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(Article begins on next page)

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INVESTIGATION OF THE ANAEROBIC DIGESTION OF COSMETIC INDUSTRIAL WASTES: FEASIBILITY AND PERSPECTIVES

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Abstract

This study assessed the anaerobic digestion (AD) of wastes deriving from cosmetics production: sludge from onsite wastewater treatment plant (sWWTP), residues of shampoo/conditioner (RSC) and sludge from mascara production (MS), considered as single substrates and as mixture according to the produced amounts (54 %-wt sWWTP, 31 %-wt RSC, 13 %-wt MS, plus 2 %-wt food waste from the canteen, FW). Total COD (COD_T) was 624-1436 g O_2/kg $_{VS,}$ while soluble COD was 5-23 %-wt of COD_T. AD tests at 35 °C achieved the following biogas yields: 0.10 Nm^3/kg_{vs} (70 %-v/v methane) for sWWTP; 0.07 Nm^3/kg_{vs} (62 %-v/v methane) for RSC; 0.04 Nm^3/kg_{vs} (67 %-v/v methane) for MS. The mixed substrates underwent physico-chemical pre-treatments (thermo-alkaline, TA: 120 min at 50 °C; thermo-alkaline-sonication, TAS: 15 min at 40 kHz and 80 °C, both based on the addition of 0.08 g NaOH per each g of total solid in the substrate), reaching 64-66 % disintegration rate, and AD tests (5 %-wt dry substance) at 35 and 52 °C. Biogas yields were (for TA and TAS respectively): 0.22 and 0.20 Nm^3/kg_{VS} (62-70 % methane); 0.21 and 0.19 Nm^3/kg_{VS} (66-66 % methane) at 52 °C. At both temperatures, methane yields considerably improved (+71-100 %), compared to mixed

untreated substrates, and 5-8 %-wt total solids reductions were observed. A technical-economic scale-up assessment completed the research. The energy analysis highlighted the crucial role of TA pre-treatment in achieving the process energetic sustainability. The economic analysis showed that the AD of the considered cosmetic waste could be sustainable anyway, thanks to the savings related to the disposal of the digestate compared to current waste management costs.

Keywords: biogas, cosmetic waste, economic analysis, pre-treatment, scale-up.

1. Introduction

According to the European Parliament regulation No. 1223/2009 (European Parliament, 2009), cosmetic products may include "any substance or mixture intended to be placed in contact with the external parts of the human body with the aim of cleaning, perfuming, protecting, keeping them in good conditions". The same EU regulation bans the use of hazardous substances in cosmetic products to ensure the highest protection of human health. Growing interest for the sustainability of raw materials employed to manufacture cosmetic products (Bom et al., 2019) has been involving in recent years the consumers, cosmetics industries and academia. Nonetheless the major concern on ensuring the highest safety and sustainability of cosmetic products, the European Waste Catalogue (EWC) and the list of wastes in art. 7 of Directive 2008/98/CE often define hazardous and mark with an asterisk the EWC codes assigned to wastes deriving from the manufacturing of fats, grease, soaps, detergents, disinfectants and cosmetics (a sub-chapter of wastes deriving from organic chemistry processes). Liquid and solid wastes originating from cosmetic products manufacturing are usually incinerated or landfilled, while wastewater is typically treated in onsite treatment plants (Demichelis et al., 2018a), because of the high concentrations of chemical oxygen demand (COD), suspended solids, fats, oils and detergents (Abidemi et al., 2018). Enhanced onsite wastewater treatment operations are necessary in cosmetic industries to mitigate the impact of the treated effluent on the receiving

water bodies (De Melo et al., 2013). The presence of scarcely biodegradable compounds in cosmetics wastewater lead to the adoption of different treatment strategies, as:

1. *the enhancement of soluble COD to promote subsequent biological processes* through physico-chemical pre-treatments (Demichelis et al., 2018a), catalytic wet peroxide oxidation (Bautista et al., 2010) or combined light/Fe^0/H_2O_2 processes (Muszyński et al., 2019);

2. *the decrease of total COD* through flocculation/coagulation (Kim et al., 2020), electro-coagulation (Müller Tones et al., 2020), combined electro-coagulation and heterogenous photo-catalysis (Boroski et al., 2009) or advanced oxidation processes, as electro-chemical oxidation (Awad and Ghany, 2013), Fenton oxidation (Bautista et al., 2007), photo Fenton-like treatment and heterogenous photo-catalysis (De Andrade et al., 2020);

3. *the application of biological processes highly efficient towards the removal of high organic loads*, as membrane bioreactors (MBR) (Banerjee et al., 2016; Friha et al., 2014; Monsalvo et al., 2014), upflow anaerobic sludge blanket (UASB) (Puyol et al., 2011) and biofilm (Fan and Wang, 2017) reactors.

On the grounds of the performed literature survey on cosmetic industrial waste management, two issues appeared noteworthy: state-of-the-art literature exclusively focused on cosmetic wastewater treatment, leaving out any other waste; cosmetic wastewater treatment applied at full-scale only involve, to our knowledge, physico-chemical pre-treatments preliminary to active sludge processes, or MBR and UASB designs, while all other options, even if characterized by high COD removal performances, are not yet mature at full-scale and technically and economically feasible (Zhen et al., 2017). The aim of this work is to fill the gap investigating the feasibility of the AD of cosmetic waste. Compared to existing literature, this research has the two general elements of novelty: it is specifically focused on cosmetic waste and not on cosmetic wastewater; it explores the feasibility of the AD of unconventional scarcely biodegradable substrates. In details, this work investigated the treatment of the cosmetic wastes

(303 t/y) produced by L'Oréal Group in a plant located in Italy; specifically, sludge from the onsite wastewater treatment plant (sWWTP), residues of shampoo and conditioner (RSC, hazardous), sludge from mascara production (MS, hazardous) and food waste (FW) from the canteen, considered singularly and as mixture according to produced amounts. At the moment of this research, these waste materials were landfilled or incinerated. This work is the follow-up of a previous study (Demichelis et al., 2018a), which investigated the technical feasibility of different physico-chemical pre-treatments, based on chemicals (NaOH or HCl), heat (50-90 °C) and sonication, and their various combinations for different times and temperatures, on the same wastes. The performances of the pre-treatments were compared for the single wastes according to the disintegration rate (DR, i.e., the % enhancement of soluble COD) (Li et al., 2012) and the best results were obtained for thermo-alkaline (TA) treatment at 50 °C for 120 min and termo-alkaline-sonication (TAS) at 80 °C for 15 min, reaching DR equal to 64 % for TA and 66 % for TAS. Further AD tests at 35 °C on the waste mixture, feeding 5 %-wt dry substance after TA and TAS, showed 44-48 % increase of methane production (0.13 Nm^3/kg_{VS}) and 5-7 %-wt reduction of waste amounts compared to the untreated wastes. The study was concluded by a preliminary analysis of the scale-up at full plant scale of TA and TAS, reaching the economic feasibility (14-20 % savings compared to the adopted waste management operations), with TA exhibiting lower investment and operational costs compared to TAS. Related to the described previous study (Demichelis et al., 2018), the common aspects (apart of the considered cosmetic wastes) with the present work are just the adoption of TA and TAS as pre-treatments and the investigation of AD at 35 °C feeding 5 %-wt dry substance; the follow up presented in this work has five significant improvements: 1. the characterization of the samples was enhanced; 2. the investigation of AD explored mesophilic (35 °C) and thermophilic (52 °C) conditions; 3. AD tests were performed on single wastes at 35 °C and on their pre-treated (TA and TAS) mixture at 35 °C and at 52 °C (while the previous study considered AD tests only of the waste mixture

at 35 °C); 4. the performances and kinetics of the AD processes were analyzed through the modified Gompertz model; 5. finally, the overall process (pre-treatment + AD) was scaled-up based on energy and economic analyses, to explore the economic sustainability of the value chain.

