACHI Thermal Analysis System STA200RV; the related Thermogravimetric (TG) and Derivative TG (DTG) analyses were performed by the following temperature program: heating at 10 °C/min up to 105 °C and dwell at 105 °C for 15 min to evaporate moisture, heating up to 815 °C at 10 °C/min and dwell at 815 °C for 7 min to devolatilize volatile matter, cooling down to 600 °C at 20 °C/ min, all under nitrogen flow; heating at 10 °C/min from 600 °C up to 700 °C and dwell at 700 °C for 15 min, heating at 10 °C/min up to 815 °C and dwell at 815 °C for 15 min, all under air flow to combust fixed carbon. Based on ASTM D5142/02, contents of volatile matter was obtained from the weight loss during nitrogen heating, ash from the final residual mass after combustion, and fixed carbon was determined as the remaining mass after accounting for moisture, volatile matter, and ash.

Ultimate analysis was performed on previously dried samples of SS_{st} and SS_{res}, using a PerkinElmer Series II CHNS/O analyzer, obtaining elemental mass percentages on dry basis (wt $\%_{dry}$) of carbon (C), hydrogen (H), nitrogen (N), sulfur (S), and oxygen (O, by difference), named $X_{j,\ i}$ with $j =$ C, H, N, S, O, and $i = st, res$.

The elemental variations in dry matter of SS, due to thermal pre-treatment at temperature T, were named $\Delta j(T)$ (with $j =$ C, H, N, S, O) and calculated by elemental balances in Eq. (8).

$$\Delta j(T) = \frac{m_0\left(\frac{100 - M_{st}}{100}\right)X_{j,\ st} - m_{res}\left(\frac{100 - M_{res}}{100}\right)X_{j,\ res}(T)}{m_0\left(\frac{100 - M_{st}}{100}\right)X_{j,\ st}} \quad \text{with } j = \text{C, H, N, S, O} \tag{8}$$

The Lower Heating Values (LHV) of SS samples were calculated using $X_{j,\ i}$ according to the Dulong equation [46].

2.3.2. *Characterizations of condensate*

The condensate was characterized in terms of: pH at room temperature (by HANNA instruments-USA); solution conductivity at 25 °C (AMEL INSTRUMENTS 160); organic acid content (identification and quantification of formic acid, lactic acid, acetic acid, propionic acid, and levulinic acid, High-Performance Liquid Chromatography-HPLC Alliance e2695 Waters USA); carbon content (tests LCK381 for Total Carbon-TC, Total Inorganic Carbon-TIC, Total Organic Carbon-TOC, spectrophotometer DR 6000 UV/VIS–HATCH); oxidable matter (tests LCK555 for Biochemical Oxygen Demand after 5 days-BOD_5 and LCK114 Chemical Oxygen Demand- COD); and nitrogen content (APAT 4030 method, 20 nm and 543 nm in Lambda 2 UV/VIS spectrometer Perkin Elmer).

2.3.3. *Data elaboration*

Replicated measurements were graphically presented in terms of mean and confidence intervals (95 % confidence level, by Student's t distribution).

2.4. *Estimates of heat contributions in the thermal pre-treatment*

A preliminary estimate of heat duties involved in the convective thermal pre-treatment of SS was performed along the lines of Fermi estimates [47]: experimental tests were framed as pseudo-continuous processes, with 40 g of SS_{st} treated for 2 h (i.e., $F_0 = 20$ g/h) and 2 NL/min of air (F_{air}), both fed at 20 °C and 0.92 bar (experimental room temperature and pressure).

The following heat contributions were estimated: (i) heating of SS_{st} dry matter (Q_{dry}, Eq. (9)) and air (Q_{air}, Eq. (10)) from 20 °C to process temperature of the thermal pre-treatment (T); (ii) heating of moisture removed from SS_{st} ($\Delta M(T)$, Eq. (7)), its evaporation (97 °C at 0.92 bar [48]), and superheating of the resulting steam up to T (Q_M, Equation 11); (iii) heat of decomposition of dry matter removed from SS_{st} (Q_{dec}, Eq. (12)), arbitrarily assumed as the oxidation of $\Delta j(T)$ (Eq. (8), $j =$ C, H, N, S, O) into CO_2, NO_2, SO_3 and H_2O. The latent heat of water vaporization (λ, Eq. (11)) and the specific heat capacities at constant pressure ($c_{p,k}$,$k = dry, air,$ $H_2O_{(l)}$, $H_2O_{(v)}$ for dry matter of SS, air, liquid water and steam, respectively, Eq. (9)-Eq. (11)) were considered constant along with T. The elemental heats of decomposition (ΔH_j, $j =$ C, H, N, S, O, Eq. (12)) were equaled to standard formation enthalpies of CO_2, NO_2, SO_3 and half of H_2O one, respectively, and considered constant along with T. Section A.5 of Supplementary Material gathers the values assumed for λ, $c_{p,k}$, ΔH_j.

