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Abstract: Fast development of centralized agricultural biogas plants leads to high amounts of digestate production. The treatment and disposal of liquid fractions after on-site digestate solid–liquid separation remains problematic due to their high organic, nutrient and aromatic contents. This work aims to study the variability of the remaining compounds in the digestate liquid fractions in relation to substrate origin, process parameters and solid–liquid separation techniques. Twenty-nine digestates from full-scale codigestion biogas plants and one waste activated sludge (WAS) digestate were collected and characterized. This study highlighted the combined effect of the solid–liquid separation process and the anaerobic digestion feedstock on the characteristics of liquid fractions of digestates. Two major clusters were found: (1) liquid fractions from high efficiency separation process equipment (e.g., centrifuge and others with addition of coagulant, flocculent or polymer) and (2) liquid fractions from low efficiency separation processes (e.g., screw press, vibrating screen and rotary drum), in this latter case, the concentration of chemical oxygen demand (COD) was associated with the proportion of cow manure and energy crops at biogas plant input. Finally, $SUVA_{254}$, an indicator for aromatic molecule content and the stabilization of organic matter, was associated with the hydraulic retention time (HRT).

Keywords: anaerobic digestion; solid waste; organic compound; solid–liquid separation; liquid phase; digestate treatment

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1. Introduction

The development of biogas plants from agricultural waste in Europe was particularly due to its energy policy to implement Clean Energy Package including Renewable Energy Directive. This policy aims to achieve a 32% share of renewable energy from total energy consumption by the year 2030 [1–3]. In consequence, this leads to a huge production of biogas plant byproducts, digestate, a renewable resource [4] which requires post-treatment for nutrient recovery to meet the latest European Union regulation proposal on fertilizers [3,5].

The most common current practice of digestate post-treatment is by volume reduction through mechanical solid–liquid separation [6,7]; producing 80–92% of liquid fraction in terms of mass; common separators on sites are the screw press, centrifuge, vibrating screen or rotary drum [8–10].

Solid fractions of digestates which contain more P are generally utilized for land application as fertilizer [6,11,12]. On the other hand, liquid fractions of digestates still contain high residual of organic compounds with the concentration of total chemical oxygen demand (COD) from 9.2 to 78 g/L; where 60 to 96% of COD are in the form of suspended particles (>1.2 μm) while the remaining are in the form of colloids (1.2 μm to 1 kDa) and dissolved matter (<1 kDa), representing 2–27% and 2–18%, respectively [13]. Owing to its poor aerobic biodegradability characteristics with high humic substance content [13], aerobic post-treatment for liquid fractions of digestates is far from feasible. Besides, the liquid fraction contains high amounts of nutrients such as; total nitrogen (TN) (1.5 to 6.5 g/L), 0.5 to 3.5 g/L for ammoniacal nitrogen (NH_4^+ and NH_3), 1.05 to 5.48 g/L for potassium (K^+) and 0 to 2.13 g/L for phosphates (PO_4^{3-})) [6,12–15] which exhibit a fertilizing potential for crops [16].

Currently, new technologies for processing liquid fractions of digestates are still being explored [17]. One of the possibilities is through nutrient recovery such as struvite (STR) precipitation [18] and ammonia stripping (to produce ammonium sulphate (AS)) [6,19–21], combined ozone treatment and ultrafiltration [22], combined system with aerobic granular sludge batch reactors and ultrafiltration [23], or utilizing fly ash as a chemical precipitant [24].

Besides, high nutrient contents mean that the liquid fractions of digestates able to be reused for microalgae cultivation for biomass [25,26] or as biomass for fertilizer [27], recycling nutrients back to digesters, soil application and subsurface injection into soils [6,28].

The appropriate post-treatments for either solid fractions or liquid fractions of digestates are very crucial for any future biogas plant that integrates part of the circular bioeconomy [17,29,30]. The aim of the circular economy is to influence material and energy flows in order to maximize environmental benefits whilst avoiding costs (grow–make—se–restore) [31]; which is currently one of the main priorities of the European Union as described in detail by Molina-Moreno et al. [32], Muradin et al. [33] and Vilardi et al. [34]. However, one type of full-scale post-treatment could not be applied to all liquid fractions of digestates mainly because the composition in organics, nutrients and aromatic compounds can strongly vary from one liquid fraction of digestate to another.

The primary aim of the research is to understand the variability of the remaining compounds in the liquid fractions of digestates specifically produced at full-scale codigestion plants in relation with substrates origin, operating conditions of the digester and types of solid–liquid separation. For the first time, a substantial number of liquid fractions (29) were sampled from full-scale anaerobic codigestion plants treating agricultural wastes and then deeply characterized. A single liquid fraction of digestate from a common anaerobic digestion plant treating waste activated sludge (WAS) was also collected as a benchmark for agricultural codigestion plants.

2. Materials and Methods

2.1. Digestate Collection and Storage

Digestates (raw digestate, solid and liquid fractions of digestates after separation) were taken from 30 full-scale anaerobic digestion plants. Eleven samples were already described in a previous paper [13]; see the plant reference marked with an asterisk in Table 1. Two liters of each raw digestate and solid fraction of digestate, respectively, and 4 liters of liquid fraction of digestate were collected from each plant for this study. In this investigation, raw digestate and solid fraction of digestate were collected for analyses of total solids (TS) and volatile solids (VS) concentrations in order to gain information on solid–liquid separation efficiency performed on-site. All samples were stored in a cold room at 4 °C for later use.

Table 1. Feedstock compositions, process parameters (temperature range, type of reactor, size of reactor, size of post reactor, feeding, retention time), methane production and types of solid–liquid separation.

Plants	**Substrates Composition (% of Each Category Presented in Table A1)**								**Temperature Range**	**Type of Reactor**	**Reactor Volume (m³)**	**Post Reactor Volume (m³)**	**Feeding (Tonnes/d)**	**Retention Time (Days)**	**Methane Production (m³/d)**	**Solid–liquid Separation**
	SS	Mnr	EnCr	CrR	Cer	FOG	AFW	Other								
A *	36	16		7		22	19		M	CSTR	2800	n.a	120	n.a	n.a	Screw press
B *		81.6			10.2	8.2			M	CSTR	1370	n.a	15	60	1550	Screw press
C *							100		M	CSTR	450	450	16.6	24	558	Vibrating screen
E *	40					30	30		M	CSTR	3300	1800	90	37	4500	Centrifuge
F *		59.7		18.5	10.9		10.9		M	CSTR	1206	n.a	15	63	1230	Screw press
H *	50	28				22			M	CSTR	930	n.a	30	31	1550	Centrifuge
I *							100		T	PF	3150	n.a	100	30	10,000	Screw press with coagulant + centrifuge
J *		5	95						T	PF	1200	n.a	35	35	3500	Screw press with coagulant
K *	38.5		20		12.5	25	3.5	0.5	M	CSTR	2800	1360	80	30–35	n.a	Screw press
L *	5	64	5	5	9		11	1	M	CSTR	2350	n.a	55	46	1450	Screw press
M *		50	50						M	CSTR	400	400	10	80	550	Vibrating screen
N	44.2	30.8	4.4		1.9	12.8	6		M	CSTR	1500	650	15.8	95 + 41	691	Screw press
O		75				8	17		M	CSTR	2 × 7500	2 × 3500	290	52	17,085	Centrifuge with flocculant
P	5	75		10		5		5	M	CSTR	1000	1000	30.5	65	n.a	Screw press
Q		76.5	16.6	0.85	0.85		5.2		M	CSTR	3900	3900	30	n.a	3915	Screw press
R		53.6	10.7	10.7	10.7		14.3		M	CSTR	2300	n.a	28	85	1450	Centrifuge
S	50	28				22			M	CSTR	920	640	30–35	26 + 18	n.a	Centrifuge
T		48	12			40			M	CSTR	2600	n.a	34.2	50	2381	Rotary drum. Solid fraction was dried
U		55.5	42.1		2.4				40–45	CSTR	2 × 718.5	682	2 × 10.6	(2 × 68) + 32.5	n.a	Screw press
V		36.2	56.9		6.9				T	CSTR	2 × 1500	3000	29	37	n.a	Screw press
W			87.5		12.5				T	CSTR	10,000	n.a	70–100	100–120	6240	Screw press
X			100						40–41	CSTR	2400	n.a	27–30	70–80	n.a	Screw press
Y					4.7	25.7	69.6		M	CSTR	3400	1600	57 + 35 recirculation	37	12,400	Screw press
Z		82	13		5				M	CSTR	1200	1200	31.3	66	1418	Screw press
I2							100		T	PF	3150	n.a	100–150	20–30	5500–8500	Screw press with flocculant + centrifuge
AA		60.1	17.9		6.2	15.8			M	CSTR	1300	n.a	65.9	45	2300	Screw press
AB		33	20			20	27		M	CSTR	2900	n.a	50	57	3090	Screw press
AC		100							M	CSTR	500	n.a	7–8	40	n.a	Centrifuge

AD	48		4	48	M	CSTR	1500	3000	75	20 + 40	2790	Filter press (150 plates) + inorganic coagulant + polymer. Solid fraction was later dried
G	100				M	CSTR	10,000	n.a	19.1	20	5583	Centrifuge with addition of polymer

* Samples were described in previous comprehensive characterization by Akhiar et al. [13]. n.a = information not available. SS: sewage sludge, Mnr: manure, EnCr: energy crops, CrR: crop residues, FOG: fats, oil and grease, AFW: agro-food waste. CSTR: continuous stirred-tank reactor, PF: plug flow. T: thermophilic, M: mesophilic.

2.2. Categorization of Substrates

The different substrates used to feed the different digesters were distributed into 7 main categories: sewage sludge (SS), manure (Mnr), energy crops (EnCr), crop residues (CrR), cereal residues (Cer), fats, oil and grease (FOG) and agro-food waste (AFW) (Table 1). The different types of substrates used to feed the digesters based on the selected categories are described in Table A1.

2.3. Operating Conditions of the Anaerobic Plants

Details on substrate composition, operating parameters and solid–liquid separation of the different plants are presented in Table 1. In this study, digestates (raw, solid and liquid fractions) from an ordinary anaerobic digestion plant fed with only Waste Activated Sludge (WAS) (Plant G) were also collected in order to compare with samples from codigestion plants.

2.4. Filtration and Size Fractionation of Liquid Fractions of Digestates

Dilution with Milli-Q® water was initially performed on each respective liquid fraction of digestate to ease filtration. Dilution factor from 0 to 1/20 was considered in order to have a final COD concentration ranged between 1 to 5 g/L. Filtration at size 1.2 μm and 1 kDa performed later on each respective liquid fraction of digestate enables us to have four fractionation sizes representing: raw liquid (without any filtration), suspended particles (size > 1.2 μm), colloids (size 1.2 μm^{-1} kDa) and dissolved matter (size < 1 kDa) [35].

2.5. Analytical Methods (Chemical, Physical and Biological)

The following analytical methods were similar to procedures performed (where detailed description can be found) in previous work by Akhiar et al. [13].

