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Experimental and modelling optimisation of sustainable techniques for the pre-treatment of the organic fraction municipal solid waste to improve anaerobic digestion

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ABSTRACT

The study investigated and compared the anaerobic digestion (AD) of real organic fraction municipal solid waste (OFMSW) prior pre-treated with four types of pre-treatments: mechanical, thermal, hydrodynamic-cavitation (HC), and ultrasound (US). The tested pre-treatments and AD configurations were selected through Design of Experiments and then regression models were built to find the most promising configurations in terms of biogas production and energetic sustainability of the whole process. The novelty of the research is the simultaneously study of the working conditions of the pre-treatments; and AD parameters like the two origins of the inoculum, its incubation time, and the substrate: inoculum ratio (SI).

The results demonstrated that the best configurations of pre-treatments and AD were the ones performed with thermal pre-treatment at 120 °C for 45 min (with inoculum incubation of 10 d at substrate: inoculum (SI) ratio of 2:1) and HC at 55 °C (with inoculum incubation of 10 d at SI of 3:1). The thermal, and to some extent the mechanical pre-treatment, evidenced as significant the interaction between the pre-treatment time and the inoculum incubation time. AD of US-OFMSW achieved the lowest performances since inhibition occurred, probably due to the lignocellulosic inhibitors release after ultrasound pre-treatment.

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1. Introduction

Anaerobic digestion (AD) is a mature technology adopted to stabilise the organic matter and convert it into biogas. Nowadays, the global warming change challenge is promoting the application of renewable alternative sources of energy, and AD can enhance biogas production, which is a renewable energy employed to produce heat-electricity and transport fuel. The above-mentioned applications of biogas are not still widely implemented due to the higher costs of biogas production rather than other renewable energy sources such as wind or photovoltaic. Nevertheless, biogas is an energy which can be stored and used directly without conversions and is able to face the problem of peak requirement and power failure (Scherzinger and Kaltschmitt, 2021). Due to these properties, AD is a mature technology to face climate change and resource depletion and produce clean energy (Hai et al., 2022) according to the Sustainable Development Goals.

The optimization and improvement of AD consists in the study of the reactor design, the process conditions (as temperature, pH, mixing, etc.), the feedstocks employed and its pre-treatment (Kainthola et al., 2019). Based on the current scientific literature, pre-treatments may significantly improve AD, considering both the efficiency of the pre-treatment

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Abbreviations: AD, anaerobic digestion; CAS, mesophilic digestate of cow-agriculture sludge; COD, Chemical Oxygen Demand; DoE, design of experiment; DR, disintegration rate; HC, hydrodynamic-cavitation; OFMSW, organic fraction municipal solid waste; TOC, Total Organic Carbon; TS, total solids; VS, volatile solids; WAS, mesophilic digestate of wastewater activated sludge.

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and its effect on the overall AD. The efficiency of the pre-treatment depends on the technique as well as on the type and composition of substrate employed. The performance of AD depends on several factors: the kinetics and the hydrolysis, the rate-limiting step, which depends on process conditions, the substrate composition, and the biodegradability (Carmona-Cabello et al., 2018). In the literature, referring to AD, the term biodegradability expresses the amount of material biologically convertible in methane. Complex substrates can range from no biodegradable, as lignin, to complete biodegradable, as starch and sugar (Cheah et al., 2019). Nevertheless, biodegradable compounds could have low degree of bioavailability since part of the bio-matter can be incorporated into complex and barely biodegradable lignocellulosic structures. In addition, substrates constituted by large particles will be slowly degraded due to the actual limited surface area (Harun et al., 2019).

Currently, the pre-treatments are investigated for substrates as wastewaters from treatment plants, crops-harvesting residues, wastes from the food industry, animal manure and organic fraction from households. The Organic Fraction Municipal Solid Waste (OFMSW), considered in the present study is a heterogeneous substrate, coming from the collection of municipal wastes including food industry and households, with an average composition including easily biodegradable compounds (as 17.5% lipids, 17.7% proteins, 17.1% starch, 10.5% free sugars), and harder biodegradable components (as 18.6% cellulose, 9.7% lignin and 8.6% hemicellulose) (Pleissner and Peinemann, 2020).

In the last 40 years the most studied pre-treatments of organic wastes from the food industry and households reported in the scientific literature available in Science Direct, concerning the laboratory scale, are mechanical (35%), thermal (26%), ultrasonic (18%), chemical (12%) and microwaves (9%).

Mechanical pre-treatments have been evaluated because the particle size significantly affects the kinetics and stability of AD, determining the success or failure of the process.

Thermal pre-treatments have been performed at mild temperature because most part of OFMSW consists of starch and hemicellulose, which can be hydrolysed at relative low temperatures (from 90 to 180 °C) with longer residence time (Li et al., 2017). Hydrothermal, steam explosion and vapour-thermal are the most adopted thermal pre-treatments; among them, the vapour-thermal pre-treatment requires a lower energy and a lower heating time compared to the hydrothermal and it could be done with jacket reactors for a wide range of substrates, without water addition (Scherzinger and Kaltschmitt, 2021).

Hydrodynamic cavitation (HC) prior to AD has provided a faster disintegration and the solubilisation of larger organic molecules, so that it could be easily digested by the microbial inoculum, promoting a lower incubation time, higher degradation rates, and higher COD reduction with higher biogas generation (Saxena et al., 2019).

According to (Demichelis et al., 2022), for AD in batch feeding mode, inoculum plays a key role and the most important parameters for the inoculum are the incubation time (Zhang et al., 2019a), and source, and the substrate: inoculum (SI) ratio (Zhang et al., 2019b).

For the best of author's knowledge, the available scientific studies about pre-treatments and AD focused on them separately as in the studies from (Cesaro et al., 2014) and (Karthikeyan et al., 2018), who reviewed the available pre-treatment methods for organic wastes before the AD process. Other studies focused on: i) the evaluation of the pre-treatment performances through the disintegration rate, as in (Demichelis et al., 2018); ii) on the effects of pre-treatments (use of acid or alkaline reagents, and effect of temperature) on the AD of waste activated sludge from the perspectives of organic matter composition, thermodynamics, and multi-omics, as in (Chen et al., 2022); iii) by considering the improvement of AD after the pre-treatments, by an approach based on Anaerobic Digestion Model 1 (ADM1), as in (Huang et al., 2021) and (Wang et al., 2020). All these studies revealed that pre-treatment is a fundamental step with recalcitrant feedstocks to improve the methane yield; but, they did not investigate the interaction between pre-treatments and AD, which can be accomplished only if the conditions of both phases are changed contemporarily. The present study evaluated the AD of real OFMSW after pre-treatment with four types of pre-treatments: mechanical, thermal, HC, and US. The study concerned experimental tests and modelling. The experimentally tested pre-treatments and AD configurations were selected through DoE, then the enhancement of AD process (pre-treatment and AD) was evaluated in terms of biogas and methane productions, VS removal, kinetic study, and ESI.

The novelty of the study was the optimization of AD considering the combined effect and the interaction between pre-treatment and AD: this was possible by varying simultaneously the conditions of the two phases according to DoE, instead of simply adopting these pre-treatments before AD. The results of the experimentation, were used to build regression model correlating the factors involved in the study, their interactions, and their quadratic effects, to the production of biogas and ESI, to obtain predictive models for the identification of the best running conditions for pre-treatment and AD. In the results section, the best running condition will be detected for each type of pre-treatment by separately considering the biogas, the kinetic and the ESI results. In the conclusion section the best overall running conditions will be identified, among all the tested pre-treatments, by combining the results obtained for biogas production, kinetics, and ESI.