$$Q_{dry} = F_0\left(\frac{100 - M_{st}}{100}\right)c_{p,dry}(T - 20^{\circ}\text{C}) \tag{9}$$

$$Q_{air} = F_{air}c_{p,air}(T - 20^{\circ}\text{C}) \tag{10}$$

$$Q_M = F_0\Delta M(T)\left(c_{p,H_2O_{(l)}}(97^{\circ}\text{C} - 20^{\circ}\text{C}) + \lambda + c_{p,H_2O_{(v)}}(T - 97^{\circ}\text{C})\right) \tag{11}$$

$$Q_{dec} = F_0\left(\frac{100 - M_{st}}{100}\right)\sum_{j=\text{C,H,N,S}}\left(X_{j,\ st}\ \Delta j(T)\ \Delta H_j\right) \tag{12}$$

The overall specific heat duty of the pre-treatment (q_{tot}) and the specific heat provided externally (q_{ext}) were estimated by Eq. (13) and Eq. (14), respectively.

$$q_{tot} = \frac{Q_{dry} + Q_{air} + Q_M + Q_{dec}}{F_0} \tag{13}$$

$$q_{ext} = \frac{Q_{dry} + Q_{air} + Q_M}{F_0} \tag{14}$$

Due to the idealized approach inherent in the method of Fermi estimates, values calculated by Eq. (9)-Eq. (14) should be considered as indicative of orders-of-magnitude that may concern SS convective thermal pretreatment, providing preliminary insights into its energy requirements.

3. Experimental results

Compositions of solid phases were presented and discussed on raw/absolute basis (wt %) or dry basis (wt $\%_{dry}$). The following abbreviations were adopted for the different states or portions of SS: SS_{ar} is as-received SS; SS_{st} is sterilized SS; SS_{res} is the solid residue after experimental tests; SS_{dry} is the dry solid part of a SS sample; SS_{daf} is the dry-ash-free solid part of a SS sample.

3.1. *Composition of SS feedstock*

Results of proximate and ultimate analyses for SS_{ar} and SS_{st} were summarized in Fig. 1a,b and Table 2. TGA/DTG measurements for proximate analyses are in Supplementary material, Section B.

The moisture content was 80.5 wt % in SS_{ar} and 77.6 wt % in SS_{st} (BS ISO 1171:2010, Fig. 1b); the mass loss due to the sterilization (Δm_{st}, Eq. (1)) equaled 12.8 wt % of SS_{ar}. The ash content of SS_{st} was 28.2 wt $\%_{dry}$ when measured by BS ISO 1171:2010 (Fig. 1a) and 29.7 wt $\%_{dry}$ by ASTM D5142/02 (Table 2).

According to proximate analysis (Table 2), the organic fraction of SS_{st} included 86.49 wt $\%_{daf}$ of volatile matter and 13.51 wt $\%_{daf}$ of fixed carbon, suggesting that SS could be significantly prone for thermal modifications. Ultimate analysis (Figs. 1a and b) highlighted that C was the dominant element of SS_{dry} (35.95 wt $\%_{dry}$); N amounted to 4.33 wt $\%_{dry}$, a content higher than that of other biomasses [49] such as wood (about 0.1 wt $\%_{dry}$), bark (0 wt $\%_{dry}$), energy crops (0.3–1.2 wt $\%_{dry}$), processed biomass (about 1.5 wt $\%_{dry}$), agricultural (about 0–2 wt $\%_{dry}$), or aquatic biomass (about 1.4–4.5 wt $\%_{dry}$) [50].

All data in Table 2 and Fig. 1 are consistent with literature information in Table 1. This might ensure that observations in this work could be generalized to some extent, despite the influence of seasonality and location on SS properties [51,52].

	As received	Sterilized	Post test at 75°C	Post test at 120°C	Post test at 150°C	Post test at 200°C	Post test at 250°C
■ Oxygen	4.37	4.37	4.05	4.37	3.80	3.39	2.02
■ Sulfur	0.35	0.35	0.32	0.33	0.31	0.28	0.21
■ Nitrogen	0.84	0.84	0.84	0.84	0.84	0.84	0.84
■ Hydrogen	1.14	1.14	1.08	1.10	1.07	0.89	0.62
■ Carbon	7.01	7.01	6.67	6.96	6.65	6.29	5.40
■ Ash	5.78	5.78	5.62	5.58	5.41	5.40	5.28
■ Moisture	80.50	67.66	27.45	4.26	1.55	0.01	0.40

Fig. 1. Composition of SS_{ar} and SS_{st} on dry basis (a); Compositions of SS in different states (b); Mass balances with 100 kg of as-received SS as the reference (c).

Table 2
Proximate analysis of SS (ASTM D5142/02): Volatile Matter, Fixed carbon, Ashes content.