For chemical methods, a WTW series inoLab pH720 probe was used for pH measurement. Calibration with pH 4 and pH 7 buffer solutions was mandatory prior to use. For alkalinity, 0.1 N hydrochloric acid was used for titration to reach pH 4.3 as described elsewhere [36]. Total solids (TS), volatile solids (VS) and mineral solids (MS) analyses were performed according to standard methods described elsewhere [36]. Commercial Aqualytic™ 420721 COD Vario Tube Test MR 0–1500 mg/L (Aqualytic, Dortmund, Germany) was used to measure Chemical Oxygen Demand (COD). Buchi AutoKjeldahl Unit K-370 (Büchi AG, Flawil, Switzerland) was used for Ammonium (NH_4^+) and Total Kjeldahl Nitrogen (TKN) measurements. For TKN measurement only, premineralization with BUCHI Digest Automat K-438 (Büchi AG, Flawil, Switzerland) was required. Shimadzu TOC-VCSN Total Organic Carbon Analyzer (Shimadzu Corporation, Kyoto, Japan) —equipped with Shimadzu ASI-V auto sampler— was utilized for Total Organic Carbon (TOC) and Inorganic Carbon (IC) measurement [37]. Ion chromatograph, ICS 3000 (Dionex, Sunnyvale, CA, USA) was used to measure cations and anions [38].

UV-2501PC UV–vis spectrophotometer (Shimadzu Corporation, Kyoto, Japan) was used to measure absorbance spectra [37]. Specific Ultraviolet Absorbance at 254 nm ($SUVA_{254}$) was calculated by dividing specific UV absorbance at wavelength 254 nm with dissolved total organic carbon (A_{254}/TOC). $SUVA_{254}$ indicates the content of aromatic carbon in dissolved organic matter and humification degree as well as linked to biological degradability [39]. Perkin Elmer LS55 fluorescence spectrometer (PerkinElmer, Waltham, MA, USA) was used for 3D fluorescence spectroscopy analysis. Fluorescence spatialization integration for spectra interpretation and quantification was according to: (1) protein-like (Tyrosine, Tyrptophane, microbial products); (2) fulvic acid-like; (3) glycolated protein-like; (4) melanoidin-like; lignocellulose-like; (5) Humic acid-like [40].

For physical methods, Beckman Coulter LS200 granulometer (Beckman Coulter, Pasadena, CA, USA) was utilized for the measurement of particle size distribution in the size range between 0 to 2000 μm [41]. HACH portable turbidimeter model 2100P (Hach, Loveland, CO, USA) precalibrated with formazin was used to measure turbidity. WTW multi 3410 digital multi parameter meter TretraCon® 925 probe (Xylem, Rye Brook, NY, USA) with was used for conductivity measurement at a fixed reference temperature of 25 °C. For biological method, WTW Oxitop® control system (Xylem, Rye Brook, NY, USA) was used for determination of Biochemical Oxygen Demand after 5 days (BOD_5) and 21 days (BOD_{21}) [42,43].

Capillary Suction Time (CST) which measures filterability and conditionability of a given liquid sample containing suspended and colloidal particles was conducted using Type 304B CST timer (Tritonel, Strmec, Croatia) equipped with funnel (18 mm diameter) and filter papers (basis weight of 440 g/m2, size 7 × 9 cm, thickness of 0.92 mm, tensile strength of 4525 m/d g/15 mm, porosity of 9 s/100 mL/sqin) purchased from Triton Electronics Ltd. (Dunmow, UK). Each respective liquid fraction of digestate was prediluted to same TS concentration of 10 g TS/kg and only 2 mL of diluted sample were used for each analysis.

The analytical results from chemical, physical and biological analyses of 18 samples combined with another 11 samples (samples A, B, C, E, F, H, I, J, K, L, M) from Akhiar et al. [13] and 1 sample from WAS (sample G) are displayed in Appendix A (Tables A2–A4). All the analytical results were used for statistical analysis in this study.

2.6. Determination of Solid–liquid Separation Efficiency

The separation efficiency indicates the removal efficiency (R) of a particular compound from a slurry to the solid fraction. The calculation for separation efficiency or removal efficiency (R) by solid–liquid separation techniques was made using Equation (1) below [44].

$$R = 1 - \frac{[TS]liq}{[TS]raw} \tag{1}$$

where $[TS]_{liq}$ = total solids concentration in liquid fraction of digestate and $[TS]_{raw}$ = total solids concentration in raw digestate.

2.7. Statistical Analysis

The classification of the parameters analyzed on liquid fractions of digestates from 29 codigestion plants and 1 WAS plant was performed via Principal component analysis (PCA), hierarchical cluster analysis (HCA) and correlation matrix using R version 3.3.2 (31 December 2016) [45]. PCA was carried out in center-scaled variables using function 'FactoMineR' package version 1.35 [46] with PCA plots package 'factoextra' version 1.0.4. For HCA, 'stats' package version 3.3.2 ('hclust' function) was applied to center-scaled variables and Euclidean distances. The clustering algorithm was referring to Ward [47] and the resulting dendrogram was plotted using function 'dendextend' package version 1.4.0. Meanwhile, correlation matrices were constructed using 'rcorr' function with the Pearson's correlation method ('rcorr' is a function of the 'Hmisc' package (version 4.0.2)).

3. Results and Discussion

All results from chemical, physical and biological analyses performed on liquid fractions of digestates are presented in Tables A2–A4. The feedstocks used, the operating parameters (types of reactor, temperature, loading rate, hydraulic retention time (HRT), methane production) and types of solid–liquid separation equipment used are presented in Table 1. The chemical, physical and biological characteristics of the liquid fractions of digestates analyzed (based on the following fractions: raw liquid, suspended particles, colloids and dissolved matter) were included in the PCA, HCA and correlation matrix.

3.1. Correlation between Parameters

Only significant correlations between parameters are shown in Table 2 (p-value < 0.01). In relation to feedstock composition, TKN in colloids was clearly observed to correlate with sewage sludge in the feeding. Indeed, sewage sludge contains very high TN based on dry matter basis due to low total solids content of the sludge after efficient centrifugation. In a study by Oliveira et al. [48], high correlations between nitrogen content in the digestate and both sludge composition or conditioning parameters were reported. Similar to this study, it was observed that as the sewage sludge proportion increased at the feed, higher colloidal TKN in liquid fractions of the digestates was observed. In comparison, none of the other feedstock categories have shown high correlation with the characteristics of liquid fractions of digestates. This remark may be supported by the high uncertainty of the quantities reported from the full-scale plants, the lack of detailed information such as VS quantities in the feeding (instead of total mass) but also by the selection of categories that might not be specific enough (i.e., there is a high variation of quality within feedstocks of the same category). Moreover, several studies reported high variabilities of digestate from the same plant over time but also that anaerobic digestion acts as a buffer for feedstock variation, producing digestates with less quality variability than inputs [49,50]. Both effects would tend to reduce correlation observation based on single samples from different plants.

Table 2. Summary of correlations (p-value < 0.01).

Parameters	Unit	Strong (Anti-)Correlation		Moderate (Anti-)Correlation	
		$\|r\| > 0.7$	r	$0.5 < \|r\| < 0.7$	r
Sewage Sludge [a]	w/w	TKN colloids	0.74	Alkalinity	0.5
				IC	0.51
				TOC dissolved	0.54
				TKN total	0.52
				NH_4^+	0.52
EnCr [a]	w/w			VS/TS liquid	0.51
				MS/TS liquid	−0.51
				$SUVA_{254}$	0.67
Cer [a]	w/w			CST	0.67
AFW [a]	w/w	MS/TS raw	0.70	VS/TS liquid	−0.52
		VS/TS raw	−0.70	MS/TS liquid	0.52
Load	t/day/m³ reactor			AFW	0.54
HRT	Days	$SUVA_{254}$	0.72	CST	0.64
				TOC dissolved	−0.5
				Turbidity	0.5
				C/N	0.55
Methane production	m³ CH_4/ton fed			TKN colloids	0.67
VS/TS raw digestate	w/w			VS/TS liquid	0.63
				MS/TS liquid	−0.63
				COD total	0.57
				COD suspended	0.51
MS/TS raw digestate	w/w	VS/TS raw	−1	VS/TS liquid	−0.63
				MS/TS liquid	0.63
				COD total	−0.57
				COD suspended	−0.51
VS/TS solid fraction	w/w	MS/TS solids	−0.99	MS/TS raw	−0.53
		Separ. Efficiency	−0.72	VS/TS raw	0.53
				COD suspended	0.51
				COD dissolved	−0.51
				Turbidity	0.64
				C/N	0.5
				Na^+	−0.60
MS/TS solid fraction	w/w	Separ. Efficiency	0.74	MS/TS raw	0.5
				VS/TS raw	−0.5
				COD dissolved	0.52

				Turbidity	−0.63
				Turbidity	−0.63
				Na^+	0.60
VS/TS liquid fraction	w/w	MS/TS liquid	−1	CST	0.57
		COD total	0.79	Turbidity	0.61
		COD suspended	0.74	C/N	0.57
				N organic dissolved	−0.51
				Na^+	−0.63
				Cl^-	−0.56
				Conductivity	−0.5
				$SUVA_{254}$	0.56
MS/TS liquid fraction	w/w	COD total	−0.79	CST	−0.57
		COD suspended	−0.74	Turbidity	−0.61
				C/N	−0.57
				N organic dissolved	0.51
				Na^+	0.63
				Cl^-	0.56
				Conductivity	0.5
				$SUVA_{254}$	−0.56
Separation efficiency	w/w	COD dissolved	0.77	CST	−0.60
		Turbidity	−0.70	Alkalinity	0.64
		TKN dissolved	0.70	IC	0.68
		Conductivity	0.72	TOC dissolved	0.61
		NH_4^+	0.7	C/N	−0.55
				TKN total	0.69
				TKN colloids	0.65
				N organic dissolved	0.57
CST	Seconds			COD suspended	0.56
				Turbidity	0.60
				Conductivity	−0.56
				$SUVA_{254}$	0.68
pH	-			COD suspended	−0.53
				C/N	−0.51
				Na^+	0.57
				Cl^-	0.63
				Glycolated-like	0.53
				Humic acid-like	0.54
Alkalinity	$gCaCO_3/gTS$	IC	0.97		
		TOC dissolved	0.71		
		C/N	−0.71		
		TKN total	0.97		
		TKN suspended	0.81		
		TKN colloids	0.71		
		TKN dissolved	0.94		
		N organic dissolved	0.8		
		NH_4^+	0.93		
		Conductivity	0.91		
IC	gC/gTS	TOC dissolved	0.79	COD dissolved	0.55
		C/N	−0.75	Turbidity	−0.5
		TKN total	0.97	$SUVA_{254}$	0.51
		TKN suspended	0.74		
		TKN colloids	0.73		
		TKN dissolved	0.95		
		N organic dissolved	0.79		
		NH_4^+	0.94		
		Conductivity	0.94		
Conductivity	(mS/cm)/(gTS/kg)			$SUVA_{254}$	−0.5
TOC dissolved	gC/gTS			COD dissolved	0.52
				C/N	−0.61
				TKN total	0.67
				TKN suspended	0.62