These results are of fundamental importance since there is a lack of information to understand the simultaneous effect of different pre-treatment techniques on AD performances, that should be urgently studied to improve the whole AD process according to (Atelge et al., 2020) and (Abraham et al., 2020).

2. Material and methods

2.1. Substrate and inoculum characterisation

AD of OFMSW, provided by San Carlo S.p.A (Fossano, Italy), was performed with two inocula: the mesophilic digestate of wastewater activated sludge (WAS), according to (Kumar Biswal et al., 2020), provided by SMAT (Torino, Italy), and the mesophilic digestate of cow-agriculture sludge (CAS), based on (Gu et al., 2020), supplied by "Cascina La Speranza" (Fossano, Cuneo, Italy).

The OFMSW, WAS and CAS were characterised in Table 1. VS/TS and TOC contents of OFMSW were higher than 90% and 8000 mg/kg, respectively; and this abundance of organic matter proved the suitability of OFMSW to be employed as feedstock for AD (L.Zhang et al., 2019). OFMSW had acid pH (5.6 ± 0.2), but the addition of WAS and CAS inocula increased the buffer capacity, since their pH were7.1 ±0.1 (for WAS) and 7.7 ±0.1 (for CAS). The physical-chemical properties of WAS agreed with (Suksong et al., 2019) and the ones of CAS agreed with mixtures of inocula from dairy manure and agricultural residues as reported in (Chen et al., 2008).

The C:N ratio of CAS was more suitable for AD than that of WAS, due to the higher carbon to nitrogen balance. CAS (a mix of cow manure and agricultural residues) could improve AD for its C:N ratio because the inhibition effect of nitrogen and ammonia from manure was limited by the carbon deriving from agricultural residues.

2.2. Physical pre-treatments

In the present study the focus was on AD performed on pre-treated OFMSW. The DoE investigated the pre-treatments and the AD, to evaluate, specifically for mechanical and thermal pre-treatments, the interactions between these two steps (pre-treatment and AD).

The mechanical pre-treatment was performed with the mixer blender (Aigostar Archer 30RKN, China) of 1.8 L, at three maximum speed values (15, 30, 45and 60 min) (Gagić et al., 2018), requiring 0.023 kWh/L.

The thermal pre-treatment was performed in the heating bath (Corio

Table 1
Physical and chemical properties of OFMSW and inocula.

	TS (%)	VS (%)	pH (–)	C (%)	H (%)	N (%)	S (%)	C/N (–)	TOC (g/kg)
OFMSW (mean)	19.32	96.76	5.31	48.42	6.76	2.97	0.20	16.3	24,995.82
OFMSW (dev.st)	0.61	0.53	0.22	0.51	0.70	0.32	0.12	1.4	114.92
WAS (mean)	5.09	70.7	7.12	35.42	3.04	4.51	0.01	7.92	9.52
WAS (dev.st)	0.11	1.0	0.11	0.51	0.02	0.11	0.01	0.14	0.12
CAS (mean)	5.82	70.3	7.74	40.62	3.09	7.92	0.03	5.12	12.04
CAS (dev.st)	0.12	1.0	0.12	0.61	0.07	0.11	0.01	0.12	0.24

C Julabo, Merck, Germany) at three temperature values 60, 90 and 120 °C (Bruni et al., 2010) and for three time periods (15, 30 and 45 min), settled according to the mechanical pre-treatment. The energies to perform the pre-treatments were 0.040 kWh/L for T = 60 °C, 0.048 kWh/L for T = 90 °C and 0.059 kWh/L for T = 120 °C.

Two hydrodynamic-cavitation (HC) pre-treatments were performed using a rotor/stator HC unit (Rotocav®, E-PIC srl – Mongrando, Italy) at two temperatures (25 and 55 °C) for 10 min (Bruni et al., 2010). The two pre-treatments required 0.022 kWh/L and 0.073 kWh/L.

The ultrasound pre-treatment (US) was performed in a 3 L powerful multiprobe reactor (Weber Ultrasonics AG, Karlsbad - Germany) for 30 min at 22 Hz and 200 W (Lauberte et al., 2021), requiring 0.020 kWh/L.

2.2.1. Pre-treatment evaluation

The evaluation of each pre-treatment was performed through the Disintegration Rate (DR) (Eq. (1) -2) (Bougrier et al., 2005).

$$DR_{COD}(\%) = \frac{SCOD_1 - SCOD_0}{TCOD - SCOD_0} \bullet 100 \tag{1}$$

$$DR_N\ (\%) = \frac{SN_1 - SN_0}{TN - SN_0} \bullet 100 \tag{2}$$

where $SCOD_0$ and $SCOD_1$ are the Soluble Chemical Oxygen Demand (SCOD) before and after pre-treatment, respectively; TCOD is the total COD; SN_0 and SN_1 are the soluble nitrogen before and after pre-treatment, respectively, and TN is the total nitrogen.

Total and soluble COD and total nitrogen were detected through a COD LCI 400 and a LCK 338 (HACH LANGE GHB, Germany) and quantified by a spectrophotometer 5000 D, (HACH, Canada).

2.3. Anaerobic digestion set up

AD was performed on OFMSW in 1.0 L Pyrex glass bottles (Duran, Germany) with a working volume of 80%, at 37 °C, placed in a 55 L thermostatic water-bath (Julabo-Corio-C, Merck, Germany), operating in batch mode with 6% total solids (TS) of OFMSW. Each bioreactor was manually shaken, and AD ended when biogas production was below 1% v/v of the total volume of biogas produced up to that time (Angelidaki et al., 2009).

Each bioreactor was connected by 6 mm Teflon tubes (PTFE, Germany) to a gasholder, made by 2 L Pyrex glass bottles (Duran, Germany). Biogas was analysed qualitatively by a biogas-analyser (GA5000, GMBH, Germany) and quantitatively by water displacement.

AD on not pre-treated OFMSW was performed as control to detect increase or decrease of the performances with respect to pre-treated OFMSW.

The WAS and CAS inocula, selected considering a previous study (Demichelis et al., 2022), were separately cultivated under anaerobic conditions at 37 °C in 2 L Pyrex glass bottles (Duran, Germany), for three different periods (0, 5 and 10 d) and then inoculated in the pre-treated OFMSW considering the Substrate: Inoculum (SI) ratio ranging from 1:2 to 2:1 for mechanical and thermal pre-treatment and from 1:3 to 3:1 for HC and US, based on volatile solids (VS) (Demichelis et al., 2022). Since HC and US pre-treatments were already optimised in (Lauberte et al., 2021), tests were performed on the effect of the SI ratio.

2.4. Analytical methods

The OFMSW and the two inocula (WAS and CAS), were physically and chemically characterized.

TS and VS content were detected according to UNI EN 15216:2021 and elemental analysis (CHNSO) was performed through an Elemental Macro Cube system (Vario, Germany).

The VS removed at the end of AD was evaluated through Eq. (3) according to (Li et al., 2018):

$$VS\ removed\ (\%) = \frac{VS\ input - VS\ output}{VS\ input - (VS\ input \bullet VS\ output)} \bullet 100 \tag{3}$$

where VS removed is the percentage of removed volatile solids, VS input and VS output are the volatile solids concentrations in the feed substrate before and after AD.