	Volatile Matter[wt %$_{dry}$]	Fixed Carbon [wt %$_{dry}$]	Ash [wt %$_{dry}$]
SS$_{st}$	60.8	9.5	29.7
SS$_{res}$ post-test at 75 °C	58.3	11.4	30.3
SS$_{res}$ post-test at 120 °C	59.0	12.1	29.0
SS$_{res}$ post-test at 150 °C	57.7	12.4	29.9
SS$_{res}$ post-test at 200 °C	54.6	13.9	31.6
SS$_{res}$ post-test at 250 °C	40.5	22.7	36.8

3.2. *Results of thermal pre-treatment tests*

Fig. 2a summarizes the main effects of the convective thermal pre-treatment on SS$_{st}$, in terms of mass shares of SS$_{res}$ (r_{res}, Eq. (2)), condensate (r_c, Eq. (3)), gases and losses (r_g, Eq. (4)).

The share r_c increased as the pre-treatment temperature was increased, more abruptly in the range 75–120 °C, much more slightly in the range 150–250 °C (Fig. 2a). This was mainly due to moisture that evaporated during the process and was condensed downstream. From a mechanistic point of view, the removal of water from SS is peculiar: Pilli et al. [53] highlight that the water in SS is held in ways that hinder its evaporation, such as intracellular water in SS bacteria, or water bound into extracellular polymers (that prevent desiccation of bacterial cells); Pilli et al. [53] report that the treatments at temperatures greater than 150 °C enhance the SS dewaterability, i.e., allow disengaging water from those hindered states; Boran Wo et al. [54] explain how higher temperature affects positively SS dewaterability primarily through its influence on viscosity, floc structure, and interaction between extracellular polymeric substances and water. It is sensible to presume that similar factors influenced the mechanism of moisture removal in the experiments discussed in this work.

Conversely, r_{res} and therefore the mass of SS$_{res}$ decreased as the treatment temperature was increased (Fig. 2a): a significant decrease occured at 120 °C (reduction of 47 %), while further changes above 150 °C were minor (reduction of 73–80 %). This could be associated with the removal of moisture assessed by r_c, not excluding the depletion of SS$_{dry}$.

According to r_g trend (Fig. 2a) and assuming negligible losses, SS released a stable share of species incondensable at test conditions, equal to approximately 11 wt % of initially loaded SS$_{st}$: the released gases never were the dominant fraction of pre-treatment products, but constituted an additional clue about depletion of SS$_{dry}$.

Overall, the trends of r_c, r_{res}, r_g suggested that the release of moisture was accompanied by other modifications in SS$_{dry}$, which was plausible when considering the volatile content of SS$_{st}$ (proximate analysis, Table 2). Deeper insights came from characterizations of solids (Section 3.2.1) and condensates (Section 3.2.2) after tests.

3.2.1. *Solids after tests*

The SS$_{res}$ samples were not sticky after the pre-treatment at 120 °C or higher temperatures (18.1 wt % or lower moisture, Fig. 1b); only SS$_{res}$ at 75 °C (59.6 wt % of moisture, Fig. 1b) was slightly sticky. This could be ascribed to different amounts of residual moisture (Fig. 1b), in agreement with Schnell et al. [8] who state that SS is highly viscous, sticky, and tough in the range 55–35 % of moisture.

Table 2 compares the proximate analyses of SS$_{res}$: the ratio between volatile matter and fixed-carbon decreased along with pre-treatment temperature (from 6.4 of SS$_{res}$ at 75 °C to 1.8 of SS$_{res}$ at 250 °C, Table 2), indicating the progressive loss of volatile compounds as the pretreatment temperature was increased; as a consequence, the ash and fixed-carbon shares increased along with the pre-treatment temperature. Data in Table 2 constitutes a solid evidence of SS$_{dry}$ depletion.

The compositions of SS$_{res}$ samples in terms of ultimate elemental analysis are in Fig. 1b; Fig. 1c summarizes experimental results about solids in the form of mass balances concerning a starting base of 100 kg of SS$_{ar}$.

The moisture content in SS$_{res}$ (M_{res}, Eq. (5)) decreased along with temperature (Figs. 1b and c) in agreement with r_c in Fig. 2a; the total moisture removal was ensured by temperatures above 200 °C (Fig. 1). 120 °C was the lowest acceptable temperature to pre-treat SS for a thermochemical conversion (Fig. 1b), considering the reference thresholds of admissible moisture discussed in the Introduction (<20 %, Table 1).

The overall mass of C, H, N, S, O in SS$_{daf}$ significantly decreased after thermal pre-treatments at temperatures of 150 °C or greater (Fig. 1c). An analysis of trends of single elements in Figs. 1b, c and 2b ($\Delta j(T)$, Eq. (8)) gave additional evidence on depletion of SS$_{dry}$.

The $\Delta C(T)$ was 5 wt % at 150 °C and 23 wt % at 250 °C (Fig. 2b). A mass balance based on Table 2 and Fig. 1c revealed that the mass of fixed carbon of SS$_{res}$ samples was not affected by the temperature of thermal pretreatment: only slight random variations of fixed carbon masses were estimated (standard deviation of 0.5 kg); therefore, C losses in Fig. 1c were attributed to volatile matter. The $\Delta H(T)$ was 6 wt % at 150 °C and 46 wt % at 250 °C (Fig. 2b). Even though the reactive environment was oxidative, $\Delta O(T)$ at 150 °C and greater temperatures was significant, up to about 54 wt % at 250 °C (Fig. 2b). Overall, trends of $\Delta C(T)$, $\Delta H(T)$, $\Delta O(T)$ can be related to the release of organic compounds from SS$_{daf}$; literature [55] findings suggest hydrocarbons and carbonyl compounds as possible released compounds.