				TKN colloids	0.66
				TKN dissolved	0.62
				N organic dissolved	0.52
				NH_4^+	0.61
				PO_4^{3-}	0.61
				Conductivity	0.66
				$SUVA_{254}$	−0.65
COD total	gO_2/gTS	COD suspended	0.93	Turbidity	0.57
				Na^+	−0.64
				Cl^-	−0.67
				BOD_{21}	0.60
COD suspended	gO_2/gTS	Turbidity	0.76	C/N	0.52
		Cl^-	−0.73	Na^+	−0.66
				Conductivity	−0.57
				BOD_{21}	0.5
COD colloids	gO_2/gTS			COD dissolved	0.66
COD dissolved	gO_2/gTS			Turbidity	−0.57
				C/N	−0.53
				TKN total	0.52
				TKN dissolved	0.53
				N organic dissolved	0.51
				NH_4^+	0.5
				K^+	0.51
				Conductivity	0.57
Turbidity	NTU/(gTS/kg)			C/N	0.57
				TKN total	−0.5
				TKN dissolved	−0.55
				N organic dissolved	−0.55
				NH_4^+	−0.53
				Na^+	−0.62
				Cl^-	−0.69
				Conductivity	−0.64
C/N	-	TKN total	−0.74	TKN suspended	−0.58
		TKN dissolved	−0.73	TC/TN dissolved	0.54
		NH_4^+	−0.71	N organic dissolved	−0.67
		Conductivity	−0.78	Na^+	−0.55
				$SUVA_{254}$	0.54
TKN suspended	gN/gTS			TKN colloids	0.52
				TKN dissolved	0.62
				N organic dissolved	0.62
				NH_4^+	0.59
				Conductivity	0.62
TKN total	gN/gTS	TKN suspended	0.73		
		TKN colloids	0.76		
		TKN dissolved	0.98		
		N organic nitrogen	0.81		
		NH_4^+	0.97		
		Conductivity	0.97		
TKN colloids	gN/gTS	NH_4^+	0.74	TKN dissolved	0.69
		Conductivity	0.71		
TKN dissolved	gN/gTS	N organic dissolved	0.83	TC/TN dissolved	−0.51
		NH_4^+	0.99	Cl^-	0.5
		Conductivity	0.97		
TC/TN dissolved	-			NH_4^+	−0.51
				Protein-like	0.56
				Fulvic acid-like	−0.58
N organic dissolved	gN/gTS	NH_4^+	0.74	Na^+	0.65
		Conductivity	0.84	K^+	0.51
				Cl^-	0.55
NH_4^+	gN/gTS	Conductivity	0.96		
Na^+	gNa/gTS			Cl^-	0.67

				PO_4^{3-}	0.55
				Conductivity	0.55
				Humic acid-like	0.54
Cl^-	gCl/gTS			Conductivity	0.58
				Protein-like	−0.61
				Glycolated-like	0.59
Mean Size	μm	Median size	0.89		
BOD_5	gO_2/TS	BOD_{21}	0.85		
Protein-like	-	Fulvic acid-like	−0.88		
		Glycolated-like	−0.84		
		Melanoidin-like	−0.72		
Glycolated-like	-	Melanoidin-like	0.71	Humic acid-like	0.5
Fulvic acid-like	-			Glycolated-like	0.5

a. AD feedstock proportion.

An anticorrelation was observed between residues of AFW and VS/TS in the raw digestates. This observation may possibly be justified by the characteristics of these feedstocks which are highly biodegradable. This leads to a lower organic matter content (VS/TS) after anaerobic digestion.

The correlation matrix highlights several high correlations between anaerobic process parameters. Specifically, HRT was observed to have a positive correlation with $SUVA_{254}$. This signifies that higher HRT used will result to a higher humification ratio. This statement will be further discussed in detail in Section 3.4.

From the observation of strong (anti-) correlations between characterization parameters ($|r| > 0.7$), VS/TS on solid fraction was anticorrelated with separation efficiency which can be linked to the fact that low performance separators are applied mostly to digestates with a high content of fibers that present a higher VS/TS ratio.

For liquid fractions of digestates, VS/TS was correlated to total and suspended COD in liquid fractions of digestates. Total COD was correlated with suspended COD which confirmed the finding by Akhiar et al. [13] that 60–96% of COD in liquid fractions of digestates are mainly in the form of suspended particles (>1.2 μm). Meanwhile, suspended COD was correlated with turbidity and anticorrelated with Cl. Separation efficiency was also observed to be correlated with dissolved COD, dissolved TKN, conductivity, NH_4^+ while anticorrelated with turbidity. This result seems coherent as higher efficiency of solid–liquid separation should tend to remove COD as suspended solids reducing turbidity, while increasing the concentration of soluble compounds. Furthermore, the utilization of coagulants and polymers in several separation techniques led to a high correlation relating separation efficiency with conductivity, with a slightly lower correlation observed between separation efficiency with alkalinity. Some commonly used coagulants are metallic salts, for instance ferric chloride or aluminum sulfate, which react with bicarbonate in order to form metallic hydroxides ($Fe(OH)_3$, $Al(OH)_3$) [9].

Other high correlations observed were between alkalinity and IC, dissolved TOC, TKN (total, suspended, colloids and dissolved), dissolved organic N, NH_4^+ and conductivity. It seems a trivial correlation as ICs consist of a major part of alkalinity (carbonates) as well as ammoniacal nitrogen, which is greatly responsible for digestate buffering capacity. Meanwhile, alkalinity was anticorrelated with C/N in liquid fractions of digestates, possibly because of high ammoniacal nitrogen contributing to alkalinity and, thus, to low C/N. Besides, the correlations between all nitrogen measurements (TKN, dissolved organic N, NH_4^+, C/N) were also observed, together with their correlation with conductivity.

BOD_5 is positively correlated with BOD_{21} ($r = 0.85$). Indeed, BOD_{21} comprises the BOD_5 parameter; which justifies the relation between these parameters. In this study, the mean value of BOD_5/BOD_{21} obtained was 0.43 ± 0.12. Notably, this value is much lower than the usual ratio of BOD_5/BOD_{21} from 0.6 to 0.9 observed for raw sewage [51]; BOD_5/BOD_{21} could be a relevant parameter for digestate characterization.

In the range of moderate correlation coefficients (0.5 < |r| < 0.7), several correlations between parameters were identified. In relation to feedstock composition, cereal residues fraction (Cer) was shown to link to CST in consequence to small particles in liquid fractions of digestates, while energy crop residues (EnCr) appeared to be interconnected to $SUVA_{254}$ as an aromatic content indicator in the digestates. The lignin content of the energy crop residue is generally discussed in the literature; explaining the low methane potential of these compounds. $SUVA_{254}$ and CST were found to be parameters which validate the organic matter residual content in digestates. Hermann et al. [52] and Dandikas et al. [53] investigated various crop silages and grassland, respectively; both have concluded that the $SUVA_{254}$ increase after anaerobic digestion process with limited biomethane potential (BMP) is due to presence of lignin found in these feedstocks.

In this study, some parameters were more signified for correlation with various parameters. Turbidity, conductivity, $SUVA_{254}$ and CST were the utmost common parameters which also justified the main part of digestates' characteristics. From a practical perspective, apart from $SUVA_{254}$, these measurements are simple to conduct with shorter time required to obtain results of the liquid fractions of digestates' characteristics (e.g., conductivity measurement to obtain separation efficiency, alkalinity, IC, C/N, TKN total, TKN colloids, TKN dissolved, N organic dissolved and NH_4^+ of liquid fractions of digestates).

3.2. Multivariate Analysis via Principal Component Analysis (PCA): Impact of Solid–Liquid Separation Techniques

A PCA was carried out including on all the 42 variables (center-scaled) of all the 30 digestates as shown in Figure 1. The first PCA component describes 32% of the variance while the second component describes 16%. Considering the high number (42) of very diverse variables included in the analysis, a description of almost 50 % of total variance with only two components highlights the power of PCA.

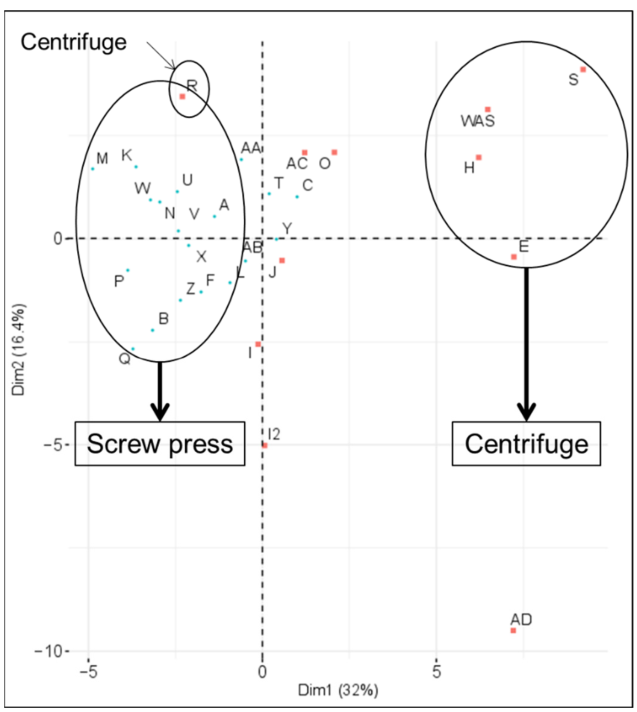


Figure 1. PCA Individuals: Impact of solid–liquid separation.

The PCA in Figure 1 shows that the liquid fractions of digestates could be categorized by the types of solid–liquid separation techniques applied. Sequentially, calculation on separation efficiency or removal efficiency (R) based on Equation (1) allowed us to evaluate the influence of solid–liquid separators and the results are presented in Figure 2.

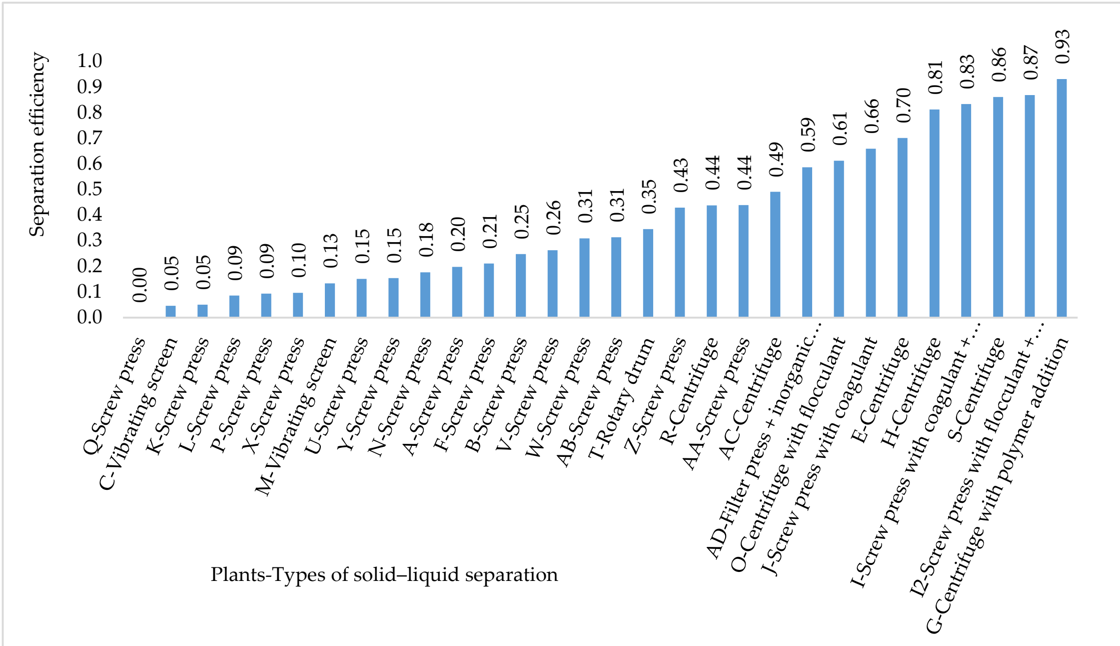


Figure 2. Separation efficiency vs. types of solid–liquid separators used for plants.