The pH was measured according to DIN 38404 C5 methodology with a pH340 WTW pH-meter (Mettler Toledo, Germany).

2.5. Design of experiments

The adopted Design of Experiment (DoE) identified the role played by the factors, their interactions, and quadratic effects, and accomplished the optimization of the system with the final identification of the best conditions for process running.

For mechanical and thermal pre-treatments, the DoE involved the simultaneous study of factors related to pre-treatment and to AD to identify the effect eventually played by the interaction between these two phases.

For HC and US pre-treatments, the DoE involved only the optimization of AD since the pre-treatments were previously optimised (Calcio et al., 2018) (Lauberte et al., 2021).

Different DoEs were adopted to optimize the four pre-treatments since they differed from the number of factors to be studied; moreover, some practical constraints needed to be taken into account: i) the maximum number of experiments that could be run simultaneously, due to the number of available AD reactors; ii) the necessity to simultaneously run all the experiments related to a single pre-treatment to guarantee a lower experimental error; iii) the necessity to add some replications of the experiments to evaluate the experimental error. See the supplementary material for the complete list of experiments established by DoE and the types of models investigated.

For AD, three experimental factors were considered.

- inoculum incubation (INOC), set at three levels: 0, 5 and 10 d (Demichelis et al., 2022).
- SI, set at: i) three levels (1:2, 1:1 e 2:1) for mechanical and thermal pre-treatments, according to the ones investigated in (Demichelis et al., 2022), and ii) at five levels (1:3, 1:2, 1:1, 2:1, 3:1) for HC and US according to (Liu et al., 2019) and (Kawai et al., 2014).
- origin of inoculum (ORIG), set at two levels, namely WAS and CAS (Demichelis et al., 2022).

ORIG was a qualitative factor, hence the experiments identified by the DOEs were repeated for CAS and WAS independently.

The DoE investigated the performances of the biodegradation of

OFMSW; and specific biogas production (NL/kg vs) and ESI (−) were selected as the experimental responses to be modelled. The response surface methodology provided the best experimental conditions through a grid search algorithm exploring the obtained models in the overall experimental domain (scaled in the range [0,1] for each factor) with a step of 0.1 for each factor included in the model.

2.5.1. DoE for mechanical and thermal pre-treatments

For mechanical and thermal pre-treatments, the DOE included both the pre-treatment and the AD. For the AD, INOC was studied at three levels (0, 5 and 10 d), SI at three levels (1:2, 1:1 and 2:1) and ORIG at two qualitative levels (WAS and CAS) (Demichelis et al., 2022).

For the pre-treatments, the experimental factors were settled according to the study of DR_{COD} and DR_N developed in the present study (section 3.2):

- For the mechanical pre-treatment: the time of pre-treatment (PT) was studied at three levels (15, 30 and 45 min)
- For the thermal pre-treatment two factors were added: the temperature (TEMP) at three levels (60, 90 and 120 °C) and the time of pre-treatment (PT) at three levels (15, 30 and 45 min).

For the mechanical pre-treatment a central composite design was adopted with two replications of the centre of the domain to evaluate the experimental error. The resulting 16 experiments are reported in Table S1.

For the thermal pre-treatment, a fractional factorial design (FFD) 2^{4-1} was adopted, where the fourth factor (SI) was confused with the interaction between the first two (PT*TEMP), providing a total of $2^3 = 8$ experiments. A star design was added, providing $2p+1 = 2*4 + 1 = 9$ experiments (p being the number of factors), to evaluate the quadratic effects. Two more replications of the centre of the domain were added to evaluate the experimental error, providing a total of 20 experiments, reported in Table S2.

2.5.2. DoE for hydrodynamic cavitation and ultrasound

Since HC and US pre-treatments were previously optimised (Calcio et al., 2018) (Lauberte et al., 2021), only AD was investigated considering the SI ratio at five levels (1:3, 1:2, 1:1, 2:1, 3:1) according to (Liu et al., 2019) and (Kawai et al., 2014), while INOC was studied at three levels (0, 5 and 10 d). The DoE involved all the possible combinations of the levels for both factors, providing a total of $5*3 = 15$ experiments with one more experiment consisting in a replication of the centre of the domain to evaluate the experimental error. The resulting 16 experiments are reported in Table S3. For HC, the DoE was repeated independently at 25 and 55 °C.

2.6. Calculation of regression models

For each DoE, regression models were assessed relating biogas production and ESI to the investigated factors, their interactions, and their quadratic effects, independently for CAS and WAS origins. Only statistically relevant (α-level <0.05) coefficients, identified by ANOVA (Analysis Of Variance), were included in the final models (Box and Hunter, 2005). See Supplementary Material for the description of the coefficients included in each evaluated model.

2.7. Kinetic study

The kinetics of AD was studied to evaluate the disintegration rate (k_d) and the biogas volumetric rate. The k_d was calculated by a first-order kinetic model Eq. (4):

$$B(t) = B_{exp}(1 - e - k_d t) \tag{4}$$

where $B(t)$ is the cumulative methane production at given time t (d), B_{exp} represents the ultimate methane potential yield (NL/kg vs) at the 5th day, k_d is the first-order disintegration rate (1/d) and t is the time of the process (1/d).

The biogas volumetric rate was calculated through Eq. 5

$$V\ biogas\ rate\left(\frac{L}{L \bullet d}\right) = \frac{Biogas\ (L)}{Volume\ of\ reactor\ (L) \bullet time\ (d)} \tag{5}$$

2.8. Energy sustainable index

The energetic sustainability of AD was measured with the energy sustainable index (ESI) calculated according to (Kovalovszki et al., 2020) and Eq. (6):

$$ESI = \frac{Q_{pro}}{Q_s} \tag{6}$$

where Q_{pro} was the energy produced from AD, considering that methane equals to 7.2 kWh/m^3 (Rillo et al., 2020) and Q_s is the system thermal load measured in kWh (Eq. (7)), and corresponded to the sum of the thermal power required for heating the substrate (Q_{sub}), the heat loss from the reactor walls (Q_{loss}), the heat loss through the tube (Q_p), according to (Mehr et al., 2017) and the energy consumed to perform the pre-treatments ($Q_{pre-treatment}$) with the specific consumption reported in sections 2.2 for each type of pre-treatment.

$$Q_s = Q_{sub} + Q_{loss} + Q_p + Q_{pre-treatment} \tag{7}$$

3. Results

3.1. Disintegration rate

The study of the DR, calculated for COD and nitrogen (Fig. 1), was performed to establish the suitable experimental domain for the factors included in the study of the four investigated pre-treatments, in terms of improvement of the available degradable matter (increase of the solubilisation of ready-digestible compounds of the OFMSW).

For the mechanical pre-treatment, the highest DR values were achieved after 45 min of pre-treatment, whereas a further increase to 60 min did not show any statistically significant improvement of DR and for the AD tests, a pre-treatment time of 60 min was not considered.

For the thermal pre-treatment, the highest DR values were achieved at the highest tested temperature (120 °C) after 45 min of pre-treatment. By increasing the thermal pre-treatment time to 60 min, for all the tested temperatures (60, 90 and 120 °C), no statistically significant differences were detected ($\alpha - level = 0.05$), and a pre-treatment time of 60 min was not investigated in AD tests.