As to potential pollutants in thermochemical conversions, the $\Delta S(T)$ reached 41 wt % after the treatment at 250 °C, whereas N remained relatively stable until 200 °C (negligible losses), decreasing by 1 wt % after the treatment at 250 °C. Even though S and N were minoritarian components of the SS (Fig. 1c), their removal from SS should be taken into account in the overall evaluation of the SS thermal pretreatment under air, since the effluents could contain polluting compounds, as shown in Section 3.2.2.

3.2.2. *Characterization of condensates*

The collected condensate samples were aqueous solutions, yellow, orange, or brown in color, and with a pungent odor. The condensate color was an additional indication that a depletion of SS$_{dry}$ occurred, as determined in Section 3.2.1.

Fig. 3 gathers the characterizations of condensates, including the pH and conductivity (Fig. 3a), ammoniacal nitrogen (Fig. 3b), organic acids (Figs. 3c and d), carbon forms (Fig. 3e), and oxidable matter (Fig. 3f).

According to mass balances on volatile matter and fixed carbon discussed in Section 3.2.1 (based on Table 2 and Fig. 1), the compounds other than water found in the condensate derived from the volatile matter in SS$_{daf}$.

Neither pH nor conductivity of condensates (Fig. 3a) substantially varied along with pre-treatment temperature: pH ranged within 9.10–10.12 and conductivity within 0.63–2.39 mS/cm (for comparison purposes, consider that mineral waters have 10^1–10^3 mS/cm). The highest value of pH was obtained at 150 °C (Fig. 3a), confirming a pH trend chemically compatible to that of organic acids (Figs. 3c and d) since high values of pH correspond to a decrease of acidity. Conductivity measurements are increasingly considered in literature as useful lumped indicators of thermal reaction progress [56]. In this perspective, our preliminary measurements could serve as a baseline for future research activities aimed at correlating condensate conductivity with solid-phase depletion and liquid-phase composition, thus enabling a rapid and low-cost monitoring tool.

Ammoniacal nitrogen ranged between 400–1166 g/l (Fig. 3b), ammonia between 514–1500 g/l. Organic acids ranged from 884 to 4997 g/l (Fig. 3c). At 200 °C, the highest content of ammonia and