2. Materials and methods

2.1 Substrates characterization

The wastes analyzed in the research were sampled in 2016 at L'Oréal Saipo Industriale (L'Oréal Group) plant located near Turin (Italy), producing shampoo and conditioner, mascara, and make-up powders for the mass market in 34 countries. Waste production in 2015 was equal to 303 t and included: sludge from onsite biological wastewater treatment plant (sWWTP) (EWC 070612), residues of shampoo and conditioner production (RSC) (EWC 070608*), sludge from mascara production (MS) (EWC 070601*) and food waste from the canteen (FW) (EWC 200108). At the moment of the research, sWWTP was landfilled, RSC and MS were incinerated, and FW was sent to composting. In this work, waste samples were considered individually and as mixture (in the following defined "Mix") according to the relative amounts produced in the plant in 2015 (54 %-wt sWWTP, 31 %-wt RSC, 13 %-wt MS, 2 %-wt FW).

The characterization involved 5 samplings for each waste, along 9 months, to assess any variability in composition. The measured parameters were: total solids (TS) and volatile solids (VS) according to reference procedures (APHA-AWWA-WEF, 2012); pH through a pH 340 WTM pH-meter; total chemical oxygen demand (COD) and soluble chemical oxygen demand (sCOD) according to a reference method (Raposo et al., 2008); ammonium through Nanocolor Ammonium Test 1-05 reagents and a UNICAM Helios Alpha UV-vis spectrophotometer; elemental analysis was performed by means of a CHNSO Thermo Fisher Flash 2000 Analyzer EA 1112. Unfortunately, only one replicate was possible in the elemental analysis of RSC and MS. The digestate was analyzed at the end of the AD tests measuring TS and VS and the lower

heating value (LHV) according to UNI EN 15170:2009 method, adopting benzoic acid as reference material. LHV analyses involved 5 replicates for each sample.

2.2. Pre-treatments

The pre-treatments tests involved the mixed wastes (Mix) according to the procedures optimized in a previous work (Demichelis et al., 2018a). Specifically, in both cases the NaOH was added to the Mix (0.08 g/g of TS of the substrate), and the two pre-treatments were performed as follows. The thermo-alkaline (TA) treatment at 50 °C for 120 min in a Julabo Corio-C thermostatic bath and termo-alkaline-sonication (TAS) at 40 kHz in a VWR USC 300TH device at 80 °C for 15 min. Full details about the optimization of the pre-treatments and the results achieved on single waste materials are available in the cited study (Demichelis et al., 2018a).

2.3. Anaerobic digestion tests

AD tests were carried out at 35 °C on the single waste materials (FW excluded) and the Mix, and at 52 °C on the Mix. In all cases in batch mode, feeding 5 %-wt TS and adopting substrate-inoculum ratio equal to 1:1 based on VS. The inoculum was provided by two local facilities: a WWTP performing mesophilic AD of the sludge and a plant performing thermophilic AD of the organic fraction of municipal solid waste; before the tests, the inoculum was stored at 20 °C and degassed for 7 days. The mesophilic inoculum was characterized by 1.71 %-wt TS, 75 % VS/TS and pH 7.3; the thermophilic inoculum by 2.65 %-wt TS, 60 % VS/TS and pH 6.9. The AD tests were performed in 0.5 L Pyrex Duran glass bottles (0.4 L working volume), connected by 6 mm Teflon tubes to 2.5 L gas-tight Tedlar bags. Each reactor was purged with nitrogen for 1 min to remove air from the headspace. A 55 L Julabo-Corio-C thermostatic water-bath allowed to control the temperature of the digesters. For each substrate (the single wastes and

the Mix), 12 reactors were engaged: 2 blanks (containing only the inoculum), 3 for biogas measurement and 3 for methane measurement. Unfortunately, only one replicate was available for methane measurement at 52 °C. During the tests, each reactor was manually shaken three times per day for 5 days per week. Biogas and methane volumes were measured 5 days per week through water displacement (10 g/L NaOH aqueous solution was employed to wash CO_2 from the reactors used for methane measurement) and referred to standard temperature and pressure (0 °C, 1 atm). The AD tests were declared completed when marginal biogas production was below 1 %.

2.4. Performance and kinetics analysis

The performance and kinetics of AD were analyzed according to Gompertz modified model (Nguyen et al., 2016) according to (eq. 1).

$$B(t) = P \exp\left\{-\exp\left[\frac{R_{max}\, e}{P}\,(\lambda - t) + 1\right]\right\} \quad (1)$$

where *B(t)* is the cumulative methane production ($Nm^3\ kgVS^{-1}$) at time *t* (day), *P* is the methane potential of the substrate ($Nm^3\ kgVS^{-1}$), R_{max} is the maximum methane production rate ($Nm^3\ kgVS^{-1}\ d^{-1}$), λ is the lag phase (day), *e* is the base of the natural logarithm. The kinetic parameters were estimated using non-linear least square regression analysis by means of the SOLVER tool pack of Microsoft Excel.

2.5. Scale-up assessment: reactor design and energy balance

The scale-up of the process applied on the Mix at 35 °C in a continuously fed digester was based on two scenarios: AD without any pre-treatment (S1) and after TA pre-treatment (S2), considering the amount of waste produced at the moment of the research (303 t/y) (section 2.1). Two sub-scenarios, based on the amount of available waste, were also considered for S2: a. investigating the size of the digester necessary to process 303 t/y of waste mixture; b.

investigating the minimum size of the digester necessary to produce enough methane to achieve energy self-sufficiency (thermal and electric energy). The reactor design parameters were derived from the results of the pre-treatments and of the AD tests presented in section 3.3.

The energy analysis was performed to estimate the net thermal energy (TE_{net}) and the net electrical energy (EE_{net}), expressed in MJ/year and considering one year of operations, as in eq. 1 and 2, adopting the parameters and calculations detailed in Table 1.

$$TE_{net} = TE_{CHP} - TE_{pre} - TE_{AD} - TE_{loss} \quad (1)$$

$$EE_{net} = EE_{CHP} - EE_{mix} \quad (2)$$

Where TE_{CHP} [MJ/year] is the thermal energy obtained from the biogas in a combined heat&power (CHP) unit; TE_{pre} [MJ/year] is the thermal energy spent during the TA pre-treatment; TE_{AD} [MJ/year] is the thermal energy required to heat the substrate prior to AD; TE_{loss} [MJ/year] is the energy lost through the walls of the reactor. EE_{CHP} [MJ/year] is the electricity obtained from the biogas in the CHP unit, and EE_{mix} [MJ/year] is the electrical energy needed for the mixing.