The results proved that, for thermal pre-treatments, the temperature played a more significant effect than time, according to (Gagić et al., 2018).

No statistical differences were detected in DR calculated for COD and nitrogen, comparing HC at 25 and 55 °C; and these two configurations were tested.

Notwithstanding the worst results achieved by the US pre-treatment on DR for COD and nitrogen (14.41% and 10.21%), compared to the other treatments, it was included in the experimentation since it is considered as promising according to the literature (Lauberte et al., 2021).

Among the tested pre-treatments, those reaching the highest DR_{COD}, were: thermal pre-treatment at 120 °C for 45 min (27.85%) and HC at 55 °C (27.92%) and 25 °C (27.86%), due to the synergic effect of temperature and time, and for HC pre-treatments, the formation of extremely reactive microenvironments generated inside the bubbles, characterized by intense pressure waves and hydraulic jets, and responsible of a series of chemical and physical transformations in the OFMSW. The DR indirectly describes the efficiency of degradation of complex organic substrates, but it only quantifies the performances of

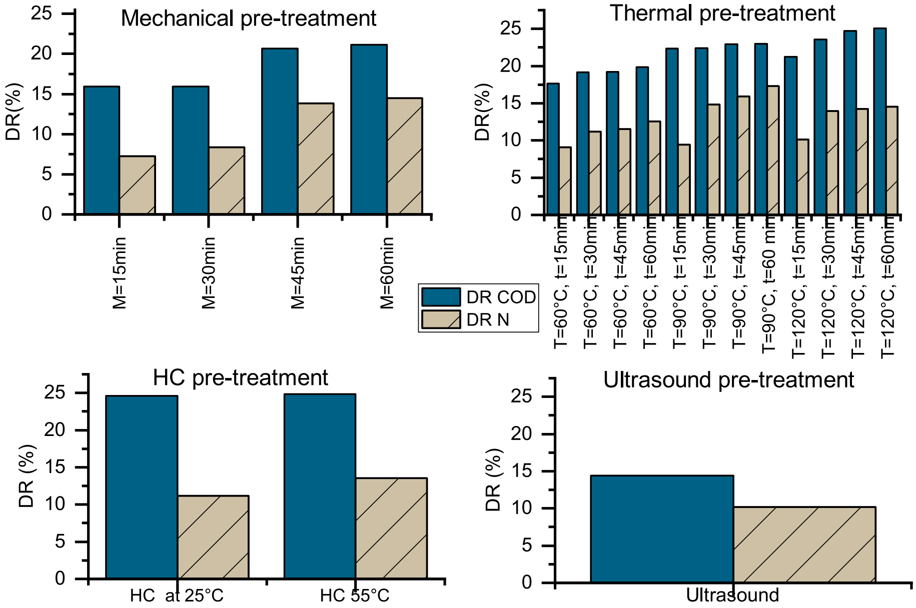

Fig. 1. Disintegration rate (DR) of pre-treatments calculated for COD and nitrogen.

the pre-treatment, neglecting its effect on AD.

3.2. *Biogas production*

The AD performance was investigated through the evaluation of the productions of biogas and methane (Fig. 2a), and the VS removal (Fig. 2b), according to (Li et al., 2019), and through the response surfaces of the models calculated for biogas production (Fig. 3).

3.2.1. *Mechanical pre-treatment*

In the case of AD of mechanically pre-treated OFMSW performed with WAS and CAS, the productions of biogas and CH_4 (Fig. 2a: A1,A2) and the VS removal (Fig. 2b: A1, A2) showed similar trends.

The surface responses (Fig. 3A1 and A2) and the built models (Table S4) proved that the interaction between INOC and SI was relevant; and that the effect of INOC was similar for WAS and CAS, but, the models for biogas production with WAS showed higher R^2 value ($R^2 = 0.9269$, Table S4) than CAS ($R^2 = 0.8161$), due to the absence of the SI parameter for the CAS origin ($\alpha < 0.05$). The biogas production (Fig. 3A1 and A2) improves by increasing INOC at high SI (2:1). The quadratic effect of INOC was also evident, while neither the linear nor the quadratic effect of the pre-treatment was relevant.

The best experimental conditions, identified by the grid search algorithm (Table 2), were, for the two inocula, at SI = 2:1, with INOC = 10 d and pre-treatment time of 15 min. For these configurations, biogas and methane productions and VS removal were in the range: 695.46–699.17 NLbiogas/kg_{VS}, 475.66–482.45 NL CH_4/kg_{VS} (Fig. 2a: A1, A2), and 71.99–73.52% w/w for VS (Fig. 2b: A1, A2), which were higher than the ones reached in (Zhang and Banks, 2013) (VS removal in the range 57–64% w/w for mesophilic AD of mechanically pre-treated OFMSW) due to the higher INOC (from 5 to 10 d).

The results proved that the particle size reduction did not notably increase the extent of degradation, because the important aspect was the formation of a homogeneous substrate to feed AD without impurities (Jain et al., 2015). The excess of smaller particles could lead to acid accumulation inside the digester as proven by (Panigrahi et al., 2020); indeed, AD of mechanically shredded OFMSW reached negligible different CH_4 content by reducing OFMSW particle size from 4 to 2 mm (respectively 0.34 and 0.31 Nm^3/kg_{VS}).

3.2.2. *Thermal pre-treatment*

For AD of thermally pre-treated OFMSW with WAS and CAS inocula, the biogas and CH_4 productions (Fig. 2a: B1, B2) and VS removal (Fig. 2b: B1, B2) showed similar trends.

The models for biogas production exhibited similar R^2 values for CAS ($R^2 = 0.9630$) and WAS ($R^2 = 0.9644$), and, for both inocula, PT showed a significant interaction with the INOC (Fig. 3B1, Table S5), while it was negligible as linear factor. This result was due to the higher buffering capacity of the incubated inoculum, which produced the acclimatised micro-organisms able to biodegrade the OFMSW (Zhang et al., 2019a).

Fig. 3B1, B2, and B3 represent only the response surfaces for WAS (the factors not represented are in turn fixed at the central value), since the two origins showed similar response surfaces. The quadratic effect of INOC was evident, and changes in INOC correspond to the most significant increases of the experimental response.

With the two inoculum origins, the configurations with pre-treatment at the highest temperature (120 °C) and the highest INOC (10 d) showed statistically negligible differences if pre-treatment was performed for 15 or 45 min, because at the highest temperature, the pre-treatment time was negligible due to the solubilisation and degradation effects played by the temperature (Li et al., 2017). This result is in agreement with the literature: the increase of temperature promoted the feedstock conversion degree, and the pre-treatment temperature affected AD performances more than pre-treatment time according to (Gagić et al., 2018). Thermal pre-treatment is usually carried out at a higher temperature (from 150 to 200 °C), but its main drawback is the high energy requirement, which usually cannot be balanced by the high biogas production, leading to the consequential reduction of the economic overall profitability of the process (Rajput and ZeshanVisvanathan, 2018). The optimal conditions identified through the grid search algorithm (Table 2) were, for the two inocula, at SI = 2:1, INOC = 10 d, and pre-treatment at 120 °C for 15 min: the biogas production was predicted to be equal to 665 NLbiogas/kg_{VS} in these conditions, which were not included in the DoE.