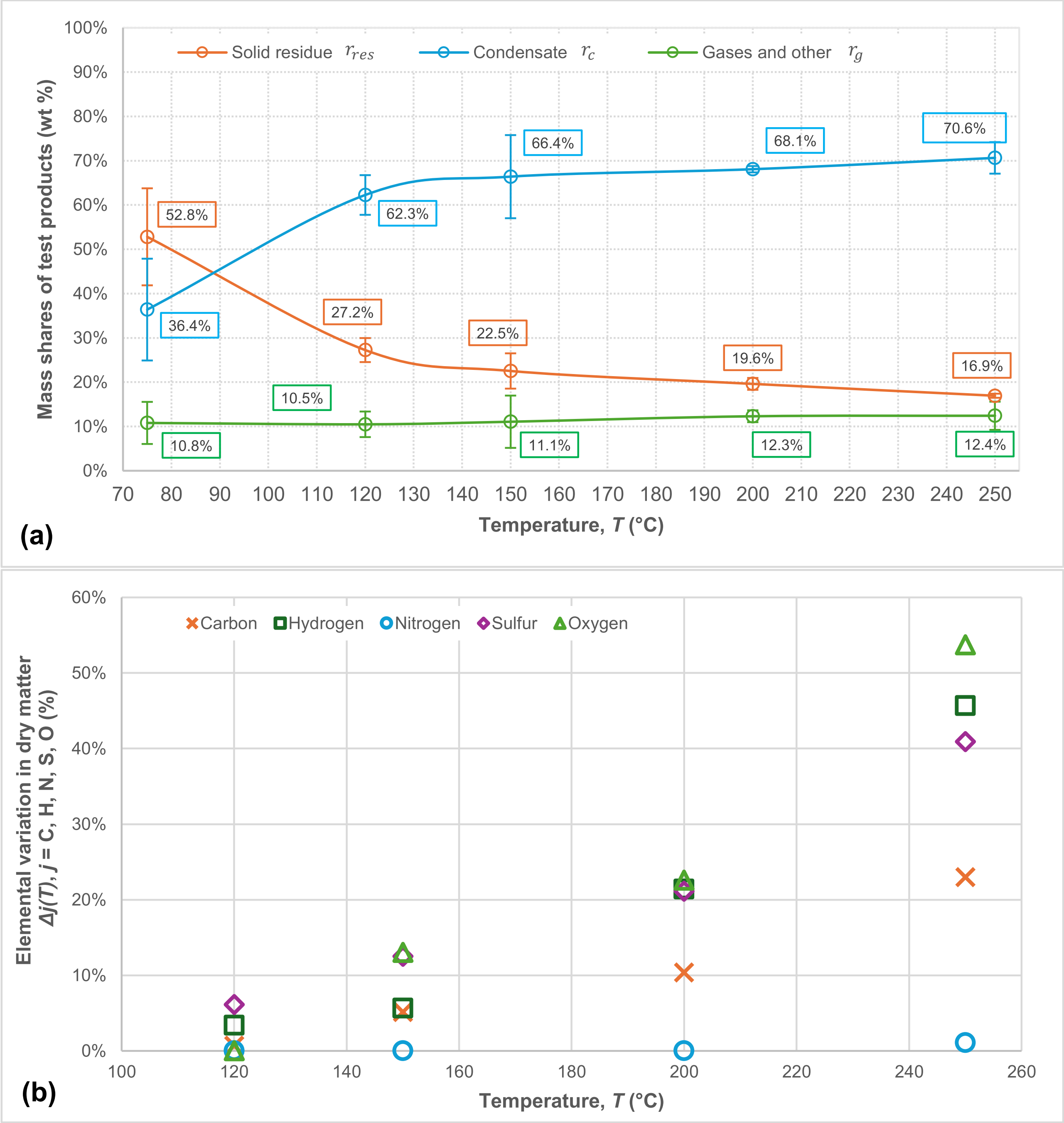

Fig. 2. Post-test mass shares of solid residue (r_{res}, Eq. (2)), condensate (r_c, Eq. (3)), and gases-losses (r_g, Eq. (4)), with error bars = 95 % confidence intervals (a); Percentage elemental variations of C, H, N, S, O in SS dry matter due to thermal pre-treatments at temperature T, Δj(T) (with $j = C$, H, N, S, O, Eq. (8)) (b).

organic acids was registered. Nitrogen content (Fig. 3b) in condensate was stable up to the treatment temperature of 150 °C, and increased at 200 °C and 250 °C, suggesting that SS_{daf} released nitrogenous compounds, in agreement with trends of elemental N in Figs. 1b and 2

The content of organic acids in the condensate varied along with the temperature of thermal pre-treatment (Fig. 3c). All identified acids contributed appreciably to the total acid content (Fig. 3d). Levulinic acid was approximately half by mass percentage compared to acetic and formic acid (Fig. 3d). The trend of organic acids content had a peculiar minimum at 150 °C (Fig. 3c), which suggested the occurrence of different mechanisms: the higher content of acids obtained at temperatures lower than 150 °C could be attributed to the loss of significant volatile matter already in the processed SS, because of physical separation and transport phenomena; on the other hand, the increase at 200 °C could be related to reactions such as hydrolysis, polymerization, and depolymerization [57], i.e., all actual chemical modifications of the fraction SS_{daf}.

The TC in condensates (Fig. 3e) had the same trends as the organic acids (Fig. 3c). Considering that the quantity of inorganic carbon was always low, the trend of TC was ascribable to TOC (Fig. 3e); indeed, TOC (Fig. 3e) showed a minimum at 150 °C similarly to organic acids (Fig. 3c). The highest value of TC was reached at 200 °C (more than 3 g/l of TC, Fig. 3e), which further suggested the enhancement of thermal depletion of SS_{daf} components at higher temperatures.

The BOD_5 (Fig. 3f) was approximately stable at about 320 mg/l, except for a minimum (196 mg/l) for the treatment at 150 °C, in line