Table 1. Parameters and calculations adopted in the energy analysis

Parameter	Calculations	Notes	Reference
net thermal energy (TE_{net}) [MJ/year]	$TE_{net} = TE_{CHP} - TE_{pre} - TE_{feed} - TE_{loss}$		
heating pre-treatment (TE_{pre})	$TE_{pre} = m_{mix}\ C_p\ (T_{pre} - T_{amb})\ t\ 365 / \eta$	specific heat capacity (C_p): 4.18 MJ/(ton·K); efficiency of the heat exchange (η): 0.65 pre-treatment temperature (T_{pre}): 50 ◦C ambient temperature (T_{amb}): 15 ◦C; treatment time (t): 2 hours/day	Gomez-Camacho et al., 2021
heating feeding AD (TE_{AD})	$TE_{AD} = m_{mix,5\%}\ C_p\ (T_{AD} - T_{amb})\ 365 / \eta$	AD temperature (T_{AD}): 35 ◦C; ambient temperature (T_{amb}): 15 ◦C	Gomez-Camacho et al., 2021
thermal loss AD (TE_{loss})	$TE_{loss} = U_{wall}\ A_{reactor}\ (T_{AD}-T_{amb})\ 31.536$	natural heat convection h air: 10 W/(m^2·K);	Gomez-Camacho et al., 2021

		concrete: conductivity (λ) 2.3 W/(m·K); thickness (s): 0.25 m; insulation: λ: 0.037 W/ (m·K); s: 0.08 m, $U_{wall:}$ 0.422 W/(m^2·K); surface reactor ($A_{reactor}$): 89 m^2	
thermal energy from CHP (TE_{CHP})	$TE_{CHP} = Q_{CH4}\ \eta_{TH}\ LHV_{CH4}\ 365$	LHV methane (LHV_{CH4}): 35.8 MJ/m^3; CHP thermal energy efficiency (η_{TH}): 0.4 methane production (Q_{CH4}) [m^3/day]	Metcalf&Eddy, 2013
net electrical energy (EE_{net}) [MJ/year]	$EE_{net} = EE_{CHP} - EE_{mix}$		
electricity mixing AD (EE_{mix})	$EE_{mix} = P_{mix}\ t\ V_{reactor}\ 31536$	power input (P_{mix}): 0.02 kW/m^3; reactor volume ($V_{reactor}$): 64 m^3 mixing time (t): 15/60 hours	Gomez-Camacho et al., 2021
electricity from CHP (EE_{CHP})	$EE_{CHP} = Q_{CH4}\ \eta_{EL}\ LHV_{CH4}\ 365$	CHP electrical efficiency (η_{EL}): 0.4	Metcalf&Eddy, 2013

2.6. Scale-up assessment: economic analysis

A preliminary economic assessment was performed according to (Demichelis et al., 2018a and b; Chiappero et al., 2019) to estimate the profitability of the two scenarios detailed in section 2.5. The profitability assessment was based on the following parameters: return of investment (ROI), i.e., the ratio between the annual net profit and the initial investment; net present values (NPV), and payback time (PBT). A plant lifetime equal to 20 years and a 5 % discount rate on the future cash flows were considered. The economic assessment involved capital costs, operational costs, and revenues. The annual net profit was determined as the difference between the revenues and the operational costs; the amortization of the initial capital cost was not considered in the present analysis. The capital costs included the fixed capital costs (FCC), and the working capital costs (WCC) assumed equal to 6.5% of FCC [4]. The cost of land was not considered since the installation was placed inside the cosmetic industrial plant. The operational costs were composed by cost of the NaOH for the pre-treatment and of the water for the dilution of the Mix, by thermal and electrical energy (obtained from the energy assessment), and labor costs. The revenues derived from the surplus electricity from biogas co-combustion in the CHP

209 unit, and from the saved costs related to the disposal of the digestate (0.12 €/kg) compared to
210 the costs of the current waste disposal for sWWTP (0.15 €/kg), RSC and MS (0.31 €/kg), and
211 FW (0.11 €/kg) (Demichelis et al., 2018a).

212 **3. Results and discussion**

213 *3.1.Substrates characterization*

214 The results of the characterization of the studied cosmetic wastes (Table 2) showed that all
215 substrates could be eventually considered suitable for AD based on their physico-chemical
216 features (VS above 75 %, TS below 35 %, pH not too far from neutrality), even if the values of
217 C/N were rather lower (respectively 10.71 for sWWTP, 5.49 for RS, 5.87 for MS and 14.40 for
218 FW) than the values (20-35) usually recommended for AD (Metcalf&Eddy, 2013). Moreover,
219 the low values of COD_S/COD_T witnessed the need of pre-treatments aimed at increasing the
220 amount of soluble organic substances. sWWTP showed the highest variability in COD values,
221 which was not unexpected; in fact, the onsite WWTP was fed by wastewater deriving from the
222 washing of reactors and pipes in the productive lines of L'Oréal plant, where industrial
223 production was organized in batches of different cosmetic products.
224 The Mix (54 %-wt sWWTP, 31 %-wt RSC, 13 %-wt MS, 2 %-wt FW) was characterized by
225 14.06 % TS, 83.68 % VS/TS and C/N equal to 8.86.

226 **Table 2.** Characterization of the cosmetic wastes (*only one replicate available)

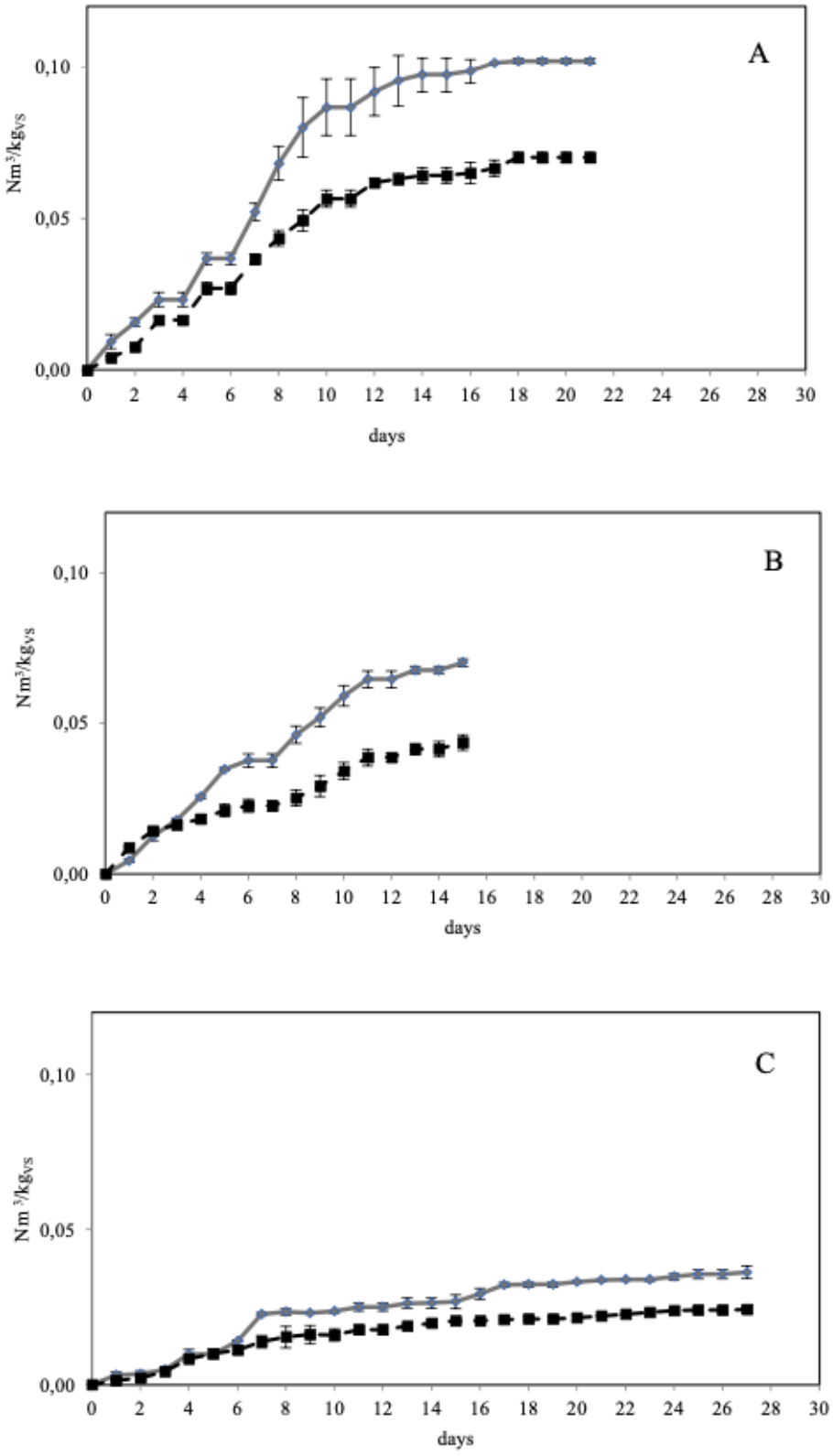
	sWWTP	RSC	MS	FW	reference
pH	6.54±0.49	6.30 ±0.17	6.69±0.79	5.08±0.34	Demichelis et al., 2018a
TS (%)	2.43±0.74	24.98±0.45	34.70±2.18	25.06 ±3.31	Demichelis et al., 2018a
VS/TS (%)	86.24±3.2	81.65± 1.51	75.46±1.28	97.17±0.95	Demichelis et al., 2018a
COD_T (mgO_2/g_{VS})	1174.06 ±94.80	1436±23.1	1148.52±17.56	624.25±48.4	Demichelis et al., 2018a
COD_S/COD_T	5.56±0.88	51.48±1.48	6.19±0.26	23.24±1.79	Demichelis et al., 2018a
NH_4^+ [mg/g_{VS}]	0.29±0.06	2.54±0.34	0.02±0.01	0.02±0.02	this study