3.2.3. *HC pre-treatment*

The AD performances on HC-treated OFMSW at 25 (Fig. 3: C1, C2) and 55 °C (Fig. 3: D1, D2) were similar.

The model for biogas with CAS reached $R^2 = 0.9394$ at 25 °C and 0.9305 at 55 °C, higher than those obtained with WAS ($R^2 = 0.8474$ at 25 and 55 °C, Table S6). For CAS, the model contained all the

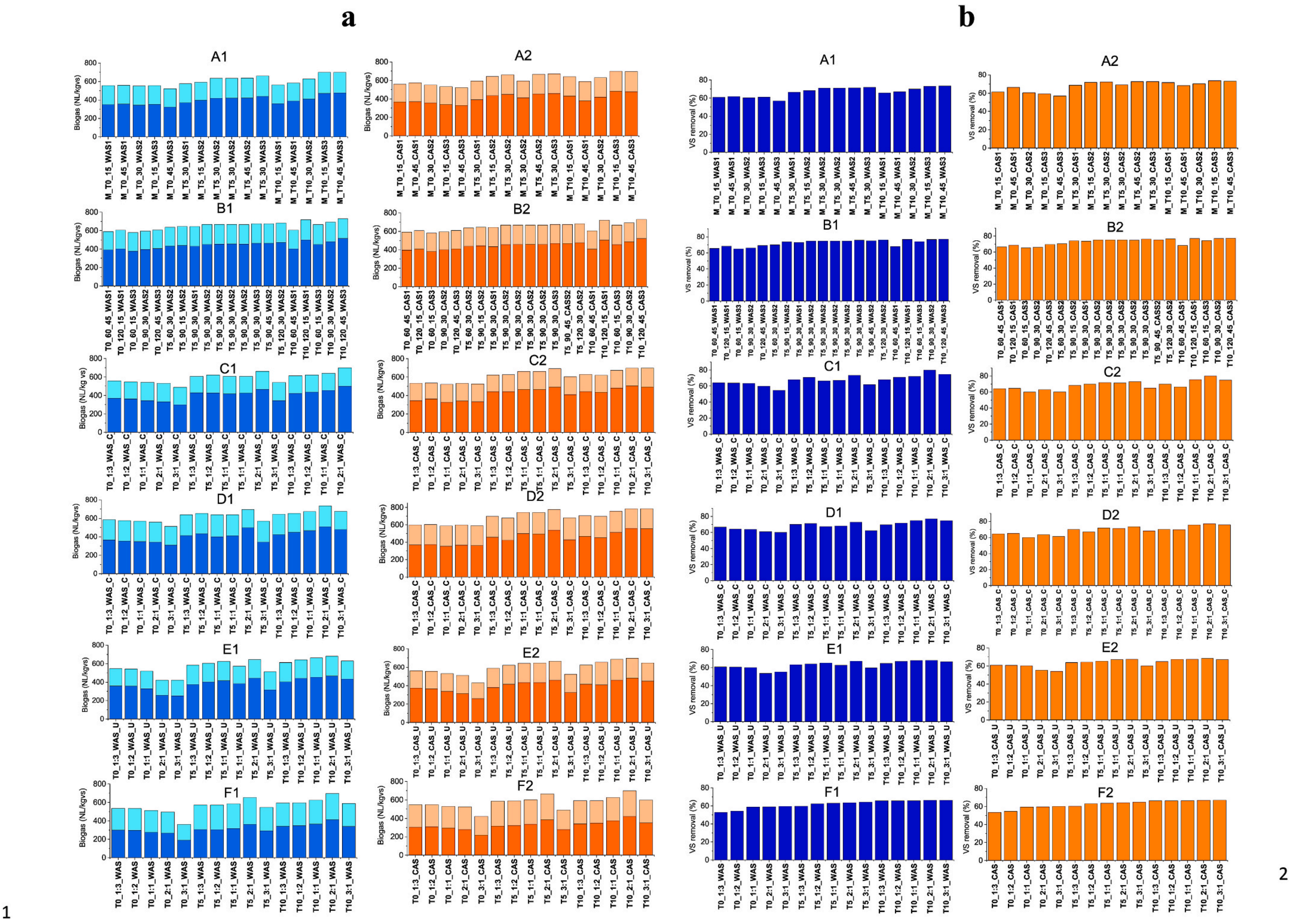

Fig. 2. a: Biogas production of AD on pre-treated and not pre-treated OFMSW: mechanical (A), thermal (B), hydrodynamic cavitation (HC) at 25 °C (C), (HC) at 55 °C (D), ultrasound (E), no pre-treatment (F). On the left, AD configurations carried out with inoculum WAS are reported (dark blue is methane, light blue is carbon dioxide), while those carried out with the CAS origin are reported on the right (dark orange is methane, light orange is carbon dioxide).
Fig. 2b: Volatile solids removal of AD performed on pre-treated and not pre-treated OFMSW: mechanical (A), thermal (B), HC at 25 °C (C), HC at 55 °C (D), ultrasound (E), no pre-treatment (F). On the left, AD configurations carried out with inoculum WAS are reported (blue), while those carried out with the CAS origin are reported on the right (orange). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

parameters, whereas, for WAS, the model excluded SI and the quadratic effect of INOC. For all the models, all the parameters were statistically significant ($\alpha < 0.05$).

The biogas productions of HC at 25 and 55 °C with CAS (Fig. 3: C2 and D2) provided comparable results: a quadratic effect was evident for SI and INOC, proving their synergic effect in increasing the biogas production. The best conditions identified by the grid search algorithm were at high levels of INOC, equal to 8 d for HC at 25 °C (predicted biogas production = 700.62 NL/kg $_{VS}$) and 9.5 d for HC at 55 °C (predicted biogas production = 798.07 NL/kg $_{VS}$), at SI = 3:1.

For HC at 25 and 55 °C with WAS (Fig. 3 C1 and D1), the maximum biogas production occurred at high INOC values (from 5 to 10 d) and medium-high value of SI (from 2:1 to 3:1), due to its evident quadratic effect. The best conditions identified by the grid search algorithm (Table 2), for HC, at 25 and 55 °C, with WAS were: INOC = 10 d at SI = 2.74 (predicted biogas production = 670.44 NL/kg vs. for HC at 25 °C and 705.36 NL/kg vs for HC at 55 °C).

The AD of HC-OFMSW at 55 °C achieved higher biogas and CH_4 productions (Fig. 2a: C, D) than HC at 25 °C, since during the collapse phase realised by HC, the highest temperature promoted the formation of more reactive microenvironments which boosted the diffuse turbulence, the phase changes, and the heat exchanges, occurring from macro to micro scales (Calcio et al., 2018).

3.2.4. *Ultrasound pre-treatment*

For US pre-treatment, the biogas and CH_4 productions and VS removal had similar trends for AD performed with WAS and CAS inocula, but the AD performed with CAS inoculum reached higher performances (Fig. 2a: E; Fig. 2b: E).

The model for biogas with CAS reached higher R^2 value (R^2 = 0.9286, Table S7) than WAS (R^2 = 0.8756), with a model containing INOC, the quadratic effect of SI and the interaction between INOC and SI for both origins ($\alpha < 0.05$). By increasing INOC, the biogas production increased independently of SI (Fig. 3: E1, E2), whereas SI showed a significant quadratic effect at high INOC values. These results proved the suitability of the incubated inoculum to treat higher OFMSW amounts and to promote its degradation (Zhang et al., 2019a).