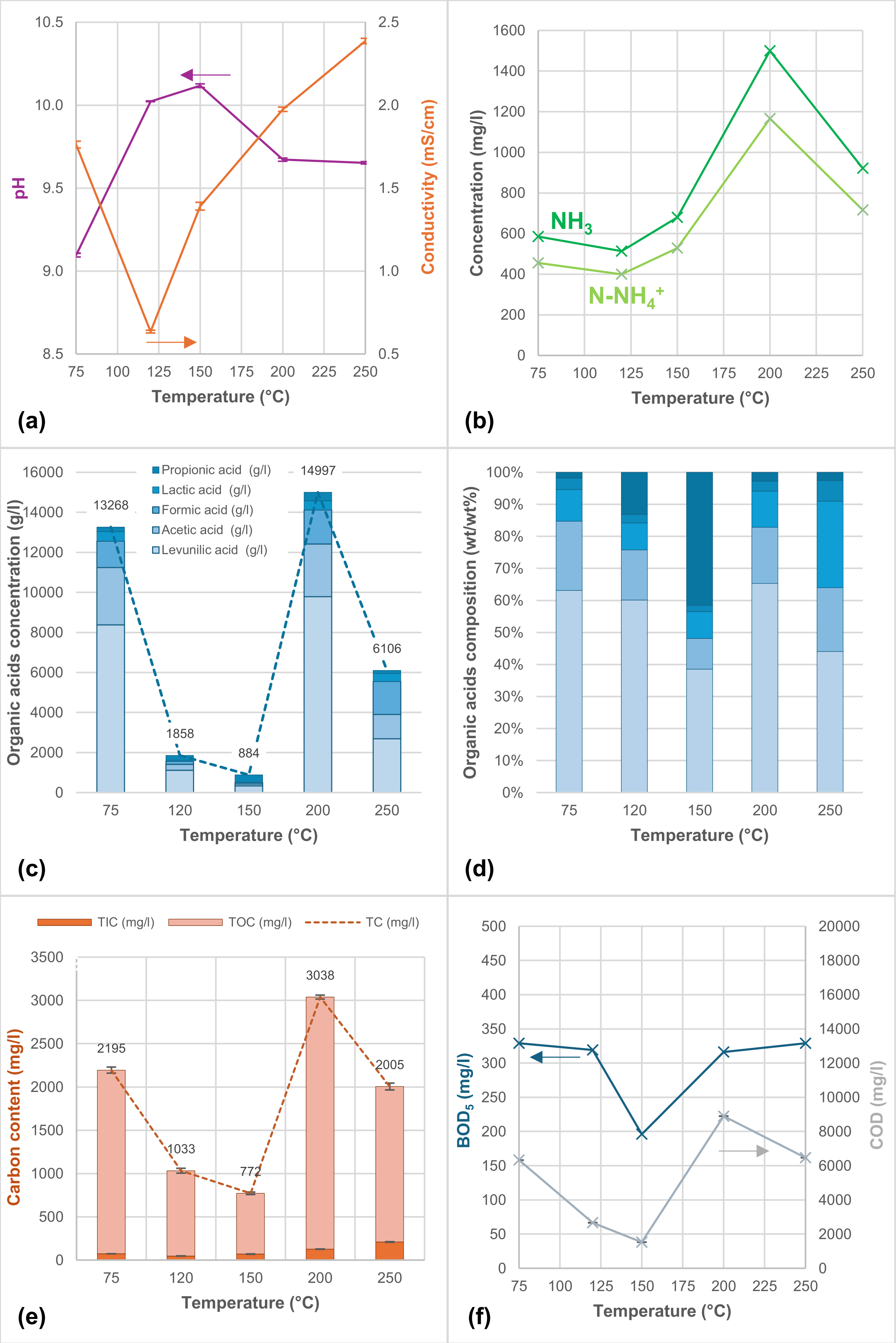

Fig. 3. Characterization of post-test condensate: pH and Conductivity (a); Ammoniacal nitrogen content (b); Organic acids content as solution concentration (c) and relative mass composition (d); Total Organic Carbon (TOC), Total Inorganic Carbon (TIC) and Total Carbon (TC) contents (e); Biological Oxygen Demand in 5 days (BOD5) and Chemical Oxygen Demand (COD); error bars = 95 % confidence intervals.

with the trends of organic acids (Fig. 3c) and TOC (Fig. 3e). Moreover, the increase of BOD_5 and COD (Fig. 3f) at higher temperatures matched well with the hypothesis about the depletion of the SS_{daf} fraction. The significant BOD_5 and COD values in the condensate obtained at 200 °C (Fig. 3f) indicate that this was the richest liquid in terms of oxidable matter, well corresponding to the fact that it was the richest in carbon (TC, TOC, TIC, Fig. 3e), ammoniacal nitrogen (Fig. 3b), organic acids (Fig. 3c).

Overall, the maximum of pH and minima of several properties investigated in Fig. 3 occurred around 120 °C and 150 °C, suggesting that SS_{daf} was depleted by two different mechanism, respectively dominant below or above a threshold temperature in the range 120–150 °C. Literature provides mechanism insights in agreement with these observations: Camacho et al. [58]. performed a literature study about oxidative thermal pre-treatments of SS between 50–420 °C, finding that 150 °C is a threshold beyond which the COD release dramatically increases, referred to as *"probably characteristic of different physico-chemical modifications"*; Foladori et al. [59] report that thermal pre-treatment around 150 °C can cause SS liquefaction, desorption, and cell lysis. Considering the correlation between COD data and those of TOC and organic acids (Figs. 3f vs. e and Fig. 3c), it is sensible to hypothesize that SS liquefaction, desorption, and cell lysis occurred at 150 °C [59] and contributed to the increase of TOC and organic acids concentrations in condensate phases at 200 °C and 250 °C (Fig. 3f, Fig. 3e and Fig. 3c).

4. Discussion and process observations

4.1. Indications for the unit operation

The temperature range of 120–150 °C brought in many advantages for the SS pre-treatment: (i) a satisfying residual moisture level in the SS below 20 % (Fig. 1), as identified in the Introduction; (ii) the avoidance of SS stickiness, making SS suitable for grinding and feeding to continuous reactors for thermochemical conversions; (iii) a generally less polluted liquid condensate (Fig. 3); (iv) the good containment of SS_{daf} depletion (Figs. 1 and 2).

The preliminary removal of N and S operated in the convective thermal pretreatment may be an advantage for the unitary process of thermochemical conversion (e.g., decreasing the shares of H_2S and NH_3 in syngas produced by gasification); on the other hand, the proper management of gaseous effluents from the thermal pre-treatment should be considered in an actual process scheme of thermochemical conversion (see Section 4.3).