C (%)	44.69±0.57	51.60*	60.50*	45.65±4.39	this study
H (%)	7.43±0.16	2.05*	0.30*	6.76±0.58	this study
N (%)	4.17±0.44	9.40*	10.30*	3.24±1.44	this study
S (%)	1.15±0.24	0.90*	0.11*	0.05±0.07	this study
formula	$C_{13}H_{26}O_{12}N$	C_6HO_6N	$C_{24}HO_{18}N$	$C_{22}H_{40}O_{20}N$	this study
C/N	11	6	6	14	this study

227

228 *3.2. Results of the anaerobic digestion tests performed on the single cosmetic wastes*

229 The AD tests performed at 35 °C on the single cosmetic wastes (Figure 1) provided some
230 interesting insights: sWWTP exhibited the highest specific biogas production (0.10 ±0.014
231 Nm^3/kg_{vs}, 70 %-v/v methane), followed by RSC (0.07 ±0.010 Nm^3/kg_{vs}, 62 %-v/v methane)
232 and MS (0.04 ±0.001 Nm^3/kg_{vs}, 67 %-v/v methane). These results demonstrated that the
233 considered cosmetic wastes could undergo AD, even if further actions are needed to improve
234 their biodegradability and therefore biogas and methane yields. TS removal was 2.1 % for
235 sWWTP, 24.2 % for RSC and 34.5 % for MS, meaning the following reduction of the amounts
236 of "raw" (i.e. as they are produced in the plant) wastes: 18.9 %-wt for sWWTP, 3.2 %-wt for
237 RSC and 1.43 %-wt for MS. As expected from the results of the characterization, sWWTP
238 showed the highest variability in biogas and methane specific daily productions. It was also
239 noteworthy the duration of the AD processes: 21 days for sWWTP, 15 days for RSC and 27
240 days for MS.



241

242 **Figure 1**. Results of AD tests at 35 °C on (A) sWWTP, (B) RSC and (C) MS (specific biogas

243 production in grey; specific methane production in black)

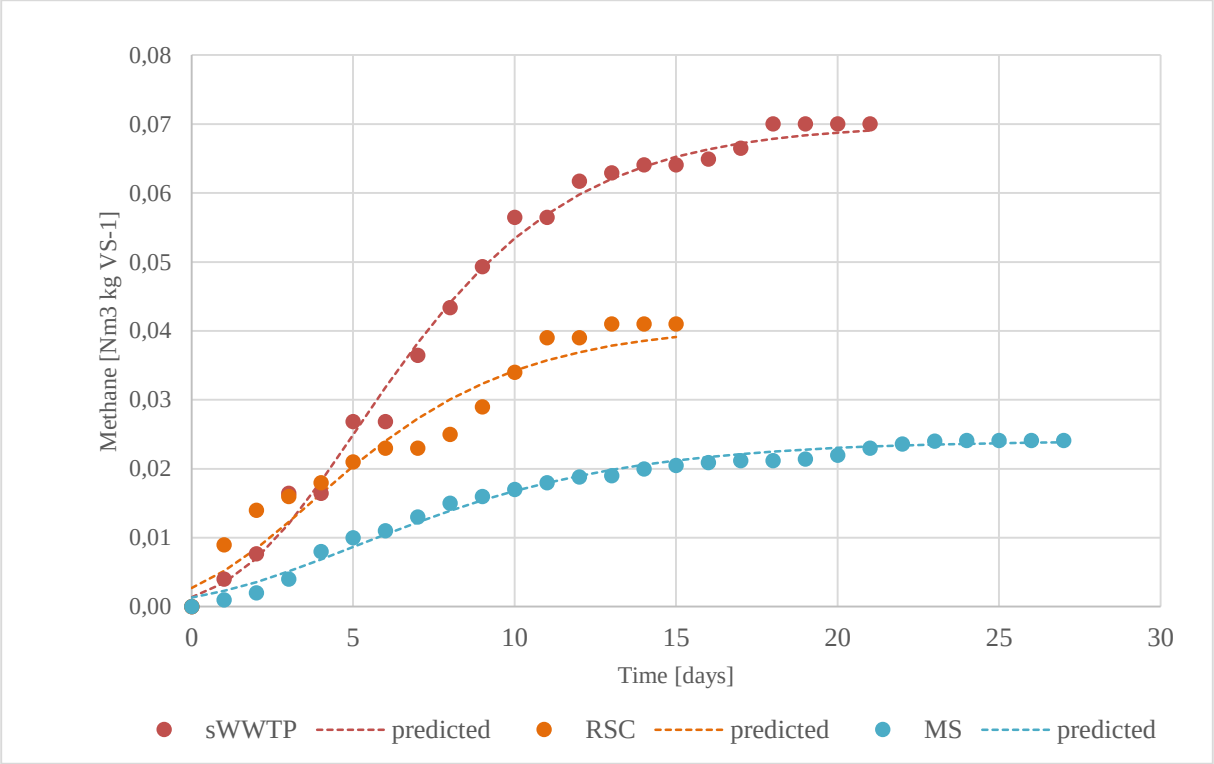


Figure 2. Results of the modeling of experimental methane production from the single cosmetic wastes at 35 °C through the modified Gompertz model

Table 3. Performance and kinetic parameters of the AD at 35 °C of single cosmetic wastes obtained through the modified Gompertz model (P: methane potential of the substrate; R_{max}: maximum methane production rate; λ: lag phase)

	P ($Nm^3\ kgVS^{-1}$)	R_{max} ($Nm^3\ kgVS^{-1}d^{-1}$)	λ (days)	R^2 (-)
sWWTP	0.070	0.007	1.385	0.993
RSC	0.041	0.004	0.000	0.933
MS	0.024	0.002	0.302	0.986