The optimal conditions identified by the grid search algorithm corresponded to INOC = 10 d: at SI = 2.74 for WAS (predicted biogas production = 683.95 NL/kg vs) and SI = 2.34 for CAS (predicted biogas production = 703.63 NL/kg vs) (Table 2). These results agreed with (Rasapoor et al., 2016) where US was carried out on 6% TS OFMSW for 30 min at 200 kHz.

Among the tested pre-treatments, US reached the lowest biogas and methane productions and VS removals because probably the lipids accumulation negatively influenced AD since their degradation was relatively slow and led to an accumulation of hydrophobic lipids which

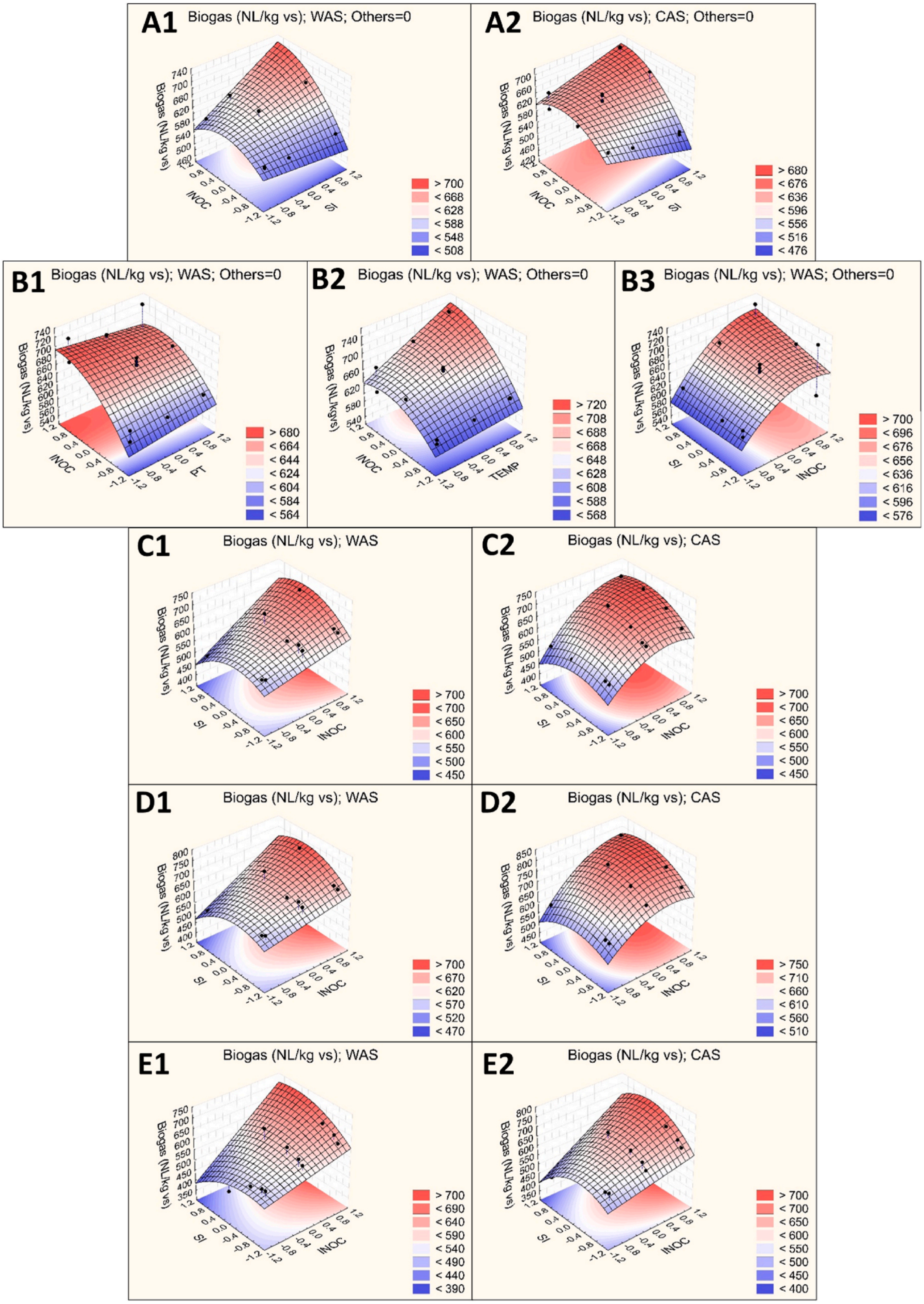

Fig. 3. Response surfaces of biogas production for the pre-treatments: mechanical (A1, WAS; A2, CAS), thermal (B1, B2, B3, WAS). HC at 25 °C (C1, WAS; C2, CAS), HC at 55 °C (D1, WAS; D2, CAS) and ultrasound (E1 for WAS; E2 for CAS). For thermal pre-treatment only the response surfaces for WAS are depicted since the models for WAS and CAS are almost identical.

Table 2
Response prediction of AD of pre-treated OFMSW (/) is reported when the parameter was not included in the optimization; - is reported when the parameter can be kept at any level).

			Best conditions ranged between −1 and +1					Best conditions reported in the original measure units			
			PT	TEMP	INOC	SI	Y best	PT (min)	TEMP (°C)	INOC (d)	SI
Mechanical	**Biogas**	WAS	–	/	1	1	703.00	–	/	10	2:1
		CAS	–	/	1	1	684.70	–	/	10	2:1
	ESI	WAS	–	/	1	–	1.01	–	/	10	–
		CAS	−1	/	1	1	1.17	15	/	10	2:1
Thermal	**Biogas**	WAS	−1	1	1	1	752.70	15	120	10	2:1
		CAS	−1	1	1	1	753.20	15	120	10	2:1
	ESI	WAS	1	1	1	0.65	2.13	45	120	10	1.74:1
		CAS	1	−1	1	1	1.20	45	60	10	2:1
HC Cavitation 25°C	**Biogas**	WAS	/	/	1	0.8	670.44	/	/	10	2.74:1
		CAS	/	/	0.8	1	700.62	/	/	8	3:1
	ESI	WAS	/	/	1	1	1.09	/	/	10	3:1
		CAS	/	/	1	0.9	1.12	/	/	10	2.87:1
HC Cavitation 55°C	**Biogas**	WAS	/	/	1	0.8	705.36	/	/	10	2.74:1
		CAS	/	/	0.9	1	798.07	/	/	9.5	3:1
	ESI	WAS	/	/	1	0.7	1.07	/	/	10	2.60:1
		CAS	/	/	1	0.8	1.12	/	/	10	2.74:1
Ultrasound	**Biogas**	WAS	/	/	1	0.8	683.95	/	/	10	2.74:1
		CAS	/	/	1	0.5	703.63	/	/	10	2.34:1
	ESI	WAS	/	/	1	1	0.90	/	/	10	3:1
		CAS	/	/	1	1	0.92	/	/	10	3:1

were adsorbed on the microorganisms' surface (Hendriks and Zeeman, 2009) with the effect of limiting the mass transfer process between microbial cells and dissolved organic matter (Scherzinger and Kaltschmitt, 2021).