Remarkably, the screw press, vibrating screen and rotary drum were classified in the group of solid–liquid separation with low separation efficiency (with values ≤44%). In contrast, the centrifuge and other types of solid–liquid separators with the addition of either coagulants, flocculents or polymers were classified as high efficiency separators with 44 to 93% separation efficiency. This study confirmed the low separation efficiencies of the screw press (<30% efficiencies) compared to the centrifuge (from 33 to 69% efficiencies) obtained previously in a study by Moller [54]. A meta-analysis study with over 60 full-scale separators resulted in a similar observation where, based on the same indicator, two efficiency groups could be observed and similarly linked to feedstock [5].

Even though digestate R was separated by centrifugation, it had a low separation efficiency of 44% only and, hence, it belonged to the group with low efficiency separation. This may possibly be owing to the inefficient centrifuge applied for digestate separation.

The liquid fractions of digestates I, I2 and J from high efficiency separators, each with 83, 87 and 66% separation efficiency, respectively (Figure 2), were, however, near to low efficiency solid–liquid separation group (Figure 1). This may possibly be due to the fact that I, I2 and J were originated from T-PF anaerobic digesters operated at high total solid content (dry-AD) where the organic matter was not completely digested during the process. As a result, the amounts of undigested organic matter remained high although an efficient solid–liquid separator was used, resulting in liquid fractions of digestates with high VS/TS, COD total/TS, COD suspended/TS and COD colloids/TS.

In contrast, T (originated from pig slurry, corn silage and fats), C (originated from fruits and vegetable waste) and Y (originated from biowaste, cereals and fats) from low efficiency solid–liquid separators (separation efficiency of 35, 5 and 15%, respectively) (Figure 2), however, were near to the cluster of solid–liquid separators with high performance, as shown in PCA in Figure 1. This may possibly be explained by the origins of the easily biodegradable substrates, resulting in lower residual organic matters in the liquid fractions of digestates.

The loading scores of measured parameters of Dimension 1 of the PCA is shown in Figure 3. Positive values were correlated to high efficiency separation, which is correlated to dissolved inorganic, alkalinity, ions with significant parameters of conductivity, TKN dissolved, TKN total, IC, NH_4^+, dissolved organic nitrogen and alkalinity with value >0.8. On the contrary, negative values were correlated to low efficiency which is correlated to organic matter and solids. The significant parameters observed were dissolved C/N, turbidity, COD suspended, VS liquid and CST with values <−0.6. A meta-analysis study on digestate quality with a database containing about 150 raw digestates, solid and liquid fractions resulted in a very similar observation [8]. This result can be associated with the fact that low efficiency separators such as screw presses are mainly applied to fibrous inputs which are mostly poorer in N content while results in digestates with greater recalcitrant organic matter (higher C content). At the same time, high performance separation equipment such as centrifuges are widely applied to non-fibrous inputs such as pig slurry and biowaste which are commonly N-rich and more biodegradable.

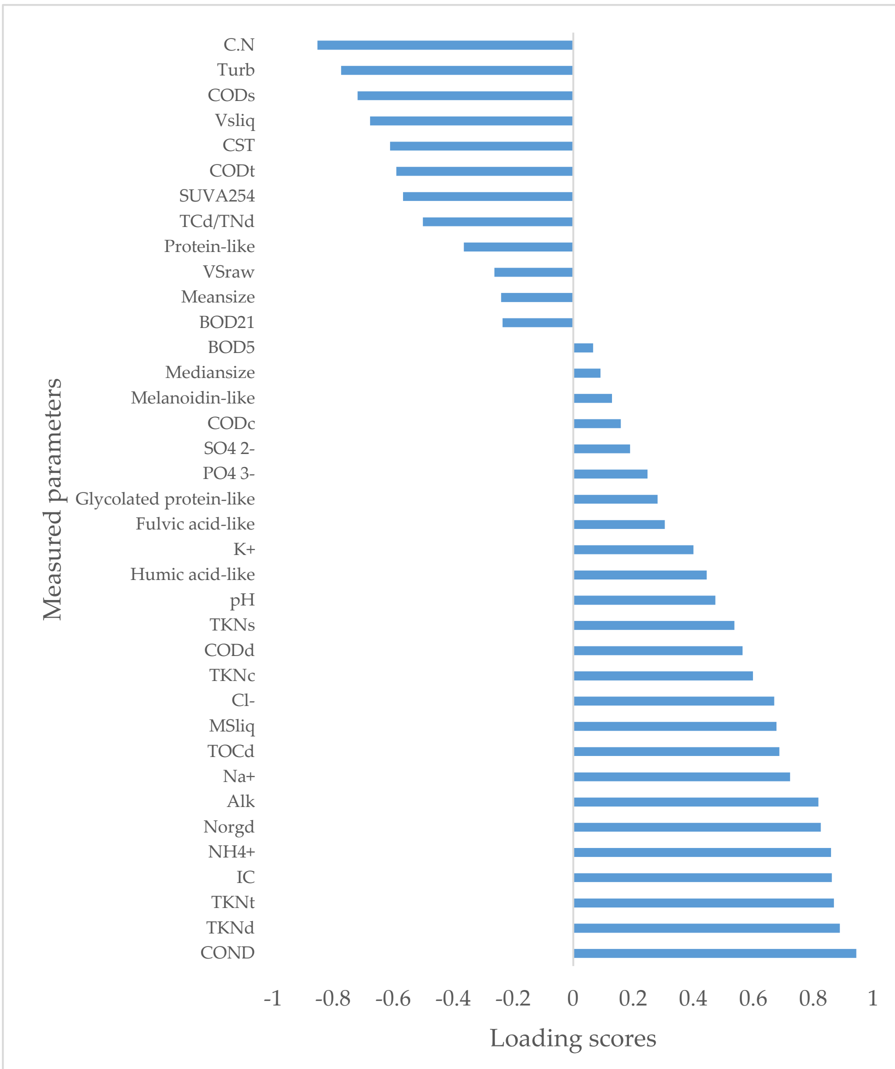


Figure 3. Loading scores of measured parameters of Dimension 1.

3.3. Influence of Feedstock Composition on Digestate Characteristics

Hierarchical Clustering Analysis (HCA) was carried out to evaluate the influence of the feedstock composition. Sequentially, to remove the influence of solid–liquid separators, HCA was separately implemented according to high performance of solid–liquid separators (centrifuge and other types of separators with addition of coagulant, flocculent or polymer) and low performance solid–liquid separators (screw press, vibrating screen and rotary drum), as shown in Figure 4. In high performance separation group (left side of Figure 4), two major clusters of liquid fractions can be identified based on AD feedstock. The first cluster was primarily from sewage sludge codigested with Mnr (pig manure), FOG and AFW. Meanwhile, the second cluster identified was primarily originated from agricultural and industrial wastes. This cluster can then be divided by two subclusters; liquid phase anaerobic digestion (L-AD) from mesophilic CSTR and solid-state anaerobic digestion (SS-AD) from T-PF reactor. From the observation in the subgroup of L-AD, the influence of higher manure proportion formed the group apart the other major group of L-AD primarily from sewage sludge.

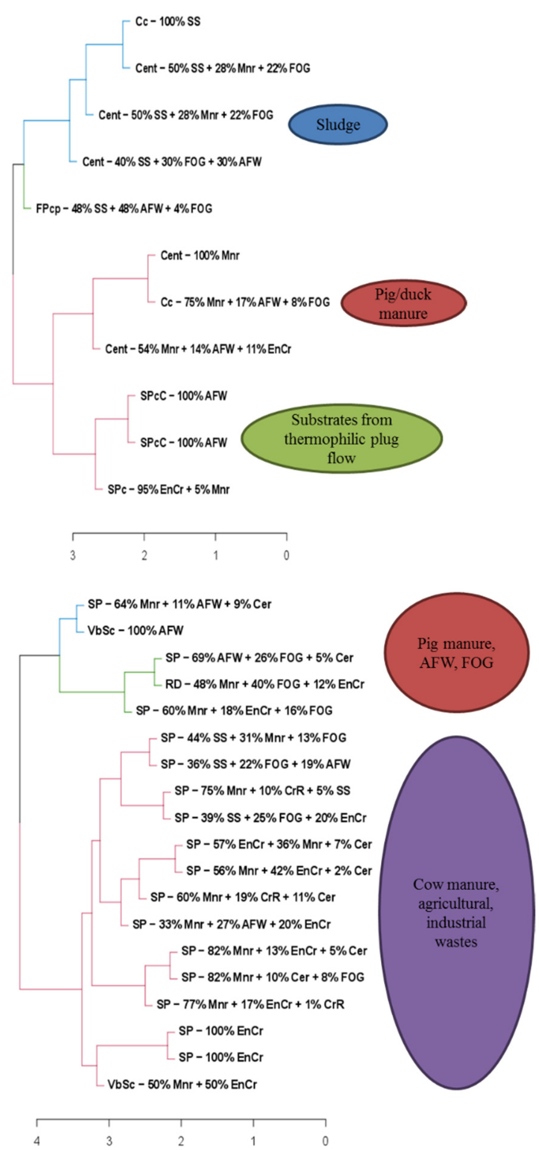


Figure 4. Clustering of feedstocks (Left: High performance solid–liquid-solid separation, Right: Low performance solid–liquid separation).

The clustering of low performance solid–liquid separation was less marked regarding AD feedstock but can also be separated into two major clusters. The first cluster was predominantly originating either from pig manure, FOG or AFW. Meanwhile, the origin of the second cluster was from the codigestion of cow manure and diverse agricultural and industrial wastes, including sewage sludge.