The performance and kinetic parameters estimated from daily methane production and methane yield of the single cosmetic wastes at 35 °C through the modified Gompertz model (Table 3, Figure 2 and Appendix) showed that sWWTP exhibited the highest methane potential (experimental values) and maximum methane production rate values, compared to RSC and

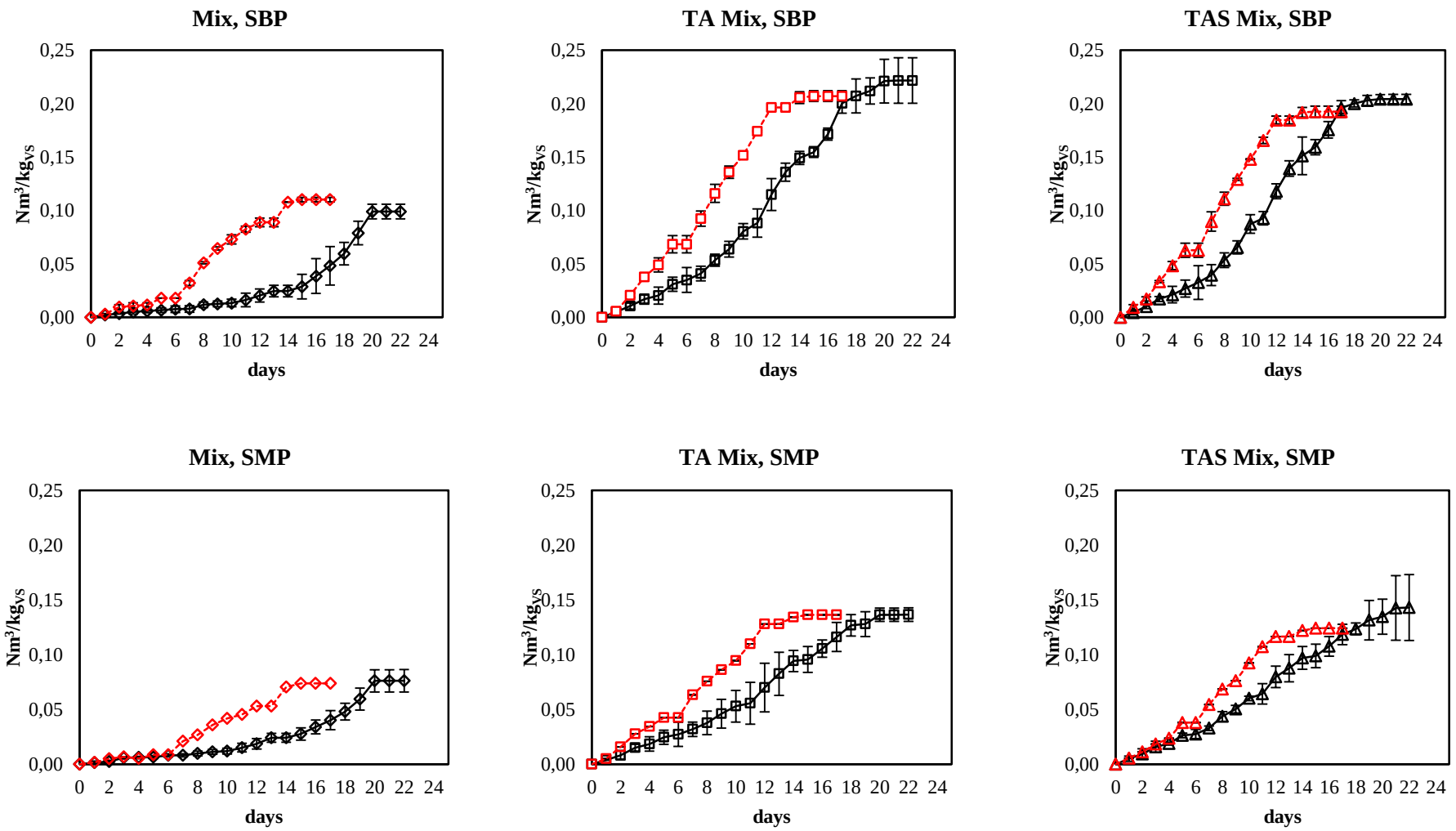
MS, even if with greater lag phase. The fitting of the experimental data through the modified Gompertz model was in all cases satisfactory, achieving coefficient of determination values between 0.93 and 0.99. Comparing the estimated biodegradability of the different waste materials, the highest methane production rate was ascribable, accordingly to the methane potential, in the order sWWTP>RSC>MS; on the contrary, the lag phase length followed the sequence sWWTP>MS>RSC. The highest lag phase estimated for the sWWTP may be due to the already mentioned high concentrations of COD, suspended solids, fats, oils and detergents in the cosmetic industry wastewater (Abidemi et al., 2018) that concentrate in the related waste activated sludge. However, the fact that sWWTP achieved the highest methane potential and production rate values, compared to the other considered samples, demonstrated that this specific waste is anyway biodegradable.

3.3. Results of the anaerobic digestion tests performed on mixed cosmetic wastes

The positive effect of the pre-treatments (DR was 64 % ± 0.01 after TA and 66 % ± 0.01 after TAS) was appreciated from the results of the AD tests involving the Mix (Table 4 and Figure 3). The cumulative biogas/methane production curves showed analogous ultimate yields and different durations of the AD process: 22 days at 35 °C and 17 days at 52 °C, with a faster process in thermophilic conditions, compared to mesophilic, according to literature (among the others, Chi et al., 2010; Ge et al., 2011; Gebreeyessus and Jenicek, 2016).

Table 4. Results of the AD tests performed on the Mix at 35 and 52 °C and after TA and TAS pre-treatments (SBP: specific biogas production; SMP: specific methane production. *only one replicate was available for SMP at 52 °C)

	Mix		TA Mix		TAS Mix	
T (°C)	35	52	35	52	35	52
AD duration (d)	22	17	22	17	22	17
SBP (Nm^3/kg_{VS})	0.10 ± 0.01	0.11 ± 0.002	0.22 ± 0.02	0.21 ± 0.005	0.20 ± 0.004	0.19 ± 0.01
SMP (Nm^3/kg_{VS})	0.08 ± 0.01	0.07*	0.14 ± 0.01	0.14*	0.14 ± 0.03	0.12*
CH_4 (% v/v)	77	67	62	66	70	65
SMP increment (%)	/	/	75	100	75	71



284 **Figure 3.** Results of AD tests performed at 35 °C (black) and 52 °C (red) on mixed cosmetic wastes (Mix) unaltered and after TA and TAS pre-
285 treatments (SBP: specific biogas production; SMP: specific methane production). Only one replicate was available for SMP at 52 °C