The decrease of biodegradability after pre-treatment occurred for two main effects: formation of refractory/toxic compounds (Carrère et al., 2009) and removal of organic material (Hendriks and Zeeman, 2009). The US pre-treatment of the lignocellulosic fraction of OFMSW can release hydroxymethylfurfural (HMF), furfural, and soluble phenols (Hendriks and Zeeman, 2009), or produce melanoidins by uncompleted Maillard reactions (Pilli et al., 2011), which inhibit the AD.

3.2.5. *Comparison of the physical pre-treatments and no pre-treated OFMSW*

The performances of AD on physical pre-treated OFMSW were higher than those on untreated OFMSW tested in (Demichelis et al., 2022) in 0.5 L bioreactors and re-tested in the present manuscript in 2 L bioreactors to evaluate the scale effect (Fig. S1).

The biogas production of mechanically pre-treated OFMSW was higher than untreated OFMSW, in the ranges 3.0 and 7.3% in agreement with (Coarita Fernandez et al., 2020), 4.6 and 9.8% for mild-thermal pre-treatments in agreement with (Chen et al., 2020), 7.8 and 11.8% with HC according to (Saxena et al., 2019), 2.5 and 4.7% with US as proved by (Rasapoor et al., 2016), since pre-treatments increase the exposure of the biodegradable matter to microorganisms and vary the composition of hardly degradable matter (Zhen et al., 2017).

3.3. *Kinetic study*

The kinetic study proved the importance of the incubation time and of the origin of the inoculum (Fig. S1).

AD performed with INOC equal to 5 and 10 d promoted hydrolysis, acidogenesis, acetogenesis and methanogenesis without inhibition, achieving the faster k_d and volumetric biogas rates.

The origin of the inoculum influenced the k_d, and CAS inoculum exhibited a faster degradation rate than WAS, in agreement with (Kumar Biswal et al., 2020): because the proper C:N ratio of CAS supported a correct development of the AD process (Calcio et al., 2018), and the incubation of the inoculum provided acclimatised micro-organisms able to biodegrade the OFMSW (Zhang et al., 2019a).

In all the tested pre-treatments, the values of k_d and biogas volumetric rate were linearly correlated.

In AD of mechanically pre-treated OFMSW, the highest volumetric biogas rate and k_d were achieved, for the two inocula, by increasing INOC (5 and 10 d) and SI, without a significant effect of the extension of the PT ($k_d = 0.33$ and 0.50 1/d), as proved by (Gagić et al., 2018). The study of (Motte et al., 2015) stated that the fine milling of organic waste may simultaneously increase the AD kinetic and failure (Victorin et al., 2020), whereas in the present study these risks were limited by the presence of the incubated inoculum.

For AD of thermally pre-treated OFMSW, the highest volumetric biogas rate and k_d were achieved by AD performed with INOC = 10 d, at SI = 2:1 and pre-treatment at 120 °C for 45 min: 3.31 with WAS and 3.33 NL/kgvs d with CAS (L. Zhang et al., 2019), and k_d equal to 0.51 and 0.53 1/d with WAS and CAS respectively (Zhang et al., 2019b). These results proved that kinetic values increased by increasing INOC (from 0 to 10 d) and pre-treatment temperature (Li et al., 2017), because the incubation promoted the formation of acclimatised micro-organisms, while the temperature boosted the solubilisation of the OFMSW improving its bio-degradation.

AD performed on HC-OFMSW at 25 and 55 °C with the two inocula, showed the same kinetic configuration trends: the volumetric biogas rate and k_d increased by increasing the INOC (from 0 to 10 d and the HC temperature, due to the simultaneously effect of the inoculum incubation and the higher capacity of extraction of bioactive compounds characteristic of HC performed at higher temperature (Calcio et al., 2018).

AD of US pre-treated OFMSW reached the highest kd and volumetric biogas rates with the highest INOC = 10 d, notwithstanding the value of SI, for the two inocula.

For AD performed with incubated inocula, the k_d and volumetric biogas rates increased by increasing the SI ratio, because the inoculum with acclimatised micro-organisms could be employed with a lower amount than a non-incubated inoculum (Zhang et al., 2019a).

The k_d of the four types of pre-treatments varied between 0.1 and 0.58 1/d, ranging from the lowest to the highest specific biogas production, according to the optimal range of 0.134–0.56 1/d stated by (Li et al., 2018). The k_d of carbohydrates ranged from 0.5 to 2.0 1/d, proteins varied between 0.25 and 0.8 1/d and lipids ranged between 0.1 and 0.7 1/d, (Victorin et al., 2020). In the present study, mechanical, thermal and US pre-treatments could promote the release of carbohydrate compounds, whereas HC pre-treatment, a mix of carbohydrates and lipids.

The inocula incubation provided the optimal consortium of anaerobic microbes able to prevent inhibition, due to the higher SI ratio (from 3:1 to 2:1) (Browne and Murphy, 2013), whereas the non-incubated inocula negatively affected the lag phase (λ) and the maximum specific biogas production (Dasgupta and Chandel, 2019).

Among the tested AD configurations, AD of HC-OFMSW reached the highest k_d, since HC is a promising strategy to overcome the non-degradability of the recalcitrant components in AD (Naran et al., 2016) (Saxena et al., 2019).

3.4. Energy sustainable index

3.4.1. Mechanical pre-treatment

The ESI major than 1 for AD performed on mechanically pre-treated OFMSW was reached by the same configurations with WAS and CAS (Fig. 4A1-A2, and Fig. S2 and Table S4), but different models were obtained for AD performed with CAS and WAS(Table S4). The model for WAS reached a lower R^2 (equal to 0.8963) and contained only INOC, while the model for CAS contained INOC, PT and two interactions (SI * PT and SI * INOC), proving the relevant interaction between the pre-treatment phase and the AD ($R^2 = 0.9369$).

In the case of WAS, no surface responses are provided since the model was simple and the best conditions were obtained with high values of INOC (10 d) notwithstanding the values applied for PT and SI; these two factors can be therefore kept at the most convenient value equal to PT = 15 min at SI = 2:1.

In the case of the CAS origin, looking at the surface responses, (Fig. 4: A1, A2), considering INOC*SI, the ESI increased by increasing INOC at high and low SI, since INOC (Table S4) played the most significant role (Zhang et al., 2019a).

The best configuration stated by the grid search algorithm (Table 2) was, for the two inoculum origins, PT15 min, SI = 2:1and INOC = 10 d, with a calculated response of 1.17 and 1.01 for CAS and WAS (close to the experimental ones, 1.14 and 1.04).

3.4.2. Thermal pre-treatment

The ESI of AD on thermally pre-treated OFMSW reached the same trends with WAS and CAS (Figs. S2 and 4B).

For thermal pre-treatment, the models with CAS and WAS reached high R^2 values ($R^2 = 0.9882$ for WAS and $R^2 = 0.9878$ for CAS, Table S5), and contained the same parameters ($\alpha < 0.05$). The two models were similar (Table S5), therefore, the response surfaces for the four significant interactions were reported just for WAS (Fig. 4: B1–B4). Considering the interactions, the trend of TEMP*PT (Fig. 4 B1) and TEMP*SI (Fig. 4 B3) was similar one to each other and the same can be observed for INOC*TEMP (Fig. 4 B2) and INOC*SI (Fig. 4 B4). Increasing TEMP, the ESI increased at low PT values, according to (Rittmann et al., 2018), while an increase of PT increased ESI at low TEMP values, without producing relevant effects at high TEMP values, in agreement with (Menardo et al., 2015).