Figure 5 plotted below aims to analyze the influence of substrates' composition on the liquid fractions of digestates, specifically on final COD concentration. From the observation, a correlation $R^2 = 0.53$ (p value < 0.1) exists between cow manure percentage in the feedstock and the COD concentration. Having a larger sample size made it possible to confirm the observations made by Akhiar et al. [13] with 11 digestates, which also confirmed a study by Ganesh et al. [55] where the increase of cow manure proportions in the feed led to higher COD concentration in the liquid fractions of digestates. It was also observed that higher energy crops' proportion in the feedstock may possibly influence the COD concentration in the liquid fractions of digestates with $R^2 = 0.24$ (p value < 0.1) (Figure 5). This correlation is not robust and should be confirmed in further work.

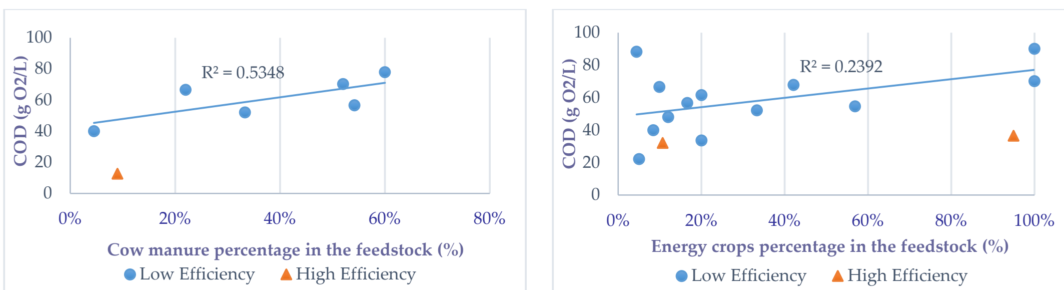
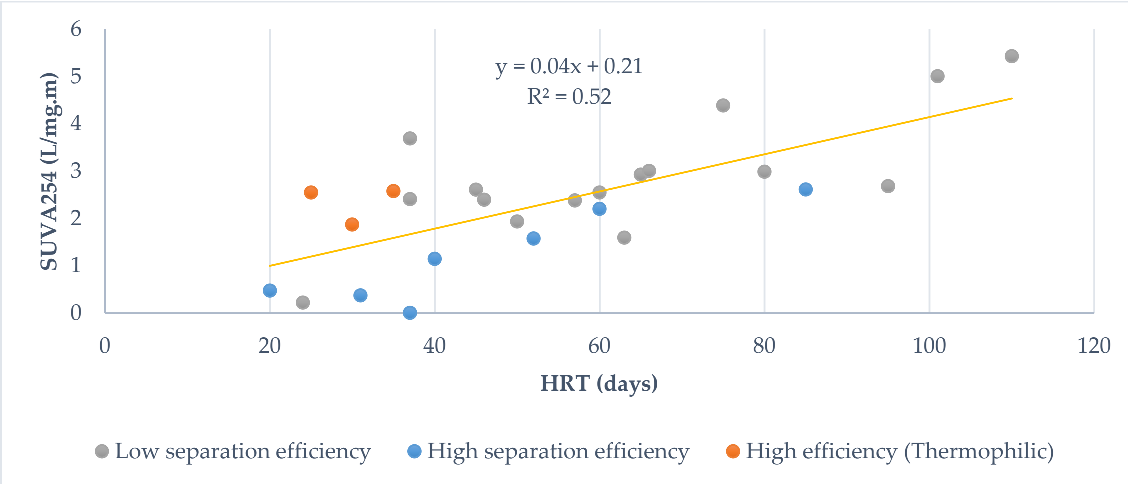


Figure 5. (Left) COD in the liquid fraction vs cow manure percentage and (right) COD in the liquid fraction vs energy crops percentage in the feedstock.

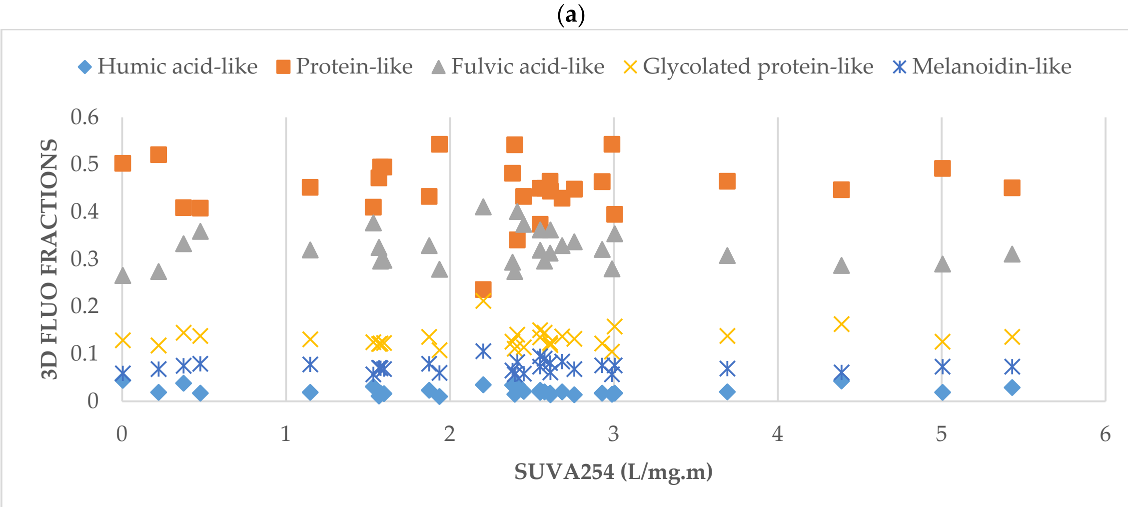
3.4. Influence of Anaerobic Digestion Operating Parameters on Digestate Characteristics: Impact of HRT on Liquid Fractions of Digestates

In this study, the parameter with the highest correlation to HRT observed was $SUVA_{254}$ with R = 0.52 (p value < 0.01) as presented in Figure 6a. The $SUVA_{254}$ is a common indicator of the aromatic content of the organic matter in water and wastewater. When an anaerobic digester is set to a longer HRT, it could be presumed that independently of the substrates at the input, the degree of humification in the digester rises proportionally with $SUVA_{254}$. This correlation was previously studied by Zheng et al. [39] with a variety of biodegradable substrates confirming proportional correlation between $SUVA_{254}$ and degradation time. Besides, the final $SUVA_{254}$ was also observed to vary depending on the types of substrates. Given that HRT correspondingly depends on the types of substrates, an indirect relation between $SUVA_{254}$ and the characteristics of the substrates can be presumed.

The degree of humification should also be represented by the measurements of fluorimetry, describing humic acidlike area. Figure 6b intended to examine $SUVA_{254}$ and its relation with 3D fluorescence spectrum zones; however, no correlation between fluorimetry fractions (in particular humic acidlike area) of digestates and $SUVA_{254}$ was observed. This was previously shown by Yang et al. [56] and Bioroza et al. [57] for the organic matter in water and drinking water, respectively. These two indicators, $SUVA_{254}$ and 3D fluorimetry, signify the humification intensity but from different molecules; both indicators are then incommutable.



(**a**)



(**b**)

Figure 6. (**a**) $SUVA_{254}$ (L/mg·m) in the liquid fraction vs HRT (days) and (**b**) 3D fluorescence spectrum zones vs $SUVA_{254}$ (L/mg·m).

3.5. Outcome of the Work

The separation technique and anaerobic digestion feedstock were identified as the major drivers of the remaining organic matter characteristics in the liquid fractions of digestates, with the separation technique being usually selected according to the feedstock. This study allowed us to define different categories of liquid fractions of digestates:

- Digestates from sewage sludge, pig manure and from thermophilic plug-flow reactor whose phase separation is carried out by high efficiency techniques (e.g., centrifugation, and other techniques using flocculant or coagulants) and
- Digestates from agricultural fibrous feedstocks which are processed by low efficiency technique processes (e.g., screw presses, vibrating screens and rotary drums). In particular, cow manure content in the feedstock was found to have high impact on the remaining COD in the liquid fraction of digestate.

These categories would set reference compositions in relation to process conditions and will support better knowledge of the liquid fractions of digestates. In addition, this work can be useful for example to practitioners when designing appropriate post-treatment of the digestates. It can also be useful to identify new solutions of the post-treatment of digestate by maximizing its utilization towards achieving circular economy.

4. Conclusions

The combined effect of the solid–liquid separation technique and anaerobic digestion feedstock were identified as the major drivers of the remaining organic matter characteristics in the liquid fractions of digestates. Two major clusters were identified in this study: (1) high-performance solid–liquid separators such as centrifuge and other separation systems with addition of coagulant, flocculent or polymer (separation efficiency from 44 to 93%) are mainly applied to digestates from sewage sludge, pig manure and from plug-flow thermophilic processes; (2) low-performance solid–liquid separators such as screw presses, vibrating screens and rotary drums (separation efficiency not more than 44%) are commonly applied to fibrous digestates; in this case, increasing the percentage of cow manure or energy crops in the feedstocks contents' were identified as the contributing factors to the increase in the remaining organic compounds in the liquid fractions of digestates. Notably, cow manure percentage in the feedstocks had a robust correlation with the concentration of COD in liquid fractions of digestates. Besides, amongst all the operational parameters observed, longer HRT applied to the reactor appears to have an impact to higher value of $SUVA_{254}$, associated with fulvic acid compounds in dissolved matter. This indicator fits to describe the organic matter stabilization after biodegradation.

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Appendix A

Table A1. Substrate categories based on substrates at the input.

Substrate Category	Substrates at the Input
Sewage sludge (SS)	Sludge, solid sludge, liquid sludge, waste activated sludge, cheese plant sludge, contents from septic tanks (and garbage), wastewater
Manure (Mnr)	Animal manures and slurry
Energy crops (EnCr)	Energy crops, catch crop, corn silage, grass silage, grass, energy crop silage, whole grain plants, sorghum silage, barley, rye
Crop residues (CrR)	Crop residues, corn withers, sweetcorn cobs, tomato leaves, apple pomace
Cereal residues (Cer)	Cereal residues, crushed grain
Fats, oil and grease (FOG)	Fats, oil, grease
Agro-food wastes (AFW)	Food wastes, fruit and vegetable wastes, municipal biowastes, biowastes, glucose, cattle feed residues, pet food, milk industry residues, mixture of cream milk, slaughterhouse wastes, blood, glycerin, whey

Table A2. Total solids (TS), volatile solids (VS) and mineral solids (MS), VS/TS, pH, alkalinity, turbidity, capillary suction time (CST) and particle sizes.