The relatively high content of ash in SS (Table 2 and Fig. 1) must be taken in due regard in future applications of SS as a fuel for thermochemical conversions [60–64]: in general, ash is a byproduct that complicates the management of the thermochemical process; in the specific case of fluidized-bed reactors, the accumulation of ashes triggers agglomeration of bed particles, jeopardizing fluidization quality.

Scale-up evaluations for the industrial-feasibility of convective thermal pre-treatment should duly consider the influences from the transport phenomena. On the other hand, experimental conditions (air medium and temperatures below 250 °C) are plausible to exert an actual convective thermal pre-treatment. This is demonstrated by several industrial facilities operating between 70 °C and 200 °C (e.g.: Belt Sludge Drying | HUBER Technology 70–150 °C [65]; Belt drying system BDS for sludge 120–150 °C [66], Biosolids/Sludge Dryer - Komline around 200 °C [67]). Therefore, the discussed experiments can inform actual unit operations at industrial scale about chemical-physical modifications of SS.

4.2. Pollutants in condensate

Condensates are potential effluents of the SS convective pre-treatment framed as a unitary operation in a thermochemical conversion plant.

Even at their measured minimum (tests at 150 °C, Fig. 3), both BOD_5 and COD exceeded their corresponding environmental thresholds of Italian legislation (i.e., 250 mg/e for BOD_5 and 500 mg/e for COD [68]). Moreover, the value of ammoniacal nitrogen greatly exceeded the Italian legal limit of 30 mg/l, and the pH was partially out of the acceptable range of 5.5–9.5 [68]. Consequently, the direct discharge of condensates in the environment is not permissible without prior treatment; in other words, condensate phases as those obtained in this work would become additional industrial wastewater. Beyond its environmental significance, condensate characterization may also be exploited as a process monitoring tool. Previous studies demonstrated its correlation with the kinetics of biomass thermal conversions [56].

From a circular-economy perspective, the disposal of condensate should be substituted by alternative recovery practices. Condensates might be a sustainable source of valuable platform chemicals, such as furfural or 5-hydroxymethylfurfural [69]. In any case, new processing routes should be evaluated from the perspective of all three aspects of sustainability (economic, social, and environmental) [70].

4.3. Energy estimations

Table 3 shows the LHV calculated for SS_{ar}, SS_{st} and SS_{res} obtained at different tested temperatures. The LHV increased along with the severity of drying, from about 3 MJ/kg of SS_{ar} to a about 15–16 MJ/kg of SS_{res} at 150 °C or higher temperatures: this effect was due to the progressively lowered moisture content. On a dry basis, the LHV value remains nearly constant with an average of about 16 MJ/kg (SS_{dry}, Table 3), an effect ascribable to the buffering by the ash fraction. The LHV values on a dry ash-free basis (SS_{dry}, Table 3) exhibited a slight increase along with the pre-treatment temperature, ranging from 23 to 25 MJ/kg, demonstrating the influence of modification in elemental composition of SS_{daf}, documented by Figs. 1 and Fig. 2.

Fig. 4 shows results of energy estimations about the SS pre-treatment unit operation: only tests at 120 °C or above were considered, since 75 °C did not allow to reach the advised SS moisture content for thermochemical conversions (see Introduction, <20 % in Table 1) and was affected by higher variability (Fig. 2a). Due to the approximation hypotheses described in Section 2.4, estimations in Fig. 4 must be mainly intended as trend indicators.

The hypotheses of total oxidation of C, H, N, and S (Q_{dec}, Eq. (12)) appeared unfit to the results shown in Fig. 3. For instance, part of C, H, O in SS was found as organic acids in condensates (see Section 3.2.2). Even though Q_{dec} was an overestimating quantity, results in Fig. 4 indicated that SS_{dry} decompositions, if exothermic, may contribute appreciably in the overall energy balance of the thermal pre-treatment (Fig. 4).

The calculation of specific heat externally provided (q_{ext}, Eq. (13)) should ensure a higher quantitative reliability. q_{ext} linearly increased along with the pre-treatment temperature between ~2800-4100 kJ/kg_{SSst} (Fig. 4). This suggested that the choice of a sufficiently low temperature can positively affect the affordability of SS pre-treatment and associated thermochemical process.

To operate an auto-thermal thermochemical conversion process, the energy needed by the pre-treatment must be provided by internal re-

Table 3
LHV of SS samples estimated by Dulong equation [46].