This result proved that the pre-treatment temperature was more effective than time since the temperature boosted the solubilisation of the OFMSW reducing the ammonia concentration as the result of caramelization or Maillard reactions occurring at temperature above 90 °C, preserving the AD process.

The best configurations identified by the grid search algorithm for thermal pre-treatment (Table 2) were AD with INOC = 10 d at SI = 2:1, TEMP = 120 °C and PT = 45 min for CAS (predicted ESI = 1.20), followed by AD with INOC = 10 d and SI = 1.74, TEMP = 120 °C and PT = 45 min for WAS (predicted ESI = 2.13).

3.5. HC pre-treatment

For HC at 25 °C, the models with the two inoculum origins reached high R^2 values ($R^2 = 0.9635$ for WAS and $R^2 = 0.9390$ for CAS, Table 2), whereas for HC at 55 °C, the model with CAS reached a R^2 value higher than WAS ($R^2 = 0.9455$ for CAS, $R^2 = 0.8842$ for WAS, Table 2).

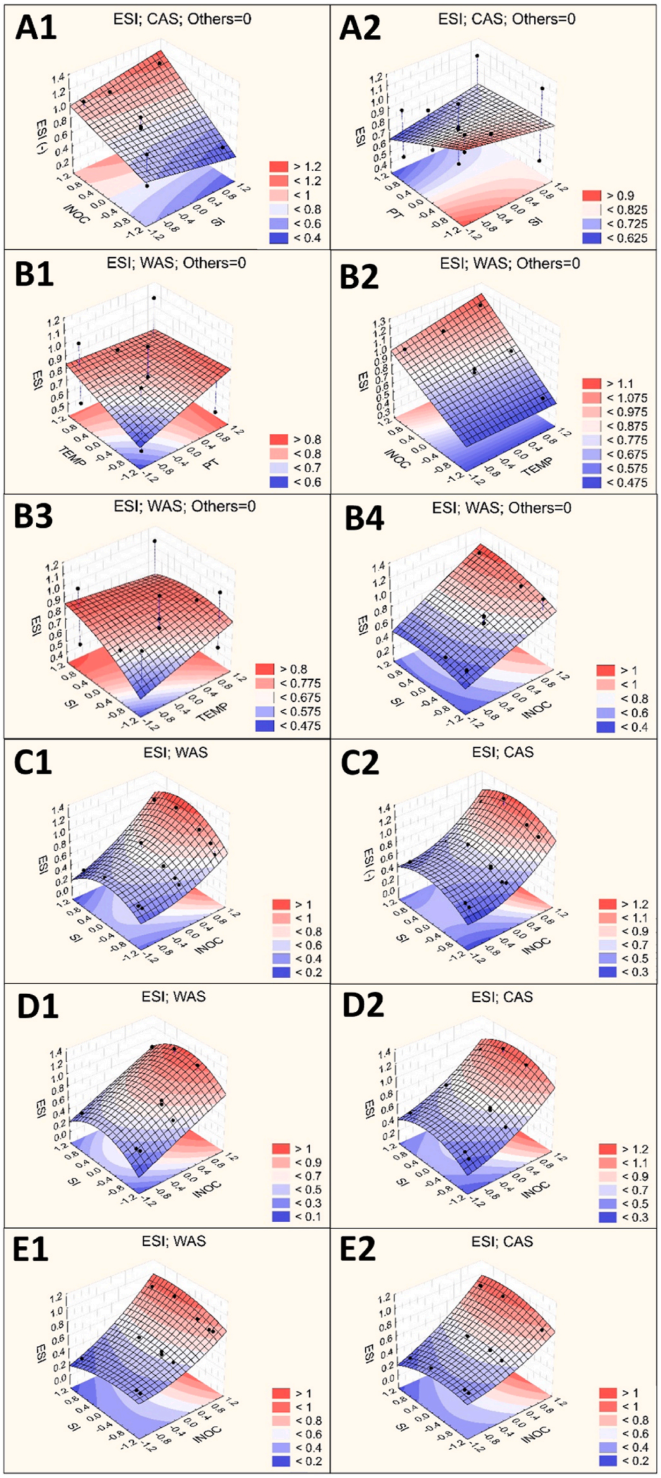

Fig. 4. Response surfaces of ESI for the four physical pre-treatments: mechanical (A1, A2), thermal (B1, B2, B3, B4) HC at 25 °C (C1, C2), HC at 55 °C (D1, D2) and ultrasound (E1, E2). For the mechanical and thermal pre-treatments only the response surfaces for WAS are reported since the models for WAS and CAS are almost identical.

The best conditions of ESI (Table 2, Fig. 4: C1, C2, D1, D2) were: INOC = 10 d and SI values ranging from 3:1 (WAS) to 2.87 (CAS) for HC at 25 °C and from 2.6 (WAS) and 2.74 (CAS) for HC at 55 °C. The predicted ESI values ranged between 1.07 and 1.12. These results proved the importance of temperature on the HC performance (Calcio et al., 2018).

The energy cost to carry out HC was covered by the biogas surplus produced by AD of HC-OFMSW, assessing HC as an effective pre-treatment (Saxena et al., 2019). The HC had a positive ESI due to lower plant and operating costs, higher process yields and energy savings due to shorter process times (Calcio et al., 2018).

3.5.1. *Ultrasound pre-treatment*

All tested configurations for AD carried out on ultrasound pre-treated OFMSW, with CAS and WAS, were energetically unsustainable (Fig. S2E).

The model with WAS and CAS contained all the parameters ($R^2 = 0.9832$ for WAS, $R^2 = 0.9775$ for CAS, Table S7). The response surface (Fig. 4: E1, E2) with WAS and CAS, proved that the increase of INOC increased ESI notwithstanding SI values, whereas ESI increased and reached almost a plateau by increasing SI at high INOC.

The best conditions identified by the grid search algorithm (Table 2) corresponded to INOC = 10 d and SI = 3:1 for both origins, but the calculated responses with these conditions reached ESI<1. The energetic unsustainability of AD performed on US pre-treated OFMSW was due to the high energy required to carry out the pre-treatments and lower CH_4 produced during AD (59.80 and 68.90 %v/v with WAS and 60.34 and 69.52 %v/v with CAS) compared to the other pre-treatments.

4. Conclusions

This study evaluated the AD of real OFMSW prior pre-treated with four types of pre-treatments: mechanical, thermal, HC, and US, to optimize the whole process. The tested pre-treatments and AD configurations were selected through DoE, considering the interaction between the pre-treatment and the AD phases. The experiments of each DoE were evaluated by measuring the biogas production, the VS removal, and the ESI. The results were used to build regression models correlating the responses to the factors involved in the study, their interactions, and their quadratic effects.

The best configurations of pre-treatments and AD were the ones performed with thermal pre-treatment at 120 °C for 45 min and inoculum incubation of 10 d at SI equal to 2:1, due to the thermal solubilisation effect, and HC at 55 °C and inoculum incubation of 10 d at SI equal to 3:1, for the combined heat-bubbling effect, which enhanced the availability of the digestible fraction of OFMSW. Pre-treatment time was significant only in the case of thermal pre-treatment and it showed a significant interaction with the inoculum incubation time.

The AD of US-OFMSW achieved the lowest performances since inhibition occurred. In the future the combined environmental and economic assessments of the four pre-treatments and AD will be investigated.

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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.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jclepro.2023.136594.

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