	Raw Digestate				Solid Fraction of Digestate				Liquid Fraction of Digestate												
Plant	TS (g/kg)	VS (g/kg)	MS (g/kg)	VS/TS (%)	TS (g/kg)	VS (g/kg)	MS (g/kg)	VS/TS (%)	TS (g/kg)	VS (g/kg)	MS (g/kg)	VS/TS (%)	pH	Alkalinity ($gCaCO_3/L$)	Turbidity (NTU)	CST (CST10g TS/kg) (s)	0.375–50 µm	50–100 µm	100–500 µm	500–1000 µm	1000–2000 µm
A	70.9	43.0	27.9	61%	234.8	208.0	26.8	89%	56.8	31.8	25.0	56%	8.22	24.8	43300	242.3	72%	18%	10%	0%	0%
B	107.5	74.1	33.4	69%	229.2	182.6	46.6	80%	80.9	52.3	28.6	65%	7.88	17.1	51400	454.6	50%	12%	32%	6%	0%
C	14.4	5.4	9.0	37%	173.2	161.1	12.2	93%	13.7	4.7	9.0	35%	8.14	7.4	6160	58.9	89%	2%	8%	0%	0%
E	55.9	27.3	28.5	49%	238.9	117.3	121.5	49%	16.7	7.5	9.2	45%	8.42	21.5	3780	46.9	35%	26%	39%	0%	0%
F	104.8	64.4	40.4	61%	309.9	243.3	66.6	79%	82.7	44.5	38.1	54%	8.30	23.6	49400	319.2	53%	14%	29%	3%	0%
H	54.6	36.2	18.5	66%	179.5	119.1	60.5	66%	10.3	6.0	4.3	58%	8.25	14.1	2960	34.0	54%	32%	14%	0%	0%
I	227.2	115.4	111.8	51%	416.2	198.9	217.2	48%	37.9	22.6	15.3	60%	8.08	13.8	12840	16.3	27%	19%	45%	5%	4%
J	94.4	74.0	20.4	78%	296.2	239.8	56.4	81%	32.1	20.9	11.3	65%	8.19	13.2	7590	16.7	40%	17%	43%	0%	0%
K	63.7	40.9	22.8	64%	264.8	218.5	46.3	83%	60.5	38.0	22.4	63%	7.61	14.0	41800	225.4	70%	13%	13%	4%	0%
L	44.4	28.3	16.1	64%	323.9	272.4	51.5	84%	40.6	24.5	16.1	60%	8.25	22.6	29640	319.3	39%	9%	32%	19%	2%

M	44.4	34.4	10.0	77%	99.2	87.6	11.5	88%	38.4	28.7	9.7	75%	8.15	9.0	38160	416.2	53%	13%	21%	8%	4%
G	30.5	17.6	12.8	58%	276.0	156.6	119.3	57%	2.1	1.2	1.0	0.55	7.95	3.7	947	12.1	59%	16%	20%	5%	0%
N	93.2	62.3	30.9	67%	218.2	189.0	29.2	87%	76.7	46.0	30.7	60%	8.12	24.0	67067	314.9	76%	9%	15%	0%	0%
O	33.8	21.0	12.8	62%	276.7	164.9	111.8	60%	13.1	7.1	6.0	54%	8.39	10.3	6835	70.8	93%	7%	0%	0%	0%
P	68.0	39.1	28.8	58%	202.4	163.0	39.4	81%	61.6	34.6	27.0	56%	7.7	10.0	33552	271.8	56%	12%	24%	8%	0%
Q	71.2	43.1	28.1	61%	267.3	202.5	64.8	76%	72.6	48.1	24.4	66%	7.93	23.3	48940	198.1	41%	9%	33%	16%	1%
R	47.4	33.4	14.0	70%	247.1	151.2	95.9	61%	26.7	18.6	8.0	70%	7.82	16.3	20712	424.7	87%	5%	8%	0%	0%
S	48.4	31.4	16.9	65%	187.6	114.5	73.1	61%	6.7	3.0	3.7	45%	8.08	13.8	1409	15.0	77%	15%	8%	0%	0%
T	71.6	42.1	29.5	59%	893.9	537.5	356.4	60%	46.9	27.3	19.5	58%	8.49	31.4	25160	402.7	67%	13%	20%	0%	0%
U	78.0	42.0	36.0	54%	243.7	173.4	70.3	71%	66.2	41.7	24.5	63%	8.04	23.4	42030	395.3	79%	11%	10%	0%	0%
V	73.8	44.8	28.9	61%	179.9	124.7	55.2	69%	54.3	32.1	22.3	59%	7.92	27.6	36260	448.7	56%	13%	30%	2%	0%
W	81.8	57.7	24.1	71%	244.4	209.0	35.5	85%	56.5	41.1	15.4	73%	8.2	20.2	30015	820.0	58%	13%	29%	0%	0%
X	94.7	58.6	36.1	62%	208.8	166.4	42.4	80%	85.5	51.3	34.2	60%	8.32	29.1	60800	665.4	55%	13%	31%	1%	0%
Y	52.7	33.7	19.0	64%	374.0	267.4	106.6	71%	44.6	26.2	18.3	59%	8.51	26.0	27000	714.6	58%	13%	25%	4%	0%
Z	117.9	78.7	39.2	67%	246.2	204.9	41.3	83%	67.3	41.2	26.1	61%	7.99	29.0	42420	404.7	45%	12%	34%	8%	1%
I2	282.8	97.9	185.0	35%	439.7	166.8	272.9	38%	37.2	20.7	16.5	56%	8.24	12.7	10100	17.5	20%	13%	43%	14%	11%
AA	61.5	39.9	21.6	65%	314.2	256.3	57.9	82%	34.5	22.1	12.4	64%	8.28	18.1	20027	250.7	82%	6%	12%	0%	0%
AB	67.4	36.6	30.9	54%	296.7	202.6	94.1	68%	46.3	23.4	22.8	51%	7.89	20.4	23093	192.6	65%	7%	22%	6%	0%
AC	31.7	19.7	12.0	62%	315.4	212.9	102.6	67%	16.1	8.7	7.4	54%	8.02	12.0	10367	98.1	85%	8%	7%	0%	0%
AD	18.9	8.7	10.2	46%	879.6	425.9	453.7	48%	7.8	2.9	4.9	37%	9.09	2.3	12	5.8	19%	41%	40%	0%	0%

Table A3. Mean, median, inorganic carbon (IC), total organic carbon (TOC), total carbon (TC), chemical oxygen demand (COD), total kjeldahl nitrogen (TKN), ammonium (NH_4^+), nitrogen (N), ammonium (NH_4^+), carbon/nitrogen ration (C/N), sodium (Na^+), potassium (K^+), chlorine (Cl^-), phosphate (PO_4^{3-}), sulfate (SO_4^{2-}), conductivity (COND).

Plant	Mean (µm)	Median (µm)	IC (g/L)	TOCd (g/L)	TCd (g/L)	CODt (g/L)	CODs (g/L)	CODc (g/L)	CODd (g/L)	CODd/TOCd	TKNt (g/L)	TKNs (g/L)	TKNc (g/L)	TKNd (g/L)	Norgd (g/L)	NH_4^+ (g/L)	Cd/Nd	Na^+ (g/L)	K^+ (g/L)	Cl^- (g/L)	PO_4^{3-} (g/L)	SO_4^{2-} (g/L)	CONDd (mS/cm)
A	48.2	30.0	2.3	0.5	2.8	47.3	44.5	1.3	1.5	3.2	6.5	3.1	0.7	2.7	0.1	2.6	1.1	0.5	1.8	0.9	0.1	0.7	25.8
B	137.6	53.0	2.7	1.5	4.2	78.0	67.3	6.4	4.3	2.8	4.7	2.6	0.4	1.7	0.3	1.3	2.5	0.5	4.7	1.3	0.9	0.1	28.6
C	30.6	14.0	1.3	0.8	2.1	9.2	8.5	0.2	0.5	0.6	1.5	0.7	0.1	0.7	0.2	0.6	2.8	0.1	3.2	1.1	0.0	0.1	14.3
E	76.0	89.0	3.2	3.2	6.3	12.1	8.1	1.7	2.2	0.7	5.1	1.6	0.5	3.0	1.0	2.1	2.1	3.0	2.0	1.0	2.1	0.1	30.6
F	105.2	44.0	3.6	2.7	6.2	70.3	57.2	4.5	8.6	3.2	5.8	2.7	0.4	2.7	0.3	2.3	2.3	1.1	4.7	1.8	0.8	0.0	38.0
H	60.8	49.0	2.4	1.2	3.6	9.8	7.2	1.3	1.4	1.1	4.3	0.5	0.4	3.4	0.3	3.1	1.1	0.4	1.0	1.6	0.1	0.0	27.4
I	208.0	112.0	2.3	1.5	3.7	39.5	28.1	7.1	4.4	3.0	4.4	1.7	0.6	2.1	0.3	1.8	1.8	1.3	2.5	2.3	0.2	0.1	30.0
J	120.8	80.0	2.4	1.7	4.1	36.6	21.4	9.9	5.2	3.0	4.6	1.5	0.9	2.1	0.3	1.8	1.9	0.1	5.5	3.8	0.1	0.1	35.3
K	82.4	30.0	1.6	0.5	2.0	61.7	59.4	1.3	1.0	2.1	4.6	2.7	0.6	1.3	0.3	1.1	1.5	0.4	1.3	0.5	0.6	0.1	16.3
L	257.2	116.0	2.9	1.1	4.0	22.2	16.3	2.7	3.2	2.9	5.2	3.0	0.4	1.8	0.4	1.4	2.3	1.0	4.3	1.8	0.8	0.7	32.8
M	179.4	46.0	1.7	1.2	2.9	52.1	47.2	1.6	3.4	2.8	2.9	1.9	0.3	0.8	0.3	0.5	3.8	0.6	3.9	1.4	0.6	0.1	16.0
G	100.7	40.0	0.5	0.3	0.8	1.7	1.4	0.1	0.2	0.8	1.1	0.2	0.2	0.7	0.1	0.6	1.2	0.1	0.1	0.1	0.0	0.0	4.9
N	53.3	21.3	2.7	0.9	3.6	88.3	82.7	3.0	2.6	2.7	6.3	3.2	0.3	2.8	0.6	2.2	1.3	0.4	3.5	1.0	0.4	0.9	33.4