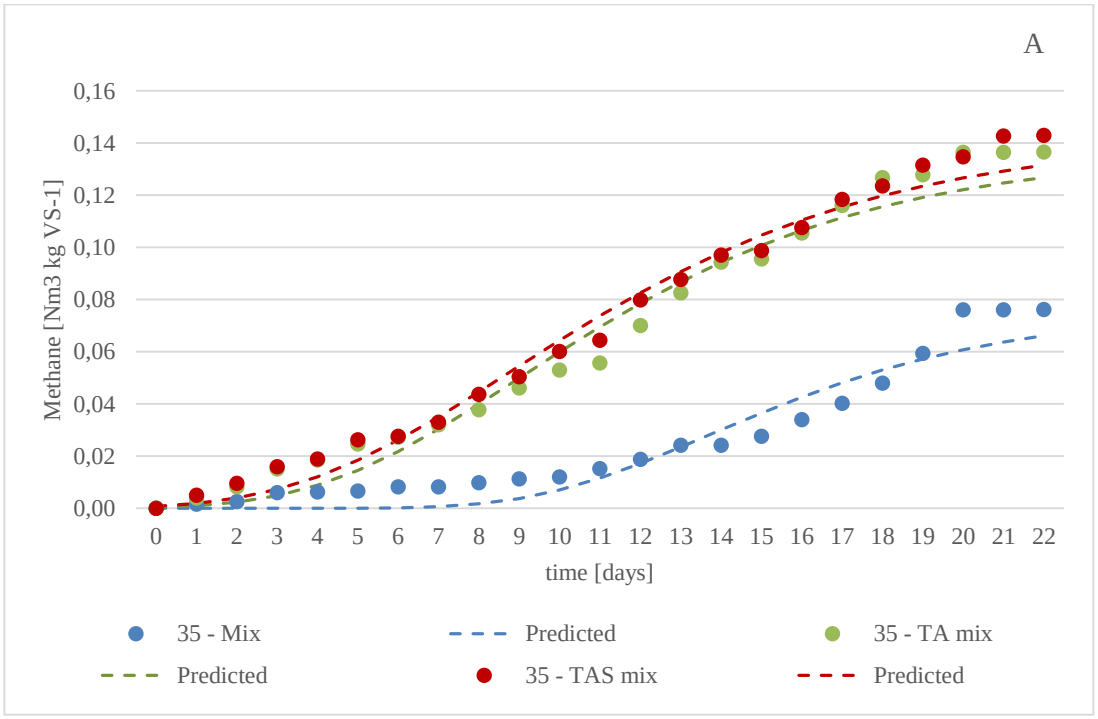
Even if the cosmetic waste materials considered in this work are largely different from any other substrate already studied, a comparison with state-of-the-art literature related to the application of pre-treatments to improve AD of sewage sludge (e.g., the most similar substrate in available literature) could be beneficial anyway. Methane production from sewage sludge resulted improved after thermal (40 %), alkaline (83 %) and ultrasounds (35-95%) pre-treatments (Zhen et al., 2017); industrial active sludge exhibited 90 and 104 % methane production increments after thermal and ultrasound pre-treatments respectively (Carrère et al., 2010).

Table 5. Performance and kinetic parameters of AD at 35 °C and 52 °C of mixed cosmetic wastes (Mix) before and after TA and TAS pre-treatments obtained through the modified Gompertz model (P: methane potential of the substrate; R_{max}: maximum methane production rate; λ: lag phase)

	P ($Nm^3\ kgVS^{-1}$)	R_{max} ($Nm^3\ kgVS^{-1}d^{-1}$)	λ (days)	R^2 (-)
35 °C				
Mix	0.076	0.007	9.4	0.915
TA Mix	0.137	0.010	4.0	0.972
TAS Mix	0.143	0.010	3.5	0.983
52 °C				
Mix	0.074	0.009	4.8	0.975
TA Mix	0.136	0.014	2.1	0.975
TAS Mix	0.124	0.014	2.7	0.981

The performance and kinetic parameters estimated from methane production deriving from the Mix before and after TA and TAS through the modified Gompertz model (Table 5, Figure 4, and Appendix) showed that TA and TAS pre-treatments were able to improve the methane production rate and lag phase. Comparing the pre-treatments, at 35 °C TA Mix exhibited lower methane potential (experimental values) and equal maximum methane production rate values,

304 compared to TAS Mix, and higher lag phase; at 52°C, TA Mix exhibited higher methane
305 potential (experimental values) and equal maximum methane production rate values, compared
306 to TAS Mix, and shorter lag phase. The modified Gompertz model fitting of the experimental
307 data was in all cases satisfactory, achieving coefficient of determination values between 0.92
308 and 0.98.



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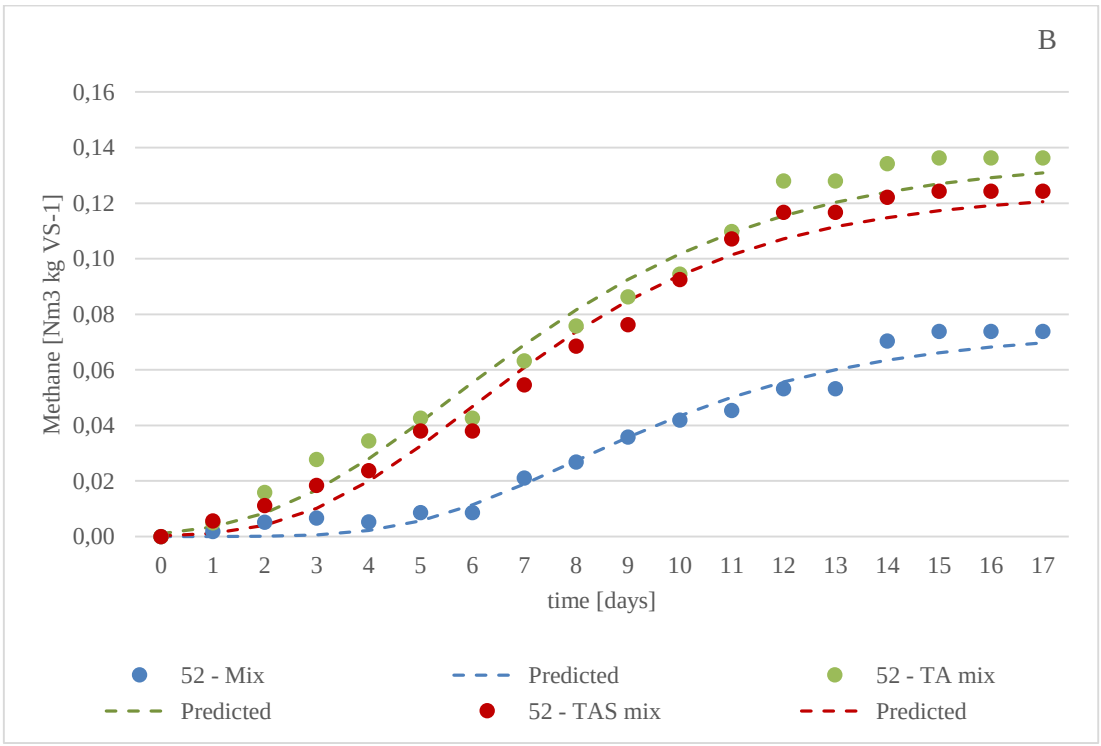

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Figure 4. Results of the modeling of experimental methane production from the mixed cosmetic wastes at 35 (A) and 52 °C (B) through the modified Gompertz model

3.4. Results of digestate characterization

The digestate samples deriving from the AD tests performed on the Mix were characterized with the aim of a preliminary investigation of their thermal valorization. A main critical issue is obviously represented by their high moisture content (95 %-wt), which requires a dehydration treatment before the thermal valorization. The LHV values of the cosmetic waste samples were as follows (values expressed in MJ/kg$_{TS}$): 20.16±1.01 for sWWTP; 25.97± 1.24 for RSC; 25.87±1.30 for MS.

Considering the digestates deriving from the Mix, all gave back LHV values similar to wood (12-19 MJ/kg$_{TS}$) (Green and Perry, 2007). Comparing the LHV values obtained from the Mix after AD at 35 and 52 °C (Figure 5), the thermophilic conditions produced lower LHV values than the mesophilic conditions, in agreement with literature (Gebreeyessus and Jenicek, 2016; Khemkhao et al., 2012).