	LHV [MJ/kg of SS]	LHV [MJ/kg of SS_{dry}]	LHV [MJ/kg of SS_{daf}]
SS_{ar}	3.1	16.0	22.8
SS_{st}	3.6	16.0	22.8
SS_{res} post-test at 75 °C	6.5	16.1	23.1
SS_{res} post-test at 120 °C	12.9	15.8	22.2
SS_{res} post-test at 150 °C	15.3	16.6	23.7
SS_{res} post-test at 200 °C	15.9	16.0	23.3
SS_{res} post-test at 250 °C	15.5	15.9	25.2

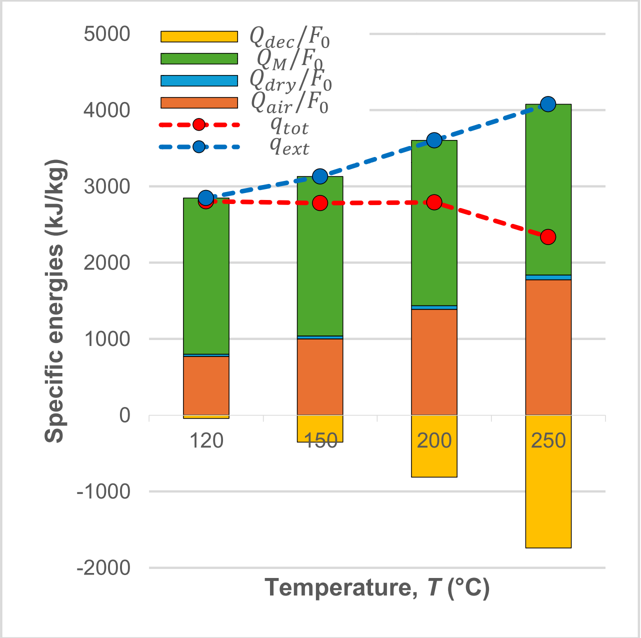

Fig. 4. Estimates of specific energy in SS thermal pre-treatment (per unit of pre-treated SS), as a function of pre-treatment temperature (from Eq. (9) to Eq. (14)).

coveries of thermal energy. The by far lowest energy share of q_{ext} is ascribable to the heating of SS_{dry} (always lower than 2 % q_{ext}), whereas those of moisture and air contribute substantially (Fig. 4).

Although the estimated energy demand q_{ext} was not negligible, its justification must be sought in the benefits to downstream thermochemical conversion processes. In fact, reducing SS moisture content would ensure autothermic operation of the system and enable more efficient combustion and gasification, improving both energy yields and process stability compared to operations with as-received SS. The estimated ratio LHV/q_{ext} ranged between 3.8 (pre-treatment at 250 °C) and 4.9 (pre-treatment at 150 °C), thus ensuring a potential positive net balance. Moreover, the air/condensate mixture produced during pre-treatment could be treated and reused as a heat stream in recovery units. In this perspective, the energy trade-off could have favorable potential, as the pre-treatment may contribute to a more sustainable and self-sufficient operation of thermochemical conversion plants.

It is worth pointing out that the pre-treatments investigated in this study may fit seamlessly into the schemes of traditional and innovative thermochemical processing plants, thanks to the use of low-temperature thermal energy that is ill-suited to other forms of recovery (e.g., sensible heat of flue gases). In this work, 120–150 °C resulted as suitable for the SS pre-treatment, indeed (see Section 4.2). For instance, in the hypothesis of plants for power production (by combustion or gasification with syngas combustion), the flue gases may be employed as a heating medium in direct contact with SS for thermal pre-treatment; otherwise, flue gases could become the service fluid to preheat the air fed in direct contact to SS, and exhaust air could be fed to the combustor.

5. Conclusions

A literature investigation suggested that SS should contain 20 wt % or less to be fed to a unitary process of thermochemical conversion. Experiments of SS convective thermal pre-treatment (2 NL/min, 2 h, 75–250 °C) showed that operations at 120–150 °C allow for reducing SS moisture below 20 wt %. The chemical-physical depletion of dry solids in SS was observed in addition to the nearly total moisture removal at 150 °C or higher temperatures. Condensates at 120–150 °C had the lowest content of pollutants, even though concentrations were still beyond the Italian legal limits for direct discharge. Temperatures of 120–150 °C are sensibly achievable by heat recoveries in plants that operate both traditional and innovative thermochemical processing.

This study provides a comprehensive, experimentally validated mapping of SS behavior under convective thermal pre-treatment across an industrially-relevant wide temperature range, highlighting that temperatures beyond 150 °C might trigger the onset of early thermochemical transformations of SS, and linking operating conditions to both solids and condensate characteristics. Notably, integrating thermal pre-treatment of SS in thermochemical conversion plants appears not only feasible, but advisable to increase energy efficiency and availability of the plant. Future efforts will aim at demonstrating this in real scale applications to highlight potential scale-up and integration issues.

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CRediT authorship contribution statement

Serena Capone: Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Methodology, Investigation, Formal analysis, Data curation. **Katia Gallucci:** Writing – review & editing, Validation, Supervision, Resources, Project administration, Methodology, Funding acquisition, Formal analysis, Conceptualization. **Luca Taglieri:** Writing – review & editing, Validation, Resources, Methodology, Investigation, Formal analysis, Data curation. **Daniel Mammarella:** Writing – review & editing, Visualization, Validation, Software, Methodology, Investigation, Formal analysis, Data curation. **Massimiliano Materazzi:** Writing – review & editing, Validation, Supervision, Project administration. **Matteo Stante:** Writing – review & editing, Resources, Funding acquisition. **Andrea Di Giuliano:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.jaecs.2025.100405.

Data availability

Data will be made available on request.

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