O	26.2	23.7	1.6	1.0	2.6	12.7	10.5	0.6	1.6	1.6	2.7	0.7	0.1	1.8	0.6	1.2	1.4	0.8	1.6	1.0	0.1	0.3	20.0
P	125.2	42.4	1.3	0.6	1.9	63.1	60.5	0.6	2.1	3.3	3.0	2.0	0.4	0.6	0.2	0.5	3.0	0.7	2.5	0.9	0.8	0.1	15.9
Q	240.5	103.4	2.1	0.9	3.0	56.7	51.9	2.3	2.5	2.7	3.8	2.0	0.2	1.6	-	1.7	1.8	0.6	3.6	1.2	0.5	0.2	24.9
R	30.7	20.5	1.7	0.9	2.5	32.2	28.0	1.5	2.6	3.0	3.2	1.3	0.1	1.8	0.2	1.6	1.4	0.3	2.8	1.0	0.5	0.0	20.5
S	38.0	26.9	1.8	0.4	2.1	4.4	2.6	0.7	1.0	2.9	3.9	0.8	0.2	2.9	0.7	2.2	0.7	0.5	1.4	1.9	0.1	0.0	21.7
T	62.0	30.1	3.7	1.6	5.3	48.1	41.9	3.3	2.9	1.8	8.3	2.8	0.2	5.2	0.6	4.7	1.0	1.4	3.3	2.9	0.4	0.0	46.3
U	35.1	14.8	3.7	1.3	5.0	67.9	56.5	5.5	5.9	4.4	5.7	2.2	0.3	3.3	0.6	2.7	1.5	0.3	6.7	1.7	1.0	0.2	39.0
V	105.1	38.1	2.5	0.9	3.4	54.7	48.0	3.4	3.3	3.8	4.3	2.0	0.2	2.1	0.4	1.8	1.6	0.2	5.4	1.5	0.9	0.2	28.9
W	82.8	36.5	3.7	1.1	4.8	70.2	59.4	4.4	6.4	5.6	5.7	2.4	0.4	2.9	-	3.0	1.7	0.1	5.0	1.0	0.4	0.3	36.0
X	98.4	43.2	4.4	1.4	5.8	90.1	73.1	8.8	8.2	5.9	7.6	4.0	0.5	3.1	-	3.2	1.9	0.2	7.1	1.0	0.8	0.2	40.8
Y	103.7	36.2	3.2	0.8	4.0	41.3	36.0	3.0	2.4	2.8	7.7	3.0	0.5	4.2	1.1	3.1	1.0	2.4	2.8	3.8	0.5	0.3	43.0
Z	174.2	69.4	2.9	1.3	4.2	66.7	58.1	4.9	3.7	2.8	5.2	2.1	0.4	2.7	0.5	2.2	1.5	0.5	5.2	1.5	0.6	0.08	33.3
I2	388.1	199.1	2.0	1.5	3.5	28.9	20.3	3.9	4.7	3.0	3.6	1.4	0.3	1.9	0.3	1.6	1.8	1.2	3.6	2.3	0.2	0.1	27.5
AA	30.2	7.6	2.8	1.2	4.0	40.1	32.4	4.3	3.3	2.7	5.0	1.5	0.3	3.1	1.1	2.0	1.3	0.7	4.3	2.1	0.4	0.1	33.9
AB	113.7	27.6	2.9	0.8	3.6	33.7	29.9	1.4	2.4	3.1	4.8	2.2	0.1	2.5	0.5	2.0	1.5	3.3	3.9	3.8	0.6	0.0	38.9
AC	23.9	7.3	1.7	1.0	2.7	15.4	12.5	1.2	1.6	1.7	2.8	0.9	0.0	1.9	0.3	1.5	1.5	0.8	2.5	1.3	0.1	0.1	21.1
AD	91.4	97.0	0.5	0.1	0.6	0.8	0.0	0.0	0.8	-	1.3	0.0	0.1	1.3	0.3	1.0	0.5	1.3	0.5	4.4	0.0	0.0	17.4

t = total, s = suspended, c = colloids, d = dissolved, orgd = dissolved organic.

Table A4. SUVA$_{254}$, BOD$_5$, BOD$_{21}$, BOD$_5$/COD, BOD$_{21}$/COD, protein-like, fulvic acid-like, glycolated protein-like, melanoidin-like, humic acid-like.

Plant	SUVA$_{254}$	BOD$_5$ (g/L)	BOD$_{21}$ (g/L)	BOD$_5$/ COD	BOD$_{21}$/ COD	Protein-Like	Fulvic Acid-Like	Glycolated Protein-Like	Melanoidin-Like	Humic Acid-Like
A	1.6	7.4	23.2	0.2	0.5	47%	33%	12%	7%	1%
B	2.6	5.6	12.9	0.1	0.2	37%	36%	15%	10%	2%
C	0.2	1.7	3.1	0.2	0.3	52%	27%	12%	7%	2%
E	0.0	1.9	4.3	0.2	0.4	50%	27%	13%	6%	4%
F	1.6	3.7	9.8	0.1	0.1	50%	30%	12%	7%	2%
H	0.4	3.7	5.3	0.4	0.5	41%	33%	15%	7%	4%
I	1.9	7.3	18.1	0.2	0.5	43%	33%	14%	8%	2%
J	2.6	8.6	22.5	0.2	0.6	45%	30%	14%	9%	2%
K	1.9	9.4	32.2	0.2	0.5	54%	28%	11%	6%	1%
L	2.4	4.8	11.1	0.2	0.5	54%	27%	11%	6%	2%
M	3.0	5.0	14.3	0.1	0.3	54%	28%	11%	6%	2%
G	0.5	0.4	1.0	0.2	0.6	41%	36%	14%	8%	2%
N	2.7	13.9	42.6	0.2	0.5	43%	33%	14%	8%	2%
O	1.6	0.3	1.3	0.0	0.1	50%	30%	12%	7%	2%
P	2.9	3.7	27.8	0.1	0.4	46%	32%	12%	8%	2%
Q	2.8	7.4	18.2	0.1	0.3	45%	34%	13%	7%	1%
R	2.6	9.6	20.7	0.3	0.6	44%	36%	12%	6%	1%
S	2.4	1.9	2.9	0.4	0.7	43%	37%	11%	6%	2%

T	1.5	13.1	28.3	0.3	0.6	41%	38%	12%	6%	3%
U	5.0	13.0	30.6	0.2	0.5	49%	29%	13%	7%	2%
V	3.7	12.0	28.2	0.2	0.5	46%	31%	14%	7%	2%
W	5.4	16.6	35.6	0.2	0.5	45%	31%	14%	7%	3%
X	4.4	30.1	54.2	0.3	0.6	45%	29%	16%	6%	4%
Y	2.4	16.2	26.0	0.4	0.6	34%	40%	14%	8%	3%
Z	3.0	14.5	35.0	0.2	0.5	39%	35%	16%	8%	2%
I2	2.6	9.6	17.8	0.3	0.6	45%	32%	14%	7%	2%
AA	2.6	7.2	17.6	0.2	0.4	46%	31%	12%	8%	2%
AB	2.4	11.4	25.4	0.3	0.8	48%	29%	13%	6%	3%
AC	1.1	3.7	8.0	0.2	0.5	45%	32%	13%	8%	2%
AD	2.2	0.1	0.4	0.1	0.4	24%	41%	21%	11%	3%

References

1. European Biogas Association (EBA). *Statistical Report of the European Biogas Association 2018*; EBA: Brussels, Belgium, 2018.
2. Akhiar, A.; Ahmad Zamri, M.F.M.; Torrijos, M.; Shamsuddin, A.H.; Battimelli, A.; Roslan, E.; Mohd Marzuki, M.H.; Carrere, H. Anaerobic digestion industries progress throughout the world. *IOP Conf. Ser. Earth Environ. Sci.* **2020**, *476*, 012074.
3. Battista, F.; Frison, N.; Bolzonella, D. Energy and Nutrients' Recovery in Anaerobic Digestion of Agricultural Biomass: An Italian Perspective for Future Applications. *Energies* **2019**, *12*, 3287, doi:10.3390/en12173287.
4. Herbes, C.; Dahlin, J.; Kurz, P. Consumer Willingness To Pay for Proenvironmental Attributes of Biogas Digestate-Based Potting Soil. *Sustainability* **2020**, *12*, 6405, doi:10.3390/su12166405.
5. Guilayn, F.; Jimenez, J.; Martel, J.-L.; Rouez, M.; Crest, M.; Patureau, D. First fertilizing-value typology of digestates: A decision-making tool for regulation. *Waste Manag.* **2019**, *86*, 67–79, doi:10.1016/J.WASMAN.2019.01.032.
6. Szymańska, M.; Szara, E.; Sosulski, T.; Wąs, A.; Van Pruissen, G.W.P.; Cornelissen, R.L.; Borowik, M.; Konkol, M. A Bio-Refinery Concept for N and P Recovery—A Chance for Biogas Plant Development. *Energies* **2019**, *12*, 155, doi:10.3390/en12010155.
7. Maurer, C.; Seiler-Petzold, J.; Schulz, R.; Müller, J. Short-Term Nitrogen Uptake of Barley from Differently Processed Biogas Digestate in Pot Experiments. *Energies* **2019**, *12*, 696, doi:10.3390/en12040696.
8. Guilayn, F.; Jimenez, J.; Rouez, M.; Crest, M.; Patureau, D. Digestate mechanical separation: Efficiency profiles based on anaerobic digestion feedstock and equipment choice. *Bioresour. Technol.* **2019**, *274*, 180–189, doi:10.1016/J.BIORTECH.2018.11.090.
9. Drosg, B.; Fuchs, W.; Al Seadi, T.; Madsen, M.; Linke, B. *Nutrient Recovery by Biogas Digestate Processing*; 2015. Available Online: http://task37.ieabioenergy.com/files/daten-redaktion/download/Technical%20Brochures/NUTRIENT_RECOVERY_RZ_web1.pdf (accessed on 23 November 2020)
10. Bauer, A.; Mayr, H.; Hopfner-Sixt, K.; Amon, T. Detailed monitoring of two biogas plants and mechanical solid–liquid separation of fermentation residues. *J. Biotechnol.* **2009**, *142*, 56–63, doi:10.1016/j.jbiotec.2009.01.016.
11. Rehl, T.; Müller, J. Life cycle assessment of biogas digestate processing technologies. *Resour. Conserv. Recycl.* **2011**, *56*, 92–104, doi:10.1016/j.resconrec.2011.08.007.
12. Tambone, F.; Orzi, V.; D'Imporzano, G.; Adani, F. Solid and liquid fractionation of digestate: Mass balance, chemical characterization, and agronomic and environmental value. *Bioresour. Technol.* **2017**, *243*, 1251–1256, doi:10.1016/J.BIORTECH.2017.07.130.
13. Akhiar, A.; Torrijos, M.; Battimelli, A.; Carrère, H. Comprehensive characterization of the liquid fraction of digestates from full-scale anaerobic co-digestion. *Waste Manag.* **2017**, *59*, 118–128, doi:10.1016/j.wasman.2016.11.005.
14. Xia, A.; Murphy, J.D. Microalgal Cultivation in Treating Liquid Digestate from Biogas Systems. *Trends Biotechnol.* **2016**, *34*, 264–275, doi:10.1016/j.tibtech.2015.12.010.
15. Baldi, M.; Collivignarelli, M.; Abbà, A.; Benigna, I. The Valorization of Ammonia in Manure Digestate by Means of Alternative Stripping Reactors. *Sustainability* **2018**, *10*, 3073, doi:10.3390/su10093073.
16. Valentinuzzi, F.; Cavani, L.; Porfido, C.; Terzano, R.; Pii, Y.; Cesco, S.; Marzadori, C.; Mimmo, T. The fertilising potential of manure-based biogas fermentation residues: Pelleted vs. liquid digestate. *Heliyon* **2020**, *6*, e03325, doi:10.1016/j.heliyon.2020.e03325.
17. Guilayn, F.; Rouez, M.; Crest, M.; Patureau, D.; Jimenez, J. Valorization of digestates from urban or centralized biogas plants: A critical review. *Rev. Environ. Sci. Biotechnol.* **2020**, *19*, 419–462.
18. Tao, W.; Fattah, K.P.; Huchzermeier, M.P. Struvite recovery from anaerobically digested dairy manure: A review of application potential and hindrances. *J. Environ. Manag.* **2016**, *169*, 46–57, doi:10.1016/j.jenvman.2015.12.006.
19. Szymańska, M.; Sosulski, T.; Szara, E.; Wąs, A.; Sulewski, P.; van Pruissen, G.W.P.; Cornelissen, R.L. Ammonium Sulphate from a Bio-Refinery System as a Fertilizer—Agronomic and Economic Effectiveness on the Farm Scale. *Energies* **2019**, *12*, 4721, doi:10.3390/en12244721.
20. Folino, A.; Zema, D.A.; Calabrò, P.S. Environmental and Economic Sustainability of Swine Wastewater Treatments Using Ammonia Stripping and Anaerobic Digestion: A Short Review. *Sustainability* **2020**, *12*, 4971, doi:10.3390/su12124971.
21. Szymańska, M.; Szara, E.; Wąs, A.; Sosulski, T.; van Pruissen, G.; Cornelissen, R. Struvite—An Innovative Fertilizer from Anaerobic Digestate Produced in a Bio-Refinery. *Energies* **2019**, *12*, 296, doi:10.3390/en12020296.
22. Gienau, T.; Ehrmanntraut, A.; Kraume, M.; Rosenberger, S. Influence of Ozone Treatment on Ultrafiltration Performance and Nutrient Flow in a Membrane Based Nutrient Recovery Process from Anaerobic Digestate. *Membranes* **2020**, *10*, 64, doi:10.3390/membranes10040064.
23. Świątczak, P.; Cydzik-Kwiatkowska, A.; Zielińska, M. Treatment of Liquid Phase of Digestate from Agricultural Biogas Plant in a System with Aerobic Granules and Ultrafiltration. *Water* **2019**, *11*, 104, doi:10.3390/w11010104.
24. Myllymäki, P.; Pesonen, J.; Romar, H.; Hu, T.; Tynjälä, P.; Lassi, U. The Use of Ca- and Mg-Rich Fly Ash as a