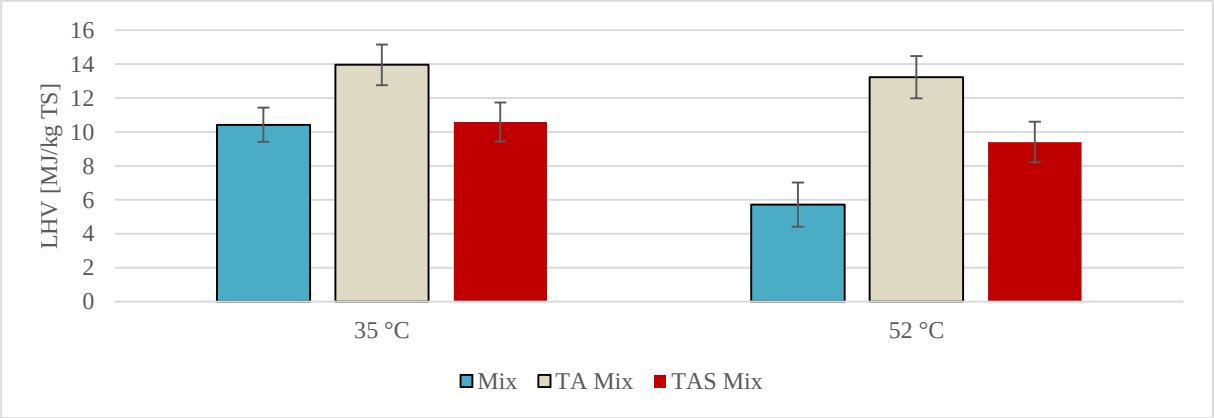


Figure 5. LHV values of digestate samples deriving from the AD tests of the Mix at 35 °C and 52 °C

329 *3.5. Scale-up and economic assessment*

330 The design parameters (Table 6) adopted in the scale-up analysis (section 2.5) considered AD
331 in continuous mode, feeding 5 % dry substance, and achieving the performances reported in
332 section 3.3. The analysis envisioned two scenarios: AD of the Mix "as it was" (S1) and after
333 TA pre-treatment (S2). Two sub-scenarios, based on the amount of available waste mixture,
334 were also considered for S2: the amount of waste produced at the moment of the research (a)
335 and the amount of waste necessary to achieve energy self-sufficiency (b) (section 2.5). The
336 scale-up assessment based on the amount of waste mixture produced in the plant led for
337 scenarios S1 and S2.a to a 64 m^3 digester, with methane production from the pre-treated Mix in
338 scenario S2.a (13.7 Nm^3/d) almost double than without any pre-treatment in scenario S1 (7.8
339 Nm^3/d). Scenario S2.b led to a 113 m^3 digester corresponding to 24.1 Nm^3/d methane
340 production.

341

342 Table 6. Design parameters considered for the scale-up assessment (Mix: waste mixture; TA:
343 thermo-alkaline pre-treatment with NaOH at 50 °C for 120 min; HRT: hydraulic retention time;
344 OLR: organic loading rate; Q: flow rate)

Parameter [measure unit]	S1 (Mix)	S2 (TA Mix)
TA temperature (T_{TA}) [°C]	-	50
dose of NaOH [g/g_{TS}]	0	0.08
temperature (T_{AD}) [°C]	35	35
HRT [days]	22	22
Total solids of the mix [kg/m^3]	140.6	140.6
Volatile solids of the mix [kg/m^3]	117.7	117.7
Total solids of the diluted mix [kg/m^3]	50	50
Volatile solids of the diluted mix [kg/m^3]	41.8	41.8

OLR [kg_{VS} /(m^3 day)]	1.90	1.90	
specific methane yield [Nm^3/kg_{VS}]	0.080	0.140	
Total solids removal [%]	5.25	7.28	
		a	b
Q NaOH [kg/day]	0	9.3	16.5
Q Mix [m^3/d]	0.8	0.8	1.5
Q water [m^3/d]	1.5	1.5	2.7
Q diluted Mix [m^3/d]	2.3	2.3	4.1
methane production (Q_{CH4}) [Nm^3/day]	7.8	13.7	24.1
working volume, 80 % [m^3]	51	51	91
volume of the reactor [m^3]	64	64	113

345

346 The energy analysis (Table 7) showed that in scenarios S1 and S2.a surplus thermal energy was
347 required to heat the digester. In details, considering 303 t/y of waste mixture, TE_{net} was negative,
348 indicating that TE_{CHP} could just partly cover the thermal energy requirements; instead, EE_{net}
349 was positive. The energy surplus needed was larger for the Mix (- 59704 MJ/year) than TA Mix
350 (- 4073 MJ/year). Instead, scenario S2.b was designed to achieve E_{tot} equal to zero, i.e., energy
351 self-sufficiency based on methane production.

352

353 Table 7. Results of energy analysis (Mix: waste mixture; TA: thermo-alkaline pre-treatment
354 with NaOH at 50 °C for 120 min)

	S1 (Mix)	S2 (TA Mix)	
		a	b
Net thermal energy (TE_{net}) [MJ/year]	- 92479	- 67507	-111786
Heating pre-treatment (TE_{pre})	0	- 5689	- 10025

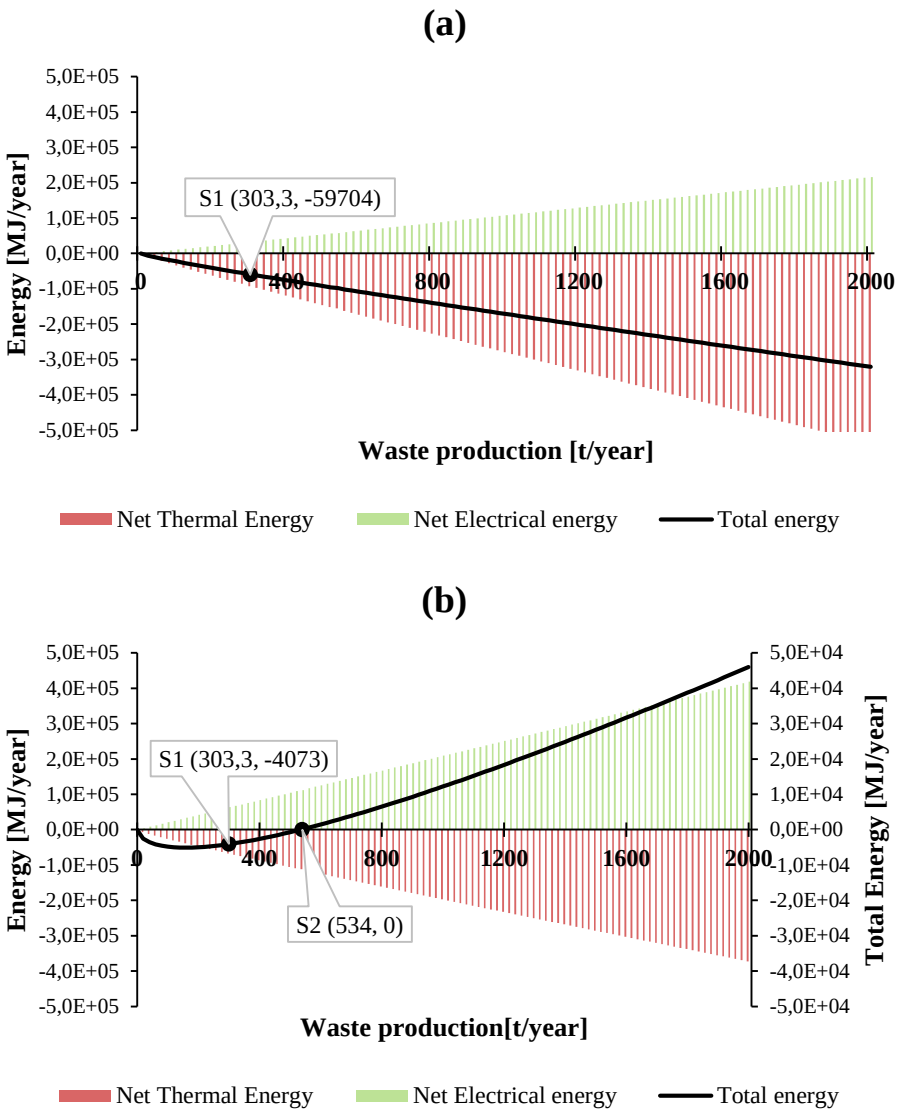
Heating feeding AD (TE_{AD})	- 109693	- 109693	- 193305
Thermal loss AD (TE_{loss})	- 23665	- 23665	- 34527
Thermal energy from CHP (TE_{CHP})	+ 40880	+ 71540	+ 126071
Net electrical energy (EE_{net}) [MJ/year]	+ 32774	+ 63434	+ 111786
Electricity mixing AD (EE_{mix})	- 8106	- 8106	- 14284
Electricity from CHP (EE_{CHP})	+ 40880	+ 71540	+ 126071
Total energy (Etot) [MJ/year]	- 59704	- 4073	0