Chemical Precipitant in the Simultaneous Removal of Nitrogen and Phosphorus—Recycling and Reuse. *Recycling* **2019**, *4*, 14, doi:10.3390/recycling4020014.
25. Krzemińska, I.; Oleszek, M.; Wiącek, D. Liquid Anaerobic Digestate as a Source of Nutrients for Lipid and Fatty Acid Accumulation by Auxenochlorella Protothecoides. *Molecules* **2019**, *24*, 3582, doi:10.3390/molecules24193582.
26. Kisielewska, M.; Zieliński, M.; Dębowski, M.; Kazimierowicz, J.; Romanowska-Duda, Z.; Dudek, M. Effectiveness of Scenedesmus sp. Biomass Grow and Nutrients Removal from Liquid Phase of Digestates. *Energies* **2020**, *13*, 1432, doi:10.3390/en13061432.
27. Jimenez, R.; Markou, G.; Tayibi, S.; Barakat, A.; Chapsal, C.; Monlau, F. Production of Microalgal Slow-Release Fertilizer by Valorizing Liquid Agricultural Digestate: Growth Experiments with Tomatoes. *Appl. Sci.* **2020**, *10*, 3890, doi:10.3390/app10113890.
28. Akhiar, A.; Zamri, M.F.M.A.; Torrijos, M.; Battimelli, A.; Roslan, E.; Hanif, M.; Shamsuddin, A.H.; Carrere, H. Current Technology on Nutrients Removal, Recovery and Reuse from Liquid Fraction of Digestate. *TEST Eng. Manag.* **2019**, *81*, 5861–5868.
29. Theuerl, S.; Herrmann, C.; Heiermann, M.; Grundmann, P.; Landwehr, N.; Kreidenweis, U.; Prochnow, A. The Future Agricultural Biogas Plant in Germany: A Vision. *Energies* **2019**, *12*, 396, doi:10.3390/en12030396.
30. Piwowar, A. Agricultural Biogas—An Important Element in the Circular and Low-Carbon Development in Poland. *Energies* **2020**, *13*, 1733, doi:10.3390/en13071733.
31. Barros, M.V.; Salvador, R.; de Francisco, A.C.; Piekarski, C.M. Mapping of research lines on circular economy practices in agriculture: From waste to energy. *Renew. Sustain. Energy Rev.* **2020**, *131*, 109958.
32. Molina-Moreno, V.; Leyva-Díaz, J.; Llorens-Montes, F.; Cortés-García, F. Design of Indicators of Circular Economy as Instruments for the Evaluation of Sustainability and Efficiency in Wastewater from Pig Farming Industry. *Water* **2017**, *9*, 653, doi:10.3390/w9090653.
33. Muradin, M.; Joachimiak-Lechman, K.; Foltynowicz, Z. Evaluation of Eco-Efficiency of Two Alternative Agricultural Biogas Plants. *Appl. Sci.* **2018**, *8*, 2083, doi:10.3390/app8112083.
34. Vilardi, G.; Bassano, C.; Deiana, P.; Verdone, N. Exergy and energy analysis of three biogas upgrading processes. *Energy Convers. Manag.* **2020**, *224*, 113323, doi:10.1016/j.enconman.2020.113323.
35. Ziyang, L.; Youcai, Z. Size-fractionation and characterization of refuse landfill leachate by sequential filtration using membranes with varied porosity. *J. Hazard. Mater.* **2007**, *147*, 257–264, doi:10.1016/j.jhazmat.2006.12.084.
36. American Public Health Association. *Standard Methods for the Examination of Water and Wastewater*, 22nd ed.; Rice, E.W., Baird, R.B., Eaton, A.D., Clesceri, L.S., Eds.; American Public Health Association: Washington, DC, USA, 2012.
37. Battimelli, A.; Loisel, D.; Garcia-Bernet, D.; Carrere, H.; Delgenes, J.-P. Combined ozone pretreatment and biological processes for removal of colored and biorefractory compounds in wastewater from molasses fermentation industries. *J. Chem. Technol. Biotechnol.* **2010**, *85*, 968–975, doi:10.1002/jctb.2388.
38. Uggetti, E.; Sialve, B.; Latrille, E.; Steyer, J.-P. Anaerobic digestate as substrate for microalgae culture: The role of ammonium concentration on the microalgae productivity. *Bioresour. Technol.* **2014**, *152*, 437–443, doi:10.1016/j.biortech.2013.11.036.
39. Zheng, W.; Lü, F.; Phoungthong, K.; He, P. Relationship between anaerobic digestion of biodegradable solid waste and spectral characteristics of the derived liquid digestate. *Bioresour. Technol.* **2014**, *161*, 69–77, doi:10.1016/j.biortech.2014.03.016.
40. Jimenez, J.; Gonidec, E.; Cacho Rivero, J.A.; Latrille, E.; Vedrenne, F.; Steyer, J.-P. Prediction of anaerobic biodegradability and bioaccessibility of municipal sludge by coupling sequential extractions with fluorescence spectroscopy: Towards ADM1 variables characterization. *Water Res.* **2014**, *50*, 359–372, doi:10.1016/J.WATRES.2013.10.048.
41. Ganesh, R.; Torrijos, M.; Sousbie, P.; Steyer, J.P.; Lugardon, A.; Delgenes, J.P. Anaerobic co-digestion of solid waste: Effect of increasing organic loading rates and characterization of the solubilised organic matter. *Bioresour. Technol.* **2013**, *130*, 559–569, doi:10.1016/j.biortech.2012.12.119.
42. WTW GmbH. *Supervision of BOD Measuring Systems According to DIN/ISO 9000 and GLP*; WTW: Weilheim, Germany, 2010.
43. WTW GmbH. *System OxiTop® Control: Operating Manual*; WTW: Weilheim, Germany, 2004.
44. Hjorth, M.; Christensen, K.V.; Christensen, M.L.; Sommer, S.G. Solid–liquid separation of animal slurry in theory and practice. A review. *Agron. Sustain. Dev.* **2010**, *30*, 153–180.
45. R Core Team. *R: A Language and Environment for Statistical Computing*; R Foundation for Statistical Computing: Vienna, Austria, 2014.
46. Husson, F.; Le, S.; Pages, J. Exploratory Multivariate Analysis by Example Using R. *J. Stat. Softw.* **2011**, *40*.
47. Ward, J.H. Hierarchical grouping to optimize an objective function. *J. Am. Stat. Assoc.* **1963**, *58*, 236–244, doi:10.1080/01621459.1963.10500845.
48. Oliveira, I.; Reed, J.P.; Abu-Orf, M.; Wilson, V.; Jones, D.; Esteves, S.R. The potential use of shear viscosity to monitor polymer conditioning of sewage sludge digestates. *Water Res.* **2016**, *105*, 320–330,

doi:10.1016/j.watres.2016.08.007.
49. Banks, C.J.; Chesshire, M.; Heaven, S.; Arnold, R. Anaerobic digestion of source-segregated domestic food waste: Performance assessment by mass and energy balance. *Bioresour. Technol.* **2011**, *102*, 612–620, doi:10.1016/j.biortech.2010.08.005.
50. Zirkler, D.; Peters, A.; Kaupenjohann, M. Elemental composition of biogas residues: Variability and alteration during anaerobic digestion. *Biomass Bioenergy* **2014**, *67*, 89–98, doi:10.1016/j.biombioe.2014.04.021.
51. Sibil, R.; Berkun, M.; Bekiroglu, S. The comparison of different mathematical methods to determine the BOD parameters, a new developed method and impacts of these parameters variations on the design of WWTPs. *Appl. Math. Model.* **2014**, *38*, 641–658, doi:10.1016/j.apm.2013.07.013.
52. Herrmann, C.; Idler, C.; Heiermann, M. Biogas crops grown in energy crop rotations: Linking chemical composition and methane production characteristics. *Bioresour. Technol.* **2016**, *206*, 23–35, doi:10.1016/j.biortech.2016.01.058.
53. Dandikas, V.; Heuwinkel, H.; Lichti, F.; Drewes, J.E.; Koch, K. Correlation between biogas yield and chemical composition of energy crops. *Bioresour. Technol.* **2014**, *174*, 316–320, doi:10.1016/j.biortech.2014.10.019.
54. Møller, H.B. Separation efficiency and particle size distribution in relation to manure type and storage conditions. *Bioresour. Technol.* **2002**, *85*, 189–196.
55. Ganesh, R.; Torrijos, M.; Sousbie, P.; Lugardon, A.; Steyer, J.P.; Delgenes, J.P. Effect of increasing proportions of lignocellulosic cosubstrate on the single-phase and two-phase digestion of readily biodegradable substrate. *Biomass Bioenergy* **2015**, *80*, 243–251, doi:10.1016/j.biombioe.2015.05.019.
56. Yang, X.; Shang, C.; Lee, W.; Westerhoff, P.; Fan, C. Correlations between organic matter properties and DBP formation during chloramination. *Water Res.* **2008**, *42*, 2329–2339, doi:10.1016/j.watres.2007.12.021.
57. Bieroza, M.Z.; Bridgeman, J.; Baker, A. Fluorescence spectroscopy as a tool for determination of organic matter removal efficiency at water treatment works. *Drink. Water Eng. Sci.* **2010**, *3*, 63–70, doi:10.5194/dwes-3-63-2010.