355

356 Despite the waste production at the moment of the research was not sufficient to reach the
357 energetic self-sustainability of AD at 35 °C, TA pre-treatment could be the key towards the
358 process energetic sustainability by significantly enhancing the specific methane yield (from
359 0.080 Nm^3/kg_{VS} to 0.140 Nm^3/kg_{VS}). In fact, investigating the energy demand as a function of
360 the amount of waste mixture undergoing AD at 35 °C (Figure 6), E_{tot} was negative for any
361 values of waste production (Figure 6a), and E_{tot} even decreased by enhancing the waste
362 production. Therefore, the energetic self-sustainability couldn't be reached for without TA pre-
363 treatment. Instead, in case of the AD of the TA mix at 35 °C (Figure 6b), E_{net} was positive for
364 waste production larger than 534 t/year.

365

366 Figure 6. Total energy, net thermal energy, and net electrical energy as a function of the waste
367 production: (a) AD at 35 °C of the Mix, (b) AD at 35 °C of the TA pre-treated Mix.



368
369

370 The results and details of the economic analysis (Table 8) showed that both the Mix and TA
371 Mix options could be profitable with the current waste production (scenarios S1 and S2.a), with
372 promising NPV and ROI of 108,694 € and 13.1 % for the Mix, and 112,936 € and 13.3 % for
373 TA Mix. In both cases the payback time of 10 years was reasonable. Despite both scenarios
374 didn't reach the energetic sustainability (natural gas was hypothesized as thermal energy
375 source), they showed promising economic performances. This is not surprising, since the major
376 contribution to the total revenues (Table 8) didn't come from the sale of electricity (representing
377 4.5 % of total revenues for the Mix and 7.6 % for TA Mix), but from the savings related to the

disposal of the digestate compared to the current waste management (95.5 % of total revenues for the Mix and 92.4 % for TA Mix). Considering S2.b, the profitability was larger than in other scenarios, with NPV of 270,294 €, ROI of 15.2 % and payback time of 9 years.

Table 8. Results of the economic analysis: operational costs, revenues, total investment costs, return of investment (ROI), net present value (NPV), payback time (PBT).

	S1(Mix)	S2 (TA mi)		Notes	References
		a	b		
Total operational costs [€/year]	9182	9910	11744		
NaOH [€/year]	0	921	1623	NaOH: cost 0.27 €/kg; dose 0.08 g/gTS	Demichelis et al., 2018a
water [€/year]	753	753	1327	water cost: 1.37 €/m^3	Demichelis et al., 2018a
Net thermal energy [€/year]	717	523	866	non-household consumers - natural gas cost average EU-27 (2nd half 2020): 0.0279 €/kWh	Eurostat, 2021
Electricity [€/year]	282	282	498	non-household consumers - electricity cost average EU-27 (2nd half 2020): 0.1254 €/kWh	Eurostat, 2021
Labour [€/year]	7430	7430	7430	labour cost average EU-27: 28.5 €/hour; 1 worker; 1 hours/day; 5 days/week	Eurostat, 2021
Total revenues [€/year]	31633	32701	57626	electricity cost average EU-27 (2nd half 2020): 0.1254 €/kWh	Eurostat, 2021
Save costs for waste disposal [€/year]	30209	30209	53235	waste disposal: sWWTP: 0.15 €/kg; RSC and MS: 0.31 €/kg; FW: 0.110 €/kg; digestate: 0.120 €/kg	Demichelis et al., 2018a

Electricity [€/year]	1424	2492	4391	electricity cost average EU-27 (2nd half 2020): 0.1254 €/kWh	Eurostat, 2021
Total investment cost [€]	171087	171087	301495	FCC: Cost digester construction: 2500 €/m^3 WCC: 6.5 % of FCC	Chiappero et al., 2019
ROI [%]	13.1	13.3	15.2		Chiappero et al., 2019
NPV [€]	108694	112936	270294	lifetime (n): 20 years; discount rate (i): 5 %	Chiappero et al., 2019
PBT [years]	10	10	9		Chiappero et al., 2019

4. Conclusions

The management of the industrial wastes deriving from cosmetic products manufacturing is challenging from the technical and economic viewpoints: wastewater, rich of scarcely biodegradable organic compounds, is typically treated in onsite treatment plants that are highly efficient in COD reduction; other wastes, including the wastewater sludge, are usually landfilled or incinerated. While the available literature on cosmetic waste management only offers few data on the AD of wastewater, this work was aimed at investigating the technical feasibility of the AD of different cosmetic wastes deriving from one of the largest plants of L'Oréal Group in Europe.

A first set of AD tests at 35 °C concerning the single waste materials provided encouraging results (up to 0.10 Nm^3/kg_{vs} of biogas, 70 %-v/v methane). Further AD tests involved a mixture (according to the amounts produced in the plant) of the wastes at 35 and 52 °C. Considering the low ratio between soluble and total COD, the wastes underwent physico-chemical pre-treatments (thermo-alkaline and thermo-alkaline-sonication). The main findings of this research were as follows: pre-treatments considerably increased (up to 100 %) methane yields compared

to untreated mixed wastes, with better performances for the thermo-alkaline pre-treatment; no significant differences were observed comparing the performances of the AD of pre-treated mixed wastes in mesophilic and thermophilic conditions, achieving in all cases biogas yields around 0.2 Nm^3/kg_{VS} and 62-70 % methane; after AD, 5-8 %-wt total solids reductions were observed; even if a dewatering process will be necessary, the thermal valorization of the digestate could be investigated in the future.

On the grounds of the technical and economic assessment of the feasibility of the scale-up of the overall process, TA pre-treatment exhibited a key role in achieving the process energetic sustainability. The economic analysis showed that with the current waste production AD of the considered cosmetic waste could be sustainable. In fact, even if the energetic sustainability wasn't achieved (natural gas was hypothesized as thermal energy source), the major contribution to the total revenues derived from the savings related to the disposal of the digestate compared to the current waste management. In conclusion, considering the lack of literature on the anaerobic digestion of cosmetic wastes, this study would like to provide an insight on the topic and stimulate further exploration of alternative management options for cosmetic wastes.

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and writing-original draft writing and manuscript review; M. Onofrio: conceptualization, methodology and writing-manuscript review.

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